AUTHENTICATED
U.S. GOVERNMENT
INFORMATION
GPO

10-26-84
Vol. 49 No. 209
Pages 43035-43442

# Federal Register

Friday
October 26, 1984

## Selected Subjects

**Air Pollution Control**
Environmental Protection Agency

**Animal Drugs**
Food and Drug Administration

**Aviation Safety**
Federal Aviation Administration

**Chemicals**
Environmental Protection Agency

**Endangered and Threatened Species**
Fish and Wildlife Service

**Food Additives**
Food and Drug Administration

**Grant Programs—Education**
Education Department

**Hazardous Waste**
Environmental Protection Agency

**Household Appliances**
Federal Trade Commission

**Income Taxes**
Internal Revenue Service

**Livestock**
Agricultural Marketing Service

**Loan Programs—Housing and Community Development**
Veterans Administration

CONTINUED INSIDE

**FEDERAL REGISTER** Published daily, Monday through Friday, (not published on Saturdays, Sundays, or on official holidays), by the Office of the Federal Register, National Archives and Records Service, General Services Administration, Washington, DC 20408, under the Federal Register Act (49 Stat. 500, as amended; 44 U.S.C. Ch. 15) and the regulations of the Administrative Committee of the Federal Register (1 CFR Ch. I). Distribution is made only by the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

The **Federal Register** provides a uniform system for making available to the public regulations and legal notices issued by Federal agencies. These include Presidential proclamations and Executive Orders and Federal agency documents having general applicability and legal effect, documents required to be published by act of Congress and other Federal agency documents of public interest. Documents are on file for public inspection in the Office of the Federal Register the day before they are published, unless earlier filing is requested by the issuing agency.

The **Federal Register** will be furnished by mail to subscribers for $300.00 per year, or $150.00 for 6 months, payable in advance. The charge for individual copies is $1.50 for each issue, or $1.50 for each group of pages as actually bound. Remit check or money order, made payable to the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

There are no restrictions on the republication of material appearing in the **Federal Register.**

Questions and requests for specific information may be directed to the telephone numbers listed under INFORMATION AND ASSISTANCE in the READER AIDS section of this issue.

# Selected Subjects

**Marketing Agreements**
Agricultural Marketing Service

**Savings and Loan Associations**
Federal Home Loan Bank Board

**Surface Mining**
Surface Mining Reclamation and Enforcement Office

**Vessels**
Customs Service

**Water Pollution Control**
Environmental Protection Agency

**Wine**
Alcohol, Tobacco and Firearms Bureau

# Contents

Federal Register
Vol. 49, No. 209
Friday, October 26, 1984

**Agricultural Marketing Service**
RULES
43039 Lemons grown in Arizona and California
Livestock; grading, certification, and standards:
43055 Slaughter lambs, yearlings, and sheep

**Agriculture Department**
*See* Agricultural Marketing Service.

**Alcohol, Tobacco and Firearms Bureau**
RULES
Alcohol; viticultural area designations:
43053 Sonoita, Arizona

**Army Department**
*See also* Engineers Corps.
NOTICES
43089 Agency information collection activities under OMB review

**Blind and Other Severely Handicapped, Committee for Purchase from**
RULES
43065 Freedom of Information Act and Privacy Act; change of address; correction
NOTICES
43087 Procurement list, 1984; additions and deletions; correction
43087 Procurement list, 1985; additions and deletions (2 documents)

**Civil Rights Commission**
NOTICES
Meetings; State advisory committees:
43078 Alaska
43078 Delaware

**Commerce Department**
*See* International Trade Administration; National Oceanic and Atmospheric Administration.

**Commodity Futures Trading Commission**
RULES
Domestic exchange-traded commodity options:
43048 Options traders; commercial categories

**Customs Service**
RULES
Vessels in foreign and domestic trades:
43049 Denmark; pleasure vessels licensing list

**Defense Department**
*See also* Army Department; Engineers Corps; Navy Department.
NOTICES
43089 Agency information collection activities under OMB review
Federally funded research and development centers:
43088 Logistics Management Institute
Meetings:
43088 DIA Scientific Advisory Committee
43088 Electron Devices Advisory Group (2 documents)

**Economic Regulatory Administration**
NOTICES
Natural gas exportation and importation petitions:
43091–43094 Northwest Alaskan Pipeline Co. (2 documents)

**Education Department**
RULES
Educational research and improvement:
43226 Discretionary program
NOTICES
Grants; availability, etc.:
43090 Handicapped special studies program

**Employment Standards Administration**
NOTICES
43170 Minimum wages for Federal and federally-assisted construction; general wage determination decisions, modifications, and supersedeas decisions (CA, CT, IL, KS, MA, MI, MO, OH, OK, OR, RI, TX, VA, WV)

**Energy Department**
*See* Economic Regulatory Administration; Federal Energy Regulatory Commission.

**Engineers Corps**
NOTICES
Environmental statements; availability, etc.:
43090 Oahu, HI

**Environmental Protection Agency**
RULES
Air programs; approval and promulgation; State plans for designated facilities and pollutants:
43057 Iowa, Kansas, and Nebraska
Air quality implementation plans:
43202 Preparation, adoption and submittal; Fugitive emissions in new source review calculations
Toxic substances:
43058 1,2-Benzenediamine, 4-ethoxy, sulfate; significant new uses
43061 Dicarboxylic acid monoester; significant new uses
Water pollution control:
43234 Pollutants analysis test procedures; guidelines; final rule and interim rule with request for comments
PROPOSED RULES
Air quality implementation plans:
43211 Preparation, adoption and submittal; Fugitive emissions, surface coal mines, etc.
Air quality implementation plans; approval and promulgation; various States:
43072 Illinois; extension of time
43072 Kentucky and Ohio; extension of time
Hazardous waste program authorizations:
43072 Maryland
Water pollution control:
43437 Pollutants analysis test procedures; guidelines

NOTICES
Air quality; prevention of significant deterioration (PSD):
43107 Permit approvals
43108 Permit extensions
Environmental statements; availability, etc.:
43108 Agency statements; review and comment
43108 Agency statements; weekly receipts
Toxic and hazardous substances control:
43105 Premanufacture notices receipts

**Federal Aviation Administration**
RULES
Airworthiness directives:
43046 Hughes Helicopters, Inc.
Airworthiness standards:
43168 Aircraft seat cushions; flammability requirements
43182 Floor proximity emergency escape path marking

**Federal Emergency Management Agency**
PROPOSED RULES
Flood elevation determinations:
43074 Alabama et al.; correction

**Federal Energy Regulatory Commission**
NOTICES
Hearings, etc.:
43096 Arkansas Louisiana Gas Co.
43097 Arkansas Power & Light Co.
43097, 43098 Columbia Gas Transmission Corp. (3 documents)
43098 National Fuel Gas Supply Corp.
43098 Northern Indiana Public Service Co.
43099 Northern Natural Gas Co. (2 documents)
43100 Northwest Pipeline Corp.
43100 Ozark Gas Transmission System
43100, 43102 Panhandle Eastern Pipe Line Co. (3 documents)
43102 Texas Eastern Transmission Corp.
43102 Trunkline Gas Co.
43103 United Gas Pipe Line Co. (3 documents)
43104 Western Gas Interstate Co.
43145 Meetings; Sunshine Act
Small power production and cogeneration facilities; qualifying status; certification applications, etc.:
43104 OLS Energy-Agnews
43105 Turbine Tech, Inc.

**Federal Home Loan Bank Board**
RULES
Federal associations:
43040 Implementation of new powers; limitation on loans to one borrower; final rule and request for comments

**Federal Maritime Commission**
NOTICES
43109 Agreements filed, etc.
Shipping Act of 1984:
43109 Controlled carriers listing; update

**Federal Reserve System**
NOTICES
Bank holding company applications, etc.:
43110 Central Banc Holding, Inc., et al.
43110 Mansura Bancshares, Inc., et al.
43145 Meetings; Sunshine Act (3 documents)

**Federal Trade Commission**
RULES
Appliances, consumer; energy cost and consumption information in labeling and advertising:
43047 Room air conditioners; comparability ranges

**Fish and Wildlife Service**
RULES
Endangered and threatened species:
43065 Smoky madtom
PROPOSED RULES
Endangered and threatened species:
43076 San Mateo thornmint and Bay Checkerspot butterfly; hearing, extension of time, etc.
NOTICES
43118 Endangered and threatened species permit applications

**Food and Drug Administration**
RULES
Animal drugs, feeds, and related products:
43051 Cefadroxil tablets
Food for human consumption:
43050 Polymers; perfluorocarbon cured elastomers
NOTICES
Food additive petitions:
43111 Eastman Kodak Co.
43111 ICI Americas, Inc.

**Health and Human Services Department**
*See also* Food and Drug Administration; Public Health Service.
PROPOSED RULES
43074 Nondiscrimination on basis of age in federally assisted programs or activities; evaluation
NOTICES
43110 Agency information collection activities under OMB review

**Interior Department**
*See* Fish and Wildlife Service; Land Management Bureau; National Park Service; Surface Mining Reclamation and Enforcement Office.

**Internal Revenue Service**
RULES
Income taxes:
43052 Diversification requirements for variable annuity, endowment, and life insurance contracts; effective date; temporary
NOTICES
Meetings:
43143 Art Advisory Panel

**International Trade Administration**
NOTICES
Antidumping:
43078 Animal glue and inedible gelatin from Yugoslavia
43079 Elemental sulphur from Mexico
43080 Cell site transceivers from Japan
Countervailing duties:
43079 Chain of iron or steel from Japan
Scientific articles; duty free entry:
43086 Arizona State University et al.
43085 Medical College of Wisconsin
43085 North Carolina State University

43086 University of Colorado
43087 University of Chicago; correction
43086 University of Wisconsin-Parkside
43086 Virginia Polytechnic Institute and State University et al.

**International Trade Commission**
NOTICES
Antidumping:
43120 Cut-to-length carbon steel plate from West Germany

**Interstate Commerce Commission**
RULES
Practice and procedure:
43065 Fees for licensing and related services; correction
NOTICES
Motor carriers:
43121 Compensated intercorporate hauling operations; intent to engage in
Railroad operation, acquisition, construction, etc.:
43121 Chesapeake & Ohio Railway Co.
43122 Northwestern Pacific Acquiring Corp. et al.
Railroad services abandonment:
43121 Burlington Northern Railroad Co.

**Justice Department**
NOTICES
43122 Agency information collection activities under OMB review

**Labor Department**
*See also* Employment Standards Administration; Pension and Welfare Benefit Programs Office.
NOTICES
43123 Agency information collection activities under OMB review

**Land Management Bureau**
NOTICES
Environmental statements; availability, etc.:
43117 Platte River Resource Area, WY
43116 Walker Resource Area, NV
Management framework plans:
43116 California
Resource management plans:
43118 Grand Junction Resource Area, CO
Survey plat filings:
43118 Iowa (2 documents)

**National Oceanic and Atmospheric Administration**
PROPOSED RULES
Fishery conservation and management:
43076 Atlantic swordfish fishery management plan, hearing

**National Park Service**
NOTICES
Land protection plans; availability, etc.:
43119 Pictured Rocks National Lakeshore, MI
Meetings:
43119 Gates of the Arctic National Park Subsistence Resource Commission

**National Science Foundation**
NOTICES
Meetings:
43135 Social/Cultural Anthropology Advisory Panel

**Navy Department**
NOTICES
43089 Privacy Act; systems of records

**Nuclear Regulatory Commission**
PROPOSED RULES
Rulemaking petitions:
43070 Nantz, John L.
NOTICES
Environmental statements; availability, etc.:
43136 Duke Power Co.
43138 Three Mile Island Unit 2 cleanup
Meetings:
43135, 43136 Reactor Safeguards Advisory Committee (2 documents)

**Pension and Welfare Benefit Programs Office**
NOTICES
Employee benefit plans; prohibited transaction exemptions:
43123 Alaska Mutual Bank et al.
43126 J.H. Kleinfelder & Associates et al.

**Postal Service**
NOTICES
43145 Meetings; Sunshine Act

**Public Health Service**
NOTICES
Grants; availability, etc.:
43112 Adolescent family life demonstration projects
Medical technology scientific evaluations:
43115 Ambulatory esophageal pH monitoring
43115 Bone mineral density studies
43111 Cardiokymography in diagnosis of coronary artery disease

**Securities and Exchange Commission**
NOTICES
Hearings, etc.:
43139 Creditanstalt-Bankverein et al.
43142 Tri-Comp Sensors, Inc.
Self-regulatory organizations; proposed rule changes:
43140 Pacific Securities Depository Trust Co.

**Small Business Administration**
NOTICES
Disaster loan areas:
43142 California
43142 Oregon
43143 Tennessee
43143 Washington
Meetings; regional advisory councils:
43143 Hawaii

**Surface Mining Reclamation and Enforcement Office**
RULES
Permanent program submission; various States:
43055 Missouri
PROPOSED RULES
Permanent program submission; various States:
43071 Montana; comment period reopened

**Tennessee Valley Authority**
NOTICES
43146 Meetings; Sunshine Act

**Trade Representative, Office of United States**
NOTICES
Import quotas and exclusions, etc.:
43139 Amorphous metal articles and alloys; inquiry

**Transportation Department**
*See* Federal Aviation Administration; Urban Mass Transportation Administration.

**Treasury Department**
*See* Alcohol, Tobacco and Firearms Bureau; Customs Service; Internal Revenue Service.
NOTICES
43143 Agency information collection activities under OMB review

**Urban Mass Transportation Administration**
NOTICES
Grants; availability, etc.:
43218 Federal Public Transportation Act; funds apportionment

**Veterans Administration**
RULES
Loan guaranty:
43055 Interest rates

---

**Separate Parts in This Issue**

**Part II**
43170 Department of Labor, Employment Standards Administration, Wage and Hour Division

**Part III**
43182 Department of Transportation, Federal Aviation Administration

**Part IV**
43188 Department of Transportation, Federal Aviation Administration

**Part V**
43202 Environmental Protection Agency

**Part VI**
43218 Department of Transportation, Urban Mass Transportation Administration

**Part VII**
43226 Department of Education

**Part VIII**
43234 Environmental Protection Agency

---

**Reader Aids**
Additional information, including a list of public laws, telephone numbers, and funding aids, appears in the Reader Aids section at the end of this issue.

## CFR PARTS AFFECTED IN THIS ISSUE

A cumulative list of the parts affected this month can be found in the Reader Aids section at the end of this issue.


**7 CFR**
53 ..... 43035
910 ..... 43039

**10 CFR**
**Proposed Rules:**
7 ..... 43070

**12 CFR**
523 ..... 43040
541 ..... 43040
545 ..... 43040
549 ..... 43040
561 ..... 43040
563 ..... 43040
584 ..... 43040

**14 CFR**
25 (2 documents) ..... 43182, 43188
29 ..... 43188
39 ..... 43046
121 (2 documents) ..... 43182, 43188

**16 CFR**
305 ..... 43047

**17 CFR**
1 ..... 43048
16 ..... 43048

**19 CFR**
4 ..... 43049

**21 CFR**
177 ..... 43050
520 ..... 43051

**26 CFR**
1 ..... 43052

**27 CFR**
9 ..... 43053

**30 CFR**
925 ..... 43055
**Proposed Rules:**
926 ..... 43071

**34 CFR**
760 ..... 43226

**38 CFR**
36 ..... 43055

**40 CFR**
51 ..... 43202
52 ..... 43202
62 ..... 43057
136 ..... 43234
721 (2 documents) ..... 43058–43061
**Proposed Rules:**
51 ..... 43211
52 (3 documents) ..... 43072, 43211
136 ..... 43437
271 ..... 43072

**41 CFR**
51–7 ..... 43065
51–8 ..... 43065

**44 CFR**
**Proposed Rules:**
67 ..... 43074

**45 CFR**
**Proposed Rules:**
90 ..... 43074

**49 CFR**
1002 ..... 43065
1011 ..... 43065
1152 ..... 43065
1177 ..... 43065
1180 ..... 43065
1182 ..... 43065

**50 CFR**
17 ..... 43065
**Proposed Rules:**
17 ..... 43076
630 ..... 43076

# Rules and Regulations

Federal Register
Vol. 49, No. 209
Friday, October 26, 1984

This section of the FEDERAL REGISTER contains regulatory documents having general applicability and legal effect, most of which are keyed to and codified in the Code of Federal Regulations, which is published under 50 titles pursuant to 44 U.S.C. 1510.
The Code of Federal Regulations is sold by the Superintendent of Documents. Prices of new books are listed in the first FEDERAL REGISTER issue of each week.

## DEPARTMENT OF AGRICULTURE

### Agricultural Marketing Service

### 7 CFR Part 53

### Standards for Grades of Slaughter Lambs, Yearlings, and Sheep

**AGENCY:** Agricultural Marketing Service (AMS), USDA.

**ACTION:** Final rule.

**SUMMARY:** This rule revises the official U.S. standards for grades of slaughter lambs, yearlings, and sheep to align them with the previously revised standards for their respective carcasses. A final rule to revise the official standards for grades of lamb, yearling mutton, and mutton carcasses was published in the **Federal Register** on September 13, 1982, 47 FR 40141. Since all of the changes to the carcass standards are not discernible in live animals, the current quality requirements for slaughter lambs, yearlings, and sheep did not require revision. However, consistent with the other carcass changes, this rule will standardize the quality and conformation compensations, add descriptions of degrees of muscling associated with each grade to the conformation descriptions, and eliminate the Cull grade for slaughter lambs and yearlings. The changes will make the live animal standards consistent with the carcass standards where possible.

**EFFECTIVE DATE:** November 26, 1984.

**FOR FURTHER INFORMATION CONTACT:** Dr. Michael L. May; Chief; Standardization and Review Branch; Livestock Division; Agricultural Marketing Service; U.S. Department of Agriculture; Washington, D.C., 20250, (202) 447–4486.

**SUPPLEMENTARY INFORMATION:** This action was reviewed under USDA procedures established to implement Executive Order 12291 and was classified as a non-major rule pursuant to sections 1(b) (1), (2), and (3) of that order because (1) it will not have an annual effect on the economy of $100 million or more, (2) it will not result in a major increase in costs or prices for consumers, individual industries, Federal, State, or local government agencies, or geographic regions; and (3) it will not have significant adverse effects on competition, employment, investment, productivity, innovation, or on the ability of United States-based enterprises to compete with foreign-based enterprises in domestic or export markets. These changes will not significantly affect the number of lambs, yearlings or sheep in any grade.

William T. Manley, Deputy Administrator, Agricultural Marketing Service, certified that this rule will not have a significant economic impact on a substantial number of small entities because the changes merely simplify the teaching and application of the quality grade standards without significantly affecting the number of lambs in any grade.

#### Background

Tentative U.S. standards for the market classes and grades of slaughter ovine (lambs, yearling, and sheep) were prepared in 1917 to provide a basis on which the Federal Market News Service might issue market quotations on these animals according to a uniform classification. Revisions were made in these standards in 1936, 1940, 1951, 1957, and 1960. Yield grades were adopted in 1969. Generally, revisions through the years in the quality grade standards for lambs have been as this rule is intended—to coordinate them with changes already made in the carcass standards. These standards have been used by the Department as the basis for market reporting of lamb and sheep prices and are also used by States which conduct official lamb grading programs.

The proposal to revise the official standards was published in the **Federal Register** on February 21, 1984 (Vol. 49, No. 35, pages 6378–6382), and comments were invited for 62 days ending on April 23, 1984. A total of three comments were received in response to the proposal. Two comments were from agricultural organizations and one was from a State Department of Agriculture and Markets.

All comments expressed support for the overall intent of the proposal with specific support for standardizing quality and conformation compensations which make the standards more understandable. After a review and analysis of the comments and all information available, the proposed revisions are finalized without change.

The quality grade standards for slaughter ovine are based on a combination of conformation and quality. Under this rule the conformation requirements would be unchanged, except that descriptions for various degrees of muscling associated with each grade are added. However, the rate of compensation of quality for conformation and conformation for quality is modified in the Choice, Good and Utility grades. In the present grades, compensation is on an equal basis in some cases, and in other cases a half grade of superior conformation or quality is needed to compensate for a third of a grade of inferior quality or conformation. Since the rate of compensation for both quality and conformation are different in different grades, the situation is often confusing. Teaching these compensations to new graders and others is especially difficult. Under these standards all compensations of conformation for quality and quality for conformation are on an equal basis. The limits and extent of compensations are not changed. These changes conform the live animal standards to the carcass standards (7 CFR Part 54, 47 FR 40141).

Because of the limited number of young ovine which fail to qualify for the Good or higher grades and because of the limited variation among such ovine, it was determined that four quality grades are sufficient for young ovine. Therefore, the Cull grade in §§ 53.132(a), 53.133(e) and 53.134(e) has been dropped for the lamb and yearling classes, and all lambs and yearlings which fail to qualify for Good are graded Utility. The Cull grade is kept for the sheep class. This change conforms the slaughter standards with the carcass standards.

Previous standards have combined the quality grade specifications for slaughter yearlings and sheep into one section. However, in the carcass standards, yearling mutton and mutton

quality grade specifications are contained in separate sections. For greater consistency with the carcass standards, the corresponding slaughter yearling and sheep specifications in § 53.134 are split into separate sections, §§ 53.134 and 53.135. Previous § 53.135 is renumbered as 53.136.

In summary, the changes involved in this revision of the standards for grades of slaughter lambs, yearlings, and sheep provide for the following:

(1) Descriptions of degrees of muscling have been added to the conformation requirements for each grade.

(2) The rate of compensation of conformation for quality and quality for conformation has been changed to be on an equal basis in all grades.

(3) In 7 CFR 53.132(a), 53.133(e) and 53.134(e) the Cull grade has been dropped for the slaughter lamb and yearling classes.

(4) The quality grade specifications for slaughter yearlings and sheep have been split into separate sections and the yield grade specification section has been renumbered to allow space for the new section.

Minor non-substantive changes also have been made throughout the standards to improve clarity and facilitate uniform interpretation.

**List of Subjects in 7 CFR Part 53**

Livestock (Grading, Certification, and Standards), Lamb.

## PART 53—LIVESTOCK (GRADING, CERTIFICATION, AND STANDARDS)

For the reasons set out in the preamble, the official U.S. standards for grades of slaughter lambs, yearlings, and sheep in 7 CFR Part 53 are amended by redesignating § 53.135 as § 53.136, adding a new § 53.135, and revising §§ 53.132, 53.133, and 53.134, as follows:

1. The authority citation for Part 53 reads as follows:

**Authority:** Agricultural Marketing Act of 1946, sec. 203, 205, as amended; 60 Stat. 1087, 1090, as amended (7 U.S.C. 1622 and 1624).

2. Section 53.135 would be redesignated as § 53.136, a new § 53.135 would be added, and §§ 53.132 through 53.134 would be revised to read as follows:

### § 53.132 Application of standards.

(a) *Grade factors.* Grades of slaughter ovine are intended to be directly related to the grades of the carcasses they produce. To accomplish this, these slaughter ovine grade standards are based on factors which are directly related to the quality grades and the yield grades of ovine carcasses. The standards are written so that the quality and yield grade standards are contained in separate sections. The quality grade standards are divided into three sections applicable to slaughter lambs, slaughter yearlings, and slaughter sheep. There are four quality grades within each class—Prime, Choice, Good, and Utility for lambs and yearlings; and Choice, Good, Utility, and Cull for sheep. Also, there are five yield grades applicable to all classes of slaughter ovine, denoted by numbers 1 through 5, with Yield Grade 1 representing the highest degree of cutability.

(b) *General princples.* (1) The determination of the carcass grade that the slaughter animal will produce requires the exercise of well-regulated judgment. Each animal presents a different combination of the grade-determining factors. Animals frequently have characteristics associated with two or more grades. Therefore, a composite evaluation of all inherent physical characteristics is essential for accuracy in determining grade.

(2) The accurate determination of the grade of a slaughter ovine requires handling in addition to visual observation. The length and density of the fleece vary greatly with individuals and the thickness and firmness of the flesh covering of wooled ovine can only be roughly estimated without handling. The technique used in handling usually varies with the degree of precision in mind as well as the experience of the grader. Experienced graders may find one quick handling satisfactory. This usually consists of placing one open hand over the back and ribs in simultaneous motion. The thumb extends just over the backbone, while the fingers, which are held close together, cover the rib section and pressure is applied very lightly with a slight lateral and forward and backward motion. The generally accepted technique of handling ovine where time permits, and especially when noting slight differences between individuals, is to handle forward from the dock to neck with the open hand, fingers together, laid flat and with a slight lateral motion. Both hands may then be used, one on each side, in a similar manner to determine the fleshing over the shoulders, ribs, and hips. Regardless of the method, considerable experience is necessary in handling ovine to accurately determine the grade.

(c) *Quality grades.* (1) The quality grade of a slaughter ovine is determined by a composite evaluation of two general considerations which influence carcass excellence: Conformation and quality—fatness, maturity, and other indicators of differences in palatability of the lean flesh.

(2) Conformation refers to the general body proportions of the animal and to the ratio of meat to bone. Although primarily determined by the inherent muscular and skeletal systems, it is also influenced by the degree of fatness. However, external fat in excess of that normally left on retail cuts is not considered in evaluating conformation. The conformation descriptions included in each of the grade specifications refer to the thickness of muscling and to an overall degree of thickness and fullness of the animal. Slaughter ovine which meet the requirements for thickness of muscling specified for a grade will be considered to have conformation adequate for that grade despite the fact that, because of a lack of fatness, they may not have the overall degree of thickness and fullness described. Conformation is evaluated by averaging the conformation of the various component parts, giving special consideration to those parts of the body producing the more desirable cuts of meat—loin, hotel rack, and leg.

(3) In grading slaughter ovine, quaity of the lean must be evaluated indirectly by considering the quantity, distribution, and type of fat or finish in relation to the maturity of the animal being graded. Limited consideration is also given to such factors as character of bone and smoothness and symmetry of body. Finish is evaluated by noting variations in the fullness and apparent thickness of the fat covering over the back, loin, ribs, and legs. A high degree of desriable finish is evidenced by a firm, smooth layer of fat which is uniformly distributed over the body.

(4) Although the market designation of slaughter ovine is usually made by classes, the quality standards are intended to apply to all classes without regard to sex condition. However, male animals which have thick heavy necks and shoulders typical of uncastrated males are discounted in grade in proportion to the extent to which these characteristics are developed. Such discounts may vary from less than half a grade in young lambs in which such characteristics are barely noticeable, to as much as two full grades in mature rams in which such characteristics are very pronounced.

(d) *Yield grades.* (1) The yield grades for slaughter ovine are based on the same factors used in the official yield grade standards for ovine carcasses. These factors are as follows:

(i) *Thickness of fat over ribeye.* As the amount of external fat increases, the percent of retail cuts decreases and the numerical yield grade increases. Assuming no change in the other factors,

each 0.15 inch change in adjusted fat thickness over the ribeye changes the yield grade by a full grade. On slaughter ovine which do not have a normal distribution of external fat, the fat thickness estimate over the ribeye may be adjusted, as necessary, to reflect unusual amounts of fat on other parts of the animal.

(ii) *Percent of kidney and pelvic fat.* As the amount of these fats increases, the percent of retail cuts decreases. A change of 4 percent of the carcass weight in kidney and pelvic fat changes the yield grade by a full grade.

(iii) *Leg conformation grade.* An increase in the conformation grade of the legs increases the percent of retail cuts. A change of two full grades in conformation of the legs changes the yield grade by approximately one-third of a yield grade.

(2) When evaluating slaughter ovine for yield grade, each of these factors can be estimated and the yield grade determined therefrom by using the equation contained in the official standards for yield grades of lamb, yearling mutton, and mutton carcasses. However, a more practical method of appraising slaughter ovine for yield grade is to use only two factors normally considered in evaluating live ovine—degree of fatness and leg conformation. In this approach, the degree of fatness largely accounts for the effects of thickness of fat over the ribeye and the percent of kidney and pelvic fat.

(3) The overall fatness of an animal can be determined best by giving particular attention to those parts on which fat is deposited at a faster-than-average rate. These include the back, loin, rump, flank, breast, and cod or udder. As ovine increase in fatness, these parts become progressively fuller, thicker, and more distended in relation to the thickness and fullness of the other parts, particularly the legs. However, since an animal's thickness of muscling also affects the development of its various parts, this also needs to be considered when evaluating the degree of fatness. In thinly muscled ovine with a low degree of finish, the width of the back usually will be greater than the width through the center of the legs. Conversely, in thickly muscled ovine with a low degree of finish, the thickness through the legs will be greater than through the back and the back will be full and rounded. At an intermediate degree of fatness, ovine which are thinly muscled will be considerably wider through the back than through the leg and will be nearly flat across the back. Thickly muscled ovine that have an intermediate degree of fatness will be about the same width through the legs as through the back and the back will appear only slightly rounded. Very fat ovine will be wider through the back than through the legs, but this difference will be greater in thinly muscled ovine than in those that are thickly muscled. As ovine increase in fatness, they also become deeper bodied because of large deposits of fat in the flanks and breast and along the underline. In determining yield grade, variations in fatness are of very much greater importance than variations in conformation of the leg.

(e) *Other considerations.* (1) Other factors such as sex, heredity, and management also may affect the development of grade-determining characteristics in slaughter ovine. Although these factors do not lend themselves to descriptions in the standards, the use of factual information of this nature is justified in determining the grade of slaughter ovine. The ability to make proper allowances for the effects of genetic and management factors on the appearance of grade-determining characteristics must be developed through experience.

(2) Slaughter ovine qualifying for any particular grade may vary with respect to the relative development of their individual grade factors. In fact, some will qualify for a particular grade although they have some characteristics more typical of ovine of another grade. Because it is impractical to describe the nearly infinite number of such recognizable combinations of characteristics, the standards describe only ovine which have a relatively similar development of the various quality and yield grade-determining factors and which are near the lower limits of quality or yield for the grade. However, examples of the extent to which superiority in quality-indicating characteristics may compensate for deficiencies in conformation, and vice versa, are indicated for each quality grade. In the slaughter lamb quality grade standards, the requirements are given for two maturity groups. In the yield grade standards, ovine with two levels of muscling are described and specific examples in terms of carcass characteristics also are included.

**§ 53.133 Specifications for Official U.S. Standards for Grades of Slaughter Lambs (Quality).**

(a) *Prime.* (1) Slaughter lambs having minimum conformation requirements for the Prime grade tend to be thickly muscled throughout, and they are moderately wide and thick in relation to their length and height. The are moderately wide over the back, loin, and rump. Shoulders and hips are usually moderately smooth. The twist is moderately deep and full, and the legs are moderately large and plump. They generally present a well-rounded appearance due to a slight fullness or plumpness over the crops, loins, and rump. Relatively young lambs, under seven months of age, tend to have a moderately thin fat covering over the back, ribs, loins, and rump. In handling, the backbone and ribs are readily discernible. Older, more mature lambs have a slightly thin fat covering over the back, ribs, loin, and rump. In handling, the backbone and ribs are slightly discernible. Prime grade lambs exhibit evidences of rather high quality. The bones tend to be proportionately small, the joints tend to be smooth, and the body tends to be smooth and symmetrical.

(2) To qualify for the Prime grade, a lamb must possess the minimum qualifications for finish regardless of the extent that its conformation may exceed the minimum requirements for Prime. However, a development of finish which is superior to that specified as minimum for the Prime grade may compensate, on an equal basis, for a development of conformation which is inferior to that specified for Prime as indicated in the following example: A lamb which has evidences of finish equivalent to the midpoint of the Prime grade may have conformation equivalent to the midpoint of the Choice grade and remain eligible for Prime. However, in no instance may a lamb be graded Prime which has a development of conformation inferior to that specified as minimum for the Choice grade.

(b) *Choice.* (1) Slaughter lambs having minimum conformation requirements for the Choice grade are slightly thick muscled throughout, and they tend to be slightly wide and thick in relation to their length and height. They tend to be slightly wide over the back, loin, and rump. The shoulders and hips are usually slightly smooth but may exhibit a slight tendency toward prominence. The twist tends to be slightly deep and full, and the legs tend to be slightly thick and plump. Relatively young lambs, under seven months of age, have a thin fat covering over the back, ribs, loin, and rump. In handling, the backbone and ribs are moderately prominent. Older, mature lambs have a moderately thin fat covering over the back, ribs, loin, and rump. In handling, the backbone and ribs are slightly prominent. Choice grade lambs usually present a moderately refined appearance.

(2) A development of quality which is superior to that specified as minimum for the Choice grade may compensate, on a equal basis, for a development of conformation which is inferior to that specified as minimum for Choice as indicated in the following example: A lamb which has evidences of quality equivalent to the midpoint of the Choice grade may have conformation equivalent to the midpoint of the Good grade and remain eligible for Choice. However, in no instance may a lamb be graded Choice which has a development of conformation inferior to that specified as minimum for the Good grade. Also, a lamb which has conformation at least one-third grade superior to that specified as minimum for the Choice grade may qualify for Choice with a development of quality equivalent to the lower limit of the upper one-third of the Good grade. Compensation of superior conformation for inferior quality is limited to one-third grade of deficient quality.

(c) *Good.* (1) Slaughter lambs having minimum conformation requirements for the Good grade are slightly thin muscled throughout, and are moderately narrow in relation to their length and height and are slightly narrow over the back, loin, and rump. Hips and shoulders are moderately prominent. The twist is slightly shallow and the legs are slightly small and thin. Relatively young lambs, under seven months of age, have slightly more than a very thin, uneven fat covering over the back, loin, and upper ribs. In handling, the shoulders, backbone, hips, and ribs are prominent. Older, more mature lambs have slightly more than a thin fat covering over the back, ribs, and loin. In handling, the bones of the shoulders, backbone, hips, and ribs are rather prominent. Good grade lambs may present evidences of slightly low quality. The bones and joints are usually moderately large, and the body is somewhat lacking in symmetry and smoothness.

(2) A development of quality which is superior to that specified as minimum for the Good grade may compensate, on an equal basis, for a development of conformation which is inferior to that specified as minimum for Good as indicated in the following example: A lamb which has evidences of quality at least one-third grade superior to that specified as minimum for the Good grade may have conformation equivalent to the minimum for the upper one-third of the Utility grade and remain eligible for Good. However, in no instance may a lamb be graded Good which has a development of conformation inferior to the minimum for the upper one-third of the Utility grade. Also, a lamb which has conformation at least one-third grade superior to that specified as minimum for the Good grade may qualify for Good with a development of quality equivalent to the lower limit of the upper one-third of the Utility grade. Compensation of superior conformation for inferior quality is also limited to one-third grade of deficient quality.

(d) *Utility.* The Utility grade consists of those lambs whose characteristics are inferior to those specified as minimum for the Good grade.

**§ 53.134 Specifications for Official U.S. Standards for Grades of Slaughter Yearlings (Quality).**

(a) *Prime.* (1) Slaughter yearlings having minimum conformation requirements for the Prime grade tend to be thickly muscled throughout, and are moderately wide and thick in relation to their length and height, and they are moderately wide over the back, loin, and rump. Shoulders and hips are usually moderately smooth. The twist is moderately deep and full, and the legs are moderately large and plump. There is a rather distinct fullness or plumpness evident over the crops, loins, and rump which contributes to a well-rounded appearance. There is a slightly thick fat covering over the back, ribs, loin, and rump. In handling, the backbone and ribs are hardly discernible. Prime grade slaughter yearlings exhibit evidences of rather high quality. The bones tend to be proportionately small, the joints tend to be smooth, and the body tends to be smooth and symmetrical.

(2) Regardless of the extent to which its conformation may exceed the minimum requirements for Prime, a yearling must have minimum Prime quality to be eligible for the Prime grade. However, a development of quality which is superior to that specified as minimum for the Prime grade may compensate, on an equal basis, for a development of conformation which is inferior to that specified as minimum for Prime as indicated in the following example: A yearling which has evidences of quality equivalent to the midpoint of the Prime grade may have conformation equivalent to the midpoint of the Choice grade and remain eligible for Prime. However, in no instance may a yearling be graded Prime which has a development of conformation inferior to that specified as minimum for the Choice grade.

(b) *Choice.* (1) Slaughter yearlings having minimum conformation requirements for the Choice grade are slightly thick muscled throughout, and they tend to be slightly wide and thick in relation to their length and height, and they tend to be slightly wide over the back, loin, and rump. The shoulders and hips are usually slightly smooth but may show a slight tendency toward prominence. The twist tends to be slightly deep and full, and the legs tend to be slightly thick and plump. They have a slightly thin fat covering over the back, ribs, loin, and rump. In handling, the backbone and ribs are readily discernible. Choice grade slaughter yearlings usually present a moderately refined appearance.

(2) A development of quality which is superior to that specified as minimum for the Choice grade may compensate, on an equal basis, for a development of conformation which is inferior to that specified as minimum for Choice as indicated in the following example: A yearling which has evidences of quality equivalent to the midpoint of the Choice grade may have conformation equivalent to the midpoint of the Good grade and remain eligible for Choice. However, in no instance may a yearling be graded Choice which has a development of conformation inferior to that specified as minimum for the Good grade. Also, a yearling which has a development of conformation at least one-third grade superior to that specified as minimum for the Choice grade may qualify for Choice with a development of quality equivalent to the lower limit of the upper one-third of the Good grade. Compensation of superior conformation for inferior quality is limited to one-third grade of deficient quality.

(c) *Good.* (1) Slaughter yearlings having minimum conformation requirements for the Good grade are slightly thin muscled throughout, and are moderately narrow in relation to their length and height, and are slightly narrow over the back, loin, and rump. Hips and shoulders are moderately prominent. The twist is slightly shallow, and the legs are slightly small and thin. They have slightly more than a moderately thin fat covering over the back, loin, and upper ribs. In handling, the shoulders, backbone, hips, and ribs are rather prominent. Good grade yearlings may present evidences of slightly low quality. The body is somewhat lacking in symmetry and smoothness.

(2) A development of quality which is superior to that specified as minimum for the Good grade may compensate, on an equal basis, for a development of conformation which is inferior to that specified as minimum for Good as indicated in the following example: A yearling which has evidences of quality

at least one-third grade superior to that specified as minimum for the Good grade may have conformation equivalent to the minimum for the upper one-third of the Utility grade and remain eligible for Good. However, in no instance may a yearling be graded Good which has a development of conformation inferior to the minimum for the upper one-third of the Utility grade. Also, a yearling which has conformation at least one-third grade superior to that specified as minimum for the Good grade may qualify for Good with a development of quality equivalent to the lower limit of the upper one-third of the Utility grade. Compensation of superior conformation for inferior quality is also limited to one-third grade of deficient quality.

(d) *Utility.* The Utility grade includes those yearlings whose characteristics are inferior to those specified as minimum for the Good grade.

**§ 53.135 Specifications for Official U.S. Standards for Grades of Slaughter Sheep (Quality).**

(a) *Choice.* (1) Slaughter sheep having minimum conformation requirements for the Choice grade are slightly thick muscled throughout, and they tend to be slightly wide and thick in relation to their length and height, and they tend to be slightly wide over the back, loin, and rump. The shoulders and hips are usually slightly smooth but may show a slight tendency toward prominence. The twist tends to be slightly deep and full, and the legs tend to be slightly thick and plump. They have a slightly thick fat covering over the back, ribs, loin, and rump. In handling, the backbone and ribs are slightly discernible. Choice grade slaughter sheep usually present a moderately refined appearance.

(2) A development of quality which is superior to that specified as minimum for the Choice grade may compensate, on an equal basis, for a development of conformation which is inferior to that specified as minimum for Choice as indicated in the following example: A sheep which has evidences of quality equivalent to the midpoint of the Choice grade may have conformation equivalent to the midpoint of the Good grade and remain eligible for Choice. However, in no instance may a sheep be graded Choice which has a development of conformation inferior to that specified as minimum for the Good grade. Also, a sheep which has conformation at least one-third grade superior to that specified as minimum for the Choice grade may qualify for Choice with a development of quality equivalent to the lower limit of the upper one-third of the Good grade. Compensation of superior conformation for inferior quality is limited to one-third grade of deficient quality.

(b) *Good.* (1) Slaughter sheep having minimum conformation requirements for the Good grade are slightly thin muscled throughout, and are moderately narrow in relation to their length and height, and they are slightly narrow over the back, loin, and rump. Hips and shoulders are moderately prominent. The twist is slightly shallow, and the legs are slightly small and thin. They have a slightly thin fat covering over the back, ribs, and loin. In handling, the bones of the shoulders, backbone, hips, and ribs are slightly prominent. Good grade sheep may present evidences of slightly low quality. The body is somewhat lacking in symmetry and smoothness.

(2) A development of quality which is superior to that specified as minimum for the Good grade may compensate, on an equal basis, for a development of conformation which is inferior to that specified as minimum for Good as indicated in the following example: A sheep which has evidences of quality equivalent to the midpoint of the Good grade may have conformation equivalent to the midpoint of the Utility grade and remain eligible for Good. However, in no instance may a sheep be graded Good which has a development of conformation inferior to that specified as minimum for the Utility grade. Also, a sheep which has conformation at least one-third grade superior to that specified as minimum for the Good grade may qualify for Good with a development of quality equivalent to the lower limit of the upper one-third of the Utility grade. Compensation of superior conformation for inferior quality is limited to one-third grade of deficient quality.

(c) *Utility.* (1) Slaughter sheep having minimum conformation requirements for the Utility grade are thinly muscled throughout, and are very angular and very narrow in relation to their length and height, and they are very thin fleshed, very narrow over the back, loin, and rump, and very shallow in the twist. The hips are very prominent, and the shoulders are usually open, rough, and prominent. The legs are very small and thin and present a slightly concave appearance. Utility grade slaughter sheep show no visible evidences of fat covering. In handling, the bones of the shoulders, backbone, hips, and ribs are so thinly covered that they are very prominent. Utility grade slaughter sheep are of rather low quality. The bones and joints are proportionately large, and the body is very rough and unsymmetrical.

(2) A development of quality which is superior to that specified as minimum for the Utility grade may compensate, on an equal basis, for a development of conformation which is inferior to that specified as minimum for Utility as indicated in the following example: A sheep which has evidences of quality at least one-third grade superior to that specified as minimum for the Utility grade may have conformation equivalent to the minimum for the upper one-third of the Cull grade and remain eligible for Utility. However, in no instance may a sheep be graded Utility which has a development of conformation inferior to the minimum for the upper one-third of the Cull grade. Also, a sheep which has conformation at least one-third grade superior to that specified as minimum for the Utility grade may qualify for Utility with a development of quality equivalent to the lower limit of the upper one-third of the Cull grade. Compensation of superior conformation for inferior quality is also limited to one-third grade of deficient quality.

(d) *Cull.* (1) The Cull grade includes those slaughter sheep whose characteristics are inferior to those specified as minimum for the Utility grade.

**§ 53.136 Specifications for Official U.S. Standards for Grades of Slaughter Lambs, Yearlings, and Sheep (Yield).**

* * * * *

Done at Washington, D.C., on October 22, 1984.

**William T. Manley,**

*Deputy Administrator, Marketing Programs.*

[FR Doc. 84-28281 Filed 10-25-84; 8:45 am]

BILLING CODE 3410-02-M

---

**7 CFR Part 910**

**[Lemon Reg. 487]**

## Lemons Grown in California and Arizona; Limitation of Handling

**AGENCY:** Agricultural Marketing Service, USDA.

**ACTION:** Final rule.

**SUMMARY:** This regulation establishes the quantity of fresh California-Arizona lemons that may be shipped to market at 200,000 cartons during the period October 28—November 3, 1984. Such action is needed to provide for orderly marketing of fresh lemons for the period due to the marketing situation confronting the lemon industry.

**EFFECTIVE DATE:** October 28, 1984.

**FOR FURTHER INFORMATION CONTACT:** William J. Doyle, Chief, Fruit Branch, F&V, AMS, USDA, Washington, D.C. 20250, telephone 202–447–5975.

**SUPPLEMENTARY INFORMATION:** This final rule has been reviewed under Secretary's Memorandum 1512–1 and Executive Order 12291, and has been designated a "non-major" rule. William T. Manley, Deputy Administrator, Agricultural Marketing Service, has certified that this action will not have a significant economic impact on a substantial number of small entities.

This final rule is issued under Marketing Order No. 910, as amended (7 CFR Part 910) regulating the handling of lemons grown in California and Arizona. The order is effective under the Agricultural Marketing Agreement Act of 1937, as amended (7 U.S.C. 601–674). The action is based upon recommendations and information submitted by the Lemon Administrative Committee and upon other available information. It is found that this action will tend to effectuate the declared policy of the Act.

This action is consistent with the marketing policy currently in effect. The committee met publicly on October 23, 1984, at Los Angeles, California, to consider the current and prospective conditions of supply and demand and recommended a quantity of lemons deemed advisable to be handled during the specified week. The committee reports that lemon demand is steady.

It is further found that it is impracticable and contrary to the public interest to give preliminary notice, engage in public rulemaking, and postpone the effective date until 30 days after publication in the **Federal Register** (5 U.S.C. 553), because of insufficient time between the date when information became available upon which this regulation is based and the effective date necessary to effectuate the declared purposes of the Act. Interested persons were given an opportunity to submit information and views on the regulation at an open meeting. It is necessary to effectuate the declared purposes of the Act to make these regulatory provisions effective as specified, and handlers have been apprised of such provisions and the effective time.

**List of Subjects in 7 CFR Part 910**

Marketing Agreements and Orders, California, Arizona, Lemons.

Section 910.787 is added as follows:

**§ 910.787 Lemon Regulation 487.**

The quantity of lemons grown in California and Arizona which may be handled during the period October 28, 1984, through November 3, 1984, is established at 200,000 cartons.

(Secs. 1–19, 48 Stat. 31, as amended; 7 U.S.C. 601–674)

Dated: October 24, 1984.

**Thomas R. Clark,**

*Deputy Director, Fruit and Vegetable Division, Agricultural Marketing Service.*

[FR Doc. 84–28485 Filed 10–25–84; 8:45 am]

BILLING CODE 3410–02–M

## FEDERAL HOME LOAN BANK BOARD

### 12 CFR Parts 523, 541, 545, 549, 561, 563, and 584

**[No. 84–580]**

### Implementation of New Powers; Limitation on Loans to One Borrower

October 19, 1984.

**AGENCY:** Federal Home Loan Bank Board.

**ACTION:** Final rule; solicitation of comments.

**SUMMARY:** The Board has adopted technical corrections to its final regulations implementing the new powers granted to federally chartered savings and loan associations and mutual savings banks by the Garn-St Germain Depository Institutions Act of 1982, and also to related provisions.

**DATES:** These amendments are effective October 26, 1984. Comments must be received by December 4, 1984.

**ADDRESS:** Send comments to Director, Information Services Section, Office of the Secretariat, Federal Home Loan Bank Board, 1700 G Street, N.W., Washington, D.C. 20552. Comments will be available for public inspection at the above address.

**FOR FURTHER INFORMATION CONTACT:** Susan McC. van den Toorn, Attorney, (202–377–6417) or Wendy B. Samuel, Deputy Director, (202–377–6445), Regulations and Legislation Division, Office of General Counsel, Federal Home Loan Bank Board, 1700 G Street, N.W., Washington, D.C. 20552.

**SUPPLEMENTARY INFORMATION:** On April 26, 1983, the Board adopted amendments to its regulations implementing new powers granted to federally chartered savings and loan associations and savings banks ("federal associations") by the Garn-St Germain Depository Institutions Act of 1982 ("DIA") (Pub. L. No. 97–320; 96 Stat. 1469 (1982)). The DIA amended the Home Owners' Loan Act of 1933 (HOLA) (12 U.S.C. 1461–1470), granting federal associations new authority to accept demand deposits, to offer NOW accounts to governmental units, to invest in time deposits in institutions the accounts of which are insured by the Federal Savings and Loan Insurance Corporation ("FSLIC"), to make commercial loans, and to engage in personal property leasing. The DIA also expanded the existing power of federal associations to make real estate loans, to invest in governmental obligations and commercial paper and corporate debt securities, and to make educational and consumer loans. While implementing these authorities, the Board also made revisions to its regulations to permit certain suretyship and trust activities and collateralization of deposits, to update the preapproved activities of service corporations and to place additional limits on loans to one borrower. In addition, the Board revised Part 545 to permit federal associations to engage in all activities authorized by statute, subject to these regulations, and incorporated various technical amendments. Board Resolution No. 83–241 (April 26, 1983), 48 FR 23032 (May 23, 1983). The Board's experience under the amended regulations has revealed the need for various technical amendments which will be discussed individually below. While the amendments pertain primarily to Part 545, technical amendments, consisting primarily of corrections to cross references to accommodate the changes to section numbers in Part 545, have been made to other Parts. The Board recognizes the need for additional corrections, particularly with regard to references in the Insurance Regulations to Part 545, and will address those corrections in a future document.

**Deposit Associations**

In revising Part 545, the Board inadvertently deleted § 545.1–2(c) regarding priority of savings deposits. The Board is taking this opportunity to insert it in § 549.5–1(b)(5).

**Real Estate Lending**

As adopted by Board Resolution No. 83–241, § 545.34 imposed various restrictions on the authority of a federal association to include in the loan contract provisions pertaining to due-on-sale clauses, late charges, and prepayment penalties. Paragraphs (a) and (c) expressly stated that an association may include a due-on-sale clause and prepayment penalty provision in its loan contract, but paragraph (b) includes no express statement with respect to late charges. The Board is therefore clarifying its intent that late charges may be imposed, subject to the conditions of § 545.34(b), if the loan contract so provides. The

Board also notes its view that these provisions are preemptive of any state laws purporting to limit or affect a federal association's authority to impose late charges and prepayment penalties, to require escrow payments and to include due-on-sale clauses in its loan documents. The Board is also taking this opportunity to correct a cross-reference contained in § 545.34 (b) and (c) which refers to the disclosure provisions of § 545.33 (Home loans).

In order to avoid the possible implication that associations are not subject to Part § 591, Preemption of State Due-On-Sale Laws, applicable to lenders generally, the Board is taking this opportunity to amend § 545.34(c) to clarify its position that the ability of a federal association to impose a prepayment penalty is subject also to the limitations of § 591.5(b)(2).

The Board is relocating a provision relating to appraisals of real property from § 545.40(b) to § 545.32(b). This makes clear that the general appraisal guidelines issued by the Board's Office of Examinations and Supervision apply to any appraisal of real estate required by Part 545. The Board is also relocating one provision from the description of security property, § 545.32(c)(4), to the section pertaining to the classification of loans or investments, § 545.31(a). This provision states that a loan may be considered to be made on the security of real estate if an association relies substantially on the real estate as the primary security for the loan. The substance of this provision has not changed, but it has been moved to make clear what is necessary in order to classify a loan as a real estate loan. The Board is also adding a new paragraph (4) to § 545.32(b), pertaining to amortization, and deleting the same language from § 545.33(c). The substance of the regulations is unchanged, but the relocation of the amortization language clarifies that any real estate loan may be fully or partially amortized, nonamortized or a line-of-credit loan, subject to the relevant regulations applicable to the particular type of loan.

The amendments also add new § 545.32(b)(5) and (6) pertaining to initial loan charges and escrow accounts. This authority had been stated in the previous regulations and is being reinserted to resolve questions that have arisen about the continued authority of federal associations to impose such charges. The Board is also amending the last sentence of § 545.32(d)(1), pertaining to the loan-to-value-ratio requirements for home loans secured by both real estate and savings accounts. For the purpose of clarifying this limitation, the Board is inserting a phrase contained in the previous regulation (§ 545.6–2(a)(5)) indicating that the limitation on the source of the funds in the account securing the loan applies when the loan is originated in excess of 90 percent of the initial appraised value. The amendment also restates the general limitation on loans in excess of the appraised value of the real estate. In addition, the Board is inserting language in § 545.345(a) from the previous regulation (§ 545.6–6(a)) to clarify that partially amortized loans shall be repayable with principal and interest payments sufficient to meet a 30-year amortization schedule. The Board is also clarifying the meaning of the phrase "development project" in § 545.36(d). The use of the words "may include" in the third sentence of that paragraph has caused some confusion as to whether development projects encompass more than all facilities comprising an integrated development plan. The intent of the amendment was merely to eliminate the previous rule's limitation (to residential, recreational and other facilities, § 545.6–2(d)(4)), but not to extend the rule beyond all facilities comprising an integrated development plan. The amendments also add to the last sentence of § 545.42 references to § 545.32(b)(4) regarding amortization and to § 545.33(f) regarding disclosures, and a clarification is inserted that no loan contract made to pursuant to § 545.42 may provide for the deferral and capitalization of interest on a loan. The amendments also make several other corrections to cross-references, language, and typographical errors.

**Finance Leasing**

It has been the Board's view that federal associations have the authority to engage in leasing activities under the lending authority contained in HOLA, provided that the leasing activities are the functional equivalent of lending. The final rule expanded this type of leasing, referred to as "finance leasing" (as distinguished from, and in addition to, the operating lease authority conferred by section 330 of the DIA), to include real as well as personal property. This authority, however, is circumscribed by requirements that reflect the guidelines established by courts for bank leasing activities that are the functional equivalent of lending. *See M&M Leasing Co.* v. *Seattle First National Bank*, 563 F.2d 1377 (9th Cir. 1977), *cert. denied*, 436 U.S. 956 (1978). One guideline limited the lessor/lender reliance on the residual value of the property at the end of the lease term. The previous regulation implemented this requirement by limiting the residual value of the property to 25% of its acquisition cost. However, this limitation was not adaptable to real property, which usually retains a far greater value over the life of a functionally equivalent real estate loan.

To continue to ensure that a federal association does not substantially rely on the residual value of the security property, the final rule limited such reliance to no more than 20% of the return (along with rental payments and tax benefits) under the full-payout requirement. As a result, the Board deemed that there was no need to require that the portion of the estimated residual value in excess of the limitation be guaranteed. However, this has led to some confusion as to whether an association can use a guarantee, or other source, to ensure that its reliance on the residual value does not exceed 20% of the return on this investment. Since the purpose of the change was to ensure that reliance on the residual value would be limited, it is appropriate to permit reliance on other sources (including guarantees), along with rental income and estimated tax benefits. The Board, therefore, is amending § 545.53(c)(2) accordingly.

**Service Corporations**

The Board has clarified its intentions with respect to the loosening of the limitation on conforming loans to service corporations. Conforming loans may be made to service corporations or their joint ventures in an aggregate amount equal to half the loans-to-one-borrower limit (12 CFR 563.9–3(b)) where the association owns ten percent or more of the service corporation or where the service corporation owns ten percent or more of the joint venture. In other words, the total amount of conforming loans made by an association to such service corporations and joint ventures may not exceed half the loans-to-one-borrower limit. With respect to service corporations in which the association owns less than ten percent or joint ventures in which the service corporation owns less than ten percent, conforming loans by the association may be made up to the loans-to-one-borrower limit to each such service corporation or joint venture. The Board is also amending this section to include functionally equivalent conforming leases. The applicable loans-to-one-borrower limit (general or commercial) will be determined by the characterization of the loans.

In addition, the Board is reinstating the requirement for preapproval of the acquisition of an ongoing business from

association insiders where the purchase price exceeds the fair market value of the tangible assets of the business. This provision had been deleted in the expectation that the conflict-of-interest regulation revision, which was being considered at the time this regulation was adopted, would cover these situations. As that did not occur, the Board is reinstating the previous provisions. For the same reason, the Board is also reinstating the requirement that Board approval be obtained before any activity of the service corporation is performed through one or more joint ventures if a director, officer, or controlling person of any stockholder of the service corporation as a direct or indirect beneficial interest in the joint venture.

Finally, the Board is reinstating authority regarding the purchase of office supplies, furniture, and equipment primarily for financial institutions which was inadvertently deleted from the list of preapproved activities.

**Commercial Paper and Corporate Debt Securities**

In adopting regulatory changes in connection with the DIA, the Board also made certain adjustments in the requirements for investment in commercial paper and corporate debt securities. While these changes for the most part removed prior restrictions, they imposed an additional requirement that these investments be rated by two nationally recognized investment rating services, replacing the previous requirement for rating by one such service. This amendment was due in part to a comment letter suggesting that a double rating was an appropriate guarantee of the security of such investments. It now appears that this is true with respect to commercial paper, but that with respect to corporate debt securities the prior requirement for a single rating is sufficient. In fact, a double rating requirement precludes purchase of many prudent corporate debt investments. Consequently, with respect to corporate debt securities, the Board is amending § 545.75(b) by reinstating its prior requirement that these instruments be rated by a single nationally recognized investment rating service. This amendment is also being made with respect to corporate debt securities which qualify as liquid assets pursuant to § 523.10(g)(9) and to corporate debt securities subject to less stringent loans-to-one-borrower limitations pursuant to § 563.9–3(b)(3)(ii).

The Board notes in making this change that questions have arisen as to what constitutes a nationally recognized investment rating service and what are the four highest rating "grades" or categories. The Board does not wish to address these questions in the regulations because it believes that to include regulatory language that would define or name rating services might unnecessarily limit the flexibility of these regulations. The Board takes this opportunity to state generally its view that entities recognized by the Securities and Exchange Commission ("SEC") as nationally recognized statistical rating organizations would qualify as nationally recognized investment rating services for purposes of its regulations. The Board also states its view that the four highest investment rating grades or categories are those rating "categories" recognized by the SEC. In order to conform the language of the regulation regarding such ratings to that of the SEC, §§ 523.10, 545.75 and § 563.9–3 have been amended to use the term "categories" rather than grades.

The Board's regulation regarding the loans-to-one-borrower limitations applicable to investment in commercial paper and corporate debt securities (12 CFR 545.75(b)(3) (1983)) references § 563.9–3 generally (loans to one borrower). The Board is clarifying its intent that the commercial loans-to-one-borrower limit, rather than the general limit, is applicable to these investments. Consequently, the reference in § 545.75(b)(3) to § 563.9–3 is redesignated as § 563.9–3(b)(2).

**Open-End Management Investment Companies**

Prior to the effective date of Board Resolution No. 83–241, three sections of the regulations permitted federal associations to invest in the shares of open-end management investment companies. Section 545.9(a) permitted investments in liquid assets as defined in § 523.10(g), which included at subparagraph (8) shares of open-end management investment companies whose investments are limited to those specified in § 523.10(g)(1)–(7). Section 545.9(i) permitted investment in open-end management investment companies whose policy limited their investments to those assets in which a federal association was permitted to invest directly without percentage-of-assets limitations. Section 545.9–4(b)(3) permitted as part of the investment authority in commercial paper and corporate debt securities purchases of shares of open-end management investment companies subject to a five-percent-of-assets limitation on purchases of the shares of any one such company. Although it did not so state, § 545.9–4(b)(3) implied that this limitation on purchases from a single issuer applied only to open-end management investment companies investing in permissible commercial paper and corporate debt securities. The statutory changes obviated the need to separate regulatory authority for investment in open-end management investment companies investing in commercial paper and corporate debt securities and those investing in assets not subject to percentage-of-assets limitations, since commercial paper and corporate debt is now included in the unlimited category. The new regulations, as adopted, while consolidating this authority inadvertently imposed the five-percent-of-assets limit on purchases from a single issuer on investments in all open-end management investment companies, not only those having commercial paper and corporate debt investments. This amendment clarifies the Board's intent to continue to apply the five-percent-of-assets limit on purchases from a single issuer only to investments in funds investing in commercial paper and corporate debt. The amendment also clarifies that this is a substitute for, rather than an addition to, imposition of the loans-to-one-borrower limit contained in 12 CFR 563.9–3(b), which applies to direct purchases of commercial paper and corporate debt securities.

**Savings Accounts**

The Board in Resolution No. 82–193 (47 FR 13776 (1982)) eliminated the ten year maximum-term restriction on certificate accounts. The current amendment removes the corresponding provision in § 563.3–1 which had limited a renewal period on a certificate account to ten years. The Board is also amending § 563.3–1(c)(2) by changing the term for which an institution may accept any fixed-term account from not less than 14 days to not less than seven days.

**Bonds for Agents**

The Board has amended § 563.20 to exempt insured institutions from the bond requirement of the section in order to more effectively allow them to perform the function of collecting and paying agent as authorized by § 545.13(c) (as amended, 48 FR 23059).

**Recourse Loans**

In Board Resolution No. 82–20 (47 FR 4049 (1982)), the Board rescinded its prohibition against the sale by insured institutions of loans, and participation interests in loans, with recourse (former § 563.23), and correspondingly deleted the exception for loans sold subject to a subordinated interest or guarantee not

exceeding ten percent if certain reserve requirements were met. This exception was included in the definition of the term "without recourse" (former § 561.8). These amendments were liberalizing in nature and were intended to increase institutions' flexibility in accommodating innovative arrangements for secondary-market sales. However, deletion of the exception for limited subordinations and guarantees inadvertently restricted use of these techniques in a number of areas, such as mobile-home loan sales and loans-to-one-borrower calculations, where recourse loans continue to be prohibited or treated differently. The Board is therefore taking this opportunity to reinstate the exception, with the reserve requirement modified to comply with the current requirement for recourse loans set forth at 12 CFR 563.13(b).

### Geographic Lending Restrictions

The Board also revised § 563.9 to eliminate geographic limitations on the lending activities of insured institutions pursuant to its authority under section 403(b) of the National Housing Act to permit an insured institution to make loans outside its normal lending territory to the extent authorized by its chartering authority. Inadvertently omitted by this revision was language contained in § 563.9(b) concerning requirements for a signed report of appraisal of any real estate relied upon as the primary security for the loan. The Board is therefore reinstating the necessary language to correct this omission, and is clarifying that the appraisal requirements apply only to loans made primarily on the security of real estate.

### Notice to Housing Creditors

The final rule incorporated a revised Notice to Housing Creditors as an Appendix to Part 545. This Notice, required by § 807(b) of the DIA, identified those rules applicable to housing creditors (other than commercial banks, credit unions, and federal associations) authorized by § 804(a) to make alternative mortgage transactions. The revised Notice was intended to identify those rules setting forth the authority to make partially amortized or nonamortized loans and to adjust the interest rate, payment, balance, or term to maturity; limitations on adjustments to loans secured by borrower-occupied property; and requirements for disclosures on loans secured by borrower-occupied property that are not fixed rate and fully amortized. While referencing § 545.33 (c), (e), and (f)(4)–(11) on the authority to make adjustments, the Notice inadvertently omitted a reference to § 545.32(b) (3) and (4), which further define the authority to make adjustments. The Board is correcting the reference by adding § 545.32(b) (3) and (4) to the Notice. The Board is also taking this opportunity to clarify that the rules identified also apply to credit sales made by housing creditors. Therefore, the Notice is revised to state that housing creditors should read the term "loan" to mean "credit sale" when appropriate.

### Correction of Typographical and Technical Errors

As published, the final rule contained typographical errors and a technical omission in a reference in paragraph (c)(2)(iii) of § 545.121 (Indemnification of directors, officers and employees), and did not delete certain unnecessary language regarding employment contracts in § 545.122, and contained a typographical error in a reference in paragraph (a)(3)(ii) of § 563.9–3 (Loans to one borrower). By its action today, the Board corrects these typographical errors. The Board also corrects references to the statutory citation of the "leeway" authority of federal associations to recognize the reinstatement of 12 U.S.C. 1464(c)(3)(D) by Pub. L. No. 97–457, 96 Stat. 2507 (1983) and in § 561.26 corrects the reference regarding service corporations to read "§ 545.74". For consistency in style, the Board is changing, in § 545.50(b), the words "are defined as" to read "include".

The Board has determined that observance of the notice and public comment procedure requirements of 5 U.S.C. 552(b) and 12 CFR 508.11 and the delay of effective date provided pursuant to 5 U.S.C. 552(d) and 12 CFR 508.14 are unnecessary and contrary to the public interest. These amendments contain minor, technical corrections and interpretations intended to clarify regulations issued after extensive opportunity for public comment.

The Board, however, solicits the public's comments on the amendments and has accordingly provided for a 60-day comment period after which, if appropriate, the rules may be modified, extended, or otherwise addressed.

### Lists of Subjects in 12 CFR Parts 523, 541, 545, 549, 561, 563 and 584

Federal Home Loan Banks, Savings and loan associations.

Accordingly, the Board hereby corrects FR Doc. 83–13317, appearing at 48 FR 23032 *et seq.* in the issue of May 23, 1983, and amends Parts 523, 541, 545, 549, 561, 563, and 584 of Subchapters B, C, D, and F, Chapter V, Title 12 of the *Code of Federal Regulations*, as set forth below.

1. Amend page 23042, first column, first full paragraph, third sentence, as follows:

"The Board believes that the prior regulatory language, which in large part simply restated the provisions of 12 U.S.C. 1464(c)(3)(C)–(D), is unnecessary and that associations desiring to make such loans may simply refer to the statute."

2. Amend page 23048, second column, the second full paragraph, by removing "12 U.S.C. 1464(c)(1)C–(F)" and by substituting "12 U.S.C. 1464(c)(1) (C)–(F)".

## SUBCHAPTER B—FEDERAL HOME LOAN BANK SYSTEM

## PART 523—MEMBERS OF BANKS

3. Amend § 523.10(g) by: removing the word "bank" the first time it appears in paragraph (1) thereof and inserting in its place the word "Bank"; by removing the word "grades" in paragraph (g)(9)(ii) and substituting the word "categories"; and by revising paragraphs (g)(9)(i)(*a*) and (h)(5); as follows:

**§ 523.10 Definitions for purposes of this section, § 523.11, and § 523.12.**

* * * * *

(g) *Liquid assets.* * * *

* * * * *

(9) * * *

(i) * * *

(*a*) continue to be rated in one of the four highest categories by the most recently published rating of such obligations by a nationally recognized investment rating service, * * *

(h) *Short-term liquid assets.* * * *

* * * * *

(5) obligations of or obligations (other than gold-related obligations) issued by a public housing agency which have the full faith and credit of the United States pledged under section 1421a(c) or section 1437i(a) of Title 42 of the United States Code, as amended, and will mature in 6 months or less;

* * * * *

## SUBCHAPTER C—FEDERAL SAVINGS AND LOAN SYSTEM

## PART 541—DEFINITIONS

**§ 541.20 [Amended]**

4. Amend § 541.20 by inserting the word "or" between the words "structure" and "unit".

## PART 545—OPERATIONS

5. Amend § 545.31 by adding a sentence at the end of paragraph (a) thereof, as follows:

**§ 545.31 Election regarding classification of loans or investments.**

(a) * * * To classify a loan as a real estate loan, an association must rely substantially upon the real estate as the primary security for the loan.

* * * * *

6. Amend § 545.32 by adding a new sentence at the end of paragraph (b)(1), adding new paragraphs (b) (4), (5), and (6), adding the word "and" at the end of paragraph (c)(3), removing paragraph (c)(4), redesignating paragraph (c)(5) as paragraph (c)(4), and revising the last sentence of paragraph (d)(1) thereof, as follows:

**§ 545.32 Real estate loans.**

* * * * *

(b) *General.* (1) *Appraisals.* * * * The appraisals of any real estate required by this Part shall be rendered in accordance with the general appraisal guidelines issued by the Office of Examinations and Supervision.

* * * * *

(4) *Amortization.* Subject to the limitations in §§ 545.33 and 545.35 of this Part, a real estate loan may be fully amortized, partially amortized, nonamortized, or a line-of-credit loan, and the loan contract may provide for the deferral and capitalization of a portion of the interest.

(5) *Initial loan charges.* Except as provided in § 563.35(d) of this Chapter, an association may require a borrower to pay necessary initial charges connected with making a loan, including the actual costs of title examination, appraisal, credit report, survey, drawing of papers, loan closing, and other necessary incidental services and costs, in such reasonable amounts as the board of directors may fix. The association may collect the charges from the borrower and pay the persons rendering services.

(6) *Escrow accounts.* An association may require that all or any part of the estimated annual taxes, assessments, insurance premiums, and other charges on any loan be paid in advance to the association, in addition to interest and principal payments on the loan, to enable the association to pay such charges as they become due, consistent with the Real Estate Settlement Procedures Act of 1974 (12 U.S.C. 2601–2617) ("RESPA").

(c) *Security property.* * * *

* * * * *

(3) The security property is capable of separate appraisal; and

(4) With regard to a security property that is a leasehold or other interest for a period of years, the term of the interest extends, or is subject to extension or renewal at the option of the association, for a term of at least five years following the maturity of the loan.

(d) *Loan-to-value ratios.* (1) * * * Home loans made on the combined security of real estate and savings accounts may be made in excess of the maximum loan-to-value ratios adopted pursuant to this paragraph (d) with such excess secured by the savings account: *Provided*, that for loans originated in excess of 90 percent of the initial appraised value of the security property, the savings account shall consist only of funds belonging to the borrower, the borrower's family, or the borrower's employer, and the loans shall not exceed the appraised value of the real estate.

* * * * *

**§ 545.33 [Amended]**

7. Amend § 545.33 by inserting before the period at the end of paragraph (a) the words "or by § 545.32(b)(4) of this Part"; by revising paragraph (c) as follows: "(c) *Amortization.* The loan contract may provide for the deferral and capitalization of all interest on loans to natural persons secured by borrower-occupied property and on which periodic advances are being made."; by changing the reference in the first sentence of paragraph (f) from "12 U.S.C. 1464(c)(3)(C)" to "12 U.S.C. 1464 (c)(3) (C) and (D)"; by removing from paragraph (f)(1) the word "Whether" in the first sentence, and inserting in its place the word "If"; by removing from the first sentence of paragraph (f)(1) the words "and, if so,"; by removing from paragraph (f)(2) the words "A statement about whether" in the first sentence, inserting in their place the word "If", and removing from the first sentence of paragraph (f)(2) the words "and, if so,"; by changing the word "or" in the first sentence of paragraph (f)(9) to the word "of".

**§ 545.34 [Amended]**

8. Amend § 545.34 by removing the reference to "§ 545.33(f)(1)(ii)" in the first sentence of paragraph (b) and inserting in its place "§ 545.33(f)(2)"; by removing the word "of" the first time it appears in the last sentence of paragraph (b) and inserting in its place the word "after"; by inserting a new first sentence of paragraph (b) to read: "An association may include in the loan contract a provision authorizing the imposition of a late charge with respect to the payment of any delinquent periodic payment."; by adding a sentence at the end of paragraph (b) to read "No form of such late charge permitted by this paragraph shall be considered as interest to the association and the association shall not deduct late charges from the regular periodic installment payments on the loan, but must collect them as such from the borrower."; by removing the reference to "§ 545.33(f)(1)(ii)" in the second sentence of paragraph (c) and inserting in its place "§ 545.33(f)(2) and § 591.5(b)(2)"; and by removing the words "of a" from the last sentence of paragraph (c) and by inserting in their place the word "following".

9. Amend § 545.35(a) by inserting a new sentence at the end of paragraph (a), as follows:

**§ 545.35 Other real estate loans.**

(a) * * * Partially amortized loans shall be repayable with principal and interest payments sufficient to meet a 30-year amortization schedule. A partially amortized loan is any loan which is repayable in full in a lump sum at the end of the loan term but which requires partial amortization during the loan term by regular monthly payments which include both principal and interest.

* * * * *

**§ 545.36 [Amended]**

10. Amend § 545.36 by inserting in the first sentence, following the words "unimproved real estate", the parenthetical "(or loans on the security of unimproved real estate already owned by the borrower)"; and by removing the phrase "may include" from the third sentence of paragraph (d) and inserting in its place the word "includes".

**§ 545.40 [Amended]**

11. Amend § 545.40 by removing paragraph (b) and removing the paragraph designation and heading of the remaining text of the section.

**§ 545.42 [Amended]**

12. Amend § 545.42 by removing from the last sentence the phrase "under § 545.33(e) of this Part" and by inserting in its place the following: "by § 545.32(b)(4) and § 545.33 (e) and (f) of this Part. No loan contract may provide for the deferral and capitalization of interest on a loan made under this section."

**§ 545.45 [Amended]**

13. Amend § 545.45(d)(3)(i) by removing the phrase "residential real estate loan under § 545.33(b)" and

inserting in its place "home loan under § 545.33".

**§ 545.50 [Amended]**

14. Amend § 545.50(b) by removing the phrase "are defined as", and inserting in its palce the word "include".

**§ 545.51 [Amended]**

15. Amend § 545.51 by changing the references to "§ 545.140" and "§ 545.140(a)(2)" in paragraph (b) to "§ 545.141" and "§ 545.141(a)(2)", respectively.

**§ 545.53 [Amended]**

16. Amend § 545.53(c)(2) by inserting after the phrase "estimated tax benefits," and before the phrase "and the estimated residual value . . ." the phrase "guarantees and other sources,".

17. Amend § 545.74 by changing the phrase "Confirming loan" in paragraph (a)(2) to read "Conforming loan"; changing the reference to "12 U.S.C. 1464(c)(3)(C)" to "12 U.S.C. § 1464(c)(3)(C)–(D)"; by redesignating paragraphs (b)(i) and (b)(ii)(*a*) and (*b*) as paragraphs (b)(1) and (b)(3)(i) and (ii); by redesignating paragraphs (b)(iii) and (b)(iv) as paragraphs (b)(4) and (b)(5); by removing the period at the end of (c)(2)(ix) and inserting in its place a semicolon; and by adding paragraphs (b)(2), (b)(6), (c)(2)(x), and revising paragraph (d)(2) as follows:

**§ 545.74 Service corporations.**

* * * * *

(b) *General.* * * *

* * * * *

(2) Approval of the Board is obtained before any activity of the service corporation is performed through one or more joint ventures if a director, officer, or controlling person of any stockholder of the service corporation has a direct beneficial interest in the joint venture;

* * * * *

(6) Prior approval of the Board must be obtained for investment in a service corporation or for investment by a service corporation in a joint venture or subsidiary if the purpose of the investment is to acquire a going business for an amount exceeding the fair market value of the tangible net assets of that business from a director or officer of an association which owns any of the capital stock of the service corporation or from an entity in which a director or officer of the association has a direct or indirect beneficial interest or is a director, officer, controlling person, partner, or trustee.

* * * * *

(c) *Services primarily for financial institutions.* * * *

(2) * * *

* * * * *

(x) Purchase of office supplies, furniture, and equipment.

* * * * *

(d) *Amount of investment.* * * *

(2) * * *

(i) An amount not to exceed the loans-to-one-borrower limit may be invested in conforming loans and functionally equivalent leases made to each service corporation of which the association owns or holds with power to vote not more than ten percent of the capital stock, and to each joint venture in which a service corporation in which the association is a stockholder, including subsidiaries of such service corporations, (*a*) owns or holds with power to vote not more than a total of ten percent of the capital stock or (*b*) is a limited partner and has contributed not more than ten percent of such joint venture's capital. (ii) An aggregate outstanding amount not to exceed fifty percent of the loans-to-one-borrower limit may be invested in conforming loans and functionally equivalent leases to all service corporations in which the association owns more than ten percent of the capital stock, and to all joint ventures in which service corporations in which the association is a stockholder, including subsidiaries of such service corporations, (*a*) own or hold with power to vote more than a total of ten percent of the capital stock, or (*b*) are partners.

* * * * *

18. Amend § 545.75 by changing the reference to "§ 563.9–3" in paragraph (b)(3) to "§ 563.9–3(b)(2)"; by removing the word "grades" in paragraph (b)(1)(ii) and substituting the word "categories"; and by revising paragraph (b)(2)(iii) as follows:

**§ 545.75 Commercial paper and corporate debt securities.**

* * * * *

(b) *Limitations.* * * *

(2) * * *

(iii) rated in one of the four highest categories by a nationally recognized investment rating service at its most recent published rating before the date of purchase of the security.

* * * * *

19. Revise § 545.76 as follows:

**§ 545.76 Investment in open-end management investment companies.**

(a) *Authorization.* Pursuant to 12 U.S.C. § 1464(c)(1)(Q), an association may invest in, redeem, or hold shares or certificates in any open-end management investment company which is registered with the Securities and Exchange Commission under the Investment Company Act of 1940 and the portfolio of which is restricted by such management company's investment policy, changeable only if authorized by shareholder vote, solely to any such investments as an association by law or regulation may, without limitation as to percentage of assets, invest in, sell, redeem, hold or otherwise deal with.

(b) *Limitations.* Where the investments of the open-end management investment company consist of commercial paper and corporate debt securities, such investments must come within the limitations of § 545.75(b) (1) and (2) of this Part. Five percent of assets shall be the maximum that may be invested in the shares of any one such company.

**§ 545.111 [Amended]**

20. Amend § 545.111 by changing the reference to "12 U.S.C. 1464(c)(3)(C)" to "12 U.S.C. 1464(c)(3)(C)–(D)."

**§ 545.121 [Amended]**

21. Amend § 545.121 by removing the word "reactors" in the first sentence of paragraph (c)(2)(iii) and inserting the words "disinterested directors".

**§ 545.122 [Amended]**

22. Amend § 545.122 by removing the phrase "savings and loan association with bylaws amended under § 544.6(k), a federal mutual savings bank or a Charter S or Charter T".

**Appendix [Amended]**

23. Amend the Appendix to Part 545 by removing the phrase "§ 545.33 (c), (e), and (f)(4)–(11)" and inserting in its place the following: "§ 545.32(b) (3) and (4), and § 545.33 (c), (e), and (f)(4)–(11). Housing creditors engaged in credit sales should read the term "loan" as "credit sale" wherever appropriate".

## PART 549—POWERS OF RECEIVER AND CONDUCT OF RECEIVERSHIPS

24. Amend §549.5–1 by adding a new paragraph (b)(5) as follows:

**§ 549.5–1 Deposit associations.**

* * * * *

(b) * * *

(5) Savings deposits and accounts shall, to the extent of their withdrawable value, be debts of the association having the same priority as debts of general creditors who have no priority, other than from consensual subordination, over other general creditors. Savings deposits of mutual associations shall have the same right to share in the remaining assets of the

association that savings share accounts would have.

* * * * *

**SUBCHAPTER D—FEDERAL SAVINGS AND LOAN INSURANCE CORPORATION**

**PART 561—DEFINITIONS**

25. Amend § 561.8 by redesignating the current text as paragraph (a), redesignating paragraphs (a), (b) and (c) therein as paragraphs (1), (2), and (3), and adding a new paragraph (b) thereto, as follows:

**§ 561.8 With recourse.**

* * * * *

(b) The term "with recourse" does not include loans or interests therein where the agreement of sale provides for the institution directly or indirectly (1) to hold or retain a subordinate interest in a specified percentage of the loans or interests; or (2) to guarantee against loss up to a specified percentage of the loans or interests, which specified percentage shall not exceed ten percent of the outstanding balance of the loans or interests at the time of sale: *Provided,* That the institution designates reserves as provided in § 563.13(b) of this Part for the subordinate interest or guarantee.

**§ 561.26 [Amended]**

26. Amend § 561.26 by changing the reference to "§ 545.9–1" to "§ 545.74".

**PART 563—OPERATIONS**

**§ 563.3–1 [Amended]**

27. Amend § 563.3–1(c)(2) by removing the number "14" and inserting the word "seven" and by removing paragraph (e) and redesignating paragraph (f) as new paragraph (e).

28. Revised § 563.9 as follows:

**§ 563.9 Geographic lending restrictions.**

(a) *Waiver of geographic restrictions.* An insured institution may invest in, sell, purchase, participate or otherwise deal in loans or interests therein beyond the 100-mile limit of 12 U.S.C. § 1726(b), without geographic restriction.

(b) *Appraisals.* An institution investing in a loan outside its normal lending territory shall obtain a signed report of appraisal of any real estate that is relied upon as the primary security for the loan. The report shall be prepared by an appraiser having no interest, direct or indirect, in that security or in any loan on that security and whose compensation is not affected by the approval or disapproval of the loan.

29. Amend § 563.9–3 by changing the reference to "(d)(3)(i)" in paragraph (a)(3)(ii) to "(a)(3)(i)"; by removing the word "grade" in paragraph (b)(3)(i) and substituting the word "category"; and by revising paragraph (b)(3)(ii) as follows:

**§ 563.9–3 Loans to one borrower.**

* * * * *

(b) Limitations. * * *

(3) * * *

(ii) corporate debt securities that may be sold with reasonable promptness at a price that corresponds reasonably to their fair value, and that are rated in one of the two highest categories by a nationally recognized investment rating service at the most recently published rating before the date of purchase of the security.

* * * * *

**§ 563.9–7 [Amended]**

30. Amend § 563.9–7(a) by removing the phrase "§ 545.6–1 or § 545.6–2(a)(3)(iii)" and inserting in its place the phrase "§ 545.38(a) or § 545.32(d)(2)".

31. Amend § 563.20 by revising the last sentence as follows:

**§ 563.20 Bonds for agents.**

* * * No bond need be obtained for any agent that is a bank that is a member of the Federal Deposit Insurance Corporation or any institution that is a member of the Federal Savings and Loan Insurance Corporation.

**SUBCHAPTER F—REGULATIONS FOR SAVINGS AND LOAN HOLDING COMPANIES**

**PART 584—REGULATED ACTIVITIES**

**§ 584.2 [Amended]**

32. Amend § 584.2(c) by removing the reference to "§ 545.9–1(c)" and substituting therefor "§ 545.74".

(Sec. 4, 80 Stat. 824, as amended (12 U.S.C. 1425a); sec. 5, 48 Stat. 132, as amended (12 U.S.C. 1464); secs. 402, 403, 48 Stat. 1256, 1257, as amended (12 U.S.C. 1725, 1726); Reorg. Plan No. 3 of 1947; 12 FR 4981, 3 CFR 1943–48 Comp., p. 1071)

By the Federal Home Loan Bank Board.

**J. J. Finn,**

*Secretary.*

[FR Doc. 84–28276 Filed 10–25–84; 8:45 am]

**BILLING CODE 6720–01–M**

---

**DEPARTMENT OF TRANSPORTATION**

**Federal Aviation Administration**

**14 CFR Part 39**

**[Docket No. 84–ASW–48; Amdt. 39–4940]**

## Airworthiness Directives; Hughes Helicopters, Inc., Model 369 Series Helicopters

**AGENCY:** Federal Aviation Administration (FAA), DOT.

**ACTION:** Final rule.

**SUMMARY:** This action publishes in the **Federal Register** and makes effective as to all persons an amendment adopting a new airworthiness directive (AD) which was previously made effective as to all known U.S. owners and operators of certain Hughes Helicopters, Inc., Model 369 series helicopters by individual letter. The AD requires an inspection and modification of certain tail rotor blades. This AD is prompted by reports of tail rotor tip cap separation which could result in loss of tail rotor control.

**EFFECTIVE DATE:** October 26, 1984, as to all persons except those persons to whom it was made immediately effective by priority letter AD 84–18–08, issued September 14, 1984, which contained this amendment.

Compliance schedule—as prescribed in body of AD.

**ADDRESSES:** The applicable service information may be obtained from Hughes Helicopters, Inc., Centinela Avenue and Teale Street, Culver City, California 90230. A copy of the Hughes Service Information Notice is contained in the Rules Docket of the Office of Regional Counsel, FAA, Southwest Region, 4400 Blue Mound Road, Fort Worth, Texas 76106.

**FOR FURTHER INFORMATION CONTACT:** Jerry Sullivan, Aerospace Engineer, ANM–172W, Airframe Section, FAA, Western Aircraft Certification Office, P.O. Box 92007, Worldway Postal Center, Los Angeles, California 90009–2007, telephone (213) 536–6166.

**SUPPLEMENTARY INFORMATION:** On September 14, 1984, priority letter AD 84–18–08 was issued and made effective immediately as to all known U.S. owners and operators of Hughes Helicopters, Inc., Model 369 series helicopters. The AD requires inspection and modification of certain tail rotor blades. The AD was prompted by reports of tail rotor blade tip cap separation with subsequent loss of tail rotor control.

Since it was found that immediate corrective action was required, notice and public procedure thereon were impracticable and contrary to public interest, and good cause existed to make the AD effective immediately by individual letter issued September 14, 1984, to all known U.S. owners and operators of Hughes Helicopters, Inc., Model 369 series helicopters. These conditions still exist and the ADF is hereby published in the **Federal Register** as an amendment to § 39.13 of Part 39 of the Federal Aviation Regulations to make it effective as to all persons.

The FAA has determined that this regulation is an emergency regulation that is not considered to be major under Executive Order 12291. It is impracticable for the agency to follow the procedures of Order 12291 with respect to this rule since the rule must be issued immediately to correct an unsafe condition in aircraft. It has been further determined that this action involves an emergency regulation under DOT Regulatory Policies and Procedures (44 FR 11034; February 26, 1979). If this action is subsequently determined to involve a significant/major regulation, a final regulatory evaluation or analysis, as appropriate, will be prepared and placed in the regulatory docket (otherwise, an evaluation or analysis is not required). A copy of it, when filed, may be obtained by contacting the person identified under the caption **"FOR FURTHER INFORMATION CONTACT."**

**List of Subjects in 14 CFR Part 39**

Air transportation, Aircraft, Aviation safety, Safety.

**Adoption of the Amendment**

Accordingly, pursuant to the authority delegated to me by the Administrator, § 39.13 of Part 39 of the Federal Aviation Regulations (14 CFR 39.13) is amended by adding the following new airworthiness directive:

**Hughes Helicopters, Inc.** (Hughes Helicopters): Applies to certain Model 369 (Army YOH–6A), 396A (Army OH–6A), D, E, H, HE, HM, and HS helicopters certificated in all categories, which have tail rotor blades installed, as identified in Hughes Service Information Notices DN–129, EN–18 and HN–195 dated August 27, 1984.

Compliance is required as indicated unless already accomplished.

To prevent possible hazards in flight associated with tail rotor blade tip rib separation, accomplish the following:

(a) For tail rotor blades with 100 or more hours' time in service on the effective date of this AD, accomplish the inspection and modification in accordance with paragraphs a through j of Hughes Service Information Notice DN–129, EN–18, HN–195, or FAA approved equivalent prior to further flight.

(b) For tail rotor blades with less than 75 hours' time in service on the effective date of this AD, accomplish the inspection and modification in accordance with paragraphs a through j of Hughes Service Information Notice DN–129, EN–18, HN–195, or FAA approved equivalent within the next 25 hours' time in service.

(c) Remove from service any tail rotor blade with visual evidence of tip cap to blade bond failure.

(d) An equivalent method of compliance with this AD may be used when approved by the Manager of the Western Aircraft Certification Office, Hawthorne, California.

(e) Special flight permits may be issued in accordance with Federal Aviation Regulations 21.197 and 21.199 to ferry aircraft to a maintenance base in order to comply with the requirements of this AD.

This amendment becomes effective October 26, 1984, as to all persons except those persons to whom it was made immediately effective by priority letter AD 84–18–08 issued September 14, 1984, which contained this amendment.

(Sections 313(a), 601, and 603, Federal Aviation Act of 1958, as amended (49 U.S.C. 1354(a), 1421, and 1423); 49 U.S.C. 106(g) (Revised, Pub. L. 97–449, January 12, 1983); and 14 CFR 11.89)

Issued in Fort Worth, Texas, on October 11, 1984.

**C.R. Melugin, Jr.,**

*Director, Southwest Region.*

[FR Doc. 84–28263 Filed 10–25–84; 8:45 am]

**BILLING CODE 4910–13–M**

## FEDERAL TRADE COMMISSION

**16 CFR Part 305**

## Rules for Using Energy Cost and Consumption Information Used in Labeling and Advertising of Consumer Appliances Under the Energy Policy and Conservation Act; Ranges of Comparability for Room Air Conditioners

**AGENCY:** Federal Trade Commission.

**ACTION:** Final rule.

**SUMMARY:** The Federal Trade Commission amends its Appliance Labeling Rule by revising the ranges of comparability used on required labels for room air conditioners.

Under the rule, each required label on a covered appliance must show a range, or scale, indicating the range of energy costs or efficiencies for all models of a size or capacity comparable to the labeled model. These ranges, which show the highest and lowest energy costs or efficiencies for the various size or capacity groupings of the appliances covered by the rule, are published in the **Federal Register** by the Commission no more often than annually, and are called "ranges of comparability." The figures to be used on the ranges are provided by the Commission after an analysis of data submitted by appliance manufacturers, who derive the energy costs or efficiencies of their appliances by following test procedures prescribed by the Department of Energy ("DOE"). Because appliance models are constantly being added, changed or dropped by manufacturers, the ranges of comparability are likely to change occasionally. This has been the case with the ranges for room air conditioners, and this notice publishes the new range figures, which, under §§ 305.10, 305.11 and 305.14 of the rule, must be used in the labeling and advertising of room air conditioners beginning January 24, 1985.

**EFFECTIVE DATE:** January 24, 1985.

**FOR FURTHER INFORMATION CONTACT:** James Mills or Lucerne D. Winfrey, 202–376–8934, Attorneys, Division of Enforcement, Federal Trade Commission, Washington, D.C. 20580.

**SUPPLEMENTARY INFORMATION:** Section 324 of the Energy Policy and Conservation Act of 1975 (EPCA) [1] required the Federal Trade Commission to consider labeling rules for the disclosure of estimated annual energy cost or alternative energy consumption information for at least thirteen categories of appliances: (1) Refrigerators and refrigerator-freezers; (2) freezers; (3) dishwashers; (4) clothes dryers; (5) water heaters; (6) room air conditioners; (7) home heating equipment, not including furnaces; (8) television sets; (9) kitchen ranges and ovens; (10) clothes washers; (11) humidifiers and de-humidifiers; (12) central air conditioners; and (13) furnaces. Under the statute, DOE is responsible for developing test procedures that measure how much energy the appliances use. In addition, DOE is required to determine the representative average cost a consumer pays for the different types of energy available.

On November 19, 1979, the Commission issued a final rule [2] covering seven of the thirteen appliance categories: refrigerators and refrigerator-freezers, freezers, dishwashers, water heaters, clothes washers, room air conditioners and furnaces.

The rule requires that energy efficiency ratings or energy costs and related information be disclosed on labels and fact sheets and in retail sales catalogs for all covered products manufactured on or after May 19, 1980. Certain point-of-sale promotional materials must disclose the availability of energy cost or energy efficiency rating information. The required disclosures and all claims concerning energy consumption made in writing or in broadcast advertisements must be based on the results of the DOE test procedures.

Pursuant to § 305.8 of the rule, manufacturers submitted reports to the Commission by January 21, 1980. These

[1] Pub. L. 94–163, 89 Stat. 871, 42 U.S.C. 6201 (1975).

[2] 44 FR 66466, 16 CFR Part 305 (November 19, 1979).

reports contained information on the estimated annual cost or energy efficiency rating for the seven categories of appliances derived from tests performed pursuant to the DOE test procedures. The reports also contained the model, the number of tests performed on each model, and the capacity of each model. From that information, the Commission compiled and published [3] ranges of comparability for each product, as required by § 305.10 of the rule.

Section 305.8(b) of the rule requires that manufacturers, after filing this initial report, shall report annually by specified dates for each product type.[4] Because manufacturers regularly add new models to their lines, improve existing models and drop others, the data base from which the ranges of comparability of costs or energy efficiency ratings are calculated is constantly changing. To keep the required information in line with these changes, the Commission is empowered, under § 305.10 of the rule, to publish new ranges (but not more often than annually), if an analysis of the new data indicates that the upper or lower limits of the ranges have changed by more than 15%.

The revised figures for the energy efficiency ratings for room air conditioners have been submitted and have been analyzed by the Commission. New ranges based upon them are herewith published.

In consideration of the foregoing, the Commission publishes the following ranges of comparability for use in the labeling and advertising of room air conditioners beginning January 24, 1985.

## PART 305—[AMENDED]

Appendix E to Part 305 is revised to read as set forth below:

### Appendix E—Room Air Conditioners

| Manufacturers rated cooling capacity in BTU's/hr. | Ranges of energy efficiency ratings | |
|---|---|---|
| | Low | High |
| Less than 4,000 | (1) | (1) |
| 4,000 to 4,299 | 5.1 | 7.5 |
| 4,300 to 4,799 | 5.5 | 7.5 |
| 4,800 to 5,299 | 5.6 | 8.7 |
| 5,300 to 5,799 | 6.3 | 9.0 |
| 5,800 to 6,299 | 6.4 | 9.5 |
| 6,300 to 6,799 | 8.5 | 9.0 |
| 6,800 to 7,299 | 5.8 | 10.1 |
| 7,300 to 7,799 | 5.4 | 9.0 |
| 7,800 to 8,299 | 5.7 | 9.8 |
| 8,300 to 8,799 | 6.5 | 9.4 |
| 8,800 to 9,299 | 5.6 | 11.0 |
| 9,300 to 9,799 | 6.1 | 10.9 |
| 9,800 to 10,299 | 6.1 | 11.5 |
| 10,300 to 10,799 | 7.2 | 9.1 |
| 10,800 to 11,299 | 6.0 | 8.8 |
| 11,300 to 11,799 | 6.0 | 9.0 |
| 11,800 to 12,299 | 5.1 | 9.5 |
| 12,300 to 12,799 | 7.6 | 9.6 |
| 12,800 to 13,299 | 5.8 | 9.0 |
| 13,300 to 13,799 | 7.2 | 9.7 |
| 13,800 to 14,299 | 6.0 | 9.9 |
| 14,300 to 14,799 | 6.3 | 8.4 |
| 14,800 to 15,299 | 5.5 | 8.6 |
| 15,300 to 15,799 | 5.7 | 7.5 |
| 15,800 to 16,499 | 5.9 | 8.5 |
| 16,500 to 17,499 | 7.5 | 8.7 |
| 17,500 to 18,499 | 5.8 | 9.2 |
| 18,500 to 19,499 | 7.9 | 9.3 |
| 19,500 to 20,499 | 6.4 | 7.8 |
| 20,500 to 21,499 | 6.7 | 8.2 |
| 21,500 to 22,499 | 6.5 | 8.6 |
| 22,500 to 24,499 | 5.9 | 9.0 |
| 24,500 to 26,499 | 7.5 | 8.2 |
| 26,500 to 28,499 | 6.0 | 8.2 |
| 28,500 to 32,499 | 5.8 | 8.3 |
| 32,500 to 36,000 | 6.6 | 7.2 |

[1] No data submitted.

### List of Subjects in 16 CFR Part 305

Advertising, Energy conservation, Household appliances, Labeling, Reporting and recordkeeping requirements.

Authority: Sec. 324 of the Energy Policy and Conservation Act (Pub. L. 94–163) (1975), as amended by the National Energy Conservation Policy Act (Pub. L. 95–619) (1978), 42 U.S.C. 6294; Sec. 553 of the Administrative Procedure Act, 5 U.S.C. 553.

Emily H. Rock,

*Secretary.*

[FR Doc. 84–28284 Filed 10–25–84; 8:45 am]

BILLING CODE 6750–01–M

[3] 45 FR 13998 (March 3, 1980), 45 FR 19520 (March 25, 1980), 45 FR 26036 (April 17, 1980), 46 FR 3829 (January 16, 1981).

[4] Reports for clothes washers are due by March 1; reports for water heaters, room air conditioners and furnaces are due by May 1; reports for dishwashers are due by June 1; reports for refrigerators, refrigerator-freezers and freezers are due by August 1.

## COMMODITY FUTURES TRADING COMMISSION

### 17 CFR Parts 1 and 16

### Commercial Categories for Option Traders

**AGENCY:** Commodity Futures Trading Commission.

**ACTION:** Rule related notice.

**SUMMARY:** On January 10, 1983, the Commission published in the **Federal Register** notification of the availability of its revised list of occupation categories (48 FR 1047). This list, as amended on February 3, 1984 (49 FR 4200) and October 15, 1984 (49 FR 48159), forms the basis from which the Commission will measure commercial participation in the pilot program for domestic exchange-traded commodity options. Futures commission merchants and members of contract markets are required under Commission rule 1.37(a), 17 CFR 1.37(a) (1982), to record for each option customer account they carry an appropriate occupation category from a list of such categories set forth by the Commission and a symbol indicating whether the option customer is commercial or noncommercial. In order to accommodate options on cotton, the Commission has determined to revise its current list of occupation categories by adding new categories for cotton, Categories 41 through 45. In addition, Category 28 has been revised to change the occupation category from "Other Elevator Operators or Merchants" to "Elevator Operator or Merchant Other Than a Producer Cooperative" and Category 34 has been revised to change the occupation category "Non-Farmer Livestock Feeder" to "Other Livestock Feeder" for purpose of clarification. Revisions have also been made to Categories 16, 24, 25, 27, 31, 32, 33, 34, 35, 36, 37 and 38 to conform all categories to the singular form.

As is the case with the existing categories, the appropriate classification for a customer is based on the primary activity of the customer in using the option market in conjunction with its case market activities.

**FOR FURTHER INFORMATION CONTACT:** Fred Linse, Chief, Agricultural Commodities Unit, Division of Economic Analysis, (202) 254–7303, Commodity Futures Trading Commission, 2033 K Street, NW., Washington, D.C. 20581.

**SUPPLEMENTARY INFORMATION:** The Commission has revised the list of occupation categories for determining commercial participation in its pilot program for options as follows:

*Commodity and Occupation Categories*

Sugar:
1. Producer
2. Merchant or Dealer
3. Refiner
4. Manufacturer or Processor
5. Other Commercial

Precious Metals:
6. Producer
7. Refiner
8. Dealer
9. Commercial End User
11. Other Commercial [1]

Petroleum:
39. Crude Oil Producer
40. Crude Oil Reseller
12. Refiner
13. Product Marketer and/or Distributor
14. End User
15. Other Commercial

Financial Instruments/Foreign Exchange

[1] Category 10 intentionally blank.

16. Savings and Loan, Mortgage Bank or Thrift Institution
17. Commercial Bank
18. Insurance Company
19. Pension and Retirement Fund
20. Mutual Fund
21. Broker/Dealer
22. Foundation or Endowment
23. Other Commercial
24. Importer/Exporter of Goods and Services
25. Investor/Issuer of Foreign Currency Denominated Securities

Grains and Soybeans:
26. Grain or Soybean Producer
27. Producer Cooperative
28. Elevator Operator or Merchant Other Than a Producer Cooperative
29. Processor, Including Feed Manufacturing and Crushing
30. Livestock Feeder or Producer
31. Other Commercial

Livestock:
32. Farmer or Rancher
33. Commercial Feedlot Operator
34. Other Livestock Feeder
35. Marketing Agency and/or Commission Merchant
36. Packer or Other Meat Processor
37. Meat Wholesaler, Retailer, or Buyer
38. Other Commercial

Cotton:
41. Producer
42. Producer Cooperative
43. Merchant
44. Mill Operator
45. Other Commercial

Issued in Washington, D.C. on October 24, 1984.

Jean A. Webb,

*Acting Secretary of the Commission.*

[FR Doc. 84-28441 Filed 10-25-84; 8:45 am]

BILLING CODE 6351-01-M

## DEPARTMENT OF THE TREASURY

### Customs Service

### 19 CFR Part 4

[T.D. 84-215]

## Customs Regulations Amendment Adding Denmark to List of Nations Whose Pleasure Vessels Are Entitled To Be Issued U.S. Cruising Licenses

**AGENCY:** Customs Service, Treasury.

**ACTION:** Final rule.

**SUMMARY:** This document amends the Customs Regulations by adding Denmark to the list of nations whose pleasure vessels may be issued U.S. cruising licenses. Customs has been informed that yachts used and employed exclusively as pleasure vessels and belonging to any resident of the U.S. are allowed to arrive at and depart from Danish ports and cruise in the waters of Denmark without being subjected to formal entry and clearance procedures. Therefore, Customs is extending reciprocal privileges to pleasure vessels belonging to any resident of Denmark.

**EFFECTIVE DATE:** These privileges became effective for Denmark on August 31, 1984.

**FOR FURTHER INFORMATION CONTACT:** Paul Hegland, Carriers, Drawback and Bonds Division (202-566-5706), U.S. Customs Service, 1301 Constitution Avenue, NW., Washington, D.C. 20229.

**SUPPLEMENTARY INFORMATION:**

### Background

Section 4.94(a), Customs Regulations (19 CFR 4.94(a)), provides that U.S. vessels documented as yachts, used exclusively for pleasure, not engaged in any trade, and not violating the Customs or navigation laws of the U.S. may proceed from port to port in the U.S. or to foreign ports without entering and clearing, as long as they have not visited hovering vessels.

Generally, foreign-flag yachts entering the U.S. are required to comply with the laws applicable to foreign vessels arriving at, departing from, and proceeding between ports of the U.S. However, as provided in § 4.94(b), Customs Regulations (19 CFR 4.94(b)), pleasure vessels from certain countries may be issued cruising licenses which exempt them from formal entry and clearance procedures (e.g., filing manifests, obtaining permits to proceed and paying entry and clearance fees) at all but the first port of entry in the U.S. Yachts or pleasure vessels not carrying passengers or merchandise in trade are exempt from paying tonnage tax and light money in any case pursuant to § 4.21(b)(5), Customs Regulations (19 CFR 4.21(b)(5)). Cruising licenses are available to pleasure vessels of countries which extend reciprocal privileges to U.S. pleasure vessels. A list of these countries is set forth in § 4.94(b).

By letter dated August 17, 1984, the Embassy of Denmark, in Washington, D.C., informed the Department of State, which in turn informed Customs Headquarters by a letter dated August 30, 1984, that the Government of Denmark permits yachts used and employed exclusively as pleasure vessels and belonging to any resident of the U.S., to arrive at and depart from ports of Denmark and cruise the waters of Denmark without entering and clearing Danish Customs, and without the payment of any charges for entering or clearing, dues, duty per ton, tonnage taxes or charges for cruising licenses. The State Department and the Carriers, Drawback and Bonds Division of Customs are of the opinion that satisfactory evidence has been furnished to establish the reciprocity required in § 4.94(b). Therefore, on September 21, 1984, the Director of that division determined that, effective retroactively to August 31, 1984, Denmark should be added to the list of countries set forth in § 4.94(b).

By virtue of the authority vested in the President by section 5 of the Act of May 28, 1908, 35 Stat. 425, as amended (46 U.S.C. 104), the President has delegated the authority to issue these cruising licenses to the Secretary of the Treasury by E.O. 10289, September 17, 1951. By Treasury Department Order 165-25, the Secretary of the Treasury delegated authority to the Commissioner of Customs to prescribe regulations relating to § 4.94(b) and other sections of the Customs Regulations relating to lists of nations entitled to preferential treatment in Customs matters because of reciprocal privileges accorded to vessels and aircraft of the U.S. Subsequently, by Customs Delegation Order No. 66 (T.D. 82-201), dated October 13, 1982, the Commissioner delegated authority to issue these cruising licenses and to amend this section to the Assistant Commissioner (Commercial Operations), who redelegated this authority to the Director, Office of Regulations and Rulings, who then redelegated it to the Director, Regulations Control and Disclosure Law Division.

### Finding

On the basis of the information received from the Embassy of Denmark and the Department of State, as described above, it has been determined that the U.S. is in possession of satisfactory evidence regarding the passage of U.S. pleasure vessels through the ports and waters of Denmark without their being subjected to formal entry and clearance procedures. Therefore, Denmark is added to the list of countries whose pleasure vessels may be issued U.S. cruising licenses.

### Inapplicability of Public Notice and Delayed Effective Date Requirements

Because this amendment merely

implements a statutory requirement and involves a matter in which the majority of the public is not particularly interested, pursuant to 5 U.S.C. 553(b)(B), notice and public procedure thereon are unnecessary. Further, for the same reasons good cause exists for dispensing with a delayed effective date under 5 U.S.C. 553(d)(1).

**Inapplicability of the Regulatory Flexibility Act**

This document is not subject to the provisions of sections 603 and 604 of Title 5, United States Code, as added by section 3 of Pub. L. 96-354, the "Regulatory Flexibility Act". That Act does not apply to any regulations such as this for which a notice of proposed rulemaking is not required by the Administrative Procedure Act (5 U.S.C. 551 *et seq.*) or any other statute.

**Executive Order 12291**

This amendment does not meet the criteria for a major regulation as defined in section 1(b) of E.O. 12291. Accordingly, a major impact analysis is not required.

**Drafting Information**

The principal author of this document was John E. Doyle, Regulations Control Branch, Office of Regulations and Rulings, U.S. Customs Service. However, personnel from other Customs offices participated in its development.

**List of Subjects in 19 CFR Part 4**

Customs inspection and duties, Imports, Vessels, Yachts.

**Regulations Amendment**

**PART 4—[AMENDED]**

**§ 4.94 [Amended]**

To reflect this change, § 4.94(b), Customs Regulations (19 CFR 4.94(b)), is amended by inserting, in alphabetical order between "Canada" and "Germany, Federal Republic of", the word "Denmark", to the list of countries whose yachts or pleasure vessels may be issued U.S. cruising licenses.

(R.S. 251, as amended, section 3, 23 Stat. 119, as amended, section 5, 35 Stat. 425, as amended (5 U.S.C. 301, 19 U.S.C. 66, 624, 46 U.S.C. 3, 104))

Dated: October 19, 1984.

**Marvin M. Amernick,**

*Acting Director, Regulations Control & Disclosure Law Division.*

[FR Doc. 84-28308 Filed 10-25-84; 8:45 am]

**BILLING CODE 4820-02-M**

**DEPARTMENT OF HEALTH AND HUMAN SERVICES**

**Food and Drug Administration**

**21 CFR Part 177**

**[Docket No. 83F-0027]**

## Indirect Food Additives; Polymers; Perfluorocarbon Cured Elastomers

**AGENCY:** Food and Drug Administration.

**ACTION:** Final rule.

**SUMMARY:** The Food and Drug Administration (FDA) is amending the food additive regulations to provide for the safe use of perfluorocarbon cured elastomers as articles or components of articles intended for repeated use in contact with food. This action responds to a petition filed by E. I. duPont de Nemours & Co., Inc.

**DATES:** Effective October 26, 1984; objections by November 26, 1984. The Director of the **Federal Register** approves the incorporation by reference of certain publications at 21 CFR 177.2400, effective on October 26, 1984.

**ADDRESS:** Written objections to the Dockets Management Branch (HFA-305), Food and Drug Administration, Rm. 4-62, 5600 Fishers Lane, Rockville, MD 20857.

**FOR FURTHER INFORMATION CONTACT:** Marvin D. Mack, Center for Food Safety and Applied Nutrition (HFF-334), Food and Drug Administration, 200 C St. SW., Washington, D.C. 20204, 202-472-5740.

**SUPPLEMENTARY INFORMATION:** In a notice published in the **Federal Register** of March 29, 1983 (48 FR 13099), FDA announced that a food additive petition (FAP 3B3683), had been filed by E. I. duPont de Nemours & Co., Inc., Wilmington, DE 19898, proposing that the food additive regulations be amended to provide for the safe use of perfluorocarbon cured elastomers as articles or components of articles intended for repeated use in contact with food.

FDA has evaluated data in the petition and other relevant material and concludes that the proposed use of the food additive is safe and that the regulations should be amended as set forth below.

In accordance with § 171.1(h) (21 CFR 171.1(h)), the petition and the documents that FDA considered and relied upon in reaching its decision to approve the petition are available for inspection at the Center for Food Safety and Applied Nutrition (address above) by appointment with the information contact person listed above. As provided in § 171.1(h) (21 CFR 171.1(h)), the agency will delete for from the documents any materials that are not available public disclosure before making the documents available for inspection.

The agency has carefully considered the potential environmental effects of this action and has concluded that the action will not have a significant impact on the human environment and that an environmental impact statement is not required. The agency's finding of no significant impact and the evidence supporting that finding may be seen in the Dockets Management Branch (address above) between 9 a.m. and 4 p.m., Monday through Friday.

**List of Subjects in 21 CFR Part 177**

Food additives, Incorporation by reference, Polymeric food packaging.

Therefore, under the Federal Food, Drug, and Cosmetic Act (secs. 201(s), 409, 72 Stat. 1784-1788 as amended (21 U.S.C. 321(s), 348)) and under authority delegated to the Commissioner of Food and Drugs (21 CFR 5.10) and redelegated to the Center for Food Safety and Applied Nutrition (21 CFR 5.61), Part 177 is amended in Subpart C by adding new § 177.2400 to read as follows:

**PART 177—INDIRECT FOOD ADDITIVES: POLYMERS**

**§ 177.2400 Perfluorocarbon cured elastomers.**

Perfluorocarbon cured elastomers identified in paragraph (a) of this section may be safely used as articles or components of articles intended for repeated use in contact with nonacid food (pH above 5.0), subject to the provisions of this section.

(a) *Identity.* (1) For the purpose of this section, perfluorocarbon cured elastomers are produced by terpolymerizing tetrafluorethylene (CAS Reg. No. 116-14-3), perfluoromethyl vinyl ether (CAS Reg No. 1187-93-5), and perfluoro-2-phenoxypropyl vinyl ether (CAS Reg. No. 24520-19-2) and subsequent curing of the terpolymer (CAS Reg. No. 26658-70-8) using the crosslinking agent, phenol, 4,4'-[2,2,2-trifluoro-1-(trifluoromethyl) ethylidene] bis-,dipotassium salt (CAS Reg. No. 25088-69-1) and accelerator, 1,4,7,10,13,16-hexaoxacyclooctadecane (CAS Reg. No. 17455-13-9).

(2) The Perfluorocarbon base polymer shall contain no less than 40 weight-percent of polymer units derived from tetrafluoroethylene, no less than 40 weight-percent of polymer units derived from perfluoromethyl vinyl ether and no more than 5 weight-percent polymer

units derived from perfluoro-2-phenoxypropyl vinyl ether.

(3) The composition limitations of the cured elastomer, calculated as parts per 100 parts of terpolymer, are as follows: Phenol, 4,4'-[2,2,2-trifluoro-1-(trifluoromethyl)-ethylidene] bis-, dipotassium salt—not to exceed 5 parts. 1,4,7,10,13,16-Hexaoxacyclooctadecane—not to exceed 5 parts.

(b) *Optional adjuvant substances.* The perfluorocarbon cured elastomer identified in paragraph (a) of this section may contain the following optional adjuvant substances, subject to any limitations cited on their use:

(1) Substances generally recognized as safe (GRAS) in food or food packaging.

(2) Substances used in accordance with a prior sanction.

(3) Substance authorized under applicable regulations in this part and in Parts 175 and 178 of this chapter and subject to any limitations prescribed therein.

(4) Substances identified in this paragraph (b)(4) subject to such limitations as are provided:

| Substances | Limitations |
|---|---|
| Carbon black (channel process of furnace combustion process) (CAS Reg. No. 1333-86-4). | Not to exceed 15 parts per 100 parts of the terpolymer. |
| Magnesium oxide (CAS Reg. No. 1309-48-4). | Not to exceed 5 parts per 100 parts of the terpolymer. |

(c) *Specifications*—(1) *Infrared identification.* Perfluorocarbon cured elastomers may be identified by the characteristic infrared spectra of the pyrolysate breakdown product that is obtained by heating and decomposing the elastomer using the method entitled "Qualitative Identification of Kalrez® by Infrared Examination of Pyrolysate." This method is incorporated by reference. Copies of the method are available from the Division of Food and Color Additives, Center for Food Safety and Applied Nutrition (HFF–330), Food and Drug Administration, 200 C St. SW., Washington, DC 20204, or available for inspection at the Office of the Federal Register, 1100 L St. NW., Washington, DC 20408.

(2) *Thermogravimetry.* Perfluorocarbon cured elastomers have a major decomposition peak occurring at 490 °±15 °C (914 °F). Less than 1.5 percent of the elastomers will volatize below 400 °C (752 °F) when run under nitrogen at a 10 °C or 18 °F per minute heating rate using a Du Pont Thermal Analyzer Model 1099 with Model 951 TGA unit or the equivalent.

(d) *Extractive limitations.* Articles fabricated from perfluorocarbon cured elastomers having a thickness of at least 1.0 millimeter (0.039 inch) when extracted at reflux temperatures for 2 hours separately with distilled water, 50 percent ethanol, and *n*-heptane, shall meet the following extractability limits:

(1) Total extractives not to exceed 3.1 milligrams per square decimeter (0.2 milligrams per square inch).

(2) Fluoride extractives calculated as fluorine not to exceed 0.47 milligram per square decimeter (0.03 milligram per square inch).

(e) *Conditions of use.* In accordance with current good manufacturing practice, finished food contact articles containing the perfluorocarbon cured elastomers shall be thoroughly cleaned prior to their first use in contact with food.

Any person who will be adversely affected by the foregoing regulation may at any time on or before November 26, 1984 submit to the Dockets Management Branch (address above) written objections thereto and may make a written request for a public hearing on the stated objections. Each objection shall be separately numbered and each numbered objection shall specify with particularity the provision of the regulation to which objection is made. Each numbered objection on which a hearing is requested shall specifically so state; failure to request a hearing for any particular objection shall constitute a waiver of the right to a hearing on that objection. Each numbered objection for which a hearing is requested shall include a detailed description and analysis of the specific factual information intended to be presented in support of the objection in the event that a hearing is held; failure to include such a description and analysis for any particular objection shall constitute a waiver of the right to a hearing on the objection. Three copies of all documents shall be submitted and shall be identified with the docket number found in brackets in the heading of this regulation. Received objections may be seen in the office above between 9 a.m. and 4 p.m., Monday through Friday.

*Effective date.* This regulation is effective October 26, 1984.

(Secs. 201(s), 409, 72 Stat. 1784–1788 as amended (21 U.S.C. 321(s), 348))

Dated: October 9, 1984.

**Richard J. Ronk,**

*Acting Director, Center for Food Safety and Applied Nutrition.*

[FR Doc. 84-27617 Filed 10-25-84; 8:45 am]

BILLING CODE 4160-01-M

## 21 CFR Part 520

## Oral Dosage Form New Animal Drugs Not Subject to Certification; Cefadroxil Tablets

**AGENCY:** Food and Drug Administration.

**ACTION:** Final rule.

**SUMMARY:** The Food and Drug Administration (FDA) is amending the animal drug regulations to reflect approval of a supplemental new animal drug application (NADA) filed by Bristol Laboratories providing for safe and effective use of cefadroxil tablets for treating certain genitourinary tract infections of dogs.

**EFFECTIVE DATE:** October 26, 1984.

**FOR FURTHER INFORMATION CONTACT:** Sandra K. Woods, Center for Veterinary Medicine (HFV–114), Food and Drug Administration, 5600 Fishers Lane, Rockville, MD 20857, 301–443–3420.

**SUPPLEMENTARY INFORMATION:** Bristol Laboratories, Division of Bristol-Meyers Co., P.O. Box 657, Syracuse, NY 13201, filed a supplemental NADA 119–688 providing for safe and effective oral use of 50-, 100-, and 200-milligram cefadroxil tablets as a canine antibacterial for treating genitourinary tract infections (cystitis) caused by susceptible strains of *Escherichia coli, Proteus mirabilis,* and *Staphylococcus aureus* in addition to its approved use for the treatment of certain skin and soft tissue infections caused by susceptible strains of *Staphylococcus aureus.* The supplemental NADA is approved and the regulations are amended to reflect the approval. The basis for approval is discussed in the freedom of information summary.

In accordance with the freedom of information provisions of Part 20 (21 CFR Part 20) and § 514.11(e)(2)(ii) (21 CFR 514.11(e)(2)(ii)), a summary of safety and effectiveness data and information submitted to support approval of this application may be seen in the Dockets Management Branch (HFA–305), Food and Drug Administration, Rm. 4–62, 5600 Fishers Lane, Rockville, MD 20857, from 9 a.m. to 4 p.m., Monday through Friday.

The Center for Veterinary Medicine has carefully considered the potential environmental effects of this action and has concluded that the action will not have a significant impact on the human environment and that an environmental impact statement therefore will not be prepared. The Center's finding of no significant impact and the evidence supporting this finding, contained in an environmental assessment (pursuant to 21 CFR 25.31, proposed December 11,

1979; 44 FR 71742) may be seen in the Dockets Management Branch (address above) between 9 a.m. and 4 p.m., Monday through Friday.

**List of Subjects in 21 CFR Part 520**

Animal drugs, Oral use.

Therefore, under the Federal Food, Drug, and Cosmetic Act (sec. 512(i), 82 Stat. 347 (21 U.S.C. 360b(i))) and under authority delegated to the Commissioner of Food and Drugs (21 CFR 5.10) and redelegated to the Center for Veterinary Medicine (21 CFR 5.83), Part 520 is amended in § 520.314 by revising paragraph (c) (1) and (3), to read as follows:

## PART 520—ORAL DOSAGE FORM NEW ANIMAL DRUGS NOT SUBJECT TO CERTIFICATION

**§ 520.314 Cefadroxil tablets.**

* * * * *

(c) * * *

(1) *Indications for use.* For the treatment of skin and soft tissue infections including cellulitis, pyoderma, dermatitis, wound infections, and abscesses due to susceptible strains of *Staphylococcus aureus.* For the treatment of genitourinary tract infections (cystitis) due to susceptible strains of *Escherichia coli, Proteus mirabilis,* and *Staphylococcus aureus.*

* * * * *

(3) *Limitations.* The drug is administered orally. For skin and soft tissue infections, treatment should be continued for a minimum of 3 days. For genitourinary tract infections, treatment should be continued for a minimum of 7 days. Continue treatment at least 48 hours after the dog has become afebrile or asymptomatic. If no response is seen after 3 days of treatment, therapy should be discontinued and the case reevaluated. Do not treat for more than 30 days. Safety for use in pregnant bitches and stud dogs has not been determined. Federal law restricts this drug to use by or on the order of a licensed veterinarian.

*Effective date.* October 26, 1984.

(Sec. 512(i), 82 Stat. 347 (21 U.S.C. 360b(i)))

Dated: October 16, 1984.

**Lester M. Crawford,**

*Director, Center for Veterinary Medicine.*

[FR Doc. 84-28287 Filed 10-25-84; 8:45 am]

BILLING CODE 4160-01-M

## DEPARTMENT OF THE TREASURY

### Internal Revenue Service

### 26 CFR Part 1

**[T.D. 7989]**

## Effective Date for Regulations Relating to Diversification Requirements for Variable Annuity, Endowment, and Life Insurance Contracts

**AGENCY:** Internal Revenue Service, Treasury.

**ACTION:** Temporary regulations.

**SUMMARY:** This document contains temporary regulations providing the effective date for forthcoming temporary regulations relating to diversification requirements for variable annuity, endowment, and life insurance contracts. Changes to the applicable tax law were made by the Tax Reform Act of 1984. The regulations will generally affect issuers and purchasers of variable contracts in taxable years beginning after December 31, 1983.

**DATES:** The regulations provide that forthcoming temporary regulations relating to diversification requirements for variable annuity, endowment, and life insurance contracts will be effective, generally, for taxable years beginning after December 31, 1983.

**FOR FURTHER INFORMATION CONTACT:** Linda M. Kroening of the Legislation and Regulations Division, Office of Chief Counsel, Internal Revenue Service, 1111 Constitution Avenue NW., Washington, D.C. 20224 (Attention: CC:LR:T) (202-566-3238, not a toll-free call).

**SUPPLEMENTARY INFORMATION:**

**Background**

This document amends the Income Tax Regulations (26 CFR Part 1) to provide the effective date for forthcoming temporary regulations relating to diversification requirements for variable annuity, endowment, and life insurance contracts under section 817(h) of the Internal Revenue Code of 1954 as added by section 211(a) of the Tax Reform Act of 1984 (Pub. L. 98-369, 98 Stat. 750).

Section 817(h) provides that a variable contract, other than a contract issued in connection with certain employee benefit plans, which is based on a segregated asset account shall not be treated as an annuity, endowment, or life insurance contract for any period for which the investments made by such account are not adequately diversified. The investments made by a segregated account will be adequately diversified if the account meets a safe-harbor test based on the diversification requirements for regulated investment companies under section 851(b)(4) or the account satisfies diversification requirements provided by regulations.

These temporary regulations provide that the forthcoming temporary regulations under section 817(h) will be effective, generally, for taxable years beginning after December 31, 1983. If an insurance company would be considered the owner of the assets of a segregated asset account under the principles of Rev. Rul. 81-225, however, the forthcoming temporary regulations will not apply to such account until 90 days after the publication of such regulations in the **Federal Register.**

**Special Analyses**

The Commissioner of Internal Revenue has determined that this temporary rule is not a major rule as defined in Executive Order 12291. Accordingly, a Regulatory Impact Analysis is not required. No general notice of proposed rulemaking is required by 5 U.S.C. 553 for temporary Regulations. Accordingly, a Regulatory Flexbility Analysis is not required (5 U.S.C. Chapter 6).

**Drafting Information**

The principal author of these regulations is Linda M. Kroening of the Legislation and Regulations Division of the Office of Chief Counsel, Internal Revenue Service. However, personnel from other offices of the Internal Revenue Service and Treasury Department participated in developing the regulations on matters of both substance and style.

**List of Subjects in 26 CFR 1.801-1-1.832-6**

Income taxes, Insurance companies.

**Amendments to the Regulations**

**PART 1—[AMENDED]**

The amendments to 26 CFR Part 1 are as follows:

**Paragraph.** New § 1.817-5T is added immediately after § 1.817-4 to read as follows:

**§ 1.817-5T Effective date for regulations relating to diversification requirements for variable annuity, endowment, and life insurance contracts (temporary).**

(a) *In general.* Any temporary regulations issued under the authority of section 817(h), relating to the diversification requirements for variable annuity, endowment, and life insurance

contracts, will be effective for taxable years beginning after December 31, 1983.

(b) *Exception.* If an insurance company would be considered the owner of the assets of a segregated asset account under the principles of Rev. Rul. 81–225, 1981–2 C.B. 12, at all times after the later of December 31, 1983, or the date on which the segregated asset account was established, the temporary regulations described in paragraph (a) of this section will not apply to such account until 90 days after their publication in the **Federal Register.**

There is need for immediate guidance with respect to the provisions contained in this Treasury decision. For this reason, it would be impractical to issue it first under the notice and comment procedure provided in 5 U.S.C. 553(b) or subject to the effective date limitation of 5 U.S.C. 553(d).

This Treasury decision is issued under the authority contained in sections 817(h) and 7805 of the Internal Revenue Code of 1954 (98 Stat. 750, 26 U.S.C. 817(h); 68A Stat. 917, 26 U.S.C. 7805).

**Roscoe L. Egger, Jr.,**

*Commissioner of Internal Revenue.*

Approved: October 15, 1984.

**Ronald A. Pearlman,**

*Acting Assistant Secretary of the Treasury.*

[FR Doc. 84–28384 Filed 10–25–84; 8:45 am]

BILLING CODE 4830–01–M

---

## Bureau of Alcohol, Tobacco and Firearms

## 27 CFR Part 9

**[T.D. ATF–189; Re: Notice No. 524]**

## Establishment of Sonoita Viticultural Area

**AGENCY:** Bureau of Alcohol, Tobacco and Firearms, Treasury.

**ACTION:** Final rule, Treasury Decision.

**SUMMARY:** This final rule establishes a viticultural area in the State of Arizona to be known as "Sonoita." The Bureau of Alcohol, Tobacco and Firearms (ATF) believes that the establishment of the Sonoita viticultural area and the subsequent use of its name in wine labeling and advertising will enable winemakers to label wines more precisely and will help consumers to better identify the wines they purchase.

**EFFECTIVE DATE:** November 26, 1984.

**FOR FURTHER INFORMATION CONTACT:** Steve Simon, FAA, Wine and Beer Branch, Bureau of Alcohol, Tobacco and Firearms, 1200 Pennsylvania Avenue, NW, Washington, DC 20226 (202–566–7626).

**SUPPLEMENTARY INFORMATION:**

### Background

ATF regulations in 27 CFR Part 4 provide for the establishment of definite viticultural areas. The regulations also allow the name of an approved viticultural area to be used as an appellation of origin on wine labels and in wine advertisements.

Part 9 of 27 CFR provides for the listing of approved American viticultural areas, the names of which may be used as appellations of origin.

Section 4.25a(e)(1), Title 27, CFR, defines an American viticultural area as a delimited grape-growing region distinguishable by geographical features. Section 4.25a(e)(2) outlines the procedure for proposing an American viticultural area. Any interested person may petition ATF to establish a grape-growing region as a viticultural area.

### Notice of Proposed Rulemaking

ATF received a petition from Mr. A. Blake Brophy of the Babocamari Ranch Company, proposing an area near Sonoita, Arizona, as a viticultural area to be known as "Sonoita." In response, ATF published a notice of proposed rulemaking, Notice No. 524, in the **Federal Register** on May 16, 1984 (49 FR 20730). That notice proposed establishment of the "Sonoita" viticultural area and solicited public comment with respect to the proposed viticultural area.

The area contains about 325 square miles. It is located in extreme southern Arizona, near the Mexican border. There are about 40 acres of grapes currently planted in the proposed area. The petitioner states that plans call for 360 additional acres to be planted. A winery is currently under construction. Soils in the area that are suitable for wine-grape production include the White House-Bernardino-Hathaway and the Caralumpi-Hathaway associations. Grapes are being grown on the floor of the proposed viticultural area at altitudes of between 4,500 feet and 5,000 feet.

### Comments

Four public comments were received in response to the notice of proposed rulemaking, Notice No. 524. Two of the public comments were letters of complete support for the proposed area. The other two, however, were from winemakers in Sonoma County, California, who expressed their concern that the proposed name, "Sonoita," could be confused with either "Sonoma County" or "Sonoma Valley," which are already established as appellations of origin for wine.

Therefore, those commenters advocated that another name be chosen to designate this viticultural area. (One of them suggested either "Sonoita-Arizona" or "Arizona Sonoita.")

In support of their contention, one of them pointed out that "5 of the 6 letters in SONOMA are used in SONOITA, in the *EXACT PLACE AND ORDER* in each of the two words. Phonetically the words sound similar. Potentially, the printed words are so similar that confusion between the words could result."

ATF does not agree that there would be a significant potential for consumer confusion with the name "Sonoita." Despite some similarity, "Sonoita" and "Sonoma" are readily distinguishable. More importantly, the two authorized appellations using the word "Sonoma" must be accompanied by a second word (either "County" or "Valley"); consequently, they are not likely to be confused with the single word "Sonoita" standing alone.

### Evidence for the Name "Sonoita"

The following evidence was submitted by the petitioner to show that the viticultural area is known by the name of "Sonoita," and that this name is associated with grape growing in the area:

(a) "Sonoita" is the name of the only viable community in the area. (The town of Sonoita is centrally located within the viticultural area.)

(b) Historically, the name "Sonoita" is derived from a *visita* established in 1691 by the missionary-explorer, Father Eusebio Francisco Kino. At that time, the name given to this small settlement of Sobaipuri Indians was "Los Santos Reyes de Sonoita."

(c) Since 1975, the Babocamari Ranch Company has been cooperating with the University of Arizona in the growing of *vitis vinifera* grapes in the area and in the making of wine from those grapes. These efforts have been described in an article in the American Journal of Enology and Viticulture, Vol. 32, No. 4, pp. 290–296, entitled "The Use of Soils for the Delineation of Viticultural Zones in the Four Corners Region." This article calls the proposed area "Sonoita"; for example: "Other sites such as Sonoita * * * produce much better fruit than expected" (p. 291).

For these reasons, and considering that there is no expectation of consumer confusion, ATF has determined that the name proposed by the petitioner—"Sonoita"—is the best name for this viticultural area.

**Geographical Description of the Area**

Topographically, the area is separated from the surrounding areas by three major mountain ranges: The Santa Rita Mountains, the Huachuca Mountains, and the Whetstone Mountains. These mountains rise from 2,500 to 4,500 feet above the floor of the viticultural area.

The "old-timers" used to call the area "Sonoita Valley," because it resembles a valley in appearance. But geologically, the area is technically a basin rather than a valley, because it comprises the headwaters for three distinct drainages: Sonoita Creek to the south, Cienega Creek to the north, and the Babocamari River to the east. (In technical geological terms, a "valley" would comprise only a single drainage.)

The most obvious geographical distinction to the area is that, in its native state, it is classified as "high desert grassland," while the surrounding terrain is either mountain or woody-shrub desert. (See Humphrey, Robert R., *The Desert Grassland,* University of Arizona Press.)

The boundaries of the Sonoita viticultural area may be found on seven U.S.G.S. quadrangle maps in the 15 minute series: Benson, Fort Huachuca, Sunnyside, Elgin, Lochiel, Mount Wrightson, and Empire Mountains. The boundaries are described in 27 CFR § 9.97, as added by this document.

**Miscellaneous**

ATF does not wish to give the impression by approving "Sonoita" as a viticultural areas that it is approving or endorsing the quality of the wine from this area. ATF is approving this area as being distinct and not better than other areas. By approving this area, wine producers are allowed to claim a distinction on labels and advertisements as to the origin of the grapes. Any commercial advantage gained can only come from consumer acceptance of "Sonoita" wines.

**Regulatory Flexibility Act**

The provisions of the Regulatory Flexibility Act relating to an initial and final regulatory flexibility analysis (5 U.S.C. 603, 604) are not applicable to this final rule because it will not have a significant economic impact on a substantial number of small entities. The final rule is not expected to have a significant secondary or incidental effects on a substantial number of small entities, or impose, or otherwise cause, a significant increase in the reporting, recordkeeping, or other compliance burdens on a substantial number of small entities.

Accordingly, it is hereby certified under the provisions of Section 3 of the Regulatory Flexibility Act (5 U.S.C. 605(b)), that this final rule will not have a significant economic impact on a substantial number of small entities.

**Executive Order 12291**

In compliance with Executive Order 12291 of Feb. 17, 1981, the Bureau has determined that this final rule is not a major rule since it will not result in:

(a) An annual effect on the economy of $100 million or more;

(b) A major increase in costs or prices for consumers, individual industries, Federal, State, or local government agencies, or geographic regions; or

(c) Significant adverse effects on competition, employment, investment, productivity, or on the ability of United States-based enterprises to compete with foreign-based enterprises in domestic or export markets.

**Paperwork Reduction Act**

The provisions of the Paperwork Reduction Act of 1980, Pub. L. 96–511, 44 U.S.C. Chapter 35, and its implementing regulations, 5 CFR Part 1320, do not apply to this final rule because no requirement to collect information is imposed.

**List of Subjects in 27 CFR Part 9**

Administrative practice and procedures, Consumer protection, Viticultural areas, Wine.

**Drafting Information**

The principal author of this document is Steve Simon, FAA, Wine and Beer Branch, Bureau of Alcohol, Tobacco and Firearms.

**Authority and Issuance**

Accordingly, under the authority in 27 U.S.C. 205, the regulations in 27 CFR Part 9 are amended as follows:

## PART 9—AMERICAN VITICULTURAL AREAS

**Paragraph 1.** The table of sections in 27 CFR Part 9, Subpart C, is amended to add the title of § 9.97, to read as follows:

* * * * *

**Subpart C—Approved American Viticultural Areas**

Sec.

* * * * *

9.97 Sonoita.

**Par. 2.** Subpart C of 27 CFR Part 9 is amended by adding § 9.97, which reads as follows:

**§ 9.97 Sonoita.**

(a) *Name.* The name of the viticultural area described in this section is "Sonoita."

(b) *Approved maps.* The appropriate maps for determining the boundaries of Sonoita viticultural area are seven U.S.G.S. maps. They are titled:

(1) Benson Quadrangle, 15 minute series, 1958.

(2) Fort Huachuca Quadrangle, 15 minute series, 1958.

(3) Elgin Quadrangle, 15 minute series, 1958.

(4) Lochiel Quadrangle, 15 minute series, 1958.

(5) Mount Wrightson Quadrangle, 15 minute series, 1958.

(6) Sunnyside Quadrangle, 15 minute series, 1958.

(7) Empire Mountains Quadrangle, 15 minute series, 1958.

(c) *Boundary*—(1) *General.* The Sonoita viticultural area is located in Arizona. The starting point of the following boundary description is the summit of Mount Wrightson (9,543 feet) in the Santa Rita Mountains.

(2) *Boundary Description*—(i) From the starting point southeastward in a straight line for approximately 24 miles, to the summit of Lookout Knob (6,171 feet) in the Canelo Hills.

(ii) From there in a straight line eastward for approximately 10 miles, to the summit of Huachuca Peak (8,410 feet) in the Huachuca Mountains.

(iii) From there north-northwestward for approximately 21 miles in a straight line to the summit of Granite Peak (7,413 feet) in the Whetstone Mountains.

(iv) From there west-southwestward in a straight line for approximately 26 miles, to the summit of Mount Wrightson (the point of beginning).

Signed: September 24, 1984.

**Stephen E. Higgins,**

*Director.*

Approved: October 17, 1984.

**Edward T. Stevenson,**

*Deputy Assistant Secretary (Operations).*

[FR Doc. 84–28324 Filed 10–25–84; 8:45 am]

BILLING CODE 4810–31–M

## DEPARTMENT OF THE INTERIOR

### Office of Surface Mining Reclamation and Enforcement

### 30 CFR Part 925

### Notice of Extension of Deadline for Submission of Program Amendments to the Missouri Permanent Program

**AGENCY:** Office of Surface Mining Reclamation and Enforcement (OSM), Interior.

**ACTION:** Final rule.

**SUMMARY:** OSM is announcing its decision to extend the deadline for Missouri to (1) promulgate rules governing the training, examination and certification of blasters and (2) to develop and adopt a program to examine and certify all persons who are directly responsible for the use of explosives in a surface coal mining operation. On August 6, 1984, Missouri requested an extension of time until August 6, 1985, for the development of a blaster certification program. All States with regulatory programs approved under the Surface Mining Control and Reclamation Act of 1977 (SMCRA or the Act) are required to develop and adopt a blaster certification program by March 4, 1984. Section 850.12(b) of OSM's regulations provides that the Director, OSM, may approve an extension of time for a State to develop and adopt a program upon a demonstration of good cause. In accordance with the State's request, the Director is granting the State an extension of time until August 6, 1985, to submit a proposed blaster certification program.

**EFFECTIVE DATE:** October 26, 1984.

**FOR FURTHER INFORMATION CONTACT:** Mr. Richard D. Rieke, Director, Kansas City Field Office, Office of Surface Mining, 818 Grand Avenue, Kansas City, Missouri 64106; Telephone: (816) 374–5527.

### Supplementary Information

On March 4, 1983, OSM issued final rules effective April 14, 1983, establishing the Federal standards for the training and certification of blasters at 30 CFR Chapter M (48 FR 9486). Section 850.12 of these regulations stipulates that the regulatory authority in each State with an approved program under SMCRA shall develop and adopt a program to examine and certify all persons who are directly responsible for the use of explosives in a surface coal mining operations within 12 months after approval of a State program or within 12 months after publication date of OSM's rule at 30 CFR Part 850, whichever is later. In the case of Missouri's program, the applicable date is 12 months after publication date of OSM's rule, or March 4, 1984.

On August 6, 1984, Missouri advised OSM that it was requesting an extension of time until August 6, 1985, to develop and adopt a blaster certification program.

The August 6, 1984, letter from the Missouri Department of Natural Resources advised OSM that it would require additional time in order to receive and coordinate technical assistance from knowledgeable parties outside the State's staff. Additionally, the State indicated a need for the additional time to accomodate its regulatory process in accordance with certain procedures established by the Missouri Secretary of State.

In the August 31, 1984, **Federal Register** (49 FR 34532), OSM proposed an extension until August 6, 1985, for Missouri to submit to OSM a proposed blaster training program. Public comment on this proposal was sought for 30 days ending October 1, 1984. No public comments were received.

### Director's Determination

In accordance with the State's request, the Director has decided to extend the deadline for Missouri to submit a proposed blaster training program until August 6, 1985. This extension will allow Missouri to develop an instructional program so that the Missouri program will be consistent with the requirements of 30 CFR Part 850. Accordingly, 30 CFR 925.16 is being amended to reflect the Director's decision.

### VI. Additional Determinations

1. *Compliance with the National Environmental Policy Act:* The Secretary has determined that, pursuant to section 702(d) of SMCRA, 30 U.S.C. 1292(d), no environmental impact statement need be prepared on this rulemaking.

2. *Executive Order No. 12291 and the Regulatory Flexibility Act:* On August 28, 1981, the Office of Management and Budget (OMB) granted OSM an exemption from Sections 3, 4, 7, and 8 of Executive Order 12291 for actions directly related to approval or conditional approval of State regulatory programs. Therefore, this action is exempt from preparation of a Regulatory Impact Analysis and regulatory review by OMB.

The Department of the Interior has determined that this rule will not have a significant economic effect on a substantial number of small entities under the Regulatory Flexibility Act (5 U.S.C. 601 *et seq.*). This rule will not impose any new requirements; rather, it will ensure that existing requirements established by SMCRA and the Federal rules will be met by the State.

3. *Paperwork Reduction Act:* This rule does not contain information collection requirements which require approval by the Office of Management and Budget under 44 U.S.C. 3507.

### List of Subjects in 30 CFR Part 925

Coal mining, Intergovernmental relations, Surface mining, Underground mining.

Dated: October 22, 1984.

**John D. Ward,**

*Director, Office of Surface Mining.*

**Authority:** Pub. L. 95–87, Surface Mining Control and Reclamation Act of 1977 (30 U.S.C. 1201 *et seq.*).

### PART 925—MISSOURI

30 CFR Part 925 is amended by adding a new paragraph (i) to read as follows:

**§ 925.16 Required program amendments.**

* * * * *

(i) Pursuant to 30 CFR 732.17, Missouri is required to submit for OSM's approval the following proposed program amendments by the dates specified:

(1) By August 6, 1985, Missouri shall submit for OSM's approval

(i) rules governing the training, examination and certification of blasters and

(ii) a program to examine and certify all persons who are directly responsible for the use of explosives in surface coal mining operations.

[FR Doc. 84–28414 Filed 10–25–84; 8:45 am]

BILLING CODE 4310–05–M

## VETERANS ADMINISTRATION

### 38 CFR Part 36

### Decrease in Maximum Permissible Interest Rates on Guaranteed Manufactured Home Loans, Home and Condominium Loans, and Home Improvement Loans

**AGENCY:** Veterans Administration.

**ACTION:** Final regulations.

**SUMMARY:** The VA (Veterans Administration) is decreasing the maximum interest rates on guaranteed manufactured home unit loans, lot loans, and combination manufactured home unit and lot loans. In addition, the maximum interest rates applicable to fixed payment and graduated payment home and condominium loans, and to home improvement and energy conservation loans are also decreased.

These decreases in interest rates are possible because of recent improvements in the availability of funds in various credit markets. The decrease in the interest rates will allow eligible veterans to obtain loans at a lower monthly cost.

**EFFECTIVE DATE:** October 22, 1984.

**FOR FURTHER INFORMATION CONTACT:** Mr. George D. Moerman, Loan Guaranty Service (264), Department of Veterans Benefits, Veterans Administration, 810 Vermont Avenue, NW., Washington, D.C. 20420 (202–389–3042).

**SUPPLEMENTARY INFORMATION:** The Administrator is required by section 1819(f), title 38, United States Code, to establish maximum interest rates for manufactured home loans guaranteed by the VA as he finds the manufactured home loan capital markets demand. Recent market indicators—including the prime rate, the general decrease in interest rates charged on conventional manufactured home loans, and the decrease of other short-term and long-term interest rates—have shown that the manufactured home capital markets have improved. It is now possible to decrease the interest rates on manufactured home unit loans, lot loans, and combination manufactured home unit and lot loans while still assuring an adequate supply of funds from lenders and investors to make these types of VA loans.

The Administrator is also required by section 1803(c), title 38, United States Code, to establish maximum interest rates for home and condominium loans including graduated payment mortgage loans, and loans for home improvement purposes. Market indicators similarly favor reductions in the maximum interest rates for these types of loans. These lower interest rates should assist more veterans in the purchase of homes and condominiums or to obtain improvement loans because of the decrease in the monthly loan payments for principal and interest.

**Regulatory Flexibility Act/Executive Order 12291**

For the reasons discussed in the May 7, 1981 **Federal Register** (46 FR 25443), it has previously been determined that final regulations of this type which change the maximum interest rates for loans guaranteed, insured, or made pursuant to chapter 37 of title 38, United States Code, are not subject to the provisions of the Regulatory Flexibility Act, 5 U.S.C. 601–612.

These regulatory amendments have also been reviewed under the provisions of Executive Order 12291. The VA finds that they are not "major rules" as defined in that Order. The existing process of informal consultation among representatives within the Executive Office of the President, OMB, the VA and the Department of Housing and Urban Development has been determined to be adequate to satisfy the intent of this Executive Order for this category of regulations. This alternative consultation process permits timely rate adjustments with minimal risk of premature disclosure. In summary, this consultation process will fulfill the intent of the Executive Order while still permitting compliance with statutory responsibilities for timely rate adjustments and a stable flow of mortgage credit at rates consistent with the market.

These final regulations come within exceptions to the general VA policy of prior publication of proposed rules as contained in 38 CFR 1.12. The publication of notice of a regulatory change in the VA maximum interest rates for VA guaranteed, insured or direct loans would deny veterans the benefit of lower interest rates pending the final rule publication date which would necessarily be more than 30 days after publication in proposed form. Accordingly, it has been determined that publication of proposed regulations prior to publication of final regulations is impracticable, unnecessary, and contrary to the public interest.

(Catalog of Federal Domestic Assistance Program numbers 64.113, 64.114, and 64.119)

These regulations are adopted under authority granted to the Administrator by sections 210(c), 1803(c)(1), 1811(d)(1) and 1819 (f) and (g) of title 38, United States Code.

These decreases are accomplished by amending §§ 36.4212(a) (1), (2), and (3), and 36.4311 (a), (b), and (c) and 36.4503(a), Title 38, Code of Federal Regulations.

**List of Subjects in 38 CFR Part 36**

Condominiums, Handicapped, Housing, Loan programs—housing and community development, Manufactured homes, Veterans.

Approved: October 19, 1984.

**Harry N. Walters,**

*Administrator.*

## PART 36—LOAN GUARANTY

The Veterans Administration is amending 38 CFR Part 36 as follows:

1. In § 36.4212, paragraph (a) is revised as follows:

**§ 36.4212 Interest rates and late charges.**

(a) The interest rate charge the borrower on a loan guaranteed or insured pursuant to 38 U.S.C. 1819 may not exceed the following maxima except on loans guaranteed or insured pursuant to guaranty or insurance commitments issued by the Veterans Administration prior to the respective effective date: (38 U.S.C. 1819(f))

(1) Effective October 22, 1984, 15½ percent simple interest per annum for a loan which finances the purchase of a manufactured home unit only.

(2) Effective October 22, 1984, 15 percent simple interest per annum for a loan which finances the purchase of a lot only and the cost of necessary site preparation, if any.

(3) Effective October 22, 1984, 15 percent simple interest per annum for a loan which will finance the simultaneous acquisition of a manufactured home and a lot and/or the site preparation necessary to make a lot acceptable as the site for the manufactured home.

* * * * *

2. In § 36.4311, paragraphs (a), (b), and (c) are revised as follows:

**§ 36.4311 Interest rates.**

(a) Excepting loans guaranteed or insured pursuant to guaranty or insurance commitments issued by the VA which specify an interest rate in excess of 13 per centum per annum, effective October 22, 1984, the interest rate on any home or condominium loan, other than a graduated payment mortgage loan, guaranteed or insured wholly or in part on or after such date may not exceed 13 per centum per annum on the unpaid principal balance. (38 U.S.C. 1803(c)(1))

(b) Excepting loans guaranteed or insured pursuant to guaranty or insurance commitments issued by the VA which specify an interest rate in excess of 13¼ per centum per annum, effective October 22, 1984, the interest rate of any graduated payment mortgage loan guaranteed or insured wholly or in part on or after such date may not exceed 13¼ per centum per annum. (38 U.S.C. 1803(c)(1))

(c) Effective October 22, 1984, the interest rate on any loan solely for energy conservation improvements or other alterations, improvements or repairs, which is guaranteed or insured wholly or in part on or after such date may not exceed 14½ per centum per annum on the unpaid principal balance. (38 U.S.C. 1803(c)(1))

3. In § 36.4503, paragraph (a) is revised as follows:

**§ 36.4503 Amount and amortization.**

(a) The original principal amount of any loan made or after October 1, 1980,

shall not exceed an amount which bears the same ratio to $33,000 as the amount of the guaranty to which the veteran is entitled under 38 U.S.C. 1810 at the time the loan is made bears to $27,500. This limitation shall not preclude the making of advances, otherwise proper, subsequent to the making of the loan pursuant to the provisions of § 36.4511. Except as to home improvement loans, loans made by the VA shall bear interest at the rate of 13 percent per annum. Loans solely for the purpose of energy conservation improvements or other alterations, improvements, or repairs shall bear interest at the rate of 14½ percent per annum. (38 U.S.C. 1811 (d)(1) and (2)(A))

* * * * *

[FR Doc. 84-28282 Filed 10-25-84; 8:45 am]
BILLING CODE 8320-01-M

## ENVIRONMENTAL PROTECTION AGENCY

### 40 CFR Part 62

[A-7-FRL-2704-6; EPA No. 1513]

### Approval and Promulgation of State Plans for Designated Facilities and Pollutants; States of Iowa, Kansas and Nebraska

**AGENCY:** Environmental Protection Agency (EPA).

**ACTION:** Final rule.

**SUMMARY:** Section 111(d) of the Clean Air Act, as amended, requires states to adopt and submit plans to EPA for the control of emissions of certain pollutants at designated facilities. If there are no sources of a designated pollutant located in a state, the state may submit a negative declaration, i.e., a certification to that effect in lieu of a plan for control of the pollutant.

EPA has received negative declarations for kraft pulp mills from the States of Iowa and Nebraska, and primary aluminum reduction plants from the States of Kansas and Nebraska. EPA is taking action today to approve these negative declarations.

**EFFECTIVE DATE:** This action is effective December 26, 1984 unless notice is received within 30 days that someone wishes to submit adverse or critical comments.

**ADDRESSES:** Comments should be addressed to Robert J. Chanslor, Environmental Protection Agency, Region VII, Air Branch, 324 East 11th Street, Kansas City, Missouri 64106. Copies of the state submissions are available for inspection during normal business hours at the above address and at the following locations:

Iowa Department of Water, Air and Waste Management, Henry A. Wallace Building, 900 East Grand, Des Moines, Iowa 50319;

Kansas Department of Health and Environment, Forbes Field, Topeka, Kansas 66620;

Nebraska Department of Environmental Control, Air Pollution Control Division, State House Station, Box 94877, 301 Centennial Mall South, Lincoln, Nebraska 68509.

**FOR FURTHER INFORMATION CONTACT:** Robert J. Chanslor at (816) 374-3791, FTS 758-3791.

**SUPPLEMENTARY INFORMATION:** Section 111(d) of the Clean Air Act requires states to prepare and submit plans to control certain pollutants (designated pollutants) at existing sources (designated facilities) whenever standards of performance have been established under Section 111(b) for those pollutants at new sources of the same type. Designated pollutants do not include those that are already listed under Section 109(a), 108(a), National Ambient Air Quality Standards, or Section 112(b)(1)(A), Hazardous Air Pollutants.

Subparts B of CFR Part 60 establishes procedures to be followed and requirements to be met in the development and submission of state plans for controlling designated pollutants. Part 62 of the Code of Federal Rgulations provides the procedural framework for the submission of these plans. When designated facilities are located in a state, the state must develop and submit a plan for the control of the designated pollutant. However, 40 CFR 62.06 provides that if there are no existing sources of the designated pollutant located in a state, a letter of certification to that effect (negative declaration) is all that is required from the state. The negative declaration is in lieu of a plan.

To date, EPA has published guideline documents for four designated facilities and pollutants. EPA published standards for control of fluoride emissions from phosphate fertilizer plants on August 6, 1975, at 40 FR 33152; standards for control of fluoride emissions from primary aluminum reduction plants on January 26, 1976, at 41 FR 3826; standards for control of sulfuric acid mist from sulfuric acid plants on October 18, 1977, at 42 FR 55796; and standards for control of total reduced sulfur from kraft pulp mills on February 23, 1978 at 43 FR 7568.

The States of Iowa, Kansas and Nebraska have submitted negative declarations for various designated pollutants. On February 7, 1983, the State of Iowa submitted a negative declaration for kraft pulp mills. The State of Kansas submitted a negative declaration for primary aluminum reduction plants on May 23, 1984. The State of Nebraska submitted a negative declaration for kraft pulp mills and primary aluminum reduction plants on March 16, 1984.

#### Action

EPA approves the negative declarations discussed in this rulemaking in lieu of Section 111(d) plans.

EPA believes these submissions are noncontroversial and is taking final action to approve them without prior proposal. The public should be advised that this action will be effective December 26, 1984. However, if notice is received within 30 days that someone wishes to submit adverse or critical comments, this action will be withdrawn and two subsequent notices will be published before the effective date. One notice will withdraw the final action and another will begin a new rulemaking by announcing a proposal of the action and establishing a comment period.

The Office of Management and Budget has exempted this rule from the requirements of Section 3 of Executive Order 12291.

Under 5 U.S.C. 605(b), I certify that these negative declarations do not have a significant economic impact on a substantial number of small entities.

Under Section 307(b)(1) of the Act, petitions for judicial review of this action must be filed in the United States Court of Appeals by (60 days from today). This action may not be challenged later in proceedings to enforce its requirements [See 307(b)(2)].

This notice is issued under the authority of Section 111(d) of the Clean Air Act, as amended.

#### List of Subjects in 40 CFR Part 62

Air pollution control, fluoride, sulfur, administrative practice and procedure, intergovernmental relations, and reporting and recordkeeping requirements.

Dated: October 22, 1984.

**William D. Ruckelshaus,**

*Administrator.*

### PART 62—[AMENDED]

Part 62 of Chapter 1, Subchapter C, Title 40 of the Code of Federal Regulations is amended by adding a new Subpart Q and amending Subparts R and CC as follows:

1. Subpart Q is added to read as follows:

### Subpart Q—Iowa

**Total Reduced Sulfur Emissions From Existing Kraft Pulp Mills**

**§ 62.3910 Identification of Plan—Negative Declaration.**

Letter from Executive Director of Iowa Department of Environmental Quality submitted on February 7, 1983, certifying that there are no Kraft pulp mills in the State of Iowa, subject ot Part 60, Subpart B of this chapter.

2. Subpart R is amended by adding an undesignated center heading and § 62.4150 to read as follows:

### Subpart R—Kansas

* * * * *

**Fluoride Emissions From Existing Primary Aluminum Reduction Plants**

**§ 62.4150 Identification of Plan—Negative Declaration.**

Letter from the Director, Division of Environment, Kansas Department of Health and Environments submitted on May 23, 1984, certifying that there are no primary aluminum reduction plants on the State of Kansas, subject to Part 60, Subpart B of this chapter.

3. Subpart CC is amended by adding undesignated center headings and §§ 62.6880 and 62.6910 to read as follows:

### Subpart CC—Nebraska

* * * * *

**Total Reduced Sulfur Emissions From Existing Kraft Pulp Mills**

**§ 62.6880 Identification of Plan—Negative Declaration.**

Letter from the Chief of the Air Pollution Control Division of the Department of Environmental Control submitted on March 16, 1984, certifying that there are no existing Kraft pulp mills in the State of Nebraska, subject to Part 60, Subpart B of this chapter.

**Fluoride Emissions From Existing Primary Aluminum Reduction Plants**

**§ 62.6910 Identification of Plan—Negative Declaration.**

Letter from the Chief of the Air Pollution Control Division of the Department of Environmental Control submitted on March 16, 1984, certifying that there are no existing primary aluminum reduction plants in the State of Nebraska, subject ot Part 60, Subpart B of this chapter.

[FR Doc. 84-28300 Filed 10-25-84; 8:45 am]
BILLING CODE 6560-50-M

---

## 40 CFR Part 721

**[OPTS-50504A; FRL-2560-8]**

## Significant New Uses of Chemical Substances 1,2-Benzenediamine, 4-Ethoxy, Sulfate

**AGENCY:** Environmental Protection Agency (EPA).

**ACTION:** Final rule.

**SUMMARY:** EPA is issuing a significant new use rule (SNUR) under section 5(a)(2) of the Toxic Substances Control Act (TSCA) for a chemical substance which was the subject of premanufacture notice (PMN) P-83-105.

**DATES:** This rule shall be promulgated for purposes of judicial review at 1:00 p.m. eastern time on November 9, 1984. This rule shall become effective January 18, 1985.

**FOR FURTHER INFORMATION CONTACT:** Edward A. Klein, Director, TSCA Assistance Office (TS-799), Office of Toxic Substances, Environmental Protection Agency, 401 M St., SW., Washington, D.C. 20460, Toll free: (800-424-9065), In Washington, D.C.: (554-1404), Qutside the USA: (Operator-202-554-1404).

**SUPPLEMENTARY INFORMATION:** OMB Control Number 2070-0012.

### I. Authority

Section 5(a)(2) of TSCA authorizes EPA to determine that a use of a chemical substance is a "significant new use." EPA must make this determination by rule, after considering all relevant factors, including those listed in section 5(a)(2). Once a use is determined to be a significant new use, persons must, under section 5(a)(1)(B), submit a notice to EPA at least 90 days before they manufacture, import, or process the substance for that use. Such a notice is subject to the same requirements and procedures as a PMN submitted under section 5(a)(1)(A) of TSCA which are interpreted at 40 CFR Part 720 published in the **Federal Register** of May 13, 1983 (48 FR 21722). In particular, these include the information submission requirements of section 5(b) and (d)(1), certain exemptions authorized by section 5(h), and the regulatory authorities of section 5 (e) and (f). If EPA does not take regulatory action under section 5, 6, or 7 to control activities on which it has received a SNUR notice, section 5(g) requires the Agency to explain in the **Federal Register** its reasons for not taking action.

Substances covered by proposed or final SNURs are subject to the export reporting requirements of TSCA section 12(b). EPA regulations interpreting section 12(b) requirements appear at 40 CFR Part 707. Substances subject to final SNURs are subject to TSCA section 13 import certification requirements at 19 CFR 12.118 through 12.127 and 127.28 published in the **Federal Register** of August 1, 1983 (48 FR 34734). The EPA policy in support of these requirements appears at 40 CFR Part 707 published in the **Federal Register** of December 13, 1983 (48 FR 55462).

### II. Applicability of General Provisions

EPA has promulgated general provisions under 40 CFR Part 721, Subpart A which are applicable to SNURs and were published in the **Federal Register** of September 5, 1984 (49 FR 35011). These general provisions will apply to this SNUR without change except as discussed in this preamble. Interested persons should refer to that document for a detailed discussion of the general provisions.

The general provisions governing SNUR reporting were promulgated subsequent to the proposal of this rule in the **Federal Register**.

Therefore, this final rule is structurally different from its proposal format because the non-substantive and procedural matters are now contained in Subpart A to Part 721.

### III. Summary of This Rule

The chemical substance subject to this rule is identified as 1,2-benzenediamine, 4-ethoxy, sulfate. It was the subject of PMN P-83-105. EPA is designating the following as a significant new use of the substance: manufacture, import, or processing in powder or dry solid form.

### IV. Background

The chemical substance subject to this rule was the subject of a PMN designated P-83-105. The notice submitter claimed the proposed import volume as confidential business information (CBI).

For purposes of clarity, the substance is referred to in this preamble by its PMN number.

The Agency is concerned that P-83-105 may possess carcinogenic potential following inhalation or ingestion, including swallowing of inhaled particles. The basis for this concern was presented in the preamble to the proposal for this rule which was published in the **Federal Register** of

August 30, 1983 (48 FR 39245). EPA believes that the carcinogenic potential of P-83-105 merits Agency review if a use of the substance may result in inhalation or ingestion of the substance. In EPA's review of the PMN exposures, the Agency determined that human exposure, including inhalational exposure, would be negligible because of the substance's physical state.

## V. Designation of Significant New Use

To determine what would constitute a significant new use of this chemical substance, EPA considered relevant information about the toxicity of the substance and likely exposures associated with possible uses, including the four factors listed in section 5(a)(2) of TSCA. In particular, EPA considered the extent to which potential uses may change or increase the exposure to humans. Based on these considerations, EPA is defining the significant new use of P-83-105 as it appears in Unit III of this preamble and in § 721.195(a)(2).

A comment on the proposed rule stated that the Agency's definition of powder or dry solid form does not include any consideration of the potential of the material to form a respirable dust. The Agency believes that by defining powder or dry solid form as having "the potential to become fine, loose, solid particles," it has addressed the potential of the material to form a respirable dust. The Agency does not believe that imposing a more stringent definition such as might be defined by percent of particles falling within a particular mass median diameter or particular micron range would improve the rule. Particle size and weight, and hence respirability, may vary as a function of not only process, but also environmental factors. As a consequence, the respirability of P-83-105, engineering controls, and protective equipment must be addressed on a case-by-case basis by SNUR notice submitters.

Given EPA's concerns about P-83-105, EPA believes the potential levels of exposure could result in significant risks to workers. Based on analogue data, any significant exposure would present a carcinogenic risk to workers.

The Agency believes that the data described in this preamble and in the preamble to the proposed rule are sufficient to substantiate that manufacture, import, or processing for use in powder or dry solid form of P-83-105 presents a potentially significant increase in the type and magnitude of human exposure. Section 5(a)(2) of TSCA does not require the Agency to make either a "may present" or a "will present" risk finding with regard to satisfying the requirements for a significant new use. The statute imposes the requirement that the Agency provide for a "consideration of all relevant factors." The Agency believes that a reasonable qualitative assessment of these factors was incorporated in the preamble of the proposed rule published in the **Federal Register** of August 30, 1983 (48 FR 39245).

A comment on the proposed rule indicated a belief that EPA's exposure estimates were exaggerated and that monitoring efforts had shown dust exposure below the 5 mg/m³ nuisance dust time weighted average (TWA) limit. The Agency has reevaluated the available information and has concluded that its original assessment was reasonable. While controls at some facilities may keep airborne concentrations below the 5 mg/m³ limit, the available Occupational Safety and Health Administration (OSHA) data suggest that similar conditions are not uniformly maintained throughout the industry. In addition, while utilizing the 5 mg/m³ limit as a benchmark, the Agency does not believe that airborne concentrations in this range would necessarily prove acceptable depending upon duration of exposure, effect of concern, and potency.

The comment also indicated that during drumming or reactor charging operations in one plant, workers will be exposed for only a few hours per day. The comment also pointed out that intermediates are manufactured in batches in that plant that would expose workers for periods significantly less than half the year. The Agency, as it did in the proposed rule, is aware that these reduced exposure situations exist. The Agency, however, believes that even at the levels of exposure described in the comment, there remains the potential for significant exposure and EPA believes review of those exposures, is necessary before they occur.

## VI. Alternatives

In the proposed SNUR, EPA considered other possible approaches. These alternatives included the promulgation of a section 8(a) reporting rule, and/or regulation under section 6. For the reasons discussed in the proposed rule, the Agency has elected to proceed with the promulgation of a SNUR covering a significant new use of P-83-105.

## VII. Exemptions to Reporting Requirements

The Agency has promulgated exemptions to SNUR reporting requirements under § 721.19 of the general SNUR provisions. In the case of P-83-105, the terms of § 721.19 apply without change.

EPA issued it final premanufacture notification rules under 40 CFR Part 720 which were published in the **Federal Register** of May 13, 1983 (48 FR 21722) including § 720.36 which contained detailed rules for the section 5(h)(3) exemption for chemical substances manufactured or imported in small quantities solely for research and development. On September 13, 1983 (48 FR 41132), EPA stayed the effectiveness of § 720.36, among other provisions of the PMN rule, pending further rulemaking to revise the provisions. Because § 720.36 was not in effect when EPA codified § 721.19, the Agency relied on the general definition of "small quantities solely for research and development" in § 720.3(cc) and section 5(h)(3) of TSCA to determine whether activities qualify under this exemption. Upon promulgation of a revised § 720.36, EPA intends to amend § 721.19 to adopt the provisions of the revised § 720.36.

Section 721.19(g) of the general SNUR provisions exempts persons from SNUR reporting when they manufacture or process the substance solely for export and label the substance in accordance with section 12(a)(1)(B) of TSCA. While EPA is concerned about worker exposure during such manufacture and processing of the substance, EPA lacks the authority under section 12(a) of TSCA to require reporting of such manufacture or processing for a significant new use. EPA does not yet have sufficient information to make the "will present an unreasonable risk" finding necessary to regulate a substance manufactured or processed solely for export. However, persons must notify EPA of such export under section 12(b) of TSCA (see § 721.7 of the general SNUR provisions). Such notification will allow EPA to monitor manufacture and processing activities which are not subject to significant new use reporting. The term "manufacture solely for export" is defined in the PMN rule (40 CFR 720.3(s)). The term "process solely for export" is defined in § 721.3 of the general SNUR provisions in a similar fashion. Thus the persons would be exempt from reporting under this SNUR if a person manufactures (the term manufacture includes import) or processes the substance solely for export from the U.S. under the following restrictions: (1) There is no use of the substance in the U.S.; (2) processing is restricted to sites under the control of the manufacturer or processor, respectively; and (3) distribution in commerce is limited to purposes of export. If a person manufactured or

processed the substance both for export and for use in the U.S., such manufacture and processing would not be "solely for export" because manufacture or processing would be for use in the U.S.

A commentor expressed concern that the inclusion of impurities in the definition of "process for commercial purposes" might prove burdensome due to the perception that finished dye products might be liable to the SNUR due to the presence of residual traces of the intermediate. The presence of P–83–105 as a minor impurity in dye products does not fall under the definition. Section 721.19 of Subpart A specifically excludes such impurities and the Agency has determined that that section will apply to this rule. This section provides that P–83–105 is not subject to the notification requirements if the substance is manufactured, imported or processed only as an impurity or byproduct.

### VIII. Applicability to Uses Which May Have Occurred Before Promulgation of Final Rule

To establish a significant new use rule, the Agency must, among other things, determine that the use is not ongoing. In this case, the chemical substance in question has already been added to the Inventory and, thus, could have been manufactured, imported, or processed for use in powder or dry solid form prior to promulgation of this final rule. However, no person has indicated to EPA that such activities have occurred subsequent to Inventory listing.

As indicated in the proposal, EPA has found that the intent of section 5(a)(1)(B) is best served by determining whether a use is a significant new use as of the proposal date of the SNUR. If a use begun during the proposal period were not considered to be a significant new use, it would be almost impossible for the Agency to establish SNUR notice requirements, since any person could defeat the SNUR by initiating the proposed significant new use before the rule becomes final. This is contrary to the general intent of section 5(a)(1)(B).

Thus, even if the substance was imported, manufactured, or processed for a significant new use between proposal and promulgation of this rule, such activities may not continue after the effective date of this rule. Any such person must cease such activities until it has complied with all SNUR notice requirements.

### IX. Test Data and Other Information

EPA recognizes that under TSCA section 5, a person is not required to develop any particular test data before submitting a notice. Rather, a person is required only to submit test data in that person's possession or control and to describe any other data known to or reasonably ascertainable by that person. However, in view of the potential health risk that may be posed by a significant new use of P–83–105, EPA encourages possible SNUR notice submitters to test the substance's potential for carcinogenicity. The Agency believes that the results of a two-year rodent bioassay would adequately characterize possible carcinogenic effects of the substance. If a SNUR notice is submitted for activities involving significant exposure without adequate test data, EPA is likely to take action under section 5(e). As an alternative to testing the substance, potential notice submitters may want to consider the use of engineering controls and/or personal protective equipment to reduce exposure to the substance.

EPA encourages persons to consult with the Agency before selecting a protocol for testing the substance. As part of this prenotice consultation, EPA will discuss the test data it believes necessary to evaluate the significant new use of the substance. Data should be developed and submitted in accordance with the TSCA good laboratory practices regulations at 40 CFR Part 792 published in the **Federal Register** of November 29, 1983 (48 FR 53922).

EPA urges SNUR notice submitters to provide detailed information on human exposure that will result from the significant new use. In addition, EPA urges persons to submit information on potential benefits of the substance and information on risks posed by the substance compared to risks posed by potential substitutes.

### X. Economic Analysis

The Agency has evaluated the potential costs of establishing significant new use reporting requirements for P–83–105. This evaluation is summarized below.

Persons who intend to manufacture, import, or process the substance for a significant new use, as defined in this rule, would be required to submit a SNUR notice with the information required by the statute. The cost of submitting a SNUR notice can be estimated from the cost of submitting a PMN, which has been estimated to range between $1,400 and $7,900 per substance.

The SNUR may also result in delay costs. The delay caused by the preparation of a SNUR notice and the statutory notice review period could reduce the value of future profits. EPA estimates that these delay costs would be about $700.

The SNUR would not require that persons submitting notices perform additional toxicity testing. However, EPA has insufficient information to determine if P–83–105 would present an unreasonable risk of injury to human health if manufactured, processed, or used in a powder or dry solid form. The Agency has determined that the results of a two-year rodent bioassay would adequately characterize possible carcinogenic effects resulting from exposure to P–83–105. EPA could require the submitter of a SNUR notice either to control exposures or limit production under section 5(e) until sufficient information is provided for EPA to make its determination.

In light of the high cost of performing a two-year bioassay (the direct cost of a two-year bioassay, inhalation route test ranges from $742,000 to $874,500 per chemical substance tested), EPA does not anticipate that a company will perform the testing. However, depending upon the potential market for P–83–105 in a powder or dry solid form, a company may produce and process the substance using certain exposure controls.

While EPA is unable to specify, prior to submission of a SNUR notice, combinations of exposure controls that may be appropriate to reduce risks for the variety of manufacturing and processing methods that may be used, the Agency estimates that a SNUR notice submitter will have to spend a maximum of $12,500 on exposure controls.

The total direct costs, including notification, complying with a section 5(e) order, purchase of exposure controls, and delay, would be between $25,700 and $74,000. These direct costs would add between 11.8 and 34 percent to the estimated price of the substance.

EPA has not estimated any indirect costs that may result from this SNUR. These indirect costs may result from decisions not to manufacture or process the substance because of uncertainty about possible Agency regulatory action. Similarly, a decision not to manufacture might result in response to the magnitude of the direct costs. The cost of this impact would be whatever profits or benefits to users that the substance would have generated. In addition, EPA has not estimated the potential public benefits gained through the avoidance of potential health problems. Such benefits include the avoidance of costs such as the medical costs of treating exposed persons. While the Agency acknowledges that indirect

costs and benefits exist, it is impossible at this time to estimate their extent precisely.

A more complete economic analysis of this SNUR and other regulatory options is included in the rulemaking record and is available for public review.

### XI. Judicial Review

Judicial review of this final rule may be available under section 19 of TSCA in the United States Court of Appeals for the District of Columbia Circuit or for the circuit in which the person seeking review resides or has its principal place of business. To provide all interested persons an equal opportunity to file a timely petition for judicial review and to avoid so called "races to the courthouse," EPA has decided to promulgate this rule for purposes of judicial review two weeks after publication in the **Federal Register**, as reflected in **DATES** in this notice. The effective date has, in turn, been calculated from the promulgation date.

### XII. Rulemaking Record

EPA has established a record for this rulemaking (docket control number OPTS-50504A). A public version of this record from which CBI has been deleted is available to the public from 8:00 a.m. to 4:00 p.m., Monday through Friday, except legal holidays, in the OTS Reading Room, Rm. E-107, 401 M St. SW., Washington, D.C.

The record includes basic information considered by the Agency in developing this rule. The record now includes the following:

1. The PMN for the substance.
2. The **Federal Register** notice of receipt of the PMN.
3. The proposed SNUR.
4. The economic analysis of this SNUR.
5. Public comments.
6. The summary of toxicity concerns for the substance.
7. The analysis of potential new uses of the substance.
8. OMB comments and EPA's response, if any.

### XIII. Regulatory Assessment Requirements

*A. Executive Order 12291*

Under Executive Order 12291, EPA must judge whether a regulation is "Major" and therefore requires a Regulatory Impact Analysis. EPA has determined that this rule is not a "Major Rule" because it does not have an effect on the economy of $100 million or more and it will not have a significant effect on competition, costs, or prices. While there is no precise way to calculate the annual cost of this rule, for the reason explained in Unit X of this preamble and the proposal for this rule, EPA believes that the cost will be low. In addition, because of the nature of the rule and the substance subject to it, EPA believes that there will be few significant new use notices submitted. Further, while the expense of a notice and the suggested testing and the uncertainty of possible EPA regulation may discourage certain innovation, that impact may be limited because such factors are unlikely to discourage an innovation which has high potential value. Finally, this SNUR may encourage innovation in safe chemical substances or highly beneficial uses.

This regulation was submitted to the Office of Management and Budget (OMB) for review as required by Executive Order 12291.

*B. Regulatory Flexibility Act*

Under the Regulatory Flexibility Act, 5 U.S.C. 605(b), EPA certifies that this rule will not have a significant economic impact on a substantial number of small businesses. The Agency cannot determine whether parties affected by this rule are likely to be small businesses. However, EPA believes that the number of small businesses affected by this rule would not be substantial even if all the potential new uses were developed by small companies. EPA expects to receive few SNUR notices for the substance.

*C. Paperwork Reduction Act*

Information collection requirements contained in this rule have been approved by the OMB under the provisions of the Paperwork Reduction Act of 1980, U.S.C. 3501 *et seq.* and have been assigned OMB control number 2070-0012.

### List of Subjects in 40 CFR Part 721

Environmental protection, Chemicals, Hazardous substances, Recordkeeping and reporting requirements, Significant new uses.

Dated: October 12, 1984.

**William D. Ruckelshaus,**

*Administrator.*

## PART 721—[AMENDED]

Therefore, Part 721 of Chapter I of Title 40, is amended as follows:

1. By adding the following definitions to § 721.3 in alphabetical sequence:

**§ 721.3 Definitions.**

* * * * *

"Powder or dry solid form" means a state where all or part of the substance would have the potential to become fine, loose, solid particles.

* * * * *

(Approved by the Office of Management and Budget under Control Number 2070-0012)

2. By adding a new § 721.195 to read as follows:

**§ 721.195 1,2-Benzenediamine, 4-ethoxy, sulfate.**

(a) *Chemical substance and significant new use subject to reporting.* (1) The following chemical substance referred to by its chemical name is subject to reporting under this section for the significant new use described in paragraph (a)(2) of this section: 1,2-benzenediamine, 4-ethoxy, sulfate, PMN P-83-105.

(2) The significant new use is: Manufacture, import, or processing in powder or dry solid form.

(b) [Reserved]

(Sec. 5, Pub. L. 94-469, 90 Stat. 2012 (15 U.S.C. 2604))

(Approved by the Office of Management and Budget under Control Number 2070-0012)

[FR Doc. 84-28292 Filed 10-25-84; 8:45 am]

BILLING CODE 6560-50-M

---

**40 CFR Part 721**

**[OPTS-50508A; FRL-2620-6]**

## Significant New Uses of Chemical Substances; Dicarboxylic Acid Monoester

**AGENCY:** Environmental Protection Agency (EPA).

**ACTION:** Final rule.

**SUMMARY:** EPA is issuing a significant new use rule (SNUR) under section 5(a)(2) of the Toxic Substances Control Act (TSCA) for a chemical substance which was the subject of premanufacture notice (PMN) P-83-255 and a TSCA section 5(e) consent order issued by EPA.

**DATES:** This rule shall be promulgated for purposes of judicial review at 1:00 p.m. eastern time on November 9, 1984. This rule shall become effective January 18, 1985.

**FOR FURTHER INFORMATION CONTACT:** Edward A. Klein, Director, TSCA Assistance Office (TS-799), Office of Toxic Substances, Environmental Protection Agency, 401 M St., SW., Washington, D.C. 20460, Toll free: (800-424-9065), In Washington, D.C.: (554-1404), Outside the USA: (Operator—202-554-1404).

**SUPPLEMENTARY INFORMATION:** OMB Control Number 2070-0012.

## I. Authority

Section 5(a)(2) of TSCA authorizes EPA to determine that a use of a chemical substance is a "significant new use." EPA must make this determination by rule, after considering all relevant factors, including those listed in section 5(a)(2). Once a use is determined to be a significant new use, persons must, under section 5(a)(1)(B), submit a notice to EPA at least 90 days before they manufacture, import, or process the substance for that use. Such a notice is subject to the same requirements and procedures as a PMN submitted under section 5(a)(1)(A) of TSCA which are interpreted at 40 CFR Part 720 published in the **Federal Register** of May 13, 1983 (48 FR 21722). In particular, these include the information submission requirements of section 5 (b) and (d)(1), certain exemptions authorized by section 5(h), and the regulatory authorities of section 5 (e) and (f). If EPA does not take regulatory action under section 5, 6, or 7 to control activities on which it has received a SNUR notice, section 5(g) requires the Agency to explain in the **Federal Register** its reasons for not taking action.

Substances covered by proposed or final SNURs are subject to the export reporting requirements of TSCA section 12(b). EPA regulations interpreting section 12(b) requirements appear at 40 CFR Part 707. Substances subject to final SNURs are subject to TSCA section 13 import certification requirements at 19 CFR 12.118 through 12.127 and 127.28 published in the **Federal Register** of August 1, 1983 (48 FR 34734). The EPA policy in support of these requirements appears at 40 CFR Part 707 published in the **Federal Register** of December 13, 1983 (48 FR 55462).

## II. Applicability of General Provisions

EPA has promulgated general provisions under 40 CFR Part 721, Subpart A which are applicable to SNURs and were published in the **Federal Register** of September 5, 1984 (49 FR 35011). These general provisions will apply to this SNUR without change except as discussed in this preamble and as provided in the rule. Interested persons should refer to the above-cited document for a detailed discussion of the general provisions.

The general provisions governing SNUR reporting were promulgated subsequent to the proposal of this rule in the **Federal Register**. Therefore, this final rule is structurally different from its proposal format because the non-substantive and procedural matters are now contained in Subpart A to Part 721.

## III. Summary of This Rule

The chemical substance subject to this rule is identified generically as dicarboxylic acid monester. It was the subject of PMN number P–83–255. EPA is designating the following as significant new uses of the substance:

1. Any manufacture in the United States for commercial purposes.

2. Failure to require the use of gloves determined to be impervious to the substance, and/or failure to require the use of clothing to prevent dermal contact for any person involved in any processing or use operation where dermal contact may occur. (Gloves may be determined to be impervious to the substance either by testing the gloves under the conditions of use or by relying on the manufacturer's specifications.)

3. Distribution in commerce by any person, including importers, processors, and distributors, without affixing to each container of any formulation containing the substance, a label as specified in § 721.290.

## IV. Background

The chemical substance subject to this rule was the subject of a PMN designated P–83–255. The notice submitter claimed the following as confidential business information (CBI): chemical identity, proposed import volume, processing methods, and use. For purposes of clarity, the substance is referred to in this preamble and the final rule by its generic chemical name and PMN number.

The Agency proposed a SNUR for this substance which was published in the **Federal Register** of January 3, 1984 (49 FR 82) as § 721.110 (now § 721.290). The background of the PMN and the reasons for proposing the SNUR are set forth in the preamble to the proposed rule.

EPA received no substantive comments during the public comment period for this SNUR. After the close of the comment period, EPA received comments from the Chemical Manufacturers' Association (CMA) which addressed this SNUR and several others recently proposed by EPA. These comments raised a number of issues about these SNURs and made general suggestions for changes. For example, CMA proposed that EPA's SNURs (1) address hazard communication issues by referencing regulations which were recently promulgated by the Occupational Safety and Health Administration, (2) provide an expedited procedure for the review of alternative types of protective measures rather than the submission of a full SNUR notice with a 90-day review period, and (3) simplify SNUR recordkeeping requirements.

EPA is considering these late comments and may propose amendments to this SNUR in the future to implement some or all of these suggestions. However, because the chemical substance which is identified in this SNUR has been on the Inventory for some time, EPA is concerned that the significant new uses of this substance could be commenced withouth EPA review. Accordingly, the Agency has decided to proceed with promulgation of this SNUR now. If EPA determines that changes to this rule are necessitated by the Agency's response to CMA's comments, amendments to this rule will be made.

## V. Designation of Significant New Uses

To determine what would constitute a significant new use of this chemical substance, EPA considered relevant information about the toxicity of the substance and likely exposures associated with possible uses, including the four factors listed in section 5(a)(2) of TSCA. In particular, EPA considered the extent to which potential uses may change or increase the exposure to humans. Based on these considerations, EPA is defining the significant new uses of P–83–255 as they appear in Unit III of this preamble and § 721.290.

Given EPA's concerns about P–83–255, EPA believes the potential levels of exposure could result in significant risks to workers. Based on data on a probable metabolite (identity CBI) of P–83–255, any significant exposure would present teratogenic and reproductive risk to workers.

The Agency believes that the data described in this preamble and in the preamble to the proposed rule are sufficient to substantiate the contention that the defined significant new uses of P–83–255, present a potentially significant increase in the magnitude and type of exposure. Section 5(a)(2) of TSCA does not require the Agency to make either a "may present" or a "will present" risk finding with regard to satisfying the requirements for a significant new use. The statute imposes the requirement that the Agency provide for a "consideration of all relevant factors." The Agency believes that a reasonable qualitative assessment of these factors was incorporated in the preamble of the proposed rule published in the **Federal Register** of January 3, 1984 (49 FR 82).

## VI. Alternatives

In the proposed SNUR, EPA considered other possible approaches.

These alternatives included the promulgation of a section 8(a) reporting rule, and/or regulation under section 6. For the reasons discussed in the proposed rule, the Agency has elected to proceed with the promulgation of a SNUR covering significant new uses of P-83-255.

### VII. Recordkeeping

To ensure compliance with this rule, and to assist enforcement efforts, EPA is requiring under its authority in sections 5 and 8(a) of TSCA, in addition to the requirements in § 21.17, the following records be maintained for five years after the date of their creation, by persons who manufacture, import, or process P-83-255.

1. The names of persons required to wear protective clothing.

2. The name and address of each person to whom the subject substance is sold or transferred and the date of such sale or transfer.

The Agency considered omitting recordkeeping requirements, but believes compliance monitoring for this SNUR would be made more difficult without them.

### VIII. Exemptions to Reporting Requirements

The Agency has promulgated exemptions to SNUR reporting requirements under § 721.19. In the case of P-83-255, the terms of § 721.19 apply without change.

EPA issued its final premanufacture notification rules under 40 CFR Part 720 which were published in the **Federal Register** of May 13, 1983 (48 FR 21722) including § 720.36 which contained detailed rules for the section 5(h)(3) exemption for chemical substances manufactured or imported in small quantities solely for research and development. On September 13, 1983 (48 FR 41132), EPA stayed the effectiveness of § 720.36, among other provisions of the PMN rule, pending further rulemaking to revise the provisions. Because § 720.36 was not in effect when EPA codified § 721.19, the Agency relied on the general definition of "small quantities solely for research and development" in § 720.3(cc) and section 5(h)(3) of TSCA to determine whether activities qualify under this exemption. Upon promulgation of a revised § 720.36, EPA intends to amend § 721.19 to adopt the provisions of the revised § 720.36.

Section 721.19(g) of the general SNUR provisions exempts persons from SNUR reporting when they manufacture or process the substance solely for export and label the substance in accordance with section 12(a)(1)(B) of TSCA. While EPA is concerned about worker exposure during manufacture and processing of the substance, EPA lacks the authority under section 12(a) of TSCA to require reporting of such manufacture or processing for a significant new use. EPA does not yet have sufficient information to make the "will present an unreasonable risk" finding necessary to regulate a substance manufactured or processed solely for export. However, persons must notify EPA of such export under section 12(b) of TSCA (see § 721.7 of the general SNUR provisions). Such notification will allow EPA to monitor manufacture and processing activities which are not subject to significant new use reporting. The term "manufacture solely for export" is defined in the PMN rule (40 CFR 720.3(s)). The term "process solely for export" is defined in § 721.3 of the general SNUR provisions in a similar fashion. Thus, persons would be exempt from reporting under this SNUR if they manufacture (the term manufacture includes import) or process the substance solely for export from the U.S. under the following restrictions: (1) There is no use of the substance in the U.S.; (2) processing is restricted to sites under the control of the manufacturer or processor, respectively; and (3) distribution in commerce is limited to purposes of export. If a person manufactured or processed the substance both for export and for use in the U.S., such manufacture or processing would not be "solely for export" because manufacture or processing would be for use in the U.S.

### IX. Applicability to Uses Which May Have Occurred Before Promulgation of Final Rule

To establish a significant new use rule, the Agency must, among other things, determine that the use is not ongoing. In this case, the chemical substance in question had just undergone premanufacture review. The Agency received no information that the significant new uses are ongoing. Therefore, at this time, the Agency has concluded that these uses are significant new uses.

As indicated in the proposal, EPA has found that the intent of section 5(a)(1)(B) is best served by determining whether a use is a significant new use as of the proposal date of the SNUR. If uses begun during the proposal period were not considered to be significant new uses, it would be almost impossible for the Agency to establish SNUR notice requirements, since any person could defeat the SNUR by initiating the proposed significant new uses before the rule becomes final. This is contrary to the general intent of section 5(a)(1)(B).

Thus, even if the substance was imported, manufactured, or processed for the significant new use between proposal and promulgation of this rule, such activities may not continue after the effective date of this rule. Any such person must cease such activity until the person has complied with all SNUR notice requirements.

### X. Test Data and Other Information

EPA recognizes that under TSCA section 5, a person is not required to develop any particular test data before submitting a notice. Rather, a person is required only to submit test data in that person's possession or control and to describe any other data known to or reasonably ascertainable by that person. However, in view of the potential health risk that may be posed by a significant new use of P-83-255, EPA encourages possible SNUR notice submitters to test the substance's potential for teratogenic and reproductive effects. The Agency believes that the results of a rodent teratology and a 2-generation reproduction study would adequately characterize possible effects of the substance. If a SNUR notice is submitted for a use involving significant exposure without adequate test data, EPA is likely to take action under section 5(e). As an alternative to testing the substance, potential notice submitters may want to consider the use of engineering controls and/or personal protective equipment to reduce exposure to the substance.

EPA encourages persons to consult with the Agency before selecting a protocol for testing the substance. As part of this prenotice consultation, EPA will discuss the test data it believes necessary to evaluate significant new uses of the substance. Data should be developed and submitted in accordance with the TSCA good laboratory practices regulations at 40 CFR Part 792 published in the **Federal Register** of November 29, 1983 (48 FR 53922).

EPA urges SNUR notice submitters to provide detailed information on human exposure that will result from the significant new uses. In addition, EPA urges persons to submit information on potential benefits of the substance and information on risks posed by the substance compared to risks posed by potential substitutes.

### XI. Economic Analysis

The Agency has evaluated the potential costs of establishing significant new use reporting requirements for P-83-255. This evaluation is summarized in the preamble to the proposed rule. The

complete economic analysis of this SNUR and other regulatory options is included in the rulemaking record and is available for public review.

**XII. Judicial Review**

Judicial review of this final rule may be available under section 19 of TSCA in the United States Court of Appeals for the District of Columbia Circuit or for the circuit in which the person seeking review resides or has its principal place of business. To provide all interested persons an equal opportunity to file a timely petition for judicial review and to avoid so called "races to the courthouse," EPA has decided to promulgate this rule for purposes of judicial review two weeks after publication in the **Federal Register**, as reflected in "DATES" in this document. The effective date has, in turn, been calculated from the promulgation date.

**XIII. Rulemaking Record**

EPA has established a record for this rulemaking (docket control number OPTS–50508A). A public version of this record from which CBI has been deleted is available to the public from 8:00 a.m. to 4:00 p.m., Monday through Friday, except legal holidays, in the OTS Reading Room, Rm. E–107, 401 M St. SW., Washington, DC.

The record includes basic information considered by the Agency in developing this rule. The record now includes the following:

1. The PMN for the substance.
2. The **Federal Register** notice of receipt of the PMN.
3. The section 5(e) Consent Order.
4. The toxicity support document for the section 5(e) Order and the SNUR.
5. The proposed SNUR.
6. The economic analysis of this SNUR.
7. Public comments.
8. OMB comments and EPA's response.

**XIV. Regulatory Assessment Requirements**

*A. Executive Order 12291*

Under Executive Order 12291, EPA must judge whether a regulation is "Major" and therefore requires a Regulatory Impact Analysis. EPA has determined that this rule is not a "Major Rule" because it does not have an effect on the economy of $100 million or more and it will not have a significant effect on competition, costs, or prices. While there is no precise way to calculate the annual cost of this rule, for the reason explained in Unit XI of this preamble and the proposal for this rule, EPA believes that the cost will be low. In addition, because of the nature of the rule and the substance subject to it, EPA believes that there will be few significant new use notices submitted. Further, while the expense of a notice and the suggested testing, and the uncertainty of possible EPA regulation may discourage certain innovation, that impact may be limited because such factors are unlikely to discourage an innovation which has high potential value. Finally, this SNUR may encourage innovation in safe chemical substances or highly beneficial uses.

This regulation was submitted to the Office of Management and Budget (OMB) for review as required by Executive Order 12291.

*B. Regulatory Flexibility Act*

Under the Regulatory Flexibility Act, 5 U.S.C. 605(b), EPA certifies that this rule will not have a significant economic impact on a substantial number of small businesses. The Agency cannot determine whether parties affected by this rule are likely to be small businesses. However, EPA believes that the number of small businesses affected by this rule would not be substantial even if all the potential new uses were developed by small companies. EPA expects to receive few SNUR notices for the substance.

*C. Paperwork Reduction Act*

Information collection requirements contained in this rule have been approved by OMB under the provisions of the Paperwork Reduction Act of 1980, U.S.C. 3501 *et seq.* and have been assigned OMB control number 2070–0012.

**List of Subjects in 40 CFR Part 721**

Environmental protection, Chemicals, Hazardous substances, Recordkeeping and reporting requirements, Significant new uses.

Dated: October 12, 1984.

**William D. Ruckelshaus,**

*Administrator.*

**PART 721—[AMENDED]**

Therefore, Chapter I of Title 40, is amended by adding § 721.290 to read as follows:

**§ 721.290 Dicarboxylic acid monoester.**

(a) *Chemical substance and significant new uses subject to reporting.* (1) The following chemical substance referred to by its premanufacture notice number and its generic chemical name is subject to reporting under this section for the significant new uses described in paragraph (a)(2) of this section: dicarboxylic acid monoester, P–83–255.

(2) The significant new uses are:

(i) Any manufacture in the United States for commercial purposes.

(ii) Failure to require the use of gloves determined to be impervious to the substance, and/or failure to require the use of clothing to prevent dermal contact for any person involved in any processing or use operation where dermal contact may occur. (Gloves may be determined to be impervious to the substance either by testing the gloves under the conditions of use or by relying on the manufacturer's specifications.)

(iii) Distribution in commerce by any person, including importers, processors, and distributors, without affixing to each container of any formulation containing the substance a label that includes, in letters no smaller than 10 point type, the following statements:

WARNING! HARMFUL IF INHALED OR ABSORBED THROUGH THE SKIN. MAY CAUSE REPRODUCTIVE EFFECTS.

—Do not get in eye, on skin, or clothing.
—Do not breathe (vapor, mist, spray, dust).
—Use with adequate ventilation.
—Wear impervious gloves and protective equipment to prevent contact or exposure.
—Promptly remove contaminated non-imprevious clothing, wash before reuse.
—Discard contaminated leather shoes.
—Wash thoroughly after handling, and before eating, drinking, or smoking.
—Keep container closed.

FIRST AID: In case of contact.

EYES: Immediately flush with water for at least 15 minutes.

SKIN: Promptly wash thoroughly with mild soap and water.

INHALATION: Remove to fresh air. If breathing is difficult, give oxygen.

INGESTION: If conscious, give water and induce vomiting.

(b) *Specific requirements.* The provisions of Subpart A of this Part apply to this section except as modified by this paragraph.

(1) *Recordkeeping.* In addition to the requirements of § 721.17, importers and processors of the chemical substance identified in paragraph (a)(1) of this section must maintain the following records for five years from their creation:

(i) The names of persons required to wear protective clothing.

(ii) The name and address of each person to whom the substance is sold or transferred and the date of such sale or transfer.

(2) [Reserved]

(Approved by the Office of Management and Budget under control number 2070-0012)

(Sec. 5, 8, Pub. L. 94-469, 90 Stat. 2012 (15 U.S.C. 2604, 2607))

[FR Doc. 84-28291 Filed 10-25-84; 8:45 am]

BILLING CODE 6560-50-M

## COMMITTEE FOR PURCHASE FROM THE BLIND AND OTHER SEVERELY HANDICAPPED

### 41 CFR Parts 51-7 and 51-8

### Public Availability of Agency Materials and Privacy Act Rules; Change of Address

*Correction*

In FR Doc. 84-25780 beginning on page 38266 in the issue of Friday, September 28, 1984, make the following corrections:

**§ 51-7.4 [Corrected]**

1. On page 38267, first column, amendatory language for § 51-7.4, sixth line, "1155" should read "1755".

**§ 51-8.405 [Corrected]**

2. On the same page, same column, amendatory language for § 51-8.405, fourth line, "1155" should read "1755".

BILLING CODE 1505-01-M

## INTERSTATE COMMERCE COMMISSION

### 49 CFR Parts 1002, 1011, 1152, 1177, 1180, and 1182

[Ex Parte No. 246 (Sub-No. 2)]

### Fees for Services Performed in Connection With Licensing and Related Services; Correction

**AGENCY:** Interstate Commerce Commission.

**ACTION:** Final rules: Correction.

**SUMMARY:** At 49 FR 18490, May 1, 1984, the Commission published rules revising its schedule of fees for services and benefits provided by the Commission under its jurisdictional statute. Those rules contained several errors which this notice corrects.

**FOR FURTHER INFORMATION CONTACT:** Kathleen King, (202) 275-7429.

**SUPPLEMENTARY INFORMATION:** At 49 FR 18493, May 1, 1984, the following correction are made to § 1002.2(f):

**§ 1002.2 [Corrected]**

1. In § 1002.2(f), item (1) under Part I is corrected to read as follows:

(1) An application for motor carrier, or water carrier operating authority, or exemption authority, a certificate of registration or broker or freight forwarder authority.....$150.

2. In § 1002.2(f), items (38), (40), and (41) under Part V are corrected by revising the cross references to "§§ 1180.50, 1180.40, and 1180.50" to read "§§ 1152.50, 1152.40, and 1152.50" respectively.

**James H. Bayne,**

*Secretary.*

[FR Doc. 84-28416 Filed 10-25-84; 8:45 am]

BILLING CODE 7035-01-M

## DEPARTMENT OF THE INTERIOR

### Fish and Wildlife Service

### 50 CFR Part 17

### Endangered and Threatened Wildlife and Plants; Determination of Endangered Status and Designation of Critical Habitat for the Smoky Madtom (Noturus baileyi)

**AGENCY:** Fish and Wildlife Service, Interior.

**ACTION:** Final rule.

**SUMMARY:** The Service determines the smoky madtom (*Noturus baileyi*), a small catfish, to be an endangered species and designates its critical habitat. This rule will implement Federal protection for the species and its critical habitat as provided by the Endangered Species Act of 1973, as amended. The smoky madtom was thought to be extinct when extirpated from Abrams Creek, Great Smoky Mountains National Park, Blount County, Tennessee, in 1957. It was rediscovered in Citico Creek in 1980, and the results of an extensive survey indicate that the species is now apparently restricted to approximately 6.5 miles of Citico Creek, primarily within the Cherokee National Forest, Monroe County, Tennessee. With this restricted range, a single catastrophic event could render the species extinct.

**DATES:** The effective date of this rule is November 26, 1984.

**ADDRESSES:** The complete file for this rule is available for inspection, by appointment, during normal business hours at the Asheville Endangered Species Field Station, 100 Otis Street, Room 224, Asheville, North Carolina 28801 (704/259-0321 or FTS 8/672-0321).

**FOR FURTHER INFORMATION CONTACT:** Mr. Richard G. Biggins, Asheville Endangered Species Field Station, U.S. Fish and Wildlife Service, 100 Otis Street, Room 224, Asheville, North Carolina 28801 (704/259-0321 or FTS 8/672-0321) or Mr. John L. Spinks, Jr., Chief, Office of Endangered Species, U.S. Fish and Wildlife Service, Washington, D.C. 20240 (703/235-2771 or FTS 8/235-2771).

**SUPPLEMENTARY INFORMATION:**

#### Background

The smoky madtom (*Noturus baileyi*) was believed extinct until September 1980 when it was discovered by a U.S. Fish and Wildlife Service survey crew sampling in Citico Creek, a tributary of the Little Tennessee River in Monroe County, Tennessee. Presently, this is the only population known to exist. The species was originally discovered in 1957 in Abrams Creek, a Little Tennessee River tributary in the Great Smoky Mountains National Park, Blount County, Tennessee, by a Service crew which was treating the creek with a fish toxicant to remove unwanted fishes from the Chilhowee Reservoir watershed prior to the closure of Chilhowee Dam. This was a routine procedure at the time, designed to enhance the chances of establishing a trout fishery in the new reservoir. The smoky madtom specimens taken from Abrams Creek during this project were used by Taylor (1969) to describe the species.

A study of the smoky madtom, funded by the Service, was completed in November 1982 (Dinkins, 1982). That survey involved extensive sampling at 44 locations in the Little Tennessee River drainage in North Carolina and Tennessee; 2 tributaries in the Hiwassee River, Tennessee; and 1 tributary in the Pigeon River, Tennessee. Although some habitat looked favorable for the species, the smoky madtom was not found outside Citico Creek.

The species is known from a total of 6.5 miles of Citico Creek, primarily within the Cherokee National Forest. One individual was found about 1 mile below the National Forest's boundary, but this area (outside the area designated as critical habitat) contains little of the species' preferred habitat. The species' prime habitat and the rest of the individuals observed during the study (a total of 67 individuals) were located on Forest Service lands above the upper Citico Creek bridge on Mountain Settlement Road.

The biology of this madtom is poorly understood. However, this small (largest known individual was 2.9 inches in total length) member of the catfish family is probably nocturnal and likely feeds on aquatic insects. The fish has been found in various stages of breeding condition during the spring and summer, and nests (containing an average of 35 eggs) have been located under large slab rocks in pool areas during July (Dinkins, 1982). During the period of May to November,

smoky madtoms are generally found associated with palm-sized slab rock at either the crest or base of riffles.

The apparent limited distribution of this species leaves it vulnerable to a single catastrophic event which could completely eliminate it. The fish's habitat could also be degraded by logging activities, road and bridge construction and maintenance, mineral exploration, and other disturbances within the Citico Creek watershed if these activities are not carefully designed and carried out with the survival of the species in mind.

On June 22, 1982, the Service published, in the **Federal Register** (47 FR 26878), a notice that a review of this species' status was being conducted. That notice requested data on the species' status and solicited information on environmental and economic impacts and the effects on small businesses that could result if the species and its critical habitat were listed. The following is a summary of each of the responses received.

*Tennessee Wildlife Resources Agency* recommended listing the species as an endangered species and designating critical habitat. They also noted that extreme care was needed to ensure that no habitat deterioration took place in the creek or its watershed.

*Tennessee Department of Public Health* recommended the species and its critical habitat in Citico Creek watershed be listed under the Endangered Species Act. They expressed concern for the species if mineral exploration occurred in the watershed. They stated that the watershed contains geologic formations of anakeesta shale. Anakeesta has a 10 percent sulfide content and forms sulfuric acid upon contact with water. They cautioned that mineral explorations could expose anakeesta and result in acid contamination of Citico Creek. They further explained that acid which enters the watershed can oxidize or bring into solution aluminum and other metals that are naturally found in the soils. These metals, especially aluminum, are extremely toxic to the aquatic ecosystem.

*U.S. Department of Agriculture, Forest Service,* stated they had no proposed activities directly involving Citico Creek. They expressed concern that designating critical habitat could have the most significant effect on future timber sales, accompanying road construction, and on possible mineral exploration in the watershed. However, they said no road crossings of Citico Creek were being planned and significant exploration for oil and gas was unlikely. In summary, they stated "* * * we know of no existing or proposed activity that would affect the quality of Citico Creek, nor do we know of significant impacts to small businesses or organizations."

*U.S. Department of the Interior, National Park Service,* responded that their agency did not have any jurisdiction over the area where the species is presently found. However, they did urge protection for the species and its habitat. The species was first discovered in Abrams Creek in Blount County, Tennessee, which is within the Great Smoky Mountains National Park. The Park Service has shown considerable interest in reestablishing the species in Abrams Creek.

*U.S. Department of Transportation, Federal Highway Administration,* informed the Service that a Federal aid secondary road system parallels Citico Creek at the lower end of the creek section where one smoky madtom was found. This creek section is below the designated critical habitat. Although no State requests are pending, Federal aid funds for this short road section may be requested in the future. In spite of potential projects which may impact the species, they state: "We see no reason why these projects could not be implemented with proper measures to prevent significant impacts on the quality of Citico Creek. Listing of the species and designating critical habitat may result in additional coordination/consultation efforts but should not have any significant effect on the Federal aid highway program."

*Department of the Army, Corps of Engineers, Office of Chief Engineer,* responded that the designation of Citico Creek as critical habitat for the smoky madtom would not have a significant effect on any Corps of Engineers program. They further stated: "The Corps of Engineers concurs with the preservation of the species through listing and the designation of its critical habitat."

*U.S. Soil Conservation Service* had no proposed or planned projects in the Citico Creek watershed.

*U.S. Nuclear Regulatory Commission* reported they had no existing or proposed activities which might affect the species or its habitat.

*Federal Energy Regulatory Commission* reported that at this time they had no licensed project or preliminary permits issued in the area inhabited by the smoky madtom.

*Tennessee Valley Authority* stated the area of Citico Creek where the madtom existed was not owned or controlled by TVA. However, the agency had been involved in planning, reviewing, and implementing proposals in this Creek's watershed. They did not report on any presently ongoing projects that would impact the species.

We received one comment on the biology and status of the species from the private individual conducting the smoky madtom status survey for the Service. He recommended the species be listed as endangered and a portion of Citico Creek be designated as critical habitat. This information was utilized in the preparation of the proposal.

On November 21, 1983, the Service published, in the **Federal Register** (48 FR 52612), a proposal to list the smoky madtom as an endangered species and to designate its critical habitat. That proposal provided information on the species' biology, status, threats, and the potential implications of listing. The proposal also solicited comments on the species and potential impacts of the proposed critical habitat determination.

**Summary of Comments and Recommendations**

In the November 21, 1983, proposed rule (48 FR 52612) and associated notifications, all interested parties were requested to submit factual reports or information which might contribute to the development of a final rule. Appropriate State agencies, county governments, Federal agencies, scientific organizations, and other interested parties were contacted and requested to comment. A newspaper notice was published in the Maryville Daily Times on December 21, 1983, which invited general public comment. Five comments were received and are discussed below.

*Tennessee Wildlife Resources Agency* concurred with the proposal to list the species and its critical habitat and stated the Service had adequately described the types of activities subject to impact by the listing action. They concluded the proposed action would be beneficial to the smoky madtom and the existing trout fishery of Citico Creek.

*U.S. Department of Agriculture, Forest Service,* responded that they envisioned no significant change in their management and protection of the Citico Creek habitat but were prepared, if necessary, to modify their management of the Creek to maintain and enhance the smoky madtom. They further stated that listing would likely benefit the species' enhancement opportunities through national recognition, increased interagency cooperation, and definitive multi-agency objectives.

*Department of the Army, Corps of Engineers, Office of Chief Engineer,* stated, "The listing of the smoky

madtom and designation of its critical habitat would not have a significant effect on U.S. Army Corps of Engineers programs. Further, the Corps of Engineers concurs with the preservation of the species through listing and the designation of its critical habitat."

*Tennessee Valley Authority* felt the listing action would not impact any of their programs or environmental considerations in the area. They also stated that, based on the apparent limited range of the species, it should receive protection under the Endangered Species Act.

*U.S. Federal Energy Regulatory Commission* stated they were forwarding our letter to their Washington, D.C., office for comments. We have received no further comments from the Commission.

The Service agrees with the comments that the smoky madtom qualifies for protection under the Endangered Species Act, and concurs that the recognition provided by listing will likely benefit the species and its habitat.

**Summary of Factors Affecting the Species**

After a thorough review and consideration of all information available, the Service has determined that the smoky madtom should be classified as an endangered species. Procedures found at Section 4(a)(1) of the Endangered Species Act (16 U.S.C. 1531 *et seq.*) and regulations promulgated to implement the listing provisions of the Act (codified at 50 CFR Part 424; under revision to accommodate 1982 Amendments—see proposal at 48 FR 36062, August 8, 1983) were followed. A species may be determined to be an endangered or threatened species due to one or more of the five factors described in Section 4(a)(1). These factors and their application to the smoky madtom (*Noturus baileyi*) are as follows:

A. *The present or threatened destruction, modification, or curtailment of its habitat or range.* The smoky madtom is presently known from only 6.5 miles of Citico Creek. With such a limited distribution, the species could be rendered extinct by a single catastrophic event, either natural or human-related. Potential threats to the species and its critical habitat could also come from logging activities, road and bridge construction and maintenance, mineral exploration and mining, and other projects in the watershed if these activities are not planned and implemented with the survival of the species in mind.

Other than the potential soil erosion and siltation problems associated with any land disturbance, a more serious problem could arise in this watershed. The Citico Creek watershed contains geologic formations of anakeesta shale, an acid-bearing rock which has caused problems in the past. Bergendahl *et al.* (1977) reported that in the 1970s a formation of anakeesta was exposed during construction of the Tellico-Robbinsville highway. Acid leaching from a road cut increased the concentration of sulfates, heavy metals, and acidity in Grassy Branch, a tributary of the South Fork Citico Creek. Surveys of Grassy Branch in 1978 revealed no fish life. Attempts have been made to mitigate this problem, but they have not been entirely successful. Other formations of anakeesta do exist in the watershed, and there is a danger that they too could be exposed during construction and mining activities.

B. *Overutilization for commercial, recreational, scientific, or educational purposes.* There is no evidence of overutilization for this species.

C. *Disease or predation.* There is no evidence of threats from disease or predation.

D. *The inadequacy of existing regulatory mechanisms.* Tennessee State law prohibits the taking of the smoky madtom without a permit. See Tenn. Code Ann. section 70–8–104. This law also provides a mechanism which encourages the protection of the fish's habitat. Federal listing would provide necessary additional protection for the species by requiring Federal agencies to consult with the Service when projects they fund, authorize, or carry out may affect the species.

E. *Other natural or manmade factors affecting its continued existence.* Several species of madtoms, for still unexplained reasons, have been extirpated from portions of their range. Etnier and Jenkins (1981) speculated that this may "* * * in addition to visible habitat degradation, be related to their being unable to cope with olfactory 'noise' being added to riverine ecosystems in the form of a wide variety of complex organic chemicals that may occur only in trace amounts." Organic pollution is minimal in the Citico Creek system. However, if madtoms are adversely impacted by increased concentrations of complex organic chemicals, any increase in these materials could cause a problem for this isolated population.

The Service has carefully assessed the best scientific and commercial information available regarding the past, present, and future threats faced by this species in determining to make this rule final. Based on this evaluation, the preferred action is to list the smoky madtom as an endangered species with critical habitat. The fish's present restricted range (6.5 river miles) makes it vulnerable to a single catastrophic event. Therefore, threatened status would not be appropriate. Critical habitat is being designated concurrently as its delineation will aid in the protection and recovery of the species (see Critical Habitat section of this rule). A decision to take no action would exclude the smoky madtom from needed protection available under the Endangered Species Act.

**Critical Habitat**

Critical habitat, as defined in Section 3(5) of the Act, means: (i) the specific areas within the geographical area occupied by a species, at the time it is listed in accordance with the Act, on which are found those physical or biological features (I) essential to the conservation of the species and (II) that may require special management considerations or protection, and (ii) specific areas outside the geographic area occupied by a species at the time it is listed, upon a determination that such areas are essential for the conservation of the species.

Section 4(a)(3) of the Act requires that critical habitat be designated to the maximum extent prudent and determinable concurrently with the determination that a species is endangered or threatened. Critical habitat is being designated for the smoky madtom to include Citico Creek, Cherokee National Forest, Monroe County, Tennesse, from the Cherokee National Forest boundary at upper Citico Bridge on Mountain Settlement Road (approximately creek mile 4.3) upstream to the confluence of Citico Creek with Barkcamp Branch (approximately creek mile 10.8).

As specified in the listing regulations (50 CFR 424.12(b)), the Service shall consider, in determining what areas are critical habitat, those physiological, behavioral, ecological, and evolutionary requirements essential to the conservation of the species and that may require special management consideration or protection. These requirements include, but are not limited to: (1) Space for individual and population growth and normal behavior; (2) food, water, air, light, minerals, or other natural or physiological requirements; (3) cover or shelter; (4) sites for breeding, reproduction, rearing of offspring, * * * and, generally (5) habitats that are protected from disturbance or are representative of the historic geographical and ecological distribution of a species.

In addition to the present high water quality in Citico Creek, the smoky madtom requires run/pool areas with pea-size gravel substrate containing scattered large flat rocks for nesting cover. The species utilizes palm-sized slab rocks for cover and relatively silt-free riffle areas during other times of the year. The area designated as critical habitat provides the smoky madtom with all of the necessary constituent elements for completion of its life cycle. If the quality of this creek section can be maintained near its present level and no catastrophic event occurs, the species will likely continue to survive in Citico Creek.

Section 4(b)(8) of the Act requires, for any proposed or final regulation that designates critical habitat, a brief description and evaluation of those activities (public or private) which may adversely modify such habitat or may be affected by such designation. Activities which presently occur within the critical habitat include fishing, swimming, camping, nature study, and scientific research. These activities, at their present use level, do not appear to be adversely impacting critical habitat.

There are activities which do or could occur within the Citico Creek watershed and which may be affected by designating critical habitat. They include, in part, mineral exploration and mining, bridge and road construction and maintenance, logging, off-road vehicle use, and stream alterations. If not controlled, these activities, along with others that alter the watershed, could degrade the water and substrate quality of Citico Creek by increasing siltation, water temperatures, organic pollutants, acidity, heavy metal concentrations, and extremes in water flow. If any of these activities may affect the critical habitat and are authorized, funded, or carried out by a Federal agency, Section 7(a)(2) of the Act, as amended, requires the agency to consult with the Service to ensure that its action(s) is (are) not likely to destroy or adversely modify critical habitat.

As required by Section 4(b)(2) of the Act, the Service has considered economic and other impacts of designating critical habitat for the smoky madtom. The information for this analysis was supplied to the Service by Federal, State, and local agencies, governmental entities, and interested parties in their responses to the Service's request for this information as part of a June 22, 1982, notice of review published in the **Federal Register** (47 FR 26878) and a November 21, 1983, proposed rule published in the **Federal Register** (48 FR 52612). Neither the notice of review or the proposal to designate critical habitat brought forth economic or other impacts to warrant the adjustment of critical habitat in this final rule.

### Available Conservation Measures

Conservation measures provided to species listed as endangered or threatened under the Endangered Species Act include recognition, recovery actions, requirements for Federal protection, and prohibitions against certain practices. Recognition through listing encourages and results in conservation actions by Federal, State, and private agencies, groups, and individuals. The Endangered Species Act provides for land acquisition and cooperation with the States and requires that recovery actions be carried out for all listed species. Such actions are initiated by the Service following listing. The protection required of Federal agencies and the prohibitions against taking and harm are discussed, in part, below.

Section 7(a) of the Act, as amended, requires Federal agencies to evaluate their actions with respect to any species that is proposed or listed as endangered or threatened. Regulations implementing this interagency cooperation provision of the Act are codified at 50 CFR Part 402 and are now under revision (see proposal at 48 FR 29990; June 29, 1983). Section 7(a)(2) requires Federal agencies to ensure that activities they authorize, fund, or carry out are not likely to jeopardize the continued existence of a listed species or to destroy or adversely modify its critical habitat. If a Federal action may affect a listed species or its critical habitat, the responsible Federal agency must enter into consultation with the Service. As covered earlier in this rule, both the U.S. Forest Service, which has jurisdiction over the Cherokee National Forest, and the Federal Highway Administration, which provides Federal aid funds for upkeep of the road paralleling the lower section of Citico Creek below the critical habitat, have stated that they have no existing or proposed projects that would significantly impact Citico Creek.

Federal activities that could impact the species and its habitat in the future include, but are not limited to, the following: Issuance of permits for mineral exploration, timber sales, recreational development, stream alterations, road and bridge construction and maintenance, and implementation of forest management plans. It has been the experience of the Service that the large majority of Section 7 consultations are resolved so that the species is protected and the project can continue.

The Act and its implementing regulations found at 50 CFR 17.21 set forth a series of general prohibitions and exceptions that apply to all endangered fish and wildlife. These prohibitions, in part, would make it illegal for any person subject to the jurisdiction of the United States to take, import or export, ship in interstate commerce in the course of a commercial activity, or sell or offer for sale in interstate or foreign commerce any listed species. It also would be illegal to possess, sell, deliver, carry, transport, or ship any such wildlife which was illegally taken. Certain exceptions would apply to agents of the Service and State conservation agencies.

Permits may be issued to carry out otherwise prohibited activities involving endangered animal species under certain circumstances. Applicable regulations governing permits are at 50 CFR 17.22. Such permits are available for scientific purposes, to enhance the propagation or survival of the species, and/or for incidental take in connection with otherwise lawful activities.

### National Environmental Policy Act

The Fish and Widlife Service has determined that an Environmental Assessment, as defined under authority of the National Environmental Policy Act of 1969, need not be prepared in connection with regulations adopted pursuant to Section 4(a) of the Endangered Species Act of 1973, as amended. A notice outlining the Service's reasons for this determination was published in the **Federal Register** on October 25, 1983 (48 FR 49244).

### Regulatory Flexibility Act and Executive Order 12291

The Department of the Interior has determined that designation of critical habitat for this species will not constitute a major rule under Executive Order 12291 and certifies that this designation will not have a significant economic impact on a substantial number of small entities under the Regulatory Flexibility Act (5 U.S.C. Section 601 *et seq.*). The entire critical habitat area is owned and administered by the Forest Service. Present and planned uses of the critical habitat area and the watershed above it are compatible with the critical habitat designation. No activities having Federal involvement are presently known that would affect or be affected by the designation. Consequently, no significant economic or other impacts are expected to result from the designation of critical habitat for the smoky madtom. No direct costs,

enforcement costs, or record-keeping requirements are imposed on small entities by the designation. Further, the rule contains no information collection or recordkeeping requirements as defined by the Paperwork Reduction Act of 1980. These determinations are based on a Determination of Effects that is available at the U.S. Fish and Wildlife Service, Office of Endangered Species, 1000 N. Glebe Road, Arlington, Virginia.

### Literature Cited


Bergendahl, B.S., J.L. Blackburn, and G.L. Klinedinst. 1977. Report on the geological and water quality of Tellico-Robbinsville Road, Station 804+85+ to 956+10+. Region 15 Federal Highway Administration.

Dinkins, G.R. 1982. Status survey of the smoky madtom (*Noturus baileyi*): final report under contract [Number 14-16-004-81-060] to U.S. Fish and Wildlife Service, Asheville, North Carolina. 33 pp.

Etnier, D.A., and R.E. Jenkins. 1981. *Noturus stanauli*, a new madtom catfish (Ictaluridae) from the Clinch and Duck Rivers, Tennessee. Bull. Ala. Mus. Nat. Hist. 5:17-22.

Taylor, W.R. 1969. A revision of the genus *Noturus Refinesque* with an analysis of higher groups of Ictaluridae. (*Noturus baileyi* pp. 141-144). Bull. U.S. Nat. Mus., No. 282, p. 315.


### Author

The primary author of this final rule is Richard G. Biggins, Asheville Endangered Species Field Station, U.S. Fish and Wildlife Service, 100 Otis Street, Room 224, Asheville, North Carolina 28801 (704/259-0321 or FTS 8/672-0321).

### List of Subjects in 50 CFR Part 17

Endangered and threatened wildlife, Fish, Marine mammals, Plants (agriculture).

## Proposed Regulations Promulgation

## PART 17—[AMENDED]

Accordingly, Part 17, Subchapter B of Chapter I, Title 50 of the Code of Federal Regulations, is amended as set forth below:

1. The authority citation for Part 17 reads as follows:

**Authority:** Pub. L. 93-205, 87 Stat. 884; Pub. L. 94-359, 90 Stat. 911; Pub. L. 95-632, 92 Stat. 3751; Pub. L. 96-159, 93 Stat. 1225; Pub. L. 97-304, 96 Stat. 1411 (16 U.S.C. 1531 *et seq.*).

2. Amend § 17.11(h) by adding the following, in alphabetical order, under fish to the List of Endangered and Threatened Wildlife:

**§ 17.11 Endangered and threatened wildlife.**

* * * * *

(h) * * *

| Species | | Historic range | Vertebrate population where endangered or threatened | Status | When listed | Critical habitat | Special rules |
|---|---|---|---|---|---|---|---|
| Common name | Scientific name | | | | | | |
| FISHES | | | | | | | |
| Madtom, Smoky | *Noturus baileyi* | U.S.A. (TN) | Entire | E | 163 | 17.95(e) | NA. |

3. Amend § 17.95(e) by adding critical habitat for the smoky madtom as follows:

**§ 17.95 Critical habitat—fish and wildlife.**

(e) *Fishes.*

* * * * *

**Smoky madtom** *(Noturus baileyi)*

Tennessee, Monroe County, Citico Creek, Cherokee National Forest. Citico Creek from the Cherokee National Forest boundary at upper Citico Bridge on Mountain Settlement Road (approximately creek mile 4.3) upstream to the confluence of Citico Creek with Barkcamp Branch (approximately creek mile 10.8).

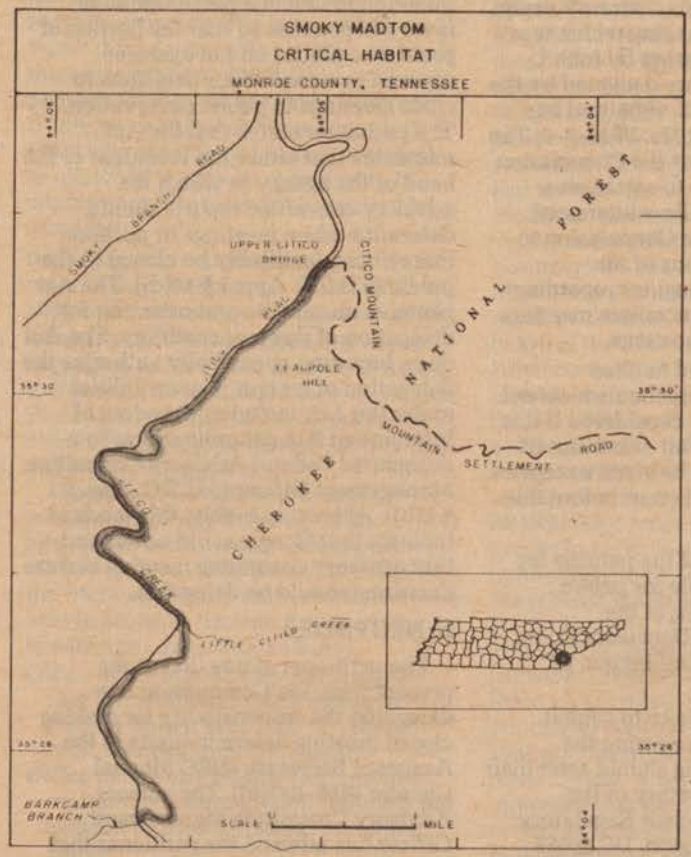
Constituent elements of the critical habitat include the present good water quality in Citico Creek and run/pool areas with relatively silt-free pea-size gravel substrate containing scattered large flat rocks for breeding habitat. The species utilizes palm-size slab rocks for cover and relatively silt-free riffle areas during other times of the year. The area designated as critical habitat provides the smoky madtom with all of the necessary constituent elements for completion of its life cycle.

* * * * *

Dated: October 10, 1984.

G. Ray Arnett,

*Assistant Secretary for Fish and Wildlife and Parks.*




[FR Doc. 84-28264 Filed 10-25-84; 8:45 am]

BILLING CODE 4310-55-M

# Proposed Rules

Federal Register
Vol. 49, No. 209
Friday, October 26, 1984

This section of the FEDERAL REGISTER contains notices to the public of the proposed issuance of rules and regulations. The purpose of these notices is to give interested persons an opportunity to participate in the rule making prior to the adoption of the final rules.

## NUCLEAR REGULATORY COMMISSION

### 10 CFR Part 7

**[Docket No. PRM-7-2]**

### John L. Nantz; Filing of Petition for Rulemaking

**AGENCY:** Nuclear Regulatory Commission.

**ACTION:** Notice of receipt of petition for rulemaking from John L. Nantz.

**SUMMARY:** The Commission is publishing for public comment this notice of receipt of a petition for rulemaking which was filed with the Commission by John L. Nantz. The petition was docketed by the Commission on July 25, 1984, and has been assigned Docket No. PRM-7-2. The petitioner requests that the Commission amend its regulations to establish a formal procedure to allow interested persons to petition the Commission to reconsider the decisions of the Commission or its delegates regarding closure of advisory committee meetings or portions of those meetings.

**DATE:** Comment period expires December 26, 1984. Comments received after this date will be considered if it is practicable to do so, but assurance of consideration cannot be given except as to comments received on or before this date.

**ADDRESSES:** A copy of the petition for rulemaking is available for public inspection and copying at the Commission's Public Document Room, 1717 H Street NW, Washington, DC 20555.

All persons who desire to submit written comments concerning the petition for rulemaking should send their comments to the Secretary of the Commission, U.S. Nuclear Regulatory Commission, Washington, DC 20555, Attention: Docketing and Service Branch.

**FOR FURTHER INFORMATION CONTACT:** John Philips, Chief, Rules and Procedures Branch, Division of Rules and Records, Office of Administration, U.S. Nuclear Regulatory Commission, Washington, DC 20555, Telephone: 301-492-7086.

**SUPPLEMENTARY INFORMATION:**

#### I. Background

The petitioner states that the Federal Advisory Committee Act (the Act) mandates that all advisory committee meetings shall be open to the Public (5 U.S.C. App. I, § 10(a)(1)). This general requirement of open deliberations is subject only to the limited exceptions contained in the Government in the Sunshine Act (5 U.S.C. 552b(c)), which were made applicable to the Act through a 1976 amendment. Further, when discussions fall within the scope of an exemption, closing the meeting to the public is not mandatory but only permissive. This was Congress's intention to create a presumption in favor of openness so that the burden of proof was placed on the agencies covered to sustain their decisions to close meetings to public observation. The petitioner states that the Act mandates that either the President or the head of the agency to which the advisory committee reports should determine when meetings or portions thereof may justifiably be closed to the public (5 U.S.C. App. I § 10(d)). The Act contains no specific authorization for delegation of this responsibility. The Act does, however, specifically authorize the delegation of certain responsibilities under the Act, including Freedom of Information Act determinations, to a designated Federal Advisory Committee Management Officer (5 U.S.C. App. I, § 8(b)). At least arguably, this tends to indicate that Congress did not intend that advisory committee meeting closure decisions should be delegable.

#### II. NRC Practice

The petitioner states that at the present time, the Commission has delegated the responsibility for making closed meeting determinations to the Assistant Secretary (NRC Manual Chapter 0105-033(b)). The current Advisory Committee Management Officer has advised the petitioner that the authority for this delegation derives for 5 U.S.C. App. I, § 8 and 10 CFR 7.5. However, neither of these provisions specifically refers to the authority to make closure determinations and the language of the manual provision in question and 10 CFR 7.9(d) appear to the petitioner to be limited solely to initial closure decisions. Neither makes any mention of authority to rule on petitions for reconsideration.

The present practice regarding advisory committee closure contains none of the safeguards to ensure adequate consideration of the public interest which exist pertaining to the Commission's own meetings. The Commission, according to the petitioner, rejected proposals to adopt a formal two-step meeting closure procedure in reliance on the awareness of the Commissioners and their advisors of countervailing public interest concerns. The petitioner further states that the present lack of involvement on the part of the Commissioners themselves regarding advisory committee closure determinations increases the possibility that the public interest in open deliberations will not receive adequate consideration.

The petitioner asserts that the Commission, as the collegial body heading the NRC, should retain the ultimate responsibility for reviewing the decisions of its delegate. The clear import of the Act, maintains the petitioner, mandates that the Commission should exercise the authority to review the closure decisions of its delegate when they are challenged. The petitioner states that providing for review of the delegate's determinations by the General Counsel is not sufficient to discharge the statutory responsibility imposed on the Commissioners themselves.

#### III. Proposed Amendment of 10 CFR 7.9

In order to establish a procedure for seeking reconsideration of decisions to close advisory committee meetings, in whole or in part, to public observation and participation, the petitioner recommends the following amendments to the regulations.

1. Present paragraph (e) of 10 CFR 7.9 would be redesignated as paragraph (g).

2. New paragraph (e) and (f) would be added to 10 CFR 7.9 to read as follows:

(e) Any person may petition the

Commission to reconsider its action under § 7.9(c) or the action of its delegate under § 7.9(d) by filing a petition for reconsideration with the Commission within seven days after the date of public announcement of such action and before the meeting in question is held.

(f) A petition for reconsideration filed pursuant to paragraph (e) of this section shall state specifically the grounds on which the action of the Commission or its delegate is claimed to be erroneous, and shall set forth, if appropriate, the public interest in the opening of the meeting. The filing of such a petition shall not act to stay the effectiveness of the action of the Commission or its delegate or to postpone or delay the meeting in question unless the Commission orders otherwise.

The petitioner points out that the proposed new paragraphs (e) and (f) establish a clear procedure for reconsideration of advisory committee meeting closure decisions similar to that available for review of decisions to close Commission meetings pursuant to 10 CFR 9.106 (b) and (c). With several exceptions, the language of the proposed amendments is identical to that employed in § 9.106.

In proposed new paragraph (e), the petitioner has also specified that a petition for reconsideration may be filed within seven days of public announcement of the closure decision. The petitioner believes that allowing at least seven days after the public announcement rather than the date of the decision itself, as is the case with 10 CFR 9.106(b), is required because Part 7 contains no provision comparable to § 9.105(b) requiring prompt public notification of the closure decision.

The petitioner believes that the proposed amendments will adequately address the concerns raised in his petition without unduly burdening the Commission. Moreover, the petitioner concludes that providing an established mechanism to invoke Commission review of advisory committee closure determinations should enhance the probability that the Commission's delegate will give adequate attention to the public interest in open deliberations when making his decisions.

Dated at Washington, DC this 23rd day of October 1984.

For the Nuclear Regulatory Commission.

Samuel J. Chilk,

*Secretary of the Commission.*

[FR Doc. 84-28328 Filed 10-25-84; 8:45 am]

BILLING CODE 7590-01-M

## DEPARTMENT OF THE INTERIOR

### Office of Surface Mining Reclamation and Enforcement

### 30 CFR Part 926

### Proposed Modifications to the Montana Permanent Regulatory Program

**AGENCY:** Office of Surface Mining Reclamation and Enforcement (OSM), Interior.

**ACTION:** Reopening of public comment period.

**SUMMARY:** OSM is reopening the period for review and comment on material submitted by the Montana Department of State Lands (DSL), that clarifies modified portions of the Montana permanent regulatory program. In the February 6, 1984 **Federal Register**, OSM announced a public comment period and procedure for requesting a public hearing on the substantive adequacy of proposed amendments to the Montana permanent regulatory program under the Surface Mining Control and Reclamation Act of 1977 (SMCRA) submitted by Montana on January 3, 1984. The clarifying material submitted by Montana consists of a letter and two forms that, among other things, address the assessment and waiver of civil penalties under the Montana Strip and Underground Mine Reclamation Act. OSM is reopening the comment period to allow the public an opportunity to comment on the material submitted by Montana.

**DATE:** Written comments not received on or before 4:00 p.m. on November 12, 1984 will not necessarily be considered.

**ADDRESSES:** Written comments should be mailed or hand delivered to William R. Thomas, Field Office Director, Casper Field Office, Office of Surface Mining, Freden Building, 935 Pendell Boulevard, Mills, Wyoming 82644.

Copies of the revised material submitted by Montana and other relevant documents are available for review at the OSM offices and office of the State regulatory authority listed below, Monday through Friday, 8:00 a.m. to 4:00 p.m., excluding holidays.

Office of Surface Mining Reclamation and Enforcement, Administrative Record, Room 5124 1100 "L" Street, NW., Washington, D.C. 20240

Office of Surface Mining Reclamation and Enforcement, Freden Building, 935 Pendell Boulevard, Mills, Wyoming 82644

Montana Department of State Lands, Reclamation Division, Capitol Station, Helena, Montana 59620.

**FOR FURTHER INFORMATION CONTACT:** William R. Thomas, Field Office Director, Office of Surface Mining, Freden Building, 935 Pendell Boulevard, Mills, Wyoming 82644; Telephone: (307) 328-5830.

**SUPPLEMENTARY INFORMATION:** The general background on the permanent regulatory program, the State program approval process, the Montana program and conditional approval can be found in the Secretary's Findings and conditional approval published in the April 1, 1980 **Federal Register** (45 FR 21560) and the February 11, 1982 **Federal Register** (47 FR 6266).

On January 3, 1984, Montana submitted to OSM a program amendment that, among other things, addressed civil penalties. The proposed amendment consists of proposed regulations governing procedures for the assessment and waiver of civil penalties; implementation of a point system for civil penalties and waivers; issuance of notice of non-compliance and cessation orders; informal hearings; operator's inability to comply with a notice of compliance or cessation order; and continuation of health and safety activities during the period an order is in effect. The February 6, 1984 **Federal Register** announced receipt of the modifications by OSM as well as a public comment period (49 FR 4385). In that same notice, OSM announced that a public hearing would be held only if requested. No requests were received and no hearing was held. The State of Montana, however, on February 2, 1984, conducted a public hearing to receive comments on the adequacy of Montana's proposed regulations. As a result of comments received at the hearing, the State, on March 6, 1984, submitted to OSM revised proposed regulations addressing civil penalties. Copies of the revised material are available in the OSM Administrative Record. OSM announced receipt of the revisions in the March 22, 1984 **Federal Register** and reopened the comment period in order to allow the public an opportunity to review and comment on the revised material submitted to OSM by the State on March 6, 1984 (49 FR 10675). The comment period closed April 23, 1984. The Montana Department of State Lands (DSL) published final rules relating to civil penalties on March 31, 1984 and submitted a copy to OSM on April 2, 1984. A review by OSM indicated that the DSL made minor revisions to the rules prior to publication in the Montana Administrative Record. Therefore, on May 7, 1984, OSM reopened the public comment period to allow the public an opportunity to

review and comment on the revised material submitted by Montana (49 FR 19340). A review by OSM identified minor deficiencies in the Montana submission. The State was notified July 25, 1984, of OSM's concern and subsequently provided clarifying material to OSM on September 20, 1984. OSM is announcing receipt of the material submitted by Montana and reopening the comment period to all the public an opportunity to comment on it.

Specifically, OSM is seeking comment on whether the clarifying material pertaining to civil penalties submitted by Montana on September 20, 1984, satisfies the criteria for approval of State program amendments at 30 CFR 732.15 and 732.17. Upon request to OSM's Field Office Director, each person may receive, free of charge, one single copy of the revised material.

**Authority:** Pub. L. 95–87, Surface Mining Control and Reclamation Act of 1977 (30 U.S.C. 1201 *et seq.*)

Dated: October 18, 1984.

**William B. Schmidt,**

*Assistant Director, Program Operations and Inspection.*

[FR Doc. 84–28344 Filed 10–25–84; 8:45 am]

BILLING CODE 4310–05–M

## ENVIRONMENTAL PROTECTION AGENCY

### 40 CFR Part 52

**[A–5–FRL 2704–3]**

### Approval and Promulgation of Implementation Plans; Illinois

**AGENCY:** Environmental Protection Agency (USEPA).

**ACTION:** Proposed rule; extension of the public comment period.

**SUMMARY:** On August 15, 1984, (49 FR 32601), USEPA proposed action on revisions to the draft Illinois 1982 ozone and carbon monoxide SIP. On October 2, 1982, the public comment period was extended until October 13, 1984. The State of Illinois has requested a further extension of the public comment period to allow a reasonable opportunity to comment on unresolved issues identified in the supplemental proposed rulemaking. Therefore, the public comment period is extended until November 12, 1984.

**DATE:** Comments must be received on or before November 12, 1984.

**ADDRESSES:** Comments should be submitted to: Gary V. Gulezian, Chief, Regulatory Analysis Section, Air and Radiation Branch, Region V, U.S. Environmental Protection Agency (5AR–26), 230 South Dearborn Street, Chicago, Illinois 60604.

**FOR FURTHER INFORMATION CONTACT:** Randolph O. Cano, (312) 886–6035.

Dated: October 18, 1984.

**Valdas V. Adamkus,**

*Regional Administrator.*

[FR Doc. 84–28295 Filed 10–25–84; 8:45 am]

BILLING CODE 6560–50–M

### 40 CFR Part 52

**[A–5–FRL–2704–4]**

### Approval and Promulgation of Implementation Plans; Ohio and Kentucky

**AGENCY:** Environmental Protection Agency.

**ACTION:** Notice announcing extension of public comment period.

**SUMMARY:** On July 25, 1984 (49 FR 29973), EPA proposed to disapprove the Ohio and Kentucky ozone attainment plans for the Cleveland and Cincinnati urban areas. A 30-day comment period was provided. In response to several requests, EPA agreed on August 27, 1984 (49 FR 33902), to extend the comment period by 30 days, to September 24, 1984.

Subsequently, EPA received a request for an additional 30-day extension of the comment period. The reason given for this request was to allow for a survey of the impact of EPA's proposed action on small entities, as described in the Regulatory Flexibility Act. In its July 25, 1984, proposal, EPA stated its belief that such impact would be small, but acknowledged the difficulty of obtaining reliable information of this nature. Because this information would not otherwise be readily available to EPA, the Agency agrees to extend the comment period to October 24, 1984, for the purpose of receiving comments on the impact of EPA's proposed action on small entities. EPA believes the 60-day comment period ending September 24, 1984, provided adequate opportunity for comment on other aspects of the proposed rule, and is therefore not extending the comment period as it relates to other aspects of the rule.

**DATE:** Comments on the impact of the proposed rule on small entities must be received by October 24, 1984. Comments on other aspects of the rule must be received on or before September 24, 1984.

**ADDRESS:** Comments should be sent to: Gary Gulezian, Chief, Regulatory Analysis Section, Air and Radiation Branch, U.S. Environmental Protection Agency, Region V, 230 South Dearborn Street, Chicago, IL 60604.

**FOR FURTHER INFORMATION CONTACT:** Thomas Lyttle, EPA Region IV, 404/881–2864.

Dated: October 15, 1984.

**John A. Little,**

*Acting Regional Administrator.*

[FR Doc. 84–28296 Filed 10–25–84; 8:45 am]

BILLING CODE 6560–50–M

### 40 CFR Part 271

**[WH–3–FRL–2703–8]**

### Maryland; Final Authorization of State Hazardous Waste Management Program

**AGENCY:** Environmental Protection Agency.

**ACTION:** Notice of Tentative Determination on Maryland's Application for Final Authorization, Public Hearing, and Public Comment Period.

**SUMMARY:** The State of Maryland has applied for Final Authorization under the Resource Conservation and Recovery Act (RCRA). EPA has reviewed Maryland's application and found that the State needs to provide additional information and assurances before Final Authorization can be granted. Maryland has agreed to provide both the assurances and the information to EPA's satisfaction prior to the public hearing on the application. Thus, EPA tentatively intends to grant Final Authorization to the State to operate its program in lieu of the Federal program. Maryland's application for Final Authorization is available for public review and comment, and a public hearing will be held to solicit comments on the tentative decision. In making its final decision, EPA will consider all public comments, both written and oral, on the tentative decision and the measures taken by the State to address the EPA concerns.

**DATE:** A public hearing is scheduled for November 28, 1984. Maryland will participate in the public hearing held by EPA on this subject. All written comments on Maryland's Final Authorization application must be received by the close of business on November 28, 1984.

**ADDRESSES:** Copies of Maryland's Final Authorization application are available during normal business hours at the following addresses for inspection and copying:

Maryland Department of Health and Mental Hygiene, Waste Management

Administration, 2nd Floor, O'Connor Building, 201 West Preston Street, Baltimore, Maryland 21201. (Contact: Bernard Bigham, Phone: 301/383–5740)
Office of Environmental Programs, Failinger Complex, Route 8 and Naves Crossroad. Cumberland, Maryland 21502. (Contact: Robert Creter, Phone: 301/777–2370)
Waste Management Administration, Centreville Multi-Service Center, 120 Broadway, Centreville, Maryland 21617. (Contact: John Chlada, Phone: 301/758–2221)
EPA Headquarters Library, 401 M Street, SW., Washington, DC 20460, (202/382–5926)
U.S. EPA, Region III, Library, 2nd Floor, 6th and Walnut Streets, Philadelphia, Pennsylvania 19106. (Contact: Diane McCreary, Phone: 215/597–0580).

Written comments on the Maryland application must be sent to: John J. Humphries, Program Manager, State Programs Section (3HW31), U.S. EPA Region III, 6th and Walnut Streets, Philadelphia, Pennsylvania 19106, (215) 597–2863.

EPA will hold a public hearing on Maryland's application for Final Authorization on Wednesday, November 28, 1984 at 7:30 p.m. in the 1st Floor Auditorium, State Office Complex, 300 West Preston Street, Baltimore, Maryland.

**FOR FURTHER INFORMATION CONTACT:** John J. Humphries, Program Manager, State Programs Section (3HW31), U.S. EPA Region III, 6th and Walnut Streets, Philadelphia, Pennsylvania 19106, (215) 597–2863.

**SUPPLEMENTARY INFORMATION:**

## A. Background

Section 3006 of the Resource Conservation and Recovery Act (RCRA) allows EPA to authorize State hazardous waste programs to operate in lieu of the Federal hazardous waste program. Two types of authorizations may be granted. The first type, known as "Interim Authorization", is a temporary authorization which is granted if EPA determines that the State program is "substantially equivalent" to the Federal program (Section 3006(c), 42 U.S.C. 6226(c)). EPA's implementing regulations at 40 CFR 271.121 through 271.137 established a phased approach to Interim Authorization: Phase I, covering the EPA regulations in 40 CFR Parts 260 through 263, and 265 (universe of hazardous wastes, generator standards, transporter standards, and standards for interim status facilities), and Phase II, covering the EPA regulations in 40 CFR Parts 124, 264, and 270 (procedures and standards for permitting hazardous waste management facilities).

Phase II, in turn, has three components. Phase II A covers general permitting procedures and technical standards for containers and tanks. Phase II B covers permitting of incinerator facilities, and Phase II C addresses permitting of landfills, surface impoundments, waste piles, and land treatment facilities. By statute, all Interim Authorizations expire on January 26, 1985, unless Congress extends the date. Responsibility for the hazardous waste program returns (reverts) to EPA on that date if the State has not received Final Authorization, as described below.

The second type of authorization is "Final Authorization" that is granted by EPA if the Agency finds that the State program is (1) "equivalent" to the Federal program, (2) consistent with the Federal program and other State programs, and (3) provides for adequate enforcement (Section 3006(b), 42 U.S.C. 6226(b)). States need not have obtained Interim Authorization in order to qualify for Final Authorization. EPA regulations for Final Authorization appear at 40 CFR 271.1 through 271.23.

## B. Maryland

The State of Maryland received Interim Authorization for Phase I on July 8, 1981, Interim Authorization for Phase II, Component A, on November 23, 1983 and Interim Authorization for Phase II, Components B and C on July 9, 1984. On January 30, 1984, the State submitted a draft application for Final Authorization to EPA. Prior to submission of the State's official application to EPA on June 29, 1984, Maryland solicited public comments and held a public hearing on April 26, 1984. The State did not receive any written or oral comments. EPA's comments on the official application were forwarded by a letter dated August 17, 1984 from Mr. Anthony J. Donatoni, Chief, State Programs Section to Mr. Ronald Nelson, Director, Maryland Waste Management Administration. That letter is a matter of public record. In brief, EPA is requesting the State to provide the following:

1. A discussion in the Program Description regarding the State's use of a permit application form. Also, a model permit should be substituted for a specific facility permit in the Program Description appendices.

2. A clear statement in the Program Description that the staff positions identified are committed to the RCRA hazardous waste program only and do not perform hazardous waste work in other program areas like Superfund.

3. The State's permit call-in strategy needs further discussion in the Program Description.

4. A discussion in the Program Description to clarify two regulatory issues; (1) what State standards are imposed on "non-regulated units" which must maintain a groundwater monitoring program and (2) that the Department of Health and Mental Hygiene is the sole State agency with authority over the construction and operation of RCRA facilities.

5. A regulatory errata published in the *Maryland Register*, before EPA's public hearing, addressing 15 regulatory discrepancies between the EPA and State program regulations.

The State of Maryland has agreed to satisfactorily address EPA's comments. The application contains amendments to the Program Description and a regulatory errata will be published by the State before EPA's public hearing.

Thus, EPA tentatively intends to grant Final Authorization to Maryland to operate its program in lieu of the Federal Program. Copies of Maryland's application are available for inspection and copying at the locations indicated in the **ADDRESSES** section of this notice.

In making its final decision, EPA will consider all public comments on its tentative determination and the measures taken by the State to address EPA's concerns. Issues raised by those comments may be the basis for a decision to deny Final Authorization to Maryland. EPA's final decision whether to approve the State's program will be based, in part, on Maryland's ability to maintain the current level of performance and fulfilling the commitments included in the Memorandum of Agreement. EPA expects to make a final decision on whether or not to approve Maryland's program by January 11, 1985, and will give notice of it in the **Federal Register.** The notice will include a summary of the reasons for the final determination and a response to all major comments.

However, this schedule will change if amendments made to Maryland's application are substantial or if they differ significantly from the above arrangements. Provisions of 40 CFR 271.20(b) require the State to provide for additional public comment if the proposed State program is substantially modified after the State comment period ends. Additionally, 40 CFR 271.5(c) provides that if the State's application materially changes during EPA's review period, the statutory review period begins again upon receipt of the revised submission. The State and EPA may also extend the review period by

agreement (see 40 CFR 271.5(d)). EPA will give notice of its final decision or of a change in schedule in the Federal Register by January 11, 1985.

**Regulatory Flexibility Act**

Pursuant to the provisions of 5 U.S.C. 605(b), I hereby certify that this authorization will not have significant economic impact on a substantial number of small entities. The authorization suspends the applicability of certain Federal regulations in favor of the State program, thereby eliminating duplicative requirements for handlers of hazardous waste in the State. This rule, therefore, does not require a regulatory flexibility analysis.

**Executive Order 12291**

The Office of Management and Budget (OMB) has exempted this rule from the requirements of Executive Order 12291, Section 3.

**List of Subjects in 40 CFR Part 271**

Hazardous materials, Indian lands, Reporting and record keeping requirements, Waste treatment and disposal, Water pollution control, Water supply, Intergovernmental relations, Penalties, Confidential business information.

(Secs. 2002(a), 3006, and 7004(b), Solid Waste Disposal Act as amended by Resource Conservation and Recovery Act of 1976, as amended, 42 U.S.C. 6912(a), 6926, and 6974(b). EPA Delegation 8-7)

Dated: September 28, 1984.

**Thomas P. Eichler,**

*Regional Administrator.*

[FR Doc. 84-28289 Filed 10-25-84; 8:45 am]

BILLING CODE 6560-50-M

## FEDERAL EMERGENCY MANAGEMENT AGENCY

**44 CFR Part 67**

**[Docket No. 6625]**

## Proposed Flood Elevation Determinations; Alabama et al.

*Correction*

In FR Doc. 84-27112 beginning on page 40901 in the issue of Thursday, October 18, 1984, make the following correction:

On page 40905, in the second entry for "Alabama", second column, insert "of Mobile County" after "Unincorporated Areas".

BILLING CODE 1505-01-M

## DEPARTMENT OF HEALTH AND HUMAN SERVICES

**Office of the Secretary**

**45 CFR Part 90**

## Nondiscrimination on the Basis of Age in Programs or Activities Receiving Federal Financial Assistance; Evaluation of Final Rules

**AGENCY:** HHS.

**ACTION:** Opportunity for public comment on effectiveness of final rules.

**SUMMARY:** This notice solicits public comments on the effectiveness of the general regulations implementing the Age Discrimination Act of 1975, as amended. These rules are at 45 CFR Part 90. The purpose of the public comment period is to help the Department conduct an evaluation of the general regulations.

**DATE:** Comments should be submitted by December 26, 1984.

**ADDRESS:** Comments should be submitted to Office for Civil Rights, Department of Health and Human Services, 330 Independence Ave., SW., Room 5400, Washington, D.C. 20201.

**FOR FURTHER INFORMATION CONTACT:** Kathleen O'Brien at the above address, telephone 245-6700 (TTY No. 202-472-2916).

**SUPPLEMENTARY INFORMATION:**

### A. Background

*1. Issuance of General Rules*

In November of 1975, Congress enacted the Age Discrimination Act prohibiting, subject to several exceptions, discrimination on the basis of age in programs or activities receiving Federal financial assistance. In October of 1978, following a study and report by the United States Commission on Civil Rights (required by the original Act), Congress enacted several amendments to the Act.

The Age Discrimination Act, as amended, provides that "no person in the United States shall, on the basis of age, be excluded from participation in, be denied the benefits of, or be subject to discrimination under, any program or activity receiving Federal financial assistance." The Act specified exceptions for: (1) Any program or activity established under the authority of any law which provided benefits or assistance or describes beneficiaries in age-related terms; (2) actions which reasonably take into account age as a factor necessary to the normal operation of the program or activity; (3) actions which reasonably take age into account as a factor necessary to the achievement of any statutory objective of the program or activity; and (4) any age distinction based upon reasonable factors other than age.

The Act also directed the Secretary of Health, Education and Welfare to issue general regulations, to be effective not earlier than July 1, 1979, to implement the Act. Thereafter, the Act required each department or agency which extends Federal financial assistance to programs or activities covered by the Act to issue agency-specific regulations, which must be consistent with the general regulations and be approved by the Secretary of HEW (now HHS).

On June 12, 1979, HEW issued final general regulations. In its most important provisions, the regulations set forth specific rules against age discrimination and for the application of the statutory exceptions. The specific rules against age discrimination prohibit any age distinction which has the purpose or effect of discriminating on the basis of age. The "any law" exception was made applicable to any Federal, State or local statute or ordinance adopted by an elected, general purpose legislative body. The "normal operation" and "statutory objective" exceptions were made applicable where the recipient of Federal funds proves that: (a) Age is used to measure other characteristics; (b) the other characteristics must be measured; (3) age is a reasonable proxy for the other characteristics; and (d) it is impractical to measure directly the other characteristics. The exemption for reasonable factors other than age was made applicable where the recipient proves the factor bears a direct and substantial relationship to the normal operation of the program or the achievement of a statutory objective.

The general regulations also provided that the Department would, after 30 months, review the effectiveness of the general regulations, and would provide an opportunity for public comment as part of this review.

*2. Implementation Activities*

Although, as discussed below, issuance of agency-specific regulations has been delayed, there have been substantial implementation activities by federal agencies pursuant to the general regulations. For example, under § 90.43(c) of the general regulations, departments and agencies have established procedures to handle age discrimination complaints. This complaint process includes referral to the Federal Mediation and Conciliation Service for mediation efforts to resolve complaints. During fiscal years 1979

through 1983, 470 age discrimination complaints have been received and handled by federal agencies. The vast majority (429) of these complaints were to five agencies: The Department of Education, Housing and Urban Development, Health and Human Services, Agriculture, and the Civil Aeronautics Board.

In addition to the handling of age discrimination complaints, a number of agencies have conducted compliance reviews of recipients. For example, in fiscal year 1983: HHS conducted 221 pre-grant reviews that included compliance with the Age Discrimination Act; the Small Business Administration conducted 831 post-award reviews; the Department of Interior conducted 102 compliance reviews; and the Tennessee Valley Authority completed a program evaluation concerning the participation of the elderly in its Home Insulation Program.

Other implementation activities include specific actions taken by a number of agencies to inform recipients of their obligations under the Age Discrimination Act and to train appropriate agency staffs in matters relating to ADA enforcement.

*3. Progress in the Issuance of Agency Regulations*

Delays have been encountered in completing the process of issuing agency regulations. These were principally associated with questions raised by the Office of Management and Budget about the requirement that each recipient employing 15 or more employees complete a written self-evaluation of its compliance with the Act. Litigation that followed resolution of the self-evaluation issue lengthened these delays.

On December 28, 1982, the HHS agency-specific regulation, which included a revised self-evaluation requirement, approved by OMB, was published. At this point, however, litigation, *Action Alliance of Senior Citizens of Greater Philadelphia* v. *Heckler*, No. 83–0285 (D.D.C., filed February 2, 1983), was instituted which challenged the validity of the self-evaluation provision and two other procedural provisions of the HHS regulations. Because the outcome of this litigation could have affected the provisions of other agency regulations, final HHS clearance of those regulations was further delayed.

On March 19, 1984, the court dismissed those aspects of the litigation challenging provisions of the HHS regulations, and on June 27, 1984, denied the plaintiffs' motion for reconsideration. Therefore, on July 13, 1984, HHS advised the 17 agencies that had submitted rules to HHS for review that their regulations were approved (some of the approvals conditioned upon certain revisions being adopted). HHS also urged the nine agencies that have not yet submitted regulations (seven of which has issued proposed rules, but have not submitted their final rules) to expedite completion of their regulations. HHS anticipates substantial progress over the next several months toward the adoption of implementing regulations by all the enforcing agencies.

**B. Comments Solicited on Provisions of General Regulations**

The Department solicits public comments on provisions of the general regulations. The Department is interested in comments concerning the effectiveness of those provisions, suggestions for changes in the provisions of the regulations, and suggestions for administrative actions to increase the effectiveness of the compliance and enforcement activities. To facilitate the comment process, the following identifies issues, grouped under headings corresponding to the respective subparts of the general regulations, on which comments are particularly solicited:

*1. General*

Subpart A describes the basic purposes of the Age Discrimination Act and the general regulations, sets forth the Department's interpretation of the statutory "any law" exception, and defines terms used in the rules. Comments are solicited on any of these issues.

*2. Standards for Determining Discriminatory Practices*

Subpart B sets forth the standards for determining discriminatory practices. Most importantly, it established a framework of strict scrutiny review of age distinctions in applying the "normal operation", "statutory objective," and "reasonable factors other than age" statutory exceptions. Under this framework, age distinctions fall within the statutory exceptions only if the recipient proves that: (a) Age is used to measure other characteristics, (b) the other characteristics must be measured, (c) age is a reasonable proxy for the other characteristics; and (d) it is impractical to measure directly the other characteristics. Comments are solicited on these standards.

*3. Responsibilities of Federal Agencies*

Subpart C of the general regulations sets forth the responsibilities of Federal agencies which extend Federal financial assistance to programs and activities. Included in the subpart is a requirement that such agency review the age distinctions it imposes on its recipients by regulations, policies and administrative practices to determine whether those age distinctions are permissible. This subpart also requires that age distinctions not contained in regulations may not be continued. In addition to comments regarding the provisions of this subpart, the Department would welcome comments from the public giving examples of age distinctions contained in regulations, policies and administrative practices of agencies which are believed to be inconsistent with the requirements of the Act and the general regulations.

*4. Procedures*

Subpart D of the general regulations sets forth procedures concerning voluntary compliance efforts, notice to recipients regarding obligations under the Act, technical assistance to recipients, educational materials to recipients and beneficiaries under the Act, self-evaluations of recipient compliance, compliance reviews, information collection and reporting authorities, remedial and affirmative action, age distinctions that provide special benefits to children or the elderly, exhaustion of administrative remedies, and other matters.

In addition to comments regarding these provisions, the Department welcomes suggestions for administrative actions pursuant to these procedures to effectuate the purpose of the Act, including examples of practices believed to be in violation of the Act of particular categories of recipients, to which enforcement procedures (such as compliance reviews, self-evaluations, and data collection) should be targeted. The Department also solicits comments on the effectiveness of the mediation process established in this subpart and on whether the "special benefits" provision adequately accomplishes the congressional intent that age distinctions reasonably providing special benefits to children or the elderly should not be disturbed.

*5. Review of Regulations*

Subpart E of the general regulation sets forth the requirement, of which this notice of opportunity for public comment is a part, for a review of the effectiveness of the regulations. Comments on any matter contained in the general regulations and on any other matter related to the implementation and enforcement of the Age Discrimination Act of 1975 are welcome.

Dated: July 18, 1984.

**Betty Lou Dotson,**

*Director, Office of Civil Rights.*

Approved: October 22, 1984.

**Margaret M. Heckler,**

*Secretary, Department of Health and Human Services.*

[FR Doc. 84-28349 Filed 10-25-84; 8:45 am]

BILLING CODE 4150-04-M

## DEPARTMENT OF THE INTERIOR

### Fish and Wildlife Service

### 50 CFR Part 17

### Endangered and Threatened Wildlife and Plants; Public Hearing and Reopening of Comment Period on Proposed Endangered Status for Acanthomintha Obovata Duttonii (San Mateo Thornmint) and Public Hearing and Extension of Comment Period on Proposed Endangered Status and Critical Habitat for the Bay Checkerspot Butterfly (Euphydryas Editha Bayensis)

**AGENCY:** Fish and Wildlife Service, Interior.

**ACTION:** Proposed rules; notice of public hearing and reopening and extension of comment periods.

**SUMMARY:** The U.S. Fish and Wildlife Service gives notice that a public hearing will be held on the proposed determination of endangered status for *Acanthomintha Obovata Duttonii* (San Mateo thornmint), and that in order to accommodate the hearing, the comment period on the proposal is reopened. The public hearing will also concern the proposed determination of endangered status and critical habitat for the bay checkerspot butterfly (*Euphydryas Editha Bayensis*), and that the comment period on the proposal is extended. The thornmint is found only in central San Mateo County near the town of Redwood City, California. The butterfly was historically known from the San Francisco Peninsula and outer Coast Range to the south and east of the peninsula. Of the 16 known colonies, only five remain. This hearing and the reopening and extension of the comment periods for these two species will allow comments on these proposals to be submitted from all interested parties.

**DATES:** The comment period on both of the proposals is reopened and extended effective (October 26, 1984). The public hearing will be held from 7 to 9 p.m., on Tuesday, November 13, 1984, in San Mateo, California. The comment period for the San Mateo thornmint and the bay checkerspot proposals, which originally closed respectively on August 7, 1984 and November 13, 1984, will now both close November 23, 1984.

**ADDRESSES:** The public hearing will be held at the Hillsdale Inn, Tudor Room, 477 E. Hillsdale Boulevard, San Mateo, California. Written comments and materials should be sent to the Regional Director, U.S. Fish and Wildlife Service, Lloyd 500 Building, 500 NE. Multnomah Street, Suite 1692, Portland, Oregon 97232. Comments and materials received will be available for public inspection during normal business hours, by appointment, at the Regional Endangered Species Division at the above Regional Office address.

**FOR FURTHER INFORMATION CONTACT:** For information on the public hearing, contact Mr. Gail Kobetich, Project Leader, U.S. Fish and Wildlife Service, Sacramento Endangered Species Office, 2800 Cottage Way, Room E-1823, Sacramento, California 95825 (916/484-4935 or FTS 468-4935).

**SUPPLEMENTARY INFORMATION:**

#### Background

The San Mateo thornmint is found only in central San Mateo County near the town of Redwood City, California. The plant is threatened by unauthorized and uncontrolled recreational activities such as disturbance by off-road vehicles and trampling by horses and people. A proposal of endangered status for the San Mateo thornmint was published in the **Federal Register** on June 18, 1984 (49 FR 24906). The bay checkerspot butterfly has suffered a reduction in number and range. Of 16 known colonies, five remain. Habitat alteration has resulted from highway and subdivision construction, overgrazing by livestock, and introduction of non-native plants. A proposal of endangered status and critical habitat for the butterfly was published in the **Federal Register** on September 11, 1984 (49 FR 35665).

Section 4(b)(5)(E) of the Endangered Species Act of 1973, as amended, requires that a public hearing be held, if requested within 45 days of the publication of a proposed rule. On July 25, 1984, a public hearing on the proposal for the San Mateo thornmint was requested by Mr. Paul M. Koenig, Department of Environmental Services, County of San Mateo. As a result of discussions with the county, the Service has decided to hold a joint public hearing for the thornmint and the bay checkerspot butterfly. The Service has scheduled this hearing for November 13, 1984, from 7 to 9 p.m., at the Hillsdale Inn, San Mateo, California (see Addresses above). Those parties wishing to make statements for the record should have available copies of their statements to be presented to the Service at the start of the hearing. Oral statements may be limited to 5 or 10 minutes, if the number of parties present that evening necessitates some limitation. There are no limits to the length of written comments presented at this hearing or mailed to the Service.

The comment peirod on the San Mateo thornmint and bay checkerspot butterfly proposals originally closed on August 17, 1984 and November 13, 1984, respectively. In order to accommodate the hearing, the Service also reopens and extends these public comment periods. Written comments may now be submitted for both proposals until November 23, 1984, to the Service office in the Addresses section.

#### Author

The primary author of this notice is Carolyn Bohan, U.S. Fish and Wildlife Service, Lloyd 500 Building, 500 N.E. Multnomah Street, Suite 1692, Portland, Oregon 97232 (503/231-6131).

#### List of Subjects in 50 CFR Part 17

Endangered and threatened wildlife, Fish, Marine mammals, Plants (agriculture).

(Endangered Species Act of 1973 (16 U.S.C. 1531 *et seq.*; Pub. L. 93-205, 87 Stat. 884; Pub. L. 94-359, 90 Stat. 911; Pub. L. 95-632, 92 Stat. 3751; Pub. L. 96-159, 93 Stat. 1225; Pub. L. 97-304, 96 Stat. 1411)

Dated: October 17, 1984.

**James W. Teeter,**

*Acting Regional Director.*

[FR Doc. 84-28355 Filed 10-25-84; 8:45 am]

BILLING CODE 4310-55-M

## DEPARTMENT OF COMMERCE

### National Oceanic and Atmospheric Administration

### 50 CFR Part 630

### Gulf of Mexico Fishery Management Council

**AGENCY:** National Marine Fisheries Service(NMFS), NOAA, Commerce.

**ACTION:** Swordfish Fishery Management Plan; notice of a public hearing and request for comments.

**SUMMARY:** The Gulf of Mexico Fishery Management Council, established by section 302 of the Magnuson Fishery Conservation and Management Act of 1976 (Pub. L. 94-265), will hold a swordfish public hearing in conjunction with the upcoming Council meeting to allow for input on the changes to the

draft Swordfish Fishery Management Plan.

**DATES:** Written comments will be accepted until November 30, 1984. The hearing portion of the meeting will convene at 9:00 a.m. and will adjourn at approximately 9:30 a.m. on Wednesday, November 14, 1984.

**ADDRESSES:** Send comments to Wayne Swingle, Executive Director, Gulf of Mexico Fishery Management Council, Lincoln Center, Suite 881, 5401 Kennedy Boulevard, Tampa, Florida 33609; or Jack Brawner, Regional Director, National Marine Fisheries Service, Southeast Region, 9450 Koger Boulevard, St. Petersburg, Florida 33702.

The hearing will take place at The Lincoln Hotel, Kennedy Square Boulevard, Tampa, Florida 44609.

**FOR FURTHER INFORMATION CONTACT:** Wayne Swingle, Executive Director, Gulf of Mexico Fishery Management Council, 813–228–2815.

Dated: October 23, 1984.

**Joseph W. Angelovic,**

*Deputy Assistant Administrator for Science and Technology, National Marine Fisheries Service.*

[FR Doc. 84-28346 Filed 10-25-84; 8:45 am]

BILLING CODE 3510-22-M

# Notices

Federal Register
Vol. 49, No. 209
Friday, October 26, 1984

This section of the FEDERAL REGISTER contains documents other than rules or proposed rules that are applicable to the public. Notices of hearings and investigations, committee meetings, agency decisions and rulings, delegations of authority, filing of petitions and applications and agency statements of organization and functions are examples of documents appearing in this section.

## COMMISSION ON CIVIL RIGHTS

### Alaska Advisory Committee; Agenda and Notice of Public Meeting

Notice is hereby given, pursuant to the provisions of the Rules and Regulations of the U.S. Commission on Civil Rights, that a meeting of the Alaska Advisory Committee to the Commission will convene at 1:00 p.m. and will end at 5:00 p.m., on November 19, 1984, at the Federal Building, Room C–105, 701 C Street, Anchorage, Alaska 99513. The purpose of the meeting is to plan for future programs.

Persons desiring additional information, or planning a presentation to the Committee, should contact the Northwestern Regional Office at (206) 442–1246.

The meeting will be conducted pursuant to the provisions of the Rules and Regulations of the Commission.

Dated at Washington, D.C., October 22, 1984.

**John I. Binkley,**

*Advisory Committee Management Officer.*

[FR Doc. 84–28287 Filed 10–25–84; 8:45 am]

BILLING CODE 6335–01–M

### Delaware Advisory Committee; Agenda and Notice of Public Conference

Notice is hereby given, pursuant to the provisions of the Rules and Regulations of the U.S. Commission on Civil Rights, that a conference of the Delaware Advisory Committee to the Commission will convene at 8:30 a.m. and will end at 6:00 p.m., on November 19, 1984, at the Radisson Hotel, Hornkill Room, 700 King Street, Wilmington, Delaware 19801. The purpose of the conference is to identify civil rights issues of major importance in the State.

Persons desiring additional information, or planning a presentation to the Committee, should contact the Mid-Atlantic Regional Office at (202) 254–6670.

The meeting will be conducted pursuant to the provisions of the Rules and Regulations of the Commission.

Dated at Washington, D.C., October 22, 1984.

**John I. Binkley,**

*Advisory Committee Management Officer.*

[FR Doc. 84–28286 Filed 10–25–84; 8:45 am]

BILLING CODE 6335–01–M

## DEPARTMENT OF COMMERCE

### International Trade Administration

[A–479–063]

### Animal Glue and Inedible Gelatin From Yugoslavia; Final Results of Administrative Review of Antidumping Finding

**AGENCY:** International Trade Administration/Import Administration, Department of Commerce.

**ACTION:** Notice of Final Results of Administrative Review of Antidumping Finding.

**SUMMARY:** On July 9, 1984, the Department of Commerce published the preliminary results of its administrative review of the antidumping finding on animal glue and inedible gelatin from Yugoslavia. The review covers the one known exporter of this merchandise to the United States and the period December 1, 1982, through November 30, 1983. There were no known shipments of this merchandise to the United States during the period and there are no known unliquidated entries

We gave interested parties an opportunity to comment on the preliminary results. We received no comments. Based on our analysis, the final results of review are unchanged from those presented in the preliminary results.

**EFFECTIVE DATE:** October 26, 1984.

**FOR FURTHER INFORMATION CONTACT:** Dennis U. Askey or Robert J. Marenick, Office of Compliance, International Trade Administration, U.S. Department of Commerce, Washington, D.C. 20230; telephone: (202) 377–2923/5255.

**SUPPLEMENTARY INFORMATION:**

#### Background

On July 9, 1984, the Department of Commerce ("the Department") published in the **Federal Register** (49 FR 27963) the preliminary results of its administrative review of the antidumping finding on animal glue and inedible gelatin from Yugoslavia (42 (FR 641167, December 22, 1977). The Department has now completed that administrative review.

#### Scope of the Review

Imports covered by the review are shipments of animal glue and inedible gelatin of which there are two principal types, hide glue and bone glue. Animal glue is an organic colloid of protein derivation. There is no significant difference between animal glue and inedible gelatin. Animal glues are odorless, dry, hard, hornlike materials. They are used as general purpose adhesives in industries producing abrasives, paper containers, book and magazine bindings, and leather goods. They are also used as sizing agents and as colloids in emulsions and cleaning compounds. Animal glue and inedible gelatin are currently classifiable under items 455.4000 and 455.4200 of the Tariff Schedules of the United States Annotated.

The review covers the one known exporter of Yugoslavian animal glue and inedible gelatin to the United States, Kemija-Impex, and the period December 1, 1982, through November 30, 1983. There were no known shipments of this merchandise to the United States during the period and there are no known unliquidated entries.

#### Final Results of the Review

We gave interested parties an opportunity to comment on the preliminary results. The Department received no written comments or requests for a hearing. Based on our analysis, the final results of our review are the same as those presented in the preliminary results of review. As provided for in § 353.48(b) of the Commerce Regulations, a cash deposit of estimated antidumping duties of 9.7 percent shall be required on all shipments of Yugoslavian animal glue and inedible gelatin entered, or withdrawn from warehouse, for consumption on or after the date of publication of this notice. This deposit requirement shall remain in effect until publication of the final results of the next administrative review. The Department intends to begin

immediately the next administrative review.

The Department encourages interested parties to review the public record and submit applications for protective orders as early as possible after the Department's receipt of the requested information.

This administrative review and notice are in accordance with section 751(a)(1) of the Tariff Act of 1930 (19 U.S.C. 1675(a)(1)) and § 353.53 of the Commerce Regulations (19 CFR 353.53).

Dated: October 18, 1984.

**Alan F. Holmer,**

*Deputy Assistant Secretary for Import Administration.*

[FR Doc. 84-28318 Filed 10-25-84; 8:45 am]

BILLING CODE 3510-DS-M

---

[A-201-034]

## Elemental Sulphur From Mexico; Final Results of Administrative Review of Antidumping Finding

**AGENCY:** International Trade Administration/Import Administration, Department of Commerce.

**ACTION:** Notice of Final Results of Administrative Review of Antidumping Finding.

**SUMMARY:** On August 6, 1984, the Department of Commerce published the preliminary results of its administrative review of the antidumping finding on elemental sulphur from Mexico. The review covers the two known exporters of this merchandise to the United States currently covered by the finding and the period June 1, 1982, through May 31, 1983.

We gave interested parties an opportunity to submit oral or written comments on the preliminary results. We received no comments. Based on our analysis, the final results of our review are the same as our preliminary results.

**EFFECTIVE DATE:** October 26, 1984.

**FOR FURTHER INFORMATION CONTACT:** Mery Helen Askins or Robert J. Marenick, Office of Compliance, International Trade Administration, U.S. Department of Commerce, Washington, D.C. 20230, telephone: (202) 377-1130/5255.

**SUPPLEMENTARY INFORMATION:**

### Background

On August 6, 1984, the Department of Commerce ("the Department") published in the **Federal Register** (49 FR 31311) the preliminary results of its administrative review of the antidumping finding on elemental sulphur from Mexico (37 FR 12727, June 28, 1972). The Department has now completed that administrative review in accordance with section 751 of the Tariff Act of 1930 ("the Tariff Act").

### Scope of the Review

Imports covered by the review are shipments of elemental sulphur. Basically there are two types of sulphur, "bright" and "dark" sulphur. Chemically these two types are almost equal, the dark sulphur being discolored by certain hydrocarbon impurities. The single greatest use of sulphur is in the manufacture of sulphuric acid. In elemental form or as sulphuric acid it enters into the production or processing of hundreds of products. Among the most important are fertilizers, chemicals, titanium and other pigments, pulp and paper, rayon, film, iron and steel, dyestuffs, vulcanized and synthetic rubber, insecticides, fungicides, fuels and explosives. Elemental sulphur is currently classifiable under item 415.4500 of the Tariff Schedules of the United States Annotated.

The review covers the two known exporters of Mexican elemental sulphur to the United States currently covered by the finding, Agro Centro, S.A. and Prohulsa, S.A., and the period June 1, 1982, through May 31, 1983.

### Final Results of the Review

We gave interested parties an opportunity to comment on the preliminary results. The Department received no written comments or requests for a hearing. Based on our analysis, the final results of our review are the same as those presented in the preliminary results of the review, and we determine that a margin of 33 percent exists for those two firms for the period June 1, 1982, through May 31, 1983.

The Department shall determine, and the Customs Service shall assess, dumping duties on all appropriate entries. The Department will issue appraisement instructions on each exporter directly to the Customs Service.

Further, as provided for in § 353.48(b) of the Commerce Regulations, a cash deposit of estimated antidumping duties of 33 percent based on the above margin shall be required for those two firms. For any future entries from a new exporter not covered in this or prior administrative reviews, whose first shipments occurred after May 31, 1983, and who is unrelated to any reviewed firm, a cash deposit of 33 percent shall be required. These deposit requirements are effective for all shipments of Mexican elemental sulphur entered, or withdrawn from warehouse for consumption, on or after the date of publication of this notice and shall remain in effect until publication of the final results of the next administrative review. The Department intends to begin immediately the next administrative review.

The Department encourages interested parties to review the public record and submit applications for protective orders as early as possible after the Department's receipt of the requested information.

This administrative review and notice are in accordance with section 751(a)(1) of the Tariff Act (19 U.S.C. 1675(a)(1)) and § 353.53 of the Commerce Regulations (19 CFR 353.53).

Dated: October 18, 1984.

**Alan F. Holmer,**

*Deputy Assistant Secretary for Import Administration.*

[FR Doc. 84-2837 Filed 10-25-84; 8:45 am]

BILLING CODE 3510-DS-M

---

[C-588-047]

## Chain of Iron or Steel From Japan; Final Results of Administrative Review of Countervailing Duty Order

**AGENCY:** International Trade Administration/Import Administration, Department of Commerce.

**ACTION:** Notice of Final Results of Administrative Review of Countervailing Duty Order.

**SUMMARY:** On June 26, 1984, the Department of Commerce published the preliminary results of its administrative review of the countervailing duty order on chain of iron or steel from Japan. The review covers the period January 1, 1983, through December 31, 1983.

We gave interested parties an opportunity to comment on the preliminary results. We received no comments. Based on our analysis, the final results of the review are the same as the preliminary results.

**EFFECTIVE DATE:** October 26, 1984.

**FOR FURTHER INFORMATION CONTACT:** Al Jemmott or Richard Moreland, Office of Compliance, International Trade Administration, U.S. Department of Commerce, Washington, DC 20230; telephone: (202) 377-2786.

**SUPPLEMENTARY INFORMATION:**

### Background

On June 26, 1984, the Department of Commerce ("the Department") published in the **Federal Register** (49 FR 26126) the preliminary results of its administrative review of the countervailing duty order on chain of iron or steel from Japan (43 FR 37685,

August 24, 1978). The Department has now completed that administrative review, in accordance with section 751 of the Tariff Act of 1930 ("the Tariff Act").

**Scope of the Review**

Imports covered by the review are shipments of Japanese chain of iron or steel, the links of which are essentially round in cross section, and parts thereof. Such merchandise is currently classifiable under items 652.2410 through 652.2450, 652.2710 through 652.2740, 652.3010 through 652.3040, 652.3310 through 652.3330, and 652.3510 through 652.3530 of the Tariff Schedules of the United States Annotated. The review covers the period January 1, 1983, through December 31, 1983, and a program of tax deferrals on funds held in the Overseas Market Development Reserve ("OMDR").

**Final Results of the Review**

We gave interested parties an opportunity to comment on the preliminary results. We received no comments. Based on our analysis, the final results of the review are the same as the preliminary results. We determine the aggregate net subsidy to be 1.95 percent *ad valorem* for the period January 1, 1983, through December 31, 1983.

On November 17, 1982, the International Trade Commission ("the ITC") notified the Department that the Government of Japan had requested an injury determination for this order under section 104(b) of the Trade Agreements Act of 1979. Should the ITC find that there would be material injury or threat of material injury to an industry in the United States if the order were revoked, the Department will instruct the Customs Service to assess countervailing duties in the amount of the estimated duties required to be deposited on all unliquidated entries of Japanese chain of iron or steel entered, or withdrawn from warehouse, for consumption on or after November 17, 1982, and through the date of the ITC's notification to the Department of its determination.

The Department will instruct the Customs Service to collect a cash deposit of estimated countervailing duties, as provided for in section 751(a)(1) of the Tariff Act, of 1.95 percent of the entered value on any shipment of Japanese chain of iron or steel entered, or withdrawn from warehouse, for consumption on or after the date of publication of this notice. This deposit requirement shall remain in effect until publication of the final results of the next administrative review. The Department intends to begin immediately the next administrative review.

The Department encourages interested parties to review the public record and submit applications for protective orders as early as possible after the Department's receipt of the requested information.

This administrative review and notice are in accordance with section 751(a)(1) of the Tariff Act (19 U.S.C. 1675(a)(1)) and § 355.41 of the Commerce Regulations (19 CFR 355.41).

Dated: October 22, 1984.

**Alan F. Holmer,**

*Deputy Assistant Secretary, Import Administration.*

[FR Doc. 84-28359 Filed 10-25-84; 8:45 am]

BILLING CODE 3510-DS-M

---

**[A-588-021]**

## Final Determination of Sales at Less Than Fair Value; Cell Site Transceivers From Japan

**AGENCY:** International Trade Administration, Import Administration.

**ACTION:** Notice.

**SUMMARY:** We determine that cell site transceivers from Japan are being sold, or are likely to be sold, in the United States at less than fair value. We have notified the United States International Trade Commission (ITC) of our determination, and we have directed the U.S. Customs Service to continue to suspend liquidation of all entries of the subject merchandise. We have directed the U.S. Customs Service to require a cash deposit or the posting of a bond for each such entry in an amount equal to the estimated dumping margins, as described in the "Suspension of Liquidation" section of this notice. We also determined that critical circumstances exist with respect to imports of cell site transceivers from Japan.

**EFFECTIVE DATE:** October 26, 1984.

**FOR FURTHER INFORMATION CONTACT:** Vincent Kane, Office of Investigations, Import Administration, International Trade Administration, Department of Commerce, 14th Street and Constitution Avenue, NW., Washington, D.C. 20230; telephone: (202) 377-5414.

**SUPPLEMENTARY INFORMATION:**

**Final Determination**

Based on our investigation and in accordance with section 735(a) of the Tariff Act of 1930, as amended (19 U.S.C. 1673d(a)) (the Act), we determine that cell site transceivers from Japan are being sold in the United States at less than fair value, within the meaning of section 731 of the Act.

We found that the foreign market value of cell site transceivers from Japan exceeded the United States price on all sales. The overall weighted-average margin on all sales compared is 59.94 percent.

**Case History**

On December 28, 1983, we received a petition from E.F. Johnson and Company on behalf of the cell site transceivers industry in the United States. In accordance with the filing requirements of § 353.36 of our regulations (19 CFR 353.36), the petition alleged that imports of cell site transceivers from Japan are being, or are likely to be, sold in the United States at less than fair value, within the meaning of section 731 of the Act, and that these imports are injuring, or threatening to injure, a United States industry. The petition alleged that sales of cell site transceivers were being made at less than the cost of production. The petition also alleged, pursuant to section 733(e) of the Act, that "critical circumstances" exist in this case.

After reviewing the petition, we determined it contained sufficient grounds to initiate an antidumping investigation. We notified the ITC of our action and initiated the investigation on January 17, 1984 (49 FR 3100). On February 13, 1984, the ITC determined that there is a reasonable indication that imports of cell site transceivers are materially injuring a United States industry.

On March 7, 1984, we presented an antidumping questionnaire to Kokusai Electric Company, Ltd. (Kokusai), the sole Japanese manufacturer selling the subject merchandise for export to the United States. We reviewed a response from Kokusai on April 16, 1984, and verified the response during the period May 9 through May 12, 1984. On June 5, 1984, we preliminarily determined that there is a reasonable basis to believe or suspect that cell site transceivers from Japan are being, or are likely to be sold at less than fair value and that "critical circumstances" do exist with respect to cell site transceivers from Japan (49 FR 24155). On August 10, 1984, we published a notice postponing our final determination from August 20, 1984, until October 19, 1984, at the request of counsel for the respondent in accordance with section 735(a)(2)(A) of the Act (49 FR 32096). We received supplementary responses on August 13 and August 20, 1984, and verified these responses in Japan during the period September 10 through September 19, 1984.

On October 1, 1984, in accordance with requests from counsel for petitioners and counsel for respondents, a public hearing was held.

### Scope of Investigation

The merchandise covered by this investigation is cell site transceivers and related subassemblies, as provided for in item 685.2976 of the Tariff Schedules of the United States Annotated. Cell site transceivers and related subassemblies are part of the radio frequency (RF) equipment in the base station (cell site) of a cellular radio communications systems. This single package RF equipment functions as a locating receiver and provides simultaneous two-way voice and data communications between the base station and the subscriber's mobile telephone by using different frequencies to transmit and receive. Subassemblies are an assemblage of component parts dedicated for use in cell site transceivers as defined above.

### Fair Value Comparison

To determine whether sales of the subject merchandise in the United States were made at less than fair value, we compared the United States price with the foreign market value.

### United States Price

As provided in section 772(b) of the Act, we used the purchase price of the subject merchandise to represent the United States price because the merchandise was sold to an unrelated U.S. purchaser prior to its importation into the United States. We calculated the purchase price based on the f.o.b. price, El Segundo, California. We deducted port charges, inland freight, ocean freight and insurance costs incurred in delivering the product.

### Foreign Market Value

In accordance with section 773(a)(2) of the Act, we used "Constructed Value" to determine the foreign market value, because Kokusai Electric Company, Ltd. has not sold a product "such or similar" to that sold in the U.S. in either its home market or in a third country. To determine constructed value we examined production costs, including materials, labor, research and development, other manufacturing costs, selling, other general expenses and profit.

On February 23, 1983, Kokusai entered into a contract to sell cell site transceiver units to a U.S. buyer. The company had not previously manufactured this product. Production began in late 1983. Consequently, cost information available at the time of our preliminary determination covered the period from the beginning of production through March 1984 and included certain costs which were related to start-up production. In order for the constructed value to reflect the appropriate production cost, it was based on "normalized costs of production". "Normalized costs" were the weighted-average costs for the units to be produced under the contract based on: (1) Actual costs (including start-up costs) incurred through March 1984; and (2) those anticipated costs which were supported by contracts or other credible documentation for the remaining number of units needed to fulfill the contract commitment. Under this method, start-up costs were amortized, on a pro-rata basis, over the total number of units to be manufactured under the contract.

In determining the constructed value in our preliminary determination, we made certain revisions to the cost elements submitted by the respondent. The Department used: (1) Actual costs incurred in the production of component parts manufactured by other divisions of Kokusai, instead of transfer prices, (2) revised cost projections which reflected estimates supported by verified information, (3) overhead costs in addition to the direct costs of research and development, and (4) in certain instances where the respondent had been unable to provide sufficient data, the best information available. We used the statutory 10 percent for general expenses because actual expenses did not meet the minimum of 10 percent of the sum of material and fabrication costs required by section 773(e)(1)(B)(i) of the Act. We calculated profit based on the 8 percent minimum, as prescribed in section 773(e)(1)(B)(ii) of the Act, since the actual profit was less than 8 percent. We made currency conversions from the Japanese yen to the U.S. dollar in accordance with § 353.56(a)(1) of our regulations.

Our final determination was based on verified cost information relating to production through the end of July 1984. We used weighted-average costs for the units produced under the contract based on the actual costs (including start-up costs) incurred for their production. We revised the costs as presented by the respondent in its submissions of August 20 related to direct labor, indirect overhead expense and factory administrative costs.

We used the actual general, administrative and selling expenses which exceeded the statutory 10 percent of the material and fabrication costs. We used 15.64 percent for profit, which was the "best information" representing the profit of a product in the "same general class or kind" as the transceiver. This profit was based on an analysis of the profit margin for several Japanese firms engaged in the production and sale of communications equipment in Japan.

### Determination of Critical Circumstances

Petitioner alleged that imports of cell site transceivers from Japan present "critical circumstances." Under section 735(a)(3) of the Act (19 U.S.C. 1673d(a)(3)), critical circumstances exist when the Department finds that: (1) (a) There is a history of dumping in the United States or elsewhere of the merchandise under investigation, or (b) the person by whom, or for whose account, the merchandise was imported knew or should have known that the exporter was selling the merchandise under investigation at less than fair value; and (2) there have been massive imports of the merchandise under investigation over a relatively short period.

Based upon our analysis of the information, we determine there is no history of dumping. We then considered whether the person by whom, or for whose account, these products were imported knew or should have known that the exporter was selling these products at less than fair value. It is the Department's position that a reasonable basis the importer knew or should have known that a product was being sold at less than its fair value exists where margins calculated on the basis of responses to the Department's questionnaire are sufficiently large. In this case the weighted-average margin is 59.94 percent. Where, as here, there is a corporate relationship between the exporter and the importer of record, margins of this size indicate that the importer of record, Kokusai Electric Company of America, knew or should have known that prices on sales for export to the United States (as adjusted according to the antidumping law) were below the foreign market value. Consequently, we find that the importer knew or should have known that the merchandise was being sold in the United States at less than fair value.

We also find that imports of the product subject to this investigation appear massive over a relatively short period. In reaching this determination, we considered the specific circumstances surrounding Kokusai's contract with its U.S. buyers. First, at the time the contract was entered into, it represented a substantial portion of the U.S. market. Second, even with increased demand, the market remains relatively small in terms of the number

of units needed to fill current demand. Third, Kokusai began deliveries shortly before the petition was filed. In the interim between the filing of the petition and the preliminary determination Kokusai's deliveries increased rapidly and significantly. Consequently, on the basis of our analysis of the information, we determine that imports of the product subject to this investigation appear massive over a relatively short period.

For reasons described above, we determine that "critical circumstances" do exist with respect to cell site transceivers from Japan.

**Petitioner's Comments**

*Comment 1*

The petitioner claims that other products manufactured by Kokusai are less sophisticated technologically and sold in higher volume, yet are sold at substantially higher prices than the transceiver.

This price disparity constitutes evidence that cell site transceivers are being sold at less than fair value and the disparity should be considered by the Department in its final determination by adopting the profit in the home market of merchandise which is in the same "general class or kind."

*DOC Response*

The products referred to by the petitioner—"mobile subscriber unit," "Japan personal radio" and "Redicom radio"—were not considered to be "such or similar merchandise" to the transceiver for purposes of our investigation. Kokusai's home market sales of these items could, therefore, not serve as the basis for foreign market value. Consequently, the Department did not obtain sales or cost information pertaining to these products for the period of investigation.

For purposes of calculating profit, however, as defined under the constructed value provision of the Act, the Department requested the profit margin for these products to be used as a reference point in establishing profit for merchandise of the same general class or kind as the transceivers. However, such data could not be satisfactorily verified by the Department, and, therefore, was not used in this determination.

*Comment 2*

The petitioner claims that Kokusia's justification for its low costs—(1) off the shelf technology, and (2) efficient production methodology—would not account for low costs because other expenses like prototype design and equipment costs would be incurred.

*DOC Response*

The Department verified the capital costs (depreciation) related to the production of transceivers. Pre-and-post production prototype expenses were included in research and development.

*Comment 3*

The petitioner suggests that the reasons for the differences in the prices paid by Kokusai and the U.S. market prices for the same type of components, such as quantity purchased and distance from vendor should be ascertained.

*DOC Response*

The Department verified the prices paid by Kokusai to unrelated suppliers for 60 percent of these components and used these actual prices for calculating the constructed value. It is not the Department's practice to compare prices paid by the manufacturer under investigation with U.S. market prices for components, since the Department bases its determination on the costs actually incurred by the manufacturer. It should be noted that the Department's product expert has indicated that all but a few parts could be considered "off the shelf" items, and that it is usual practice for the prices of components to be negotiated.

*Comment 4*

The petitioner claims that a decrease in the number of labor hours because of a decrease in the amount of testing is unlikely because: (1) The contract specifies the amount of testing required, and (2) economic principles would dictate that Kokusai test throughout the process.

*DOC Response*

The Department verified the actual hours required to produce the transceiver. These hours included testing. Kokusai does test throughout the process for early detection of defects and malfunctioning of the transceiver.

*Comment 5*

The petitioner claims that labor expenses should include fringe benefits, year-end bonuses and Japanese payroll-taxes.

*DOC Response*

It is the usual practice of the Department to include all fringe benefits as part of labor expenses. All fringe benefits, including year-end bonuses and any taxes paid by Kokusai, have been included in labor expenses.

*Comment 6*

The petitioner claims that the Department must examine Kokusai's basis of allocating manufacturing overhead costs to product groups and must be satisfied that overhead costs are allocated only to those products and product group which properly bear these costs.

*DOC Response*

The Department, as is its usual practice, reviewed Kokusai's basis for identifying and allocating overhead expenses of the Radio Communications Division, the Division in which the transceiver is produced. The Radio Communications Division is divided into a number of cost centers, one of which includes the costs pertaining to the transceivers and all other products manufactured on the "automatic" equipment. Certain of the overhead costs in question could be identified directly with the Division and others could be identified directly to the cost centers within the Division.

Kokusai allocated these overhead costs accordingly. However, Kokusai allocated certain other factory overhead costs to the Radio Communications Division based on full-time employees, production value or building space. It also allocated certain costs to the cost centers based on full-time employees or production value.

The Department did not accept the allocation based on full-time employees and reallocated the expenses to the cost centers within the Division on the basis of total labor hours. Allocation of overhead costs on the basis of full-time employees to the cost center where the employee is permanently assigned was not accepted because this method: (1) Would not attribute overhead costs, such as supervision and heating, to the many hours worked by the part-time employees, and (2) would disproportionately allocate these overhead costs to the areas where the full-time employees have been assigned.

The Department notes that: (1) The employees considered by the company to be "part-time" are not incidental or temporary but have been employed for many years, work a regular work week and are a significant part of the company's permanent work force, and (2) employees may work in areas other than those to which they are permanently assigned.

*Comment 7*

Petitioner claims that where it is possible to identify research and development costs (R&D) as being specific to a product rather than as

generally beneficial to overall operations, such costs must be directly allocated to that product and included in its manufacturing costs, regardless of the manufacturer's accounting practices concerning such costs.

*DOC Response*

To determine if R&D expense should be considered manufacturing expense of the product or general expense of the corporation, the Department considers the purpose of the expenditure. R&D expenses which can be identified directly with the product under investigation or to the area in which the product is manufactured are considered manufacturing expenditures and are part of "fabrication" costs in the constructed value calculation. R&D expenditure incurred for general corporate purposes are classified as a "general" expenses.

The Department reviewed the purposes for which Kokusai incurred its R&D expenses. For expenses which could be identified with the transceiver, the Department attributed these costs to the product and amortized such expenses over the production of transceivers ordered. Kokusai also identified such costs with the transceiver in its records as manufacturing expenses.

With respect to expenses which were identified with the Radio Communications Division, but not with a specific product, the Department allocated the R&D expenses to the products based on the relationship of R&D to direct costs of each group, i.e., R&D expenses as a percentage of material, labor and certain transportation costs. The Department did not accept Kokusai's basis (full-time employees) for allocating such costs to the groups and used instead for its allocation the total direct labor hours for each group, as discussed above.

Both the R&D costs directly identified with specific products and the R&D costs allocated among the products in the Radio Communications Division were considered manufacturing overhead by Kokusai and were part of the costs of manufacturing the product.

The company also incurred research and development expenses which were considered to be general expenses of the company and these were recorded as part of "general, selling and administrative" expenses on its financial statements.

*Comment 8*

The petitioner states that because Kokusai did not maintain inventory records of all components purchased for the transceivers, 5 percent of the invoiced costs for materials should be added as an inventory cost for waste. Alternatively, the yield rate for production of one of the components manufactured by Kokusai could be used.

*DOC Response*

Although inventory records for these components were not maintained by Kokusai, the company tracked the receipt of components by purchase slips. These slips reflected the quantity of components needed for each batch as well as additional components ordered in the event of spoilage.

The Department's product expert indicated that he found no evidence during his observations of the receipt and distribution of these parts to dispute the company's claims that it did not maintain inventories of the components. Therefore, the Department relied on the purchase slips for each batch and used the waste costs provided by the respondent.

*Comment 9*

The petitioner claims that tooling costs associated with the production of one of the components which were incurred prior to the current financial period should be included in the cost of that component.

*DOC Response*

The Department agrees and has made the appropriate adjustment.

**Respondent's Comment**

*Comment 1*

The respondent claims that the Department should: (1) Use the actual average labor costs incurred over the entire contract, or labor costs incurred in the most recent lots of completed production, and (2) use that labor rate, including part-time labor, which the company could actually attribute to the transceiver.

*DOC Response*

The Department used the average labor hours which were presented in the respondent's submission for its construced value calculation. The average was based on the total actual number of labor hours used to produce all the units required under the contract.

For the labor cost rate, the Department used the average labor cost rate for the Radio Communications Divisions of Kokusai, the division in which cell site transceivers are produced. This rate included full-time and part-time employees within the division. These employees were identified with the Radio Communications Division by the company's employee records during verification.

In this case where (1) the company has a permanent work force which consists of full-time and part-time employees, (2) workers generally could be, and in many cases are, interchangeable, (3) there is a material difference in the full labor cost rate between these types of employees, and (4) the difference in the full labor cost rate is a result of the employees being classified as full- or part-time employees, the Department applies the average labor rate of the Division.

*Comment 2*

The respondent claims that the Department erroneously included in its preliminary determination certain costs for internally produced parts, specifically (1) factory overhead which duplicated costs already included in overhead, and (2) labor costs, by failing to account for the "coefficient of efficiency" (an integral part of Kokusai's standard time calculation methodology).

*DOC Response*

Regarding the duplication of factory overhead costs, we note that Kokusai in both its original and its revised responses excluded general research and development costs incurred by a support division which produced certain components for the cell site transceiver. In both our preliminary and final determinations, we included a share of these general R & D expenses in our calculation of constructed value. We note that the company's books and records reflected the general R & D expenses as part of the factory overhead of the support division.

The Department did not accept the company's calculation for the "coefficient of efficiency" in the preliminary determination because the company did not provide source documentation to support the amount. The company provided in its revised response the actual hours worked in the mechanical division to produce certain parts. The Department used these actual labor hours for its final determination.

*Comment 3*

The respondent claims that for the preliminary determination the Department erred in: (1) Allocating the research and development expenses identified with the transceivers over only the transceivers produced for the initial contract, and (2) adjusting the company's submission for overhead expenses, by adding an "overhead" factor to Kokusai's R & D costs for the cell site transceiver.

*DOC Response*

Since the preliminary determination, the company has received additional orders for transceivers. The Department has accepted the respondent's basis of allocation and, accordingly, allocated research and development expenses identified with the transceivers over all orders.

During the second verification overhead expenses for research and development directly identified with the transceiver were reviewed. For the final determination the Department accepted the costs as presented by Kokusai and did not, as in the preliminary determination, adjust these costs for additional overhead.

*Comment 4*

On February 23, 1983, Kikusai entered into a contract with AT&T Technologies (then Western Electric) for the production, sale, and delivery of a specified number of cell site transceivers at a specified price. Subsequent to the Department's initiation of this investigation, the contracting parties agreed to an increase in the contract price to cover a modification in Kokusai's performance terms under the contract at the request of AT&T Technologies. Because the price increase applied retroactively, Kokusai claims that this price increase should serve as the basis for a circumstance of sale adjustment in the amount of the price increase for units shipped prior to the formal contract amendment. Additionally, purchase price should be based on the amended contract price for shipments after the contract amendment.

*DOC Response*

In conducting antidumping investigations, we normally select as our period of investigation a historic period which predates the filing of the petition. In the present investigation the original sale of the subject merchandise occurred prior to our initiation of the investigation. We have selected this sale price as the proper subject of our investigation. It was only after initiation of our investigation that the contracting parties agreed on a price increase based on a modification of Kokusai's performance terms under the contract.

The Department is necessarily very cautious in adopting price increases which occur after receipt of a petition alleging sales at less than fair value. We have concluded for this investigation, that the proper basis of purchase price remains the original contract price, which predated filing of the petition.

*Comment 5*

The respondent claims that only the actual costs identified with developing the prototype required to meet AT&T specifications should be used, although the company relied upon prior knowledge for production technique and other expertise.

*DOC Response*

The Department used those actual costs which could be identified directly with the development of the transceiver.

*Comment 6*

The respondent claims that R&D costs should be treated as general expenses and no part of the costs should be considered processing costs.

*DOC Response*

The Department applied Kokusai's method, used in its ordinary course of business for: (1) Identifying specific types of R&D costs with the product, and (2) accounting for such R&D costs as part of the direct cost of manufacturing of the product. These were considered by Kokusai as part of the manufacturing costs of the product. Kokusai also recognized other R&D costs as being general to the corporation and including these costs in "selling, general and administrative costs." See response to petitioner's comment 6.

*Comment 7*

Respondent states that petitioner, E.F. Johnson, seeks to insert the present antidumping investigation into the context of overall telecommunications trade policy between the United States and Japan. Whatever the merits of the issues being raised in other fora about United States telecommunications policy, those issues are not part of the present antidumping proceeding and should not be allowed to color the Department's analysis in the present investigation.

*Response*

We agree with the respondent.

*Comment 8*

The respondent claims that since Kokusai's sale of the transceiver would be considered a "purchase price" transaction and there were no products sold in the home or third country markets which were considered to be "such or similar" merchandise, the constructed value should include the selling expenses incurred for the U.S. sale as a substitute for selling expenses incurred on home market sales.

*DOD Response*

The Department agrees with the respondent in this case and has used the selling expenses incurred in the U.S. market as a substitute for home market selling expenses of the product, because there were no home market or third country sales of the product. Additionally selling expenses for products considered to be of the same general class or kind could not be verified. Because the corporation sold many different products, average home market selling expense for the corporation, as a whole, were not considered to be representative of products of the same general class or kind since. The Radio Communication, Division's sales were clearly less than a majority of sales and the transceiver sales would be an insignificant part of this Division's sales.

*Comment 9*

Kokusai claims that its shipments were not massive when considered within the context of the rapidly expanding U.S. market.

*DOD Response*

At the time it was entered into, the Kokusai contract with AT&T represented a substantial portion of the U.S. market. In the interim between the filing of the petition and the preliminary determination, deliveries under the contract accelerated rapidly and significantly, such that the bulk of the contract was delivered prior to our preliminary determination. Because of the magnitude of the contract and accelerated delivery schedule, we have determined that massive imports were made over a relatively short period of time.

*Comment 10*

Kokusai claims that in its preliminary critical circumstance determination the Department focused on the wrong party in imputing knowledge of less than fair value sales.

*DOC Response*

In imputing knowledge of sales at less than fair value, the Department considered whether the importer of record, Kokusai Electric Company of America (Kokusai America), knew or should have knwon that sales were at less than fair value. Section 735(a)(3)(A)(ii) of the Act requires that the person by whom, or for whose account, the merchandise is imported knew, or should have known that the sale was at less than fair value. Kokusai America, as importer of record, clearly qualifies as the person by whom the

merchandise was imported, notwithstanding the fact that Kokusai America was not the purchaser or consumer of the goods. We note that Kokusai America was the importer of record for all deliveries under investigation.

**Verification**

In accordance with section 776(a) of the Act, we verified the information used in making this determination. We were granted access to the books and records of Kokusai and to its related importer in the U.S. We used standard verification procedures including examination of accounting records, financial records, and selected documents containing relevant information. In addition, we secured the services of a product expert who advised us on technical matters relating to production of the subject merchandise.

**Continuation of Suspension of Liquidation**

In accordance with section 733(d) and 733(e) of the Act, we are directing the United States Customs Service to continue to suspend liquidation of all entries of cell site transceivers from Japan which are subject to this investigation. This suspension of liquidation applies to unliquidated entries of merchandise entered, or withdrawn from warehouse, for consumption on or after March 7, 1984, which date is 90 days before the date of publication of our preliminary determination in the **Federal Register**. The U.S. Customs Service shall continue to require a cash deposit or the posting of a bond equal to the estimated amount of the weighted-average margin by which the foreign market value of the merchandise subject to this investigation exceeds the United States price. The suspension of liquidation will remain in effect until further notice. The weighted-average margins are as follows:

| *Manufacturers/Producers/Exporters* | *Weighted-Average Margins (%)* |
|---|---|
| Kokusai | 59.94% |
| All other manufacturers/producers/exporters | 59.94% |

**ITC Notification**

In accordance with section 735(d) of the Act, we are notifying the ITC of our determination. In addition, we are making available to the ITC all non-privileged and non-confidential information relating to this investigation. We will allow the ITC access to all privileged and confidential information in our files, provided the ITC confirms that it will not disclose such information, either publicly or under administrative protective order, without the written consent of the Deputy Assistant Secretary for import Administration. If the ITC determines that material injury does not exist, this proceeding will be terminated and all deposits or securities posted as a result of the suspension of liquidation will be refunded or cancelled. If, however, the ITC determines that such injury does exist, we will issue an antidumping order directing Customs officers to assess an antidumping duty on cell site transceivers from Japan entered, or withdrawn from warehouse, for consumption after March 7, 1984, equal to the amount by which the foreign market value of the subject merchandise exceeds the United States price. This determination is being published pursuant to section 735(d) of the Act [19 U.S.C. 1673d(d)].

Dated: October 19, 1984.

**Alan F. Holmer,**

*Acting Assistant Secretary for Trade Administration.*

[FR Doc. 84-28360 Filed 10-25-84; 8:45 am]

BILLING CODE 3510-DS-M

## Decision on Application for Duty-Free Entry of Scientific Instrument; Medical College of Wisconsin

This decision is made pursuant to section 6(c) of the Educational, Scientific, and Cultural Materials Importation Act of 1966 (Pub. L. 89-651, 80 Stat. 897; 15 CFR Part 301). Related records can be viewed between 8:30 AM and 5:00 PM in Room 1523, U.S. Department of Commerce, 14th and Constitution Avenue, NW., Washington, D.C.

Docket No.: 84-204. Applicant: Medical College of Wisconsin, Milwaukee, WI 53226. Instrument: Fluorescence Lifetime Instrument. Manufacturer: Photochemical Research Associates, Canada. Intended use: See notice at 49 FR 24911.

Comments: None received. Decision: Approved. No instrument of equivalent scientific value to the foreign instrument, for such purposes as it is intended to be used, is being manufactured in the United States. Reasons: The foreign instrument operates in the pulsed light mode and is capable of time-correlated single photon counting. The National Institutes of Health advises in its memorandum dated August 28, 1984 that (1) the capability of the foreign instrument described above is pertinent to the applicant's intended purpose and (2) it knows of no domestic instrument or apparatus of equivalent scientific value to the foreign instrument for the applicant's intended use.

We know of no other instrument or apparatus of equivalent scientific value to the foreign instrument which is being manufactured in the United States.

(Catalog of Federal Domestic Assistance Program No. 11.105, Importation of Duty-Free Educational and Scientific Materials)

**Frank W. Creel,**

*Acting Director, Statutory Import Programs Staff.*

[FR Doc. 84-28312 Filed 10-25-84; 8:45 am]

BILLING CODE 3510-DS-M

## Decision on Application for Duty-Free Entry of Scientific Instrument; North Carolina State University

This decision is made pursuant to section 6(c) of the Educational, Scientific, and Cultural Materials Importation Act of 1966 (Pub. L. 89-651, 80 Stat. 897; 15 CFR Part 301). Related records can be viewed between 8:30 AM and 5:00 PM in Room 1523, U.S. Department of Commerce, 14th and Constitution Avenue, NW., Washington, D.C.

Docket No.: 84-222. Applicant: North Carolina State University, Raleigh, NC 27695-8208. Instrument: Automatic Inlet Ports (12) & Test Kit for Mass Spectrometer. Manufacturer: Finnigan MAT, West Germany. Intended use: See notice at 49 FR 28288.

Comments: None received. Decision: Approved. No instrument of equivalent scientific value to the foreign instrument, for such purposes as it is intended to be used, is being manufactured in the United States. Reasons: These are compatible accessories for an instrument previously imported by the applicant. The instrument and accessories were made by the same manufacturer. National Bureau of Standards advises in its memorandum dated September 4, 1984 that the accessories are pertinent to the intended uses and that it knows of no comparable domestic accessories.

We know of no domestic accessories which can be readily adapted to the instrument.

(Catalog of Federal Domestic Assistance Program No. 11.105, Importation of Duty-Free Educational and Scientific Materials.)

**Frank W. Creel,**

*Acting Director, Statutory Import Programs Staff.*

[FR Doc. 84-28313 Filed 10-25-84; 8:45 am]

BILLING CODE 3510-DS-M

### Decision on Application for Duty-Free Entry of Scientific Instrument

This decision is made pursuant to section 6(c) of the Educational, Scientific, and Cultural Materials Importation Act of 1966 (Pub. L. 89–651, 80 Stat. 897; 15 CFR Part 301). Related records can be viewed between 8:30 AM and 5:00 PM in Room 1523, U.S. Department of Commerce, 14th and Constitution Avenue, NW., Washington, D.C.

Docket No. 84–190. Applicant: University of Colorado, Denver, CO 80262. Instrument: Used Mass Spectrometer with Data System, Model 7070 EHF. Manufacturer: VG Micromass, Ltd. United Kingdom. Intended use: See notice at 49 FR 20351.

Comments: None received. Decision: Approved. No instrument of equivalent scientific value to the foreign instrument, for such purposes as it is intended to be used, is being manufactured in the United States. Reasons: The foreign instrument provides a mass range of 1 to 3100 atomic mass units at 5000 volts and a scan rate of 0.1 seconds per decade. The National Institutes of Health advises in its memorandum dated July 26, 1984 that (1) the capability of the foreign instrument described above is pertinent to the applicant's intended purpose and (2) it knows of no domestic instrument or apparatus of equivalent scientific value to the foreign instrument for the applicant's intended use.

We know of no other instrument or apparatus of equivalent scientific value to the foreign instrument which is being manufactured in the United States.

(Catalog of Federal Domestic Assistance Program No. 11.105, Importation of Duty-Free Educational and Scientific Materials.)

**Frank W. Creel,**

*Acting Director, Statutory Import Programs Staff.*

[FR Doc. 84–28311 Filed 10–25–84; 8:45 am]

**BILLING CODE 3510–DS–M**

---

### Decision on Application for Duty-Free Entry of Scientific Instrument; University of Wisconsin-Parkside

This decision is made pursuant to section 6(c) of the Educational, Scientific, and Cultural Materials Importation Act of 1966 (Pub. L. 89–651, 80 Stat. 897; 15 CFR Part 301). Related records can be viewed between 8:30 AM and 5:00 PM in Room 1523, U.S. Department of Commerce, 14th and Constitution Avenue, NW., Washington, D.C.

Docket No.: 84–204. Applicant: University of Wisconsin-Parkside, Kenosha, WI 53141. Instrument: Counter Current Distribution Apparatus: Manufacturer: University of Sheffield, United Kingdom. Intended use: See notice at 49 FR 24911.

Comments: None received. Decision: Approved. No instrument of equivalent scientific value to the foreign instrument, for such purposes as it is intended to be used, is being manufactured in the United States. Reasons: The foreign instrument, an Albertson type thin layer counter-current apparatus for separating cell membranes, provides machined partitioning plates capable of 120 counter-current exchanges. The National Institutes of Health advises in its memorandum dated August 28, 1984 that (1) the capability of the foreign instrument described above is pertinent to the applicant's intended purpose and (2) it knows of no domestic instrument or apparatus of equivalent scientific value to the foreign instrument for the applicant's intended use.

We know of no other instrument or apparatus of equivalent scientific value to the foreign instrument which is being manufactured in the United States.

(Catalog of Federal Domestic Assistance Program No. 11.105, Importation of Duty-Free Educational and Scientific Materials)

**Frank W. Creel,**

*Acting Director, Statutory Import Programs Staff.*

[FR Doc. 84–28310 Filed 10–25–84; 8:45 am]

**BILLING CODE 3510–DS–M**

---

### Consolidated Decision on Applications for Duty-Free Entry of Electron Microscopes; Virginia Polytechnic Institute and State University, et al.

This is a decision consolidated pursuant to section 6(c) of the Educational, Scientific, and Cultural Materials Importation Act of 1966 (Pub. L. 89–651, 80 Stat. 897; 15 CFR Part 301). Related records can be viewed between 8:30 A.M. and 5:00 P.M. in Room 1523, U.S. Department of Commerce, 14th and Constitution Avenue, NW., Washington, D.C.

Docket No.: 84–227. Applicant: Virginia Polytechnic Institute and State University, Blacksburg, VA 24061. Instrument: Electron Microscope, Model JEM–100CX with SEGZ Side Entry Goniometer with Accessories. Manufacturer: JEOL, Ltd., Japan. Intended use: See notice at 49 FR 30985. Instrument ordered: May 17, 1984.

Docket No.: 84–228. Applicant: Columbia University, New York, NY 10027. Instrument: Electron Microscope, Model JEM–1200EX with SEG–10 Eucentric Side Entry Goniometer State and Accessories. Manufacturer: JEOL, Ltd., Japan. Intended use: See notice at 49 FR 30985. Instrument ordered: February 1, 1984.

Docket No.: 84–232. Applicant: Lebanon Valley College, Annville, PA 17003. Instrument: Electron Microscope, Model EM–109 with Accessories. Manufacturer: Carl Zeiss, West Germany. Intended use: See notice at 49 FR 30986. Application received by Commissioner of Customs: June 25, 1984.

Docket No.: 84–249. Applicant: Colorado State University, Fort Collins, CO 80523. Instrument: Electron Microscope, Model JEM–1200 EX with Accessories. Manufacturer: JEOL, Ltd., Japan. Intended use: See notice at 49 FR 32638. Instrument ordered: February 22, 1984.

Docket No.: 83–303. Applicant: University of Miami, Miami, FL 33101. Instrument: Electron Microscope, JEM–100CX and Accessories. Manufacturer: JEOL, Ltd., Japan. Intended use: See notice at 48 FR 40933. Instrument ordered: July 27, 1984.

Comments: None received. Decision: Approved. No instrument of equivalent scientific value to the foreign instrument, for such purposes as these instruments are intended to be used, was being manufactured in the United States at the time the instruments were ordered. Reasons: Each foreign instrument is a conventional transmission electron microscope (CTEM) and is intended for research or scientific educational uses requiring a CTEM. We know of no CTEM, or of any other instrument suited to these purposes, which was being manufactured in the United States either at the time of order of each instrument or at the time of receipt of application by the U.S. Customs Service.

(Catalog of Federal Domestic Assistance Program No. 11.105, Importation of Duty-Free Educational and Scientific Materials.)

**Frank W. Creel,**

*Acting Director, Statutory Import Programs Staff.*

[FR Doc. 84–28314 Filed 10–25–84; 8:45 am]

**BILLING CODE 3510–DS–M**

---

### Consolidated Decision on Applications for Duty-Free Entry of Electron Microscopes; Arizona State University, et al.

This decision is made pursuant to section 6(c) of the Educational, Scientific, and Cultural Materials Importation Act of 1966 (Pub. L. 89–651, 80 Stat. 897; 15 CFR Part 301). Related records can be viewed between 8:30 A.M. and 5:00 P.M. in Room 1523, U.S.

Department of Commerce, 14th and Constitution Avenue, NW., Washington, D.C.

Docket No.: 84–261. Applicant: Arizona State University, Tempe, AZ 85287. Instrument: Electron Microscope, Model JEM–4000EX with Accessories. Manufacturer: JEOL, Ltd., Japan. Intended use: See notice at 49 FR 35168. Instrument ordered: January 20, 1984.

Docket No.: 84–263. Applicant: Brown University, Providence, RI 02912. Instrument: Electron Microscope, Model EM 420 with Accessories. Manufacturer: N.V. Philips, The Netherlands. Intended use: See notice at 49 FR 35168. Instrument ordered: April 26, 1984.

Docket No.: 84–264. Applicant: University of Arizona, Tucson, AZ 85721. Instrument: Electron Microscope, Model EM 420T with Accessories. Manufacturer: N.V. Philips, The Netherlands. Intended use: See notice at 49 FR 35168. Instrument ordered: June 27, 1984.

Docket No.: 84–267. Applicant: Albert Einstein Medical Center, Philadelphia, PA 19141. Instrument: Electron Microscope, Model H–600–2 with Accessories. Manufacturer: Hitachi, Japan. Intended use: See notice at 49 FR 37136. Instrument ordered: January 19, 1984.

Docket No.: 84–268. Applicant: National Institute of Mental Health, Washington, DC 20232. Instrument: Electron Microscope, Model 10CA with Accessories. Manufacturer: Carl Zeiss, West Germany. Intended use: See notice at 49 FR 35397. Instrument ordered: June 8, 1984.

Docket No.: 84–269. Applicant: University of Maryland, Catonsville, MD 21228. Instrument: Electron Microscope, Model EM 10CA/C/CR with Accessories. Manufacturer: Carl Zeiss, West Germany. Intended use: See notice at 49 FR 37136. Instrument ordered: May 25, 1984.

Comments: None received. Decision: Approved. No instrument of equivalent scientific value to the foreign instrument, for such purposes as these instruments are intended to be used, was being manufactured in the United States at the time the instruments were ordered. Reasons: Each foreign instrument is a conventional transmission electron microscope (CTEM) and is intended for research or scientific educational uses requiring a CTEM. We know of no CTEM, or of any other instrument suited to these purposes, which was being manufactured in the United States either at the time of order of each instrument or at the time of receipt of application by the U.S. Customs Service.

(Catalog of Federal Domestic Assistance Program No. 11.105, Importation of Duty-Free Educational and Scientific Materials)

**Frank W. Creel,**

*Acting Director, Statutory Import Programs Staff.*

[FR Doc. 84–28315 Filed 10–25–84; 8:45 am]

BILLING CODE 3510–DS–M

## Applications for Duty-Free Entry of Scientific Instruments; University of Chicago

*Correction*

In FR Doc. 84–27619 beginning on page 41079 in the issue of Friday, October 19, 1984, make the following correction on page 41079: In the third column, in the seventh complete paragraph, in the last line, "June 27, 1984" should read "August 27, 1984".

BILLING CODE 1505–01–M

## COMMITTEE FOR PURCHASE FROM THE BLIND AND OTHER SEVERELY HANDICAPPED

## Proposed Additions to Procurement List

**SUMMARY:** The Committee has received proposals to add to Procurement List 1985 military resale commodities to be produced by and services to be provided by workshops for the blind and other severely handicapped.

Comments must be received on or before: November 28, 1984.

**ADDRESS:** Committee for Purchase from the Blind and Other Severely Handicapped, Crystal Square 5, Suite 1107, 1755 Jefferson Davis Highway, Arlington, Virginia 22202.

**FOR FURTHER INFORMATION CONTACT:** C.W. Fletcher, (703) 557–1145.

**SUPPLEMENTARY INFORMATION:** This notice is published pursuant to 41 U.S.C. 47(a)(2), 85 Stat. 77. Its purpose is to provide interested persons an opportunity to submit comments on the possible impact of the proposed actions.

*Additions*

If the Committee approves the proposed additions, all entities of the Federal Government will be required to procure the military resale commodities and services listed below from workshops for the blind or other severely handicapped.

It is proposed to add the following military resale commodities and services to Procurement List 1985, October 19, 1984 (49 F.R. 41195):

**Military Resale Item Nos. and Names**

No. 921 Mop, Anglematic
No. 931 Refill, for #921

*SIC 0782*

Grounds Maintenance, Ash Woods, French Dr. & Independence Avenue to 17th St. & Independence Avenue, Washington, D.C.

*SIC 5812*

Catering Service, Hot Meal, Military Entrance Processing Station, Jackson, Mississippi

*SIC 7349*

Janitorial/Custodial, Federal Building, 536 South Clark Street, Chicago, Illinois

*SIC 7369*

Commissary Shelf Stocking and Custodial Service, Reese Air Force Base, Texas

Commissary Shelf Stocking and Custodial Service, Francis E. Warren Air Force Base, Wyoming

**C.W. Fletcher,**

*Executive Director.*

[FR Doc. 84–28305 Filed 10–25–84; 8:45 am]

BILLING CODE 6820–33–M

## Procurement List 1984; Correction of Proposed Additions

In FR Doc. 84–27024 appearing on page 40077 in the issue of Friday, October 12, 1984, make the following correction:

In the third column under SIC 7349, the first item should read, Janitorial/Custodial, Federal Building and U.S. Courthouse, 312 North Spring Street, Los Angeles, California.

**C.W. Fletcher,**

*Executive Director.*

[FR Doc. 84–28306 Filed 10–25–84; 8:45 am]

BILLING CODE 6820–33–M

## Procurement List 1985; Additions

**AGENCY:** Committee for Purchase From the Blind and Other Severely Handicapped.

**ACTION:** Additions to procurement list.

**SUMMARY:** This action adds to Procurement list 1985 commodities to be produced by and services to be provided by workshops for the blind and other severely handicapped.

**EFFECTIVE DATE:** October 26, 1984.

**ADDRESS:** Committee for Purchase from the Blind and other Severely Handicapped, Crystal Square 5, Suite 1107, 1755 Jefferson Davis Highway, Arlington, Virginia 22202.

**FOR FURTHER INFORMATION CONTACT:** C.W. Fletcher, (703) 557–1145.

**SUPPLEMENTARY INFORMATION:** On June 8 and July 9, 1984, the Committee for Purchase from the Blind and Other Severely Handicapped published notices (49 FR 23907 and 49 F.R. 27969) of proposed additions to Procurement List 1985, October 19. 1984. (49 FR 41195).

After consideration of the relevant matter presented, the Committee has determined that the commodities and services listed below are suitable for procurement by the Federal Government under 41 U.S.C. 46–48c, 85 Stat. 77.

I certify that the following actions will not have a significant impact on a substantial number of small entities. The major factors considered were:

a. The actions will not result in any additional reporting, recordkeeping or other compliance requirements.

b. The actions will not have a serious economic impact on any contractors for the commodities and services listed.

c. The actions will result in authorizing small entities to produce or provide the commodities and services procured by Government.

Accordingly, the following commodities and services are hereby added to Procurement List 1985:

*Class 7530*

Tape, Postage Meter
7530–00–912–3925
7530–00–912–3924

*Class 7920*

Towel, Paper
7920–00–832–9772
(For GSA Regions 1,3 (excluding New Cumberland,
Pennsylvania depot), 5 and the Navy Pack for Charleston South Carolina Depot only)

*Sic 7349*

SIC 7349
Janitorial/Custodial U.S. Courthouse, 68 Court Street,
Buffalo, New York.
Janitorial/Custodial U.S. Courthouse West 920 Riverside Avenue Spokane, Washington

**C.W. Fletcher,**

*Executive Director.*

[FR Doc 84–28304 Filed 10–25–84; 8:45 am]

**BILLING CODE 6820–33–M**

## DEPARTMENT OF DEFENSE

### Office of the Secretary

### Defense Intelligence Agency Scientific Advisory Committee; Closed Meeting

**SUMMARY:** Pursuant to the provisions of Subsection (d) of Section 10 of Pub. L. 92–463, as amended by Section 5 of Pub. L. 94–409, notice is hereby given that a closed meeting of a panel of the DIA Scientific Advisory Committee has been scheduled as follows:

**DATE:** December 4, 1984, 1:00 p.m. to 5:00 p.m.

**ADDRESS:** INCA Program Office, McLean, VA.

**FOR FURTHER INFORMATION CONTACT:** Major Harold E. Linton, USAF, Executive Secretary, DIA Scientific Advisory Committee, Washington, D.C. 20301 (202/373–4930).

**SUPPLEMENTARY INFORMATION:** The entire meeting is devoted to the discussion of classified information as defined in Section 552b(c)(1), Title 5 of the U.S. Code and therefore will be closed to the public. Subject mattter will be used in a study on Intelligence Communications Architecture.

Dated: October 22, 1984.

**Patricia H. Means,**

*OSD Federal Register Liaison Officer, Department of Defense.*

[FR Doc. 84–28270 Filed 10–25–84; 8:45 am]

**BILLING CODE 3810–01–M**

### DoD Advisory Group on Electron Devices; Advisory Committee Meeting

**SUMMARY:** Working Group C (Mainly Opto-Electronics) of the DOD Advisory Group on Electron Devices (AGED) will meet in closed door session on 29–30 October 1984 at Naval Ocean Systems Center, 271 Catalina Blvd., San Diego, California 92152.

The mission of the Advisory Group is to provide the Under Secretary of Defense for Research and Engineering, the Director, Defense Advanced Research Projects Agency and the Military departments with technical advice on the conduct of economical and effective research and development programs in the area of electron devices.

The Working Group C meeting will be limited to review and development programs which the military propose to initiate with industry, universities or in their laboratories. This opto-electronic devices area includes such programs as imaging devices, infrared detectors and lasers. The review will include classified program details throughout.

In accordance with Section 10(d) of Pub. L. No. 92–463, as amended (5 U.S.C. App. I § 10(d) (1976)), it has been determined that this Advisory Group Meeting concerns matters listed in 5 U.S.C. 552b(c)(1) (1976), and that accordingly, this meeting will be closed to the public.

Dated: October 22, 1984.

**Patricia H. Means,**

*OSD Federal Register Liaison Officer.*

[FR Doc. 84–28272 Filed 10–25–84; 8:45 am]

**BILLING CODE 3810–01–M**

### DoD Advisory Group on Electron Devices; Advisory Committee Meeting

**SUMMARY:** Working Group A (Mainly Microwave Devices) of the DOD Advisory Group on Electron Devices (AGED) will meet in closed session on November 7, 1984 at Palisades Institute for Research Services, Inc., 1215 Jefferson Davis Highway, Crystal Gateway #3, Suite 1203, Arlington, Virginia 22202.

The mission of the Advisory Group is to provide the Under Secretary of Defense for Research and Engineering, the Director, Defense Advanced Research Projects Agency and the Military Departments with technical advice on the Conduct of economical and effective research and development programs in the area of electron devices.

The Working Group A meeting will be limited to review of research and development programs which the military proposed to initiate with industry, universities or in their laboratories. This microwave device area includes programs on developments and research related to microwave tubes, solid state microwave, electronic warfare devices, millimeter wave devices, and passive devices. The review will include classified program details throughout.

In accordance with Section 10(d) of Pub. L. No. 92–463, as amended, (5 U.S.C. App. 1 § 10(d)(1976)), it has been determined that this Advisory Group meeting matters listed in 5 U.S.C. 552b(c)(1) (1976), and that accordingly, this meeting will be closed to the public.

Dated: October 22, 1984.

**Patricia H. Means,**

*OSD Federal Register Liaison Officer, Department of Defense.*

[FR Doc. 84–28275 Filed 10–25–84; 8:45 am]

**BILLING CODE 3810–01–M**

### Establishment of a Federally Funded Research and Development Center (FFRDC)

**ACTION:** Notice of proposed designation of FFRDC.

**SUMMARY:** The Department of Defense, in compliance with the procedures of OFPP Policy Letter No. 84–1, "Federally Funded Research and Development Centers" (April 4, 1984), announces its intention to designate the Logistics

Management Institute an FFRDC to perform research, studies, and analyses in the area of logistics and weapon systems acquisition. Such work includes research and analyses to: (1) Reduce costs and increase the effectiveness of military procurement, materiel management, logistics and manpower support activities and other related areas; (2) formulate and recommend changes in DOD policy relating to acquisitions and support of weapons systems and other defense resources requirements; (3) develop mathematical models and other management tools for the evaluation of logistics and manpower plans and materiel requirements, and (4) appraise the readiness of the Armed Forces.

Dated: October 22, 1984.

**Patricia H. Means,**

*OSD Federal Register Liaison Officer, Department of Defense.*

[FR Doc. 84-28269 Filed 10-25-84; 8:45 am]

BILLING CODE 3810-01-M

## Public Information Collection Requirement Submitted to OMB for Review

**SUMMARY:** The Department of Defense has submitted to OMB for review the following proposal for the collection of information under the provisions of the Paperwork Reduction Act (44 U.S.C. Chapter 35). Each entry contains the following information: (1) Type of Submission; (2) Title of Information Collection and Form Number if applicable; (3) Abstract statement of the need for and the uses to be made of the information collected; (4) Type of Respondent; (5) An estimate of the number of responses; (6) An estimate of the total number of hours needed to provide the information; (7) To whom comments regarding the information collection are to be forwarded; and (8) The point of contact from whom a copy of the information proposal may be obtained.

New

Medical Information Questionnaire: DIS FL 14 and 14a.

The Defense Investigative Service (DIS) is responsible for conducting personnel security investigations to determine an individual's suitability for a position of trust. This form is used to gather medical information when there is an indication of a history of mental or nervous disorder, use or abuse of prescribed or illegal drugs, or abuse or excessive use of alcohol.

Medical Professionals

Responses: 14,000

Burden hours: 7,000

**ADDRESSES:** Comments are to be forwarded to Mr. Edward Springer, Office of Management and Budget, Desk Officer, Room 3235, New Executive Office Building, Washington, DC 20503, and Mr. Daniel J. Vitiello, DOD Clearance Officer, WHS/DIOR, Room 1C535, The Pentagon, Washington, DC 20301-1155, telephone (202) 694-0187.

**SUPPLEMENTARY INFORMATION:** A copy of the information collection proposal may be obtained from Mr. Fred A. Schonert, Defense Investigative Service, Administrative Services Division, V0240, 1900 Half Street, SW, Washington, DC 20324-1700, telephone (202) 693-0881.

Dated: October 22, 1984.

**Patricia H. Means,**

*OSD Federal Register Liaison Officer, Department of Defense.*

[FR Doc. 84-28274 Filed 10-25-84; 8:45 am]

BILLING CODE 3810-01-M

## Department of the Army

## Public Information Collection Requirement Submitted To OMB for Review

**SUMMARY:** The Department of Defense has submitted to OMB for review the following proposal for the collection of information under the provisions of the Paperwork Reduction Act (44 U.S.C. Chapter 35). Each entry contains the following information: (1) Type of submission; (2) Title of Information Collection and Form Number if applicable; (3) Abstract statement of the need for and the uses to be made of the information collected; (4) Type of Respondent; (5) An estimate of the number of responses; (6) An estimate of the total number of hours needed to provide the information; (7) To whom comments regarding the information collection are to be forwarded; and (8) The point of contact from whom a copy of the information proposal may be obtained.

Revision

Uniform Cost Accounting and Reporting System (UCARS) MIL-STD 1260; DA Forms 4812-R, 4813-R and 4814-R.

The Military Standard (UCARS) was needed to bring uniformity to the accounting systems at the Government Owned Contractor Operated (GOCO) Ammunition plants. UCARS provides uniform cost accounting data that can be used to evaluate individual contractor performance, and to make comparisons among the plants of cost and manpower information.

Businesses

Responses 132

Burden hours 2,640

**ADDRESSES:** Comments are to be forwarded to Mr. Edward Springer, Office of Management and Budget, Desk Officer, Room 3235, New Executive Office Building, Washington, DC 20503 and Mr. Daniel J. Vitiello, DOD Clearance Officer, WHS/DIOR, Room 1C535, The Pentagon, Washington, DC 20301-1155, telephone (202) 694-0187.

**SUPPLEMENTARY INFORMATION:** A copy of the information collection proposal may be obtained from Mr. David O. Cochran, DAIM-ADI, Room 1D667, The Pentagon, Washington, DC 20301, telephone (202) 695-5111.

Dated: October 22, 1984.

**Patricia H. Means,**

*OSD Federal Register Liaison Officer, Department of Defense.*

[FR Doc. 84-26273 Filed 10-25-84; 8:45 am]

BILLING CODE 3810-01-M

## Department of the Navy

## Privacy Act of 1974; Deleted System of Records

**AGENCY:** Department of the Navy, DOD.

**ACTION:** Notice of deletion of systems of records.

**SUMMARY:** The Department of the Navy proposes to delete one system of records in its inventory of systems of records subject to the Privacy Act of 1974.

**DATE:** The proposed action will be effective without further notice November 26, 1984, unless comments are received which would result in a contrary determination.

**ADDRESS:** Send any comments to the systems manager identified in the system notice.

**FOR FURTHER INFORMATION CONTACT:** Mrs. Gwendolyn R. Aitken, Privacy Act Coordinator, Office of the Chief of Naval Operations (Op-09B30), Department of the Navy, The Pentagon, Washington, DC 20350. Telephone: (202) 697-1459.

**SUPPLEMENTARY INFORMATION:** The Department of the Navy systems notices for records systems subject to the Privacy Act of 1974 (5 U.S.C. 552a) Pub. L. 93-579 were published in the **Federal Register** as follows:

FR Doc. 83-109 (48 FR 26029) June 6, 1983
FR Doc. 84-2616 (49 FR 3901) January 31, 1983
FR Doc. 83-2828 (49 FR 4124) February 2, 1983
FR Doc. 83-4908 (49 FR 6967) February 24, 1983
FR Doc. 84-8893 (49 FR 13350) April 4, 1984
FR Doc. 84-8901 (49 FR 13399) April 4, 1984
FR Doc. 84-10509 (49 FR 15601) April 19, 1984

FR Doc. 84–10681 (49 FR 16777) April 20, 1984
FR Doc. 84–14818 (49 FR 23107) June 4, 1984
FR Doc. 84–16521 (49 FR 25507) June 21, 1984
FR Doc. 84–23495 (49 FR 35172) September 6, 1984
FR Doc. 84–25459 (49 FR 37825) September 26, 1984

The proposed deletion is not within the purview of the provision of 5 U.S.C. 552a(0) which requires the submission of altered systems reports.

**Patricia H. Means,**

*OSD Federal Register Liaison Officer, Department of Defense.*

October 22, 1984.

**DELETION**

**N05100–9**

*System name:*

Personnel Data Base Application/ Student Instructor Performance Module (48 FR 26058) June 6, 1983.

*Reason:*

This system has been discontinued.

[FR Doc. 84–28271 Filed 10–25–84; 8:45 am]

BILLING CODE 3810–01–M

## Corps of Engineers, Department of the Army

## Intent To Prepare a Draft Environmental Impact Statement (DESI); Department of the Army (DA) Regulatory Permit Action for the Proposed Ewa Marina Community, Ewa District, Oahu, HI

**AGENCY:** U.S. Army Corps of Engineers, Honolulu District, Pacific Ocean Division.

**ACTION:** Notice of intent to prepare a DEIS for a regulatory permit action.

**SUMMARY:**

*1. Background of Previous Actions*

A Notice of Intent to prepare a DEIS was previously prepared by the U.S. Army Corps of Engineers and published in the Federal Register, Vol. 45, No. 96 on May 15, 1980. This was followed by the distribution of the DEIS in May and June, 1980. Based on the initial reactions and comments from the reviewing agencies, the applicant, MSM and Associates, Inc., requested the withdrawal of the DEIS pending incorporation of additional consultant efforts in addressing the agency concerns.

*2. Description of the Proposed Action*

The applicant proposes to construct a recreational marina as part of a 556-acre permanent residential-recreational development consisting of 3,500 dwelling units. The proposed marina will consist of 98 acres of internal waterways with 1,600 boat slips. The waterway system will be created inland of the existing shoreline with a primary channel located at the present Kaloi Gulch, on the west end of Oneula Beach Park. Two jetties up to 700 feet long will be constructed along the entrance channel to protect the marina from waves penetrating the basin and to prevent littoral drift from shoaling the channel. The proposed marina requires a DA permit under Section 10 of the River and Harbor Act of 1899 (33 U.S.C. 403) and Section 404 of the Clean Water Act (33 U.S.C. 1344).

*3. Description of Reasonable Alternatives*

Because the project is in its preliminary planning stages, details on reasonable alternatives have not been finalized. The applicant is in the process of determining various alternatives, which may include:

a. Full scale development as proposed.

b. Reduced development.

c. Alternative design plans, without the marina.

d. No development alternatives, such as maintaining the existing land use, or enhancing agricultural, open space, or park uses.

*4. Description of the Scoping Process for the DEIS*

a. The public and affected Federal, State and local agencies, and other interested private organizations and parties are invited to provide comments identifying specific concerns which should be addressed in the DEIS. Upon preparation of the DEIS, a public notice shall be issued summarizing the facts of the case and announcing the availability of the DEIS. If a public hearing is requested, it will be held after completion of the DEIS. A public notice announcing the time, date, location and nature of the hearing would be issued at least 30 days prior to the hearing date.

b. The applicant's DEIS may also satisfy the State of Hawaii's requirement for an Environmental Impact Statement pursuant to Chapter 343, Hawaii Revised Statutes, and the State Environmental Quality Commission's *Envrionmental Impact Statement Regulations.*

c. The significant issues to be analyzed in depth in the DEIS will include:

(1) Impacts of the marina on the coastal zone and shoreline and on wetlands.

(2) Susceptibility of the marina project location in the tsunami hazard zone.

(3) Impacts of the marina on flora and fauna and on rare or endangered species.

(4) Impacts of the marina on surface water runoff and drainage.

(5) Alterations of coastal water quality and oceanographic conditions caused by the marina.

(6) Effects of the marina on air quality and noise.

(7) Effects of Navy and commercial aircraft noise on the proposed residential community.

(8) Aesthetic considerations and socioeconomic impacts, including impacts on public facilities such as transportation and utilities related to the marina development.

(9) Impacts of the marina on historic, archaeological and paleontological resources.

(10) Land use considerations and impacts, including the loss of agricultural lands.

(11) Recreational impacts related to the marina development.

5. It is estimated that the DEIS will be made available to the public in November 1984.

**ADDRESS:** Questions about the proposed action and DEIS can be answered by: Mr. Stanley T. Arakaki, Chief, Operations Branch, Construction-Operations Division, U.S. Army Corps of Engineers, Fort Shafter, Hawaii 96858–5440, Telephone: (808) 438–9258.

Dated: October 18, 1984

**Michael M. Jenks,**

*Colonel, Corps of Engineers, District Engineer.*

[FR Doc. 84–28303 Filed 10–25–84; 8:45 am]

BILLING CODE 3710–NN–M

## DEPARTMENT OF EDUCATION

## Office of Special Education and Rehabilitative Service

## Handicapped Special Studies Program

**AGENCY:** Department of Education.

**ACTION:** Notice of proposed annual evaluation priorities.

**SUMMARY:** The Secretary proposes annual evaluation priorities for the Handicapped Special Studies program. Studies have been selected to ensure effective use of program funds and to meet study requirements included in the Education of the Handicapped Act.

**DATE:** Comment must be received on or before January 24, 1985.

**ADDRESS:** Comments should be addressed to: Nancy Safer, Research

Projects Branch, Division of Educational Services, Office of Special Education Programs, Department of Education, 400 Maryland Avenue SW. (Switzer Building, Room 3513), Washington, D.C. 20202.

**FOR FURTHER INFORMATION CONTACT:** Nancy Safer. Telephone: (202) 732–1084.

**SUPPLEMENTARY INFORMATION:** The Handicapped Special Studies program, authorized by section 618 of Part B of the Education of the Handicapped Act as amended, supports studies to evaluate the impact of the Act including States' efforts towards the provision of a free appropriate public education to handicapped children (20 U.S.C. 1401, 1411 et seq.). Section 618 of the Act requires that the results of these studies be included in the annual report submitted to the Congress by the Department.

Under section 618(c) of the Act, as amended by the Education of the Handicapped Act Amendments of 1983, Pub. L. 98–199, the Secretary is expressly required to submit to the appropriate committees of each House of the Congress and publish in the Federal Register for review and comment proposed annual priorities for evaluations conducted under section 618. A State and local financing study mandated under section 618(e)(2), as added by the Education of the Handicapped Act Amendments of 1983, was initiated in fiscal year 1984 and will be continued in fiscal year 1985.

Priorities

(a) *Educational Progress of Handicapped Students.* This proposed priority would support a longitudinal study of a sample of handicapped students encompassing the full range of handicapping conditions, to examine their educational progress while in special education and their occupational, educational, and independent living status after graduating from secondary school or otherwise leaving special education. This study is specifically required by section 618(e)(1), as added by the Education of the Handicapped Act Amendments of 1983.

(b) *Programming Features of Special Purpose Facilities.* Projects. This proposed priority would support a project to examine the educational programming and other services available to handicapped students in separate schools or facilities, or public or private residential facilities to clarify the criteria used in selecting those placement options, and to profile the characteristics of the handicapped students in those placements.

(c) *State Educational Agency/Federal Evaluation Studies Projects.* This proposed priority would support evaluation studies to assess the impact and effectivenes of programs assisted under the Education of the Handicapped Act. Within this priority, studies will be invited that address: (1) The impact and effectiveness of criteria used to detemine eligibility and placement of students in various program options; (2) the effectiveness of instructional programming options and screening procedures used prior to referral for placement of children in special education; and (3) the impact of secondary program options provided handicapped students in relation to competency testing, graduation, and transition. In accordance with section 618(d) of the Act, as added by the Education of the Handicapped Act Amendments of 1983, the Secretary proposes to enter into cooperative agreements with State educational agencies to carry out these studies.

(d) *Identification and Clarification of Emerging Issues.* This proposed priority would support activities to obtain and analyze imformation on emerging issues related to the Act. This information is essential for both immediate program administrative needs and for inclusion in the annual report to Congress.

(e) *Evaluation Assistance.* This proposed priority would support a project to assist States in designing, conducting, and reporting evaluations of the impact and effectiveness of the implementation of the Act in their States. The project will also perform an analysis of these findings which will be included in the annual report to Congress. This priority is required under section 618(d)(3) of the Act, as added by Educaton of the Handicapped Amendments of 1983, when State Education Agency/Federal Evaluation Studies Projects are funded.

Intergovernmental Review

This program is subject to the requirements of Executive Order 12372 and the regulations in 34 CFR Part 79 (48 FR 29158; June 24, 1983). The objective of the Executive Order is to foster an intergovernmental partnership and a strengthened federalism by relying on State and local processes for State and local government coordination and review of proposed Federal financial assistance.

In accordance with the Order, this document provides early notification of the Department's plans and actions for this program.

Executive Order 12291

These proposed priorities have been reviewed in accordance with Executive Order 12291. They are not classified as major regulations because they do not meet the criteria for major regulations established in the Order.

Invitation to Comment

Interested persons are invited to submit comments and recommendations regarding the proposed evaluation priorities.

All comments submitted in response to these proposed priorities will be available for public inspection, during and after the comment period, in Room 3517, Switzer Building, 330 C Street, SW., Washington, D.C. between the hours of 8:30 a.m. and 4:00 p.m., Monday through Friday of each week except Federal holidays.

(20 U.S.C. 1418)

(Catalog of Federal Domestic Assistance Number 84.159; Handicapped Special Studies)

Dated: October 22, 1984.

**T.H. Bell,**

*Secretary of Education.*

[FR Doc. 84-28330 Filed 10-25-84; 8:45 am]
BILLING CODE 4000-01-M

## DEPARTMENT OF ENERGY

### Economic Regulatory Administration

**[ERA Docket No. 84-14-NG]**

### Northwest Alaskan Pipeline Co.; Application To Amend Authorization To Import Natural Gas From Canada

**AGENCY:** Department of Energy, Economic Regulatory Administration.

**ACTION:** Notice of Application to Amend and Extend Authorization to Import Natural Gas from Canada through the Eastern Leg of the Alaska Natural Gas Transportation System.

**SUMMARY:** The Economic Regulatory Administration (ERA) gives notice of receipt on October 16, 1984, of an application of Northwest Alaskan Pipeline Company (Northwest Alaskan). In light of a renegotiated contract with its Canadian natural gas supplier, Pan-Alberta Gas Ltd. (Pan-Alberta), Northwest Alaskan requests removal of conditions imposed on its current authorization to import on an average annual daily basis up to 800,000 Mcf per day of Canadian natural gas to the United States for transportation through the Eastern Leg of the Alaska Natural Gas Transportation System (ANGTS). Northwest Alaskan also requests an extension of its authorization to October 31, 2002. The natural gas imported by Northwest Alaskan is received at the international boundary near Monchy, Saskatchewan, resold to Northern Natural Gas Company, Division of InterNorth, Inc. (Northern), Panhandle

Eastern Pipe Line Company (Panhandle), and United Gas Pipeline Company (United), and transported through the Eastern Leg of the ANGTS. Although Northwest Alaskan and Pan-Alberta negotiated a single Gas Sales Contract, separate and distinct pricing and take provisions have been negotiated for Northwest Alaskan's three repurchasers of gas. Northwest Alaskan requests that the ERA act on its application by December 15, 1984, and that the contract amendments be made effective on November 1, 1984.

The application is filed with the ERA pursuant to Section 3 of the Natural Gas Act (NGA), Section 9 of the Alaska Natural Gas Transportation Act of 1976 (ANGTA), and DOE Delegation Order No. 0204–111. Protests, motions to intervene, notices of interventions, and written comments are invited.

**DATE:** Protests, motions to intervene or notices of intervention, as applicable, and written comments are to be filed no later than 4:30 p.m., on November 26, 1984.

**FOR FURTHER INFORMATION CONTACT:**

John Glynn, Natural Gas Division, Office of Fuels Programs, Economic Regulatory Administration, Forrestal Building, Room GA–007, 1000 Independence Avenue, S.W., Washington, D.C. 20585, (202) 252–9482

Diane Stubbs, Office of the General Counsel, Natural Gas and Mineral Leasing, U.S. Department of Energy, Forrestal Building, Room 6E–042, 1000 Independence Avenue, S.W., Washington, D.C. 20585, (202) 252–6667

**SUPPLEMENTARY INFORMATION:**

Northwest Alaskan purchases Canadian natural gas from Pan-Alberta pursuant to a Gas Sales Contract dated March 9, 1978, as amended (Eastern Contract). The gas is resold to Northern, Panhandle, and United under three separate Gas Purchase Agreements and is transported through the prebuilt section of the Eastern Leg of the ANGTS for resale and delivery to midwestern and eastern markets. Deliveries of gas to Northwest Alaskan began on September 1, 1982.

### Current Export/Import Authorizations

Pan-Alberta has licenses from the Canadian National Energy Board (NEB) to export natural gas at Monchy, Saskatchewan, to Northwest Alaskan through October 31, 1992. On July 23, 1984, Pan-Alberta filed an application with the NEB to extend its export authority through October 31, 1996. However, the total volume of gas to be exported daily during the proposed extended term would remain unchanged.

In 1980 the Federal Energy Regulatory Commission (FERC), pursuant to Section 3 of the NGA and Section 9 of the ANGTA, issued two orders authorizing Northwest Alaskan to import 800,000 Mcf per day through October 31, 1988. On December 15, 1983, the FERC extended the import authorization through October 31, 1992, to be consistent with the export license granted to Pan-Alberta by the NEB. However, the FERC conditioned its extension by requiring Northwest Alaskan to (1) renegotiate its Eastern Contract with Pan-Alberta and its Purchase Agreements with Northern, Panhandle, and United to provide for market-responsive prices and volume obligations; (2) submit contract amendments and necessary tariff changes; and (3) obtain regulatory approval from the ERA, FERC, and NEB.

Northwest Alaskan states in its application that the first two conditions imposed by the FERC in its December 15 order have been met by its instant filing and that regulatory approval by the ERA, FERC, and NEB will satisfy the third condition.

### Jurisdiction

On February 15, 1984, the Secretary of Energy (Secretary), in Delegation Order No. 0204–110 (49 FR 6690, February 22, 1984), rescinded Delegation Order No. 0204–8 (42 FR 61441, December 5, 1977), which delegated to the FERC sole authority under Section 3 of the NGA to approve the importation of Canadian natural gas related to the construction and operation of the ANGTS. At the same time the Secretary, in Delegation Order 0204–111 (49 FR 6690, February 22, 1984), delegated the authority to the Administrator of the ERA to regulate the importation and exportation of natural gas under Section 3 of the NGA, including imports through the ANGTS.

### Application

Northwest Alaskan states in its application that it has renegotiated with Pan-Alberta and the U.S. purchasers three amendments to the Eastern Contract and corresponding amendments to each of the Northern, Panhandle, and United Gas Purchase Agreements which together provide for market-responsive pricing and volume obligations. Although similar in effect, the amendments were negotiated separately and contain distinct pricing and take provisions for Northern, Panhandle, and United. In addition, a fourth amendment to the Eastern Contract allocates the maximum daily quantity of 800,000 Mcf among the three U.S. purchasers according to their volume entitlements under the Gas Purchase Agreements.

#### *Northern*

Specifically, the amendment directed to Northern establishes a minimum daily volume obligation requiring Northern to take-*and*-pay for 20 percent of Northern's maximum daily quantity in the seven months of April through October and 40 percent in the five winter months of November through March. For the 1984–85 contract year, the minimum annual take-*and*-pay obligation is 50 percent, and for the 1985–86 contract year, it is 60 percent. In addition, there is a 60 percent take-*or*-pay obligation with a $0.32 (U.S.) per Mcf settlement to be paid for any deficiency below the take-or-pay level.

The amendment also provides for a demand-commodity rate structure for the volumes sold to Northern. The demand component, which is estimated to be $2.8 million monthly, will consist of (1) the administrative costs incurred by Pan-Alberta in connection with securing and arranging for the transportation of the gas; (2) the cost of transporting Northern's volumes through Zones 6 and 9 of the ANGTS prebuilt facilities of Foothills Pipeline (Yukon) Ltd. (Foothills); (3) the cost of gathering and transporting Northern's volumes through the facilities of NOVA, an Alberta Corporation (NOVA); and (4) the administrative costs incurred by Northwest Alaskan for purchase and resale of Canadian gas at the U.S.-Canadian border. The demand charge will be redetermined every six months on January 16 and July 1, provided that all Canadian incurred costs have been reviewed and found acceptable by the NEB. If actual costs differ from those used to compute the demand charge, any overcharges or undercharges would be determined and applied to the next six months at the time of the semi-annual redetermination. The Foothills and NOVA charges would be renegotiated if they are substantially increased for reasons including cost allocation, major expansion, or rate design.

The applicant states that the commodity component will provide for a price at the international border which will enable the gas to be competitive in Northern's market area. For the 1984–85 contract year, the commodity rate will be $2.40 (U.S.) per MMBtu for volumes up to 85 percent of Northern's maximum daily quantity times the number of days in the year. For the 1985–86 contract year, the commodity rate will be $2.45 (U.S.) for such volumes. An incentive

rate of $2.30 (U.S.) for volumes purchased each year above 85 percent will apply for both years. Commencing November 1, 1986, the commodity charge and the minimum volume obligations applicable during each contract year will be redetermined through renegotiation or failing agreement, arbitration.

*Panhandle*

The minimum daily volume obligation requires Panhandle to take-*and*-pay for 30 percent of Panhandle's maximum daily quantity. For the 1984–85 contract year, there is a minimum annual take obligation of approximately 37 percent, and a minimum annual take-*or*-pay of 50 percent, with a $0.32 (U.S.) per Mcf settlement to be paid for any deficiency below the take-or-pay level.

The amendment includes a demand-commodity rate structure. The demand component is estimated to be $2.1 million monthly. The provisions governing the demand component are the same as for Northern.

The commodity component for the 1984–85 contract year will be $2.14 (U.S.) per MMBtu for volumes purchased up to Panhandle's annual take obligation (37 percent), $2.20 (U.S.) per MMBtu for any additional volumes purchased up to the take-*or*-pay level of 50 percent, and for all volumes over 50 percent, the price will be set quarterly by Pan-Alberta after consultation with Northwest Alaskan and Panhandle concerning the anticipated condition of Panhandle's market. Commencing November 1, 1985, the commodity charge and the minimum volume obligations applicable during each contract year will be redetermined by negotiation, or failing agreement, arbitration.

*United*

The United agreement establishes different price and take provisions, with three tiers. The minimum daily volume obligation (Tier I) requires United to take-*and*-pay for 33⅓ percent of the maximum daily quantity. There is also a minimum annual volume obligation of 33⅓ percent. There is no take-*or*-pay obligation.

For Tier I volumes, the initial price will equal the Alberta Border Price ($2.31 U.S. effective August 1, 1984), plus (1) Foothills' transportation charges for the volumes, and (2) United's share of the cost of fuel and line pack on Foothill's system. This price is a minimum floor designed to cover the cost of Canadian prebuilt facilities and return a price at the Alberta border consistent with the price established by the Alberta Petroleum Marketing Commission. This price is subject to renegotiation each May 1 and November 1, if either party requests it. If no agreement is reached, the matter will be resolved by arbitration.

Additional volumes (Tier II) up to United's maximum daily quantity which, when added to the Tier I volumes, make the weighted average price of United's purchases under this contract equal the weighted average price of United's domestic gas purchases, may be purchased at a price equal to the Alberta Border Price plus the cost of fuel required to transport the gas through the Foothills system.

United will have an obligation to purchase any remaining volumes beyond those needed to equal United's weighted average cost of domestic gas (Tier III volumes) if the price offered by Pan-Alberta is less than that for any of the domestic gas supplies purchased by United. If the price for such Tier III volumes is equal to that of any of United's domestic supplies, United must purchase the Tier III volumes on a proratable basis with such equally priced domestic gas. Each month Pan-Alberta will determine the price it wishes to offer for Tier III volumes based on prevailing conditions in United's market.

In addition to the above, United will also pay Northwest Alaskan each month for the administrative costs it incurs in the purchase and resale of United's volumes at the U.S.-Canadian border.

Northwest Alaskan states in its application that the proposed contract amendments are tailored to each of the three U.S. purchasers' markets in order to make Canadian natural gas continually market-sensitive throughout the term of the contracts. Under the proposed contract amendments and based upon the minimum taken-and-pay volume obligations of each of the three U.S. purchasers, Northwest Alaskan estimates that the average unit cost of gas purchased under the Eastern Contract for the 1985 contract year would be $3.35 (U.S.) per MMBtu at the border. Northwest Alaskan maintains that the per unit cost to its three U.S. purchasers would approximate $4.00 (U.S.) per MMBtu if the proposed contract amendments were not approved. If each of the three U.S. purchasers took 100 percent of their contracted volumes under the amendment for the 1985 contract year, Northwest Alaskan estimates the unit cost of the gas would be about $2.71 (U.S.) per MMBtu for United and Panhandle, and about $2.92 (U.S.) per MMBtu for Northern. Northwest Alaskan further maintains that approval of these proposed amendments would result in a savings of $617 million (U.S.).

Northwest Alaskan maintains that the proposed contract amendments will allow Canadian gas to compete in U.S. markets, while still providing an enhanced economic benefit to Canada. As a result, Northwest Alaskan requests that the ERA find its amendments in compliance with the ERA's new policy guidelines issued by the Secretary of Energy on February 17, 1984 (49 FR 6684, February 22, 1984), remove the conditions imposed by the FERC in the December 15, 1983 order, and unconditionally extend its import authority for a term to coincide with the terms of its Eastern Contract and Gas Purchase Agreements with its three U.S. purchasers which extend through October 31, 2002. Alternatively, Northwest Alaskan requests an extension through October 31, 1996, consistent with the export authorization sought by Pan-Alberta before the NEB.

Furthermore, Northwest Alaskan contends that approval of this application is in the public interest. It asserts that the amendments ensure that the prices are responsive to conditions in the indiviudal U.S. markets, and that they will remain market-sensitive throughout the life of the contracts. Northwest Alaskan claims the gas will be needed as the present surplus of gas dissipates by the late 1980s, and that the extension will not cause undue reliance on Canadian imports. Northwest Alaskan also claims that Canadian imports represent a more secure and dependable energy supply than OPEC oil. Northwest Alaskan also asserts that since its purchasers and their customers have borne the early initial costs of transportation of Canadian gas through the Eastern Leg of the prebuilt ANGTS system, they should also receive the benefits that will accrue to them from the proposed extension—an additional secure and dependable supply of Canadian gas through the ANGTS system with the attendant lower transportation charges resulting from declining depreciation and related expenses.

Finally, Northwest Alaskan stresses the importance of the import authorization extension by asserting that it will provide Northern Border Pipeline Company, the transporter of the Eastern Contract volumes, an opportunity to lower its cost of service on a long-term basis and thus make the transportation of these volumes more economical.

The decision on this application will be made consistent with the Secretary of Energy's gas import policy guidelines, under which the competitiveness of an import arrangement in the markets

served is the primary consideration in determining whether it is in the public interest. The applicant has asserted that this import arrangement is competitive. Parties that may oppose this application should address their comments to the issue of competitiveness as set forth in the policy guidelines. parties opposing the arrangement bear the burden of overcoming this assertion.

**Other Information**

In response to this notice, any person may file a protest, motion to intervene or notice of intervention, as applicable, and written comments. Any person wishing to become a party to the proceeding and to have the written comments considered as the basis for any decision on the application must, however, file a motion to intervene or notice of intervention, as applicable. The filing of a protest with respect to this application will not serve to make the protestant a party to the proceeding, although protests and comments received by persons who are not parties will be considered in determining the appropriate procedural action to be taken on the application. All protests, motions to intervene, notices of intervention, and written comments must meet the requirements that are specified by the regulations in 10 CFR Part 590. They should be filed with the Natural Gas Division, Office of Fuels Programs, Economic Regulatory Administration, Room GA-033-B, RG-43, Forrestal Building, 1000 Independence Avenue, S.W., Washington, D.C. 20585. They must be filed no later than 4:30 p.m., November 26, 1984.

A decisional record on the application will be developed through responses to this notice by parties, including the parties' written comments and replies thereto. Additional procedures will be used as necessary to achieve a complete understanding of the facts and issues. A party seeking intervention may request that additional procedures be provided, such as additional written comments, an oral presentation, a conference, or a trial-type hearing. Any request to file additional written comments should explain why they are necessary. Any request for an oral presentation should identify the substantial question of fact, law, or policy at issue, show that it is material and relevent to a decision in the proceeding, and demonstrate why an oral presentation is needed. Any request for a conference should demonstrate why the conference would materially advance the proceeding. Any request for a trial-type hearing must show that there are factual issues genuinely in dispute that are relevant and material to a decision and that a trial-type hearing is necessary for a full and true disclosure of the facts.

If an additional procedure is scheduled, the ERA will provide notice to all parties. If no party requests additional procedures, a final opinion and order may be issued based on the official record, including the application and responses filed by parties pursuant to this notice, in accordance with 10 CFR § 590.316.

A copy of Northwest Alaskan's application is available for inspection and copying in the Natural Gas Division Docket Room, Room GA-033-B, at the above address. The docket room is open between the hours of 8:00 a.m. and 4:30 p.m., Monday through Friday, except Federal holidays.

Issued in Washington, D.C., on October 23, 1984.

**James W. Workman,**

*Director, Office of Fuels Programs Economic Regulatory Administration.*

[FR Doc. 84-28428 Filed 10-25-84; 8:45 am]

BILLING CODE 6450-01-M

---

**[ERA Docket No. 84-15-NG]**

## Northwest Alaskan Pipeline Co.; Application To Amend Authorization To Import Natural Gas From Canada

**AGENCY:** Department of Energy, Economic Regulatory Administration.

**ACTION:** Notice of Application to Amend and Extend Authorization to Import Natural Gas from Canada through the Western Leg of the Alaska Natural Gas Transportation System.

**SUMMARY:** The Economic Regulatory Administration (ERA) gives notice of receipt on October 16, 1984, of an application by Northwest Alaskan Pipeline Company (Northwest Alaskan). In light of a renegotiated contract with its Canadian natural gas supplier, Pan-Alberta Gas Ltd. (Pan-Alberta), Northwest Alaskan requests removal of conditions imposed on its current authorization to import on an average annual daily basis up to 240,000 Mcf per day of Canadian natural gas to the United States for transportation through the Western Leg of the Alaska Natural Gas Transportation System (ANGTS). Northwest Alaskan also requests an extension of its authorization to October 31, 2001. The natural gas imported by Northwest Alaskan is received at the international boundary near Kingsgate, British Columbia, where it is sold to Pacific Interstate Transmission Company (PIT) for further transmission over the Western Leg of the ANGTS and then over other pipelines for sale to Southern California Gas Company (SoCal). Northwest Alaskan and Pan-Alberta also renegotiated a demand-commodity rate structure, and a reduction in daily and annual take-and-pay provisions to 60 percent of contract quantity. The revised contract also provides for revision of the components of the rate structure every six months. Northwest Alaskan requests the ERA act on its application by December 15, 1984, and that the contract amendment be made effective on November 1, 1984.

The application was filed with the ERA pursuant to Section 3 of the Natural Gas Act (NGA), Section 9 of the Alaska Natural Gas Transportation Act (ANGTA), and DOE Delegation Order No. 0204-111. Protests, motions to intervene, notices of interventions, and written comments are invited.

**DATE:** Protests, motions to intervene or notices of intervention, as applicable, and written comments are to be filed no later than 4:30 p.m., on November 26, 1984.

**FOR FURTHER INFORMATION CONTACT:**

Chuck Boehl, Natural Gas Division, Office of Fuels Programs, Economic Regulatory Administration, Forrestal Building, Room GA-007, 1000 Independence Avenue, S.W., Washington, D.C. 20585, (202) 252-6050

Diane Stubbs, Office of General Counsel, Natural Gas and Mineral Leasing, U.S. Department of Energy, Forrestal Building, Room 6E-042, 1000 Independence Avenue, S.W., Washington, D.C. 20585, (202) 252-6667

**SUPPLEMENTARY INFORMATION:** Northwest Alaskan purchases Canadian natural gas from Pan-Alberta pursuant to a sales contract dated March 9, 19178, as amended (Western Contract). The gas is resold to PIT under a Purchase Agreement and is transported through the prebuilt section of the Western Leg of the ANGTS for resale to SoCal and resale and delivery to Southern California markets. Deliveries at Kingsgate began on October 1, 1981.

**Current Export/Import Authorizations**

Pan-Alberta has licenses from the Canadian National Energy Board (NEB) to export natural gas at Kingsgate, British Columbia, to Northwest Alaska through October 31, 1992. On July 23, 1984, Pan-Alberta filed an application with the NEB to extend its import authority through October 31, 1996. However, the total volume of gas to be exported daily during the proposed extended term would remain unchanged.

In orders issued January 11, 1980, and June 13, 1980, pursuant to Section 3 of the NGA and Section 9 of the ANGTA, the Federal Energy Regulatory Commission (FERC) authorized Northwest Alaskan to import natural gas from Pan-Alberta at Kingsgate, British Columbia, through October 31, 1988. On December 15, 1983, the FERC extended the import authorization through October 31, 1992, to correspond to an export authorization to Pan-Alberta by the NEB. However, the FERC conditioned is extension by requiring Northwest Alaskan to (1) renegotiate its Western Contract with Pan-Alberta and its Purchase Agreement with PIT to provide for market-responsive prices and volume obligations, (2) submit contract amendments and necessary tariff changes, and (3) obtain regulatory approval from the ERA, the FERC, and the NEB.

Northwest Alaskan states in its application that the first two conditions have been met by its instant filing and that regulatory approval by the ERA, FERC, and the NEB will satisfy the third condition.

### Jurisdiction

On February 15, 1984, the Secretary of Energy (Secretary), in Delegation Order No. 0204-110 (49 F.R. 6690, February 22, 1984), rescinded Delegation Order No. 0204-8 (42 F.R. 61441, December 5, 1977) which delegated to the FERC sole authority under Section 3 of the NGA to approve the importation of Canadian natural gas related to the construction and operation of the ANGTS. At the same time the Secretary, in Delegation Order 0204-111 (49 F.R. 6690, February 22, 1984), delegated the authority to the Administrator of the ERA to regulate the importation and exportation of natural gas under Section 3 of the NGA, including imports through the ANGTS.

### Application

Northwest Alaska states in its application that it has negotiated an amendment to its Western Contract with Pan-Alberta to respond to the needs of PIT's market, thus assuring PIT a secure, market-responsive long-term supply of natural gas. The amendment provides for a rate structure comprised of a demand and a commodity component. The demand component consists of a combination of (1) administrative costs incurred by Pan-Alberta in connection with securing the gas and arranging transportation and sale of the gas from the Province of Alberta; (2) the cost of transporting the volumes resold to PIT through Zones 7 and 8 of the ANGTS prebuilt facilities of Foothills Pipe Lines (Yukon) Ltd. (Foothills); (3) the cost of gathering and transporting PIT's volumes through the facilities of NOVA, AN ALBERTA CORPORATION (NOVA); and (4) the administrative costs incurred by Northwest Alaskan for purchase and resale of the gas at the U.S.-Canadian border. The demand charge is currently projected to be $4 million a month. The contract specifies that approval of these provisions, including the time extension, by all parties would prevent the immediate increase in transportation charges related to accelerated depreciation of the Foothills pipeline.

The demand charge would be redetermined every six months on January 1 and July 1, provided that all Canadian incurred costs have been reviewed and found acceptable by the NEB. If actual costs differ from those used to compute the demand charge, any overcharges or undercharges would be determined and applied to the next six months at the time of the six-month redetermination. PIT would have the right to audit the charges. The Foothills and NOVA charges would be subject to renegotiation if they are substantially increased for reasons including cost allocation, major expansion or rate design.

The commodity charge, also subject to recalculation every six months, would be a price at the U.S.-Canadian border based on a formula which takes into consideration changes in the recent cost of all other gas supplies purchased by SoCal or its affiliates for resale in the Southern California gas market. Pan-Alberta would have the right to verify this calculation. This would be, in effect, the price the Alberta producers receive for the gas, to which transportation charges to the border and then to the PIT's system would be added as the demand charge.

The commodity charge will initially be established at $2.40 (U.S.) per MMBtu. The amendment also establishes an incentive price of $2.30 (U.S.) per MMBtu for volumes purchased per year in excess of 85 percent of the contract volumes. If purchases exceed 100 percent of contract volume, the price reverts to the basic commodity charge. The incentive rate will be renegotiated at the same time the base commodity rate is redetermined.

The amendment further provides for a reduction in the minimum daily and annual volume purchase obligations from 85 percent of contract volume to a 60 percent take-*and*-pay requirement. There is no take-*or*-pay requirement.

Northwest Alaskan has asked the ERA to remove the conditions imposed by the FERC on the four-year authorization extension to 1992. In addition, the applicant requests the ERA to extend the authorization to October 31, 2001, to correspond to the term of its purchase contract with Pan-Alberta. Alternatively, Northwest Alaskan requests the ERA to extend the authorization through October 31, 1996, to correspond to the export authorization currently being sought by Pan-Alberta from the NEB.

Northwest Alaskan states that the amendment establishes market-responsive, flexible pricing terms for importing Canadian gas, and that the amendment is consistent with the new policy guidelines issued by the Secretary of Energy on February 17, 1984 (49 FR 6684, February 22, 1984). The applicant contends that use of a demand-commodity rate structure will enable Canadian gas to compete with alternate fuels in the Southern California area. Because the commodity price will be the price the Alberta producers receive for the gas, price signals in the marketplace will be directly relayed back to the producers. The applicant claims that since the commodity price will increase or decrease in relationship to other gas supplies delivered into the Southern California market, the Canadian gas will continue to be responsive to the market throughout the life of the contract.

Northwest Alaskan states that the amendment to its contract with Pan-Alberta and the corresponding change to its Purchase Agreement with PIT will result in significant cost reductions. Under the amendments, using the proposed demand-commodity rate and the minimum take requirement of 60 percent, Northwest Alaskan estimates that the total cost of gas purchased at the border during the contract year ending October 31, 1985, would be $173 million—a savings of $124 million compared with the estimated $297 million cost under the existing contract with a higher minimum take requirement at the present border price. On a unit basis, Northwest Alaskan calculates the present border price of $4.40 (U.S.) coupled with the Volume Related Incentive Price (VRIP) of $3.40 (U.S.), to result in a price at the border for Northwest Alaskan of $4.00 per MMBtu. Under the amendments, the unit cost at the border would be $3.29 (U.S.) per MMBtu at the minimum 60 percent take and pay level, would decrease to $3.00 (U.S.) per MMBtu at an 85 percent take level, and would decrease further to $2.92 (U.S.) per MMBtu if the full 1985 contract volume were taken.

The applicant asserts that the Canadian gas proposed to be imported under this amendment will provide the

Southern California market with a secure and reliable long-term supply of gas. The applicant contends that supply deficiencies are anticipated in PIT's market areas in the late 1980's, and that PIT will need Canadian imports in the late 1980's and 1990's, although the proposed import extension will not cause undue reliance on Canadian imports. Northwest Alaskan also claims that Canadian imports represent a more secure and dependable energy supply than OPEC oil. Finally, Northwest Alaskan claims that since PIT and its customers have borne the early initial costs of transportation of Canadian gas through the Western Leg of the prebuilt ANGTS system, they should also receive the benefits that will accrue to them from the proposed extension—an additional secure and dependable supply of Canadian gas through the ANGTS system with the attendant lower transportation charges resulting from declining depreciation and related expenses.

The decision on this application will be made consistant with the Secretary of Energy's gas import policy guidelines, under which the competitiveness of an import arrangement in the markets served is the primary consideration in determining whether it is in the public interest. The applicant has asserted that this import arrangement is competitive. Parties that may oppose this application should address their comments to the issue of competitiveness as set forth in the policy guidelines. Parties opposing the arrangement bear the burden of overcoming this assertion.

**Other Information**

In response to this notice, any person may file a protest, motion to intervene, or notice of intervention, as applicable, and written comments. Any person wishing to become a party to the proceeding and to have the written comments considered as the basis for any decision on the application must, however, file a motion to intervene or notice of intervention, as applicable. The filing of a protest with respect to this application will not serve to make the protestant a party to the proceeding, although protests and comments received by persons who are not parties will be considered in determining the appropriate procedual action to be taken on the application. All protests, motions to intervene, notices of intervention, and written comments must meet the requirements that are specified by the regulations in 10 CFR Part 590. They should be filed with the Natural Gas Division, Office of Fuels Program, Economic Regulatory Administration, Room GA–033–B, Forrestal Building, 1000 Independence Avenue, S.W., Washington, D.C. 20585. They must be filed no later than 4:30 p.m., November 26, 1984.

A decisional record on the application will be developed through response to this notice by parties, including the parties' written comments and replies thereto. Additional procedures will be used as necessary to achieve a complete understanding of the facts and issues. A party seeking intervention may request that additional procedures be provided, such as additional written comments, an oral presentation, a conference, or a trial-type hearing. Any request to file additional written comments should explain why they are necessary. Any request for an oral presentation should identify the substantial question of fact, law, or policy at issue, show that it is material and relevant to a decision in the proceeding, and demonstrate why an oral presentation is needed. Any request for a conference should demonstrate why the conference would materially advance the proceeding. Any request for a trial-type hearing must show that there are factual issues genuinely in dispute that are relevant and material to a decision and that a trial-type hearing is necessary for a full and true disclosure of the facts.

If an additional procedure is scheduled, the ERA will provide notice to all parties. If no party requests additional procedures, a final opinion and order may be issued based on the official record, including the application and responses filed by parties pursuant to this notice, in accordance with 10 CFR § 590.316.

A copy of Northwest Alaskan's application is available for inspection and copying in the Natural Gas Division Docket Room, Room GA–033–B, at the above address. The docket room is open between the hours of 8:00 a.m. and 4:30 p.m., Monday through Friday, except Federal holidays.

Issued in Washington, D.C., on October 23, 1984.

**James W. Workman,**

*Director, Office of Fuels Program, Economic Regulatory Administration.*

[FR Doc. 84–28427 Filed 10–25–84; 8:45 am]

BILLING CODE 6450–01–M

## Federal Energy Regulatory Commission

**[Docket No. CP85–5–000]**

## Arkansas Louisiana Gas Co., a Division of Arkla, Inc.; Request Under Blanket Authorization

October 23, 1984.

Take notice that on October 3, 1984, Arkansas Louisiana Gas Company, a division of Arkla, Inc. (Arkla), P.O. Box 21734, Shreveport, Louisiana 71151, filed in Docket No. CP85–5–000 a request pursuant to § 157.205 of the Commission's Regulations under the Natural Gas Act (18 CFR 157.205) for authorization to construct and operate sales taps on two jurisdictional lines to permit direct retail sales of gas to end-users under the certificate issued in Docket Nos. CP82–384–000 and CP82–384–000 pursuant to section 7 of the Natural Gas Act, all as more fully set forth in the request on file with the Commission and open to public inspection.

Arkla proposes to construct and operate one sales tap in Pope County, Arkansas, to enable Arkla to serve the River Oaks Subdivision near Russellville, Arkansas, and one sales tap in Conway County, Arkansas, to enable Arkla to serve various residential and small commercial customers in a small rural unincorporated community known as Jerusalem, Arkansas. Arkla estimates total peak day deliveries of 619 Mcf of gas and total annual deliveries of 29,368 Mcf.

Arkla states that these would all be retail sales of gas for use and consumption in the ordinary course of its retail gas business in the area, which is subject to the jurisdiction of the Arkansas Public Service Commission. The gas would be delivered from Arkla's general system supply, which, it is stated, is adequate to provide the service.

Any person or the Commission's staff may, within 45 days after issuance of the instant notice by the Commission, file pursuant to Rule 214 of the Commission's Procedural Rules (18 CFR 385.214) a motion to intervene or notice of intervention and pursuant to § 157.205 of the Regulations under the Natural Gas Act (18 CFR 157.205) a protest to the request. If no protest is filed within the time allowed therefor, the proposed activity shall be deemed to be authorized effective the day after the

time allowed for filing a protest. If a protest is filed and not withdrawn within 30 days after the time allowed for filing a protest, the instant request shall be treated as an application for authorization pursuant to section 7 of the Natural Gas Act.

Kenneth F. Plumb,

*Secretary.*

[FR Doc. 84-28361 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

[Docket Nos. ER83-297-003 and ER81-577-012]

## Arkansas Power & Light Co.; Refund Compliance Report

October 23, 1984.

Take notice that on September 4, 1984, Arkansas Power and Light Company (AP&L) submitted for filing its refund compliance report pursuant to the Commission's order issued July 20, 1984.

AP&L states that this report shows monthly billing determinants; revenue receipt dates; revenues under the prior, present and compliance rates; the monthly revenue refund; computation of interest and a summary of such information for each refund period in the above dockets.

Any person desiring to be heard or to protest this filing should file comments with the Federal Energy Regulatory Commission, 825 North Capitol Street, NE., Washington, DC 20426, on or before November 1, 1984. Comments will be considered by the Commission in determining the appropriate action to be taken. Copies of this filing are on file with the Commission and are available for public inspection.

Kenneth F. Plumb,

*Secretary.*

[FR Doc. 84-28362 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

[Docket No. CP84-735-000]

## Columbia Gas Transmission Corp.; Request Under Blanket Authorization

October 23, 1984.

Take notice that on September 25, 1984, Columbia Gas Transmission Corporation (Columbia), 1700 MacCorkle Avenue, SE., Charleston, West Virginia 25314, filed in Docket No. CP84-735-000 a request pursuant to § 157.205 of the Commission's Regulations under the Natural Gas Act (18 CFR 157.205) for authorization to transport natural gas on behalf of Union Camp Corporation (Union) under the certificate issued in Docket No. CP83-76-000 pursuant to section 7 of the Natural Gas Act, all as more fully set forth in the request which is on file with the Commission and open to public inspection.

Columbia proposes to transport up to 290 million Btu equivalent of natural gas per day for Union through June 30, 1985. Columbia states that the gas to be transported would be purchased from Liberty Oil & Gas Corp. (Liberty) and would be used as boiler fuel in Union's Eighty-Four, Pennsylvania, plant.

It is indicated that Columbia has released certain gas supplies of Liberty and that these supplies are subject to the ceiling price provisions of sections 103, 107, and 108 of the Natural Gas Policy Act of 1978. It is further indicated that Union has made arrangements to purchase this released gas from Liberty. Columbia states that it would receive the gas from Liberty and redeliver the gas to Columbia Gas of Pennsylvania, Inc. (CPA), the distribution company serving Union, near Eighty-Four, Pennsylvania. Further, Columbia states that depending upon whether its gathering facilities are involved, it would charge either (1) 40.11 cents per dt equivalent for storage and transmission, exclusive of company-use and unaccounted-for gas, as set forth in Columbia's Rate Schedule TS-1. Columbia states that it would retain 2.85 percent of the total quantity of gas delivered into its system for company-use and unaccounted-for gas, as set forth in Columbia's Rate Schedule TS-1.

Any person or the Commission's staff may, within 45 days after issuance of the instant notice by the Commission, file pursuant to Rule 214 of the Commission's Procedural Rules (18 CFR 385.214) a motion to intervene or notice of intervention and pursuant to § 157.205 of the regulations under the Natural Gas Act (18 CFR 157.205) a protest to the request. If no protest is filed within the time allowed therefor, the proposed activity shall be deemed to be authorized effective the day after the time allowed for filing a protest. If a protest is filed and not withdrawn within 30 days after the time allowed for filing a protest, the instant request shall be treated as an application for authorization pursuant to section 7 of the Natural Gas Act.

Kenneth F. Plumb,

*Secretary.*

[FR Doc. 84-28363 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

[Docket No. CP84-741-000]

## Columbia Gas Transmission Corp.; Request Under Blanket Authorization

October 23, 1984.

Take notice that on September 26, 1984, Columbia Gas Transmission Corporation (Columbia), 1700 MacCorkle Avenue, SE, Charleston, West Virginia 25314, filed in Docket No. CP84-741-000 a request pursuant to § 157.205 of the Commission's Regulations under the Natural Gas Act (18 CFR 157.205) for authorization to transport natural gas on behalf of Tru Fit Products Corporation (Tru Fit) under the certificate issued in Docket No. CP83-76-000 pursuant to section 7 of the Natural Gas Act, all as more fully set forth in the request which is on file with the Commission and open to public inspection.

Columbia proposes to transport up to 136 million Btu equivalent of natural gas per day for Tru Fit through June 30, 1985. Columbia states that the gas to be transported for Tru Fit is Tru Fit's own production and would be used as boiler fuel in Tru Fit's Medina, Ohio, plant.

Columbia states that it would receive the gas from Tru Fit and deliver the gas to Columbia Gas of Ohio, Inc. (COH), the distribution company serving Tru Fit, near Medina, Ohio. Further, Columbia states that depending upon whether its gathering facilities are involved, it would charge either (1) 40.11 cents per dt equivalent for storage and transmission, exclusive of company-use and unaccounted-for gas, as set forth in Columbia's Rate Schedule TS-1. Columbia states that it would retain 2.85 percent of the total quantity of gas delivered into its system for company-use and unaccounted-for gas, as set forth in Columbia's Rate Schedule TS-1.

Any person or the Commission's staff may, within 45 days after issuance of the instant notice by the Commission, file pursuant to Rule 214 of the Commission's Procedural Rules (18 CFR 385.214) a motion to intervene or notice of intervention and pursuant to § 157.205 of the regulations under the Natural Gas Act (18 CFR 157.205) a protest to the request. If no protest is filed within the time allowed therefor, the proposed activity shall be deemed to be authorized effective the day after the time allowed for filing a protest. If a protest is filed and not withdrawn within 30 days after the time allowed for filing a protest, the instant request shall be treated as an application for authorization pursuant to section 7 of the Natural Gas Act.

Kenneth F. Plumb,

*Secretary.*

[FR Doc. 84-28364 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

[Docket No. CP84-743-000]

## Columbia Gas Transmission Corp.; Request Under Blanket Authorization

October 23, 1984.

Take notice that on September 26, 1984, Columbia Gas Transmission Corporation (Columbia), 1700 MacCorkle Avenue, SE., Charleston, West Virginia 25314, filed in Docket No. CP84-743-000 a request pursuant to § 157.205 of the Commission's Regulations under the Natural Gas Act (18 CFR 157.205) authorization to transport natural gas on behalf of W.R. Grace & Co., Davison Chemical Division (W.R. Grace), under the certificate issued in Docket No. CP83-76-000 pursuant to section 7 of the Natural Gas Act, all as more fully set forth in the request which is on file with the Commission and open to public inspection.

Columbia proposes to transport up to 2 billion Btu equivalent of natural gas per day for W.R. Grace through June 30, 1985. Columbia states that the gas to be transported would be purchased from Ohio Gas Marketing, Inc. (OGM), and Oxford Oil Company (Oxford) and would be used as boiler fuel and process gas in W.R. Grace's Cincinnati, Ohio plant.

It is indicated that Columbia has released certain gas supplies of Oxford and that these supplies are subject to the ceiling price provisions of sections 103, 107 and 108 of the Natural Gas Policy Act of 1978. It is further indicated that W.R. Grace has made arrangements to purchase the released gas from Oxford and other gas from OGM. Columbia states that it would receive the gas from Oxford and OGM and redeliver the gas to Cincinnati Gas & Electric Company, the distribution company service W.R. Grace, near Cincinnati, Ohio. Further, Columbia states that depending upon whether its gathering facilities are involved, it would charge either (1) 40.11 cents per dt equivalent for storage and transmission, exclusive of company-use and unaccounted-for gas, or (2) 44.93 cents per dt equivalent for storage, transmission and gathering, exclusive of company-use and unaccounted-for gas, as set forth in Columbia's Rate Schedule TS-1. Columbia states that it would retain 2.85 percent of the total quantity of gas delivered into its system for company-use and unaccounted-for gas, as set forth in Columbia's Rate Schedule TS-1.

Any person or the Commission's staff may, within 45 days after issuance of the instant notice by the Commission, file pursuant to Rule 214 of the Commission's Procedural Rules (18 CFR 385.214) a motion to intervene or notice of intervention and pursuant to § 157.205 of the regulations under the Natural Gas Act (18 CFR 157.205) a protest to the request. If no protest is filed within the time allowed therefor, the proposed activity shall be deemed to be authorized effective the day after the time allowed for filing a protest. If a protest is filed and not withdrawn within 30 days after the time allowed for filing a protest, the instant request shall be treated as an application for authorization pursuant to section 7 of the Natural Gas Act.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84-28365 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

[Docket No. CP84-724-000]

## National Fuel Gas Supply Corp.; Application

October 23, 1984.

Take notice that on September 19, 1984, National Fuel Gas Supply Corporation (Applicant), Ten Lafayette Square, Buffalo, New York 14203, filed in Docket No. CP84-724-000 an application pursuant to section 7(c) of the Natural Gas Act for a certificate of public convenience and necessity authorizing Applicant to sell natural gas for resale to Elizabethtown Gas Company (Elizabethtown), all as more fully set forth in the application which is on file with the Commission and open to public inspection.

Applicant proposes to sell Elizabethtown a maximum daily quantity of 10,000 dt equivalent of gas and a maximum annual quantity of 3,000,000 dt equivalent of gas. It is explained that this service would be provided on a firm basis and for a ten-year period of time. Applicant states that it would sell these volumes under Rate Schedule RQ, in Applicant's FERC Gas Tariff, plus applicable adjustments and surcharges.

It is explained that Elizabethtown has experienced a substantial increase in the temperature sensitive, firm, high priority requirements on its system since 1978. Applicant states that this proposal is designed to meet that winter heating demand by permitting Elizabethtown to inject gas into storage during the summer and also to aid Elizabethtown in providing winter service.

Any person desiring to be heard or to make any protest with reference to said application should on or before November 9, 1984, file with the Federal Energy Regulatory Commission, Washington, D.C. 20426, a motion to intervene or a protest in accordance with the requirements of the Commission's Rules of Practice and Procedure (18 CFR 385.214 or 385.211) and the Regulations under the Natural Gas Act (18 CFR 157.10). All protests filed with the Commission will be considered by it in determining the appropriate action to be taken but will not serve to make the protestants parties to the proceeding. Any person wishing to become a party to a proceeding or to participate as a party in any hearing therein must file a motion to intervene in accordance with the Commission's Rules.

Take further notice that, pursuant to the authority contained in and subject to jurisdiction conferred upon the Federal Energy Regulatory Commission by sections 7 and 15 of the Natural Gas Act and the Commission's Rules of Practice and Procedure, a hearing will be held without further notice before the Commission or its designee on this application if no motion to intervene is filed within the time required herein, if the Commission on its own review of the matter finds that a grant of the certificate is required by the public convenience and necessity. If a motion for leave to intervene is timely filed, or if the Commission on its own motion believes that a formal hearing is required, further notice of such hearing will be duly given.

Under the procedure herein provided for, unless otherwise advised, it will be unnecessary for Applicant to appear or be represented at the hearing.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84-28366 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

[Docket No. ER85-37-000]

## Northern Indiana Public Service Co.; Filing

October 23, 1984.

The filing Company submits the following:

Take notice that on October 10, 1984, Northern Indiana Public Service Company (NIPSCO) tendered for filing Second Revised Volume No. 3 to its FERC Electric Service Tariff-Fourth Revised Volume No. 1 which has been revised to include an additional delivery point for the Town of Bremen #2 69 Kv). NIPSCO also tendered for filing the following:

Exhibit B, Sheet B-2, a supplement to the Service Agreement between NIPSCO and the Town of Bremen, which covers the supply of

electric energy for resale at the delivery point located on the south side of second Street in the Industrial Addition to the Town of Bremen.

NIPSCO requests an effective date of August 1, 1984, and therefore requests waiver of the Commission's notice requirements.

Copies of this filing were served upon all customers receiving electric service under NIPSCO's FERC Electric Service Tariff-Fourth Revised Volume No. 1 and the Public Service Commission of Indiana.

Any person desiring to be heard or to protest said filing shoud file a motion to intervene or protest with the Federal Energy Regulatory Commission, 825 North Capitol Street NE., Washington, D.C. 20426, in accordance with Rules 211 and 214 of the Commission's Rules of Practice and Procedure (18 CFR 385.211, 385.214). All such motions or protests should be filed on or before November 2, 1984. Protests will be considered by the Commission in determining the appropriate action to be taken, but will not serve to make protestants parties to the proceeding. Any person wishing to become a party must file a motion to intervene. Copies of this filing are on file with the Commission and are available for public inspection.

Kenneth F. Plumb,

*Secretary.*

[FR Doc. 84-28367 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

[Docket No. CP84-729-000]

## Northern Natural Gas Co., Division of InterNorth, Inc.; Application

October 23, 1984.

Take notice that on September 21, 1984, Northern Natural Gas Company, Division of InterNorth, Inc. (Northern), 2223 Dodge Street, Omaha, Nebraska 68102, filed in Docket No. CP84-729-000 an application pursuant to section 7(c) of the Natural Gas Act for a certificate of public convenience and necessity authorizing the construction and operation of facilities which would allow Northern to deliver natural gas to a new gas distribution company, Boundary Natural Gas Company (BNG), and authorizing the sale of natural gas to BNG for resale, all as more fully set forth in the application which is on file with the Commission and open to public inspection.

Northern proposes to construct and operate approximately 61 miles of branchline from a point of interconnection with Northern Border Pipeline System at valve no. 41 in Deuel County, South Dakota, to a delivery point with BNG near Ortonville, Big Stone County, Minnesota. Northern states that nine town border stations would be established along the proposed branchline as delivery points to BNG. Northern estimates the total cost of the facilities at $5,016,213 which cost would be financed out of cash on hand.

Northern also requests authorization to sell natural gas to BNG under Northern's Rate Schedules CD-1 and SS-1. Northern estimates that its sales to BNG would grow from 558,924 Mcf of gas during the first year of service, with 2,122 Mcf in sales on a peak day, to 1,192,865 Mcf during the fifth year of service, with peak day sales at 4,855 Mcf. According to Northern, BNG would use these volumes to serve residential and industrial customers in South Dakota and Minnesota.

Any person desiring to be heard or to make any protest with reference to said application should on or before November 9, 1984, file with the Federal Energy Regulatory Commission, Washington, D.C. 20426, a motion to intervene or a protest in accordance with the requirements of the Commission's Rules of Practice and Procedure (18 CFR 385.214 or 385.211) and the Regulations under the Natural Gas Act (18 CFR 157.10). All protests filed with the Commission will be considered by it in determining the appropriate action to be taken but will not serve to make the protestants parties to the proceeding. Any person wishing to become a party to a proceeding or to participate as a party in any hearing therein must file a motion to intervene in accordance with the Commission's Rules.

Take further notice that, pursuant to the authority contained in and subject to jurisdiction conferred upon the Federal Energy Regulatory Commission by sections 7 and 15 of the Natural Gas Act and the Commission's Rules of Practice and Procedure, a hearing will be held without further notice before the Commission or its designee on this application if no motion to intervene is filed within the time required herein, if the Commission on its own review of the matter finds that a grant of the certificate is required by the public convenience and necessity. If a motion for leave to intervene is timely filed, or if the Commission on its own motion believes that a formal hearing is required, further notice of such hearing will be duly given.

Under the procedure herein provided for, unless otherwise advised, it will be unnecessary for Northern to appear or be represented at the hearing.

Kenneth F. Plumb,

*Secretary.*

[FR Doc. 84-28368 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

[Docket No. CP84-738-000]

## Northern Natural Gas Co., Division of InterNorth, Inc.; Application

October 23, 1984.

Take notice that on September 25, 1984, Northern Natural Gas Company, Division of InterNorth, Inc. (Northern), 2223 Dodge Street, Omaha, Nebraska 68102, filed in Docket No. CP84-738-000 an application pursuant to section 7(b) of the Natural Gas Act for permission and approval to abandon by sale certain facilities in Weld County, Colorado, to Pantera Energy Company (Pantera), all as more fully set forth in the application which is on file with the Commission and open to public inspection.

Northern requests that it be permitted to abandon by sale to Pantera approximately 4 miles of 6-inch pipeline for $120,000. The pipeline is located in Weld County, Colorado. Northern states that it would not experience any interruption of service nor increased costs as a result of the abandonment of the proposed sale.

Any person desiring to be heard or to make any protest with reference to said application should on or before November 9, 1984, file with the Federal Energy Regulatory Commission, Washington, D.C. 20426, a motion to intervene or a protest in accordance with the requirements of the Commission's Rules of Practice and Procedure (18 CFR 385.214 or 385.211) and the Regulations under the Natural Gas Act (18 CFR 157.10). All protests filed with the Commission will be considered by it in determining the appropriate action to be taken but will not serve to make the protestants parties to the proceeding. Any person wishing to become a party to a proceeding or to participate as a party in any hearing therein must file a motion to intervene in accordance with the Commission's Rules.

Take further notice that, pursuant to the authority contained in and subject to jurisdiction conferred upon the Federal Energy Regulatory Commission by sections 7 and 15 of the Natural Gas Act and the Commission's Rules of Practice and Procedure, a hearing will be held without further notice before the Commission or its designee on this application if no motion to intervene is

filed within the time required herein, if the Commission on its own review of the matter finds that permission and approval for the proposed abandonment are required by the public convenience and necessity. If a motion for leave to intervene is timely filed, or if the Commission on its own motion believes that a formal hearing is required, further notice of such hearing will be duly given.

Under the procedure herein provided for, unless otherwise advised, it will be unnecessary for Northern to appear or be represented at the hearing.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84-28369 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

**[Docket No. CP84-754-000]**

## Northwest Pipeline Corp.; Request Under Blanket Authorization

October 23, 1984.

Take notice that on September 28, 1984, Northwest Pipeline Corporation (Northwest), 295 Chipeta Way, Salt Lake City, Utah 84110, filed in docket No. CP84-754-000 a request pursuant to § 157.205(b) of the Regulations under the Natural Gas Act (18 CFR 157.205) for authorization to construct and operate certain natural gas facilities and the reallocation of natural gas service for Cascade Natural Gas Company (Cascade), an existing customer of Northwest, under the certificate issued in Docket No. CP82-433-000 pursuant to section 7 of the Natural Gas Act, all as more fully set forth in the request on file with the Commission and open to public inspection.

Northwest proposes to construct and operate the Selah meter station in Yakima County, Washington, to provide natural gas service to Cascade. Northwest states that the additional volumes of natural gas for resale to Cascade would be within the certificated volumes which Northwest is authorized to sell and deliver to Cascade pursuant to Northwest's presently effective Rate Schedule ODL-1. No increase in the total daily contract demand, which Northwest is authorized to sell and deliver, is proposed nor would any such increase result from the authorizations sought herein, it is submitted. Cascade would reimburse Northwest for all actual costs, estimated to be $138,500, it is stated.

Northwest states that the volumes of natural gas to be sold through the Selah meter station would be reallocated from the existing Longview-Kelso delivery point. It is explained that Cascade has requested a transfer of 20,000 therms per day to provide for the sales at the proposed meter station.

Any person or the Commission's staff may, within 45 days after issuance of the instant notice by the Commission, file pursuant to Rule 214 of the Commission's Procedural Rules (18 CFR 385.214) a motion to intervene or notice of intervention and pursuant to § 157.205 of the Regulations under the Natural Gas Act (18 CFR 157.205) a protest to the request. If no protest is filed within the time allowed therefor, the proposed activity shall be deemed to be authorized effective the day after the time allowed for filing a protest. If a protest is filed and not withdrawn within 30 days after the time allowed for filing a protest, the instant request shall be treated as an application for authorization pursuant to section 7 of the Natural Gas Act.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84-28370 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

**[Docket No. CP84-735-000]**

## Ozark Gas Transmission System; Application

October 23, 1984.

Take notice that on September 28, 1984, Ozark Gas Transmission System (Ozark), First City Center, 1700 Pacific Avenue, Dallas, Texas, filed in Docket No. CP84-753-000 an application pursuant to section 7(c) of the Natural Gas Act for a certificate of public convenience and necessity authorizing the construction and operation of tap and measurement facilities along with approximately 60 feet of twelve-inch pipeline located in White County, Arkansas, all as more fully set forth in the application which is on file with the Commission and open to public inspection.

Ozark states that it proposes to construct and operate the subject facilities to attach facilities of Ozark in White County, Arkansas, to facilities to be constructed by Tennessee Gas Pipeline Company, a Division of Tenneco Inc. (Tennessee), which would intersect with facilities of Texas Eastern Transmission Corporation. Ozark, which indicates that it transports gas for Columbia Gas Transmission Corporation and Tennessee, states that currently all gas transported through its system is delivered into the facilities of Natural Gas Pipeline Company of America (Natural) which then redelivers or exchanges gas with the shippers. Ozark states that the ability to receive shippers' gas into the new pipeline would relieve dependence on Natural's facilities, promote lower costs and help assure operational flexibility. Ozark also states that Tennessee would reimburse Ozark for the cost of the facilities.

Any person desiring to be heard or to make any protest with reference to said application should on or before November 9, 1984, file with the Federal Energy Regulatory Commission, Washington, D.C. 20426, a motion to intervene or a protest in accordance with the requirements of the Commission's Rules of Practice and Procedure (18 CFR 385.214 or 385.211) and the Regulations under the Natural Gas Act (18 CFR 157.10). All protests filed with the Commission will be considered by it in determining the appropriate action to be taken but will not serve to make the protestants parties to the proceeding. Any person wishing to become a party to a proceeding or to participate as a party in any hearing therein must file a motion to intervene in accordance with the Commission's Rules.

Take further notice that, pursuant to the authority contained in and subject to jurisdiction conferred upon the Federal Energy Regulatory Commission by sections 7 and 15 of the Natural Gas Act and the Commission's Rules of Practice and Procedure, a hearing will be held without further notice before the Commission or its designee on this application if not motion to intervene is filed within the time required herein, if the Commission on its own review of the matter finds that a grant of the certificate is required by the public convenience and necessity. If a motion for leave to intervene is timely filed, or if the Commission on its own motion believes that a formal hearing is required, further notice of such hearing will be duly given.

Under the procedure herein provided for, unless otherwise advised, it will be unnecessary for Ozark to appear or be represented at the hearing.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84-28372 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

**[Docket No. CP84-472-001]**

## Panhandle Eastern Pipe Line Co.; Request Under Blanket Authorization

October 23, 1984.

Take notice that on October 2, 1984, Panhandle Eastern Pipe Line Company (Applicant), P.O. Box 1642, Houston

Texas 77001, filed in Docket No. CP84–472–001 a request pursuant to § 157.205 of the Commission's Regulations under the Natural Gas Act (18 CFR 157.205) for flexible authority to add or delete sources of supply or receipt/delivery points under the certificate issued in Docket No. CP83–83–000 pursuant to section 7 of the Natural Gas Act, all as more fully set forth in the request which is on file with the Commission and open to public inspection.

Applicant states that it made its prior notice filing on June 8, 1984, in Docket No. CP84–472–000 and no protests were received during the 45-day prior notice period ending July 29, 1984, and accordingly, authority was granted pursuant to § 157.205 of the Commission's Regulations effective on July 30, 1984. Applicant indicates that under that authority it receives gas at the tailgate of Union Texas Petroleum Corporation Chaney Dell plant in Major County, Oklahoma, and then transports and redelivers the subject gas to Central Illinois Light Company which in turn makes ultimate redelivery to Midwest Solvents Company for its end-use at its facility in Pekin, Illinois.

By this petition to amend the existing authorization, Applicant requests flexible authority to add or delete sources of supply or receipt/delivery points, if such altered service is on behalf of the same end-user, at the same end-user location, within the maximum daily and annual volumes authorized in this docket, and under the same terms and conditions authorized for the basic service. Within 30 days of the addition or deletion of any gas suppliers and/or receipt/delivery points, Applicant states that it would file the following information in this docket, where applicable to the changes in service:

(i) Copy of the gas purchase contract between the seller and the end-user;

(ii) Statement as to whether the supply is attributable to gas under contract and released by a pipeline or distributor and if so, identification of the parties, and specification of the current contract price;

(iii) Statement of the Natural Gas Policy Act of 1978 (NGPA) pricing categories of the added supply, if released gas, and the volumes attributable to each category;

(iv) Statement that the gas is not committed or dedicated within the meaning of the NGPA Section 2(18);

(v) Location of the receipt/delivery points being added or deleted, and the name of the producer/supplier;

(vi) Where an intermediary participates in the transaction between the seller and the end-user, the information required by § 157.209(c)(1)(ix) of the Commission's Regulations;

(vii) Identity of any other pipeline involved in the transport.

Other than its request for flexible authority, Applicant proposes no other change to the activity as previously granted in this proceeding.

Any person or the Commission's staff may, within 45 days after issuance of the instant notice by the Commission, file pursuant to Rule 214 of the Commission's Procedural Rules (18 CFR 385.214) a motion to intervene or notice of intervention and pursuant to § 157.205 of the Regulations under the Natural Gas Act (18 CFR 157.205) a protest to the request. If no protest is filed within the time allowed therefor, the proposed activity shall be deemed to be authorized effective the day after the time allowed for filing a protest. If a protest is filed and not withdrawn within 30 days after the time allowed for filing a protest, the instant request shall be treated as an application for authorization pursuant to section 7 of the Natural Gas Act.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84–28373 Filed 10–25–84; 8:45 am]

**BILLING CODE 6717–01–M**

---

**[Docket No. CP84–757–000]**

## Panhandle Eastern Pipe Line Co.; Application

October 23, 1984.

Take notice that on September 28, 1984, Panhandle Eastern Pipe Line Company (Applicant), P.O. Box 1642, Houston, Texas 77001, filed in Docket No. CP84–757–000 an application pursuant to section 7(c) of the Natural Gas Act for a certificate of public convenience and necessity authorizing the transportation of natural gas for Kansas Industrial Energy Supply Company (Kansas Industrial), all as more fully set forth in the application which is on file with the Commission and open to public inspection.

Applicant proposes, pursuant to a transportation agreement dated August 20, 1984, to transport, on an interruptible basis, up to 3,000 Mcf of natural gas per day for Kansas Industrial which gas is being purchased by Kansas Industrial from Hoppy Toad Production Company (Hoppy Toad) pursuant to a gas purchase contract dated December 14, 1983. Applicant states that it would receive the gas from Oklahoma Natural Gas Company (ONG) for Kansas Industrial's account in Dewey County, Oklahoma, and would redeliver equivalent volumes, less 1 percent for fuel and unaccounted-for line loss, to Getty Gas Gathering (Getty) for Kansas Industrial's account in Harper County, Kansas. Applicant asserts that Kansas Industrial has made separate arrangements with ONG and Getty, respectively, to deliver the gas to and receive the gas from Applicant.

It is asserted that Kansas Industrial would pay Applicant a rate of 3.90 cents per Mcf plus 1.25 cents per Mcf for the GRI Funding Unit.

Any person desiring to be heard or to make any protest with reference to said application should on or before November 9, 1984, file with the Federal Energy Regulatory Commission, Washington, DC 20426, a motion to intervene or a protest in accordance with the requirements of the Commission's Rules of Practice and Procedure (18 CFR 385.214 or 385.211) and the Regulations under the Natural Gas Act (18 CFR 157.10). All protests filed with the Commission will be considered by it in determining the appropriate action to be taken but will not serve to make the protestants parties to the proceeding. Any person wishing to become a party to a proceeding or to participate as a party in any hearing therein must file a motion to intervene in accordance with the Commission's Rules.

Take further notice that, pursuant to the authority contained in and subject to jurisdiction conferred upon the Federal Energy Regulatory Commission by sections 7 and 15 of the Natural Gas Act and the Commission's Rules of Practice and Procedure, a hearing will be held without further notice before the Commission or its designee on this application if no motion to intervene is filed within the time required herein, if the Commission on its own review of the matter finds that a grant of the certificate is required by the public convenience and necessity. If a motion for leave to intervene is timely filed, or if the Commission on its own motion believes that a formal hearing is required, further notice of such hearing will be duly given.

Under the procedure herein provided for, unless otherwise advised, it will be unnecessary for Applicant to appear to be represented at the hearing.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84–28374 Filed 10–25–84; 8:45 am]

**BILLING CODE 6717–01–M**

[Docket No. CP84-758-000]

## Panhandle Eastern Pipe Line Co.; Application

October 23, 1984.

Take notice that on September 28, 1984, Panhandle Eastern Pipe Line Company (Panhandle), P.O. Box 1642, Houston, Texas 77001, filed in Docket No. CP84-758-000 an application pursuant to section 7(c) of the Natural Gas Act for a certificate of public convenience and necessity authorizing the transportation of natural gas on behalf of UGI Corporation (UGI), all as more fully set forth in the application which is on file with the Commission and open to public inspection.

It is asserted that pursuant to a transportation agreement dated June 30, 1984, Panhandle has agreed to transport, on an interruptible basis, up to 40,000 Mcf of natural gas per day on behalf of UGI. Panhandle states it would receive the gas for UGI's account in Dewey and Major Counties, Oklahoma. Panhandle explains that it would redeliver the gas for UGI's account to Columbia Gas Transmission Corporation in Drake, Paulding and Lucas Counties, Ohio. It is submitted that UGI would pay Panhandle 39.0 cents per Mcf for this service and that the term of the proposed service would be until June 30, 1985, and for successive terms of three months, unless cancelled by either party giving three-months written notice.

Panhandle states that the subject gas is purchased by UGI for its system supply from Delhi Gas Pipeline Corporation.

Any person desiring to be heard or to make any protest with reference to said application should on or before November 9, 1984, file with the Federal Energy Regulatory Commission, Washington, D.C. 20426, a motion to intervene or a protest in accordance with the requirements of the Commission's Rules of Practice and Procedure (18 CFR 385.214 or 385.211) and the Regulations under the Natural Gas Act (18 CFR 157.10). All protests filed with the Commission will be considered by it in determining the appropriate action to be taken but will not serve to make the protestants parties to the proceeding. Any person wishing to become a party to a proceeding or to participate as a party in any hearing therein must file a motion to intervene in accordance with the Commission's Rules.

Take further notice that, pursuant to the authority contained in and subject to jurisdiction conferred upon the Federal Energy Regulatory Commission by sections 7 and 15 of the Natural Gas Act and the Commission's Rules of Practice and Procedure, a hearing will be held without further notice before the Commission or its designee on this application if no motion to intervene is filed within the time required herein, if the Commission on its own review of the matter finds that a grant of the certificate is required by the public convenience and necessity. If a motion for leave to intervene is timely filed, or if the Commission on its own motion believes that a formal hearing is required, further notice of such hearing will be duly given.

Under the procedure herein provided for, unless otherwise advised, it will be unnecessary for Panhandle to appear to be represented at the hearing.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84-28375 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

[Docket No. CP84-732-000]

## Texas Eastern Transmission Corp.; Request Under Blanket Authorization

October 23, 1984.

Take notice that on September 24, 1984, Texas Eastern Transmission Corporation (Texas Eastern), Post Office Box 2521, Houston, Texas 77252, filed in Docket No. CP84-732-000 a request pursuant to § 157.205 of the Regulations under the Natural Gas Act (18 CFR 157.205) for authorization to construct a new sales delivery point to Mississippi Valley Gas Company (Mississippi Valley), an existing customer, under the certificate issued in Docket No. CP82-535-000 pursuant to section 7 of the Natural Gas Act, all as more fully set forth in the request on file with the Commission and open to public inspection.

Texas Eastern states that the proposed sales tap would be located in Hinds County, Mississippi. Texas Eastern states that the maximum daily delivery obligation would be 6,000 dt equivalent of gas which would be within the volume Texas Eastern is presently authorized to sell and deliver to Mississippi Valley. Texas Eastern asserts that it would be reimbursed by Mississippi Valley for the estimated $140,600 cost of constructing the metering and regulating facilities.

Any person or the Commission's staff may, within 45 days after issuance of the instant notice by the Commission, file pursuant to Rule 214 of the Commission's Procedural Rules (18 CFR 385.214) a motion to intervene or notice of intervention and pursuant to § 157.205 of the Regulations under the Natural Gas Act (18 CFR 157.205) a protest to the request. If no protest is filed within the time allowed therefor, the proposed activity shall be deemed to be authorized effective the day after the time allowed for filing a protest. If a protest is filed and not withdrawn within 30 days after the time allowed for filing a protest, the instant request shall be treated as an application for authorization pursuant to section 7 of the Natural Gas Act.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84-28376 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

[Docket No. CP84-759-000]

## Trunkline Gas Co.; Application

October 23, 1984.

Take notice that on September 28, 1984, Trunkline Gas Company (Applicant), P.O. Box 1642, Houston, Texas 77001, filed in Docket No. CP84-759-000 an application pursuant to section 7(c) of the Natural Gas Act for a certificate of public convenience and necessity authorizing the transportation of public natural gas for Louisiana Industrial Gas Supply System (LIGS), all as more fully set forth in the application which is on file with the Commission and open to public inspection.

It is stated that Applicant and LIGS have entered into a transportation agreement dated April 17, 1984, wherein Applicant has agreed to transport up to 75,000 Mcf of natural gas per day for LIGS. It is indicated that Applicant would receive transportation quantities for LIGS' account at a point on its system in Jefferson Davis Parish, Louisiana, and would redeliver equivalent quantities to Southern Natural Gas Company for LIGS' account in West Carroll Parish, Louisiana. It is explained that the gas to be transported is gas which LIGS has available from Tennessee Gas Pipeline Company, a Division of Tenneco Inc.

Applicant further indicates that the transportation service for LIGS was initiated pursuant to Subpart B of Part 284 of the Commission's Regulations as reported in Docket No. ST84-762-000. For the transportation service Applicant proposes to charge LIGS 7.58 cents per Mcf, plus 1.25 cents per Mcf representing the Gas Research Institute surcharge. The term of the proposed service would be two years with the provision for successive terms of 6 months.

Any person desiring to be heard or to make any protest with reference to said application should on or before

November 9, 1984, file with the Federal Energy Regulatory Commission, Washington, D.C. 20426, a motion to intervene or a protest in accordance with the requirements of the Commission's Rules of Practice and Procedure (18 CFR 385.214 or 385.211) and the Regulations under the Natural Gas Act (18 CFR 157.10). All protests filed with the Commission will be considered by it in determining the appropriate action to be taken but will not serve to make the protestants parties to the proceeding. Any person wishing to become a party to a proceeding or to participate as a party in any hearing therein must file a motion to intervene in accordance with the Commission's Rules.

Take further notice that, pursuant to the authority contained in and subject to jurisdiction conferred upon the Federal Energy Regulatory Commission by sections 7 and 15 of the Natural Gas Act and the Commission's Rules of Practice and Procedure, a hearing will be held without further notice before the Commission or its designee on this application if no motion to intervene is filed within the time required herein, if the Commission on its own review of the matter finds that a grant of the certificate is required by the public convenience and necessity. If a motion for leave to intervene is timely filed, or if the Commission on its own motion believes that a formal hearing is required, further notice of such hearing will be duly given.

Under the procedure herein provided for, unless otherwise advised, it will be unnecessary for Applicant to appear or be represented at the hearing.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84-28377; Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

**[Docket No. CP81-212-002]**

## United Gas Pipe Line Co.; Petition To Amend

October 23, 1984.

Take notice that on October 1, 1984, United Gas Pipe Line Company (Petitioner), P.O. Box 1478, Houston, Texas 77001, filed in Docket No. CP81-212-002 a petition to amend the order issued December 2, 1981, in Docket No. CP81-212-000 pursuant to section 7(c) of the Natural Gas Act so as to authorize the transportation of natural gas to an additional delivery point for Shell Oil Company (Shell), all as more fully set forth in the petition to amend.

It is stated that by order issued December 2, 1981, in Docket No. CP81-212-000, Petitioner was authorized to transport natural gas for Shell from a mutually agreeable point in Natchitoches Parish, Louisiana, to points of redelivery in St. Charles and Assumption Parishes, Louisiana, and Rankin County, Mississippi, under a gas transportation agreement between Petitioner and Shell dated October 28, 1980. Petitioner states that May 11, 1984, amendment to the transportation agreement provides for an additional point of redelivery at the outlet side of several measuring and regulating stations where Petitioner presently delivers gas to Mobile Gas Service Corporation in Mobile County, Alabama. Petitioner proposes to deliver gas to Shell at the proposed point in Mobile County, Alabama.

Any person desiring to be heard or to make any protest with reference to said petition to amend on or before November 9, 1984, file with the Federal Energy Regulatory Commission, Washington, D.C. 20426, a motion to intervene or a protest in accordance with the requirements of the Commission's Rules of Practice and Procedure (18 CFR 385.214 or 385.211) and the Regulations under the Natural Gas Act (18 CFR 157.10). All protests filed with the Commission will be considered by it in determining the appropriate action to be taken but will not serve to make the protestants parties to the proceeding. Any person wishing to become a party to a proceeding or to participate as a party in any hearing therein must file a motion to intervene in accordance with the Commission's Rules.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84-28378; Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

**[Docket No. CP84-424-001]**

## United Gas Pipe Line Co.; Petition To Amend

October 22, 1984.

Take notice that on October 10, 1984, United Gas Pipe Line Company (United), Post Office Box 1478, Houston, Texas 77001, filed in Docket No. CP84-424-001 a petition pursuant to section 7(c) of the Natural Gas Act to amend the order issued September 19, 1984, in Docket No. CP84-424-000, so as to authorize a new location for the construction and operation of certain facilities necessary to initiate gas service to the Town of Arnaudville, Louisiana (Arnaudville), all as more fully set forth in the petition to amend which is on file with the Commission and open to public inspection.

United states that the order of September 19, 1984, authorized it to construct a sales meter station and appurtenant facilities on its 36-inch pipeline located south of the Arnaudville compressor station in St. Martin Parish, Louisiana. United now proposes to make its deliveries and construct the authorized facilities on its 30-inch loop line located north of the Arnaudville compressor station in St. Landry Parish, Louisiana. United also submitted revised Exhibits F, F-1, and G-1, in Docket No. CP84-424-001. The requested change in location is to comply with Arnaudville's desire for a new location as Arnaudville would construct the necessary lateral of its distribution system to connect to United's proposed facilities, it is asserted.

Any person desiring to be heard or to make any protest with reference to said petition to amend on or before Nov. 9, 1984, file with the Federal Energy Regulatory Commission, Washington, D.C. 20426, a motion to intervene or a protest in accordance with the requirements of the Commission's Rules of Practice and Procedure (18 CFR 385.214 or 385.211) and the Regulations under the Natural Gas Act (18 CFR 157.10). All protests filed with the Commission will be considered by it in determining the appropriate action to be taken but will not serve to make the protestants parties to the proceeding. Any person wishing to become a party to a proceeding or to participate as a party in any hearing therein must file a motion to intervene in accordance with the Commission's Rules.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84-28380 Filed 10-25-84; 8:45 am]

BILLING CODE 6717-01-M

---

**[Docket No. CP84-749-000]**

## United Gas Pipe Line Co.; Request Under Blanket Authorization

October 23, 1984.

Take notice that on September 27, 1984, United Gas Pipe Line Company (United), P.O. Box 1478, Houston, Texas 77001, filed in Docket No. CP84-749-000 a request pursuant to § 157.205 of the Regulations under the Natural Gas Act (18 CFR § 157.205) for authorization to construct and operate a sales tap for the delivery of gas to Entex, Inc., in St. Mary Parish, Louisiana, under the certificate issued in Docket No. CP82-430-000 pursuant to section 7 of the Natural Gas

Act, all as more fully set forth in the request on file with the Commission and open to public inspection.

United states that its proposed 8-inch tap would be installed on United's 30-inch Bayou Sale Junction line in St. Mary Parish, Louisiana. It is indicated United would use the tap to supply up to 25,000 Mcf of natural gas per day to Entex, Inc., for resale to the Central Louisiana Electric Company for use as boiler fuel in an electric power generation station. It is further indicated that the service would be provided under United's Rate Schedule DG–S. United avers that the sale volumes would be within Entex, Inc.'s existing entitlement and that sufficient capacity exists on United's system to render the service without detriment or disadvantage to its existing customers.

Any person or the Commission's staff may, within 45 days after issuance of the instant notice by the Commission, file pursuant to Rule 214 of the Commission's Procedural Rules (18 CFR 385.214) a motion to intervene or notice of intervention and pursuant to § 157.205 of the Regulations under the Natural Gas Act (18 CFR 157.205) a protest to the request. If no protest is filed within the time allowed therefor, the proposed activity shall be deemed to be authorized effective the day after the time allowed for filing a protest. If a protest is filed and not withdrawn within 30 days after the time allowed for filing a protest, the instant request shall be treated as an application for authorization pursuant to section 7 of the Natural Gas Act.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84–28381 Filed 10–25–84; 8:45 am]

BILLING CODE 6717–01–M

---

**[Docket No. CP84–623–000]**

## Western Gas Interstate Co.; Application

October 23, 1984.

Take notice that on July 30, 1984, Western Gas Interstate Company (Western), 400 West 15th Street, Suite 900, Austin, Texas 78701, filed in Docket No. CP84–623–000 an application pursuant to section 7(b) of the Natural Gas Act for permission and approval to abandon certain facilities and associated services rendered in connection with Western's sales to and transportation for Gas Company of New Mexico (GCNM) within the State of New Mexico, and the abandonment of certain facilities and associated services rendered in the tranportation of gas for Southern Union Exploration Company (SX) within the State of New Mexico, all as more fully set forth in the application which is on file with the Commission and open to public inspection.

Western proposes to abandon the transportation and sale of gas to GCNM under Western's Rate Schedule G–R. Further, Western seeks to abandon facilities used to transport gas purchased from Shell Western E&P, Inc. (Shell) and Continental Oil Company (Conoco) from the outlet of the Shell Antelope Ridge processing plant to Transwestern Pipeline Company's pipeline facilities, all in Lea County, New Mexico. It is stated that the contract with Shell and Conoco under which Western purchased gas for resale to GCNM under Rate Schedule G–R expired on July 12, 1983, and has not been renewed. Western asserts that GCNM has acquired supplies from other sources to meet requirements previously furnished by Western so that no diminution of service to GCNM's customers would occur as a result of the proposed abandoment.

Western also seeks Commission authorization to abandon transportation services provided for SX in Lea and San Juan Counties, New Mexico, under Western's Rate Schedules T–2 and T–3, and to abandon the facilities used to perform such services. Currently, it is indicated, under the Rate Schedule T–2 arrangement, Western transports SX's production from three wells in Lea County, New Mexico, through approximately 10 miles of 4-inch transmission line for delivery to El Paso Natural Gas Company (EPNG) and then EPNG transports the gas to GCNM and Southern Union Company (SUG). It is explained that the Lea County production is sold to GCNM and SUG. Western asserts that CGNM and SUG do not want to continue purchasing the gas from SX under the current arrangements. Western's transmission line is not used for service other than the one provided for SX, it is asserted; and Western requests authorization to abandon the line in place.

Finally, Western requests authorization to abandon a transportation service and the facilities used in performing the service for SX in San Juan County, New Mexico, under Rate Schedule T–3. Western states that it transports gas to EPNG's facilities for SX through approximately 1.5 miles of transmission line and that the gas is sold by SX by GCNM. Upon abandonment authorization, Western proposes to transfer the line of SX for use by SX as a gathering line.

Any person desiring to be heard or to make any protest with reference to said application should on or before November 9, 1984, file with Federal Energy Regulatory Commission, Washington, D.C. 20426, a motion to intervene or a protest in accordance with the requirements of the Commission's Rules of Practice and Procedure (18 CFR 385.214 or 385.211) and the Regulations under the Natural Gas Act (18 CFR 157.10). All protests filed with the Commission will be considered by it in determining the appropriate action to be taken but will not serve to make the protestants parties to the proceeding. Any person wishing to become a party to a proceeding or to participate as a party in any hearing therein must file a motion to intervene in accordance with the Commission's Rules.

Take further notice that, pursuant to the authority contained in and subject to the jurisdiction conferred upon the Federal Energy Regulatory Commission by sections 7 and 15 of the Natural Gas Act and the Commission's Rules of Practice and Procedure, a hearing will be held without further notice before the Commission or its designee on this application if no motion to intervene is filed within the time required herein, if the Commission on its own review of the matter finds that permission and approval for the proposed abandonment are required by the public convenience and necessity. If a motion for leave to intervene is timely filed, or if the Commission on its own motion believes that a formal hearing is required, further notice of such hearing will be duly given.

Under the procedure herein provided for, unless otherwise advised, it will be unnecessary for Western to appear or be represented at the hearing.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84–28382 Filed 10–25–84; 8:45 am]

BILLING CODE 6717–01–M

---

**[Docket No. OF85–9–000]**

## OLS Energy-Agnews; Application for Commission Certification of Qualifying Status of a Cogeneration Facility

October 23, 1984.

On October 5, 1984, OLS Energy-Agnews (Applicant) of 450 Sansome-Suite 210, San Francisco, California 94111, submitted for filing an application for certification of a facility as a qualifying cogeneration facility pursuant to § 292.207 of the Commission's regulations. No determination has been made that the submittal constitutes a complete filing.

The topping-cycle cogeneration facility will be located at the Agnews State Hospital, San Jose, California. The primary energy source will be natural gas. The facility will consist of a combustion turbine generator unit rated 20,600 kilowatts, and a heat recovery steam generator supplying an extraction turbine generator unit rated 6,600 kilowatts. The extracted steam will be used to supply the hospital's process heating load. The facility is scheduled to be installed by the end of 1986.

Any person desiring to be heard or objecting to the granting of qualifying status should file a petition to intervene or protest with the Federal Energy Regulatory Commission, 825 North Capitol Street NE., Washington, D.C. 20426, in accordance with rules 211 and 214 of the Commission's Rules of Practice and Procedure. All such petitions or protests must be filed within 30 days after the date of publication of this notice and must be served on the applicant. Protests will be considered by the Commission in determining the appropriate action to be taken but will not serve to make protestants parties to the proceeding. Any person wishing to become a party must file a petition to intervene. Copies of this filing are on file with the Commission and are available for public inspection.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84-28371 Filed 10-25-84; 8:45 am]

**BILLING CODE 6717-01-M**

---

**[Docket No. QF84-442-000]**

## Turbine Tech., Inc.; Application for Commission Certification of Qualifying Status of a Small Power Production Facility

October 23, 1984.

On October 4, 1984, Turbine Tech., Inc., (Applicant) of 9000 Eucalyptus Street, Bakersfield, California 93306, submitted for filing a revised application for certification of a facility as a qualifying small power production facility pursuant to § 292.207 of the Commission's regulations. On August 8, 1984, the Applicant initially submitted for filing an application for certification of the same facility as a qualifying cogeneration facility. No determination has been made that the submittal constitutes a complete filing.

The demonstration facility is located at Ringling, Oklahoma in Jefferson County. The primary energy resource is claimed to be waste inertia as a result of imbalance between the upstroke and downstroke in oil well pump jack operations. The applicant states that the facility has a power production capacity of 15 kilowatts.

Any person desiring to be heard or to objecting to the granting of qualifying status should file a petition to intervene or protest with the Federal Energy Regulatory Commission, 825 North Capitol Street NE., Washington, D.C. 20426, in accordance with rules 211 and 214 of the Commission's Rules of Practice and Procedure. All such petitions or protests must be filed within 30 days after the date of publication of this notice and must be served on the applicant. Protests will be considered by the Commission in determining the appropriate action to be taken but will not serve to make the protestants parties to the proceeding. Any person wishing to become a party must file a motion to intervene. Copies of this filing are on file with the Commission and are available for public inspection.

**Kenneth F. Plumb,**

*Secretary.*

[FR Doc. 84-28378 Filed 10-25-84; 8:45 am]

**BILLING CODE 6717-01-M**

---

## ENVIRONMENTAL PROTECTION AGENCY

**[OPTS-51542; BH-FRL 2703-3]**

## Certain Chemicals; Premanufacture Notices

**AGENCY:** Environmental Protection Agency (EPA).

**ACTION:** Notice.

**SUMMARY:** Section 5(a)(1) of the Toxic Substances Control Act (TSCA) requires any person who intends to manufacture or import a new chemical substance to submit a premanufacture notice (PMN) to EPA at least 90 days before manufacture or import commences. Statutory requirements for section 5(a)(1) premanufacture notices are discussed in EPA statements of the final rule published in the **Federal Register** of May 13, 1983 (48 FR 21722). This notice announces receipt of twenty-six PMNs and provides a summary of each.

**DATES:** Close of Review Period:

PMN 85-28, 85-29, 85-30 and 85-31; January 9, 1985.

PMN 85-32; January 12, 1985.

PMN 85-33, 85-34, 85-35, 85-36, 85-37, 85-38, 85-39, 85-40, 85-41, 85-42, 85-43, 85-44 and 85-45; January 13, 1985.

PMN 85-46, 85-47, 85-48, 85-49 and 85-50; January 14, 1985.

PMN 85-51, 85-52 and 85-53; January 15, 1985.

Written comments by:

PMN 85-28, 85-29, 85-30 and 85-31; December 10, 1984.

PMN 85-32; December 13, 1984.

PMN 85-33, 85-34, 85-35, 85-36, 85-37, 85-38, 85-39, 85-40, 85-41, 85-42, 85-43, 85-44 and 85-45; December 14, 1984.

PMN 85-46, 85-47, 85-48, 85-49 and 85-50; December 15, 1984.

PMN 85-51, 85-52 and 85-53; December 16, 1984.

**ADDRESS:** Written comments, identified by the document control number "[OPTS-51542]" and the specific PMN number should be sent to: Document Chemical Officer (TS-793), Chemical Information Branch, Information Management Division, Office of Toxic Substances, Environmental Protection Agency, Rm. E-201, 401 M St., SW., Washington, DC 20460, (202-382-3532).

**FOR FURTHER INFORMATION CONTACT:** Wendy Cleland-Hamnett, Premanufacture Notice Management Branch, Chemical Control Division (TS-794), Office of Toxic Substances, Environmental Protection Agency, Rm. E-611, 401 M St., SW., Washington, DC 20460, (202-382-3729).

**SUPPLEMENTARY INFORMATION:** The following notice contains information extracted from the non-confidential version of the submission provided by the manufacturer on the PMNs received by EPA. The complete non-confidential document is available in the Public Reading Room E-107 at the above address.

*PMN 85-28*

*Importer.* Confidential.

*Chemical.* (G) Trisubstituted benzene.

*Use/Import.* (S) Site-limited and industrial intermediate for pesticide. Import range: Confidential.

*Toxicity Data.* Acute oral: Male—>5,000 mg/kg, Female—3,299 mg/kg; Acute dermal: >5,000 mg/kg; Irritation: Skin—Not an irritant, Eye—Not an irritant; $LC_{50}$ inhalation: > 2,100 mg/m³.

*Exposure:* Use: Dermal, a total of 1 worker, up to .2 hr/da, up to .04 da/yr.

*Environmental Release/Disposal.* .35 kg/day released to air with 10 kg/batch to land.

*PMN 85-29*

*Importer.* Confidential.

*Chemical.* (G) Trisubstituted benzene.

*Use/Import.* (S) Site-limited and industrial intermediate for pesticide. Import range: Confidential.

*Toxicity Data.* Acute oral: Male—266 mg/kg, Female—114 mg/kg; dermal: Male—2,073 ul/kg, Female—3,439 ul/kg; Irritation: Skin—Moderate, Eye—Slight; $LC_{50}$ inhalation: Severe irritation.

*Exposure.* Use: Dermal, a total of 1 worker, up to .2 hr/da, up to .04 da/yr.

*Environmental Release/Disposal.* Less than $10^{-4}$ kg/batch released to air.

*PMN 85–30*

*Manufacturer.* Confidential.

*Chemical.* (G) Carbopolycycle sulfonate of substituted phenyl azo substituted heteromonocycle.

*Use/Production.* (G) Open, non-dispersive use. Prod. range: Confidential.

*Toxicity Data.* Acute oral: 21 g/kg.

*Exposure.* Confidential.

*Environmental Release/Disposal.* Confidential. Disposal by navigable waterway.

*PMN 85–31*

*Manufacturer.* Confidential.

*Chemical.* (G) Carbopolycycle sulfonate of substituted heteropolycycle.

*Use/Production.* (G) Open, non-dispersive use. Prod. range: Confidential.

*Toxicity Data.* Irritation: Eye—Negative.

*Exposure.* Confidential.

*Environmental Release/Disposal.* Confidential. Disposal by navigable waterway.

*PMN 85–32*

*Manufacturer.* Confidential.

*Chemical.* (G) Alkyl mercaptothiadiazole.

*Use/Production.* (G) Contained and destructive use. Prod. range: Confidential.

*Toxicity Data.* No data on the PMN substance submitted.

*Exposure.* Manufacturer: Dermal.

*Environmental Release/Disposal.* Confidential.

*PMN 85–33*

*Manufacturer.* Confidential.

*Chemical.* (G) Polymonocyclic urethane.

*Use/Production.* (G) Industrial coating having a non-dispersive use. Prod. range: 250,000–1,000,000 kg/yr.

*Toxicity Data.* No data submitted.

*Exposure.* Manufacture and processing: Dermal, a total of 27 workers, up to 4 hrs/da, up to 260 da/yr.

*Environmental Release/Disposal.* 10 to 150 kg/batch released to land. Disposal by incineration and landfill.

*PMN 85–34*

*Manufacturer.* Monsanto Company.

*Chemical.* (G) Polyoxypropylene polyoxyethylene block copolymer ester acyl lactam.

*Use/Production.* (S) Industrial reaction injection molded plastics parts. Prod. range: Confidential.

*Toxicity Data.* Acute oral: > 5,000 mg/kg; Acute dermal: > 5,000 mg/kg; Irritation: Skin—Slight, Eye—Slight.

*Exposure.* Manufacture: Dermal, a total of 6 workers, up to 0.04 hr/da, up to 40 da/yr.

*Environmental Release/Disposal.* 0.25 to 2 kg/batch released. Disposal by incineration.

*PMN 85–35*

*Manufacturer.* Monsanto Company.

*Chemical.* (G) Polybutadiene ester acyl lactam.

*Use/Production.* (S) Industrial reaction injection molded plastics part. Prod. range: Confidential.

*Toxicity Data.* Acute oral: > 5,000 mg/kg; Acute dermal: > 5,000 mg/kg; Irritation: Skin-Slight, Eye—Slight.

*Exposure.* Manufacture: Dermal, a total of 6 workers, up to 0.25 hr/da, up to 160 da/yr.

*Environmental Release/Disposal.* No release. Disposal by incineration.

*PMN 85–36*

*Manufacturer.* The Dow Chemical Company.

*Chemical.* (G) Substituted pyridine.

*Use/Production.* (G) Site-limited chemical intermediate. Prod. range: Confidential.

*Toxicity Data.* Acute oral: Between 130 and 250 mg/kg; Acute dermal: Between 100 and 200 mg/kg; Irritation: Skin—Slight/moderate, Eye—Slight.

*Exposure.* Confidential.

*Environmental Release/Disposal.* Release to air. Disposal by incineration.

*PMN 85–37*

*Manufacturer.* Confidential.

*Chemical.* (G) Isocyanate-terminated polyurethane.

*Use/Production.* (S) Used internally as an intermediate in the production of an adhesive. Prod. range: Confidential.

*Toxicity Data.* No data submitted.

*Exposure.* Manufacture: Dermal a total of 5 workers, up to 1 hr/da, up to 70 da/yr.

*Environmental Release/Disposal.* 5 to 200 gm released. Disposal by incineration.

*PMN 85–38*

*Manufacturer.* Confidential.

*Chemical.* (G) Hydroxyl-terminated polyurethane.

*Use/Production.* (S) Industrial laminating adhesive in ethyl alcohol solution. Prod. range: Confidential.

*Toxicity Data.* No data submitted.

*Exposure.* Manufacture: Dermal, a total of 2 workers, up to 2 hrs/da, up to 42 da/yr.

*Environmental Release/Disposal.* 5 kg/batch released to land. Disposal by landfill.

*PMN 85–39*

*Importer.* Confidential.

*Chemical.* (G) Benzoquinolinyl-sulfoindendione, substituted ammonium salt.

*Use/Import.* (S) Industrial paper dye. Import range: 5,500–16,000 kg/yr.

*Toxicity Data.* No data on the PMN substance submitted.

*Exposure.* Processing: Dermal.

*Environmental Release/Disposal.* No release.

*PMN 85–40*

*Importer.* Confidential.

*Chemical.* (G) Aliphatic ketone.

*Use/Import.* (G) Highly dispersive use. Import range: Confidential.

*Toxicity Data.* Acute oral: > 8,000 mg/kg; Acute dermal; 8,000 mg/kg; Irritation: Eye—Moderate; Skin sensitization: Weak/moderate sensitizer; Phototoxicity test: No phototoxic potential; Photosensitization test: No photosensitizing potential; Repeated insult patch test: Non-irritant/non-sensitizer; Open epicutaneous test: Negative.

*Exposure.* Confidential.

*Environmental Release/Disposal.* Confidential. Disposal by publicly owned treatment works (POTW).

*PMN 85–41*

*Manufacturer.* Emery Industries.

*Chemical.* (s) Carboxylic acids, $C_6$–$C_{18}$ mono and $C_8$–$C_{15}$ di-, polymers with adipic acid, 1.4-butanediol and propylene glycols.

*Use/Production.* (S) Industrial plasticizers for polyvinyl chloride resin. Prod. range: 100,000–150,000 kg/yr.

*Toxicity Data.* No data submitted.

*Exposure.* Manufacture: Dermal, a total of 3–5 workers, up to 4 hrs/da, up to 4–8 da/yr.

*Environmental Release/Disposal.* 75 per 10,000 charge released to water with 185 per 10,000 charge to land. Disposal by POTW and approved landfill.

*PMN 85–42*

*Manufacturer.* Emery Industries.

*Chemical.* (S) Carboxylic acids, $C_6$–$C_{18}$ mono and $C_8$–$C_{15}$ di-, polymers with adipic acid, 1.4-butanediol, propylene glycols and acetic anhydride.

*Use/Production.* (S) Industrial plasticizer for polyvinyl chloride resin. Prod. range: 100,000–150,000 kg/yr.

*Toxicity Data.* No data submitted.

*Exposure.* Manufacture: Dermal, a total of 3–5 workers, up to 4 hrs/da, up to 4–8 da/yr.

*Environmental Release/Disposal.* 75 per 10,000 charge released to water with 185 per 10,000 charge to land. Disposal by POTW and approved landfill.

*PMN 85–43*

*Manufacturer.* Confidential.

*Chemical.* (S) Polymer of phenol, triphenyl ehtyl phosphonium iodide, trimellitic anhydride and EPON 828.

*Use/Production.* (S) Industrial expoxy resin curative agent for powdered coating formulations. Prod. range: 6,800–20,000 kg/yr.

*Toxicity Data.* No data submitted.

*Exposure.* Manufacture and processing: Dermal, a total of 33 workers, up to 8 hrs/da, up to 12 da/yr.

Environmental Release/Disposal. Less than 0.68 to less than 136 kg/batch released with 16.25 kg/da to land. Disposal by incineration and landfill.

*PMN 85–44*

*Manufacturer.* The Minnesota Mining and Manufacturing Company.

*Chemical.* (G) Substituted benzocyazoleydine ethylidine.

*Use/Production.* (G) Dye in coated article. Prod. range: Confidential.

*Toxicity Data.* No data submitted.

*Exposure.* Manufacture and processing: Dermal, a total of 21 workers, up to 8 hrs/da, up to 5 da/yr.

*Environmental Release/Disposal.* Less than 0.002 to less than 0.03 kg/batch released with less than 0.008 kg/batch to land. Disposal by incineration and landfill.

*PMN 85–45*

*Manufacturer.* Confidential.

*Chemical.* (G) 3-substituted propanoic acid, glycol ester.

*Use/Production.* (G) Open-non dispersive. Prod. range: Confidential.

*Toxicity Data.* No data on the PMN substance submitted.

*Exposure.* Manufacture and use: Dermal, a total of 8 workers, up to 2 hrs/da, up to 177 day/yr.

*Environmental Release/Disposal.* 0.5 kg/batch released to water. Disposal by biological treatment system.

*PMN 85–46*

*Manufacturer.* The Dow Chemical Company.

*Chemical.* (G) Sulfonated polycyclic aromatics.

*Use/Production.* (G) Site-limited chemical intermediate. Prod. range: Confidential.

*Toxicity Data.* No data on the PMN substance submitted.

*Exposure.* Confidential.

*Environmental Release/Disposal.* Release to air, water and land. Disposal by incineration and navigable waterway after treatment.

*PMN 85–47*

*Manufacturer.* The Dow Chemical Company.

*Chemical.* (G) Sulfonated polycyclic aromatics, sodium salt.

*Use/Production.* (G) Water reducer and dispersant. Prod. range: Confidential.

*Toxicity Data.* Acute oral: > 5,000 mg/kg; Irritation: Skin—Essentially no irritation, Eye—Slight; Ames test: Negative; $LC_{50}$ 48 hr (Daphnia magna): 140 mg/L; $LC_{50}$ 96 hr (Fathead minnow): 220 mg/L.

*Exposure.* Confidential.

*Environmental Release/Disposal.* Release to air and land. Disposal by incineration.

*PMN 85–48*

*Manufacturer.* The Dow Chemical Company.

*Chemical.* (G) Sulfonated polycyclic aromatics, ammonium salt.

*Use/Production.* (G) Water reducer and dispersant. Prod. range: Confidential.

*Toxicity Data.* No data submitted.

*Exposure.* Confidential.

*Environmental Release/Disposal.* Release to air and land. Disposal by incineration.

*PMN 85–49*

*Manufacturer.* The Dow Chemical Company.

*Chemical.* (G) Sulfonated polycyclic aromatics, calcium salt.

*Use/Production.* (G) Water reducer and dispersant. Prod. range: Confidential.

*Toxicity Data.* No data submitted.

*Exposure.* Confidential.

*Environmental Release/Disposal.* Release to air and land. Disposal by incineration.

*PMN 85–50*

*Manufacturer.* The Dow Chemical Company.

*Chemical.* (G) Sulfonated polycyclic aromatics, zinc salt.

*Use/Production.* (G) Water reducer and dispersant. Prod. range: Confidential.

*Toxicity Data.* No data submitted.

*Exposure.* Confidential.

*Environmental Release/Disposal.* Release to air and land. Disposal by incineration.

*PMN 85–51*

*Manufacturer.* Westvaco Corporation.

*Chemical.* (G) Monoethanolamine salt of lignin.

*Use/Production.* (G) Carrier and binder for coatings, use as colorant or pigment and dispersant/emulsifier. Prod. range: Confidential.

*Manufacturer.* Confidential.

*Toxicity Data.* Acute oral: > 15 g/kg; Irritation: Skin—Non-irritant Eye—Non-irritant.

*Exposure.* Confidential.

*Environmental Release/Disposal.* Confidential. Disposal by navigable waterway.

*PMN 85–52*

*Manufacturer.* Confidential.

*Chemical.* (G) Modified fatty acid polyamine condensate.

*Use/Production.* (G) An additive used in the energy production industry. Prod. range: Confidential.

*Toxicity Data.* No data submitted.

*Exposure.* Manufacture: Dermal, a total of 1 worker, up to 4 hrs/da, up to 8 da/yr.

*Environmental Release/Disposal.* 25 kg/batch released by washout method.

*PMN 85–53*

*Manufacturer.* Confidential.

*Chemical.* (G) Crosslinked acrylic copolymer.

*Use/Production.* (G) For use with aqueous solutions in a contained use. Prod. range: Confidential.

*Toxicity Data.* No data submitted.

*Exposure.* Manufacture: Dermal, a total of 16 workers, up to 4 hrs/da, up to 25 da/yr.

*Environmental Release/Disposal.* 20 to 45 kg/batch released to water. Disposal by publicly POTW.

Dated: October 22, 1984.

**Linda A. Travers,**

*Acting Director, Information Management Division.*

[FR Doc. 84–28203 Filed 10–25–84; 8:45 am]

BILLING CODE 6560–50–M

---

[A–9–FRL–2704–5]

## Approval of Prevention of Significant Air Quality Deterioration (PSD) Permit to Valley Power Associates (EPA Project Number SJ 84–02)

**AGENCY:** Environmental Protection Agency (EPA), Region 9.

**ACTION:** Notice.

**SUMMARY:** Notice is hereby given that on September 24, 1984 the Environmental Protection Agency issued a PSD permit to the applicant named above granting approval to construct a 49.9-megawatt biomass-fired electrical generating facility to be located near the city of Delano, Kern County, California. This permit has been issued under EPA's PSD regulations (40 CFR 52.21) and is subject to certain conditions, including all allowable emission rate as follows: $SO_2$ at 70 lbs/hr, $NO_x$ at 191 lbs/hr, and CO at 260 lbs/hr.

**FOR FURTHER INFORMATION CONTACT:** Copies of the permit are available for

public inspection upon request; address request to: Rhonda Rothschild (M-5), U.S. Environmental Protection Agency, Region 9, 215 Fremont Street, San Francisco, CA 94105, (415) 974-7413, FTS 454-7413.

**SUPPLEMENTARY INFORMATION:** Best Available Control Technology (BACT) requirements include the use of limestone injection and combustion controls. Air Quality Impact modeling was required for $SO_2$, $NO_2$ and CO. Continuous monitoring is required and the source is not subject to New Source Performance Standards.

**DATE:** The PSD permit is reviewable under Section 307(b)(1) of the Clean Air Act only in the Ninth Circuit Court of Appeals. A petition for review must be filed by December 26, 1984.

Dated: October 18, 1984.

**Robert M. Stanburg,**

*Air Management Division, Region 9.*

[FR Doc. 84-28287 Filed 10-25-84; 8:45 am]

BILLING CODE 6560-50-M

---

[A-6-FRL-2704-1]

## Extension of the Expiration Date of a PSD Permit

Notice is hereby given that the Environmental Protection Agency (EPA), Region 6, has extended the expiration date of the following Prevention of Significant Determination (PSD) permit:

1. PSD-TX-385—Central Power and Light; this permit authorized the addition of a 781 MM Btu/hr coal-fired boiler to the existing Coleto Creek Power Plant located near U.S. Highway 59, approximately 3 miles northeast of Fannin, Goliad County, Texas; construction has not commenced because changes in land growth projection and financial considerations have resulted in several schedule modifications; the permit was extended to a new expiration date of November 26, 1984.

A notice of EPA's proposed action to extend this PSD permit was published in a newspaper in the affected area of the facility. No comments were received regarding the proposed extension. Documents relevant to the extension request are available for public inspection during normal business hours at the Air and Waste Management Division, U.S. Environmental Protection Agency, Region 6, 1201 Elm Street, Dallas, Texas 75270.

This extension is a final action reviewable under Section 307(b)(1) of the Clean Air Act only in the Fifth Circuit Court of Appeals. Any petition for review must be filed on or before December 26, 1984.

This notice will have no effect on the National Ambient Air Quality Standards.

The Office of Management and Budget has exempted this information notice from the requirements of Section 3 of Executive Order 12291.

Dated: October 17, 1984.

**Dick Whittington,**

*Regional Administrator, Region 6.*

[FR Doc. 84-28301 Filed 10-25-84; 8:45 am]

BILLING CODE 6560-50-M

---

[ER-FRL-2703-6]

## Environmental Impact Statements; Availability of Weekly Statements

Responsible agency: Office of Federal Activities, General Information (202) 382-5073 or (202) 382-5075.

Availability of Environmental Impact Statements filed October 15, 1984 through October 19, 1984 Pursuant to 40 CFR 1506.9.

EIS No. 840465, Draft, COE, AL, Coffeeville Lock and Dam Hydropower Facility, Construction, Black Warrior-Tombigbee Rivers, Clarke and Choctaw Cos., Due: December 10, 1984, Contact: Ingrid Nester (205) 690-2726.

EIS No. 840469, Draft, FHW, OR, S.E. Hubbard Road Extension, 122nd Avenue to US 212, Clackamas County, Due: December 10, 1984, Contact: Dale Wilken (503) 399-5749.

EIS No. 840471, Final, CDB, CA, Oakland Hotel Two/Parking Garage, Development, UDAG, Alameda County, Due: November 26, 1984, Contact: Charles Bryant (415) 273-3941.

EIS No. 840472, Final, COE, SC, South Carolina Oil Refinery/Underwater Pipeline Installation, Permit, Sampit River, Georgetown County: Due: November 26, 1984, Contact: John Carothers (803) 724-4258.

EIS No. 840473, Draft, BLM, NV, Walker Planning Area Resource Management Plan, Mineral, Lyon, and Douglas Counties, Due: December 10, 1984, Contact: Dave Harmon (702) 470-5748.

EIS No. 840474, Final, FHW, MN, West River Parkway Construction, Franklin Avenue to Plymouth Avenue, Hennepin County, Due: November 26, 1984, Contact: Steven Bahler (612) 725-7001.

EIS No. 840475, Draft, FHW, IN, Lynch Road Extension, Oak Hill Road to IN-62 Intersection, Vanderburgh and Warrick Counties, Due: December 10, 1984,Contact: Lawrence Tucker 1 (317) 269-7492.

EIS No. 840476, Final, COE, OH, Reno Beach Howard Farms Flood Control, Lucas County, Due: November 30, 1984, Contact: William Butler (716) 876-5654.

EIS No. 840477, Draft, UMT, CA, San Jose Multimodel Transportation Terminal, Construction and Development, Santa Clara County, Due: December 12, 1984, Contact: Brigid Hynes-Cherin (415) 556-9368.

EIS No. 840478, Draft, USN, NY, MA, RI, Battleship Surface Action Group Homeport, Construction and Operation, Stapleton-Fort Wadsworth Complex, Staten Island, Richmond County, New York; Boston Army Base, Massachusetts; Quonset Point/Davisville Pier One, Rhode Island and Davisville/Landfill Bulkhead, Rhode Island, Due: December 10, 1984, Contact: Commander T.W. Boone (215) 697-6270.

EIS No. 840479, DRevised, BLM, WY, North Fork Well, Oil and Gas Exploration, Permit, Shoshone National Forest, Park County, Due: December 17, 1984, Contact: John Thompson (307) 347-9871.

Amended Notices:

EIS No. 800763, Draft, NPS, DE, PA, Delaware Water Gap National Recreation Area, Land Management Plan, Published FR 10-14-80 Officially withdrawn.

Dated: October 22, 1984.

**David G. Davis,**

*Acting Director, Office of Federal Activities.*

[FR Doc. 84-28389 Filed 10-25-84; 8:45 am]

BILLING CODE 6560-50-M

---

[ER-FRL-2705-1]

## Environmental Impact Statements; Availability of EPA Comments

Availability of EPA comments prepared October 9, 1984 through October 12, 1984 pursuant to the Environmental Review Process (ERP); under Section 309 of the Clean Air Act and Section 102(2)(c) of the National Environmental Policy Act, as amended. Requests for copies of EPA comments can be directed to the Office of Federal Activities at (202) 382-5075/76. Explanation of the ratings assigned to draft environmental impact statements (EISs) was published in FR dated October 19, 1984 (49 FR 41108).

### Draft EISs

ERP No. D-COE-B32006-00, Rating EO2, Narrows of Lake Champlain Federal Channel Navigation, Maintenance Dredging, VT NY. Summary: EPA finds the document very general and lacking clear and useful environmental data necessary to fully evaluate maintenance dredging. EPA requests additional basic information

and will participate in the review of specific environmental assessments prepared for each particular dredging operation. EPA would oppose any selection of wetlands as disposal sites, since the EIS clearly does not demonstrate the use of wetlands is unavoidable.

ERP No. D–COE–C36055–PR, Rating EC2, Rio Puerto Nuevo Basin, Flood Control Study, PR. Summary: EPA requests that additional information be submitted concerning any ocean dumping permits, method by which dredged spoils will be contained, potential impacts to groundwater, and compensation for loss of mangrove swamps. EPA also requests testing of dredged materials be conducted and clearer maps be provided.

ERP No. DA/COE–E32022–NC, Rating EC2, Manteo (Shallowbag) Bay, Navigation Improvement, NC. Summary: EPA remains concerned about the long-term environmental consequences of the project as well as its practical feasibility over its proposed lifetime. The selected alternative does not appear to be evaluated using the more recent insights regarding the inadvisability of modifying natural shoreline processes.

ERP No. D–FAA–B51010–MA, Rating LO, Barnstable Municipal Airport, Runway 15–33 Extension and Navigation Aid Installation, MA. Summary: The DEIS adequately assesses the environmental impact of the proposed airport extension and improvement, and has proposed the necessary mitigation measures to reduce potential impacts. The FAA needs to commit to implementing those mitigation measures identified in the DEIS.

ERP No. D–FHW–E40657–FL, Rating EC2, FL–44 Upgrading, CR–581 to Eastern Intersection of FL–44/45 (US 41), FL. Summary: EPA has suggested that the noise section and related proposed mitigation measures of this projet need additional analysis and development. Particular emphasis should be given to the reconsideration of using mitigation methods that deal with reduced truck traffic, speed limits, and noise barrier use. Other environmental issues were adequately dealt with.

ERP No. D–MMS–A02207–00, Rating (Sale 94: EC2, Sale 98 and 102: EO2), 1985 Eastern (No. 94), Central (No. 98), and Western (No. 102) Gulf of Mexico OCS Oil and Gas Lease Sales. Summary: Environmental concerns regarding adequate buffer around the Florida Middle Grounds in Eastern Gulf; environmental objections regarding unrestricted leasing near banks in the Central and Western Gulf.

**Final EISs**

ERP No. F–FHW–J40068–CO, South Platte River Crossing, Construction, Canyon Rd. to Santa Fe Dr., CO. Summary: EPA made no formal comments. EPA's recommendations for minor improvements to the draft EIS were adapted in the final.

Dated: October 23, 1984.

**Daivd G. Davis,**

*Acting Director, Office of Federal Activities.*

[FR Doc. 84–28357 Filed 10–25–84; 8:45 am]

BILLING CODE 6560–50–M

## FEDERAL MARITIME COMMISSION

### Agreement(s) Filed

The Federal Maritime Commission hereby gives notice of the filing of the following agreement(s) pursuant to section 5 of the Shipping Act of 1984.

Interested parties may inspect and obtain a copy of each agreement at the Washington, D.C. Office of the Federal Maritime Commission, 1100 L Street, NW., Room 10325. Interested parties may submit comments on each agreement to the Secretary, Federal Maritime Commission, Washington, D.C. 20573, within 10 days after the date of the **Federal Register** in which this notice appears. The requirements for comments are found in § 572.603 of Title 46 of the Code of Federal Regulations. Interested persons should consult this section before communicating with the Commission regarding a pending agreement.

Agreement No.: 202–008770–014.

Title: U.K./U.S.A. Gulf Westbound Rate Agreement.

Parties: Atlantic Cargo Services, AB, Compagnie Generale Maritime, Hapag-Lloyd AG, Intercontinental Transport (ICT) BV, Lykes Bros. Steamship Co., Inc., Sea-Land Service, Inc., Trans Freight Lines, Inc.

Synopsis: The proposed amendment would terminate the agreement 60 days after the North Europe-U.S. Gulf Freight Association (Agreement No. 202–010656) becomes effective.

Agreement No.: 207–009882–002.

Title: Pacific Australia Direct Line Joint Service Agreement.

Parties: Associated Container Transportation (Australia) Ltd., PAD Shipping Australia PTY. Ltd., Rederiaktiebolaget Transatlantic.

Synopsis: The proposed amendment would modify the provisions governing notice of termination.

Agreement No.: 202–009988–016.

Title: Continental/U.S. Gulf Freight Association.

Parties: Atlantic Cargo Services, AB, Compagnie Generale Maritime, Hapag-Lloyd AG, Intercontinental Transport (ICT) BV, Lykes Bros. Steamship Co., Inc., Sea-Land Service, Inc., Trans Freight Lines, Inc.

Synopsis: The proposed amendment would terminate the agreement 60 days after the North Europe-U.S. Gulf Freight Association (Agreement No. 202–010656) becomes effective.

By order of the Federal Maritime Commission.

Dated: October 23, 1984.

**Francis C. Hurney,**

*Secretary.*

[FR Doc. 84–28354 Filed 10–25–84; 8:45 am]

BILLING CODE 6730–01–M

### Controlled Carriers Under the Shipping Act of 1984

**AGENCY:** Federal Maritime Commission.

**ACTION:** Listing of controlled carriers.

**SUMMARY:** The Federal Maritime Commission is adding Flota Bananera Ecuatoriana S.A. to the list of controlled carriers subject to the advanced tariff filing and other regulatory requirements of section 9 of the Shipping Act of 1984.

**DATE:** None.

**FOR FURTHER INFORMATION CONTACT:** Robert G. Drew, Director, Bureau of Tariffs, Federal Maritime Commission, Washington, D.C. 20573.

**SUPPLEMENTARY INFORMATION:** Section 3(8) and 9 of the Shipping Act of 1984 [46 U.S.C. 1702, 1708) provide for the regulation of rates or charges by certain state-owned or so-called "controlled carriers" in the foreign commerce of the United States. Based on information submitted by Flota Bananera Ecuatoriana S.A. (Flota), the Commission determined that Flota meets the definition of a controlled carrier as set forth in section 3(8) of the Act. Flota was so notified by letter dated August 24, 1984 and did not contest this determination. The Commission is therefore adding Flota to the list of controlled carriers published in the **Federal Register** on July 11, 1983 on July 11, 1983 [48 FR 31733].

The process of identification and classification of controlled carriers is continuous. The controlled carrier list is therefore amended as such carriers enter and leave the United States trades or become exempt from the regulatory requirements of section 9.

By the Commission, October 18, 1984.

**Francis C. Hurney,**

*Secretary.*

[FR Doc. 84-28343 Filed 10-25-84; 8:45 am]

BILLING CODE 6730-01-M

## FEDERAL RESERVE SYSTEM

### Central Banc Holding, Inc., et al.; Formations of; Acquisitions by; and Mergers of Bank Holding Companies

The companies listed in this notice have applied for the Board's approval under section 3 of the Bank Holding Company Act (12 U.S.C. 1842) and § 225.14 of the Board's Regulation (12 CFR 225.14) to become a bank holding company or to acquire a bank or bank holding company. The factors that are considered in acting on the applications are set forth in section 3(c) of the Act (12 U.S.C. 1842(c)).

Each application is available for immediate inspection at the Federal Reserve Bank indicated. Once the application has been accepted for processing, it will also be available for inspection at the offices of the Board of Governors. Interested persons may express their views in writing to the Reserve Bank or to the offices of the Board of Governors. Any comment on an application that requests a hearing must include a statement of why a written presentation would not suffice in lieu of a hearing, identifying specifically any questions of fact that are in dispute and summarizing the evidence that would be presented at a hearing.

Unless otherwise noted, comments regarding each of these applications must be received not later than November 16, 1984.

**A. Federal Reserve Bank of Dallas** (Anthony J. Montelaro, Vice President) 400 South Akard Street, Dallas, Texas 75222:

1. *Central Banc Holding, Inc.*, Balch Springs, Texas; to become a bank holding company by acquiring 95 percent of the voting shares of Central Banc Corporation, Balch Springs, Texas, thereby indirectly acquiring First Bank, Balch Springs, Texas, and Central National Bank, Dallas, Texas.

**B. Federal Reserve Bank of San Francisco** (Harry W. Green, Vice President) 101 Market Street, San Francisco, California 94105:

1. *FirstBank Holding Company of California*, Lakewood, California; to become a bank holding company by acquiring 100 percent of the voting shares of First National Bank, Palm Desert, California (in organization).

Board of Governors of the Federal Reserve System, October 23, 1984.

**James McAfee,**

*Associate Secretary of the Board*

[FR Doc. 84-28341 Filed 10-25-84; 8:45 am]

BILLING CODE 6210-01-M

### Mansura Banshares, Inc., et al.; Applications To Engage de Novo in Permissible Nonbanking Activities

The companies listed in this notice have filed an application under § 225.23(a)(1) of the Board's Regulation Y (12 CFR 225.23(a)(1)) for the Board's approval under section 4(c)(8) of the Bank Holding Company Act (12 U.S.C. 1843(c)(8)) and § 225.21(a) of Regulation Y (12 CFR 225.21(a)) to commence or to engage *de novo*, either directly or through a subsidiary, in a nonbanking activity that is listed in § 225.25 of Regulation Y as closely related to banking and permissible for bank holding companies. Unless otherwise noted, such activities will be conducted throughout the United States.

Each application is available for immediate inspection at the Federal Reserve Bank indicated. Once the application has been accepted for processing, it will also be available for inspection at the offices of the Board of Governors. Interested persons may express their views in writing on the question whether consummation of the proposal can "reasonably be expected to produce benefits to the public, such as greater convenience, increased competition, or gains in efficiency, that outweigh possible adverse effects, such as undue concentration of resources, decreased or unfair competition, conflicts of interests, or unsound banking practices." Any request for a hearing on this question must be accompanied by a statement of the reasons a written presentation would not suffice in lieu of a hearing, identifying specifically any questions of fact that are in dispute, summarizing the evidence that would be presented at a hearing, and indicating how the party commenting would be aggrieved by approval of the proposal.

Unless otherwise noted, comments regarding the applications must be received at the Reserve Bank indicated or the offices of the Board of Governors not later than November 15, 1984.

**A. Federal Reserve Bank of Atlanta** (Robert E. Heck, Vice President) 104 Marietta Street, N.W., Atlanta, Georgia 30303:

1. *Mansura Bancshares, Inc.*, Mansura, Louisiana; to engage *de novo* through its subsidiary, Mansura Insurance Agency, Inc., Mansura, Louisiana, in selling all types of general insurance in a town with a population not exceeding 5,000. These activities would be conducted in central Louisiana.

**B. Federal Reserve Bank of Dallas** (Anthony J. Montelaro, Vice President) 400 South Arkard Street, Dallas, Texas 75222:

1. *Independent Community Financial Corp.*, Dallas, Texas; to engage *de novo*, directly in making, acquiring or revising, for its own account or the account of its subsidiary banks, commercial loans, such as would be made, for example, by any commercial, mortgage or consumer finance company; real estate loans, or consumer loans.

2. *Texana Bancshares, Inc.*, Austin, Texas; to engage *de novo* through a division of the corporation, in lease brokerage activities, including serving as a broker for leases of personal property.

Board of Governors of the Federal Reserve System, October 23, 1984.

**James McAfee,**

*Associate Secretary of the Board.*

[FR Doc. 84-28342 File 10-25-84; 8:45 am]

BILLING CODE 6210-01-M

## DEPARTMENT OF HEALTH AND HUMAN SERVICES

### Office of the Secretary

### Agency Forms Submitted to the Office of Management and Budget for Clearance

Each Friday the Department of Health and Human Services (HHS) publishes a list of information collection packages it has submitted to the Office of Management and Budget (OMB) for clearance in compliance with the Paperwork Reduction Act (44 U.S.C. Chapter 35). The following are those packages submitted to OMB since the last list was published on October 19.

**Public Health Service**

*National Institutes of Health*

Subject: National Library of Medicine Reader Service Document Form—Reinstatement—(0925-0169)

Respondents: Individuals Using the NLM Reading Room

Subject: Assessment of Leukemia and Thyroid Disease in Relation to Fallout in Utah. Estimation of the Radieiodiner in Milk—New

Respondents: Individuals, Farms, Businesses, or Other for Profit; Small Businesses or Organizations

OMB Desk Officer: Fay S. Iudicello

Subject: Application for Child's Insurance Benefits—Revision—(SSA–4BK) (0960–0010)
Respondents: Individuals

Subject: Supplemental Security Income Referral Notice—Extension—No Change (SSA–L–8050–U3)-(0960–0324)
Respondents: Individuals

Subject: Transitional Employment Training Demonstration Data (conceptual clearance)—New
Respondents: Supplemental Security Income Recipients and Demonstration Program Staff
OMB Desk Officer: Robert J. Fishman

**Office of Human Development Services**

Subject: Program Performance Report for Title III of the Older Americans Act—Reinstatement (0980–0004)
Respondents: States
OMB Desk Officer: Robert J. Fishman

**Health Care Financing Administration**

Subject: Evaluation of Medicare Competition Demonstration—New Collection—HCFA–403
Respondents: Medicare Beneficiaries
OMB Desk Officer: Fay S. Iudicello

**Social Security Administration**

Subject: Reporting Events—SSI—Extension—No Change (SSA 8150) (0960–0128)
Respondents: Individuals

Subject: Health Insurance Information Request—Revision—(SSA–8019–U2) (0960–0323)
Respondents: Individuals

Subject: Application for Parent's Insurance Benefits—Revision—(SSA–7) (0960–0012)
Respondents: Individuals

**Office of Human Development Services**

Subject: Programs Performance Report for Title III of the Older Americans Act—Reinstatement (0980–0004)
Respondents: States
OMB Desk Officer: Robert J. Fishman

Copies of the above information collection clearance packages can be obtained by calling the HHS Reports Clearance Officer on 202–245–6511.

Written comments and recommendations for the proposed information collections should be sent directly to the appropriate OMB Desk Officer designated above at the following address: OMB Reports Management Branch, New Executive Office Building, Room 3208, Washington, D.C. 20503, ATTN: (name of OMB Desk Officer.)

Dated: October 22, 1984.

**Joseph F. Costa,**

*Acting Deputy Assistant Secretary for Management Analysis and Systems.*

[FR Doc. 84–28285 Filed 10–25–84; 8:45 am]

BILLING CODE 4150–04–M

## Food and Drug Administration

**[Docket No. 84F–0345]**

### Eastman Chemical Division, Eastman Kodak Co.; Filing of Food Additive Petition

**AGENCY:** Food and Drug Administration.

**ACTION:** Notice.

**SUMMARY:** The Food and Drug Administration (FDA) is announcing that Eastman Kodak Co. has filed a petition proposing that the food additive regulations be amended to provide for the safe use of poly (2-vinylpyridine-co-styrene) as a coating in the preparation of rumen-stable, abomasum-dispersible nutrient products for ruminants.

**FOR FURTHER INFORMATION CONTACT:** William D. Price, Center for Veterinary Medicine (HFV–221), Food and Drug Administration, 5600 Fishers Lane, Rockville, MD 20857, 301–443–5362.

**SUPPLEMENTARY INFORMATION:** Under the Federal Food, Drug, and Cosmetic Act (sec. 409(b)(5), 72 Stat. 1786 (21 U.S.C. 348(b)(5))), notice is given that a petition (FAP 2197) has been filed by Eastman Chemicals Division, Eastman Kodak Co., Kingsport TN 37662, proposing that the food additive regulations be amended to provide for the safe use of poly (2-vinylpyridine-co-styrene) as a coating in the preparation of rumen-stable, abomasum-dispersible nutrient products for ruminants.

The potential environmental impact of this action is being reviewed. If the agency finds that an environmental impact statement is not required and this petition results in a regulation, the notice of availability of the agency's finding of no significant impact and the evidence supporting that finding will be published with the regulation in the **Federal Register** in accordance with 21 CFR 25.40(c) (proposed December 11, 1979; 44 FR 71742).

Dated: October 19, 1984.

**Lester M. Crawford,**

*Director, Center for Veterinary Medicine.*

[FR Doc. 84–28286 Filed 10–25–84; 8:45 am]

BILLING CODE 4160–01–M

**[Docket No. 84F–0330]**

### ICI Americas, Inc.; Filing of Food Additive Petition

**AGENCY:** Food and Drug Administration.

**ACTION:** Notice.

**SUMMARY:** The Food and Drug Administration (FDA) is announcing that ICI Americas, Inc., has filed a petition proposing that the food additive regulations be amended to provide for the safe use of a copolymer of ethyl acrylate, methyl methacrylate, and methacrylamide in combination with melamine-formaldehyde resin as components of coatings for polyethylene phthalate films intended for use in contact with food.

**FOR FURTHER INFORMATION CONTACT:** James H. Maryanski, Center Food Safety and Applied Nutrition (HFF–334), Food and Drug Administration, 200 C St. SW., Washington, DC 20204, 202–472–5740.

**SUPPLEMENTARY INFORMATION:** Under the Federal Food, Drug, and Cosmetic Act (sec. 409(b)(5), 72 Stat. 1786 (21 U.S.C. 348(b)(5))), notice is given that a petition (FAP 4B3786) has been filed by ICI Americas, Inc., Wilmington, DE 19897, proposing that § 177.1630 *Polyethlene phthalate polymers* (21 CFR 177.1630) be amended to provide for the safe use of a copolymer of ethyl acrylate, methyl methacrylate, and methacrylamide in combination with melamine-formaldehyde resin for use in contact with food in coatings for polyethylene phthalate films as defined by 21 CFR 177.1630(a).

The potential environmental impact of this action is being reviewed. If the agency finds that an environmental impact statement is not required and this petition results in a regulation, the notice of availability of the agency's finding of no significant impact and the evidence supporting that finding will be published with the regulation in the **Federal Register** in accordance with 21 CFR 25.40(c) (proposed December 11, 1979; 44 FR 71742).

Dated: October 17, 1984.

**Richard J. Ronk,**

*Acting Director, Center for Food Safety and Applied Nutrition.*

[FR Doc. 84–28265 Filed 10–25–84; 8:45 am]

BILLING CODE 4160–01–M

## Public Health Service

### National Center for Health Services Research; Assessment of Medical Technology

The Public Health Service (PHS), through the Office of Health Technology Assessment (OHTA), announces that it is coordinating an assessment of what is known of the safety, clinical effectiveness, appropriateness, and use of cardiokymography. Specifically, we

are interested in the clinical utility of cardiokymography as it is used in diagnosing coronary artery disease and its sensitivity and specificity when compared with other methods of cardiac screening and diagnosis.

The PHS assessment consists of a synthesis of information obtained from appropriate organizations in the private sector and from PHS agencies and others in the Federal Government. PHS assessments are based on the most current knowledge concerning the safety and clinical effectiveness of a technology. Based on this assessment, a PHS recommendation will be formulated to assist the Health Care Financing Administration in establishing Medicare coverage policy. Any person or group wishing to provide OHTA with information relevant to this assessment should do so in writing no later than January 15, 1985, or within 90 days from the date of publication of this notice.

The information being sought is a review and assessment of past, current, and planned research related to this technology, a bibliography of published, controlled clinical trials and other well-designed clinical studies. Information related to the characterization of the patient population most likely to benefit, the clinical acceptability, and the effectiveness of this technology is also being sought.

Written material should be submitted to: National Center for Health Services Research, Office of Health Technology Assessment, Park Building, Room 3–10, 5600 Fishers Lane, Rockville, Maryland 20857.

Dated: October 19, 1984.

**Enrique D. Carter,**

*Director, Office of Health Technology Assessment, National Center for Health Services Research.*

[FR Doc. 84–28206 Filed 10–25–84; 8:45 am]

BILLING CODE 4160–17–M

## Announcement of Availability of Grants for Adolescent Family Life Demonstration Projects

**AGENCY:** Office of Adolescent Pregnancy Programs, PHS, HHS.

**ACTION:** Notice.

**SUMMARY:** This is to announce the availability of grant funds for the Adolescent Family Life Demonstration Grants Program for the states and territories listed below. These grants are for demonstration projects which test new approaches to providing care services for pregnant adolescents and adolescent parents or prevention services to encourage the postponement of premarital adolescent sexual activity, as authorized by Title XX of the Public Health Service Act (42 U.S.C. 300z, et seq.).

**ADDRESS:** Application kits may be obtained from and applications must be submitted to: Grants Management Office, Office of Adolescent Pregnancy Programs, OPA, Room 1351, HHS North Building, 330 Independence Avenue SW., Washington, D.C. 20201.

**DATE:** Applications must be postmarked or received at the above address no later than January 31, 1985.

**FOR FURTHER INFORMATION CONTACT:** Donald Underwood, Grants Management Officer, at (202) 245–0146, he is available to answer questions and provide limited technical assistance in the preparation of grant applications.

Technical Assistance Workshop: On November 19 and 20, 1984 in Denver, Colorado, a workshop will be conducted for potential applicants from the States and territories listed in the eligible applicant section which follows. At this workshop emphasis will be placed on understanding the AFL legislation, the grant process and the application. Organizations interested in attending this workshop will notify Mr. Donald Underwood of the above address of their intent to attend. Space is limited and will be allocated on a first come first served basis.

**SUPPLEMENTARY INFORMATION:** Title XX of the Public Health Service Act, 42 U.S.C. 300z, et seq. authorizes the Secretary of Health and Human Services to award grants for AFL demonstration projects (Catalog of Federal Domestic Assistance Number 13.995). This notice announces the availability of approximately $400,000 in funding for such projects, which will be made available for *care* and *prevention* projects in certain designated states and areas as set forth below. It is anticipated that 4 to 6 projects will be funded pursuant to this announcement ranging between $50,000 and $100,000. Grants may be approved for project periods of up to 5 years but funded in annual increments (budget periods).

Funding for all approved budget periods beyond the first year of the grant is contingent upon satisfactory progress of the project, adequate stewardship of Federal funds and availability of funds. A grant award may not exceed 70% of the costs of the project for the first and second years, 60% of the costs for the third year, 50% for the fourth year and 40% for the fifth year. Non-Federal contributions may be in cash or in kind, fairly evaluated, including plant, equipment, or services. Summarized below is the statutory background of the grant program and description of the procedures for applying for grants pursuant to this notice.

### Statutory Background

Title XX authorizes grants for three types of demonstration projects: (1) Projects which provide "care services" only (2) projects which provide "prevention services" only and (3) projects which provide a combination of care and prevention services.

However, in this program notice we do not propose to consider or fund any combination projects. The specific services (termed "necessary services") which may be funded under Title XX are the following:

(1) Pregnancy testing and maternity counseling;

(2) Adoption counseling and referral services which present adoption as an option for pregnant adolescents, including referral to licensed adoption agencies in the community if the eligible grant recipient is not a licensed adoption agency;

(3) Primary and preventive health services including prenatal and postnatal care;

(4) Nutrition information and counseling;

(5) Referral for screening and treatment of venereal disease;

(6) Referral to appropriate pediatric care;

(7) Educational services relating to family life and problems associated with adolescent premarital sexual relations, including:

(a) Information about adoption;

(b) Education on the responsibilities of sexuality and parenting;

(c) The development of material to support the role of parents as the provider of sex education; and

(d) Assistance to parents, schools, youth agencies, and health providers to educate adolescents and preadolescents concerning self-discipline and responsibility in human sexuality;

(8) Appropriate educational and vocational services;

(9) Referral to licensed residential care or maternity home services;

(10) Mental health services and referral to mental health services and to other appropriate physical health services;

(11) Child care sufficient to enable the adolescent parent to continue education or to enter into employment;

(12) Consumer education and homemaking;

(13) Counseling for the immediate and extended family members of the eligible person;

(14) Transportation;

(15) Outreach services to families of adolescents to discourage sexual relations among unemancipated minors; (and)

(16) Family planning services * * * (Sec. 2002(a)(4)) [1]

Under the statute, the services described in subparagraphs (1), (4), (5), (7), (8), (13), (14), and (15) above are "prevention services." (Sec. 2002(a)(8)). Grantees which provide "care services" must provide those "necessary services" which are "core services." (Sec. 2002(a)(5)). In accordance with sec. 2002(b), the regulations promulgated under Title VI of the Health Services and Centers Amendments of 1978 must presently be used to determine which of the above services are core services. Accordingly, the services described in subparagraphs (1), (2), (3), (4), (5), (6), and (7) above are core services. In addition, the referrals described by subparagraphs (8) and (10) are also core services. The tenth core service is counseling and referral for family planning services.

**Eligible Applicants**

Any public or private nonprofit organization or agency is eligible to apply for a grant if the organization or agency demonstrates "in the case of an organization which will provide care services, the capability of providing all core services in a single setting or the capability of creating a network through which all core services would be provided; or * * * in the case of an organization which will provide prevention services, the capability of providing such services" (Sec. 2002(a)(3)).

As this is a demonstration program, OAPP is interested in testing a variety of ways to deliver services and various combinations of services in order to achieve the objectives of the legislation. In order to complement existing models, the OAPP encourages the submission of applications from volunteer organizations, i.e., those organizations that provide services primarily by volunteers rather than by paid staff. The Office also welcomes applications from organizations, volunteer or otherwise, that provide alternate living arrangements, such as maternity homes, family settings for an individual or a small group, or other types of temporary shelters for pregnant adolescents and/or adolescent parents and their children. Other innovative proposals which test methods of service delivery or specialized services are also welcome.

[1] Statutory citations are to Title XX of the Public Health Service Act.

The July 21, 1981 report of the Senate Committee on Labor and Human Resources, which accompanied the Senate Bill (S. 1090) proposing the Adolescent Family Life Demonstration Grants Program, stated that one of the reasons the new legislation was necessary was to provide the states with workable models of comprehensive programs. The report further states that "without a functioning demonstration project operating within a state, public officials will not have the opportunity to examine this innovative approach to a serious problem confronting State and local governments." S. Rep. No. 97–161 at 9. Accordingly, *only entities from those states* which have never had an Adolescent Family Life Demonstration project, will be eligible to apply under this announcement. The states are Alaska, Delaware, Nevada, North Dakota, South Dakota, and Wyoming. Organizations from the U.S. Virgin Islands, American Samoa, and the Trust Territory of the Pacific Islands are also eligible to apply under this announcement. It is our intention to issue a subsequent program announcement later this fiscal year which will afford an opportunity to apply for AFL funding to States which have had an AFL project but will not have one as of October 1, 1985.

**Application Requirements**

Applications must be submitted on the forms supplied in the application kits available from the Office of Adolescent Pregnancy Programs (OAPP). Applicants are required to submit an application signed by an individual authorized to act for the applicant agency or organization and to assume for that organization the obligations imposed by the terms and conditions of the grant award. Applicants are required to submit an original application and two copies.

A copy of the legislation governing this program and proposed rules will be sent to applicants as part of the application kit package. Applicants will be expected to revise their applications to comply with any regulations issued. In the interim, applicants should use the legislation, and the proposed regulations to guide them in developing their applications. All applicants should review and must comply with the requirements for applications in 2006(a). Awards will be made only to those applicants who have met all applicable statutory requirements.

In an attempt to encourage the development and submission of applications to complement those program models currently being tested, OAPP will consider applications providing *care* or *prevention* services only, but *not a combination* of care and prevention services.

Applicants should in particular provide the following:

(1) A description of the objectives, models and strategies for delivering services and expected results. (Care programs should described services to be delivered before and after the baby's birth and should delineate the length of time after the baby's birth that clients will participate in program services.)

(2) A description of innovative approaches, as appropriate, for encouraging and supporting the involvement of families, and private and public organizations and voluntary associations in the provision of services.

(3) A description of the target groups to be served, client recruitment methods, selection criteria, case management and follow-up methods, selection criteria, case management and follow-up procedures.

(4) The numbers and types of clients expected to be served.

(5) Provision for the statutory evaluation requirements.

*Care Programs*—Under the statute the purpose of care programs is to establish innovative, comprehensive, and integrated approaches to the delivery of care services for pregnant adolescents, with primary emphasis on unmarried adolescents who are seventeen years of age or under, and for adolescent parents, which shall be based upon an assessment of existing programs and, where appropriate, upon efforts to establish better coordination, integration, and linkages among such existing programs in order to:

(A) Enable pregnant adolescents to obtain proper care and assist pregnant adolescents and adolescent parents to become productive independent contributors to family and community life; and

(B) Assist families of adolescents to understand and resolve the societal causes which are associated with adolescent pregnancy.

Within the context of providing the required core plus necessary supplemental services and developing evaluation strategies, care applicants should pay particular attention to these aspects of Title XX:

(1) The promotion of adoption as an alternative to early parenting

(2) involvement of the families of pregnant adolescents and adolescent parents, including the adolescent father; and

(3) provision of services after delivery. (This is the continuation of necessary services to clients until adolescent

parents have become or are well on their way to becoming "productive independent contributors to family and community life" and their children are developing normally physically, intellectually, and emotionally. Proposals should specify the services to be provided, the means of identifying clients' need for services, and the system for tracking clients for a period of at least two years following delivery.)

(4) core services are statutorily required. Applicants should identify the provider of each core service. If the provider is an organization other than the applicant, the applicant should include in the proposal letters and/or signed agreements indicating the other organizations' willingness to provide the services.

*Prevention Programs*—The purpose of prevention programs is to find an effective means, within the context of the family, of reaching adolescents before they become sexually active in order to maximize the guidance and support available to adolescents from parents and other family members, and to promote self-discipline and other prudent approaches to the problem of adolescent premarital sexual relations, including adolescent pregnancy.

In order to complement existing program models, OAPP will consider only those prevention programs which prepare parents to support and educate their children to postpone sexual activity. Only applications which are limited to providing services to parents regarding encouraging postponement to their own children will be considered.

All proposed prevention services must be clearly related to the objective of encouraging the postponement of premarital adolescent sexual activity. Applicants proposing activities which require a curriculum must provide a copy with the application. If the curriculum is not yet developed, a topical outline of the proposed curriculum must be provided. Applicants should explain the theoretical basis for how the proposed prevention services will result in postponement of sexual activity and include any existing research findings in support of this explanation.

**Evaluation**

Each grantee receiving funds for a services demonstration project is required to expend between one and five percent of the grant award on program evaluation. (See sec. 2006(b)). While the statute allows waiver of the 5% limit on evaluation (see Section 2006(b)(1)), waivers are rarely granted. Therefore, applicants who anticipate evaluation costs in excess of the limit should exhaust all possible alternative sources of funds before considering requesting a waiver for an evaluation amount in excess of 5%. Applicants should provide a plan for meeting the evaluation requirement, describing in detail measures of program performance, data collection methods, and a plan for analyzing the data. Applicants should provide evidence of consultation or other arrangements with a college or university located in the applicant's State. The independent entity responsible for the evaluation should also be identified. Resumes should be provided for the University consultant and the independent evaluator (if different from consultant).

**Additional Requirements**

In addition to the above, applicants for grants must meet the following requirements:

(1) Requirements for Review of an Application by the Governor

Section 2006(e) of the Public Health Service Act requires that—

"Each applicant shall provide the Governor of the State in which the applicant is located a copy of each application submitted to the Secretary for a grant for a demonstration project for services under this Title. The Governor shall submit to the applicant comments on any such application within the period of sixty days beginning on the day when the Governor receives such copy. The applicant shall include the comments of the Governor with such application."

An applicant may comply with this requirement by submitting a copy of the application to the Governor of the State in which the application is located at the same time the application is submitted to OAPP. To inform the Governor's office of the reason for the submission, a copy of this notice should be attached to the application. The Governor has sixty days in which to provide comments to the applicant.

The applicant must provide a copy of the comments or verification that there were no comments to the above address by April 30, 1985.

*(2) Review Under Executive Order 12372*

Applications under this announcement are subject to the review requirements of Executive Order 12372, State Review of Applications for Federal Financial Assistance, as implemented by 45CFR100. As soon as possible, applicants should discuss their project/s with the State Single Point of Contact (SPOC) for each State in the area to be served. The application kit contains the currently available listing of the SPOCs which have elected to be informed of the submission of applications. For those States not represented on the listing, further inquiries should be made by the applicant regarding the submission to the relevant SPOC. The SPOCs comment/s should be forwarded to the Grants Management Office, Office of Population Affairs, Room 1351, HHS North Building, 330 Independence Avenue, S.W., Washington, D.C. 20201. Such comments must be received by the Office of Population Affairs by April 30, 1985 to be considered. In the event that an application is submitted to the Office of Population Affairs without notification to the SPOC, the SPOC will be notified of the submission.

*(3) Health Systems Agency (HSA) Review.*

In order to comply with the HSA review requirements under section 1513(e) of the Public Health Service Act, 42 U.S.C. 3001–2(e), as amended, applicants must contact the HSA responsible for the area to be served by the proposed project to determine whether or not the HSA desires to review the application. If so, a copy of the application must be submitted to the HSA for review no later than January 31, 1985. Applicants are advised to contact the local HSA as soon as a decision is made to apply for a grant for detailed information on meeting this review requirement.

**Application Consideration and Assessment**

Applications which are judged to be late or which do not conform to the requirements of this program announcement will not be accepted for review. Applicants will be so notified, and the applications will be returned.

All other applications will be subjected to a competitive review and assessment. The results of this review will assist the Director of the Office of Adolescent Pregnancy Programs in considering competing applications and in making the final funding decisions.

Eligible competing grant applications will be reviewed and assessed against the following criteria:

1. The applicant's provision for the requirements set forth in sec. 2006(a) of Title XX of the Public Health Service Act.

2. The capacity of the proposed applicant organization and staff to provide the appropriate services and to evaluate the results.

3. The applicant's presentation of the project's objectives, the methods for achieving project objectives, the

workplan and the results or benefits expected.

4. The applicant's documentation of the innovativeness of the program approach, its worth for testing and replication, and its suitability to measurement and evaluation.

5. The estimated cost of the project to the government is reasonable considering the anticipated results.

6. The applicant's detailed evaluation plan indicates an understanding of program evaluation methods and reflects a practical, technically sound approach to assessing the project's achievement of program objectives. A workplan should be included to indicate the extent and nature of the involvement of a local State college or university in this effort.

In making grant award decisions the Director of OAPP will take into account the extent to which grants approved for funding will provide an appropriate distribution of resources throughout the country taking into consideration such things as the following factors:

1. The priorities in sec. 2005(a) of Title XX of the Public Health Service Act.

2. The geographic area to be served.

3. The community commitment to and involvement in the planning and implementation of the demonstration project.

4. The nature of the organization applying.

5. The population to be served.

6. The organizational models for delivery of service.

7. The usefulness for policy makers and service providers of the proposed project and its potential for complementing existing AFL demonstration models.

When final funding decisions have been made, all applicants will be notified by letter of the outcome of their applications. The official document notifying an applicant that a project application has been approved for funding is the Notice of Grant Award, which specifies to the grantee the amount of money awarded, the purpose of the grant, the terms and conditions of the grant award, the budget period for which support is being given, and the amount of funding to be contributed by the grantee to project costs.

Dated: October 23, 1984.

Majority E. Mecklenburg,

*Deputy Assistant Secretary for Population Affairs.*

[FR Doc. 84-28350 Filed 10-25-84; 8:45 am]

BILLING CODE 4160-17-M

## National Center for Health Services Research; Assessment of Medical Technology

The Public Health Service (PHS), through the Office fo Health Technology Assessment (OHTA), announces that it is coordinating an assessmeent of what is known of the safety, clinical effectiveness, and appropriateness of 24 hour ambulatory esophageal pH monitoring, and selective criteria for patients who might benefit from the use of this diagnostic procedure.

The PHS assessment consists of a synthesis of information obtained from appropriate organizations in the private sector and from PHS agencies and others in the Federal Government. PHS assessments are based on the most current knowledge concerning the safety and clinical effectiveness of a technology. Based on this assessment, a PHS report will be formulated to assist the Health Care Financing Administration (HCFA) in establishing Medicare coverage policy. Any person or group wishing to provide OHTA with information relevant to this assessment should do so in writing no later than January 30, 1985, or within 90 days from the date of publication of this notice.

The information being sought is a review and assessment of past, current, and planned research related to this technology, a bibliography of published, controlled clinical trials and other well-designed clinical studies. Information related to the characterization of the patient population most likely to benefit from, the clinical acceptability, and the effectiveness of this technology is also being sought.

Written material should be submitted to: National Center for Health Services Research, Office of Health Technology Assessment, Park Building, Room 3-10, 5600 Fishers Lane, Rockville, Maryland 20857.

Date: October 17, 1984.

Harry Handelsman, D.O.,

*Acting Director, Office of Health Technology Assessment, National Center for Health Services Research.*

[FR Doc. 84-28338 Filed 10-25-84; 8:45 am]

BILLING CODE 4160-17-M

## National Center for Health Services Research; Assessment of Medical Technology

The Public Health Service (PHS), through the Office of Health Technology Assessment (OHTA), announces that it is coordinating an assessment of what is known of the safety, clinical effectiveness, appropriateness, and use of bone mineral density studies. Included in the assessment are bone biopsy, single photon absorptiometry, dual photon absorptiometry, radiographic absorptiometry and computed tomography, as well as any other established study used to determine bone mineral content. We are interested in the clinical effectiveness and appropriate use of these devices in the determination of bone mineral content for patients with end-stage renal disease and in other conditions where disease affecting bone mineralization is suspected (i.e., osteoporosis, osteomalacia, metabolic disorders of bone, etc). We also wish to determine what constitutes a reasonable and necessary frequency for performing these procedures in patients with end-stage renal disease and other conditions. Specifically, we are interested in data that address the efficacy of these technologies as methods of: (1) Diagnosing metabolic bone diseases, (2) determining therapeutic regimens, (3) assessing therapeutic responses, (4) discriminating between diseased and normal populations (screening), (5) identifying individuals at risk (i.e., postmenopausal osteoporosis and likelihood of developing fractures), (6) determining the severity of disease.

This assessment seeks to determine where bone mineral content evaluation fits into the overall scheme of bone disease prevention, diagnosis and treatment. Moreover, it seeks to identify specific and unique applications of any of the methods used to measure bone mineral content in order to determine the optimal uses and ultimate benefit of this technology.

The PHS assessment consists of a synthesis of information obtained from appropriate organizations in the private sector and from PHS agencies and others in the Federal Government. PHS assessments are based on the most current knowledge concerning the safety and clinical effectiveness of a technology. Based on this assessment, a PHS recommendation will be formulated to assist the Health Care Financing Administration in establishing Medicare coverage policy. Any person or group wishing to provide OHTA with information relevant to this assessment should do so in writing no later than January 15, 1985, or within 90 days from the date of publication of this notice.

The information being sought is a review and assessment of past, current, and planned research related to this technology, a bibliography of published, controlled clinical trials and other well-designed clinical studies. Information related to the characterization of the

patient population most likely to benefit, the clinical acceptability, and the effectiveness of this technology is also being sought.

Written material should be submitted to: National Center for Health Services Research, Office of Health Technology Assessment, Park Building, Room 3–10, 5600 Fishers Lane, Rockville, Maryland 20857.

Dated: October 17, 1984.

**Harry Handelsman, D.O.,**

*Acting Director, Office of Health Technology Assessment, National Center for Health Services Research.*

[FR Doc. 84–28387 Filed 10–25–84; 8:45 am]

BILLING CODE 4160–17–M

## DEPARTMENT OF THE INTERIOR

### Bureau of Land Management

[INT DRMP/EIS 84–56]

### Availability of the Draft Resource Management Plan Environmental Impact Statement for the Walker Resource Area, NV

**AGENCY:** Bureau of Land Management, Interior.

**ACTION:** Notice of Availability of and Public Hearings on the Draft Resource Management Plan/Environmental Impact Statement for the Walker Resource Area, Carson City District, Nevada.

**SUMMARY:** Pursuant to section 102(2)(c) of the National Environmental Policy Act of 1969 and section 202 of the Federal Land Policy and Management Act of 1976, the BLM, Carson City District has prepared a combined Resource Management Plan/Environmental Impact Statement for the Walker Resource Area, Carson City District, Nevada.

**SUPPLEMENTARY INFORMATION:** The Walker Resource Management Plan/Environmental Impact Statement is a comprehensive land use planning document which establishes management actions and objectives for resource condition and use levels, the standards for monitoring and evaluating the plan's effectiveness, and the need for more detailed management plan(s) and support actions. It also is an environmental impact statement which analyzes the effects of implementing a multiple use resource management plan on 1.9 million acres of public land in the southern third of the Carson City District in Nevada. Four alternatives are being considered along with the Preferred Alternative. The Preferred Alternative includes a proposal to designate 16,000 acres of the Stewart Valley Fossil Area an Area of Critical Environmental Concern, withdrawing 1280 acres of that area from mineral entry, and limiting off-road vehicle use in the 16,000 acres to existing roads, trails, and washes. The Preferred Alternative proposes that none of the 92,995 acres in two wilderness study areas is suitable for wilderness designation. The affected environment is discussed, and the environmental consequences occurring from each alternative are documented.

**FOR FURTHER INFORMATION CONTACT:** Tom Owen, District Manager, Attn: RMP/EIS Team Leader, Carson City District Office, 1050 E. William St., Ste. 335, Carson City, NV 89701 (702) 882–1631.

Copies of the draft document are available for review at the following locations:

Office of Public Affairs, Bureau of Land Management, 18th and C Streets, Washington, D.C. 20240
Bureau of Land Management, Nevada State Office, 300 Booth Street, P.O. Box 12000, Reno, Nevada 89520, (702) 784–5448
Bureau of Land Management, Las Vegas District Office, 4765 West Vegas Drive, Las Vegas, Nevada 89102, (702) 385–6403
Bureau of Land Management, Winnemucca District Office, 705 East 4th Street, Winnemucca, Nevada 89445, (702) 623–3676
Bureau of Land Management, Elko District Office, 2002 Idaho Street, Elko, Nevada 89801
Bureau of Land Management, Ely District Office, Star Route 5, Box 1, Ely, Nevada 89301, (702) 289–4965
Bureau of Land Management, Carson City District Office, 1050 E. William Street, Carson City, Nevada 89701, (702) 635–5181
Bureau of Land Management, Battle Mountain District Office, North 2nd and Scott Streets, Battle Mountain, Nevada 89820, (702) 635–5181

Also, copies are available for review at the following public libraries:

Carson City Library, 900 N. Roop St., Carson City, Nevada 89701
Churchill County Library, 553 South Maine Street, Fallon, Nevada 89406
Government Publications Dept., University of Nevada, Reno, Reno Library, Reno, Nevada 89557
Nevada State Library, Library Building, Carson City, Nevada 89710
Lyon County Library, 20 Nevin Way, Yerington, Nevada 89447
University of Nevada, Reno, Getchell Library, Reno, Nevada 89507

**SUPPLEMENTARY INFORMATION:**

University of Nevada, Las Vegas, James R. Dickinson Library, 4505 Maryland Parkway, Las Vegas, Nevada 89154
Mineral County Library, 1st and D Streets, Hawthorne, Nevada 89415
Nye County Library, Tonopah, Nevada 89049
Washoe County Library, 1301 South Center Street, Reno, Nevada 89505

A copy of the Draft RMP/EIS or summary will be sent to all individuals, agencies, and groups who have expressed interest in the Walker Resource Area planning process, and a limited number of copies are available upon request to the District Manager at the above address.

**Dates:**

Written comments concerning issues pertinent to the Walker Resource Area RMP/EIS will be accepted until January 25, 1985. Public hearings have been scheduled for December 4, 1984, 7:30 p.m. at the El Dorado Hotel, 345 No. Virginia in Reno, Nevada, and December 5, 1984, 7:30 p.m. at El Capitan Club in Hawthorne, Nevada. Testimony concerning the issues will be accepted at these hearings. Interested individuals, representatives of organizations, and public officials wishing to testify are requested to contact the District Manager for advance registration by 4:15 p.m., November 30, 1984. Oral testimony will be limited to 10 minutes.

Dated: October 15, 1984.

**Edward F. Spang,**

*State Director, Nevada.*

[FR Doc. 84–27840 Filed 10–25–84; 8:45 am]

BILLING CODE 4310–HC–M

### Availability of the Public Summary and Rangeland Program Summary for the South Sierra Foothills Planning Area, Bakersfield District, CA

**AGENCY:** Bureau of Land Management, Interior.

**ACTION:** Notice of availability.

**SUMMARY:** Notice is hereby given of the availability of the Public Summary/Rangeland Program Summary for 463,000 acres of BLM-administered public land within the South Sierra Foothills Planning Area, encompassing portions of Kern and Tulare counties in California.

**SUPPLEMENTARY INFORMATION:** The Public Summary includes the Management Framework Plan (MFP) land use decisions which will guide future management actions for public land in the South Sierra Foothills Planning Area of the Caliente Resource Area. Decisions presented in the document are the culmination of several years of intensive resource inventory, evaluation, public involvement, planning and Environmental Impact Statement efforts.

The rangeland management decisions covered in the land use plan include forage allocation and season of use by allotment for livestock. These decisions,

including forage utilization standards and proposed grazing systems, are described in the Rangeland Program Summary (RPS) attached as an appendix to the document.

The management decisions in the South Sierra Foothills land use plan are now in effect. The plan will be monitored on a regular basis to assess its effectiveness and continued applicability. Any amendment to the plan that may occur will be based on the information obtained from monitoring the plan. The public will be given many opportunities for participation in any significant amendment.

Copies of the Public Summary/RPS are available for review at the following BLM offices and libraries:

U.S. Bureau of Land Management, Caliente Resource Area, 520 Butte St., Bakersfield, CA 93305, (805) 861–4236
U.S. Bureau of Land Management, Bakersfield District Office, 800 Truxtun Ave., Rm. 311, Bakersfield, CA 93301, (805) 861–4191
Beale Memorial Library, 1315 Truxtun Ave., Bakersfield, CA 93301, (805) 861–2135
Visalia County Library, 200 W. Oak St., Visalia, CA 93291, (209) 733–8440

**DATES:** Individuals or other interested groups not scheduled to receive allotment specific grazing decisions may protest specific portions of the RPS to the Caliente Resource Area Manager within 15 days of publication of this notice (November 12, 1984). If no protest is received within this time frame, the proposed decision will become final without further notice. If a protest is received, the points of the protest will be considered by the Area Manager and a final decision issued.

A period of 30 days after receipt of the final decision is provided for filing an appeal with the Area Manager for the purpose of a hearing before an administrative law judge.

**FOR FURTHER INFORMATION CONTACT:** Glenn Carpenter, Area Manager, Bureau of Land Management, Caliente Resource Area, 520 Butte Street, Bakersfield, California, 93305, (805) 861–4236.

Dated: October 10, 1984.

**Robert D. Rheiner, Jr.,**

*District Manager.*

[FR Doc. 84-27528 Filed 10-25-84; 8:45 am]

**BILLING CODE 4310-40-M**

---

## Platte River Resource Area, Casper District, WY; Availability of Final Resource Management Plan (RMP) and Environmental Impact Statement (EIS)

**AGENCY:** Bureau of Land Management, Interior.

**ACTION:** Public notice that the proposed Resource Management Plan and final Environmental Impact Statement for the Platte River Resource Area, Casper District, Wyoming is available for public review.

**SUMMARY:** The proposed Resource Management Plan (RMP) and final Environmental Impact Statement (EIS) presents a management plan and the consequences of implementing the plan for the 1.4 million acres of public land in the Platte River Resource Area (PRRA).

### Location of Documents

Source material for the proposed RMP and the final EIS is available for public review at the PRRA office at the address noted below.

Additional information or requests to be placed on the mailing list should be addressed to: Jim Melton, Area Manager, Platte River Resource Area, Bureau of Land Management, 111 South Wolcott, Casper, Wyoming 82601. Phone: (307) 261–5191.

### Public Participation

There will be a 30-day protest period on the proposed RMP and final EIS. Any person who participated in the planning process and has an interest which is or may be adversely affected by the approval of the proposed plan may protest the approval. A protest may raise only those issues which were submitted to the record during the planning process.

Protests should be sent to the Director (202), Bureau of Land Management, 1800 C Street, NW., Washington, D.C., 20240 before December 14, 1984 (end of the 30-day protest period). A protest must contain:

1. The name, mailing address, telephone number and interest of the person filing the protest.
2. A statement of the issue or issues being protested.
3. A statement of the part or parts of the plan being protested.
4. A copy of all documents addressing the issue or issues that were submitted during the planning process by the protesting party, or an indication of the date the issue or issues were discussed for the record.
5. A concise statement explaining why the proposed management plan is believed to be wrong.

At the end of the 30-day protest period noted above, the proposed management plan, excluding any portion under protest, will be finalized in a Record of Decision. Approval will be withheld on any portion of the plan under protest until final action has been completed.

**SUPPLEMENTARY INFORMATION:** The Resource Management Plan will guide management actions on the public lands within the Platte River Resource Area which includes Natrona, Converse, Platte, and Goshen counties. Within these four counties, BLM manages 16% of the surface, approximately 1.4 million acres, and about 55% of the mineral estate, approximately 4.7 million acres.

The major purpose in preparing the Platte RMP was to provide a comprehensive framework for managing and allocating resources in the PRRA for the next ten years or more. The planning process included the identification of issues, the development of planning criteria, inventory and data collection, an analysis of the management situation, the formulation of alternatives, and an analysis of the alternatives.

The four alternatives in the draft EIS included the continuation of present management, low level, moderate level, and high level management. The consequences of implementing each alternative was presented in the draft environmental statement. A preferred management plan was presented in the draft that best addressed each of the issues.

The proposed plan presented in the final environmental statement is essentially the same as the preferred alternative in the draft. In some cases, it has been revised as a result of public comments received during the 90-day comment period. The consequences of implementing the proposed management plan is presented in the final environmental statement.

The Platte RMP/EIS was prepared by an interdisciplinary team of specialists from the PRRA and the Casper District Office. Disciplines included cultural, energy and minerals, fire, forestry, grazing (range), lands, recreation, soil water, air, wildlife, and socioeconomics. Reviews for consistency were provided by both the district office and state office staffs. Consultation, coordination, and public involvement have occurred throughout the process through public meetings, informal meetings, individual contracts, newsletters, and **Federal Register** notices.

The 13 major issues addressed in the RMP/EIS are the protection of cultural resources; sand and gravel extraction, fire management, timber harvest and pine beetle control; grazing management; disposal, acquisition and leasing; withdrawals; corridors; access; recreation management; watershed protection; wildlife habitat management; and areas with special designations.

**Special Management**

The proposed plan includes two areas of critical environmental concern (ACEC). These are described below.

**Salt Creek ACEC**

The Salt Creek drainage to its confluence with the South Fork of the Powder River is managed as an ACEC. Salt Creek and portions of Teapot Creek that have been identified as sensitive drainages are included in the Salt Creek ACEC. Implementation of the ACEC plan will be in the following priority order: Salt Creek oil field, Smokey Gap oil field, and East Teapot oil field.

The ACEC contains about 75,000 acres located in Converse, Natrona, and Goshen counties.

The Salt Creek ACEC plan will provide for inventory and evaluation of historic oil and gas sites, structures and townsites that may be eligible for nomination to the National Register. The Salt Creek oil field will be designated as a historic district if applicable. In no case will the designation interfere with oil and gas development or production in producing fields within this area.

**Jackson Canyon ACEC**

The Jackson Canyon ACEC contains 3,600 federal surface acres and 11,150 federal mineral acres, for a total of 13,760 acres in the ACEC. Jackson Canyon provides an essential roosting area for approximately 60 bald eagles from December through March.

Management in the ACEC would be directed at controlling pine beetle infestations through a more active forest management program and designating bald eagle roosts for priority fire suppression.

A mineral withdrawal from the operation of the 1872 Mining Law will be recommended on 3,600 acres of federal mineral estate in bald eagle roost areas. No rights-of-way will be permitted in this ACEC. ORV use will be allowed only on designated roads and only from April 1 through October 31. There will be no increase or improvement in roads or legal access. The ACEC will be managed in accordance with the ACEC Wildlife Habitat Management plan.

**James Monroe,**

*District Manager.*

[FR Doc. 84-27896 Filed 10-25-84; 8:45 am]

BILLING CODE 4310-22-M

## Grand Junction Resource Management Plan

**AGENCY:** Bureau of Land Management, Interior.

**ACTION:** Proposed changes to planning criteria.

**SUMMARY:** Pursuant to 43 CFR 1610.2 a 30 day public comment period shall be provided for any proposed changes to planning criteria.

**ADDRESS:** Copies of proposed planning criteria changes are available upon request from: Grand Junction Resource Area Office, Bureau of Land Management, 764 Horizon Dr., Grand Junction, CO 81501.

**SUPPLEMENTARY INFORMATION:** The Grand Junction Resource Area is proposing to change the planning criteria developed for the Grand Junction Resource Management Plan. Planning criteria were approved following public review in March 1984. According to the Bureau's planning regulations, changes in planning criteria must be made available for public review and comment 30 days prior to approval. The changes are being proposed because a few of the planning criteria are not being followed or do not adequately address the issues. Proposed changes to the planning criteria for coal, land tenure adjustment and public utilities are explained in detail in a fact sheet available upon request.

Comments on the proposed changes will be accepted until December 7, 1984.

**FOR FURTHER INFORMATION CONTACT:** Forest W. Littrell, Area Manager, Bureau of Land Management, Grand Junction Resource Area Office, 764 Horizon Dr., Grand Junction, Colorado 81501. (303) 243-6552.

**Wright Sheldon,**

*District Manager, Grand Junction District.*

[FR Doc. 84-28323 Filed 10-25-84; 8:45 am]

BILLING CODE 4310-JB-M

[ES-34162, Group 6]

## Filing of Plat of Dependent Resurvey and Subdivision; Iowa

October 22, 1984.

1. The plat of the dependent resurvey of a portion of the subdivisional lines and the subdivision of sections 13 and 25, T. 83 N., R. 16 W., Fifth Principal Meridian, Iowa, will be officially filed in the Eastern States Offices, Alexandria, Virginia at 7:30 a.m., on December 6, 1984.

2. The dependent resurvey was made at the request of the Bureau of Indian Affairs.

3. All inquires or protests concerning the technical aspects of the dependent resurvey must be sent to the Deputy State Director for Cadastral Survey, Eastern States Offices, Bureau of Land Management, 350 South Pickett Street, Alexandria, Virginia 22304, prior to 7:30 a.m., December 6, 1984.

4. Copies of the plat will be made available upon request and prepayment of the reproduction fee of $4.00 per copy.

**Lane J. Bouman,**

*Deputy State Director for Cadastral Survey.*

[FR Doc. 84-28340 Filed 10-25-84; 8:45 am]

BILLING CODE 4310-GJ-M

[ES-34163, Group 5]

## Filing of Plat of Dependent Resurvey and Subdivision; Iowa

October 22, 1984.

1. The plat of the dependent resurvey of a portion of the south the west boundaries, a portion of the subdivisional lines, and the subdivision of sections 19, 20, 29, 31, and 32, T. 83 N., R. 15 W., Fifth Principal Meridian, Iowa, will be officially filed in the Eastern States Office, Alexandria, Virginia at 7:30 a.m., on December 6, 1984.

2. The dependent resurvey was made at the request of the Bureau of Indian Affairs.

3. All inquiries or protests concerning the technical aspects of the dependent resurvey must be sent to the Deputy State Director for Cadastral Survey, Eastern States, Office, Bureau of Land Management, 350 South Pickett Street, Alexandria, Virginia 22304, prior to 7:30 a.m., December 6, 1984.

4. Copies of the plat will be made available upon request and prepayment of the reproduction fee of $4.00 per copy.

**Lane J. Bouman,**

*Deputy State Director for Cadastral Survey.*

[FR Doc. 84-28339 Filed 10-25-84; 8:45 am]

BILLING CODE 4310-GJ-M

## Fish and Wildlife Service

## Endangered Species; Receipt of Application for Permit; University of Michigan; et al.

The following applicants have applied for permits to conduct certain activities with endangered species. This notice is provided pursuant to section 10(c) of the Endangered Species Act of 1973, as amended (16 U.S.C 1531, *et seq.*):

Applicant: University of Michigan-Museum of Zoology, Ann Arbor, MI; PRT-684673.

The applicant requests a permit to import a salvaged dugong (*Dugong dugon*) skull from Egypt for scientific research.

Applicant: Herp Osteo Specimens, Canoga Park, CA; PRT-684371.

The applicant requests a permit to export to Japan one preserved salt water

crocodile (*Crocodylus porosus*) for scientific research purposes.

Applicant: San Francisco Zoo, San Francisco, CA; PRT–684747.

The applicant requests a permit to import six captive Francois' langurs (*Presbytis francoisi*) from the Shanghai Zoo, China, for enhancement of propagation.

Applicant: Zoological Society of Cincinatti, Cincinatti, OH; PRT–684546.

The applicant requests a permit to import one captive-born male black-footed cat (*Felis nigripes*) from the Rotterdam Zoo, the Netherlands, for enhancement, of propagation.

Applicant: Gulf Islands National Seashore, Ocean Springs, MS: PRT–684746.

The applicant request a permit to take (capture, band and release) brown pelicans (*Pelicanus occidentalis*) for scientific research.

Documents and other information submitted with these applications are available to the public during normal business hours (7:45 am to 4:15 pm) Room 601, 1000 North Glebe Road, Arlington, Virginia, 22201.

Interested persons may comment on any of these applications within 30 days of the date of this publication by submitting written views, arguments, or data to the Director at the above address. Please refer to the appropriate PRT/APP number when submitting comments.

Dated. October 23, 1984

**R.K Robinson,**

*Chief, Branch of Permits Federal Wildlife Permit Office.*

[FR Doc. 84–28302 Filed 10–25–84; 8:45 am]

BILLING CODE 4310–55–M

## National Park Service

### Pictured Rocks National Lakeshore, Michigan; Public Review, Meetings, and Comment Period for Draft Land Protection Plan

**AGENCY:** National Park Service, Interior.

**ACTION:** Notice.

**SUMMARY:** A draft Land Protection Plan has been completed for Pictured Rocks National Lakeshore in response to the Department of the Interior's policy for the Federal portion of the Land and Water Conservation Fund (47 FR 19784, May 7, 1982).

This notice sets forth the public comment period and the public meeting schedule for review of this plan. The public is invited to participate in the review of this draft document.

**DATE:** A 30-day public comment period will begin on October 27, 1984, and will conclude on November 25, 1984. Three public meetings will be held during the comment period. These meetings will be conducted as open houses at the following locations:

*November 5:* Recreation Center, Grand Marais, Michigan—7:30 p.m.

*November 7:* Ziegert's Restaurant, Grand Island Room, Munising, Michigan—7:30 p.m.

*November 13:* Sheraton Oaks Hotel, Amphitheater, Novi, Michigan—7:30 p.m.

**ADDRESS:** Written comments should be addressed to the Superintendent, Pictured Rocks National Lakeshore, P.O. Box 40, Sand Point, Munising, MI 49862. Copies of the plan may also be obtained by visiting or writing this office.

**FOR FURTHER INFORMATION CONTACT:** Superintendent Grant Petersen at the lakeshore headquarters address or call (906) 387–2607.

**SUPPLEMENTARY INFORMATION:** National Park Service staff will be available at the open house sessions to answer questions concerning the plan. Detailed segment maps will be available to indicate property tracts covered by the plan.

Citizens may also address questions and view segement maps and provide comments during the public comment period at the lakeshore headquarters, Sand Point Road, Munising, Michigan, 8:00 a.m. to 4:30 p.m. Monday through Friday.

Copies of the plan are being mailed to landowners of record within the boundary of the lakeshore, as well as agencies, organizations and individuals with an expressed interest in management of the National Lakeshore.

**Charles H. Odegaard,**

*Regional Director, Midwest Region, National Park Service.*

[FR Doc. 84–28338 Filed 10–25–84; 8:45 am]

BILLING CODE 4310–70–M

### Gates of the Artic National Park Subsistence Resource Commission; Meeting

**SUMMARY:** The Superintendent of the Gates of the Arctic National Park and Preserve, National Park Service, announces forthcoming meetings of the Gates of the Arctic National Park Subsistence Resource Commission:

**DATES:** The meeting will be held at the following locations and times:

1. November 16, 1984, in the Community Hall in Allakaket, Alaska. The meeting will begin at 10:00 a.m.
2. November 17, 1984, in the Community Hall in Evansville, Alaska. The meeting will begin at 10:00 a.m.
3. November 18, 1984, in (To be determined) Wiseman, Alaska. The meeting will begin at 10:00 a.m.

Agendas: The following agenda items will be undertaken:

*Allakaket*

1. Introduction of Commission members and responsibilities. Approval of minutes and agenda.
2. Commission procedures.
3. Presentation by Citizens' Advisory Commission on Federal Areas.
4. State role in fish and wildlife management within the park.
5. Identification of subsistence hunting program components.
6. Discussion of traditional subsistence use areas.
7. New business.
8. Public testimony.

*Evansville*

1. Introduction of commission members and responsibilities.
2. Gates of the Arctic General Management Plan.
3. NPS cabin policies and decision.
4. Timetable for submission of recommendations to the Secretary of the Interior.
5. Commissioner/Committee assignments.
6. Traditional subsistence use areas.
7. Public testimony.

*Wiseman*

1. Introduction of Commission members and responsibilities.
2. Review of Allakaket and Evansville meetings.
3. Unfinished business.
4. Traditional subsistence use areas.
5. Public testimony.

Written comments and recommendations received prior to November 7, 1984, will be considered at the meetings.

All comments should be addressed to: Chairman, Gates of the Arctic National Park, Subsistence Resource Commission, c/o Box 74680, Fairbanks, Alaska 99707.

**FOR FURTHER INFORMATION CONTACT:** Richard G. Ring, Superintendent, Gates of the Arctic National Park and Preserve, P.O. Box 74680, Fairbanks, Alaska 99707, Phone (907) 456–0351.

**SUPPLEMENTARY INFORMATION:** The Denali National Park Subsistence Resource Commission is authorized under Title VIII, Section 808, of the Alaska National Interest Lands Conservation Act Pub. L. 96–487.

Dated: October 16, 1984.

**Robert L. Peterson,**

*Regional Director, Alaska Region.*

[FR Doc. 84-28337 Filed 10-25-84; 8:45 am]

BILLING CODE 4310-70-M

## INTERNATIONAL TRADE COMMISSION

**[Investigation No. 731-TA-147 (Final)]**

### Cut-to-Length Carbon Steel Plate From the Federal Republic of Germany

**AGENCY:** United States International Trade Commission.

**ACTION:** Institution of a final antidumping investigation and scheduling of a hearing to be held in connection with the investigation.

**SUMMARY:** As a result of an affirmative preliminary determination by the U.S. Department of Commerce that there is a reasonable basis to believe or suspect that imports of carbon steel plate, not in coils, from the Federal Republic of Germany (West Germany), provided for in item 607.66 of the Tariff Schedules of the United States, are being, or are likely to be, sold in the United States at less than fair value (LTFV) within the meaning of section 731 of the Tariff Act of 1930 (19 U.S.C. 1673), the United States International Trade Commission hereby gives notice of the institution of investigation No. 731-TA-147 (Final) under section 735(b) of the act (19 U.S.C. 1673d(b)) to determine wether an industry in the United States is materially injured, or is threatened with material injury, or the establishment of an industry in the United States is materially retarded, by reason of imports of such merchandise. Unless the investigation is extended, the Department of Commerce will make its final dumping determination in the case on or before December 14, 1984, and the Commission will make the final injury determination by February 5, 1985 (19 CFR 207.25).

**EFFECTIVE DATE:** October 9, 1984.

**FOR FURTHER INFORMATION CONTACT:** Lawrence Rausch, (202-523-0286), Office of Investigations, U.S. International Trade Commission.

**SUPPLEMENTARY INFORMATION:**

#### Background

On November 14, 1983, the Commission notified the Department of Commerce that, on the basis of the information developed during the course of its preliminary investigation, there was a reasonable indication that an industry in the United States was materially injured by reason of imports of certain hot-rolled carbon steel plate from West Germany. The preliminary investigation was instituted in response to a petition filed on September 29, 1983, by counsel on behalf of the Gilmore Steel Corp., Portland, OR.

#### Participation in the investigation

Persons wishing to participate in this investigation as parties must file an entry of appearance with the Secretary to the Commission, as provided in § 201.11 of the Commission's Rules of Practice and Procedure (19 CFR 201.11), not later than 21 days after publication of this notice in the **Federal Register.** Any entry of appearance filed after this date will be referred to the Chairwoman, who shall determine whether to accept the late entry for good cause shown by the person desiring to file the entry.

Upon the expiration of the period for filing entries of appearance, the Secretary shall prepare a service list containing the names and addresses of all persons, or their representatives, who are parties to the investigation, pursuant to § 201.11(d) of the Commission's rules (19 CFR 201.11(d)). Each document filed by a party to this investigation must be served on all other parties to the investigation (as identified by the service list), and a certficate of service must accompany the document. The Secretary will not accept a document for filing without a certificate of service (19 CFR 201.16(c)).

#### Staff report

A public version of the staff report containing preliminary findings of fact in this investigatin will be placed in the public record on December 4, 1984, pursuant to § 207.21 of the Commission's rules (19 CFR 207.21).

#### Hearing

The Commission will hold a hearing in connection with this investigation beginning at 10:00 a.m., on December 19, 1984, at the U.S. International Trade Commission Building, 701 E Street NW., Washington, D.C. 10436. Requests to appear at the hearing should be filed in writing with the Secretary to the Commission not later than the close of business (5:15 p.m.) on November 30, 1984. All persons desiring to appear at the hearing and make oral presentations should file prehearing briefs and attend a prehearing conference to be held at 10:00 a.m., on December 10, 1984, in room 117 of the U.S. International Trade Commission Building. The deadline for filing prehearing briefs is December 14, 1984.

Testimony at the public hearing is governed by § 207.23 of the Commission's rules (19 CFR 207.23). This rule requires that testimony be limited to a nonconfidential summary and analysis of material contained in prehearing briefs and to information not available at the time the prehearing brief was submitted. All legal arguments, economic analyses, and factual materials relevant to the public hearing should be included in prehearing briefs in accordance with section 207.22 (19 CFR 207.22). Posthearing briefs must conform with the provisions of section 207.24 (19 CFR 207.24) and must be submitted not later than the close of business on December 21, 1984.

#### Written submissions

As mentioned, parties to this investigation may file prehearing and posthearing briefs by the dates shown above. In addition, any person who has not entered an appearance as a party to the investigation may submit a written statement of information pertinent to the subject of the investigation on or before December 21, 1984. A signed original and fourteen (14) true copies of each submission must be filed with the Secretary to the Commission in accordance with § 201.8 of the Commission's rules (19 CFR 201.8). All written submissions except for confidential business data will be available for public inspection during regular business hours (8:45 a.m. to 5:15 p.m.) in the Office of the Secretary to the Commission.

Any business information for which confidential treatment is desired shall be submitted separately. The envelope and all pages of such submissions must be clearly labeled "Confidential Business Information." Confidential submissions and requests for confidential treatment must conform with the requirements of § 201.6 of the Commission's rules (19 CFR 201.6).

For further information concerning the conduct of the investigation, hearing procedures, and rules of general application, consult the Commission's Rules of Practice and procedure, Part 207, Subparts A and C (19 CFR Part 207), and Part 201, Subparts A through E (19 CFR Part 201).

This notice is published pursuant to § 207.20 of the Commission's rules (19 CFR 207.20).

By order of the Commission.

Issued: October 22, 1984.

**Kenneth R. Mason,**

*Secretary.*

[FR Doc. 84-28330 Filed 10-25-84; 8:45 am]

BILLING CODE 7020-02-M

## INTERSTATE COMMERCE COMMISSION

### Motor Carriers; Intent To Engage In Compensated Intercorporate Hauling Operations

This is to provide notice as required by 49 U.S.C. 10524(b)(1) that the named corporations intend to provide or use compensated intercorporate hauling operations as authorized in 49 U.S.C. 10524(b).

1. Parent corporation and address of principal office: Coachmen Industries, Inc., Coachmen Drive, Middlebury, Indiana 46540.

2. Wholly owned subsidiaries which will participate in the operations, and State of incorporation:

(A) Coachmen Industries of California, Inc., Perris, California, a California corporation.

(B) Coachmen Industries of Georgia, Inc., Fitzgerald, Georgia, a Georgia corporation.

(C) Coachmen Industries of Indiana, Middlebury, Indiana, an Indiana corporation.

(D) Coachmen Industries of Oregon, Inc., Mt. Angel, Oregon, an Oregon corporation.

(E) Coachmen Industries of Texas, Inc., Grapevine, Texas, a Texas corporation.

(F) Consolidated Leisure Industries, Elkhart, Indiana, an Indiana corporation.

(G) Fan Coach Company, Goshen, Indiana, an Indiana corporation.

(H) Shasta Industries of Pennsylvania, Inc., Leola, Pennsylvania, a Pennsylvania corporation.

(I) Sportscoach Corporation of America, Elkhart, Indiana, a California corporation.

(J) Viking Recreational Vehicles, Inc., Centreville, Michigan, a Michigan corporation.

(K) Coach-Lite Supply Company, Elkhart, Indiana, an Indiana corporation.

(L) United Sales and Warehouse, a Texas corporation.

(M) The Lux Company, Inc., Elkhart, Indiana, an Indiana corporation.

(N) All American Homes, Inc., Decatur, Indiana, an Indiana corporation.

(O) Marlette Homes, Inc., Middlebury, Indiana, a Michigan corporation.

1. Parent corporation and address of principal office: Wetterau Incorporated, 8920 Pershall Road, Hazelwood, Missouri 63042.

2. Wholly-owned subsidiaries and divisions which will participate in the operations, and states of incorporation:

| Name and Location | State |
|---|---|
| Wetterau Incorporated, Hazelwood, MO | MO |
| Wetterau Incorporated, Wetterau Food Distribution Division, Hazelwood, MO. | MO. |
| Wetterau Incorporated, Wetterau Printing Division, Hazelwood, MO. | MO. |
| Wetterau Incorporated, Wetterau Food Distribution Division, Mexico, MO. | MO. |
| Wetterau Incorporated, Wetterau Food Distribution Division, Scott City, MO. | MO. |
| Wetterau Incorporated, Wetterau General Merchandise Division, Desloge, MO. | MO. |
| Wetterau Incorporated, Wetterau Information Services Division, Florissant, MO. | MO. |
| Wetterau Incorporated, Wetterau Food Distribution Division, Keene, NH. | MO. |
| Wetterau Incorporated, Wetterau Food Distribution Division, Bloomington, IN. | MO. |
| Wetterau Incorporated, Wetterau Food Distribution Division, Charleston, SC. | MO |
| Wetterau Incorporated, Wetterau Food Distribution Division, Greenville, KY. | MO. |
| Hazelwood Farm Bakeries, Inc. d.b.a. Wetterau Bakery Products, Hazelwood, MO. | MO. |
| Wetterau Builders, Inc., d.b.a. Wetterau Development & Construction, Hazelwood, MO. | MO. |
| Hazelwood Farm Bakeries, Inc. d.b.a. Wetterau Bakery Products, Atlanta, GA. | MO. |
| Fox Grocery Company, d.b.a. Wetterau-Pittsburgh Division, Monroeville, PA. | WV. |
| Fox Grocery Company, d.b.a. Wetterau-Pittsburgh Group Headquarters, Pittsburgh, PA. | WV. |
| Fox Grocery Company, Non-Food Division, Butler, PA. | WV. |
| Fox Grocery Company, d.b.a. Wetterau-West Virginia Division, Milton, WV. | WV. |
| Fox Grocery Company, d.b.a. Wetterau-Clarksburg Division, Clarksburg, WV. | WV. |
| Foodland International Corp., Belle Vernon, PA | PA. |
| Wetterau Food Services, Inc., Temple, PA | PA. |
| Laneco, Inc., Easton, PA | PA. |
| Milliken Tomlinson Company, Portland Division, Portland, ME. | ME. |
| Milliken Tomlinson Company, Presque Isle Division, Presque Isle, ME. | ME. |
| Shop 'N Save Warehouse Foods, Inc., St. Louis, MO. | MO. |
| Millgram Food Stores, Inc., Kansas City, MO | MO. |
| Meyer Dairy, Inc., Basehor, KS | KS. |
| College Club Dairy, Inc., Fayetteville, AK | AK. |
| Acee Dairy, Inc., Fort Smith, AK | AK. |
| W.T. Sistrunk & Co., Lexington, KY | KY. |
| Trans Continental Leasing, Ltd., Hazelwood, MO | MO. |
| Wetterau Transportation, Inc., Hazelwood, MO | MO. |
| Corporate Freight Brokerage, Inc., Hazelwood, MO | MO. |
| Foxtran, Inc., Belle Vernon, PA | PA. |

James H. Bayne,

*Secretary.*

[FR Doc. 84-28277 Filed 10-25-84; 8:45 am]

BILLING CODE 7035-01-M

---

[Docket No. AB-6; Sub-213]

### Railroads; Burlington Northern Railroad Company; Abandonment; in Linn, Buchanan, Clinton, DeKalb, Caldwell, and Livingston Counties, MO

The Commission has issued a certificate authorizing the Burlington, Northern Railroad Company (BN) to abandon its 91.82 mile rail line between milepost 109.18 near Laclede and milepost 201.0 near St. Joseph, in Linn, Buchanan, Clinton, DeKalb, Caldwell, and Livingston Counties, MO. The abandonment certificate will become effective 30 days after this publication unless the Commission also finds that: (1) A financially responsible person has offered financial assistance (through subsidy or purchase) to enable the rail service to be continued; and (2) it is likely that the assistance would fully compensate the railroad.

Any financial assistance offer must be filed with the Commission and the applicant no later than 10 days from publication of this Notice. The following notation shall be typed in bold face on the lower left-hand corner of the envelope containing the offer: "Rail Section, AB–OFA." Any offer previously made must be remade within this 10-day period.

Information and procedures regarding financial assistance for continued rail service are contained in 49 U.S.C. 10905 and 49 CFR 1152.27.

James H. Bayne,

*Secretary.*

[FR Doc. 84-28278 Filed 10-25-84; 8:45 am]

BILLING CODE 7035-01-M

---

[Finance Docket No. 30565]

### Railroads; the Chesapeake and Ohio Railway Company; Trackage Rights Exemption

On September 26, 1984, The Chespeake and Ohio Railway Company (C&O) filed a notice of exemption for trackage rights over a line of track of the Seaboard System Railroad, Inc. (SSR) between Covington and Anchorage, KY, a distance of 94.6 miles, and between Latonia, KY and the north end of SBD's DeCoursey Yard, a distance of 1.8 miles.

On October 4, 1984, the Railway Labor Executives' Association requested the imposition of employee protective conditions.

The involved transaction also contemplates that C&O traffic moving between Cincinnati, OH and Louisville, KY, over the subject trackage rights route, will be handled under an Agency Agreement among C&O, SSR, and The Baltimore and Ohio Railroad Company (B&O). Under this agreement, B&O will handle, as C&O's agent, cars of C&O, loaded and empty, revenue and non-revenue, between Cincinnati and Louisville over a route that is comprised, in principal part, of SSR's line that is the subject of the instant trackage rights agreement. The other lines of railroad that comprise the remainder of the route between Cincinnati, OH and Louisville, KY are described in the Agency Agreement; C&O possesses rights to use such other lines under existing agreements.

The subject transaction will permit C&O to reroute the subject overhead traffic by utilizing existing facilities, trains, and services. Such rerouting will produce operating economies for C&O and improve its financial viability. In

addition, by rerouting the subject overhead traffic, C&O will be able to rationalize its existing facilities and services by its intent to abandon its line between Winchester and Coalton, KY. This line allegedly generates a *de minimis* amount of traffic but is presently required for the movement of the subject overhead traffic.[1]

This transaction is within a corporate family and comes within that class of transactions described in 49 CFR 1180.2(d)(3) that has been exempted from Commission regulation. The C&O purchase of the line will not result in changes in service levels, significant operation changes, or a change in the competitive balance with carriers outside the corporate family.

As a condition to the use of this exemption, any employees affected by the trackage rights agreement will be protected pursuant to *Norfolk and Western Ry. Co.—Trackage Rights—BN*, 354 I.C.C. 605 (1978), as modified by *Mendocino Coast Ry., Inc.—Lease and Operate*, 360 I.C.C. 653 (1980).

Decided: October 18, 1984.

By the Commission, Heber P. Hardy, Director, Office of Proceedings.

**James H. Bayne,**

*Secretary.*

[FR Doc. 84-28280 Filed 10-25-84; 8:45 am]

BILLING CODE 7035-01-M

---

[Finance Docket No. 30555]

## Northwestern Pacific Acquiring Corporation and Eureka Southern Railroad Company; Exemption From 49 U.S.C. 10901 and 11301

**AGENCY:** Interstate Commerce Commission.

**ACTION:** Notice of exemption.

**SUMMARY:** The Interstate Commerce Commission exempts Northwestern Pacific Acquiring Corporation (Acquiring) from the requirements of (1) 49 U.S.C. 10901 in connection with the acquisition from the Northwestern Pacific Railroad Company of its Eel River Line, extending from milepost 142.5, near Outlet, CA, to milepost 284.1, near Eureka, CA, and three short branch lines, and (2) 49 U.S.C. 11301 in connection with (a) the issuance of two promissory notes to accomplish the acquisition, and (b) the issuance of certain stock. The Commission also exempts Eureka Southern Railroad Company (Eureka) from the requirements of (1) 49 U.S.C. 10901 in connection with the lease and operation of the line, and (2) 49 U.S.C. 11301 in connection with the issuance of certain stock.

**DATES:** These exemptions will be effective on October 25, 1984. Petitions to reopen must be filed by November 15, 1984.

**ADDRESSES:** Send pleadings referring to Finance Docket No. 30555 to:

(1) Office of the Secretary, Case Control Branch, Interstate Commerce Commission, Washington, DC 20423
(2) Petitioners' representative: Sander M. Bieber, 1730 Pennsylvania Ave., NW., Washington, DC 20006.

**FOR FURTHER INFORMATION CONTACT:** Louis E. Gitomer, (202) 275-7245.

**SUPPLEMENTARY INFORMATION:** Additional information is contained in the Commission's decision. To purchase a copy of the full decision, write to T.S. InfoSystems, Inc., Room 2227, Interstate Commerce Commission, Washington, DC 20423, or call 289-4357 (DC Metropolitan area) or toll free (800) 424-5403.

Decided: October 18, 1984.

By the Commission, Chairman Taylor, Vice Chairman Andre, Commissioners Sterrett, Gradison, Simmons, Lamboley, and Strenio.

**James H. Bayne,**

*Secretary.*

[FR Doc. 84-28279 Filed 10-25-84; 8:45 am]

BILLING CODE 7035-01-M

[1] The abandonment of the Winchester-Coalton line will be the subject of an abandonment proceeding in Docket No. AB-18 (Sub-No. 53).

---

## DEPARTMENT OF JUSTICE

### Information Collection(s) Under Review by OMB

October 23, 1984.

The Office of Management and Budget (OMB) has been sent for review the following proposals for the collection of information under the provisions of the Paperwork Reduction Act (44 U.S.C. Chapter 35) since the last list was published. The list has all entries grouped into new forms, revisions, or extensions. Each entry contains the following information:

(1) The name and telephone number of the Agency Clearance Officer (from whom a copy of the form and supporting documents is available);

(2) The office of the agency issuing the form;

(3) The title of the form;

(4) The agency form number, if applicable;

(5) How often the form must be filled out;

(6) Who will be required or asked to report;

(7) An estimate of the number of responses;

(8) An estimate of the total number of hours needed to fill out the form;

(9) An indication of whether section 3504(h) of Pub L. 96.511 applies; and,

(10) The name and telephone number of the person or office responsible for the OMB review.

Copies of the proposed form(s) and the supporting documentation may be obtained from the Agency Clearance Officer whose name and telephone number appear under the agency name. Comments and questions regarding the items contained in this list should be directed to the reviewer listed at the end of each entry *AND* to the Agency Clearance Officer. If you anticipate commenting on a form but find that time to prepare will prevent you from submitting comments promptly, you should advise the reviewer and the Agency Clearance Officer of your intent as early as possible.

**Department of Justice**

Agency Clearance Officer: Larry E. Miesse, 202/633-4312.

• *Extension of the Expiration Date of a Currently Approved Collection Without any Change in the Substance or in the Method of Collection*

(1) Larry E. Miesse, 202/633-4312
(2) Immigration and Naturalization Service, Department of Justice
(3) Report of Status by Treaty Trader or Investor
(4) I-126
(5) On occasion
(6) Individuals or households. Information is used to determine whether an alien admitted to the United States as a treaty trader or investor under section 3(6) of the Act of 1924 or section 101(a)(15(e) of the INA is maintaining status.
(7) 35,000 respondents
(8) 17,500 burden hours
(9) Not applicable under 3504(h)
(10) Robert Veeder—395-4814

(1) Larry E. Miesse, 202/633-4312
(2) Correctional Programs Branch, Bureau of Justice Statistics, Department of Justice
(3) Capital Punishment, Report of Inmates Under Sentence of Death
(4) NPS-8, NPS-8A, NPS-8B, NPS-8C, NPS-8(L)
(5) Annually
(6) State or local governments, Federal agencies or employees. This program is concerned with a study of capital punishment statutes and of persons under sentence of death in state and federal correctional institutions.
(7) 1,552 respondents
(8) 401 burden hours
(9) Not applicable under 3504(h)

(10) Robert Veeder—395–4814

(1) Larry E. Miesse, 202/633–4312
(2) National Institute of Justice, Office of Justice Assistance, Research and Statistics, Department of Justice
(3) National Needs Assessment Survey
(4) NIJ Series 2300
(5) Biennially
(6) State or local governments. This data will assist the National Institute of Justice in meeting its Congressional mandate—performing research in the problems of state and local justice systems—by providing information on current and future criminal justice needs.
(7) 1,501 respondents
(8) 650 burden hours
(9) Not applicable under 3504(h)
(10) Robert Veeder—395–4814

(1) Larry E. Miesse, 202/633–4312
(2) Office of the Comptroller, Office of Justice Assistance, Research and Statistics, Department of Justice
(3) Accounting System and Financial Capability Questionnaire
(4) OJARS 7120/1
(5) On occasion
(6) Non-profit institutions, small businesses or organizations. Completed by applicants that are newly formed firms or established firms with no previous Federal government business, and used as an aid to determine those applicants/grantees that may require special attention in matters relating to the accountability of Federal funds.
(7) 60 respondents
(8) 240 burden hours
(9) Not applicable under 3504(h)
(10) Robert Veeder—395–4814

Δ *New Collection*

(1) Larry E. Miesse, 202/633–4312
(2) Bureau of Justice Statistics, Department of Justice
(3) National Crime Survey (Series Pretest)
(4) NCS–2S, NCS–2T(1), (2), (3), (4)
(5) Monthly (after pretest)
(6) Individuals or households. The Pretest is for a program for collecting and analyzing statistics on the nature of incidents reported as a series of crimes. Data used to gain insight into the diversity that may exist within a series of crimes.
(7) 420 respondents
(8) 70 burden hours
(9) Not applicable under 3504(h)
(10) Robert Veeder—395–4814

Larry E. Miesse,

*Agency Clearance Officer, Department of Justice.*

[FR Doc. 84–28386 Filed 10–25–84; 8:45 am]

BILLING CODE 4410–01–M

## DEPARTMENT OF LABOR

### Office of the Secretary

### Agency Forms Under Review by the Office of Management and Budget (OMB)

Background: The Department of Labor, in carrying out its responsibility under the Paperwork Reduction Act (44 U.S.C. Chapter 35), considers comments on the proposed forms and recordkeeping requirements that will affect the public.

List of Forms Under Review: On each Tuesday and/or Friday, as necessary, the Department of Labor will publish a list of the Agency forms under review by the Office of Management and Budget (OMB) since the last list was published. The list will have all entries grouped into new collections, revisions, extensions, or reinstatements. The Departmental Clearance Officer will, upon request, be able to advise members of the public of the nature of any particular revision they are interested in.

Each entry will contain the following information:

The Agency of the Department issuing this form.

The title of the form.

The OMB and Agency form numbers, if applicable.

How often the form must be filled out.

Who will be required to or asked to report.

Whether small businesses or organizations are affected.

An estimate of the number of responses.

An estimate of the total number of hours needed to fill out the form.

The number of forms in the request for approval.

An abstract describing the need for and uses of the information collection.

Comments and Questions: Copies of the proposed forms and supporting documents may be obtained by calling the Department Clearance Officer, Paul E. Larson, Telephone 202–523–6331. Comments and questions about the items on this list should be directed to Mr. Larson, Office of Information Management, U.S. Department of Labor, 200 Constitution Avenue, NW., Room S–5526, Washington, D.C. 20210. Comments should also be sent to the OMB reviewer, Arnold Strasser, Telephone 202–395–6880, Office of Information and Regulatory Affairs, Office of Management and Budget, Room 3208, NEOB, Washington, D.C. 20503.

Any member of the public who wants to comment on a form which has been submitted to OMB should advise Mr. Larson of this intent at the earliest possible date.

### Collection of Information in Current Rules

Occupational Safety and Health Administration.

Standard for Occupational Exposure to Ethylene Oxide.

OSHA 265.

On occasion.

Businesses or other for profit; small businesses or organizations.

6,453 respondents; 355,172 hours.

These regulations require employers to establish and maintain accurate records of exposure monitoring and medical surveillance for employees exposed to ethylene oxide. These records will be used by employers, employees, physicians and the Government to assure that workplace exposure to ethylene oxide does not adversely affect the health of employees.

Signed at Washington, D.C. this 23rd day of October 1984.

**Richard Glesener,**

*Acting Departmental Clearance Officer.*

[FR Doc. 84–28356 Filed 10–25–84; 8:45 am]

BILLING CODE 4510–26–M

### Office of Pension and Welfare Benefit Programs

**[Prohibited Transaction Exemption 84–161; Exemption Application No. D–3067 et al.]**

### Grant of Individual Exemptions; Alaska Mutual Bank, et al.

**AGENCY:** Pension and Welfare Benefit Programs, Labor.

**ACTION:** Grant of individual exemptions.

**SUMMARY:** This document contains exemptions issued by the Department of Labor (the Department) from certain of the prohibited transaction restrictions of the Employee Retirement Income Security Act of 1974 (the Act) and/or the Internal Revenue Code of 1954 (the Code).

Notices were published in the **Federal Register** of the pendency before the Department of proposals to grant such exemptions. The notices set forth a summary of facts and representations contained in each application for exemption and referred interested persons to the respective applications for a complete statement of the facts and representations. The applications have been available for public inspection at the Department in Washington, D.C. The notices also invited interested persons to submit

comments on the requested exemptions to the Department. In addition the notices stated that any interested person might submit a written request that a public hearing be held (where appropriate). The applicants have represented that they have complied with the requirements of the notification to interested persons. No public comments and no requests for a hearing, unless otherwise stated, were received by the Department.

The notice of pendency were issued and the exemptions are being granted solely by the Department because, effective December 31, 1978, section 102 of Reorganization Plan No. 4 of 1978 (43 FR 47713, October 17, 1978) transferred the authority of the Secretary of the Treasury to issue exemptions of the type proposed to the Secretary of Labor.

**Statutory Findings**

In accordance with section 408(a) of the Act and/or section 4975(c)(2( of the Code and the procedures set forth in ERISA Procedure 75–1 (40 FR 18471, April 28, 1975), and based upon the entire record, the Department makes the following findings:

(a) The exemptions are administratively feasible;

(b) They are in the interest of the plans and their participants and beneficiaries; and

(c) They are protective of the rights of the participants and beneficiaries of the plans.

**Alaska Mutual Bank (AMB) Located in Anchorage, Alaska**

[Prohibited Transaction Exemption 84–161, Application No. D–3067]

*Exemption*

I. Effective January 1, 1975, the restrictions of section 406(a) of the Act and the sanctions resulting from the application of section 4975 of the Code, by reason of section 4975(c)(1) (A) through (D) of the Code shall not apply to the past and proposed sale, exchange or transfer between AMB and certain employee benefit plans (the Plans) of multi-family residential and commercial mortgage loans (the Mortgages) or participation interests therein (the Participation Interests) which are originated by AMB provided that:

A. Such sale, exchange or transfer is expressly approved by a fiduciary independent of AMB who has authority to manage or control those Plan assets being invested in Mortgages or Participation Interests;

B. The terms of all transactions between the Plans and AMB involving the Mortgages or Participation Interests are not less favorable to the Plans than the terms generally available in arm's length transactions between unrelated parties;

C. No investment management, advisory, underwriting fee or sales commission or similar compensation is paid to AMB with regard to such sale, exchange or transfer;

D. The decision to invest in a Mortgage or Participation Interest is not part of an arrangement under which a fiduciary of a Plan, acting with the knowledge of AMB, causes a transaction action to be made with or for the benefit of a party in interest (as defined in section 3(14) of the Act) with respect to the Plan; and

E. AMB shall maintain for the duration of any Mortgage or Participation Interest which is sold to a Plan pursuant to this exemption, records necessary to determine whether the conditions of this exemption have been met. The records referred to above must be unconditionally available at their customary location for examination, for purposes reasonably related to protecting rights under the Plans, during normal business hours by: any trustee, investment manager, employer of Plan participants, employee organization whose members are covered by a Plan, participant or beneficiary of a Plan.

II. Effective January 1, 1975, the restriction of section 406(a) of the Act and the sanctions resulting from the application of section 4975 of the Code by reason of section 4975(c)(1) (A) through (D) of the Code shall not apply to any transactions to which such restrictions or taxes would otherwise apply merely because a person is deemed to be a party in interest (including a fiduciary) with respect to a Plan by virtue of providing services to the Plan (or who has a relationship to such service provider described in section 3(14), (F), (G), (H), or (I) of the Act) solely because of the ownership of a Mortgage or Participation Interest by such Plan.

For a more complete statement of the facts and representations supporting the Department's decision to grant this exemption refer to the notice of proposed exemption published on July 6, 1984 at 49 FR 27844.

*Effective Date:* The effective date of this exemption is January 1, 1975.

Written Comments: The only comment received by the Department was from AMB. AMB noted the following factual inaccuracies in the notice of proposed exemption. Since the filing of the application for exemption AMB was converted from a mutual savings bank owned by its depositors (as stated in the notice of proposed exemption) to a shareholder-owned bank. Also, AMB states that the notice incorrectly represents that its headquarters are located in Fairbanks, Alaska instead of the correct site, which is Anchorage, Alaska. The applicant represents that the plans that had previously been involved in the transactions covered by this exemption were notified of the publication in the **Federal Register** of the notice of pendency and their rights to comment with respect to the proposed exemption on September 14, 1984. Therefore, the Department extended the 30 day comment period with respect to the proposed exemption to October 14, 1984.

For Further Information Contact: Louis Campagna of the Department, telephone (202) 523–8971. (This is not a toll-free number.)

**Texas Automatic Sprinklers Pension Plan (the Plan)**

[Prohibited Transaction Exemption 84–162, Exemption Application No. D–4813]

*Exemption*

The restrictions of section 406(a), 406 (b)(1) and (b)(2) of the Act and the sanctions resulting from the application of section 4975 of the Code, by reason of section 4975(c)(1) (A) through (E) of the Code, shall not apply to (1) the continuation past June 30, 1984 of two leases (the Leases) of certain improved real property by the Plan to Texas Automatic Sprinklers, Inc. (the Employer), the sponsor of the Plan; and (2) the guaranty of the Employer's obligations under one of the Leases by Sam P. Wallace Company, Inc., the parent company of the Employer; provided that such transactions are on terms and conditions at least as favorable to the Plan as the Plan could obtain in dealing with an unrelated party.

For a more complete statement of the facts and representations supporting the Department's decision to grant this exemption refer to the notice of proposed exemption published on June 8, 1984 at 49 FR 23959.

*Effective Date:* This exemption is effective July 1, 1984.

For Further Information Contact: Ronald Willett of the Department, telephone (202) 523–8194. (This is not a toll-free number.)

*Mayfair Super Markets, Inc. Employees Retirement Plan (the Plan) Located in Elizabeth, NJ*

[Prohibited Transaction Exemption 84–163, Exemption Application No. D–5048]

*Exemption*

The restrictions of section 406(a), 406 (b)(1) and (b)(2) of the Act and the sanctions resulting from the application of section 4975 of the Code, by reason of section 4975(c)(1) (A) through (E) of the Code, shall not apply to the sales on May 4, 1981, April 21, 1982, and November 12, 1982 of certain bonds (the Bonds) by the Plan to Mayfair Super Markets, Inc., a party in interest with respect to the Plan, provided the sales prices, which aggregated $91,000, were not less than the aggregate fair market value of the Bonds on the date of each sale.

For a more complete statement of the facts and representations supporting the Department's decision to grant this exemption refer to the notice of proposed exemption published on August 13, 1984 at 49 FR 32279.

*Effective Date:* The exemption is effective May 4, 1981, April 21, 1982, and November 12, 1982.

For Further Information Contact: Miriam Freund of the Department, telephone (202) 523–8971. (This is not a toll-free number.)

*John G. Beck Agency, Inc. Employees' Profit Sharing Retirement Trust (the Plan) Located in Pittsburgh, PA*

[Prohibited Transaction Exemption 84–164, Exemption Application No. D–5141]

*Exemption*

The restrictions of section 406(a), 406 (b)(1) and (b)(2) of the Act and the sanctions resulting from the application of section 4975 of the Code, by reason of section 4975(c)(1) (A) through (E) of the Code, shall not apply to the sale, on April 1, 1977, by the Plan of certain common stock (the Superior Stock) to Mr. and Mrs. Robert A. Beck and Mr. and Mrs. Paul C. Miller, provided the consideration given for the Superior Stock was not less than its fair market value at the time the sale was executed.

For a more complete statement of the facts and representations supporting the Department's decision to grant this exemption refer to the notice of proposed exemption published on August 14, 1984 at 49 FR 32477.

*Effective Date:* The exemption is effective April 1, 1977.

For Further Information Contact: Ms. Jan D. Broady of the Department, telephone (202) 523–8971. (This is not a toll-free number.)

**DIAL ONE of Northern California Retirement Plan (the Plan) Located in Sacramento, CA**

[Prohibited Transaction Exemption 84–165; Exemption Application No. D–5154]

*Exemption*

The restrictions of section 406(a) and 406 (b)(1) and (b)(2) of the Act and the sanctions resulting from the application of section 4975 of the Code, by reason of section 4975(c)(1) (A) through (E) of the Code, shall not apply to the cash purchase from Mr. Oliver F. Speraw (Mr. Speraw) by the Transferred Account of Mr. Speraw under the Plan of 100,000 shares of common stock (the Stock) of DIAL ONE International, Inc., provided the purchase price of the Stock does not exceed its fair market value on the date of the purchase.

For a more complete statement of the facts and representatives supporting the Department's decision to grant this exemption refer to the notice of proposed exemption published on August 31, 1984 at 49 FR 32283.

For Further Information Contact: Miriam Freund of the Department, telephone (202) 523–8971. (This is not a toll-free number.)

**Winchester Optical Company Pension Plan (the Winchester Plan); Winchester Optical Company of Geneva, New York, Inc. Pension Plan (the Geneva Plan); Winchester Optical Company of Rochester, New York, Inc. Pension Plan (the Rochester Plan); and Winchester Optical Company of Williamsport, Pennsylvania, Inc., Pension Plan (the Williamsport Plan, collectively, the Plans) Located in Elmira, New York; Geneva, NY; Rochester, NY; and Williamsport, PA**

[Prohibited Transaction Exemption 84–166; Exemption Application No. D–5307]

*Exemption*

The restrictions of section 406(a) and 406 (b)(1) and (b)(2) of the act and the sanctions resulting from the application of section 4975 of the Code, by reason of section 4975(c)(1) (A) through (E) of the Code, shall not aply to the proposed loans (the Loans) of 25% of the assets of each Plan to Winchester Optical Company, a party in interest with respect to the Plans, provided that the terms and conditions of the loans are no less favorable to the Plans than those obtainable in a similar transaction with an unrelated party.

For a more complete statement of the facts and representatives supporting the Department's decision to grant this exemption refer to the notice of proposed exemption published on September 6, 1984 at 49 FR 35266.

For Further Information Contact: David M. Cohen of the Department, telephone (202) 523–8671. (This is not a toll-free number.)

**Grant of an Individual Exemption; Bell System Pension Plan Trust**

[Prohibited Transaction Exemption 84–142]

*Correction*

In FR Doc. 84–25854, published at page 38381, on Friday, September 28, 1984, on page 38382, in the first column, the sixth full paragraph shall read as follows:

*Effective Date:* This exemption is effective December 1, 1982.

**General Information**

The attention of interested persons is directed to the following:

(1) The fact that a transaction is the subject of an exemption under section 408(a) of the Act and/or section 4975(c)(2) of the Code does not relieve a fiduciary or other party in interest or disqualified person from certain other provisions of the Act and/or the Code, including any prohibited transaction provisions to which the exemption does not apply and the general fiduciary responsibility provisions of section 404 of the Act, which among other things require a fiduciary to discharge his duties respecting the plan solely in the interest of the participants and beneficiaries of the plan and in a prudent fashion in accordance with section 404(a)(1)(B) of the Act; nor does it affect the requirement of section 401(a) of the Code that the plan must operate for the exclusive benefit of the employees of the employer maintaining the plan and their beneficiaries;

(2) These exemptions are supplemental to and not in derogation of, any other provisions of the Act and/or the Code, including statutory or administrative exemptions and transitional rules. Furthermore, the fact that a transaction is subject to an administrative or statutory exemption is not dispositive of whether the transaction is in fact a prohibited transaction.

(3) The availability of these exemptions is subject to the express condition that the material facts and representations contained in each application accurately describes all material terms of the transaction which is the subject of the exemption.

Signed at Washington, D.C., this 23rd day of October, 1984.

**Elliot I. Daniel,**

*Acting Assistant Administrator for Regulations and Interpretations, Office of Pension and Welfare Benefit Programs, U.S. Department of Labor.*

[FR Doc. 84-28347 Filed 10-25-84; 8:45 am]

BILLING CODE 4510-29-M

[Application No. D-5088 et al.]

## Proposed Exemptions; J.H. Kleinfelder & Associates, et al.

**AGENCY:** Pension and Welfare Benefit Programs, Labor.

**ACTION:** Notice of proposed exemptions.

**SUMMARY:** This document contains notices of pendency before the Department of Labor (the Department) of proposed exemptions from certain of the prohibited transaction restrictions of the Employee Retirement Income Security Act of 1974 (the Act) and/or the Internal Revenue Code of 1954 (the Code).

### Written Comments and Hearing Requests

All interested persons are invited to submit written comments or requests for a hearing on the pending exemptions, unless otherwise stated in the Notice of Pendency, within 45 days from the date of publication of this **Federal Register** Notice. Comments and requests for a hearing should state the reasons for the writer's interest in the pending exemption.

**ADDRESS:** All written comments and requests for a hearing (at least three copies) should be sent to the Office of Fiduciary Standards, Pension and Welfare Benefit Programs, Room C-4526, U.S. Department of Labor, 200 Constitution Avenue, NW., Washington, D.C. 20216. Attention: Application No. stated in each Notice of Pendency. The applications for exemption and the comments received will be available for public inspection in the Public Documents Room of Pension and Welfare Benefit Programs, U.S. Department of Labor, Room N-4677, 200 Constitution Avenue, NW., Washington, D.C. 20216

### Notice to Interested Persons

Notice of the proposed exemptions will be provided to all interested persons in the manner agreed upon by the applicant and the Department within 15 days of the date of publication in the **Federal Register.** Such notice shall include a copy of the notice of pendency of the exemption as published in the **Federal Register** and shall inform interested persons of their right to comment and to request a hearing (where appropriate).

**SUPPLEMENTARY INFORMATION:** The proposed exemptions were requested in applications filed pursuant to section 408(a) of the Act and/or section 4975(c)(2) of the Code, and in accordance with procedures set forth in ERISA Procedure 75-1 (40 FR 18471, April 28, 1975). Effective December 31, 1978, section 102 of Reorganization Plan No. 4 of 1978 (43 FR 47713, October 17, 1978) transferred the authority of the Secretary of the Treasury to issue exemptions of the type requested to the Secretary of Labor. Therefore, these notices of pendency are issued solely by the Department.

The applications contain representations with regard to the proposed exemptions which are summarized below. Interested persons are referred to the applications on file with the Department for a complete statement of the facts and representations.

### J.H. Kleinfelder & Associates Employee Profit Sharing Plan (the Plan) Located in Walnut Creek, California

[Application No. D-5088]

*Proposed Exemption*

The Department is considering granting an exemption under the authority of section 408(a) of the Act and section 4975(c)(2) of the Code and in accordance with the procedures set forth in ERISA Procedure 75-1 (40 FR 18471, April 28, 1975). If the exemption is granted the restrictions of section 406(a), 406 (b)(1) and (b)(2) and 407(a) of the Act and the sanctions resulting from the application of section 4975 of the Code, by reason of section 4975(c)(1) (A) through (E) of the Code shall not apply to a lease, effective July 1, 1984, by the Plan of certain improved real property (the Property) to J.H. Kleinfelder & Associates (the Employer), the sponsor of the Plan; provided that the terms of such lease are at least equivalent to those which the Plan would receive in an arm's length transaction with an unrelated party.

Effective Date: If granted, this exemption will be effective July 1, 1984.

*Summary of Facts and Representations*

1. The Plan is a defined contribution plan with 73 participants and total assets of $1,283,025 as of January 31, 1984. The Employer is a California professional corporation engaged in engineering consultation services and has a net worth of $921,000 as of March 31, 1984. The trustee of the Plan is the Union Safe Deposit Bank (the Trustee) in Stockton, California, which represents that it is independent of and unrelated to the Employer except as Trustee of the Plan. The Property is a two-acre parcel of real property located at 2824 East Myrtle Avenue in an industrial district of Stockton, California, improved with a one-strony concrete and frame office and laboratory building which serves as one of the Employer's principal places of business. The Property has been appraised for its fair market value by Craig Hubbard, M.A.I. (Hubbard), an independent professional real estate appraiser located in Stockton, California. Hubbard represents that as of March 15, 1984 the Property had a fair market value of $220,000. Since its acquisition by the Plan in 1970, the Property has been leased by the Plan to the Employer continuously under a ten-year lease (the Original Lease), effective June 1, 1970, which was renewed and extended by its own terms in 1980 for an additional ten-year term to expire May 30, 1990. The Employer represents that the Original Lease was statutorily exempt until June 30, 1984 from the prohibitions of section 406 of the Act.[1] The Employer is requesting an exemption to permit the Plan to continue leasing the Property to the Employer past June 30, 1984 under a modification of the Original Lease (the New Lease) effective July 1, 1984.

2. The interests of the Plan for all purposes under the New Lease are represented by the Trustee. The New Lease is a triple net lease for an initial term of 15 years, renewable only with the approval of the Trustee for one additional term of 15 years. Under the New Lease the Employer assumes responsibility for all taxes, all costs of maintenance and repair, and full fire and extended coverage insurance on the Property. The New Lease requires the Employer to indemnify the Plan and hold the Plan harmless against all claims, demands and liabilities resulting from the Employer's use of the Property. The annual rental under the New Lease, payable monthly, will be no less than the fair market rental value of the Property, and will be adjusted annually to reflect the greater of: (1) The increase in the appraised fair market rental value of the Property, as determined by a qualified independent real estate appraiser selected by the Trustee; or (2) the increase in the San Francisco/Oakland area Consumer Price Index for All Urban Consumers, All Items (the CPI), as reported by the Bureau of Labor

[1] The Department expresses no opinion as to whether the Original Lease constitutes a lease within the definition of section 414(c)(2) of the Act.

Statistics of the U.S. Department of Labor. Annual rental during the renewal term, if any, shall be determined in the same manner. Initial rental under the New Lease, commencing July 1, 1984, is set at $2,237.16 per month, the fair market rental value of the Property having been appraised by Hubbard as $1,575 per month as of June 12, 1984. The Trustee represents that the initial rental under the New Lease exceeds the appraised fair market value of the Property due to previous rental adjustments under the Original Lease, reflecting changes in the CPI, and the desire of the parties to assure the Plan a return under the New Lease on a basis as favorable as that under the Original Lease. Other than the amount of rental, all terms and conditions of the New Lease for its renewal term, if any, shall be the same as those under the initial term.

3. The Trustee will represent the Plan in the enforcement of all terms and conditions of the New Lease and on behalf of the Plan will monitor the Employer's performance thereunder for the duration of the New Lease and any renewal thereof. The continued leasing of the Property past June 30, 1984 by the Plan to the Employer was reviewed and evaluated as of July 1, 1974, by O.B. Wied (Wied), Senior Vice President and Trust Officer of the Trustee. Wied represents that such continued leasing is in the best interests of the Plan for the following reasons: (1) The Property has a likelihood of continuing substantial appreciation due to developments in the surrounding area, (2) the Employer could easily lease similar property for lower rentals than that required under the New Lease and it does not appear that the Employer is receiving any particular advantage in leasing the Property from the PLan, (3) the Property represents approximately 17 percent of total Plan assets as of March 31, 1984 and it constitutes the Plan's sole investment in real property, (4) in light of the Plan's overall assets mix, continued investment in the Property does not adversely affect the Plan's liquidity needs, and (5) under the terms of the New Lease the Plan is likely to receive, as it did under the Original Lease, rentals which are in excess of those which the Plan could expect in the local market dealing at arm's length, due to the use of the CPI as an additional rent accelerator.

4. In summary, the applicant represents that the criteria of section 408(a) of the Act are satisfied in the subject transaction because: (1) The interests of the Plan under the New Lease are represented by the Trustee, an independent fiduciary which will monitor the Employer's performance and represent the Plan for all purposes under the New Lease; (2) the Trustee has reviewed and evaluated the subject transaction and has determined that it is in the best interests of the Plan; (3) the New Lease ensures that the Plan will continue to receive a rate of return on the Property which is no less than its fair market rental value as determined by an independent, qualified real estate appraiser, and such return will be net of all expenses related to the Property; (4) the New Lease is protective of the Plan's interests in the Property by requiring the Employer to carry full fire and extended coverage insurance on the Property and to indemnify the plan against all claims, demands and liabilities which may result from the Employer's use of the Property; and (5) as the Plan's sole investment in real property, the Property represents approximately 17 percent of total Plan assets and does not adversely afect the Plan's liquidity needs in light of the overall mix of Plan assets.

For Further Information Contact: Ronald Willett of the Department, telephone (202) 523–8194. (This is not a toll-free number.)

**InterFirst Corporation Retirement Program (the Plan) Located in Dallas, Texas**

[Application No. D–5216]

*Proposed Exemption*

The Department is considering granting an exemption under the authority of section 408(a) of the Act and section 4975(c)(2) of the Code and in accordance with the procedures set forth in ERISA Procedure 75–1 (40 FR 18471, April 28, 1975). If the exemption is granted the restrictions of section 406(a), 406 (b)(1) and (b)(2) of the Act and the sanctions resulting from the application of section 4975 of the Code, by reason of section 4975(c)(1) (A) through (E) of the Code shall not apply to the lease of real property by the Plan to InterFirst Bank Dallas, N.A. (the Bank), a party in interest with respect to the Plan, provided that such lease was and will continue to be on terms and conditions at least as favorable to the Plan as those obtainable by the Plan in an arm's length transaction with an unrelated party.

Effective Date: This proposed exemption, if granted, will be effective July 1, 1984.

*Summary of Facts and Representations*

1. The Plan is a separate defined benefit pension plan for the employees of InterFirst Corporation (InterFirst), the Bank, a wholly-owned subsidiary of InterFirst and certain of their Dallas affiliates. The Plan had approximately 1,630 participants as of January 1, 1983 and assets of approximately $62,024,600 as of December 31, 1983. The trustee of the Plan is the Bank. The Plan is administered by a retirement committee, consisting of eight-persons appointed by the board of directors of InterFirst.

2. The Plan leases a parcel of real property located at the northwest corner of Pacific Avenue and Field Street in Dallas, Texas to the Bank. The property under lease consists of two adjoining tracts of land containing approximately 26,998 and 3,667 square feet, respectively (the Land). The lease initially was entered into between the Bank, in its corporate capacity, and the Bank, in its capacity as trustee, on May 15, 1967, and was supplemented by written agreement of those parties on July 28, 1970 (the Lease).[2]

The larger first tract of Land was purchased by the Plan from an unrelated third party in 1967 for $1,335,000.00. The purchase was contingent upon a State District Court of Dallas County, Texas, approving the proposed lease of the tract by the Plan to the Bank. To obtain such approval, a suit was brought in District Court, 101st Judicial District, Dallas County, Texas, by the Bank in its private corporate capacity against representatives of each class of Plan beneficiaries and participants. Counsel represented each class, and every Plan beneficiary and participant received notice of the suit. A hearing was held and evidence was presented with regard to the purchase and lease. In a written judgment dated May 15, 1967 (Cause No. 67–3427–E), the Court found the lease to be fair and in the best interest of the Plan and its beneficiaries. Upon obtaining approval of the District Court, the initial lease was executed.

The smaller second tract of Land was purchased by the Plan from an unrelated third party in 1970 for $183,500.00. The same procedure that had been used in 1967 was used in 1970 to obtain the approval of the District Court, 44th Judicial District, Dallas County, Texas, for the purchase of the second trace and the extension of the Lease to cover the additional land. In a written judgment dated July 28, 1970 (Cause No. 70–5618–B–44th), the Court found the purchase of the Land and the proposed supplement to the Lease to be fair and reasonable and in the best interest of the Plan and

[2] The Bank assigned an undivided 50% of its interest in the Lease to Prudential Insurance Company of America (Prudential) on September 5, 1974. Such assignment shall not become effective, however, until the termination of the sublease from the Bank to First International Building Joint Venture (Joint Venture).

its beneficiaries. Upon obtaining the approval of the District Court, a supplement to the Lease was executed to include the additional land.

3. In 1973, the Bank, in its corporate capacity, sub-leased its interest in the Lease to the Joint Venture in which it now holds a 0.1% interest. The other venturers were InterFirst, which holds a 49.9% interest, and Prudential which holds a 50% interest. The Joint Venture constructed a twelve-story parking garage on the Land after it was sub-leased. The garage is used primarily by tenants and employees of InterFirst and the Bank. Thus, the Plan holds the fee interest in the Land, and the Joint Venture currently owns the parking garage constructed on that Land.

4. As sub-lessee, the Joint Venture agreed to pay directly to the Plan all rent due under the Lease and to perform and comply with all obligations, covenants, agreements, and undertakings of the lessee under the Lease. The only rent the Bank receives as sub-lessor is an amount equal to the sum of all ad valorem property taxes and assessments, general and special, levied, assessed and payable against the Land. The sub-lease of the Land to the Joint Venture, however, did not relieve the Bank of its obligations under the Lease; it still remains liable to the Plan for all the lessee's obligations.

5. The Lease was a net lease under which the lessee was responsible for all insurance, taxes and maintenance. The Plan, as lessor, had no obligations with respect to the leased property. The Lease expires on December 31, 2059, and the sub-lease to the Joint Venture expires on December 31, 2047. Rent payable to the Plan under the Lease was expressed as a percentage of the fair market value of the Land (i.e., the value of the Land in fee simple, exclusive of the value of any improvements). The annual rent was 6½% of the value of the first tract purchased by the Plan and leased to the Bank in 1967, plus 8% of the value of the adjoining second tract purchased by the Plan and added to the Lease in 1970. The rent was payable monthly. The monthly rent, however, could never fall below $8,453.58 (the initial rent in 1970). The amount of rent was adjusted every ten years. In computing the amount of rent, the cost of the Land was used as its value from the inception of the Lease through January 1, 1978. The Land was appraised as of January 1, 1978, to make the first rent adjustment. It was to be re-appraised as of January 1, 1988, and each ten years thereafter. Pursuant to the January 1, 1979, appraisal, the fair market value of the Land was $2,146,550. Accordingly, rent through December 31, 1987, was set at $11,948.01 per month.

6. The applicants represent that the Lease until June 30, 1984 was covered by the statutory exemption provided by section 414(c)(2) of the Act.[3] The applicants further represent that the leased land is employer real property but is not qualifying employer real property, and therefore the applicants request an exemption in order to continue the Lease after June 30, 1984. The Employer, effective July 1, 1984, executed an agreement (Agreement) with the Plan updating the prior lease.

7. The Agreement provides that the Bank will make additional monthly payments (Additional Payments) to the Plan equal to the amount by which the current fair market rental value of the Land per month exceeds the lesser of (i) the monthly rent payable to the Plan pursuant to the Lease, or (ii) in the event the Bank should ever assign all or a part of its interest in the Lease to an affiliate (as that term is defined in Code section 1504(a)), the actual amount of monthly rent paid to the Plan pursuant to the Lease. The Bank's obligation to make such payments will take effect on July 1, 1984. The Additional Payments made to the Plan from July 1, 1984, to December 31, 1987, shall be $39,029.49 each month. This amount is based on an appraisal of the Land made by Mr. Wilson K. Mason, an unrelated MAI appraiser with the firm of Wilson K. Mason Company, Dallas, Texas. The appraisal, dated June 1, 1984, indicates that the unencumbered fair market value of the Land is $5,826,000, and that the current market rate of return for long term ground leases is between 9.5% and 10.5%. In determining the Additional Payment required during the first three-and-one-half years of the Agreement, the Bank used 10.5%, the highest rate shown in the appraisal. Computation of this Additional Payment is as follows:

| | |
|---|---|
| Value of land | $5,826,000 |
| Market rate of return (percent) | ×10.5 |
| Annual fair market rental value | 611,730 |
| | 12 |
| Monthly fair market rental value | 50,977.50 |
| Monthly rent paid pursuant to lease | −11,948.01 |
| Additional payment required | 39,029.49 |

The Additional Payment will be adjusted at least once every five years to reflect increases, if any, in the fair market rental value of the Land. The first appraisal will be made during calendar year 1987 and shall be effective for the five-year period beginning January 1, 1988. The Agreement requires that other appraisals will then be made every five years thereafter.

8. The Agreement takes effect on July 1, 1984, and automatically terminates on the earlier to occur of the following: (i) Termination of the Lease, or (ii) the sale by the Plan of its complete interest in the Land. If the Lease continues until the end of its term, and the Plan continues to be the fee owner of any portion of the Land, the Bank will be obligated to make payments as required by this Agreement until 2059. If the Bank should assign its interest in the Lease while the Plan owns the Land, the Bank's obligations under the Agreement would continue until an event causing termination of the Lease occurred or the Plan disposed of the Land. The purpose of this provision is to ensure that the Plan will continue to receive all required payments until it no longer has a right to receive such amounts, either because the Lease has expired, or because the Plan has conveyed its complete interest in the Land to another person.

9. The Agreement requires the Bank to indemnify the Plan upon the Plan's sale of its complete interest in the Land if, at the time of such sale, the Land remains encumbered by the Lease. The amount to be paid pursuant to this provision, equals the excess of the current fair market value of the Land, as determined pursuant to an independent appraisal at the time of sale, over the actual sales price received by the Plan. The applicant represents that this provision will ensure that the Plan will receive an amount equal to the fair market value of the Land upon sale, since a prospective purchaser may not be willing to pay as much for the Land encumbered by the Lease as he would if the Land was unencumbered by such Lease.

10. Prior to the effective date of the transaction, the Plan appointed Mr. Raymond L. Thompson (Mr. Thompson) of Dallas, Texas to serve as independent fiduciary with respect to the lease transaction. Mr. Thompson represents that he has 35 years of extensive experience with Prudential in various aspects of real estate, including active participation in the production and property management of Property Investment Separate Account (PRISA) investments.[4] Mr. Thompson is completely independent of, and has no association or relationship with the Bank, the Plan or InterFirst, except as an

[3] The Department expresses no opinion as to the applicability of section 414 of the Act to the prior lease.

[4] Mr. Thompson states that PRISA which invests in equity and debt interests in real estate, is a variable contract account available only to qualified plans.

independent fiduciary with respect to this transaction, and except as to four certificates of deposit and a small checking account at the Bank.[5] In addition, Mr. Thompson has also terminated his employment with Prudential, and his only remaining association with that company is as a recipient of retirement benefits from its pension and medicare supplement plan. Mr. Thompson states that he has consulted with counsel experienced with the duties, responsibilities and liabilities imposed by the Act on plan fiduciaries, and that he understands and acknowledges his duties, responsibilities, and liabilities in acting as a fiduciary with respect to the Plan.

Mr. Thompson in formulating his opinion as to the merits of the leasing transaction, examined the following items:

a. The Lease between the Plan and the Bank.

b. The Agreement between the Bank and the Plan.

c. The appraisal report on the Land prepared by Lonell M. Turner, Jr. and Joe E. Lindsley, effective January 1, 1978, and appraisal reports on the Land prepared by Wilson K. Mason Company, effective January 1, 1982, January 1, 1984, and June 1, 1984.

d. The prohibited transaction exemption application, dated February 15, 1984, and supplementary letter to such application, dated June 25, 1984.

e. The improvements, the Land and the surrounding area near the Land were inspected for quality of maintenance of real estate, development in the area, and general trends of growth.

f. The Plan's investment portfolio.

g. The sub-lease between the Bank and the Joint Venture and the assignment of lease dated September 5, 1974, from the Bank to Prudential.

11. Based on his examination and review of the above items, Mr. Thompson concludes that:

a. The continuation of the leasing transaction (and the execution of the Agreement) will be an excellent investment for the Plan and will be in the best interests of, and protective of, the Plan and its participants and beneficiaries. The additional payments to be made under this Agreement and the monthly rent being paid pursuant to the Lease will equal the current fair market rental value of the Land as of July 1, 1984 and the 10.5% rate of return is currently the fair market rental value of ground leases in the Dallas downtown area.

[5] Mr. Thompson's total investments with the Bank equal $174,000, but total assets of the Bank are approximately $10.5 billion, so his holdings in the Bank represent only .002% of total Bank assets.

b. Mr. Thompson states that the rental payments to the Plan will equal or exceed those obtainable from a similar transaction with an unrelated party. Mr. Thompson also states that he will monitor the Lease and Agreement, assume responsibility for ensuring that the Bank complies with all the terms and conditions contained in the Lease and the Agreement, and take any steps necessary to enforce the rights of the Plan with respect to the subject transaction.

c. The Plan's portfolio of investments is well diversified, and the Land represents a relatively low percentage (less than 10%) of the Plan's total assets.

12. As independent fiduciary, Mr. Thompson will be responsible for deciding whether the Lease and the Agreement continue to be in the best interest and protective of the Plan and its participants and beneficiaries, recommending that the Plan divest itself of the Land if, and when, divestiture becomes appropriate. He will also be responsible for monitoring the terms of the Lease and the Agreement, such monitoring to include verifying the timely payment of rent, confirming that adequate insurance coverage is in effect, overseeing the periodic reappraisal of the Land in accordance with the Lease, appointing appraisers when required to do so by the Agreement, and enforcing those legal remedies that may become necessary or available to the Plan as lessor.

13. In summary; the applicants represent that the transaction satisfied the statutory criteria of section 408(a) of the Act because:

a. The Plan will receive the fair market rental for the Land as determined pursuant to an independent appraisal;

b. The terms and conditions of the Lease and the Agreement have been approved and will be monitored and enforced by Mr. Thompson; and

c. Mr. Thompson represents that the Lease is in the best interests of the Plan and its participants and beneficiaries.

For Further Information Contact: Allen H. Levitas of the Department, telephone (202) 523-8971. (This is not a toll-free number.)

**Muesco, Inc. Profit Sharing Plan (the Plan) Located in Houston, Texas**

[Application No. D-5333]

Proposed Exemption

The Department is considering granting an exemption under the authority of section 408(a) of the Act and section 4975(c)(2) of the Code and in accordance with the procedures set forth in ERISA Procedure 75-1 (40 FR 18471, April 28, 1975). If the exemption is granted the restrictions of section 406(a) and 406 (b)(1) and (b)(2) of the Act and the sanctions resulting from the application of section 4975 of the Code, by reason of section 4975(c)(1) (A) through (E) of the Code shall not apply to the proposed sale of certain real property to the Plan by Muesco, Inc. (the Plan Sponsor) and the proposed leaseback of the Property to the Plan Sponsor provided the terms of the sale and leaseback are as favorable to the Plan as those obtainable in an arms-length transaction with an unrelated party on the date of the consummation of the transactions.

*Summary of Facts and Representations*

1. The Plan is a profit sharing plan with approximately 207 participants. The Plan had total assets of $1,818,201 as of February 29, 1984. The trustees of the Plan are Messrs. William Henry, Harold R. Strickland, William Henry II and J. B. Malley (the Trustees). The Trustees are officers of the Plan Sponsor.

2. The Plan Sponsor manufactures valves and metering equipment for both the energy and ecology fields.

3. The Plan Sponsor owns property located at 8331 Northern Street, Houston, Texas (the Property). Two buildings which contain offices, a machine shop and warehouse facilities are located on the Property. The Plan Sponsor utilized the Property until April, 1980 when its operations were moved to another location. Since that time, the Property has remained vacant. The Plan Sponsor has decided to move its California subsidiary to Houston and plans to reopen assembly and warehousing operations within the Property. The Plan Sponsor is now repairing and upgrading the buildings and parking lots. Air conditioning is being installed in most of the buildings, the offices are being refurbished and the parking lot is being resurfaced. All of the improvements will be paid for by the Plan Sponsor, and no mechanics liens will be filed against the Property.

4. The Plan Sponsor wishes to sell the Property to the Plan for its fair market value of $367,500. This amount represents approximately 23.8% of the Plan's assets. The Plan will pay the entire purchase price in cash at closing.

5. It is then proposed that the Plan lease the Property back to the Plan Sponsor through a triple net, ten-year lease (the Lease). The rent will be $46,920 per annum, for the first three years. This amount is based upon the fair market rental value of the Property as determined by an independent

appraisal. The Property will be reappraised and the rental rate adjusted after the third year and again after the sixth year.

5. An independent appraisal of the Property was performed by Mr. D.L. Pemberton, a real estate appraiser and a senior member of the American Society of Appraisers (the Independent Appraiser). The Independent Appraiser has established the fair market value of the Property at $367,500 as of January 31, 1984 and the fair market rental value of the Property at $46,920 per annum as of January 31, 1984. The valuations by the Independent Appraiser reflect the completion of the improvements on the Property currently being made by the Plan Sponsor.

6. Capital Bank, N.A., will act as the independent fiduciary (the Independent Fiduciary) through its Vice President and Manager of Trust Administration, Ms. Lynne L. Arnold. The Independent Fiduciary has not had any prior business relationships with either the Plan Sponsor or the Plan. Before the sale is consummated, the Independent Fiduciary must approve the completed renovations of the buildings located on the Property. The Independent Fiduciary will establish a trust account for the Plan. Each month, the Plan Sponsor will send its rent payment to the Independent Fiduciary who will deposit the payment in the trust account and subsequently issue a check to the Plan. In the event of default on the Lease, the Independent Fiduciary, acting on behalf of the Plan, shall have, in addition to standard legal remedies, the right to demand the Plan Sponsor repurchase the Property at its then fair market value if a new lessee cannot be found. The Independent Fiduciary shall also monitor payment of taxes by the Plan Sponsor.

7. The Trustees will select a qualified, independent appraiser to reappraise the Property after the third year and after the sixth year of the Lease. In the event the Trustees fail to select a qualified, independent appraiser or such appraiser is not approved by the Independent Fiduciary, the Independent Fiduciary is authorized to select an appraiser to value the Property. The Independent Fiduciary must approve the appraiser's report. After the fair market rental value of the Property has been established, the Independent Fiduciary will notify the Plan Sponsor of the new rental rate in writing and verify that the new rental rate is paid.

8. The Independent Fiduciary has examined both the proposed purchase of the Property by the Plan and the Lease. It believes that the terms of these transactions are in the best interests of the Plan and its participants and beneficiaries. It represents that the rate of return under the Lease is very good, based upon present economic conditions, and the Lease terms are at least as favorable to the Plan as it would have received from an independent, third party leassee in an arm's length transaction. The Independent Fiduciary represents that it has reviewed the Plan's portfolio and has examined the impact that the proposed transactions will have on the diversity of Plan assets, on the Plan's liquidity requirements, and on the overall investment objectives of the Plan and after such review recommends that the Plan enter into the purchase and leaseback of the Property.

9. In summary, the applicant represents that the proposed transactions meet the statutory criteria of section 408(a) because:

(a) the sale of the Property will be for cash;

(b) the Independent Fiduciary will process and monitor the payments and terms under the Lease;

(c) the fair market value and the fair rental value of the Property has been determined by the Independent Appraiser; and

(d) the Independent Fiduciary has determined that the proposed transactions are in the interest of the Plan and its participants and beneficiaries.

For Further Information Contact: Ms. Linda Hamilton of the Department, telephone (202) 523–8881. (This is not a toll-free number.)

**Dynalectron Corporation Pension Trust (the Plan) Located in McLean, Virginia**

[Application No. D–5352]

*Proposed Exemption*

The Department is considering granting an exemption under the authority of section 408(a) of the Act and section 4975(c)(2) of the Code and in accordance with the procedures set forth in ERISA Procedure 75–1 (40 FR 18471, April 28, 1985). If the exemption is granted the restrictions of sections 406(a), 406 (b)(1) and (b)(2) and 407(a) of the Act and the sanctions resulting from the application of section 4975 of the Code, by reason of section 4975(c)(1) (A) through (E) of the Code shall not apply, effective July 1, 1984, to the lease of an improved parcel of real property (the Property) by the Plan to Dynalectron Corporation (Dynalectron), the sponsor of the Plan, provided that the lease is on terms at least as favorable to the Plan as those obtainable by the Plan in an arm's-length transaction with an unrelated party.

Effective Date: If granted, this exemption will be effective July 1, 1984.

*Summary of Facts and Representations*

1. The Plan is a defined benefit plan with 1,325 participants as of March 1, 1984. As of December 31, 1983, the Plan has net assets of $19,317,557. Messrs. C.G. Gulledge, T.E. Blanchard, and J.P. Schelling, officers and/or directors of Dynalectron, serve as the trustees (the Trustees) of the Plan. Pursuant to guidelines established by the Trustees, Redwood Capital Management, Inc. and Chase Investment Counsel Corporation are responsible for investment decisions involving the Plan. The majority of the securities of the Plan are held by the Riggs National Bank of Washington D.C. as custodian. Dynalectron is a Delaware corporation with, as of December, 1983, net assets in excess of $238 million and shareholder's equity in excess of $99 million.

2. The Plan owns the Property located at 6801 Calmont Street, Fort Worth, Texas. The Property consists of a three-story office building located on 48,750 square feet of land. The Plan acquired the Property pursuant to the assignment to the Plan of a purchase contract from Dynalectron in December, 1973, and entered into a ten-year lease with Dynalectron effective January 2, 1974. Dynalectron has been leasing the Property on a month to month basis since the expiration of the initial lease term on December 31, 1983. The Trustees represent that at the time the lease was entered into the lease terms were not favorable to the Plan than terms available with an unrelated party. The applicant represents that this leasing arrangement was provided relief until June 30, 1984, from sections 406 and 407(a) of the Act by virtue of the transitional rule contained in section 414(c)(2) of the Act.[6] The applicant recognizes that the continuing leasing of the Property by the Plan to Dynalectron beyond June 30, 1984, would constitute a prohibited transaction. Accordingly the parties have entered into a new lease (the New Lease), effective July 1, 1984.

3. The Texas American Bank-Fort Worth N.A. (the Bank) has been appointed to serve as the independent fiduciary the Plan with respect to the continuing holding of the Property and lease thereof to Dynalectron. Texas Longhorn Managment Company, Ltd. (Texas Longhorn) has been chosen by the Bank to provide technical assistance and advice to it with respect to the

[6] The Department expresses no opinion herein as to whether the past leasing arrangement is provided relief by section 414(c)(2) of the Act.

transactions. Both the Bank and Texas Longhorn have no business, commercial or other relationships to Dynalectron. The Bank is experienced in matters pertaining to employee benefit plan administration under the Act and understands its fiduciary duties, liabilities, and responsibilities with respect to the Plan. Texas Longhorn is a private company engaged in the business of managing real estate in the Fort Worth area.

4. The New Lease is for a ten year term, effective July 1, 1984, and provides for one five year renewal option exercisable by the lessee. The New Lease is an "absolute net" lease thereby providing that the lessee be responsible for all costs associated with the Property. Mr. Kenneth L. Huffman, MAI, SRPA, located in Forth Worth, Texas, appraised the Property and determined as of October 6, 1983, that the Property had a fair market value of $900,000. The Property represents less than 5% of the assets of the Plan. Mr. Huffman determined that the fair market rental of the Property on an "absolute net" lease basis equals $90,400 per year. This amount will be the initial rental for the first three years of the lease term. The rental will be adjusted at the 37th month and the 85th month of the lease in accordance with appraisals performed by an independent, licensed real estate appraiser selected by the Plan's independent fiduciary.

5. The New Lease provides that any exercise by the lessee of the option to renew will be subject to approval by the Plan's independent fiduciary. The initial rental of the option period will be determined by an independent appraiser selected by the independent fiduciary, and such rental will be adjusted correspondingly at the 37th month of the extension period. In no event will the rental in the succeeding rental periods by less than the rental during the initial ten year period of the New Lease.

6. The Bank has examined the New Lease documents and the appraisal and has determined that the continuing holding of the Property and the New Lease to be appropriate, suitable, and in the best interests of the Plan. The Bank reviewed a lease evaluation by Texas Longhorn and determined that each and every item of the New Lease is appropriate, suitable and in the interest of the Plan. Texas Longhorn will monitor the New Lease and will enforce the terms and conditions of the New Lease on behalf of the Plan.

7. In summary, the applicant represents that the continued holding of the Property and execution of the New Lease satisfies the conditions of section 408(a) of the Act because (a) the Property represents less than 5% of the assets of the Plan; (b) the Bank, an independent, qualified party, with the assistance of Texas Longhorn, an independent real estate company, determined, prior to the execution of the New Lease that the continued holding of the Property and the terms and conditions of the New Lease are appropriate, suitable, and in the best interests of the Plan; and (c) Texas Longhorn will monitor the obligations of Dynalectron under the New Lease and enforce the terms and conditions of the New Lease on behalf of the Plan.

For Further Information Contact: Mr. David Stander of the Department, telephone (202) 523–8881. (This is not a toll-free number.]

**Citation Box and Paper Co. Profit Sharing Plan and Retirement Trust (the Plan) Located in Chicago, Illinois**

[Application No. D–5360]

*Proposed Exemption*

The Department is considering granting an exemption under the authority of section 408(a) of the Act and section 4975(c)(2) of the Code and in accordance with the procedures set forth in ERISA Procedure 75–1 (40 FR 18471, April 28, 1975). If the exemption is granted the restrictions of sections 406(a), 406 (b)(1) and (b)(2) and 407(a) of the Act and the sanctions resulting from the application of section 4975 of the Code, by reason of section 4975(c)(1) (A) through (E) of the Code shall not apply to the continued leasing beyond June 30, 1984 of certain improved real property (the Property) to Citation Box and Paper Co. (the Employer), the sponsor of the Plan, provided that the terms and conditions of such leasing are at least as favorable to the Plan as those which the Plan could receive in a similar transaction with an unrelated party.

Effective Date: If the exemption is granged, the effective date will be July 1, 1984.

*Summary of Facts and Representations*

1. The Plan is a profit sharing plan previously determined by the Internal Revenue Service to meet the requirements of section 401(a) of the Code and consequently the companion trust is exempted under section 501(a) of the Code. As of December 31, 1983, the Plan had 23 participants and assets including the Property of $1,046,246. The trustees of the Plan are UnibancTrust Company (Corporate Trustee, formally Sears Bank and Trust Company), Robert C. Welchko and Samuel LaSpisa, officers of the Employer.

The Employer, an Illinois corporation, is in the business of wholesaling paper boxes and packaging materials with its primary need for commercial property being to warehouse its outstanding inventory. As of June 30, 1983, the Employer had a net worth of approximately $250,000 and an after tax income of $35,525. The Employer made a $40,000 contribution to the Plan for the 1983 Plan year.

2. The Plan initially acquired the Property on November 18, 1971 at a cost of $249,000 and has continuously leased the Property to the Employer since that date. There is no outstanding mortgage on the subject property. The Property consists of a masonry warehouse structure originally built in 1927 with additions made in 1930, 1945 and 1958. The land upon which the improvement is located comprises 76,000 square feet in an area suitable for various warehousing and manufacturing uses. The Corporate Trustee has acted as land trustee for the Plan under a land trust agreement (the Agreement) dated November 3, 1966. The Plan is the sole beneficiary under the terms of the Agreement. The Plan had a beneficial interest in another parcel of real estate under the Agreement at the time it acquired the Property; however, the other real estate was sold in June of 1972. The Employer and the Plan have executed several leases since the Property was originally acquired. The successive leases contained only one substantive change or modification from the prior lease, specifically that of an increased rental rate. The applicant represents that based on the mutual intention of the parties such leases have always constituted a renewal of the former lease and accordingly the leasing arrangement is exempt under section 414(c)(2) of the Act.[7]

3. The applicant is requesting an exemption which will permit the continued leasing (the Lease) of the Property by the Plan to the Employer until June 30, 1994 with three five year options from such date. While the lessee has the initial right to exercise the option provision, the Corporate Trustee, acting for the Plan as lessor, has the right to concur in the exercise of any option by the lessee. The continued leasing of the Property will be subject to the terms and conditions of an existing lease with the following amendment. The Lease provides that for each three year period during the initial ten year term and during each option period thereafter the rental amount would be adjusted based upon an MAI appraisal

[7] The Department expresses no opinion as to whether the existing leasing arrangement is exempt under section 414(c)(2) of the Act.

report as to the then current fair market rental value. In no event during the entire term of the Lease (including the option periods) shall the rent be less than the present fair market rental value of $4,500. The Lease continues to be an absolute net lease requiring the Employer to be responsible for all upkeep, repair, maintenance and taxes. Additionally, the Employer, as lessee, shall maintain liability and extended coverage fire insurance in an amount equal to the full replacement value of the Property. Any installment of rent accruing under the Lease which is not paid when due shall bear interest at a rate of ten percent until it is paid. The Lease also provides that, if, upon default by the lessee of any of its obligations which is not cured within a period of sixty (60) days, then the Plan may terminate the Lease. The Corporate Trustee has obtained a complete appraisal of the current value of the Property from Robert J. Heinzen of Wilmette, Illinois, an independent MAI appraiser. Mr. Heinzen placed a market value on the Property of $250,000 as of March, 1984. This market value resulted in a determination that fair market rental value should be $4,500 per month. Based on the appraised value, the Property represents twenty four (24) percent of Plan assets.

4. The Plan was amended and restated on April 3, 1984 to give the Corporate Trustee the sole discretion to purchase and hold employer real property in the Plan in an amount not to exceed twenty five (25) percent of the value of all Plan assets. The Corporate Trustee also has the sole discretion to lease such employer real property and to monitor any such lease and enforce the rights of the Plan under the terms and conditions of any such lease. The Corporate Trustee represents that it has evaluated the Lease in the context of the entire Plan portfolio and believes that the leasing of the Property is an appropriate and commercially reasonable investment for the Plan. The Corporate Trustee acknowledged that the Property had depreciated from its acquisition price and noted there is currently little market for the Property because of the depressed condition of the surrounding neighborhood. The rate to be paid by the Employer represents a favorable return on the Property and therefore the Corporate Trustee is of the opinion that the continued leasing of the Property is in the best interests of the Plan and its participants and beneficiaries. The Corporate Trustee agrees to monitor the Lease and enforce the Plan's rights for the sole and exclusive benefit of the Plan. The Corporate Trustee notes a minor lending arrangement ($53,000) between itself and the Employer also indicates that such loan is minimal percentage of its $144 million out standing in commercial loans. The Corporate Trustee considers itself to be independent of the Employer and quite capable of serving as an impartial trustee for the Plan.

5. The Lease terms include a provision which enables the Corporate Trustee acting for the Plan to sell the Property at any time; however, the Lease further states that if the Property is considered for sale that the Employer be given written notice of such intention and offered an option to purchase the Property for the same price and upon whatever terms and conditions the Plan might otherwise offer the Property for sale to a third party.[8] The Employer shall have thirty (30) days from the receipt of written notice to indicate its intentions regarding the future purchase of the Property.

6. In summary, the applicant represents that the proposed leasing arrangement satisfies the exemption criteria set forth in section 408(a) of the Act for the following reasons: (a) The initial rental rate has been determined by a qualified independent appraiser and such rate will be redetermined every three years by an independent appraiser selected by the Corporate Trustee; (b) the Corporate Trustee, an independent fiduciary, believes the leasing arrangement is appropriate for and in the best interest of the Plan and its participants and beneficiaries; and (c) the Corporate Trustee will exercise authority and control over and have responsibility for the operation of the Lease.

For Further Information Contact: Paul R. Antsen of the Department, telephone (202) 523–6915. (This is not a toll-free number.)

**Employees' Retirement Plan of the Bulova Watch Company, Inc. (the Plan) Located in Flushing, New York**

[Application No. D–5458]

*Proposed Exemption*

The Department is considering granting an exemption under the authority of section 408(a) of the Act and section 4975(c)(2) of the Code and in accordance with the procedures set forth in ERISA Procedure 75–1 (40 FR 18471, April 28, 1975). If the exemption is granted the restrictions of section 406(a), 406 (b)(1) and (b)(2) and 407 (a) of the Act and the sanctions resulting from the application of section 4975 of the Code, by reason of section 4975(c)(1) (A) through (E) of the Code, shall not apply to the continuation beyond June 30, 1984 of a lease of certain real property (the Property) by the Plan to Bulova Systems and Instruments Corporation (BSIC), provided that such lease is on terms and conditions at least as favorable to the Plan as those obtainable by the Plan in an arm's-length transaction with an unrelated party, and the possible cash sale of the Property by the Plan to BSIC or the Bulova Watch Company, Inc. (Bulova), pursuant to the terms of the lease, provided that the Plan receives no less than fair market value for the Property at the time of any subsequent sale to BSIC or Bulova.

Effective Date: If the proposed exemption is granted, it will be effective July 1, 1984.

**Summary of Facts and Representations**

1. The Plan is a defined benefit plan with approximately 1,778 participants. As of December 31, 1983, the Plan had net assets of approximately $75,575,627. Bulova, the Plan sponsor, distributes and sells watches and clocks for consumer use, timepiece parts and power cells. BSIC, a wholly-owned subsidiary of Bulova, manufactures and sells fuzes for national defense.

2. On January 9, 1969, Bulova's Board of Directors authorized the sale of the Property to the Plan for $1,900,000. The Property consists of certain real estate, buildings, and appurtenances thereto, located in Valley Stream, New York. On May 9, 1969, the Plan leased the Property back to Bulova for a period of 20 years. The annual rent under that lease (the Original Lease) was $170,445.16. The Original Lease was amended on November 5, 1973 to increase the annual rent to $180,175.68 in consideration of the Plan's expenditure of $86,000 for improvements to the Property. On October 27, 1976, Bulova sublet the Property to BSIC for a term commencing October 1, 1976 and expiring September 30, 1986, at a net annual rental of $180,175.68. BSIC has been in possession of the Property since October 1, 1976.

3. Bulova asserts that the Original Lease of the Property was a lease involving a party in interest pursuant to a binding contract in effect on July 1, 1974, as defined under sections 414(c)(2) and 2003(c)(2)(B) of the Act, and therefore was statutorily exempt until June 30, 1984 from the prohibitions of

[8] This proposed exemption provides no relief with respect to any future sale of the Property to the Employer or some other party in interest. However, at the time such sale is contemplated, either the Plan or any party in interest involved therein may apply for an administrative exemption pursuant to the procedures set forth in ERISA Procedure 75–1 (40 FR 18471, April 28, 1975).

sections 406 and 407 of the Act by virtue of sections 414(c)(2) and 2003(c)(2)(B) of the Act.[9] Bulova recognized that the statutory exemption for the Original Lease would expire on June 30, 1984, and therefore entered into a new lease (the New Lease) which would extend beyond that date. Bulova assigned to BSIC all of its right, title and interest as tenant under the Original Lease, thereby extinguishing the sublease, so that the New Lease is between the Plan and BSIC.

4. The New Lease will continue until May 8, 1989. The interest of the Plan under the New Lease for all purposes is represented by Security Pacific National Bank (the Bank), located in New York, New York. The Bank represents that it is independent of Bulova and BSIC, and that it is an institutional fiduciary experienced in serving as a fiduciary of employee retirement plans under the Act. The initial annual rental under the New Lease is $665,000, such amount having been determined to be the fair market rental value of the Property on May 17, 1984 by James Felt Realty Services (Felt), an independent appraiser located in New York, New York. The New Lease provides for a yearly review of the annual rental to reflect the increase, if any, but not any decrease, in the fair market rental value of the Property. Such review will be conducted by an independent real estate appraiser selected by the Bank. BSIC will bear the cost of the yearly appraisal. All rent payments are net to the Plan; all taxes, maintenance and other expenses are to be paid by BSIC. In addition, BSIC is obligated by the terms of the New Lease to carry comprehensive general liability and hazard insurance on the Property for the protection of the Plan and to protect the Plan from any damage or waste to the Property.

5. Under the terms of the New Lease, BSIC has the option to purchase the Property for cash upon giving the Plan not less than 30 days notice of its intention to purchase the Property. The applicants represent that no sale of the Property shall take place between BSIC or Bulova and the Plan without the independent fiduciary determining that the consideration to be received by the Plan is at least as favorable to the Plan as the Plan can obtain in a sale with an unrelated party. Further, the applicants represent that no such sale shall take place unless and until the Bank has determined that such sale is appropriate for the Plan and in the best interests of its participants and beneficiaries.

6. The Bank will monitor on behalf of the Plan the performance of BSIC under the New Lease and will represent the Plan in the enforcement of the terms and conditions of the New Lease. The Bank represents that it has examined the terms and conditions of the New Lease, including appraisals of the Property's fair market value and fair market rental value, and has determined that, as of June 30, 1984, the terms are equivalent to arm's-length terms between unrelated parties. The Bank represents that it has determined that the New Lease arrangement between the Plan and BSIC is in the interest of the Plan and its participants and beneficiaries. The fair market value of the Property has been appraised by Felt, which represents that as of May 17, 1984, the Property had a fair market value of approximately $5,000,000. Thus, the Property represents approximately 7% of the Plan's total assets.

7. In summary, the applicants represent that the New Lease satisfies the criteria of section 408(a) of the Act because: (1) The New Lease is a triple net lease requiring BSIC to pay all costs of repair and maintenance and all taxes and insurance on the Property; (2) the interests of the Plan under the New Lease are represented by the Bank, an independent fiduciary which is unrelated to BSIC and Bulova and which will monitor performance of the terms and conditions of the New Lease on behalf of the Plan; (3) the New Lease will require periodic appraisals to ensure that the rental remains at least the fair market rental value of the Property; (4) the Bank has evaluated the New Lease and determined that it is appropriate for the Plan and in the best interests of the Plan's participants and beneficiaries; and (5) no sale of the Property by the Plan to BSIC or Bulova pursuant to the terms of the lease will take place unless and until the Bank has determined that such sale is in the best interest of the Plan.

For Further Information Contact: Gary H. Lefkowitz of the Department, telephone (202) 523-8881. (This is not a toll-free number.)

[9] The Department expresses no opinion as to whether the Original Lease constituted a lease pursuant to a binding contract in effect on July 1, 1974 as defined under sections 414(c)(2) and 2003(c)(2)(B) of the Act or whether the Original Lease was statutorily exempt until June 30, 1984 from the prohibitions of sections 406 and 407 of the Act and section 4975 of the Code by virtue of Sections 414(c)(2) and 2003(c)(2)(B) of the Act.

**Forest Oil Corporation Pension Trust (the Plan) Located in Bradford, Pennsylvania**

[Application No. D-5629]

*Proposed Exemption*

The Department is considering granting an exemption under the authority of section 408(a) of the Act and section 4975(c)(2) of the Code and in accordance with the procedures set forth in ERISA Procedure 75-1 (40 FR 18471, April 28, 1975). If the exemption is granted the restrictions of section 406(a), 406(b)(1) and (b)(2) and 407(a) of the Act and the sanctions resulting from the application of section 4975 of the Code, by reason of section 4975(c)(1) (A) through (E) of the Code shall not apply to the lease (the Lease), effective June 30, 1984, of a parcel of improved real property (the Property) by the Plan to Forest Oil Corporation (the Employer), the sponsor of the Plan, provided the terms of the transaction are no less favorable to the Plan than those obtainable in an arm's length transaction with an unrelated third party.

Effective Date: The effective date of this proposed exemption, if granted, will be June 30, 1984.

**Summary of Facts and Representations**

1. The Plan is a defined benefit plan with 484 participants. As of May 31, 1983, the Plan has net assets of $17,549,490. The trustees of the Plan (the Trustees) are William F. Higie, Everardo Goyannes and W. Jeffrey Burford, all of whom are employees of the Employer.

2. The Property is a 0.482 acre parcel improved with a three-story 21,774 square foot office building located at 405 North Marienfeld Street, Midland, Texas. The Plan has owned the Property since November 1, 1973, and has leased it to the Employer at all times since that date (the 1973 Lease). The applicant represents that under the transitional rules of section 414 (c) of the Act, the 1973 Lease does not constitute a prohibited transaction.[10]

3. An exemption is requested, effective June 30, 1984, for the new Lease between the Plan and the Employer. The Lease will be of 10 years duration with two five year renewal options exercisable by Texas Commerce Bank, N.A. (the Bank), the Plan's independent fiduciary (see below). The annual rent during the initial 12 months of the Lease is $208,980, payable monthly in advance. On the first anniversary of the Lease

[10] In this proposed exemption, the Department expresses no opinion as to the applicability of section 414 (c) of the Act to the 1973 Lease.

and every anniversary thereafter during the initial 10 year term of the Lease, the rental rate shall be increased by an amount equal to the rental for the preceding twelve calendar month period multiplied by the percentage change in the Consumer Price Index (the CPI), or comparable index if the CPI is discontinued, that has occurred between the beginning of the preceding twelve calendar month period and the end of such period. The rental rate for the two successive five year renewal periods shall be determined as provided above, with the exception that the rental rate for the initial twelve calendar month period of each renewal term shall be determined by two independent appraisers selected by the Bank and paid by the Employer. There will be no decrease in the rental rate resulting from any decrease in the CPI, or comparable index if the CPI is discontinued. The Lease is triple net with the Employer responsible for the payment of all taxes, utilities, maintenance, repairs and insurance costs. The Employer will purchase and maintain, during the entire term of this Lease, fire and extended coverage insurance on the Property, for the full replacement value and naming the Employer and the Plan, as their respective interests shall appear, as insureds.

4. On May 31, 1984, Keith Barlow, MAI, appraised the Property and determined that it had a fair market value of $2,000,000. On June 25, 1984, Mr. Barlow conducted a market rent study of the Property and determined that as of that date, the fair market rental rate for the Property on a triple net basis was $208,980 per year. Mr. Barlow further determined that annual rental adjustments based on the Consumer Price Index and a ten year initial term with two additional and successive five year renewal terms were appropriate terms and conditions for the Lease.

5. The Bank has accepted appointment as independent fiduciary on behalf of the Plan with respect to the Lease transaction. The parent holding company of the Bank is the twentieth largest banking organization in the United States. The trust department of the Bank is the largest of the 17 trust departments in the holding company organization and is responsible for over $8 billion in assets. The Bank represents that there is no commercial banking relationship between the Employer, the Trustees and the Bank nor is there any relationship of common ownership of stock or securities. The Bank further represents that there are no joint ventures between either the Employer or the Trustees and the Bank and that no officer or employee of the Employer sits on any decision making committee of the Bank.

6. By letter dated June 29, 1984, the Bank represents that the Lease is in the best interests of and protective of the rights of the participants and beneficiaries of the Plan. In reaching this determination the Bank reviewed the Lease and Mr. Barlow's appraisals. The Bank also consulted with several other appraisers familiar with the Midland real estate market. In addition, officers of the Bank conducted an on-site inspection of the Property and reviewed the financial statements of the Plan and the Employer. Based upon this information the Bank determined the terms of the Lease are at least as favorable to the Plan as an arm's length transaction with an unrelated party. The Bank further represents that: (a) The average rate of return, assuming a conservative annual average growth in the CPI of 5% per year is projected, based on income alone, at 13.14% per annum, which compares favorably with a typical fixed income investment, in addition the Bank projects an overall average annual rate of return of 19.35% when including both rental income and growth in the value of the Property; (b) the Lease is more favorable to the Plan than most comparable leases in the Midland market because it is triple-net with annual rental increases floating with changes in the CPI and because the entire Property is under lease to a single tenant when the normal vacancy rate in the Midland market is 20%; (c) there is no current reason why it would be prudent, necessary or expedient for the Plan to sell the Property where the Plan has no liquidity problems, the Plan's portfolio is stable, the Property adds diversity and the potential for capital gain to the Plan's portfolio and the growth in value of the Property makes it one of the most attractive assets of the Plan; and (d) based upon the Employer's financial information, the Bank concludes that the Employer will be a satisfactory tenant capable of fulfilling its lease obligations. In addition, the Bank represents that it will monitor and enforce the terms and conditions of the Lease on behalf of the participants and beneficiaries of the Plan.

7. In summary, the applicant represents that the proposed Lease meets the statutory requirements of section 408(a) of the Act because: (a) The initial rent under the Lease is fair market value rent as determined by a qualified, M.A.I. appraiser; (b) the rent shall be adjusted annually for any rise in the CPI; (c) the Lease may be renewed only if the Bank, acting as independent fiduciary on behalf of the Plan, approves the renewal; (d) the initial rent during any renewal period will be determined by two M.A.I. appraisers; and (e) the Bank, acting on behalf of the Plan, determined that the Lease was in the best interests and protective of the participants and beneficiaries of the Plan.

For Further Information Contact: David M. Cohen of the Department, telephone (202) 523–8671. (This is not a toll-free number.)

**General Information**

The attention of interested persons is directed to the following:

(1) The fact that a transaction is the subject of an exemption under section 408(a) of the Act and/or section 4975(c)(2) of the Code does not relieve a fiduciary or other party in interest or disqualified person from certain other provisions of the Act and/or the Code, including any prohibited transaction provisions to which the exemption does not apply and the general fiduciary responsibility provisions of section 404 of the Act, which among other things require a fiduciary to discharge his duties respecting the plan solely in the interest of the participants and beneficiaries of the plan and in a prudent fashion in accordance with section 404(a)(1)(B) of the Act; nor does it affect the requirement of section 401(a) of the Code that the plan must operate for the exclusive benefit of the employees of the employer maintaining the plan and their beneficiaries;

(2) Before an exemption may be granted under section 408(a) of the Act and/or section 4975(c)(2) of the Code, the Department must find that the exemption is administratively feasible, in the interests of the plan and of its participants and beneficiaries and protective of the rights of participants and beneficiaries of the plan; and

(3) The proposed exemptions, if granted, will be supplemental to, and not in derogation of, any other provisions of the Act and/or the Code, including statutory or administrative exemptions and transitional rules. Furthermore, the fact that a transaction is subject to an administrative or statutory exemption is not dispositive of whether the transaction is in fact a prohibited transaction.

(4) The proposed exemptions, if granted, will be subject to the express condition that the material facts and representations contained in each application are true and complete, and that each application accurately describes all material terms of the

transaction which is the subject of the exemption.

Signed at Washington, D.C., this 23rd day of October, 1984.

**Elliot I. Daniel,**

*Acting Assistant Administrator for Regulations and Interpretations, Office of Pension and Welfare Benefit Programs, U.S. Department of Labor.*

[FR Doc. 84-28348 Filed 10-25-84; 8:45 am]

BILLING CODE 4510-29-M

## NATIONAL SCIENCE FOUNDATION

### Advisory Panel for Social/Cultural Anthropology; Meeting

In accordance with the Federal Advisory Committee Act, Pub. L. 82-463; as amended, the National Science Foundation announces the following meeting:

Name: Advisory Panel for Social/Cultural Anthropology.

Date and Time: November 13-14, 1984; 9:00 a.m.—5:00 each day.

Place: Holiday Inn, 1450 Glenarm, Denver, CO 80202, Room No.—Not yet assigned.

Type of Meeting: Closed.

Contact Person: Dr. Daniel R. Gross, Anthropology Program, Room 320, National Science Foundation, Washington, DC 20550, (202) 357-7804.

Purpose of Advisory Panel: To provide advice and recommendations concerning support for research in social and cultural anthropology.

Agenda: To review and evaluate research proposals as part of the selection process for awards.

Reason for Closing: The proposals being reviewed include information of a proprietary or confidential nature, including technical information, financial data, such as salaries, and personal information concerning individuals associated with the proposals. These matters are within exemptions (4) and (6) of 5 U.S.C. 552b(c), Government in the Sunshine Act.

Authority to Close Meeting: This determination was made by the Committee Management Officer pursuant to provisions of section 10(d) of Pub. L. 92-463. The Committee Management Officer was delegated the authority to make such determinations by the Director, NSF, on July 6, 1979.

Dated: October 23, 1984.

**M. Rebecca Winkler,**

*Committee Management Officer.*

[FR Doc. 84-28336 Filed 10-25-84; 8:45 am]

BILLING CODE 7555-01-M

## NUCLEAR REGULATORY COMMISSION

### Advisory Committee on Reactor Safeguards; Meeting

In accordance with the purposes of sections 29 and 182b. of the Atomic Energy Act (42 U.S.C. 2039, 223b.), the Advisory Committee on Reactor Safeguards will hold a meeting on November 1-3, 1984, in Room 1046, 1717 H Street, NW., Washington, DC. Notice of this meeting was published in the **Federal Register** on September 18, 1984.

The agenda for the subject meeting will be as follows:

*Thursday, November 1, 1984*

*8:30 a.m.—8:45 a.m.: Report of ACRS Chairman (Open)*—The ACRS Chairman will report briefly on items of current interest.

*8:45 a.m.—11:00 a.m.: Consideration of Severe Accidents (Open)*—The members will discuss proposed ACRS comments regarding the frequency and severity of accidents resulting in severe core damage.

*11:00 a.m.—12:00 Noon: Water Chemistry in BWRs (Open)*—The members will hear a report regarding control of water chemistry in BWRs to help control pipe cracking in the primary system.

Portions of this session may be closed to discuss Proprietary Information applicable to this subject.

*1:00 p.m.—2:00 p.m. NRC Severe Accident Policy (Open)*—The members of the Committee will discuss the latest revision of the proposed NRC policy statement on severe accident issues in nuclear power plant regulation. Representatives of the NRC Staff will participate as appropriate.

*2:00 p.m.—6:00 p.m.: Limerick Generating Station, Units 1 and 2 (Open)*—The members of the Committee will discuss the resolution of items required for approval of a full power license for this facility. Aspects of the probabilistic risk assessment for this facility will also be discussed.

Portions of this session will be closed as required to discuss Proprietary Information related to this matter.

*Friday, November 2, 1984*

*8:30 a.m.—9:30 a.m.: Emergency Planning (Open)*—The members will hear the report of the cognizant ACRS Subcommittee Chairman regarding consideration of seismic events in emergency planning (10 CFR 50, 50.47, and Appendix E).

*9:30 a.m.—10:00 a.m.: Topics for ACRS-NRC Commissioners' Meeting (Open)*—The members of the Committee will discuss proposed ACRS comments and recommendations regarding the proposed NRC policy statement on consideration of severe accidents in the regulation of nuclear power plants.

*10:00 a.m.—11:30 a.m.: Meeting with NRC Commissioners (Open)*—The members of the Committee will discuss ACRS comments regarding the proposed NRC policy statement on severe accidents.

*11:30 a.m.—12:30 p.m.: ACRS Subcommittee Activities (Open/Closed)*—The members will hear and discuss the reports of designated ACRS Subcommittee members regarding recent activities including those related to decay heat removal provisions in nuclear plants and decommissioning of the Humbolt Bay Nuclear Power Plant.

Portions of this session will be closed as necessary to discuss information provided in confidence by a foreign source and Proprietary Information related to the matters being discussed.

*1:30 p.m.—2:30 p.m.: Activities of the NRC Office of Inspection and Enforcement (Open)*—The members of the Committee will hear and discuss a report by the Director, IE, regarding activities of the NRC Office of Inspection and Enforcement.

*2:30 p.m.—3:00 p.m.: Future Activities (Open)*—The members will discuss anticipated ACRS Subcommittee activities and proposed items for consideration by the full Committee.

*3:00 p.m.—5:30 p.m.: Preparation of ACRS Reports (Open)*—The members of the Committee will discuss proposed reports to the NRC regarding matters considered during this meeting.

Portions of this session will be closed as necessary to discuss Propietary Information applicable to the topics being discussed.

*Saturday, November 3, 1984*

*8:30 a.m.—12:30 p.m.: ACRS Reports to NRC (Open)*—The members will discuss proposed ACRS reports regarding matters considered during this meeting.

Portions of this session will be closed as necessary to discuss Proprietary Information applicable to the matters being discussed.

Procedures for the conduct of and participation in ACRS meetings were published in the **Federal Register** on October 3, 1984 (49 FR 193). In accordance with these procedures, oral or written statements may be presented by members of the public, recordings will be permitted only during those portions of the meeting when a transcript is being kept, and questions may be asked only by members of the Committee, its consultants, and Staff. Persons desiring to make oral statements should notify the ACRS Executive Director as far in advance as practicable so that appropriate arrangements can be made to allow the necessary time during the meeting for such statements. Use of still, motion

picture and television cameras during this meeting may be limited to selected portions of the meeting as determined by the Chairman. Information regarding the time to be set aside for this purpose may be obtained by a prepaid telephone call to the ACRS Executive Director, R.F. Fraley, prior to the meeting. In view of the possibility that the schedule for ACRS meetings may be adjusted by the Chairman as necessary to facilitate the conduct of the meeting, persons planning to attend should check with the ACRS Executive Director if such rescheduling would result in major inconvenience.

I have determined in accordance with Subsection 10(d) Pub. L. 92–463 that it is necessary to close portions of this meeting as noted above to discuss Proprietary Information (5 U.S.C. 552b(c)(4)) and information provided in confidence by a foreign source (5 U.S.C. 552b(c)(4)).

Further information regarding topics to be discussed, whether the meeting has been cancelled or rescheduled, the Chairman's ruling on requests for the opportunity to present oral statements and the time allotted can be obtained by a prepaid telephone call to the ACRS Executive Director, Mr. Raymond F. Fraley (telephone 202/634–3265), between 8:15 a.m. and 5:00 EST.

Dated: October 22, 1984.

**John C. Hoyle,**

*Advisory Committee Mangement Officer.*

[FR Doc. 84–28327 Filed 10–25–84; 8:45 am]

BILLING CODE 7590–01–M

## Advisory Committee on Reactor Safeguards Combined Subcommittees on Reactor Operations and Reliability and Probabilistic Assessment; Meeting

The ACRS Subcommittees on Reactor Operations and Reliability and Probabilistic Assessment will hold a combined meeting on November 14, 1984, Room 1046, 1717 H Street, NW, Washington, DC.

The entire meeting will be open to public attendance.

The agenda for subject meeting shall be as follows:

*Wednesday, November 14, 1984—9:00 a.m. until the conclusion of business.*

The Subcommittees will discuss the current status of the work related to steam generator overfill, systems interaction, and the NRC Staff's work on the generic issue related to, "piping and use of highly combustible gases in vital areas".

Oral statements may be presented by members of the public with the concurrence of the Subcommittee Chairman; written statements will be accepted and made available to the Committee. Recordings will be permitted only during those portions of the meeting when a transcript is being kept, and questions may be asked only by members of the Subcommittee, its consultants, and Staff. Persons desiring to make oral statements should notify the ACRS staff member named below as far in advance as practicable so that appropriate arrangements can be made.

During the initial portion of the meeting, the Subcommittee, along with any of its consultants who may be present, may exchange preliminary views regarding matters to be considered during the balance of the meeting. The Subcommittee will then hear presentations by and hold discussions with representatives of the NRC Staff, Subcommittee consultants, and other interested persons regarding this review.

Further information regarding topics to be discussed, whether the meeting has been cancelled or rescheduled, the Chairman's ruling on requests for the opportunity to present oral statements and the time allotted therefor can be obtained by a prepaid telephone call to the cognizant ACRS staff member, Mr. Richard Major (telephone 202/634–1413) between 8:15 a.m. and 5:00 p.m., EDT. Persons planning to attend this meeting are urged to contact the above named individual one or two days before the scheduled meeting to be advised of any changes in schedule, etc., which may have occurred.

Dated: October 22, 1984.

**Morton W. Libarkin,**

*Assistant Executive Director for Project Review.*

[FR Doc. 84–28328 Filed 10–25–84; 8:45 am]

BILLING CODE 7590–01–M

**[Docket Nos. 50–369 and 50–370]**

## Duke Power Co.; Environmental Assessment and Finding of No Significant Impact

The U.S. Nuclear Regulatory Commission (the Commission or staff) is considering approval of a procedure to dispose of certain very low level radioactive waste pursuant to 10 CFR 20.302 proposed by Duke Power Company (the licensee) for the William B. McGuire Nuclear Station, Units 1 and 2, located in Mecklenburg County, North Carolina.

### Environmental Assessment

*Identification of Proposed Action:* The proposed action would approve the periodic removal and relocation of sludge consisting of slightly contaminated water treatment residues from the initial holdup pond of the wastewater treatment system and the disposal of this sludge at a state-approved landfarming site contiguous to the McGuire Station site. The sludge (residues) would be spread about six inches deep over a surface area of no more than one acre each year to dry and then incorporated into the underlying six inches of soil. After incorporation, a covering of topsoil would be applied as needed to support establishment of a year-round vegetative cover. The proposed action is in accordance with the licensee's request by letter dated June 18, 1984, as supplemented July 18, 1984.

*The Need for the Proposed Action:* Water treatment systems needed to support operation of nuclear power plants create residues containing low-levels radioactivity or other contaminants which require periodic disposal. Sanitary, potable, and demineralized water systems at McGuire Nuclear Station are supplied with water that has been filtered through diatomaceous earth (DE) pressure filters. There are two DE filters, each with a capacity of 750 gpm for 24 hours, but normal filtered water usage requires operation of one filter at 750 gpm approximately 6 hours per day. The filters are backwashed as needed. With each backwash, 0.15 cubic yard of spent DE is flushed to the conventional wastewater treatment system.

The McGuire condensate demineralization system, which removes impurities from the steam cycle water, consist of four mixed bed demineralizer filters that use powdered ion-exchange resin. Under normal operation, three condensate demineralizers are operated continuously and each cell is backwashed every 10 days. With each backwash, 0.67 cubic yard of spent resin is flushed to the conventional wastewater treatment system.

Raw water intended for condensate makeup at McGuire is pretreated to remove chlorine and organic material. Two 270 $ft^3$ beds of organic removal type activated charcoal are used for this purpose and generate approximately 540 $ft^3$ of spent charcoal per year.

All non-radioactive McGuire Station waste streams, except sanitary waste, are routed to the conventional wastewater treatment system, which uses sedimentation, chemical addition, and aeration to treat the wastewater prior to discharge to the Catawba River via the National Pollutant Discharge Elimination System (NPDES) discharge point. Primary sedimentation occurs in the initial holdup pond, a 200,000-gallon

concrete basin. Spent powered resin, charcoal, and DE accumulate in this pond and must be removed periodically to maintain proper settling and retention of wastewater, thus enabling the system to comply with its NPDES permit limits for Total Suspended Solids of 30 mg/l daily average concentration and 100 mg/l daily maximum concentration.

The sludge collected in the initial holdup pond is a semi-solid mixture of powdered resins, diatomaceous earth and associated residues. The resins are styrene divinylbenzene polymers and contain the ions removed in the condensate demineralization system. The radioactivity concentrations of the sludge are very low, consisting of Co–58 and Co–60, about 0.12 pCi/cm³ of each. The volume of sludge being generated per year is projected to range from 8,500 to 13,500 cubic feet with a total activity of 0.05 mCi of Co–58, and 0.05 mCi of Co–60.

**Environmental Impacts of the Proposed Action**

*A. Licensee's Procedures to Minimize Potential Adverse Environmental Impact*

The environmental assessment by the staff recognizes the following aspects of the licensee's procedures for transportation, disposal and administration to minimize or preclude potential adverse environmental impacts:

*Transportation Procedure*

- To remove this sludge, the pond shall be drained and the sludge dredged from the bottom and moved by dump truck to the disposal site.
- The sludge shall be transported to or from the disposal site in such a way that liquid or solid spills will be kept to a minimum.
- The preparation and shipment of radioactive material shall be in accordance with Station health physics procedures and Station directives.

*Disposal Procedure*

- During and after the disposal process, access to the proposed disposal site shall be controlled. Proper warnings shall be maintained as described in the state Landfarming Permit for Water Treatment Residues (7641R2, September 7, 1984).
- The waste sludge (water treatment residues) shall be spread on the surface of the proposed disposal site over an approximate area and depth of one acre and six inches, respectively.
- The sludge shall be incorporated approximately six inches into the soil after drying to the extent practical.
- A suitable year-round vegetative cover shall be established and maintained after the waste has been incorporated and covered with topsoil as needed to support this vegetative cover.
- The workers handling the waste disposal shall be dressed and trained in accordance with Station health physics procedure and Station directives.

*Administration Procedure*

- The waste volume of each batch disposed shall be established and documented and records maintained.
- For each batch of waste generated, a composite sample from different locations shall be taken for radiological analysis, and results shall be documented and records maintained. The analysis of the sludge shall be obtained before it is transferred, and no batch shall be landspread if its average Co–60 content exceeds 0.5 pCi/cm³.
- The total accumulated waste volume and radioactivity inventories shall be documented and records maintained.
- The disposal rates shall be limited to 500 cubic yards per year (6 inches on no more than one acre each year).
- Provisions shall be taken and maintained to prevent wind erosion and surface runoff from conveying pollutants from the waste material application disposal area onto the adjacent property.
- Upon retirement, the site shall be covered with topsoil, as necessary to support revegetation, and grassed.
- The operational procedure to minimize the risk of unexpected or hazardous exposures shall follow the guidelines provided by System Health Physics Manual and Station directives on radiation exposure control and radioactive material control. All radioactive-waste release and disposal operations shall be performed under the technical guidance and review of the Station Health Physicist.

*B. Radiological Impacts*

Licensee's analyses of the sludge indicate that the content of licensed material is only about 0.12 pCi Co–58 and 0.12 pCi Co–60 per cubic centimeter. The staff's evaluation of potential gamma radiation exposure from such material is that a person spending 2000 hours per year in an effectively infinite area of such contamination would receive a dose less than 1.0 mrem/year. Incorporating the sludge in the underlying soil and/or covering it with topsoil would reduce the exposure rate from the contamination. Such an exposure rate is, in any case, insignificant compared to the background exposure from naturally occurring radioisotopes in average soils.

The staff also estimated potential doess from ingestion of vegetables grown in such contamination; the largest potential organ dose would be less than 0.1 mrem/year.

Similarly, the staff estimated that a worker inhaling airborne dust with such contamination levels would receive a maximum dose to the lung much less than 0.1 mrem per year even if exposed 2000 hours per year to ten times the EPA Total Suspended Particulates standard of 260 $\mu g/m^3$, all respirable. Doses to other internal organs would be smaller.

The staff also judges, based on the information furnished in the licensee's letter of June 18, 1984, relative to the hydrology and geology of the site and on the control measures proposed, that the disposal would not create any significant radiological contamination hazard for either surface water or groundwater users.

Because the doses estimated by the staff are so small, even though made with conservative assumptions, the staff also judges that the proposed disposal would be acceptable if the concentrations of licensed material in the sludge were as much as ten times higher. In application of the principle of keeping radiation exposures as low as reasonably achievable, the staff, as noted above, would condition approval such that the concentration of Co–60 in sludge to be landspread would be limited to 0.5 pCi/cm³ or less.

Based on review and evaluation of the proposed disposal, the staff concludes that:

(1) The radiation risks to workers involved in the disposal would be small compared to the routine occupational exposures at the McGuire Nuclear Station.

(2) The possible radiation risks to members of the general public as a result of such disposal would be well below regulatory limits and small in comparison to the doses they receive each year from natural background radiation.

The staff further notes that at such very low levels of radioactivity, no change in decommissioning aspects of the facility and only insignificant changes in the handling or transport of radioactive material (sludge) would be associated with this proposed action.

*C. Non-radiological Impacts*

Because of the characteristics of the disposal location selected and the controls imposed by the permit from the State of North Carolina, no impact would be expected on utilized supplies of groundwater or surface waters.

Because of the water-retaining characteristics of the material to be spread, impacts associated with inhalation of windblown material (dust) will be minimal. The chance of exposure of members of the public to any inhalation effect is further reduced by the licensee's control of access to the site.

*D. Land Use Impacts*

The proposed landfarming site is located northwest of the McGuire conventional treatment system outside the security fence but within the company controlled area. It is about 1700 feet due west of the Unit 1 turbine building. The entry to the area is controlled by fencing and is accessible only from the station site. The proposed disposal site is approximately 600 feet long and 450 feet wide, and is used for deposition of soil removed during construction activities at McGuire Nuclear Station.

The proposal by the licensee is to landfarm not more than one acre per year with sludge. After the sludge has been incorporated into the underlying six inches of soil, the disturbed site will be covered with topsoil as needed to support vegetation and a suitable year-round vegetative cover will be established and maintained. This process will continue periodically until the remaining available disposal site area has been depleted. Upon retirement, the disposal site will be covered with topsoil, if needed to support vegetation, and grassed.

The staff finds that the changes in land usage from disposal of excess soil material during contruction at the site to use as a landfarming site for disposal of very low level radioactivity waste, as proposed by the licensee, would not significantly alter the character or appearance of the temporarily disturbed disposal site.

*Alternatives to the Proposed Action:* An alternative to landfarming would be landfilling. Because the sludge is slow to dry, it is unsuitable for landfilling, where the waste must be covered with soil the same day it is deposited. It is preferable to landspread the sludge at a suitable site and incorporate it into the soil after it has dried.

The "no action" alternative is essentially the same as that associated with denial of the licensee's request for approval; namely that other, more costly alternatives for disposal of the sludge would be necessary to support continued plant operation. The licensee estimates that this sludge (10,000 ft $^3$), if packaged and disposed of as radioactive waste, would cost approximately $750,000 without solidification, and would cost more than $1,140,000 if solidification were required depending on radioactive waste packaging and waste form requirements. The actual burial space would be more than 12,000 ft $^3$ in the licensed radioactive waste burial site. Considering the generation rate of this type of waste (8,500 to 13,500 ft $^3$ per year), the total cost saving using the proposed landfarming procedure could range from $620,000 to $1,500,000 per year with a burial site space saving of 10,000 to 15,000 cubic feet per year.

The "no action" alternative also entails increased risks during transportation associated with off-site shipments, whereas transport for the proposed action is for short distances entirely within controlled access areas.

*Alternative Use of Resources:* The principal action involving use of resources not previously considered in connection with the Final Environmental Statement for Operation of McGuire Nuclear Station, Units 1 and 2, or its Addendum, is a minor change in land use associated with operating support of the facility. As noted above, this change in land usage is insignificant. As further noted above, the change also involves a minor addition to the operational radiological monitoring and recordkeeping program during plant operation.

*Agencies and Persons Consulted:* The staff reviewed the licensee's request and has not consulted other agencies or persons.

*Finding of No Significant Impact:* The Commission has determined not to prepare an environmental impact statement for the proposed action.

Based upon this environmental assessment, we conclude that the proposed action will not have a significant effect on the quality of the human environment.

For further details with respect to this action, see the request for approval dated June 18, 1984, with its July 18, 1984, supplement, which is available for public inspection at the Commission's Public Document Room, 1717 H Street, NW., Washington, D.C. and at the Atkins Library, University of North Carolina, Charlotte (UNCC Station), North Carolina 28242.

Dated at Bethesda, Maryland, this 23rd day of October 1984.

For the Nuclear Regulatory Commission.

**Thomas M. Novak,**

*Assistant Director for Licensing, Division of Licensing, Office of Nuclear Reactor Regulation.*

[FR Doc. 84-28329 Filed 10-25-84; 8:45 am]

BILLING CODE 7590-01-M

## Supplement No. 1, Programmatic Environmental Impact Statement (PEIS) Related to Decontamination and Disposal of Radioactive Wastes Resulting From March 28, 1979 Accident, Three Mile Island Nuclear Station, Unit 2 Final Report; Notice of Availability

The Nuclear Regulatory Commission has published its final report, Supplement 1, Programmatic Environmental Impact Statement (PEIS) related to decontamination and disposal of radioactive wastes resulting from March 28, 1979 accident, Three Mile Island Nuclear Station, Unit 2 (NUREG-0683). The supplement to the PEIS re-evaluates the estimation of occupational radiation dose during the cleanup and the associated potential health effects on the cleanup workers.

Copies of the supplement are being placed in NRC's Public Document Room, 1717 H Street, NW, Washington, DC, and in the NRC TMI Program Office, 100 Brown Street, Middletown, PA, for review by interested persons. Copies of the report may be purchased from the Sales Agent, Division of Technical Information and Document Control, Office of Administration, U.S. Nuclear Regulatory Commission, Washington, DC 20555, for $6.00 each. Your check or money order should be made payable to Superintendent of Documents. GPO deposit account holders may charge orders by calling (301) 492-9530. Copies will also be available at the National Technical Information Service, Springfield, Virginia 22161.

Dated at Washington, DC, this 23 day of October, 1984.

For the Nuclear Regulatory Commission.

**Bernard J. Snyder,**

*Program Director, Three Mile Island Program Office, Office of Nuclear Reactor Regulation.*

[FR Doc. 84-28325 Filed 10-25-84; 8:45 am]

BILLING CODE 7590-01-M

## OFFICE OF THE UNITED STATES TRADE REPRESENTATIVE

### Request for Public Comments: Certain Amorphous Metal Articles and Amorphous Metal Alloys

On October 18, 1984, the United States International Trade Commission referred to the President for review its determination that there is a violation of section 337 of the Tariff Act of 1930 (19 U.S.C. 1337) in the importation into the United States, and in the sale, of certain amorphous metal articles produced by a process that, if practiced in the United States, would infringe a United States patent. The Commission found that the importations in question have the tendency to injure substantially an efficiently and economically operated United States industry. The Commission directed the U.S. Customs Service to exclude from entry into the United States amorphous metal articles manufactured abroad in accordance with the process claimed by the patent.

Under section 337(g), the President, for policy reasons, may disapprove the Commission's determination within sixty days following receipt of the determination and record. If disapproved by the President, the determination, and any order issued under its authority, would be without force or effect. The President also may approve the determination, making it, and any order issued under its authority, final on the date the Commission receives notice. The determination and related orders become final automatically following the sixty day review period, if the President has not disapproved.

Interested parties may submit comments concerning foreign or domestic policy issues that should be considered by the President in making his decision regarding this case. Parties commenting on domestic policy issues should refer to the portion of the Commission's record related to that issue. Parties should provide a rationale if the domestic policy issue was not raised before the Commission.

Comments may not exceed 15 letter-sized pages, including attachments. Twenty copies of the submission must be provided. Comments must be delivered by the close of business, Friday, November 16, 1984, to the Secretary, Trade Policy Staff Committee, 600 17th Street, NW., Washington, D.C. 20506. For further information, call Warren Maruyama (202) 395–6800.

**Frederick L. Montgomery,**

*Chairman, Trade Policy Staff Committee.*

[FR Doc. 84-28268 Filed 10-25-84; 8:45 am]

BILLING CODE 3190-01-M

[Rel. No. 14208; 812-5897]

## Creditanstalt Bankverein and Creditanstalt Finance, Inc.; Filing of Application for an Order Pursuant to Section 6(c) of the Act Exempting Applicants From All Provisions of the Act

October 22, 1984.

Notice is hereby given that Creditanstalt-Bankverein (the "Bank") and Creditanstalt Finance Inc. (the "Issuer") (the Bank and the Issuer hereinafter collectively referred to as "Applicants") Michael Gruson, Esq., Shearman & Sterling, Citicorp Center, 153 East 53rd Street, New York, New York 10022, filed an application on July 12, 1984, for an order of the Commission pursuant to Section 6(c) of the Investment Company Act of 1940 ("act"), exempting Applicants from all provisions of the Act. All interested persons are referred to the application on file with the Commission for a statement of the representations contained therein, which are summarized below, and to the Act for the text of the applicable provisions.

According to the application, the Issuer was incorporated in Delaware in 1984, as a subsidiary of the Bank for the purpose of issuing and selling the Issuer's short-term commercial paper notes (the "Notes") and lending to or depositing with the Bank, for use in the Bank's current lending operations, the proceeds from the sale of the Notes. All of the authorized, issued and outstanding shares of capital stock of the Issuer are and will be owned by the Bank. Applicants state that the Bank is the largest commercial bank in Austria and that the Republic of Austria owns the majority of the issued and outstanding shares of the Bank. Applicants state that at December 31, 1983, deposits (including demand, time and savings deposits) totaled approximately 81.6% of the Bank's total liabilities (including capital) of approximately $15.55 billion, while debentures outstanding totalled approximately 14.7% of such liabilities. Applicants state that at December 31, 1983, the aggregate amount of loans extended by the Bank, and deposits with banks, constituted approximately 84.3% of the Bank's total assets and of this amount 23.8% represented deposits with banks. Applicants state that for the year ended, December 31, 1983, the Bank's total revenues were $1.291 billion and that of this amouunt $1.093 billion, or 84.7%, represented revenues from loans, including loans to, and deposits with, banks.

Applicants represent that the Bank is subject to extensive regulation in Austria that is comparable in many respects to the supervision of United States banks. Applicants assert that, by maintaining a Federal branch in the United States (which is subject to regulation by the Comptroller of the Currency under the International Banking Act of 1978), the Bank is supervised by the Board of Governors of the Federal Reserve System under the Bank Holding Company Act of 1956.

According to the application, it is proposed that the Issuer will offer, issue and sell in the United States the Notes in bearer form and denominated in United States dollars. Applicants represent that the Notes will be unconditionally guaranteed by the Bank. Applicants state that the terms of such loans and deposits will be substantially similar to those of the Notes and will allow the Issuer to make timely payment on the Notes. Applicants state that it is intended that the Notes will be sold without registration under Section 5 of the Securities Act of 1933 (the "Securities Act"), in reliance upon an opinion of United States counsel that the offering will qualify for the exemption from the registration requirements of the Securities Act provided by Section 3(a)(3) thereof. Applicants do not request Commission review or approval of such opinion, and the Commission expresses no opinion as to the availability of any such exemption.

Applicants represent that the presently proposed issue of Notes and any future issue of debt securities offered for sale in the United States by the Bank or the Issuer will be conditioned upon the receipt, prior to issuance, of one of the three highest investment grade ratings from at least one of the nationally recognized statistical rating organizations. Applicants state that their United States counsel will certify that such a rating has been received.

Applicants state that the Notes will be sold through one or more United States commercial paper dealers to institutional investors and other entities and individuals in the United States who normally purchase commercial paper. Applicants undertake to insure that the Notes will not be advertised or otherwise offered for sale to the general public. Applicants represent that the purpose in making the proposed offering of the Notes is to provide an additional source of supply of United States dollars for the Bank to supplement dollars currently obtained by it in the Eurodollar market.

Applicants undertake to insure that each offeree who has indicated an

interest in the Notes will receive, prior to any sale of Notes to such offeree, a memorandum that describes the business of the Issuer and the Bank and provides the Bank's most recent publicly available annual financial statement audited in accordance with Austrian accounting principles. Applicants represent such memorandum will describe the material differences between Austrian accounting principles applicable to Austrian banks and "generally accepted accounting principles" applicable to United States commercial banks. Applicants further represent such memorandum will be at least as comprehensive as those customarily used in commercial paper offerings in the United States and will be updated periodically to reflect material changes in the Bank's business and financial condition. Applicants consent to any order granting the relief requested in the application being expressly conditioned upon Applicants compliance with the foregoing undertaking.

Applicants state that the Notes will rank *pari passu* among themselves and equally with all other unsecured and unsubordinated indebtedness of the Issuer, and ahead of its share capital. The guarantee of the Bank will rank *pari passu* with all other unsecured and unsubordinated indebtedness of the Bank.

Applicants state that they will appoint an issuing agent as authorized agent to issue the Notes from time to time. Applicants will expressly accept the jurisdiction of any state or federal court in the Borough of Manhattan in the City and State of New York in respect of any action based on the Notes or arising out of the offering and sale of the Notes, or any action based on or arising out of the guarantee of the Bank, instituted in any state or federal court by the holder of any Notes. In that connection, Applicants will appoint Creditanstalt-Bankverein, New York Branch, as agent to accept any process which may be served in any such action. Such appointment of an authorized agent to accept service of process and such consent to jurisdiction will be irrevocable until all amounts due and to become due with respect to the Notes have been paid. Applicants will also be subject to suit in any other court in the United States which would have jurisdiction because of the manner of the offering of the Notes or otherwise. Applicants state no such submission to jurisdiction or appointment of agent for service of process will affect the right to any holder of a Note to bring suit in any court which shall have jurisdiction over Applicants by virtue of the offer and sale of the Notes, the guarantee of the Bank or otherwise.

According to the application, either Applicant may, from time to time, offer other debt securities for sale in the United States. Applicants represent any such debt securities issued by the Issuer will be unconditionally guaranteed by the Bank. Applicants undertake that any future offering of debt securities in the United States will be made only pursuant to a registration statement under the Securities Act or pursuant to an applicable exemption from registration under the Securities Act. Applicants further undertake that any such offering will be done on the basis of disclosure documents that are at least as comprehensive in their description of the Applicants and their respective businesses and financial statements as the disclosure memorandum referred to above in the case of the commercial paper notes, but in no event will such future disclosure documents be less comprehensive than is customary for United States offerings of similar debt securities. Applicants further undertake to update promptly any such documents to reflect material changes in their financial conditions, respectively. Applicants consent to any order granting the relief requested in the application being expressly conditioned upon Applicants' compliance with the foregoing undertaking.

In addition, Applicants undertake, in connection with any future offering of debt securities in the United States by Applicants, or either of them, to appoint Creditanstalt-Bankverein, New York Branch, to accept any process which may be served in any action based on such securities and instituted in any state or federal court by any holder of any such security. Applicants further undertake that they will expressly accept the jurisdiction of any state or federal court in the Borough of Manhattan in the City and State of New York in respect of any such action. Such appointment of Creditanstalt-Bankverein, New York Branch, to accept service of process and such consent to jurisdiction will be irrevocable so long as such securities remain outstanding and until all amounts due and to become due in repsect of such securities have been paid. Applicants will also be subject to suit in any other court in the United States which would have jurisdiction because of the manner of the offering of such securities or otherwise.

Notice is further given that any interested person wishing to request a hearing on the application may, not later than November 16, 1984, at 5:30 p.m., do so by submitting a written request setting forth the nature of his interest, the reasons for his request, and the specific issues, if any, of fact or law that are disputed, to the Secretary, Securities and Exchange Commission, Washington, D.C. 20549. A copy of the request should be served personally or by mail upon Applicants at the address stated above. Proof of service (by affidavit or, in the case of an attorney-at-law, by certificate) shall be filed with the request. After said date an order disposing of the application will be issued unless the Commission orders a hearing upon request or upon it own motion.

For the Commission, by the Division of Investment Management, pursuant to delegated authority.

**Shirley E. Hollis,**

*Acting Secretary.*

[FR Doc. 84-28352 Filed 10-25-84; 8:45 am]

BILLING CODE 8010-01-M

**[File No. SR-PSDTC-84-9; Rel. No. 21421]**

## Order Approving a Proposed Rule Change of Pacific Securities Depository Trust Co.

October 22, 1984.

On August 13, 1984, the Pacific Securities Depository Trust Company ("PSDTC") filed with the Securities and Exchange Commission a proposed rule change under Section 19(b)(1) of the Securities Exchange Act of 1934 (the "Act"), 15 U.S.C. § 78s(b)(1). The Commission published notice of the proposal to solicit comment.[1] No comment was received. This Order approves the proposal.

The proposal establishes PSDTC as a "qualified registered securities depository" for purposes of Securities Exchange Act Rule 17Ad-14, 17 C.F.R. § 240.17Ad-14. Rule 17Ad-14 requires a registered transfer agent to open special accounts with qualified registered securities depositories when acting as a tender agent or exchange agent in connection with tender or exchange offers for depository eligible securities. Currently, the only qualified registered securities depositories are Depository

[1] Securities Exchange Act Release No. 21271 (August 28, 1984), 49 FR 34995 (September 4, 1984).

Trust Company ("DTC") and Midwest Securities Trust Company ("MSTC").

The proposal includes PSDTC procedures governing the special transfer agent accounts, particularly the processing of movements to and from the accounts to reflect tenders and withdrawals. More specifically, the proposal establishes PSDTC procedures for opening the special accounts, tender of shares by participants, withdrawals from tendered positions, delivery of physical certificates, and payment for tendered shares.

### I. Description

Under the proposal, a tender or exchange agent (the "agent") must contact PSDTC as soon as a tender or exchange offer is announced and must enter into a Letter of Agreement with PSDTC making PSDTC procedures binding on the agent. The special account must be established within two business days after commencement of the tender or exchange offer.

From commencement of an offer through the day before the offer's expiration or proration date, participants may tender their share positions by submitting letters of authorization to PSDTC. Each day, for all letters of authorization submitted by 3:00 p.m. Pacific time, PSDTC will make book-entry movements of shares from each tendering participant's account to a single PSDTC internal account.[2] After that time, PSDTC will make a single book-entry movement to transfer those shares from the internal account to the agent's account. By 8:00 a.m. Pacific time each day, PSDTC will release to the agent a position report reflecting the prior day's activity and a grand total of all tendered shares since the beginning of the offer. Also, PSDTC will send a weekly letter of transmittal to the agent indicating all shares tendered that week, unless the agent requires letters of transmittal more frequently.[3]

The proposal also enables participants to tender shares indirectly to the agent by sending the agent a letter of transmittal guaranteeing later delivery through PSDTC. Participants then "cover" such letters by sending a copy to PSDTC along with a letter of authorization. PSDTC then will make book-entry movement of the shares in the same manner as shares tendered through PSDTC, *i.e.*, first into the internal PSDTC account and then into the agent's account. The daily position reports to agents will indicate which letters of transmittal are covered by the prior day's movements.

Participants also may withdraw shares previously tendered by submitting withdrawal instructions to PSDTC. The proposal provides that withdrawal instructions must be submitted "timely."[4] PSDTC will submit corresponding instructions to the agent the day after receiving the instructions, together with the daily position report. Upon confirmation of the instructions by the agent, which should be made by noon the same day, PSDTC will return withdrawn shares to participants' accounts by book-entry movement. These movements will be reflected the next day on the agent's position report.

PSDTC generally will make physical delivery of tendered shares to the agent two business days after the expiration of the offer or any protection period. These time frames, however, can be adjusted by agreement of PSDTC and the agent. The agent immediately must confirm to PSDTC the total number of shares delivered and verify that all shares are in good deliverable form.

Finally, the proposal provides that agents must inform PSDTC as soon as a date for release of payment to tendering participants is determined. Agents also must provide the proration date for acceptance of shares, if applicable.

### II. PSDTC's Rationale

PSDTC believes that the proposal is consistent with the Act and, in particular, Section 17A of the Act and Rule 17Ad–14 under the Act. PSDTC believes that its proposal provides an efficient method of handling tender and exchange offers by centralized book-entry movement of tendered shares. In addition, PSDTC believes that the establishment of depository accounts at PSDTC by tender and exchange agents furthers the prompt and accurate clearance and settlement of securities transactions while safeguarding funds and securities in PSDTC's custody or control. Finally, PSDTC believes that because its proposal is sufficiently similar to already approved programs at DTC and MSTC, agents will not be burdened in dealing with multiple depositories during tender or exchange offers.

### III. Discussion

For the following reasons, the Commission believes that PSDTC's proposal should be approved. First, the Commission believes that the proposal will not adversely affect PSDTC's ability to safeguard securities and funds. PSDTC has submitted to the Commission detailed internal procedures governing processing under the proposal. PSDTC also submitted these procedures, together with the proposed rule change, to the Board of Governors of the Federal Reserve System ("BOG"), PSDTC's "appropriate regulatory agency" under the Act.[5] After consulting with BOG staff, the Commission believes that the proposal, including the procedures, are consistent with PSDTC's statutory duty to safeguard securities and funds.

Second, the Commission believes that PSDTC's proposal will promote prompt and accurate clearance and settlement of securities transactions.[6] Tender offer processing can occur with substantial efficiency and cost savings within a centralized, automated, book-entry environment. Use of automated facilities substantially reduces certificate control problems otherwise experienced by a depository and the securities industry as a whole when processing must occur by means of physical certificate delivery. In addition, when depository services for the subject company's securities can continue uninterrupted throughout a tender or exchange offer, customer-side and steet-side settlement of secondary market trades in these securities can occur quickly and efficiently. The proposal is designed to meet these goals.

Third, the Commission is satisfied that the proposal's procedures are substantially identical to tender and exchange offer procedures at DTC and MSTC, and other qualified registered securities depositories under Rule 17Ad–14. The primary differences in PSDTC's program concern the timing of inputs and reports, and most of these differences are due to the time differences and distances between California, Chicago and New York. For example, MSTC participants may tender shares to MSTC until 10:30 a.m. Central time on the expiration date or proration date of the offer for same-day delivery to the agent's account. A report will be issued at 4:00 p.m. Central time reflecting these tenders. PSDTC participants may tender shares until 3:00 p.m. Pacific time on the day before either expiration of the offer or the

[2] If a Participant's PSDTC position in that issue is insufficient to cover submitted tender instructions, the instructions will be rejected.

[3] PSDTC will not collect any solicitation fees payable to eligible participants. Rather, PSDTC will identify eligible participants and will forward appropriate documentation of those claims to the agent at the same time letters of transmittal are forwarded to the agent. The agent must pay eligible participants directly.

[4] Deadlines for withdrawal instructions will be set by PSDTC according to the terms of the offer.

[5] *See* Section 3(a)(34)(B) of the Act.

[6] For a detailed discussion of how tender and exchange agent programs promote the prompt and accurate clearance and settlement of securities transactions, *see* Securities Exchange Act Release No. 20581 (January 19, 1984), 49 FR 3064 (January 25, 1984), adopting Rule 17Ad–14.

proration date for same-day delivery to the agent's account, and a report reflecting these transfers will be issued by 8:00 a.m. Pacific time the next day. The Commission believes that these time differences are necessary for timely depository reports to agents, particularly reports of Participant tenders and withdrawals.[7] In any event, the proposal provides sufficient flexibility to meet special needs of agents. For example, PSDTC will supplement weekly letters of transmittal to agents with more frequent letters upon request of the agent. Also, daily position reports to agents can be hand delivered, sent by facsimile transmission, or sent via overnight courier, depending on the location and needs of an agent. This flexibility, in the Commission's view, blunts the impact of procedural differences among the several tender and exchange agent programs.

In summary, the Commission believes that PSDTC has designed an efficient automated tender and exchange offer processing program that will function harmoniously with MSTC's and DTC's programs. By establishing PSDTC as the third qualified registered securities depository under Securities Exchange Act Rule 17Ad–14, the proposal substantially completes a nationwide automated tender and exchange offer processing system.[8]

## IV. Conclusion

On the basis of the foregoing, the Commission finds the proposed rule change consistent with the Act and, more specifically, Section 17A of the Act and Securities Exchange Act Rule 17Ad–14.

Accordingly, it is therefore ordered, pursuant to Section 19(b)(2) of the Act, that the proposed rule change (SR–PSDTC–84–9) be, and it hereby is, approved.

For the Commission, by the Division of Market Regulation pursuant to delegated authority.

**Shirley E. Hollis,**

*Acting Secretary.*

[FR Doc. 84–28353 Filed 10–25–84; 8:45 am]

BILLING CODE 8010–01–M

[7] There are additional differences that the Commission believes are insignificant. For example, PSDTC and MSTC allow withdrawal of previously tendered shares by submitting withdrawal instructions to the depository; DTC requires withdrawal instructions to be submitted directly to the agent. PSDTC and DTC do not collect solicitation fees from agents on behalf of participants; MSTC collects these fees.

[8] Philadelphia Depository Trust Company also plans to file a proposed rule change with the Commission to become a qualified registered securities depository under Rule 17Ad–14.

## Tri-Comp Sensors, Inc.; Order of Trading Suspension

October 23, 1984.

It appears to the Securities and Exchange Commission that there is a lack of current and accurate information relating to the securities of Tri-Comp Sensors, Inc. ("Tri-Comp") and that questions have been raised about the adequacy and accuracy of publicly-disseminated information concerning the uses of the proceeds of Tri-Comp's initial public offering of securities, Tri-Comp's financial condition, transactions between Tri-Comp and other companies, potential mergers between Tri-Comp and other companies, the involvement of certain Tri-Comp executives in companies with which Tri-Comp has engaged in material transactions, and other matters, and the Commission is of the opinon that the public interest and the protection of investors require a summary suspension of trading in the securities of Tri-Comp.

Therefore, it is ordered, pursuant to Section 12(k) of the Securities Exchange Act of 1934, that over-the-counter trading in the securities of Tri-Comp Sensors, Inc. is suspended, for the period from 2:00 P.M. (EDT) on October 23, 1984, and terminating at midnight (EST) on November 1, 1984.

By the Commission.

**Shirley E. Hollis,**

*Acting Secretary.*

[FR Doc. 84–28351 Filed 10–25–84; 8:45 am]

BILLING CODE 8010–01–M

# SMALL BUSINESS ADMINISTRATION

**[Designation of Disaster Loan Area No. 6210]**

## Designation of Disaster Loan Area; California

Humboldt and Mendocino Counties in the State of California constitute a disaster area pursuant to Pub. L. 98–473 because of El Nino related ocean conditions in the Pacific Ocean beginning December 1982. Eligible small businesses involved in the fishing industry, without credit elsewhere and small agricultural cooperatives involved in the fishing industry, without credit elsewhere may file applications for economic injury assistance until the close of business on July 22, 1985, at the address listed below:

Disaster Area 4 Office, Small Business Administration, 77 Cadillac Drive, Suite 158, Sacramento, California 95825

or other locally announced locations. The interest rate for eligible small business applicants without credit elsewhere is 4% and 10.5% for eligible small agricultural cooperatives without credit elsewhere. Eligible small businesses involved in the fishing industry are defined by Pub. L. 98–473 as "* * * any trade or business involved in (i) the catching, taking, or harvesting of fish (whether or not sold on a commercial basis), (ii) any operation at sea or on land, in preparation for, or substantially dependent upon, the catching, taking, or harvesting of fish, and (iii) the processing or canning of fish (including storage, refrigeration, and transportation of fish before processing or canning) * * *."

Dated: October 22, 1984.

**Irene Castillo,**

*Acting Administrator.*

[FR Doc. 84–28333 Filed 10–25–84; 8:45 am]

BILLING CODE 8025–01–M

**[Designation of Disaster Loan Area No. 6223]**

## Designation of Disaster Loan Area; Oregon

Douglas, Lane, Curry, Lincoln, Clatsop, Tillamook, and Coos Counties in the State of Oregon constitute a disaster area pursuant to Pub. L. 98–473 because of El Nino related ocean conditions in the Pacific Ocean beginning December 1982. Eligible small businesses involved in the fishing industry, without credit elsewhere and small agricultural cooperatives involved in the fishing industry, without credit elsewhere may file applications for economic injury assistance until the close of business on July 22, 1985, at the address listed below:

Disaster Area 4 Office, Small Business Administration, 77 Cadillac Drive, Suite 158, Sacramento, California 95825

or other locally announced locations. The interest rate for eligible small business applicants without credit elsewhere is 4% and 10.5% for eligible small agricultural cooperatives without credit elsewhere. Eligible small businesses involved in the fishing industry are defined by Pub. L. 98–473 as "* * * any trade or business involved in (i) the catching, taking, or harvesting of fish (whether or not sold on a commercial basis), (ii) any operation at sea or on land, in preparation for, or substantially dependent upon, the catching, taking, or harvesting of fish,

and (iii) the processing or canning of fish (including storage, refrigeration, and transportation of fish before processing or canning) * * *."

Dated: October 22, 1984.

(Catalog of Federal Domestic Assistance Program Nos. 59002 and 59008)

Irene Castillo,

*Acting Administrator.*

[FR Doc. 84-28335 Filed 10-25-84; 8:45 am]

BILLING CODE 8025-01-M

### Hawaii; Region IX Advisory Council; Public Meeting

The Small Business Administration Region IX Advisory Council, located in the geographical area of Honolulu, Hawaii, will hold a public meeing at 9:00 a.m. on Wednesday, November 28, 1984, at the Prince Kuhio Federal Building, 300 Ala Moana Boulevard, Room C-270 (2nd Floor), Honolulu, Hawaii, to discuss such matters as may be presented by members, staff of the Small Business Administration, or others present.

For further information, write or call David K. Nakagawa, District Director, U.S. Small Business Administration, 300 Ala Moana Boulevard, Room 2213, Honolulu, Hawaii 96850, (808) 546-8950.

Jean M. Nowak,

*Director, Office of Advisory Councils.*

October 22, 1984.

[FR Doc. 84-28331 Filed 10-25-84; 8:45 am]

BILLING CODE 8025-01-M

### Tennessee; Region IV Advisory Council; Public Meeting

The Small Business Administration Region IV Advisory Council, located in the geographical area of Nashville, Tennessee, will hold a public meeting at 9:00 a.m. on Wednesday, October 31, 1984, at the Board Room of Third National Bank, 201 Fourth Avenue North, Nashville, Tennessee, 37244, to discuss such matters as may be presented by members, staff of the Small Business Administration, or others present.

For further information, write or call Robert M. Hartman, District Director, U.S. Small Business Administration, Suite 1012 Parkway Towers, 404 James Robertson Parkway, Nashville, Tennessee 37219. Telephone (615) 251-5850.

Jean M. Nowak,

*Director, Office of Advisory Councils.*

October 22, 1984.

[FR Doc. 84-28332 Filed 10-25-84; 8:45 am]

BILLING CODE 8025-01-M

**[Designation of Disaster Loan Area No. 6224]**

### Designation of Disaster Loan Area; Washington

Benton, Chelan, Clallam, Clark, Colwitz, Ferry, Franklin, Grant, Grays Harbor, Island, Jefferson, King, Kitsap, Lewis, Mason, Okanogan, Pacific, Pierce, San Juan, Skagit, Skamania, Snohomish, Spokane, Thurston, Wahkiakum, Whatcom, Whitman, and Yakima Counties in the State of Washington constitute a disaster area pursuant to Pub. L. 98-473 because of El Nino related ocean conditions in the Pacific Ocean beginning December 1982. Eligible small businesses involved in the fishing industry, without credit elsewhere and small agricultural cooperatives involved in the fishing industry, without credit elsewhere may file applications for economic injury assistance until the close of business on July 22, 1985, at the address listed below:

Disaster Area 4 Office, Small Business Administration, 77 Cadillac Drive, Suite 158, Sacramento, California 95825

or other locally announced locations. The interest rate for eligible small business applicants without credit elsewhere is 4% and 10.5% for eligible small agricultural cooperatives without credit elsewhere. Eligible small businesses involved in the fishing industry are defined by Pub. L. 98-473 as " . . . any trade or business involved in (i) the catching, taking, or harvesting of fish (whether or not sold on a commercial basis), (ii) any operation at sea or on land, in preparation for, or substantially dependent upon, the catching, taking, or harvesting of fish, and (iii) the processing or canning of fish (including storage, refrigeration, and transportation of fish before processing or canning) . . . "

Dated: October 22, 1984.

(Catalog of Federal Domestic Assistance Program Nos. 59002 and 59008)

Irene Castillo,

*Acting Administration.*

[FR Doc. 84-28334 Filed 10-25-84; 8:45 am]

BILLING CODE 8025-01-M

## DEPARTMENT OF THE TREASURY

### Office of the Secretary

### Public Information Collection Requirements Submitted to OMB for Review

Dated: October 22, 1984.

The Department of Treasury has submitted the following public information collection requirement(s) to OMB (listed by submitting bureau(s)), for review and clearance under the Paperwork Reduction Act of 1980, Pub. L. 95-511. Copies of these submissions may be obtained by calling the Treasury Bureau Clearance Officer listed under each bureau. Comments regarding these information collections should be addressed to the OMB reviewer listed at the end of each bureau's listing and to the Treasury Department Clearance Officer, Room 7225, 1201 Constitution Avenue, N.W., Washington, D.C. 20220.

**Internal Revenue Service**

*OMB Number:* 1545-0041
*Form Number:* IRS Form 966
*Type of Review:* Revision
*Title:* Corporate Dissolution or Liquidation

*OMB Number:* 1545-0226
*Form Number:* IRS Form 6249
*Type of Review:* Revision
*Title:* Computation of Overpaid Windfall Profit Tax

Clearance Officer: Garrick Shear (202) 566-6254, Room 5571, 1111 Constitution Avenue, NW., Washington, D.C. 20224

OMB Reviewer: Norman Frumkin, (202) 395-6880, Office of Management and Budget, Room 3208 New Executive Office Building, Washington, D.C. 20503

Joseph F. Maty,

*Departmental Reports Management Office.*

[FR Doc. 84-28283 Filed 10-25-84; 8:45 am]

BILLING CODE 4810-25-M

### Internal Revenue Service

### Art Advisory Panel; Closed Meeting

**AGENCY:** Internal Revenue Service, Treasury.

**ACTION:** Notice of Closed Meeting of Art Advisory Panel.

**SUMMARY:** A closed meeting of the Art Advisory Panel will be held in Washington, D.C.

**DATE:** The meeting will be held November 28, 1984.

**FOR FURTHER INFORMATION CONTACT:** Karen Carolan, CC:C:E:V, 1111 Constitution Avenue, NW., Room 2575, Washington, D.C., 20224, Telephone No. (202) 566-4138, (not a toll free number).

Notice is hereby given pursuant to section 10(a)(2) of the Federal Advisory Committee Act, 5 U.S.C. app. (1976), that a closed meeting of the Art Advisory Panel will be held on November 28, 1984 beginning at 9:30 a.m. in Room 3313,

Internal Revenue Service Building, 1111 Constitution Avenue, NW., Washington, D.C., 20224.

The agenda will consist of the review and evaluation of the acceptability of fair market value appraisals of works of art involved in federal income, estate, or gift tax returns. This will involve the discussion of material in individual tax returns made confidential by the provisions of section 6103 of Title 26 of the United States Code.

A determination as required by section 10(d) of the Federal Advisory Committee Act has been made that these meetings are concerned with matters listed in section 552(c) (3), (4), (6), and (7) of Title 5 of the United States Code, and that the meeting will not be open to the public.

This document does not meet the criteria for significant regulations set forth in paragraph 8 of the Treasury Directive appearing in the Federal Register for Wednesday, November 8, 1978. (43 FR 52122.)

**James I. Owens,**

*Acting Commissioner.*

[FR Doc. 84-28385 Filed 10-25-84; 8:45 am]

BILLING CODE 4830-01-M

# Sunshine Act Meetings

Federal Register
Vol. 49, No. 209
Friday, October 26, 1984

This section of the FEDERAL REGISTER contains notices of meetings published under the "Government in the Sunshine Act" (Pub. L. 94-409) 5 U.S.C. 552b(e)(3).

## CONTENTS

| | Item |
|---|---|
| Federal Energy Regulatory Commission | 1 |
| Federal Reserve System | 2, 3, 4 |
| Postal Service | 5 |
| Tennessee Valley Authority | 6 |

1

**FEDERAL ENERGY REGULATORY COMMISSION**

**"FEDERAL REGISTER" CITATION OF PREVIOUS ANNOUNCEMENT:** 49 FR 41327, October 22, 1984.

**PREVIOUSLY ANNOUNCED TIME AND DATE OF MEETING:** 10:00 a.m., October 24, 1984.

**CHANGE IN THE MEETING:** The following Docket No. has been added to the Items listed below:

*Item No., Docket No., and Company*

CP-1: RP84-75-000, Columbia Gas Transmission Company

Kenneth F. Plumb,

*Secretary.*

[FR Doc. 84-28383 Filed 10-3-84; 4:51 pm]

BILLING CODE 6717-02-M

2

**FEDERAL RESERVE SYSTEM**

**TIME AND DATE:** 10:00 a.m., Wednesday, October 31, 1984.

**PLACE:** Marriner S. Eccles Federal Reserve Board Building, C Street entrance between 20th and 21st Streets NW., Washington, D.C. 20551.

**STATUS:** Open.

**MATTERS TO BE CONSIDERED:**

Summary Agenda

Because of their routine nature, no substantive discussion of the following items is anticipated. These matters will be voted on without discussion unless a member of the Board requests that an item be moved to the discussion agenda.

1. Proposed amendment to Regulation D (Reserve Requirements of Depository Institutions) to index the low reserve tranche for transactions accounts and the reserve requirement exemption amount for 1985.

2. Proposed extension of Statement of Purpose for an Extension of Credit Secured by Margin Stock (FR U-1).

Discussion Agenda

3. Proposed policy statement concerning priced services surpluses and deficits.

4. Proposals regarding the 1985 Private Sector Adjustment Factor.

5. Proposals regarding Federal Reserve check collections services: (A) 1985 fee schedules and (B) publication for comment of price structure changes.

6. Any items carried forward from a previously announced meeting.

**Note.**—This meeting will be recorded for the benefit of those unable to attend. Cassettes will available for listening in the Board's Freedom of Information Office, and copies may be ordered for $5 per cassette by calling (202) 452-3684 or by writing to: Freedom of Information Office, Board of Governors of the Federal Reserve System, Washington, D.C. 20551.

**CONTACT PERSONS FOR MORE INFORMATION:** Mr. Joseph R. Coyne, Assistant to the Board; (202) 452-3204.

Dated: October 23, 1984.

James McAfee,

*Associate Secretary of the Board.*

[FR Doc. 84-28409 Filed 10-24-84; 8:45 am]

BILLING CODE 6210-01-M

3

**FEDERAL RESERVE SYSTEM**

(Board of Governors)

**TIME AND DATE:** Approximately 11:30 a.m., Wednesday, October 31, 1984, following a recess at the conclusion of the open meeting.

**PLACE:** Marriner S. Eccles Federal Reserve Board Building, C Street entrance between 20th and 21st Streets, NW., Washington, D.C. 20551.

**STATUS:** Closed.

**MATTERS TO BE CONSIDERED:**

1. Personnel actions (appointments, promotions, assignments, reassignments, and salary actions) involving individual Federal Reserve System employees.

2. Any items carried forward from a previously announced meeting.

**CONTACT PERSON FOR MORE INFORMATION:** Mr. Joseph R. Coyne, Assistant to the Board; (202) 452-3204. You may call (202) 452-3207, beginning at approximately 5 p.m. two business days before this meeting, for a recorded announcement of bank and bank holding company applications scheduled for the meeting.

Dated: October 23, 1983.

James McAfee,

*Associate Secretary of the Board.*

[FR Doc. 84-28410 Filed 10-24-84; 8:45 am]

BILLING CODE 6210-01-M

4

**FEDERAL RESERVE SYSTEM**

Board of Governors

**FEDERAL REGISTER CITATION OF PREVIOUS ANNOUNCEMENT:** 49 FR 40997, October 18, 1984.

**PREVIOUSLY ANNOUNCED TIME AND DATE OF THE MEETING:** 10:00 a.m., Wednesday, October 24, 1984.

**CHANGES IN THE MEETING:** One of the items announced for inclusion at this meeting was consideration of any agenda items carried forward from a previous meeting; the following such closed item(s) was added:

Federal Reserve Bank and Branch director appointments. (This item was previously announced for a closed meeting on October 1, 1984.)

**CONTACT PERSON FOR MORE INFORMATION:** Mr. Joseph R. Coyne, Assistant to the Board; (202) 452-3204.

Dated: October 24, 1984.

James McAfee,

*Associate Secretary of the Board.*

[FR Doc. 84-28484 Filed 10-24-84; 3:51 pm]

BILLING CODE 6210-01-M

5

**POSTAL SERVICE**

Board of Governors

The Board of Governors of the United States Postal Service, pursuant to its Bylaws (39 C.F.R. Section 7.5) and the Government in the Sunshine Act (5 U.S.C. Section 552b), hereby gives notice that it intends to hold meetings at 1:00 p.m. on Tuesday, November 13, 1984, in Washington, D.C., and at 8:30 a.m. on Wednesday, November 14, 1984, in the Benjamin Franklin Room, U.S. Postal Service Headquarters, 475 L'Enfant Plaza, SW., Washington, D.C. As indicated in the following paragraph, the November 13 meeting is closed to public observation. The November 14 meeting is open to the public. The Board expects to discuss the matters stated in the agenda which is set forth below. Requests for information about the meetings should be addressed to the

Secretary of the Board, David F. Harris, at (202) 245–3734.

At its meeting on October 2, 1984, the Board voted in accordance with the provisions of the Government in the Sunshine Act to close to public observation its meeting scheduled for November 13. (See 49 FR 39636, October 9, 1984.) The agenda items of the meeting to be closed concern: (1) Discussion of personnel matters; (2) strategic planning in connection with possible continued collective bargaining negotiations involving the Postal Service and four labor organizations representing certain postal employees; and (3) further consideration of the Postal Rate Commission's September 7, 1984, Opinion and Recommended Decision in Docket No. R84–1.

**Agenda**

*Tuesday Session, November 13 (Closed)*

1. Discussion of Personnel Matters.
2. Strategic Planning—Collective Bargaining.
3. Consideration of Postal Rate Commission recommended decision in the omnibus rate case, Docket No. R84–1.

*Wednesday Session, November 14 (Open)*

1. Minutes of the Previous Meeting, October 2–3, 1984.
2. Remarks of the Postmaster General. (In keeping with its consistent practice, the Board's agenda provides this opportunity for the Postmaster General to inform the Members of miscellaneous current developments concerning the Postal Service. Northing that requires a decision by the Board is brought up under this item.)
3. Briefing on Postal Rate Commission Recommended Decision in Docket No. R84–1. (Mr. Coughlin, Senior Assistant Postmaster General, will present management's analysis of the Postal Rate Commission's September 7, 1984, recommendations in the omnibus rate case.)
4. Annual Report of the Postmaster General. (The Board will consider the Annual Report of the Postmaster General to the Board concerning the operation of the Postal Service, as required by 39 U.S.C. 2402. Upon arrival thereof, or after making such changes as it considers appropriate, the Board is to transmit this record to the President and the Congress. Ms. Layton, Assistant Postmaster General, Public and Employee Communications Department, will present the proposed Annual Report for Fiscal Year 1984.)
5. Review of the Comprehensive Statement. (Pub. L. 94–421 amended 39 U.S.C. 2401(g) to require the Postal Service to present a "Comprehensive Statement" to the Legislative and Appropriations Committee of the Congress having cognizance over postal matters. The Comprehensive Statement is to describe the plans and policies of the Postal Reorganization Act; postal operations generally and financial summaries and projections. Mr. Johnstone, Assistant Postmaster General, Government Relations Department, will present the proposed Comprehensive Statement for the Board's approval.)
6. Quarterly Report on Service Performance. (Mr. Jellison, Senior Assistant Postmaster General, Operations Group, will present the quarterly summary on service performance.)
7. Report on Employee and Labor Relations. (Mr. Morris, Senior Assistant Postmaster General, Employee and Labor Relations Group, will present the annual report to the Board on developments in the Employee and Labor Relations area.)
8. Report of Regional Postmaster General. (Mr. Horgan, Regional Postmaster General, Eastern Region, will report on postal operations in the Eastern Region.)
9. Update on the Long Life Vehicle Plan. (Mr. St. Francis, Director, Office of Fleet Management, Delivery Services Department, will present a report on the status of the long life delivery vehicle plan.)
10. FY 1985 Vehicle Capital Plan. (Mr. Hagburg, Assistant Postmaster General, Delivery Services Department, will present the FY 1985 vehicle capital plan.)
11. Capital Investments.
   a. Norman (OK) Training Facility;
   b. Springfield, MO General Mail Facility.
12. Consideration of Tentative Agenda for the December 3–4, 1984, meeting in Washington, D.C.
13. Consideration of Postal Rate Commission recommended decision approving the stipulation and agreement in Docket No. MC84–1, Special Fourth-Class Mail, Phase I.

**David F. Harris,**

*Secretary.*

[FR Doc. 84–28422 Filed 10–24–84; 1:51 pm]

**BILLING CODE 7710–12–M**

---

**6**

**TENNESSEE VALLEY AUTHORITY**

(Meeting No. 1340)

**TIME AND DATE:** 6:00 p.m. (CST), Tuesday, October 30, 1984.

**PLACE:** Parkway Junior High School, 1341 North Parkway, Jackson, Tennessee.

**STATUS:** Open.

**Agenda Items**

Approval of minutes of meeting held on October 10, 1984.

*Action Items*

A—Budget and Financing

A1. Fiscal year 1985 operating budget financed from power revenues.

A2. Fiscal year 1985 operating budget financed from nonpower proceeds.

A3. Fiscal year 1985 operating budget financed from regular appropriations.

A4. Fiscal year 1985 capital budget financed from regular appropriations.

B—Purchase Awards

B1. Proposal 38–834896—Steam generator 160 MW atmospheric fluidized bed combustion demonstration plant for Shawnee Fossil Plant. The award of this contract contemplates the contractor's participation as a contributor to the atmospheric fluidized bed combustion project.

C—Power Items

C1.[1] Proposed refinancing of the purchase money and construction notes of the purchaser/lessor of the Office of Power building.

C2. Proposed increase of amount of and revision in terms for limited interruptible power available to TVA's directly served customers and distributor-served industrial customers

E—Real Property Transactions

E1. Sale of permanent waterline easement to the Hamilton County, Illinois, Water District, affecting approximately .04 acre of TVA's Eads Mine property in south central Illinois—Tract No. XICOR–7P.

E2. Grant of permanent easement to Scottsboro Water Works, Sewer and Gas Board for construction of a sewerline, affecting approximately 2.75 acres of Guntersville Reservoir land in Jackson County, Alabama—Tract No. XTGR–145S.

E3. Grant of permanent easement to the State of Tennessee, Department of Conservation, for public recreation purposes, affecting approximately 1.2 acres of Pickwick Dam Reservation land in Hardin County, Tennessee—Tract No. XTPR–52RE.

E4. Filing of condemnation cases.

F—Unclassified

F1. Fertilizer distribution agreement between AmFert, Inc., Kansas City, Missouri, and TVA providing for the company's participation in TVA's fertilizer industry demonstration program.

F2. TVA policy code relating to electronic funds transfer.

**CONTACT PERSON FOR MORE INFORMATION:** Craven H. Crowell, Jr., Director of Information, or a member of his staff can respond to requests for information about this meeting. Call (615) 632–8000, Knoxville, Tennessee. Information is also available at TVA's Washington Office (202) 245–0101.

Dated: October 23, 1984.

**John G. Stewart,**

*Manager of Corporate Administration and Planning.*

[FR Doc. 84–28434 Filed 10–24–84; 1:51 pm]

**BILLING CODE 8120–01–M**

---

[1] Item approved by individual Board members. This would give formal ratification to the Board's action.

federal register

Friday
October 26, 1984

Part II

# Department of Labor

**Employment Standards Administration, Wage and Hour Division**

**Minimum Wages for Federal and Federally Assisted Construction; General Wage Determination Decisions, Notice**

## DEPARTMENT OF LABOR

### Employment Standards Administration, Wage and Hour Division

### Minimum Wages for Federal and Federally Assisted Construction; General Wage Determination Decisions

General wage determination decisions of the Secretary of Labor specify, in accordance with applicable law and on the basis of information available to the Department of Labor from its study of local wage conditions and from other sources, the basic hourly wage rates and fringe benefit payments which are determined to be prevailing for the described classes of laborers and mechanics employed on construction projects of the character and in the localities specified therein.

The determinations in these decisions of such prevailing rates and fringe benefits have been made by authority of the Secretary of Labor pursuant to the provisions of the Davis-Bacon Act of March 3, 1931, as amended (46 Stat. 1494, as amended, 40 U.S.C. 276a) and of other Federal statutes referred to in 29 CFR 5.1 (including the statutes listed at 36 FR 306 (1970) following Secretary of Labor's Order No. 24–70) containing provisions for the payment of wages which are dependent upon determination by the Secretary of Labor under the Davis-Bacon Act; and pursuant to the provisions of part 1 of subtitle A of title 29 of Code of Federal Regulations. Procedure for Predetermination of Wage Rates, 48 FR 19533 (1983) and of Secretary of Labor's Orders 9–83, 48 FR 35736 (1983), and 6–84, 49 FR 32473 (1984). The prevailing rates and fringe benefits determined in these decisions shall, in accordance with the provisions of the foregoing statutes, constitute the minimum wages payable on Federal and federally assisted construction projects to laborers and mechanics of the specified classes engaged on contract work of the character and in the localities described therein.

Good cause is hereby found for not utilizing notice and public procedure thereon prior to the issuance of these determinations as prescribed in 5 U.S.C. 553 and not providing for delay in the effective date as prescribed in that section, because the necessity to issue construction industry wage determination frequently and in large volume causes procedures to be impractical and contrary to the public interest.

General wage determination decisions are effective from their date of publication in the **Federal Register** without limitation as to time and are to be used in accordance with the provisions of 29 CFR Parts 1 and 5. Accordingly, the applicable decision together with any modifications issued subsequent to its publication date shall be made a part of every contract for performance of the described work within the geographic area indicated as required by an applicable Federal prevailing wage law and 29 CFR Part 5. The wage rates contained therein shall be the minimum paid under such contract by contractors and subcontractors on the work.

#### Modifications and Supersedeas Decisions to General Wage Determination Decisions

Modifications and supersedeas decisions to general wage determination decisions are based upon information obtained concerning changes in prevailing hourly wage rates and fringe benefit payments since the decisions were issued.

The determinations of prevailing rates and fringe benefits made in the modifications and supersedeas decisions have been made by authority of the Secretary of Labor pursuant to the provisions of the Davis-Bacon Act of March 3, 1931, as amended (46 Stat. 1494, as amended, 40 U.S.C. 276a) and of other Federal statutes referred to in 29 CFR 5.1 (including the statutes listed at 36 FR 306 (1970) following Secretary of Labor's Order No. 24–70) containing provisions for the payment of wages which are dependent upon determination by the Secretary of Labor under the Davis-Bacon Act; and pursuant to the provisions of Part 1 of Subtitle A of Title 29 of Code of Federal Regulations. Procedure for Predetermination of Wage Rates, 48 FR 19533 (1983) and of Secretary of Labor's Order, 6–84, 49 FR 32473 (1989). The prevailing rates and fringe benefits determined in foregoing general wage determination decisions, as hereby modified, and/or superseded shall, in accordance with the provisions of the foregoing statutes, constitute the minimum wages payable on Federal and federally assisted construction projects to laborers and mechanics of the specified classes engaged in contract work of the character and in the localities described therein.

Modifications and supersedeas decisions are effective from their date of publication in the **Federal Register** without limitation as to time and are to be used in accordance with the provisions of 29 CFR Parts 1 and 5.

Any person, organization, or governmental agency having an interest in the wages determined as prevailing is encouraged to submit wage rate information for consideration by the Department. Further information and self-explanatory forms for the purpose of submitting this data may be obtained by writing to the U.S. Department of Labor, Employment Standards Administration, Wage and Hour Division, Office of Program Operations, Division of Government Wage Determinations, Washington, D.C. 20210. The cause for not utilizing the rulemaking procedures prescribed in 5 U.S.C. 553 has been set forth in the original General Determination Decision.

#### Modifications to General Wage Determination Decisions

The numbers of the decisions being modified and their dates of publication in the **Federal Register** are listed with each State.

| | |
|---|---|
| California: | |
| CA84–5007 | May 18, 1984. |
| CA83–5119 | Sept. 16, 1983. |
| Connecticut: CT84–3016 | June 8, 1984. |
| Illinois: IL82–2049 | Oct. 15, 1982. |
| IL83–2037 | Apr. 29, 1983. |
| Massachusetts: MA84–3007 | Apr. 6, 1984. |
| Ohio: OH84–5024 | Aug. 24, 1984. |
| Oklahoma: | |
| OK84–4049 | Sept. 7, 1984. |
| OK84–4050 | Sept. 7, 1984. |
| Oregon: OR84–5020 | June 22, 1984. |
| Rhode Island: RI83–3042 | Aug. 19, 1984. |
| Texas: | |
| TX84–4028 | May 4, 1984. |
| TX84–4005 | Feb. 22, 1984. |
| TX84–4020 | Apr. 13, 1984. |
| TX84–4045 | Aug. 10, 1984. |
| TX84–4015 | Mar. 16, 1984. |
| West Virginia: WV83–3022 | Nov. 18, 1983. |

#### Supersedeas Decisions to General Wage Determination Decisions

The numbers of the decisions being superseded and their dates of publication in the **Federal Register** are listed with each State. Supersedeas decision numbers are in parentheses following the number of the decisions being superseded.

| | |
|---|---|
| Kansas: KS84–4051 (KS84–4101) | Aug. 24, 1984. |
| Missouri: MO83–4046 (MO84–4102) | June 10, 1983. |
| Virginia: VA79–3050 (84VA–3040) | Nov. 9, 1979. |

Signed at Washington, D.C. this 19th day of October 1984.

**James L. Valin,**

*Assistant Administrator.*

BILLING CODE 4510–27–M

MODIFICATIONS P. 1

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| DECISION NO. CA84-5007 - MOD. #7 (49 FR 21245 - May 18, 1984) Imperial, Inyo, Kern, etc. Counties, California | | |
| CHANGE: | | |
| Boilermakers | $21.60 | $4.25 |
| Bricklayers; Stonemasons: Area 2 - change Area Description to read: Inyo, Kern and Mono Counties except for Edwards Air Force Base and China Lake Naval Weapons Center | | |
| Electricians: | | |
| Area 4: | | |
| Electrician | 22.40 | 3% + 3.55 |
| Cable Splicer | 23.93 | 3% + 3.55 |
| Area 6: | | |
| Contracts over $2 million: | | |
| Electrician | 20.80 | 3%+5.40 |
| Cable Splicer | 21.30 | 3%+5.40 |
| Contracts $2 million and under: | | |
| Electrician | 17.80 | 3% + 5.40 |
| Cable Splicer | 18.30 | 3% + 5.40 |
| Area 7: | | |
| Contracts over $100,000: | | |
| Electrician | 21.56 | 3% + 3.63 |
| Cable Splicer | 22.72 | 3% + 3.63 |
| Contracts $100,000 or less: | | |
| Electrician | 16.49 | 3% + 3.63 |
| Cable Splicer | 18.14 | 3% + 3.63 |
| Residential Electrician | 11.06 | 3% + 3.63 |
| Area 8: | | |
| Electrician | 23.13 | 3% + 3.75 |
| Cable Splicer | 24.63 | 3% + 3.75 |

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| DECISION NO. CA84-5007 - (Cont'd) | | |
| CHANGE: | | |
| Elevator Constructors: | | |
| Area 1: | | |
| Mechanics | $23.14 | $3.29+ a |
| Helpers | 16.20 | 3.29+ a |
| Probationary Helpers | 11.57 | |
| Area 2: | | |
| Mechanics | 29.39 | 3.29+ a |
| Helpers | 20.57 | 3.29+ a |
| Probationary Helpers | 14.695 | |
| Glaziers: | | |
| Area 1 | 18.59 | 6.21 |
| Line Construction: | | |
| Area 7: | | |
| Cable Splicers | 23.72 | 4% + 3.58 |
| Equipment Operators; lineman | 21.56 | 4% + 3.58 |
| Groundman | 16.17 | 4% + 3.58 |
| Painters: | | |
| Area 4: | | |
| High Iron & Steel Construction, Bridges over 30 ft.: Brush; Steeple - jack (brush) | 19.82 | 4.14 |
| Steel, Sand blaster, water blaster, power cleaning, steam cleaning | 20.32 | 4.14 |
| Steeplejack (Spray) | 21.32 | |
| Power Equipment Operators: | | |
| Dredging (Hydraulic Suction Dredges): Change Fringe Benefits only to read: | | 7.25 |
| Refrigeration & Air Conditioning: | | |
| Area 1 | 20.19 | 8.40 |
| Roofers: | | |
| Area 2 | 14.55 | 3.85 |
| Area 3 | 17.65 | 5.50 |
| Area 4 | 18.97 | 1.585 |

MODIFICATIONS P. 2

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| DECISION NO. CA84-5007 - (Cont'd) | | |
| Terrazzo Workers: Area 2 | $19.52 | $3.80 |
| Tile Setters: Area 2 | 19.65 | 3.89 |
| DELETE: | | |
| Line Construction: Area 6: Groundman | | |
| Painters: Area 3: Brush; Brush or Roller (Swing Stage); Paperhangers; Tapers (Sheet Rock); Spray; Sandblaster; Steeplejack | | |
| Plasterers: Area 2 | | |
| Plumbers; Steamfitters: Area 1; Area 2 | | |
| Tile Setters: Area 3 | | |
| ADD: | | |
| Bricklayers; Stonemasons: Area 8 Edwards Air Force Base and China Lake Naval Weapons Center | 21.44 | 4.31 |
| Carpenters: Area 1: Piledrivermen | 19.275 | 7.275 |
| Area 2: Piledrivermen | 19.275 | 7.275 |
| Line Construction: Area 6: Cable Splicer | 18.17 | 3% + 4.75 |
| Equipment Operator | 17.67 | 3% + 4.75 |
| Groundman | 13.25 | 3% + 4.75 |
| Lineman | 17.67 | 3% + 4.75 |
| Marble & Tile Finishers: Area 3: | 15.21 | 5.27 |
| Area 3 to cover: Kern, Los Angles, Orange, Riverside, San Bernardino, San Luis Obispo, Santa Barbara & Ventura Counties | | |

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| DECISION NO. CA84-5007 - (Cont'd) | | |
| ADD: (Cont'd) | | |
| Painters: Area 3: Brush | $15.00 | $4.12 |
| Brush or Roller (Swing Stage); Paperhangers; Tapers (Sheet Rock) | 15.50 | 4.12 |
| Spray; Sandblaster | 16.00 | 4.12 |
| Spray & Sandblast (Swing Stage) | 16.50 | 4.12 |
| Steeplejack | 17.00 | 4.12 |
| Plasterers: Area 2: All work expect Residential work | 25.40 | - |
| Residential Plasterers | 22.40 | - |
| Area 2 to cover: Imperial, Riverside & San Bernardino Counties | | |
| Plumbers; Steamfitters: Area 1: | | |
| Zone 1 | 21.24 | 8.42 |
| Zone 2 | 23.49 | 8.42 |
| Zone 3 | 24.74 | 8.42 |
| Zone Definitions: Zone 1: Remainder of area | | |
| Zone 2: Camp Pendleton, Camp Roberts, Edwards Air Force Base, George Air Force Base, Vandenburg Air Force Base | | |
| Zone 3: Fort Irwin Army Base, Marine Corps Logistic Base at Nebo, Marine Corps Logistic Base at Yermo, Nicolaus Island Naval Base, 29 Palms Marine Base | | |

MODIFICATIONS P. 3

| DECISION NO. CA83-5119 - MOD. #6 (48 FR 41702 - September 16, 1983) San Diego County, California | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| CHANGE: | | |
| Electricians: | | |
| Electricians | $20.00 | 3%+ 3.90 |
| Cable Splicers | 20.45 | 3% 3.90 |
| Utility Technician | 16.50 | 3%+.74 |
| Sound Installers | 16.47 | 3% + 1.46 |
| Elevator Constructors: | | |
| Mechanics | 23.14 | 3.29+a |
| Helpers | 16.20 | 3.29+a |
| Probationary Helpers | 11.57 | - |
| Tile, Marble & Terrazzo Setters | 17.35 | 4.40 |
| DELETE: | | |
| Plasters | | |
| Plumbers; Pipefitters; Steamfitters Air Condition, Refrigeration | | |
| ADD: | | |
| Plasters: | | |
| All work except residential work | 17.16 | 4.27 |
| Residential work | 14.61 | 4.27 |
| Plumbers; Pipefitters: | | |
| Steamfitters; Air Condition, Refrigeration: | | |
| Zone 1 | 21.24 | 8.42 |
| Zone 2 | 23.49 | 8.42 |
| Zone Descriptions: | | |
| Zone 1: Remainder of County | | |
| Zone 2: Camp Pendleton | | |

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| | | |

MODIFICATIONS P. 4

| DECISION NO. CT84-3016 MOD. #8 (49 FR 23980 - June 8, 1984) Statewide Connecticut | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| CHANGE: | | |
| BOILERMAKER | 18.16 | 3.691 |
| CARPENTERS; MILLWRIGHTS; PILEDRIVERMEN; LATHERERS; RESILIENT FLOOR LAYERS: | | |
| BUILDING CONSTRUCTION: | | |
| Area 1 | 15.75 | 3.05 |
| Area 2 | 15.47 | 3.05 |
| Area 3 | 15.55 | 2.55 |
| Area 4 | 15.05 | 3.15 |
| Area 5 | 15.15 | 3.15 |
| Area 6: | | |
| Carpenters | 16.20 | 1.93 |
| Millwrights | 16.70 | 1.93 |
| ELECTRICIANS | | |
| Area 1 | 19.64 | .02+ 26%+b |
| Area 2 | 16.18 | 2.98+ 9.25% |
| Residential | 10.40 | 2.55+ 9.25% |
| Area 3 | 18.25 | 3.05+ 3.75% |
| Area 4 | 18.70 | 2.07+ 3.25% |
| Area 5 | 16.33 | 3.78+ 3% |
| Area 6 | 17.90 | 4.85+ 3 1/2% |
| LINE CONSTRUCTION: | | |
| Area 1: | | |
| Lineman | 14.43 | .02+e 19.5% |
| Compressor operator | 12.00 | .02+e 19.5% |
| Lamp Changer | 11.21 | .02+e 19.5% |
| Area 2: | | |
| Lineman, cable splicer, dynamite man | 17.02 | 2.90+ 4.75% |
| Heavy equipment Op. | 15.32 | 2.90+ 4.75% |
| Equipment operator, tractor trailer driver, field mechanic | 14.47 | 2.90+ 4.75% |
| Driver groundman | 12.77 | 2.90+ 4.75% |
| Groundman | 9.36 | 2.90+ 4.75% |
| Area 3: | | |
| Lineman, cable splicer dynamite man | 18.22 | 2.90+ 4.75% |
| Heavy equipment Op. | 16.40 | 2.90+ 4.75% |
| Equipment operator, tractor trailer driver, field mechanic | 15.49 | 2.90+ 4.75% |
| Driver groundman | 13.67 | 2.90+ 4.75% |
| Groundman | 10.02 | 2.90+ 4.75% |
| Area 4: | | |
| Lineman cable splicer, dynamite man | 17.74 | 2.90+ 4.75% |
| Heavy equipment Op. | 15.97 | 2.90+ 4.75% |
| Equipment operator, tractor trailer driver field mechanic | 15.08 | 2.90+ 4.75% |
| Driver groundman | 13.31 | 2.90+ 4.75% |
| Groundman | 9.76 | 2.90+ 4.75% |
| Area 5: | | |
| Lineman, cable splicer, dynamite man | 16.93 | 2.90+ 4.75% |
| Heavy Equipment Op. | 15.24 | 2.90+ 4.75% |
| Equipment operator tractor trailer driver, field mechanic | 14.39 | 2.90+ 4.75% |
| Driver groundman | 12.80 | 2.90+ 4.75% |
| Area 6: | | |
| Lineman, cable splicer, dynamite man | 19.58 | 2.90+ 4.75% |
| Heavy Equipment Op. | 17.62 | 2.90+ 4.75% |
| Equipment operator, tractor trailer driver, field mechanic | 16.64 | 2.90+ 4.75 |
| Driver groundman | 14.69 | 2.90+ 4.75% |
| Groundman | 10.77 | 2.90+ 4.75% |

| DECISION NO. CT84-3016 MOD. #8 (continued) | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| LINE CONSTRUCTION: (CONT'D) | | |
| Areas 2, 3, 4, 5 & 6: Traffic Control, Illumination & Maintenance: Linemen, technicians & cable splicers | 17.20 | 2.90+ 4.75% |
| Equipment operator | 14.62 | 2.90+ 4.75% |
| Driver groundman | 12.90 | 2.90+ 4.75% |
| Groundman | 12.04 | 2.90+ 4.75% |
| Railroad Construction (including electrical distrubution, transmission lines, substations and signal construction: Lineman, cable splicer & dynamite man | 17.95 | 2.90+ 4.75% |
| Heavy Equipment Op. | 16.16 | 2.90+ 4.74% |
| Equipment Operator, tractor trailer driver, field mechanic | 15.26 | 2.90+ 4.74% |
| Driver groundman | 13.46 | 2.90+ 4.74% |
| Groundman | 9.87 | 2.90+ 4.74% |
| PLUMBERS & STEAMFITTERS Area 3 | 19.12 | 2.80 |
| Area 9 Total Mechanical Contract of $325,000 or less | 21.81 | 3.40+g |
| | 16.36 | 3.40+g |
| SHEETMETAL WORKERS: Area 1 | 17.99 | 5.57 |

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| | | |



| DECISION NO. IL82-2049 - Mod #5 (47 FR 46221 - Oct 15, 1982) Peoria and Tazewell Counties | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| CHANGE: | | |
| ASBESTOS WORKERS | $18.00 | 3.78 |
| BRICKLAYERS; Stonemasons | 15.91 | 3.22 |
| CEMENT MASONS | 15.43 | 3.15 |
| ELECTRICIANS: | | |
| Electricians | 17.12 | 2.05+ 3.75% |
| Cable Splicers | 18.82 | 2.05+ 3.75% |
| MARBLE SETTERS; Terrazzo Workers & Tile Setters | 15.56 | 3.22 |
| LABORERS: | | |
| Peoria Co. and the City of E. Peoria in Tazewell Co. | | |
| Group 1 | 14.07 | 2.95 |
| Group 2 | 14.195 | 2.95 |
| Group 3 | 14.32 | 2.95 |
| Group 4 | 14.245 | 2.95 |
| Group 5 | 14.445 | 2.95 |
| OMIT: | | |
| PLUMBERS; PIPEFITTERS and STEAMFITTERS: | | |
| Peoria & Tazewell (North of Hwy #98) Cos: | | |
| Commercial Building | 16.69 | 2.00 |
| Residential | 15.92 | 2.00 |
| Tazewell (Remainder of Co.) | 13.55 | 1.64 |
| ADD: | | |
| PLUMBERS; PIPEFITTERS; and STEAMFITTERS: | | |
| Peoria and Tazewell Cos. | 16.89 | 3.50 |

| DECISION NO. IL83-2037 - MOD. #2 (48 FR 19583 - April 29, 1983) Illinois, Indiana, Michigan, Minnesota, New York, Ohio, Pennsylvania, and Wisconsin | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| DELETE: | | |
| Floating Equipment (Clamshell, Dragline, & Marine Construction) from Modification #1 Published 9-14-84. | | |

MODIFICATIONS P. 7

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| DECISION NO. MA84-3007 | | |
| MOD. NO. 6 | | |
| (49 FR 13804 - April 6, 1984) | | |
| Hampshire, Franklin, Hampden, and Hampshire Counties, Massachusetts | | |
| CHANGE: | | |
| CARPENTERS; SOFT FLOOR LAYERS; DRYWALL CARPENTERS; LATHERS | | |
| AREA 2 | $15.91 | 4.47 |
| AREA DESCRIPTION | | |
| Electricians & Line Construction: | | |
| Area 2: FRANKLIN (Remainder of County); HAMPSHIRE (Remainder of County); HAMPDEN (Chester, Holyoke and North Portion of Chicopee - north of Mckinstry Avenue from the Connecticut River to Grattan Street, Lafayette Street to York Street, McKinstry to Granby Road, and projected to the Westover Air Force Base fence, continuing northeastly along this boundary to the county line.) | | |
| Area 3: HAMPDEN (Remainder of County - including Westover Air Force Base); HAMPSHIRE (Belchertown, Ware) | | |

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| DECISION NO. OH84-5024 - | | |
| MOD. #1 | | |
| (49 FR 33787 - August 24, 1984 | | |
| Clermont & Hamilton Counties, Ohio | | |
| Change: | | |
| Carpenters | $ 8.11 | |
| Electricians | 9.92 | |
| Laborers | 6.06 | |
| Painter, Brush | 8.19 | |
| Roofers | 8.86 | |
| Sheet Metal Workers | 9.00 | $1.43 |
| Add: | | |
| Drywall Hangers | 7.00 | |
| Insulators | 6.06 | |
| Truck Drivers | 7.30 | |

MODIFICATIONS P. 8

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| DECISION NO. OK84-4050 | | |
| MOD #4 (49 FR 35477 - September 7, 1984) | | |
| Alfalfa, Beckham, Blaine, Caddo, Canadian, Carter, Cleveland, Comanche, Cotton, Custer, Dewey, Ellis, Garfield, Garvin, Grady, Grant, Greer, Harmon, Harper, Jackson, Jefferson, Johnston, Kay, Kingfisher, Kiowa, Lincoln, Logan, Love, McClain, Major, Marshall, Murray, Noble, Oklahoma, Payne, Pontotoc, Roger Mills, Pottawatomie, Seminole, Stephens, Tillman, Washita, Woods and Woodward Counties, Oklahoma | | |
| CHANGE: | | |
| Fringe benefits for Elevator Constructors to read "$3.00+a" which was omitted in Mod. #2 | | |
| OMIT: | | |
| Area I | | |
| Marble Masons, Tile Setters and Terrazzo workers | 16.84 | 1.50 |
| Published FR 10/12/84 | | |
| ADD: | | |
| Marble Masons, Tile Setters and Terrazzo workers | 16.84 | 1.50 |

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| DECISION # OK84-4049 | | |
| MOD. #3 | | |
| (49 FR 35473 - September 7, 1984 | | |
| Adair, Atoka, Bryan, Coal, Cherokee, Craig, Creek, Delaware, Haskell, Hughes, Leflore, Latimer, McIntosh Mayes, Muskogee, Nowata, Okfuskee, Okmulgee, Osage, Ottawa, Pawnee, Pittsburg, Pushmataha, rogers, Tulsa, Sequoyah, Wagoner, and Washington Cos., Oklahoma | | |
| CHANGE: | | |
| Fringe Benefits for elevator Constructors, Area II to read "$3.00+a" which was omitted from Mod. #2 | | |

DECISION NO. OR84-5020 - Mod #3
(49 FR 25821 - June 22, 1984)
Statewide Oregon

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| CHANGE: | | |
| BOILERMAKERS: | | |
| Work on Storage Tanks: | | |
| Erection | $17.25 | $4.25 |
| Repair | 16.05 | 4.25 |
| All other work | 19.67 | 4.25 |
| PLASTERERS: | | |
| Area 2 | 15.23 | 3.56 |
| SHEET METAL WORKERS: | | |
| Area 1 | 18.39 | 4.54 |
| Area 2 | 16.97 | 2.29+3% |
| SOFT FLOOR LAYERS: | | |
| AREA 1 | 14.022 | 2.50+b |
| TILE SETTERS: | | |
| Area 2 | 15.43 | 2.40 |
| OMIT: | | |
| LATHERS: | | |
| Area 1 | 13.55 | 4.87 |
| ELECTRICIANS: | | |
| Area 3: | | |
| Cable Splicers | 20.95 | 4.35+3% |
| ADD: | | |
| LATHERS (Statewide) | 13.55 | 4.87 |

DECISION NO. RI83-3042 - MOD #9
(48 FR 37809 - August 19, 1983)
Statewide, Rhode Island

CHANGE:

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| BRICKLAYERS & STONEMASONS & PLASTERING | 15.62 | 4.78 |



DECISION NO. TX84-4028 - MOD. #5
(49 FR 19207 - May 4, 1984)
Brazos County, Texas

Change:

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| Boilermakers | $16.125 | $2.95 |
| Glaziers | 13.67 | 2.50 |

DECISION NO. TX84-4005 - MOD. #8
(49 FR 7069 - 2-22-84)
Collin, Dallas, Denton, Ellis, Grayson, Hood, Hunt, Johnson, Kaufman, Palo, Pinto, Rockwell, Tarrant & Wise Counties, Texas

Change:

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| Sheet Metal Workers Zone 1 | $16.905 | $2.08 |
| Plumbers & Pipefitters Zone 1 | 16.75 | 2.19 |
| Lathers (excluding Grayson Co.) | 14.00 | 2.22 |
| Carpenters: | | |
| Zone 2: | | |
| Carpenters, pile-drivermen | 14.00 | 2.22 |
| Accoustical, Drywall, Insulation and Metal Door Frames | 11.78 | 2.22 |

DECISION NO. TX84-4020 - MOD. #8
(49 FR 14839 - April 13, 1984)
Galveston and Harris Counties, Texas

Change:

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| Boilermakers | $16.125 | $2.95 |
| Glaziers | 13.67 | 2.50 |

DECISION NO. TX84-4045 - MOD. #2
(49 FR 32168 - August 10, 1984)
Travis County, Texas

Change:

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| Carpenters | $14.91 | $2.18 |
| Millwrights | 15.16 | 2.18 |

DECISION NO. TX84-4015 - MOD. #4
(49 FR 10008 - March 16, 1984)
Wichita County, Texas

Change:

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| Bricklayers & Stonemasons | $14.25 | $1.75 |
| Boilermakers | 16.125 | 2.95 |

DECISION NO. WV83-3022
MOD. #13
(48 FR 52547 - November 18, 1983)
Statewide West Virginia excluding the counties of Berkley, Jefferson and Morgan.

CHANGE:

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| IRONWORKERS: | | |
| Area 3 | 15.97 | 4.25 |

SUPERSEDEAS DECISION

STATE: KANSAS
COUNTY: SHAWNEE
DECISION NO. KS84-4101
DATE: Date of Publication
Supersedes Decision No. KS84-4051, dated August 24, 1984 in 49FR 33782
Description of Work: Building Construction Projects (Excluding single family homes and apartments up to and including 4 stories)

| | BASIC HOURLY RATE | FRINGE BENEFITS |
|---|---|---|
| Asbestos workers | $11.61 | $ .89 |
| Bricklayers/Stonemasons | 14.14 | 1.25 |
| CARPENTERS | | |
| Carpenters | 12.45 | 1.80 |
| Millwrights | 13.45 | 1.80 |
| Cement Masons/Finishers | 12.60 | 1.05 |
| Electricians | 15.55 | 3% + 2.13 |
| Glaziers | 12.83 | 5.54 |
| Ironworkers | 16.25 | 4.25 |
| LABORERS: | | |
| Laborers, General | 8.12 | 1.18 |
| Mason Tenders | 9.60 | 2.30 |
| Painters | 13.60 | 1.25 |
| Plasterers | 15.70 | .01 |
| Plumbers/Pipefitters | 15.83 | 2.34 |
| Roofers | 14.66 | 1.29 |
| Sheet Metal Workers | 14.68 | 3% + 3.10 |
| Soft Floor Layers | 11.00 | 10% + 1.76 |
| Sprinkler Fitters | 16.47 | 3.23 |
| Tile Setters | 15.00 | |
| Truck Drivers | 11.10 | 1.47 |
| POWER EQUIPMENT OPERATORS: | | |
| Backhoe Operators | 11.80 | 3.77 |
| Crane Operators | 13.75 | 3.77 |
| Forklift Operators | 13.35 | 3.77 |
| Front End Loader Operator | 11.55 | 3.77 |

WELDERS: Receive rate prescribed for craft performing operation to which welding is incidental.

Unlisted classifications needed for work not included within the scope of the classifications listed may be added after award only as provided in the labor standards contract clauses (29 CFR, 5.5(a)(1)(ii)).

SUPERSEDEAS DECISION

STATE: Missouri & Kansas
COUNTIES: Cass,Clay,Jackson, Platte & Ray Missouri;Johnson & Wyandotte, Kansas
DECISION NO.: MO84-4102
DATE: Date of Publication
Supersedes Decision No. MO83-4046 dated June 10, 1983 in 48 FR 26995.
DESCRIPTION OF WORK: Residential construction consisting of single family homes and apartments up to and including 4 stories.

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| ASBESTOS WORKERS | 17.29 | 4.35 |
| BOILERMAKERS | 17.345 | 3.25 |
| BRICKLAYERS & STONEMASONS | 15.94 | 3.00 |
| CARPENTERS | 14.55 | 2.07 |
| CEMENT MASONS | 15.395 | 2.33 |
| ELECTRICIANS: | | |
| Buildings of (3) stories or less: | | |
| ZONE 1 - Johnson County, Kansas (that portion of Johnson County west of Aubry,Oxford & Shawnee Twps.) Cass,Clay,Jackson,Platte & Ray Counties, Missouri; & Wyandotte County,Kansas & remainder of Johnson County, Kansas | 11.37 | 10%+ 2.51 |
| Buildings of over (3) stories: | | |
| ZONE 1 - Western half of Clay & Jackson Cos., Missouri not including Blue Springs;Northern half of Platte County; Northwestern portion of Cass County,Missouri; not including Pleasant Hill | 16.18 | 10%+ 2.51 |
| ZONE 2 - Remainder of Cass,Clay,Jackson & Platte Counties, Missouri: | | |
| Electricians (Contracts exceeding 2000 man hours) | 16.18 | 10%+ 2.51 |
| Buildings of over (3) stories: | | |
| ZONE 3 - Electricians (contracts not exceeding 2000 man hours) | 15.18 | 10%+ 2.51 |
| ELECTRICIANS (CONT'D): | | |
| ZONE 4 - Ray County, Missouri | | |
| Electricians (contracts exceeding 2000 man hours | 16.18 | 10%+ 2.51 |
| Electricians (contracts not exceeding 2000 man hours | 14.58 | 10%+ 2.51 |
| ELEVATOR CONSTRUCTORS' | 16.89 | 2.69+a |
| ELEVATOR CONSTRUCTORS' HELPERS | 70%JR | 2.69+a |
| ELEVATOR CONSTRUCTORS' HELPER (PROB.) | 50%JR | |
| GLAZIERS | 14.24 | 2.96+ 17.14% |
| IRONWORKERS | 17.25 | 3.25 |
| LABORERS: | | |
| Building Construction: | | |
| ZONE I Cass,Clay, Jackson & Platte Cos. Missouri;Johnson & Wyandotte Cos., Kansas | | |
| GROUP I | 13.35 | 2.15 |
| GROUP II | 13.50 | 2.15 |
| GROUP III | 13.65 | 2.15 |
| ZONE II (Ray Co. Missouri) | | |
| GROUP I | 12.35 | 2.15 |
| GROUP II | 12.50 | 2.15 |
| GROUP III | 12.65 | 2.15 |
| LABORERS: | | |
| Site Preparation & Grading | | |
| ZONE 3 - Johnson & Wyandotte Counties, Kansas;Site Preparation, incidental paving & utilities Clay,Jackson,Platte & Ray Counties, Missouri | | |
| GROUP I | 12.52 | 3.45 |
| GROUP II | 13.32 | 3.45 |

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| TILE & MARBLE SETTERS | 16.55 | 10% |
| TILE SETTERS & MARBLE FINISHERS | 14.40 | |
| PAINTERS: | | |
| Brush,roller & tapers | 15.54 | 1.00 |
| Spray | 16.54 | 1.00 |
| PLASTERERS | 17.60 | |
| PIPEFITTERS | 17.44 | 3.22 |
| PLUMBERS | 15.02 | 2.70 |
| POWER EQUIPMENT OPERATORS: | | |
| Building Construction | | |
| (ZONE 1) | | |
| GROUP I | 15.86 | 2.85 |
| GROUP II | 15.51 | 2.85 |
| GROUP III: | | |
| (a) | 10.45 | 2.85 |
| (b) | 13.61 | 2.85 |
| (c) | 11.25 | 2.85 |
| (d) | 13.86 | 2.85 |
| GROUP IV | 16.11 | 2.85 |
| GROUP V | 15.76 | 2.85 |
| GROUP VI | 16.36 | 2.85 |
| GROUP VII: | | |
| (a) | 15.71 | 2.85 |
| (b) | 15.46 | 2.85 |
| (c) | 13.46 | 2.85 |
| GROUP VIII | 16.86 | 2.85 |
| GROUP IX | 16.36 | 2.85 |
| POWER EQUIPMENT OPERATORS: | | |
| Site preparation & grading, Johnson & Wyandotte Counties, Kansas;Site Preparation, incidental paving & utilities Clay, Jackson, Platte & Ray Cos., Missouri | | |
| (ZONE 2) | | |
| GROUP I | 14.60 | 4.07 |
| GROUP II | 14.35 | 4.07 |
| GROUP III | 13.65 | 4.07 |
| GROUP IV | | |
| (a) | 9.63 | 4.07 |
| (b) | 12.65 | 4.07 |
| GROUP V | 14.85 | 4.07 |
| ROOFERS: | | |
| Roofers | 16.48 | 2.91 |
| SHEET METAL WORKERS | 16.75 | 2.78 |
| SOFT FLOOR LAYERS | 12.44 | 1% + 3.34 |
| SPRINKLER FITTERS | 19.28 | 3.21 |

| | Basic Hourly Rates | Fringe Benefits |
|---|---|---|
| TERRAZZO WORKERS: | | |
| Terrazzo Workers | 15.31 | 10% |
| Terrazzo Finishers | 13.58 | |
| Terrazzo Base Machine Operator | 13.93 | |
| TRUCK DRIVERS: | | |
| Building Construction: | | |
| (ZONE 1) | | |
| GROUP I | 14.335 | 2.75 |
| GROUP II | 14.385 | 2.75 |
| GROUP III | 14.585 | 2.75 |
| GROUP IV | 14.46 | 2.75 |
| GROUP V | 14.485 | 2.75 |
| GROUP VI | 14.685 | 2.75 |
| GROUP VII | 14.535 | 2.75 |
| GROUP VIII | 14.435 | 2.75 |
| TRUCK DRIVERS: | | |
| Site preparation & grading, Johnson & Wyandotte Counties, Kansas; Site preparation, Incidental paving & utilities Clay,Jackson,Platte & Ray Cos., Missouri | | |
| (ZONE 2) | | |
| GROUP I | 13.82 | 4.00 |
| GROUP II | 13.67 | 4.00 |
| GROUP III | 13.36 | 4.00 |
| GROUP IV | 13.16 | 4.00 |
| GROUP V | 12.94 | 4.00 |

FOOTNOTES:
a - Employer contributes 8% of basic hourly rate for over 5 years of service & 6% of basic hourly rate for 6 mos. to 5 years service as Vacation Pay Credit. Also 7 paid holidays.



CLASSIFICATION DEFINITIONS

LABORERS: Building Construction (Zones 1 and 2)
GROUP I - General laborer;wiremesh handlers or setters; carpenter tender; track men; salamander tenders; landscape man; sod layers; wrecker (for alterations or entire projects); plumber labors (conduit pipe, sewer work, drain tile and duck lines, digg g and backfilling), power tool operators; pier hole diggers (over 10 ft.), jackhammer, and chipping hammer operators; chain saw operators; concrete saw operators; brush feeders on pulverizers; reinforcing steel handlers; air tamp operators; ditch winch operators; swinging scaffolds; georgie buggies (self-propelled); fork lift; hosemen; insulation man
GROUP II - Vibrator operator; fork lift (masonry), brick tender, plaster tender, stonemasons tender (includes all hod carriers classifications previously shown as mortar men and scaffolding)
GROUP III - Barco, Jackson or similar tamp operators; asphalt rakers; power men; mastic hot kettle men; sandblasting and gunnite nozzlemen; wagon and churn drill operators; cutting torch or burner men

LABORERS: Site Preparation (Zone 3)
GROUP I - Carpenter tenders; salamander tenders; loading trucks under bins, hoppers and conveyors, track men and all other general laborers; air tool operators; cement handler (bulk or sack); chain or concrete saw; deck hands; dump man on earth fill; grade checkers on cuts and fills; georgie buggies man; material batch hopper man; scale man; material mixer man (except on manholes); coffer dams, abutments and pier hole men working below ground; riprap pavers rock, block or brick; scaffolds over 10 ft. not self-supported from ground up; skipman on concrete paving; vibrator man; wire mesh setters on concrete paving; all work in connection with sewer, water, gas, gasoline, oil, drainage pipe, conduit pipe, tile and duct lines and all other pipe lines; power tool operator; all work in connection with hydraulic or general dredging operations; puddlers (paving only), crusher feeder; man handling creosote ties on creosote materials; men working with and handling epoxy material or materials (where special protection is required); topper of standing trees; batter board man on pipe and ditch work; feeder man on wood pulverizers; board and willow mat weavers and cable tiers on river work; all laborers working on underground tunnels where compressed air is not used.
GROUP II - Spreader or screed man on asphalt machine; asphalt raker; laser beam man; barco tamper; jackson or any other similar tamp wagon driller; churn drills; air track drills and all other similar drills; form setters; cutting torch man; liners and stringline men on concrete paving, curbs, gutters and etc.; hot mastic kettleman; hot tar application; hand blade operators; manhole builders tenders and mortar men on brick or block manholes; sandblasting and gunnite nozzlemen; rubbing concrete; air tool operator in tunnels; manhole builder (brick or block); dynamite and powderman; welder; head pipe layer or sewer work

**CLASSIFICATION DEFINITIONS** - (Cont'd)

POWER EQUIPMENT OPERATORS - Building Construction (Zone 1)

Group I - Asphalt paver and spreader; asphalt plant mixer operator; asphalt plant operator; back fillers; backhoe; barber-greene loader; blade-power; boats-power; boilers (2); boring machines; cableways; cherry pickers; chip spreader; concrete ready-mixed plant, portable (job site); concrete mixer paver; crane-overhead; crusher, rock; derricks and derricks cars (power operated); ditching machines; dozers; dredges - any type power; grade-all - similar type; hoist, endless chain-power operator with power travel; loaders; mechanic and welder; mucking machine; orange peels; pumps - material; push cats; scoops; self-propelled rotary drill; shovel, power; side boom; skimmer scoop; testhole machine; throttle man; locomotives

GROUP II Boilers (1); Brooms - power operated; chip spreader (front man); clef plane operator; compressors (1) 125' or over; concrete saws, self-propelled; crab - power operated; curb finishing machine; firemen on rigs; flex plane; floating machine; form grader; greaser; hoist, endless chain - power operated; hopper - power operated; hydra hammer; lad-a-vator - similar type; rollers; siphons, jets, and jennies; sub-grader; tractors over 50 h.p.; compressors (2) 125' ft. or over not more than 20' apart; compressors-tandem; compressors single, truck mounted; elevator; finishing machine

Group III -

(a) Oilers
(b) Fork lift-masonry
(c) Oiler driver
(d) A-frame trucks; fork lift-all types (except masonry); mixers (w/side loaders); pumps (w/well points) dewatering systems, test or pressure pumps; tractors (except when hauling material) less than 50 h.p.

Group IV -

Clamshells, 100 ft. of boom or over (excl. jib); crane or rigs, 100 ft. of boom or over (excl. jib); draglines, 100 ft. of boom or over (excl. jib); pile drivers, 100 ft. of boom or over (excl. jib)

Group V

Hoists-each additional drum over 1 drum

Group VI

Crane or rigs, over 200 ft. of boom

Group VII

Ready Mixed Concrete Plants:

(a) Crane operator
(b) Loader operator & plant man
(c) Conveyor Operator

Group VIII

Master Mechanic

Group IX

Crane-tower or climbing



**CLASSIFICATION DEFINITIONS** - (Cont'd)

POWER EQUIPMENT OPERATORS: Site Preparation (Zone 2)

GROUP I - Asphalt paver and spreader; asphalt plant console operator; auto grader; backhoe; blade operator, all types; boilers-2; booster pump on dredge; boring machine (truck or crane mounted); bulldozer operator; clamshell operator; compressor maintenance operator-2; concrete plant operator, central mix; concrete mixer paver; crane operator; derrick or derrick trucks; ditching machine; dragline operator; dredge engineman; dredge operator; drill-cat with compressor mounted on cat; drilling or boring machine, rotary, self-propelled; high loader-fork lift; hoistline engine-2 active drums; locomotive operator, standard gauge; mechanics and welders; maintenance operator; mucking machine; pile driver operator; pitman crane operator; pump-2; push cat operator; quad-track; scoop operator-all types; scoops in tandem; self-propelled rotary drill (leroy or equal-not air trac); shovel operator; side discharge spreader; sideboom cats; skimmer scoop operater; slip-form paver (CMI, REX, or equal); throttle man; truck crane; welding machine maintenance operator-2.

GROUP II - A-frame truck; asphalt hot mix silo; asphalt plant fireman, drum or boiler; asphalt plant mixer operator; asphalt plant man; asphalt roller operator; back filler operator; chip spreader; concrete batch plant, dry-power operated; concrete mixer operator, skip loader; concrete pump operator; crusher operator elevating grader; greaser; hoisting engine-1 drum; letourneau rooter; multiple compactor; pavement breaker, self propelled, of the hydra-hammer or similar type; power shield; pug mill operator; stump cutting machine; towboat operator, tractor operator over 50 h.p..

GROUP III - Boilers-1; chip spreader (front man); churn drill operator; compressor maintenance operator-1; concrete saws, self-propelled; conveyor operator; distributor operator; finishing machine operator; fireman, rig; float operator; form grader operator; pump; pump maintenance operator, other than dredge; roller operator, other than high type asphalt; screening and washing plant operator; self-propelled street broom or sweeper; siphons and jets; sub-grading machine operator; tank car heater operator-combination boiler and booster; tractor, 50 h.p. or less, without attachments; vibrating machine operator, not hand; welding machine maintenance operator-1.

GROUP IV

(a) Oiler
(b) Oiler drivers, all types

GROUP V - Clamshells, 3 yds. capacity or over; crane or rigs, 80 ft. of boom or over (including jib); draglines, 3 yds. capacity or over; piledrivers, 80 ft. of boom or over (including jib); shovels & backhoes, 3 yds. capacity or over

DECISION NO.: MO84-4102 Page 6

CLASSIFICATION DEFINITIONS (Cont'd)

TRUCK DRIVERS - Building Construction (Zone 1)
GROUP I - Warehousemen and stock man.
GROUP II - Flat beds; pick-ups; dump trucks, under 10 yds.
GROUP III - Straddle trucks, wheel tractors (when used for towing); hydro lift trucks, hydraulically operated serial lifts; heavy hauling, A-frame and winch fork trucks; heavy excavating (dumpster, euclid, etc.); double bottom units (20 tons capacity and over)
GROUP IV - Dump trucks, 10 yds. and over; steel drivers; semi truck drivers
GROUP V - Distributor truck drivers and operators; oilers, greasers and mechanics' tenders
GROUP VI - Mechanics; transitmix;tractor trailer
GROUP VII - Transit mix, 5 yds. and over
GROUP VIII - Transit mix, under 5 yds.

TRUCK DRIVERS - Site Preparation (Zone 2)
GROUP I - Mechanics and welders
GROUP II - A-frame, lowboy and boom truck driver
GROUP III - Material trucks, tandem; two teams; semi-trailers; winch trucks-fork trucks; distributor drivers and operators; agitator and transit mix; tank wagon drivers; single axle; tank wagon drivers tandem or semi-trailers; insley wagons; dump trucks, excavating, 5 cu. yds. and over; dumpsters; half-tracks; speedace; euclids and other similar excavating equipment.
GROUP IV - One team; station wagons; pickpu trucks; material trucks, single axle; tank wagon drivers, single axle
GROUP V - Mechanics tender, oilers and greasers, field

WELDERS: Receive rate prescribed for craft performing operation to which welding is incidental.

Unlisted classifications needed for work not included within the scope of the classifications listed may be added after award only as provided in the labor standards contract clauses (29 CFR, 5.5(a)(1)(ii)).

SUPERSEDEAS DECISION

STATE: VIRGINIA

COUNTIES: Amherst, Appomattox, Buckingham, Campbell, Charlotte, Cumberland, Halifax, Nelson, Pittsylvania, Prince Edward, and The Cities of Danville, Lynchburg, and So. Boston

DECISION NO. 84-VA-3040 DATE: Date of Publication

SUPERSEDES DECISION NO. VA79-3050 dated November 9, 1979 in 44 FR 63614.

DESCRIPTION OF WORK: Highway Construction

| | Basic Hourly Rates | Fringe Benefits | Basic Hourly Rates | Fringe Benefits |
|---|---|---|---|---|
| Asphalt Raker | 5.42 | | | |
| Carpenter, Structure | 6.04 | | | |
| Carpenter Helper, Structure | 4.86 | | | |
| Concrete Finisher | 6.00 | | | |
| Concrete Finisher Helper | 5.15 | | | |
| Fence Erector/Guard Rail | 5.43 | | | |
| Form Setter Road | 5.03 | | | |
| Ironworker, Reinforcing | 6.03 | | | |
| Laborer, Unskilled | 4.36 | | | |
| Landscape Worker | 4.50 | | | |
| Mechanic | 6.57 | | | |
| Mechanic Helper | 5.48 | | | |
| Pipelayer | 4.36 | | | |
| Powderman Blaster | 4.36 | | | |
| Asphalt Distributor Op. | 5.71 | | | |
| Asphalt Paver Op. | 5.80 | | | |
| Backhoe Operator | 5.37 | | | |
| Bulldozer Operator | 5.53 | | | |
| Crane, Derrick, Dragline Op. 1 yd. and under | 7.00 | | | |
| Over 1 yard | 7.00 | | | |
| Drill Operator | 4.55 | | | |
| Loader Operator: 2 yds. & under | 5.54 | | | |
| Over 2 yds | 5.54 | | | |
| Motor Grader Operator-Fine Grade | 5.81 | | | |
| Motor Grader Operator-Rough Grade | 5.71 | | | |
| Roller Operator-Rough | 4.59 | | | |
| Roller Operator-Finish | 5.31 | | | |
| Scraper-Pan Operator | 5.37 | | | |
| Slurry Seal Machine Op. | 5.37 | | | |
| Slurry Seal Paver Truck Driver | 4.93 | | | |
| Stone Spreader Operator | 5.45 | | | |
| Tractor Operator-Utility | 4.58 | | | |
| Truck Driver, Heavy Duty: 7. c.y. and under | 4.82 | | | |
| over 7. c.y. | 4.82 | | | |
| Truck Driver-Single Rear Axle | 4.72 | | | |
| Truck Driver Multi Rear Axle | 4.72 | | | |

[FR Doc. 84-28086 Filed 10-25-84; 8:45 am]

BILLING CODE 4510-27-C



Welders - Receive rate prescribed for craft performing operation to which welding is incidental.

"Unlisted classifications needed for work not included within the scope of the classifications listed may be added after award only as provided in the labor standards contract clauses (29 CFR, 5.5(a)(ii))."

federal register

Friday
October 26 198

Part III

# Department of Transportation

## Federal Aviation Administration

14 CFR Parts 25 and 121
Floor Proximity Emergency Escape Path Marking; Final Rule

**DEPARTMENT OF TRANSPORTATION**

**Federal Aviation Administration**

**14 CFR Parts 25 and 121**

**[Docket No. 23792: Amdt. Nos. 25-58 and 121-183]**

## Floor Proximity Emergency Escape Path Marking

**AGENCY:** Federal Aviation Administration (FAA), DOT.
**ACTION:** Final rule.

**SUMMARY:** This amendment establishes new performance standards for floor proximity emergency escape path marking to provide visual guidance for emergency cabin evacuation when all sources of cabin lighting more than 4 feet above the aisle floor are totally obscured by smoke. This amendment makes the standards applicable to future type certification of transport category airplanes and, after November 26, 1986, to airplanes type certificated after January 1, 1958, and operating under Part 121. These standards represent a significant improvement in aircraft cabin safety and are in addition to the emergency lighting standards currently in the regulations.

**EFFECTIVE DATE:** November 26, 1984.

**FOR FURTHER INFORMATION CONTACT:** Henri Branting, Technical Analysis Branch (AWS-120), Aircraft Engineering Division, Office of Airworthiness, Federal Aviation Administration, 800 Independence Avenue, SW., Washington, D.C. 20591; Telephone (202) 426-8382.

**SUPPLEMENTARY INFORMATION:**

### Background

On August 23, 1983, the Federal Aviation Administration (FAA) issued Notice of Proposed Rulemaking (NPRM) No. 83-15 (48 FR 46218; October 11, 1983). This notice proposed new performance standards for floor proximity emergency escape path marking to provide visual guidance for emergency cabin evacuation when all sources of cabin lighting more than 4 feet above the aisle floor are totally obscured by smoke. The notice proposed to make the standards applicable to future type certification of transport category airplanes and to most airplanes operating under Part 121 of the Federal Aviation Regulations (FAR). These proposed standards would be in addition to the emergency lighting standards currently in the regulations.

The notice responded to certain findings of the Special Aviation Fire and Explosion Reduction (SAFER) Advisory Committee and was based on the results of research, development, and testing conducted by the FAA.

The SAFER Advisory Committee was established in June 1978 by the FAA as a result of information from public hearings on aircraft fire safety. The FAA directed the Committee to "examine the factors affecting the ability of the aircraft cabin occupant to survive in the post-crash environment and the range of solutions available." The Committee consisted of 24 representatives of a wide range of aviation and general public interests. Technical support groups included approximately 150 of the world's top experts in fire research, accident investigation, materials development, and related fields. The Committee found that accident experience indicates smoke from burning fuel and cabin material can obscure overhead emergency lighting and make cabin evacuation difficult. Therefore, the Committee recommended that consideration be given to placing additional sources of lighting at a lower level in the relatively clear air near the cabin floor. The FAA accepted the Committee recommendation and conducted the research, testing, and design studies necessary to develop this proposed floor proximity marking concept.

Current regulations require that emergency lighting provide specific illumination at seat armrest level. The sources of this emergency illumination are typically located overhead in the cabin ceiling area. Service experience shows that the current regulations effectively ensure that the airplane's main aisles, cross aisles, passageways, and emergency exits are capable of sustaining raid mass evacuation under critical conditions over a reasonably extended period of time. However, the regulations do not adequately cover the brief interval between the time buoyant hot smoke and gases might begin to fill the upper portion of the cabin and extend down to near floor level, obscuring all overhead lighting, and the time the cabin is not survivable. While this condition is extreme, it is considered desirable to address this in the aircraft design, and safety could be improved through the use of lights, lights and reflectors, or other devices to provide floor proximity emergency escape path marking.

The FAA conducted a series of laboratory tests to look into the problems of emergency lighting in conditions of dense smoke and to study practical ways of developing improved lighting systems for transport category airplane cabins. A design feasibility and cost study of floor proximity emergency escape path marking was conducted under FAA contract. The results of this study are published in FAA Report No. DOT/FAA/CT-83/31, Improved Interior Emergency Lighting Study, dated September 1983, available from the National Technical Information Service, Springfield, Virginia 22161. A copy of this report is in the docket available for inspection upon request. The economic analysis for the notice was based on data from this study. Eleven candidate systems were considered in this study; and although individual systems were found to have certain advantages compared to others, no system was so clearly superior to the others that it warranted its establishment through regulation as the single standard for floor proximity emergency escape path marking in general. Notice 83-15 pointed out that there might be any number of combinations of point lighting, flood lighting, strip lighting, markers, signs, reflective materials, and other components that could adequately serve the objective of floor proximity marking. Therefore, the notice proposed an objective performance standard rather than requiring a particular system. A performance standard in this case would allow industry the flexibility to choose among the various existing systems or to develop new systems.

The standard proposed in Notice 83-15 would require that floor proximity emergency escape path marking provide emergency evacuation guidance for passengers when all sources of illumination more than 4 feet above the cabin aisle floor are totally obscured. It proposed that in dark of the night conditions, the floor proximity emergency escape path marking must enable each passenger to: (1) Visually identify the emergency escape path along the aisle of the cabin floor after leaving a cabin seat; and (2) Readily identify each exit from the emergency escape path by reference only to markings and visual features not more than 4 feet above the cabin floor. The marking system performance would be proven under dark of the night conditions the same as those specified in the emergency evacuation demonstration requirements of § 25.803 of the Federal Aviation Regulations (FAR).

The proposal would require that airplanes type certificated after January 1, 1958, and operating under Part 121 comply with the new standard within 2 years after the standard became effective. The limited number of airplanes type certificated before January 1, 1958, operating under Part 121 were not included because the relatively advanced age and smaller sizes of these

airplanes would make compliance impractical from an economic standpoint. The 2-year period was intended to allow air carriers lead time to schedule the modifications necessary for compliance to coincide with major maintenance inspections and, therefore, avoid an undue compliance burden.

Comments and data on marking systems capable of meeting the proposed performance standard were specifically requested in the notice, with the stipulation that they would be considered in the publication of advisory material on acceptable means of compliance.

**Public Participation**

These amendments are based on Notice 83–15. All interested parties have been given an opportunity to participate in the making of these amendments, and due consideration has been given to all matters presented. Except for the changes discussed below, these amendments and the reasons for their adoption are the same as those stated in Notice 83–15.

**Discussion of Comments**

Twenty-four comments were received in response to Notice 83–15, representing the views of aircraft and equipment manufacturers, aircraft operators, aircraft crew organizations, U.S. and foreign government organizations, and consumer interests. All of the comments support the safety objective of the proposal, although various commenters disagree with certain technical aspects of the proposal.

Eight commenters disagree with the proposed applicability of the new marking requirements to airplanes certificated under FAR Part 25 and those operated under FAR Part 121. These commenters contend in general that applicability should depend on aircraft seating capacity, cabin size, type of operation, or similar factors. Four of these commenters oppose applicability to the relatively smaller airplanes with maximum seating capacities ranging from 30 to 60, depending on the view of the commenter. Three of the commenters favor extending the applicability to the type certification of rotorcraft and the operation of aircraft under regulations other than FAR Part 121, such as FAR Part 135.

Notice 83–15 did not address aircraft certificated under Parts 23, 27, or 29 or operations conducted under Part 135 because it was made in response to a recommendation by the SAFER Advisory Committee which limited its investigation to transport category airplanes. Notice 83–15 intended to establish additional requirements in Part 25 and Part 121 for the type of aircraft and operation for which emergency lighting is recognized as most critical and for which the most stringent lighting requirements have been established. Other regulations contain lighting and marking requirements considerably less stringent. The research, development, and testing and the feasibility and cost study which supported the proposal were based on the type of transport category airplane typically used in Part 121 operations. The proposal did not include rotorcraft or the types of airplanes operated under Part 135 because of their relatively smaller cabin sizes, shorter aisle lengths, and shorter seat-to-exit distances compared to transport category airplanes.

The FAA does not agree that applicability of these lighting requirements should be determined by the passenger capacity of an airplane operated under Part 121 or type certificated under Part 25. The transport category airworthiness standards require extensive emergency lighting and evacuation markings for all transport category airplanes, regardless of passenger capacity, although they do recognize that for relatively small cabins seating 9 or less passengers smaller evacuation markings provide the required level of safety and obviate the imposition of an impractical standard (since small cabins may not be able to accommodate larger exit signs). These cabins may use smaller emergency exit signs with lower illumination than the larger cabins. The objective performance standard will accommodate the "smaller" cabins by permitting the design of a marking system to suit the cabin size. Therefore, the applicability of the requirement is adopted as proposed.

Nine commenters express views on the 2-year compliance period in proposed § 121.310(c)(3). Three commenters favor increasing it to 3 years. Three favor accelerating compliance. Three concur with the 2 years. The comments favoring an increase to 3 years contend that the marking requirements are complex and that additional time should be allowed for development and approval of new designs, procurement of equipment, and the installation of systems.

The 2-year period will provide operators lead time to schedule modifications to coincide with major maintenance checks and therefore to avoid an undue compliance burden. The 2-year compliance period recognizes that the scheduling of major maintenance checks varies widely among carriers and individual airplanes depending on route structure and airplane utilization rate and in many cases exceeds 1 year. A significant reduction in the compliance period would conflict with the intent of the 2-year period and likely result in added airplane down-time for a number of carriers. The FAA anticipates that carriers operating large fleets of various types of aircraft may need more than 1 year to design, procure, and install the new lighting systems and to revise their technical manuals to include appropriate information and guidance regarding the new systems. Concerning extending the compliance period to 3 years, there is no indication in the comments that long-range modification scheduling could not begin some time during the design development and approval phase. Although the marking systems are not necessarily complex in design and construction, the 2-year period provides sufficient time for development and approval of new designs, procurement of equipment, and the installation of systems. Therefore, the 2-year compliance period is adopted as proposed.

One commenter contends the proposed standard infers that a separate floor proximity marking system is required. The standard, as adopted, does not require a separate system. A single system might meet the requirements for both general illumination and floor proximity marking.

Several commenters contend the wording of the proposed standard implies that marking/lighting is required along the aisle. One commenter says that aisle marking/lighting should not be necessary because a person leaving a seat needs no guidance to find the aisle, even in total darkness. The commenter says that with information provided by the pre-takeoff briefing and the passenger information card, the passenger will know the location of exits and, once in the aisle, can proceed to an exit, using the aisle as tactile guidance. Several commenters contend that marking/lighting should be necessary only for exits located off the main aisle.

The FAA agrees that visual guidance is not needed to enable a passenger to move from the seat to the aisle. The standard does not require this. The standard requires visual guidance for the aisle escape path and the exits. It does not require marking/lighting along the aisle, although this might be one means of complying with the standard. The standard does not preclude compliance by the use of conspicuous lighting or marking near the ends of the aisle or at other critical points along the

aisle. The FAA does not agree that passenger exits on the main aisle need no floor proximity marking since without marking there would be no means by which passengers could readily identify the exits when overhead lighting is obscured. Notice 83–15 explains that under this objective standard, there might be many different combinations of point lighting flood lighting, strip lighting, markers, signs, reflective materials, and other components that could provide adequate visual guidance. The FAA does not agree with the commenter that tactile guidance alone for the aisle is sufficient. Under § 25.811(c), tactile guidance currently may be used for locating exits in dense smoke. For typical airline passengers unfamiliar with cabin features, visual guidance is more effective than tactile.

One commenter points out that in an actual fire, smoke might occur below the 4-foot level and that visual performance requirements should be based on defined optical density for smoke.

The FAA does not agree. It is impractical to define the fire scenario to this extent for the purpose of floor proximity marking design. In an actual fire, the obscuring layer might vary along the length of the cabin well above and below 4 feet. Notice 83–15 acknowledges that when lights are totally obscured at 4 feet, the effective height of clear air would be somewhat less. Four feet is a nominal design height, and the air below this is deemed clear for the purpose of floor proximity marking design.

Two commenters point out that floor proximity marking should not indicate a particular direction in which the passenger should move in an emergency, as this would depend on the nature of the emergency.

The FAA agrees. The proposed standard was intended to provide an indication to each passenger of the nearest exits forward and aft, thus giving the passenger a choice, depending on the location of the passenger's seat. The rule has been revised to make this intent clear. The direction in which the passenger chooses to move in an actual emergency would depend on conditions in the cabin.

In response to one comment, § 25.812(e) has been clarified by using the term "cabin aisle floor" throughout.

Many commenters express views regarding the objective form of the standard. While several commenters support the objective form, others disagree, contending that the lack of specific requirements leaves much of the compliance finding to subjective judgment and that this will not permit a uniform application of the standard throughout industry. Several commenters recommend revising the standard to specify the location, illumination, and luminosity of lighting, markers, and signs, similar to current regulations on emergency lighting.

Notice 83–15 explains why the proposed standard, for practical reasons, defines objective rather than specific requirements. The notice explains that no marking system appears so superior to others that it warrants establishment as the single standard for marking in general. There is a notable difference between the general illumination, which is required in specific terms by current regulations, and floor proximity marking. General illumination is intended to enable a large number of passengers to orient themselves within the cabin, receive instructions and assistance from crewmembers, and queue up quickly at usable exits. General illumination is necessary for this. Non-illuminating markers will not suffice. Current regulations prescribe specific illumination for aisles and passageways. Floor proximity marking is intended to allow passengers who have become familiar with the cabin layout during the period of general overhead illumination prior to an accident to find their way to exits unassisted should the general overhead illumination become obscured by smoke. There are many combinations of lights, markers, and signs which might serve this objective in a cost beneficial manner, and each must be shown adequate for the particular cabin interior and exit arrangement in question. Therefore, a performance standard is used to allow design flexibility and at the same time ensure the necessary safety.

**Discussion Regarding Compliance With the Objective Performance Standard**

Several commenters contend that because the objective performance standard does not specifically define a means of compliance, the FAA should publish advisory material concurrently with the rule adoption to provide guidance for the application of the standard. The FAA recognizes the value of guidance material for the introduction of a new performance standard. Notice 83–15 specifically requested comments and data on marking systems capable of meeting the proposed performance standard for use in the development of advisory material on means of compliance. Although the response to this request did not provide information on marking concepts which have been evaluated or found acceptable under the standard, it did identify those aspects of the standard for which additional clarification is appropriate.

In response to the comments, the discussion below addresses all aspects of the standard necessary for the development of a means of compliance. This discussion centers on the objective of floor proximity marking and the emergency conditions the marking is intended to counter. This information in conjunction with established FAA airworthiness evaluation and approval procedures will permit the determination of means of compliance which will ensure the consistent and uniform application of the standard. With this information, publication of additional guidance material is not necessary at this time.

Notice 83–15 explains that floor proximity marking is intended to counter conditions which might occur some time after the start of the type of emergency evacuation which is simulated in the demonstration required by § 25.803. In this demonstration, test subjects representing typical airline passengers in a cabin filled to capacity must evacuate the cabin in dark of the night conditions within 90 seconds, using emergency lighting only and with one-half the number of emergency exits rendered inoperative. Typically in a mass evacuation of this type, passengers immediately leave their seats and form queues at operable exits which generally are ready for use within 15 seconds. For the next 1 to 1½ minutes, the passengers in queues await their turn to escape through emergency exits and descend to the ground by escape slides. Emergency lighting is sufficient to enable the passengers to see the features of the cabin interior.

In actual emergency evacuation involving a fuel spill fire or cabin fire, smoke might begin to fill the upper portion of the cabin and eventually obscure emergency lighting and signs. This is the condition floor proximity marking is intended to counter. By the time this condition prevails in an emergency evacuation, the evacuee flows are most likely well established or completed. The floor proximity escape path marking is intended to enable unassisted passengers who might remain in the airplane to find emergency exits when overhead lighting is obscured.

A floor proximity emergency escape path marking system might be shown to meet the objective of the standard by means of the types of demonstrations discussed below or by means of analysis based on comparison of the marking system and cabin features with a marking system and cabin features

previously approved by such types of demonstrations.

Demonstrations, if used, are a means to show that test subjects representing typical passengers can leave any passenger seat in the cabin and once in the walkway area immediately adjacent to the seat can proceed to the first exit, or pair of exits, forward and aft of the seat, and can make positive identification of the exits. A walkway area in this sense is an aisle or any other area beyond the seats which a passenger traverses to reach an exit. While demonstrations should not be necessary for each seat or seat row, they are appropriate for those walkway areas adjacent to passenger seats which are determined to be the more critical from the standpoint of visual reference and the objective of the performance standard. A sufficient number of demonstrations should be conducted to ascertain that test subjects can identify all of the passenger emergency exits in the cabin.

In a fire situation severe enough to obscure all overhead lighting, some passengers might find themselves alone or in nearly vacated sections of the cabin, without the benefit of crewmembers, queues of passengers, individual passengers, human voices, or other cues to aid them in finding and identifying exits. This would be the most adverse situation from the standpoint of orientation and sense of direction and would require the passenger, alone and unassisted, to rely solely on the visual guidance provided by the floor proximity marking system and the passenger's familiarity with the cabin exit arrangement acquired during the passenger briefing under conditions of general illumination. This situation should be accounted for in the demonstration of compliance.

Demonstrations should be conducted either during the dark of the night or during daylight with dark of the night conditions simulated. If the demonstrations are conducted during daylight hours, each window, door, emergency exit (open and closed), and other openings should have provisions to prevent daylight from entering the passenger cabin. Each internal door and curtain should be in the takeoff configuration. During the demonstrations, only the marking system subject to the show of compliance should provide light. Since the demonstrations pertain to visual reference and orientation, and not to egress performance and evacuation rate, the distribution of articles to create minor obstructions in the aisle, as mentioned in § 23.803(c)(11) for full-scale evacuation demonstrations, should not be necessary.

These demonstrations are intended to confirm the efficacy of floor proximity markings when all lighting more than 4 feet above the cabin aisle floor is totally obscured by dense smoke. Obviously, in an actual fire situation, illumination from the floor proximity system would be confined to within the 4 feet beneath the overlaying smoke and would not illuminate or reflect throughout the cabin in general. In a demonstration in which there is no overlaying smoke, illumination from the floor proximity system might reflect into the upper cabin and produce unrealistic illumination for the cabin and escape path. A single light might be sufficient to illuminate a large area of the cabin. Unrealistic reflections and illumination should be accounted for in demonstrations, either through a rational determination that they do not change the validity of the demonstration results or through the use of shielding or shrouding, if necessary, to minimize or eliminate their effects.

Persons used as subjects for the demonstrations should be ambulatory adults in normal health. Except for information obtained from the pre-takeoff briefing and passenger information card, and from the instructions given immediately prior to the demonstration, test subjects should not have practiced or rehearsed or have had the demonstration procedures described to them within the past 6 months. Crewmembers, mechanics, training personnel, and any other persons who are familiar with the interior features of the cabin through the normal course of their duties should not be used as test subjects. A test subject should not perform demonstrations for more than one walkway area.

For each critical walkway area in the cabin, demonstrations should be performed by at least the following test subjects individually: one male, one female, and one person over 60 years of age, male or female. Test subjects need not include children.

Prior to the demonstration, the test subject should be given a passenger information card and be in a seating area in a normally illuminated cabin in a position to see and hear the pre-takeoff passenger briefing required by § 121.571. One or more observers should be in the cabin. After the pre-takeoff briefing and shortly before the demonstration, the subject should be informed of the objective and procedures of the demonstration.

In each demonstration, the test subject acting alone and without assistance should be able to : (1) Leave the passenger seat or seat row and enter the walkway area immediately adjacent (Visual reference to the escape path marking need not be used to assist the test subject in locating the walkway area immediately adjacent to the seat or seat row); (2) Standing or stooping in the adjacent walkway area, identify from visual reference to the floor proximity marking system the direction(s) of the first exit or pair of exists forward and aft and indicate to the observer the means by which identification is made; (3) Traverse to those exits without significant hesitation, delay, or evidence of confusion; and (4) Make positive identification of the exits by visual reference to features not more than 4 feet above the cabin floor and indicate to the observer the means by which identification is made. The exits may be open or closed for the demonstration. Safety precautions should be taken for open exists. Identification should be made for at least one exit of each type and marking system in the cabin, in both the open and closed positions.

**Economic Analysis**

One commenter states that the cost of engineering design, documentation, and demonstration should be added to the estimated costs. The FAA agrees and has increased the cost estimates by about 10 percent. For the 1983 aircraft fleet, the cost to retrofit is estimated to be $22.95 million, including the cost impact of additional weight. Another commenter states that the benefits cannot be quantified with any degree of accuracy. Clearly, it is impossible to accurately determine the number of persons who would have died in accidents involving fire who will now live because of this rule. However, decisions must be made relating to safety based on the best available information. The historical fire fatalities for the period 1965 through 1983 were 712. Extrapolating this fire fatality rate over the next 10 years, the expected life of the lighting system, the FAA estimates that about 10 percent of that number will have to be saved to justify the cost. The FAA's judgment is that a sufficient number of persons will be saved to justify the cost. The Regulatory Evaluation which has been placed in the docket contains a cost benefit analysis of the rule.

*Trade Impact*

These rules will have little or no impact on U.S. or foreign trade. In the United States, both foreign and domestic manufacturers must meet the proposed requirements, and there will be no competitive advantage to either. In

foreign countries, there would be a minor cost advantage only if the foreign country did not require the floor proximity emergency escape path marking system. Since the cost of the marking system is negligible compared to the total costs of new aircraft, there is essentially no impact on trade.

**Conclusion**

Under the terms of the Regulatory Flexibility Act (the Act), the FAA has reviewed this proposal to determine what impact it might have on small entities. Since the projected cost of compliance could be between $5,500 and $17,400 for each aircraft in the Part 121 fleet, the FAA has determined that this rule, if adopted, may have a significant economic impact on a substantial number of small entities. Consequently, a regulatory flexibility analysis and regulatory evaluation has been prepared. It is contained in the docket which is open to public inspection. A copy of the evaluation may be obtained by contacting the person identified under the caption **"FOR FURTHER INFORMATION CONTACT."**

As required by the Act, various regulatory alternatives were considered, such as: Making the requirements applicable only to new airplanes, having different standards based on the size of the air carrier, letting the air carrier industry decide whether to use the new systems, and requiring all airplanes operating under Part 121 to come into compliance with the requirements within a certain time period. Safety needs are such that the FAA has selected the latter alternative set forth in these amendments. The alternative of making the requirements applicable only to new airplanes was rejected because of the delay this would cause implementing the new standards throughout the fleet. The alternative of having different standards based on the size of the air carrier was rejected because the FAA believes all members of the traveling public should be equally protected. The alternative of letting the air carrier industry decide whether to use the new systems was rejected because in the past a voluntary approach to new equipment has not resulted in fleetwide implementation of desired safety advances.

These amendments are not likely to result in an annual effect on the economy of $100 million or more or a major increase in costs for consumers; industry; or Federal, State, or local government agencies. In addition, these amendments will have little or no impact on trade opportunities for U.S. firms doing business overseas or for foreign firms doing business in the United States. Accordingly, it has been determined that this is not a major regulation under Executive Order 12291. In addition, the FAA has determined that this action is significant under Department of Transportation Regulatory Policy and Procedures (44 FR 11034; February 26, 1979).

**List of Subjects**

*14 CFR Part 25*

Air transportation, Aircraft, Aviation safety, Safety, Tires.

*14 CFR Part 121*

Aviation safety, Safety, Air carriers, Air transportation, Aircraft, Airplanes, Airworthiness directives and standards, Flammable materials, Transportation, Common carriers.

**Adoption of the Amendment**

Accordingly, Parts 25 and 121 of the Federal Aviation Regulations (14 CFR Parts 25 and 121) are amended as follows effective November 26, 1984:

## PART 25—AIRWORTHINESS STANDARDS: TRANSPORT CATEGORY AIRPLANES

1. By amending § 25.812(a)(1) by removing the phrase "and interior lighting in emergency exit areas" and inserting, in its place, the phrase "interior lighting in emergency exit areas, and floor proximity escape path marking".

2. By amending § 25.812 by redesignating present paragraphs (e) through (k) as paragraphs (f) through (l).

3. By amending § 25.812 by adding a new paragraph (e) as follows:

**§ 25.812 Emergency lighting.**

* * * * *

(e) Floor proximity emergency escape path marking must provide emergency evacuation guidance for passengers when all sources of illumination more than 4 feet above the cabin aisle floor are totally obscured. In the dark of the night, the floor proximity emergency escape path marking must enable each passenger to—

(1) After leaving the passenger seat, visually identify the emergency escape path along the cabin aisle floor to the first exits or pair of exits forward and aft of the seat; and

(2) Readily identify each exit from the emergency escape path by reference only to markings and visual features not more than 4 feet above the cabin floor.

* * * * *

4. By changing the reference in the introductory text of newly designated paragraph (f) of § 25.812 from "paragraph (g)" to "paragraph (h)".

## PART 121—CERTIFICATION AND OPERATIONS: DOMESTIC, FLAG, AND SUPPLEMENTAL AIR CARRIERS AND COMMERCIAL OPERATORS OF LARGE AIRCRAFT

5. By amending § 121.310 by revising paragraph (c) to read as follows:

**§ 121.310 Additional emergency equipment.**

* * * * *

(c) *Lighting for interior emergency exit markings.* Each passenger-carrying airplane must have an emergency lighting system, independent of the main lighting system. However, sources of general cabin illumination may be common to both the emergency and the main lighting systems if the power supply to the emergency lighting system is independent of the power supply to the main lighting system. The emergency lighting system must—

(1) Illuminate each passenger exit marking and locating sign;

(2) Provide enough general lighting in the passenger cabin so that the average illumination when measured at 40-inch intervals at seat armrest height, on the centerline of the main passenger aisle, is at least 0.05 foot-candles; and

(3) For airplanes type certificated after January 1, 1958, after November 26, 1986, include floor proximity emergency escape path marking which meets the requirements of § 25.812(e) of this chapter in effect on November 26, 1984.

* * * * *

6. By changing the reference in the introductory text of paragraph (d) of § 121.310 from "§ 25.812(g)" to "§ 25.812(h)".

(Secs. 313(a), 314(a), 601 through 610, and 1102 of the Federal Aviation Act of 1958 (49 U.S.C. 1354(a), 1355(a), 1421 through 1430, and 1502); 49 U.S.C. 106(g) (Revised, Pub. L. 97-449, January 12, 1983))

Issued in Washington, D.C., on October 22, 1984.

**Donald D. Engen,**

*Administrator.*

[FR Doc. 84-28293 Filed 10-23-84; 2:09 pm]

BILLING CODE 4910-13-M

federal register

Friday
October 26, 1984

Part IV

# Department of Transportation

## Federal Aviation Administration

14 CFR Parts 25, 29, and 121
Flammability Requirements for Aircraft Seat Cushions; Final Rule

**DEPARTMENT OF TRANSPORTATION**

**14 CFR Parts 25, 29, and 121**

**[Docket No. 23791; Amdt. Nos. 25-59, 29-23, and 121-184]**

## Flammability Requirements for Aircraft Seat Cushions

**AGENCY:** Federal Aviation Administration (FAA), DOT.

**ACTION:** Final rule.

**SUMMARY:** These amendments establish new flammability requirements for seat cushions used in transport category aircraft certificated under Part 25 and Part 29 and require that the cushions in transport category airplanes type certificated after January 1, 1958, and operating under Part 121 comply with these new requirements after November 26, 1987. These new requirements are in addition to the present flammability requirements contained in the Federal Aviation Regulations and represent a significant advancement in aircraft fire safety.

**EFFECTIVE DATE:** November 26, 1984.

**FOR FURTHER INFORMATION CONTACT:** Henri Branting, Technical Analysis Branch (AWS-120), Aircraft Engineering Division, Office of Airworthiness, Federal Aviation Administration, 800 Independence Avenue, SW., Washington, D.C. 20591; telephone (202) 426-8382.

**SUPPLEMENTARY INFORMATION:**

### Background

On August 23, 1983, the FAA issued Notice of Proposed Rulemaking No. 83-14 (48 FR 46250; October 11, 1983). This notice proposed to establish additional flammability requirements for seat cushions used in transport category aircraft certificated under Part 25 and Part 29 of the Federal Aviation Regulations (FAR) and to require that the cushions in most transport category airplanes operating under Part 121 comply with these new requirements 3 years after the effective date of the amendments.

The notice responded to certain findings and a recommendation of the Special Aviation Fire and Explosion Reduction (SAFER) Advisory Committee and was based on research and development carried out by the Federal Aviation Administration (FAA) Technical Center and the Ames Research Center of the National Aeronautics and Space Administration.

The SAFER Advisory Committee was established in June 1978 by the FAA as a result of information from public hearings on aircraft fire safety. The FAA directed the Committee to "examine the factors affecting the ability of the aircraft cabin occupant to survive in the post-crash environment and the range of solutions available." The Committee consisted of 24 representatives of a wide range of aviation and general public interests. Technical support groups included approximately 150 of the world's top experts in fire research, accident investigation, materials development, and related fields. At the conclusion of its investigation into cabin materials technology, the Committee issued findings and formal recommendations pertaining to long-range research, design, testing, and the problems of smoke and toxic gas emission. One recommendation was that the fire blocking layer concept be developed for aircraft seat cushions as a means of retarding flame spread. The FAA concurred in this recommendation and carried out the research and development necessary for implementation of the concept.

As a result of regulatory amendments adopted in 1972, aircraft seat cushions are typically constructed of fire-retardant polyurethane foam and upholstery covering, all of which must presently pass the Bunsen burner test prescribed in § 25.853 of the FAR. In a prolonged full-scale cabin fire condition, however, severe thermal radiation can break down the outer upholstery covering and penetrate into the relatively large fuel mass of the polyurethane foam core. This causes the core to become involved in the fire, spreading flame and producing potentially lethal smoke, combustable gases, and toxic gases. The results of accident investigations and experimental fire tests conducted by the FAA have demonstrated that this involvement of foam cushion material is a dominant factor in the spread of cabin fire. To counter this, fire retardant performance standards for seat cushions based on the level of protection that can be achieved by the fire blocking layer concept were proposed in Notice 83-14.

The fire blocking layer concept involves the use of a thin layer of highly fire-resistant material to completely encapsulate and protect the larger mass of foam core seat cushion material from involvement in the cabin fire. This layer of fire-resistant material delays the onset of ignition and retards the involvement of the core in the fire.

The initial phase of the FAA research program for fire blocking layers consisted of a series of instrumented controlled environment cabin fire tests which confirmed the efficacy and practicality of fire blocking layers for aircraft seat cushions.

The subsequent phase of the program developed the test for evaluation and certification of cushions, using an adaptation of the type of 2 gallon/hour kerosene burner which is currently in standard use throughout industry as a test for metallic tubing assemblies and components. This test subjects the cushion test specimen to temperature and heat typical of full-scale cabin fire and is far more realistic and severe than the Bunsen burner test currently required in Part 25 for cushion materials.

Notice 83-14 proposed the detailed procedures of the kerosene burner test developed by the FAA. The proposed test would subject seat bottom and seat back cushion specimens to a 2-minute burner flame impingement. The proposed criteria for acceptance were based, in part, on the percentage weight loss of the cushion specimen during the test. While the proposal was based on the performance attained by fire blocking construction, the proposal would not require that seat cushions be constructed in that way. Rather, it proposed objective standards of performance for seat cushions so that if other or improved means of accomplishing the fire safety objective are developed, they can be used without a need for regulatory amendment. The notice proposed to incorporate the new cushion flammability requirements as additions to the type certification standards for both transport category airplanes and transport category rotorcraft since the flammability requirements for these two categories of aircraft are identical. The notice also proposed that 3 years from the effective date of the final regulation, seat cushions in airplanes type certificated after January 1, 1958, and operated under Part 121 meet the new requirements.

### Public Participation

These amendments are based on Notice 83-14. All interested parties have been given an opportunity to participate in the making of these amendments, and due consideration has been given to all matters presented. Except for the changes discussed below, these amendments and the reasons for their adoption are the same as those stated in Notice 83-14.

### Discussion of Comments

Forty-two comments were received in response to Notice 83-14, representing the views of aircraft and equipment manufacturers, aircraft operators, material producers and testing laboratories, aircraft crew organizations, U.S. and foreign

government organizations, and consumer interests. The comments strongly support the objective of reducing the fire potential of seat cushion materials.

Several commenters believe the new cushion requirements should set limits on smoke and toxic gas emission. One commenter suggests using the National Bureau of Standards (NBS) smoke density chamber for this.

The FAA recognizes that reduction in smoke and toxic gas emission is an important issue in fire safety. Notice 83–14 explains that the new cushions will greatly reduce emissions by virtue of their reduced heat and flame spread potential. This has been proven by full-scale cabin fire tests. However, addressing the emissions issue in quantitative terms and setting limits on emissions based on a defined test procedure are beyond the scope of Notice 83–14. The NBS chamber mentioned by one commenter is a small-scale laboratory test which is not suitable for testing large cushion assemblies.

Several commenters contend the requirements should not apply to relatively small transport category airplanes such as executive airplanes and airplanes seating less than 44 passengers. Several of these commenters contend the basis for the justification for the requirements is the 40 seconds which can be gained in usable evacuation time through use of improved cushions to delay fire spread. They say while this gain might apply to larger aircraft, it cannot be realized in the smaller aircraft which generally have short evacuation times. Other commenters recommend extending the requirements to airplanes certificated under FAR Part 23 and those operated under FAR Part 135.

The FAA does not agree that benefits of the new requirements will be realized only in larger aircraft. The new requirements will greatly improve the fire safety of those furnishings which make up a major part of the cabin by reducing the potential for ignition and occurrence of fire and by inhibiting flame spread and smoke and toxic gas emission in the event fire does occur. Ignition, flame spread, smoke, and toxic gases are all potential hazards in inflight fires as well as in those post-crash fires involving emergency evacuation. Although the potential gain in evacuation time is more pronounced in larger aircraft, the new requirements will significantly benefit smaller aircraft as well. Notice 83–14 explains that the FAA is considering the need to propose similar requirements for small airplanes and rotorcraft used in Part 135 operations. Regulatory action for this would be the subject of a separate notice if found to be appropriate.

Several commenters contend the requirements should not apply to flight crewmember seats and flight attendant seats. These commenters point out that seat comfort has a significant influence on flight crewmember performance and efficiency and that there is the possibility fire blocking layers could compromise comfort on flights of long duration. They point out that the risk of fire involvement of flight crewmember seats is low because the seats are isolated from passengers and fuel, located near a fire extinguisher, and occupied at all times by personnel trained in fire prevention and control. One commenter points out that cushions of a flight attendant seat usually are thin and that the added thickness and weight of a fire blocking layer might interfere with the seat-retract mechanism.

The FAA agrees with the commenters on the issue of flight crewmember seats. Since inservice evaluation of fire blocking materials has not been completed, and those materials with optimum comfort properties have not been identified, it would be premature at this time to require the retrofit of seats the comfort of which might affect performance of the flight crewmembers. Since flight attendants do not usually remain in their seats for the duration of the flight, flight attendant seats are not considered as critical as flight crewmember seats from the standpoint of comfort and are not excluded from the requirements. There are several commercially available fire blocking materials which are thin and lightweight. These should have no effect on seat-retract mechanisms. The rule, as adopted, excludes flight crewmember seats from the requirements but does not exclude flight attendant seats.

Several commenters contend the 3-year compliance period proposed in § 121.312(b) should be extended to allow operators sufficient time to handle technical and logistical problems and to account for longer cushion life spans which they say exceed 3 years in many cases. The commenters contend the fire blocking requirements involve essentially a new technology and untested materials and that the proposed 3-year period does not allow sufficient time for cushion development, inservice testing, certification, production, and installation. They contend the added cost of an accelerated 3-year compliance period would be significant.

The FAA does not agree the compliance period should be extended. The FAA closely monitors industry progress and, while recognizing the concerns of the commenters, has not found any foreseeable technical problem to suggest that retrofit cannot be accomplished smoothly within 3 years. Although the 3-year period was taken as the life span of a typical cushion, as explained in Notice 83–14, the longer life spans of some cushions mentioned by commenters would have no adverse impact on the regulatory action since the addition of fire blocking layers does not necessarily result in discarding cushions.

Several commenters contend the 3-year compliance period proposed in § 121.312(b) is too long and that fleet retrofit should be completed in a much shorter time. They contend the safety benefits of a shorter compliance time would exceed costs and that this justifies the faster retrofit. Several commenters recommend that all newly manufactured airplanes comply with the requirements within 1 year.

The FAA generally recognizes that benefits from safety improvements are maximized the sooner required retrofits are completed. However, as pointed out by several commenters, the subject regulatory action involves a new technology, and there must be sufficient lead time in the compliance period to enable all parties affected to attain reasonable proficiency, develop design alternatives, produce finished articles, and phase in installations. Fire blocking technology entails new test equipment and criteria and advanced state-of-the-art materials, many of which have not been service tested. The FAA believes a substantial reduction in the compliance period recommended by commenters would be impractical. The recommendation that newly manufactured airplanes comply within 1 year will effectively be achieved since, as a matter of practice, seat and aircraft manufacturers would meet the operational rules which govern their market. It is highly unlikely that manufacturers would produce noncomplying seat cushions after 1 year has passed, knowing the cushions would require retrofit in less than 2 years. It is equally unlikely that older aircraft being refurbished would be refurbished with noncomplying seat cushions, knowing that they would need to be replaced before the end of their normal useful life. These commercial considerations will cause manufacturers and operators who are refurbishing older aircraft to introduce seat cushions with fire blocking layers (or other equivalent means of fire protection) soon after the effective date of this rule. The 3-year

compliance period is adopted as proposed.

Several commenters express concern that the addition of a fire blocking layer to a seat cushion approved under Technical Standard Order (TSO) C72b for flotation devices on TSO–C39a for seats might constitute a major modification of the cushion which could invalidate the TSO approvals.

The FAA has conducted cyclical flotation tests of several fire blocked cushions to determine the effect fire blocking layers might have on the buoyancy of cushions. The typical lightweight, highly fire-resistant materials being used as a fire blocker should have negligible effect on buoyancy. The use of heavy blocking material might reduce buoyancy to the extent which could require requalification under TSO–C72b. Provided the layer does not significantly reduce buoyancy or interfere with grasp straps, markings, or other flotation device features and the cushion foam core is not altered, the addition of fire blocking material is considered a minor modification and does not affect approval under TSO–C72b. Since the fire blocking layer requirements are additional to the requirements of § 25.853 and are in no way expected to affect seat cushions' eligibility to meet the standards of TSO–C39a and be so marked, approval under TSO–C39a is not affected.

Several commenters contend cushions which meet the new flammability requirements should not be required to meet § 25.853(b) as this would be redundant. Commenters contend also that if fire blocking layer material is required to meet § 25.853(b), it should be tested separately and not as part of a cushion assembly.

The FAA believes the new flammability requirements based on fire blocking performance and the requirements of § 25.853(b) are both necessary. Notice 83–14 explains that fire blocking delays, but does not prevent, ignition of cushion foam material and its involvement in cabin fire. The fire resistance required by § 25.853(b) is necessary in the event fire does penetrate the cushion. Under § 25.853(b), fire blocking material would be considered as upholstery in general and would be tested separately if it is not bonded or permanently affixed to the cushion foam. In view of the sound experience which backs up § 25.853(b), highly fire-resistant fire blocking materials should have no difficulty qualifying, whether tested separately or as part of a cushion assembly.

Several commenters contend the proposed requirements of § 25.853(c) and Appendix F, as written, are inflexible and would require an unnecessary amount of testing with the full-scale oil burner apparatus. Commenters point out there are numerous variations in color, weight, blend, texture, and other properties of cushion dress covering which have a negligible effect on fire safety. The commenters contend that once a cushion assembly is qualified by the oil burner test, minor changes in dress covering should be allowed without requalification by full-scale testing.

The FAA agrees with the commenters that once a cushion is qualified by full-scale oil burner tests, additional tests are not necessary for minor changes in dress covering provided the replacement covering is similar to the original covering in fire resistance. The FAA recognizes that as experience is gained in the testing of various fire blocking materials and material combinations, the purposes served by full-scale testing and the situations which warrant it will become clearly focused. Therefore, paragraph (a)(3) of Part II of Appendix F is revised to allow that for a cushion which has been qualified by the oil burner test, the dress covering of that cushion may be replaced with a similar dress covering if the burn length of the replacement covering, as determined by the test specified in § 25.853(b), does not exceed the burn length of the original covering.

Several commenters contend the oil burner test is impractical for aircraft certification and that there should be provisions for testing small-scale laboratory specimens with smaller equipment such as the Meker gas burner, the Ohio State University Heat Release Chamber, or a radiant panel type test. Several commenters are concerned that the oil burner test is not suitable for quality control testing.

The FAA does not agree the oil burner test is impractical or should be replaced by some other test. It is intended as a design qualification test to substantiate the performance of an assembly product. The test subjects specimens to temperature and heat flux typical of cabin fire, as determined by full-scale cabin fire tests. For seat cushions, as for other aircraft components and assemblies, the required quality level of constituent materials is assured by use of small-scale tests or other assay methods selected by the manufacturer for the particular materials in question. The FAA does believe that eventually other tests may be developed which could be used for the qualification of cushions. While the commenters do not substantiate the validity or equivalency of another test at this time, the FAA believes this option should be left open to encourage future developments. Accordingly, § 25.853(c) and § 29.853(b) are specifically revised to allow a finding of equivalency.

Several commenters contend the ten percent weight loss limit is not a realistic measure of a cushion's resistance to fire and is not an appropriate criterion for acceptance. The commenters suggest using an absolute weight loss of around one-half pound per specimen. One commenter suggests using a rate of weight loss, although no specific rate is suggested. Several commenters contend that under the 10 percent criterion, an adequate supply of fire blocking materials will not be available to meet airline needs.

The FAA believes the 10 percent weight loss criterion is appropriate. The FAA has tested over 300 candidate fire blocking materials, of which over 100 passed the 10 percent criterion. The use of absolute weight loss in lieu of percent weight loss as the criterion for these materials had an insignificant effect on the overall pass/fail results. Percent weight loss normalizes test results according to specimen weight and affords a safeguard against the use of materials which might have a lower resistance to fire in combination with a lower weight. There is no indication a rate of weight loss as suggested by one commenter is more appropriate than percent weight loss. Rate of weight loss alone in this case would not provide a relevant indication of fire resistance unless related to time. The 10 percent criterion relates to test duration which, as adopted, does not exceed 7 minutes. In view of the FAA materials tests and industry's progress in implementing the fire blocking concept, the FAA believes there is an adequate supply of materials to meet airline needs.

Several commenters contend the dimensionally standard specimens specified in Appendix F are not a realistic representation of cushions with complex curvatures and unique shapes. The commenters recommend testing actual cushions.

The FAA believes only dimensionally standard specimens should be used in the subject test to ensure a consistent baseline for comparison of cushion fire blocking performance. The test measures the effectiveness of material, or materials in combination, in delaying involvement of cushion foam in fire. For this, standard specimens of the materials are needed. The FAA evaluated the testing of nonstandard cushion shapes and found this can produce results unsuitable for the comparison of materials.

One commenter contends the requirements do not make clear if the seat bottom and seat back cushions must be constructed of identical fire blocking materials or may have different materials and different levels of fire blocking performance. This is a critical consideration since the test is more severe to the seat bottom specimen than the back specimen.

The requirements do not intend that materials in the back cushion necessarily be the same as those in the bottom cushion since material selection might be governed by comfort, durability, and other factors pertinent to the particular cushion. However, the requirements do intend that the materials in both the bottom and the back cushions be able to satisfactorily withstand the flame impingement of the test burner since in an actual cabin fire, flame impingement might be equally severe to both cushions. To clarify this intent, paragraph (a)(3) of Part II of Appendix F is revised to require that if different material configurations are used in the bottom and back cushions, each configuration must be tested as a complete specimen set.

Several commenters point out that the back sides of many seat back cushions are bonded to metal which effectively provides blocking layer protection. The commenters question whether in such cases the back side of the cushion must be enclosed by the same fire blocking material used to enclose the other sides.

The rule does not require the same blocking layer material be used to enclose all sides of a cushion, nor does it preclude the use of metal blocking layers. As adopted, it requires that the cushion meet the prescribed test requirements or equivalent. Seat structure in combination with some other material would be an acceptable combination of fire blocking materials, provided adequate performance of the combination is substantiated.

Numerous comments were submitted regarding the details of the proposed new test criteria of Appendix F. As a result, there are many revisions in the criteria, most of which are simple refinements to increase test repeatability. The most significant revisions are in section (a), Criteria for Acceptance, and these have only a minor effect on the performance level required of cushion specimens. Paragraph (a)(2) is revised to delete the requirement for venting internal cushion pressure. This requirement is not necessary since aircraft cushions inherently are self-venting by construction to accommodate cabin altitude changes. Paragraph (a)(4) is clarified by changing the term "flame spread" to the term "burn length," as currently used in Appendix F and by specification of a maximum permissible burn length based on specimen width. Also, paragraph (a)(4) is clarified regarding the number of specimens which must pass the test. Notice 83–14 proposed that one-half of the required three specimens, or two, pass. The rule as adopted specifies two out of three. Paragraph (a)(5) is revised to clarify the procedure for determining specimen weight after the test and to ensure that wide fluctuations in test results of marginal specimens do not unduly influence the pass/fail outcome of combined test results. The proposed requirement that there be no flaming accumulation of melted material beneath the test specimen is deleted. This was found to be impractical.

Flaming material accumulation is as much a function of the test apparatus as of specimen material properties.

Numerous clarifications are made in sections (b) through (h), all of which have a negligible effect on test requirements. The method for determining ventilation rate of the test area is clarified. Tolerances for length, weight, temperature, and heat flux are specified, and additional descriptive information on equipment is provided. A requirement for conditioning the specimen at 55 percent relative humidity is specified. The type of fuel used for the test is specified as #2 Grade kerosene or equivalent. The time and means are specified for terminating the test for those specimens which do not self-extinguish.

**Regulatory Evaluation**

This amendment is expected to provide a net benefit to society, as likely benefits are expected to exceed likely costs. This evaluation relies heavily on information developed in a study done by the National Bureau of Standards (NBS), Center for Fire Research entitled *Decision Analysis Model for Passenger—Aircraft Fire Safety With Application to Fire—Blocking of Seats,* published in March 1984. A copy of this study is available in the docket of this rulemaking action.

The NBS study reviewed an accident data base which included all world aircraft accidents where fire was a factor in fatalities, as well as major aircraft hull property damage incidents where a fire blocking seat interior might have lessened or eliminated property loss. The NBS study report lists all of these accidents, as well as the rationale for estimating the effectiveness of fire blocking layers in saving lives and lessening property damage.

The benefit effectiveness of fire blocking layers is basically a function of the increased time that is made available for aircraft evacuation, as a result of fire-blocking layers. This time is varied, ranging between 20 seconds and 60 seconds, in the NBS study. Table 1 below summarizes three basic values for fire-blocking benefits, based on assumptions of increased evacuation time and different levels of property damage. The only adjustment to the NBS study data is the use of a value of life of $650,000 compared to the $500,000 value in the NBS study. The higher number is used in FAA evaluations.

FIRE BLOCKING SEAT ALTERNATIVES
ANNUALIZED BENEFIT SUMMARY

[Values in millions of 1983 dollars]

| Additional evacuation time (seconds) | High | Middle | Low |
|---|---|---|---|
| 20 | 16.9 lives | 10.8 lives | 4.4 lives. |
| | $3.87 damage | $2.21 damage | $1.76 damage. |
| | $14.85 total | $9.23 total | $4.60 total. |
| 43 | 20.1 lives | 13.6 lives | 4.7 lives. |
| | $3.87 damage | $2.21 damage | $1.76 damage. |
| | $16.92 total | $11.05 total | $4.81 total. |
| 60 | 22.3 lives | 13.6 lives | 4.7 lives. |
| | $3.87 damage | $2.21 damage | $1.76 damage. |
| | $18.37 total | $11.05 total | $4.81 total. |

NOTE.—Lives saved are valued at $650,000 per life.
Source: NBS study p. 28 (except as per note).

For purposes of this evaluation, we will concentrate on the middle and high benefit range and limit analysis to the 20- and 43-second added evacuation time summaries. In this approach, we eliminate the extremes of very long evacuation times and very low benefit rates.

The NBS study estimated the costs of fire blocking seat alternatives much as the NASA study cited in FAA's preliminary regulatory evaluation did. The important elements of incremental cost are the incremental costs of refurbishing seats with seat blocking materials and the operating cost of carrying added weight around in the aircraft.

The following table summarized the high, middle, and low cost estimates of the incremental cost of material and installation for three blocking alternatives. The first is Norfab, a weave of 25 percent Nomex, 70 percent Kevlar, and 5 percent Kynol, aluminized on one side. The second is a loosely woven fiberglass scrim and a lightweight fiberglass paper bonded with a fire retardant adhesive. The third is 3/16" Neoprene foam, bonded to urethane. The manufacturing costs are based on estimates provided by two seat manufacturers.

INCREMENTAL COST OF FIRE BLOCKING LAYERS FOR U.S. FLEET (MATERIALS, INSTALLATION AND OPERATING COSTS)

[Data are in millions of 1983 dollars]

| Fire block alternative | High | Middle | Low |
|---|---|---|---|
| Norfab | | | |
| Materials and installation | $16.56 | $11.83 | $9.75 |
| Operating cost | 9.93 | 9.93 | 9.93 |
| Total cost | 26.49 | 21.76 | 19.68 |
| Fiberglass | | | |
| Materials and installation | 11.17 | 6.68 | 4.81 |
| Operating cost | 2.92 | 2.92 | 2.92 |
| Total cost | 14.09 | 9.60 | 7.73 |
| Neoprene | | | |
| Materials and installation | 16.95 | 9.29 | 5.40 |
| Operating cost | 19.49 | 19.49 | 19.49 |
| Total cost | 36.44 | 28.78 | 24.89 |

Source: Tables G-10 and 6 of NBS study.

The results of the NBS study indicate that there are fire blocking alternatives for which likely benefits clearly exceed likely costs. The fiberglass fabric alternative has a benefit/cost ratio greater than one except in those instances where comparisons use low benefits or high costs and middle benefit. Comparing middle costs with middle benefits, the benefit/cost ratio is 1.15.

There is some uncertainty about the predicted ultimate costs and benefits of the fire blocking rule which is adopted by this amendment. The major questions result from the uncertainties as to which technically feasible solutions will be practical. Several different solutions are being tried by industry, each of which appears promising. The optimum solutions will be known only after having fire blocking alternatives put into widescale utilization and testing with the airlines. On balance, however, FAA believes that this evaluation and the NBS study show that the amendment will create a net benefit to society.

It is expected that the airline supplies and materials industries will work with the airlines to develop a relatively inexpensive, lightweight fire blocking material. Even if practical problems are encountered with a fiberglass material, these problems will likely be solved, or alternatives will likely be developed with have weight and expense factors similar to fiberglass fabrics.

*Regulatory Flexibility Act Determination*

A final Regulatory Flexibility Analysis was conducted in compliance with section 604(a) of the Regulatory Flexibility Act. The conclusion in the initial regulatory evaluation, that the rule may cause a significant economic impact on a substantial number of small entities, is not altered by the present evaluation.

There were no public comments in response to the initial regulatory flexibility analysis, and there are no alternatives which lessen the impact on small entities while providing all members of the traveling public with an equal level of protection.

*Paperwork Reduction Act*

Information collection requirements in this regulation (Part 25, Appendix F) have been approved by the Office of Management and Budget under the provisions of the Paperwork Reduction Act of 1980 (Pub. L. 96-511) and have been assigned OMB Control Number 2120-0018.

*Conclusion*

Under the terms of the Regulatory Flexibility Act (the Act), the FAA has reviewed this amendment to determine the impact it might have on small entities.

Since the estimated impact on the small unscheduled air carriers could be approximately $9,000 per year, it has been determined that this rule may have a significant economic impact on a substantial number of small entities, such as small air carriers operating under Part 121. As required by the Act, the FAA has completed a regulatory flexibility analysis as part of the regulatory evaluation. A copy of the analysis/evaluation is contained in the regulatory docket. A copy of it may be obtained by contacting the person identified under the caption **"FOR FURTHER INFORMATION CONTACT."**

The Act also requires that when there is a significant impact on small entities the agency must consider alternatives in the rulemaking process. In the case of flammability requirements, the alternatives are limited in number. One alternative would be to lessen the impact on small entities by making the more stringent requirements apply only to the larger air carriers or by allowing the smaller entities a longer period to come into compliance. These alternatives were rejected because of the importance of passenger safety, whether traveling on a large, scheduled airline or on a smaller, unscheduled airline. As alternative approaches, the FAA considered both regulations that would specify the only materials and construction processes permitted to be used and regulations that set performance standards to be met. The FAA has proposed performance standards to permit those operating under Part 121 the opportunity to choose and install the most economical materials and processes capable of meeting the flammability performance standards.

This rule is not likely to result in an annual effect on the economy of $100 million or more, or a major increase in costs for consumers, industry, or Federal, State, or local government agencies. In addition, this rule would have little or no impact on trade opportunities for United States firms doing business overseas or for foreign firms doing business in the United States. Accordingly, it has been determined that this is not a major regulation under Executive Order 12291. In addition, the FAA has determined that this action is significant under Department of Transportation Regulatory Policy and Procedures (44 FR 11034; February 26, 1979).

**List of Subjects**

*14 CFR Part 25*

Air transportation, Aircraft, Aviation safety, Safety, Tires.

*14 CFR Part 29*

Air transportation, Aircraft, Aviation safety, Safety, Tires, Rotorcraft.

*14 CFR Part 121*

Aviation safety, Safety, Air carriers, Air transportation, Aircraft, Airplanes, Airworthiness directives and standards, Flammable materials, Transportation, Common carriers.

**Adoption of the Amendment**

Accordingly, Parts 25, 29, and 121 of the Federal Aviation Regulations (14 CFR Parts 25, 29, and 121) are amended as follows, effective November 26, 1984:

## PART 25—AIRWORTHINESS STANDARDS: TRANSPORT CATEGORY AIRPLANES

1. By amending § 25.853 by redesignating present paragraphs (c) through (e) as paragraphs (d) through (f) and adding a new paragraph (c) as follows:

**§ 25.853 Compartment interiors.**

* * * * *

(c) In addition to meeting the requirements of paragraph (b), seat cushions, except those on flight crewmember seats, must meet the test requirements of Part II of Appendix F of this part, or equivalent.

* * * * *

2. By amending Appendix F to Part 25 by removing the introductory sentence and by designating the text of Appendix F to Part 25 as Part I as follows:

Appendix F

*Part I—An Acceptable Test Procedure for Showing Compliance With §§ 25.853, 25.855, and 25.1359*

* * * * *

3. By amending Appendix F to Part 25 by adding a new Part II to read as follows:

* * * * *

*Part II—Flammability of Seat Cushions*

(a) *Criteria for Acceptance.* Each seat cushion must meet the following criteria:

(1) At least three sets of seat bottom and seat back cushion specimens must be tested.

(2) If the cushion is constructed with a fire blocking material, the fire blocking material must completely enclose the cushion foam core material.

(3) Each specimen tested must be fabricated using the principal components (i.e., foam core, flotation material, fire blocking material, if used, and dress covering) and assembly processes (representative seams and closures) intended for use in the production articles. If a different material combination is used for the back cushion than for the bottom cushion, both material combinations must be tested as complete specimen sets, each set consisting of a back cushion specimen and a bottom cushion specimen. If a cushion, including outer dress covering, is demonstrated to meet the requirements of this appendix using the oil burner test, the dress covering of that cushion may be replaced with a similar dress covering provided the burn length of the replacement covering, as determined by the test specified in § 25.853(b), does not exceed the corresponding burn length of the dress covering used on the cushion subjected to the oil burner test.

(4) For at least two-thirds of the total number of specimen sets tested, the burn length from the burner must not reach the side of the cushion opposite the burner. The burn length must not exceed 17 inches. Burn length is the perpendicular distance from the inside edge of the seat frame closest to the burner to the farthest evidence of damage to the test specimen due to flame impingement, including areas of partial or complete consumption, charring, or embrittlement, but not including areas sooted, stained, warped, or discolored, or areas where material has shrunk or melted away from the heat source.

(5) The average percentage weight loss must not exceed 10 percent. Also, at least two-thirds of the total number of specimen sets tested must not exceed 10 percent weight loss. All droppings falling from the cushions and mounting stand are to be discarded before the after-test weight is determined. The percentage weight loss for a specimen set is the weight of the specimen set before testing less the weight of the specimen set after testing expressed as the percentage of the weight before testing.

(b) *Test Conditions.* Vertical air velocity should average 25 fpm±10 fpm at the top of the back seat cushion. Horizontal air velocity should be below 10 fpm just above the bottom seat cushion. Air velocities should be measured with the ventilation hood operating and the burner motor off.

(c) *Test Specimens.* (1) For each test, one set of cushion specimens representing a seat bottom and seat back cushion must be used.

(2) The seat bottom cushion specimen must be 18± ⅛ inches (457±3 mm) wide by 20± ⅛ inches (508±3 mm) deep by 4± ⅛ inches (102±3 mm) thick, exclusive of fabric closures and seam overlap.

(3) The seat back cushion specimen must be 18± ⅛ inches (432±3 mm) wide by 25± ⅛ inches (635±3 mm) high by 2± ⅛ inches (51±3 mm) thick, exclusive of fabric closures and seam overlap.

(4) The specimens must be conditioned at 70±5 °F (21±2 °C) 55%±10% relative humidity for at least 24 hours before testing.

(d) *Test Apparatus.* The arrangement of the test apparatus is shown in Figures 1 through 5 and must include the components described in this section. Minor details of the apparatus may vary, depending on the model burner used.

(1) *Specimen Mounting Stand.* The mounting stand for the test specimens consists of steel angles, as shown in Figure 1. The length of the mounting stand legs is 12± ⅛ inches (305±3 mm). The mounting stand must be used for mounting the test specimen seat bottom and seat back, as shown in Figure 2. The mounting stand should also include a suitable drip pan lined with aluminum foil, dull side up.

(2) *Test Burner.* The burner to be used in testing must—

(i) Be a modified gun type;

(ii) Have an 80-degree spray angle nozzle nominally rated for 2.25 gallons/hour at 100 psi;

(iii) Have a 12-inch (305 mm) burner cone installed at the end of the draft tube, with an opening 6 inches (152 mm) high and 11 inches (280 mm) wide, as shown in Figure 3; and

(iv) Have a burner fuel pressure regulator that is adjusted to deliver a nominal 2.0 gallon/hour of # 2 Grade kerosene or equivalent required for the test.

Burner models which have been used successfully in testing are the Lennox Model OB–32, Carlin Model 200 CRD, and Park Model DPL 3400. FAA published reports pertinent to this type of burner are: (1) Powerplant Enginering Report No. 3A, Standard Fire Test Apparatus and Procedure for Flexible Hose Assemblies, dated March 1978; and (2) Report No. DOT/FAA/RD/76/213, Reevaluation of Burner Characteristics for Fire Resistance Tests, dated January 1977.

(3) *Calorimeter.*

(i) The calorimeter to be used in testing must be a (0–15.0 BTU/ft²-sec. 0–17.0 w/cm²) calorimeter, accurate ±3%, mounted in a 6-inch by 12-inch (152 by 305 mm) by ¾-inch (19 mm) thick calcium silicate insulating board which is attached to a steel angle bracket for placement in the test stand during burner calibration, as shown in Figure 4.

(ii) Because crumbling of the insulating board with service can result in misalignment of the calorimeter, the calorimeter must be monitored and the mounting shimmed, as necessary, to ensure that the calorimeter face is flush with the exposed plane of the insulating board in a plane parallel to the exit of the test burner cone.

(4) *Thermocouples.* The seven thermocouples to be used for testing must be 1/16- to ⅛-inch metal sheathed, ceramic packed, type K, grounded thermocouples with a nominal 22 to 30 American wire gage (AWG)-size conductor. The seven thermocouples must be attached to a steel angle bracket to form a thermocouple rake for placement in the test stand during burner calibration, as shown in Figure 5.

(5) *Apparatus Arrangement.* The test burner must be mounted on a suitable stand to position the exit of the burner cone a distance of 4± ⅛ inches (102±3 mm) from one side of the specimen mounting stand. The burner stand should have the capability of allowing the burner to be swung away from the specimen mounting stand during warmup periods.

(6) *Data Recording.* A recording potentiometer or other suitable calibrated instrument with an appropriate range must be used to measure and record the outputs of the calorimeter and the thermocouples.

(7) *Weight Scale.* Weighing Device—A device must be used that with proper procedures may determine the before and after test weights of each set of seat cushion specimens within 0.02 pound (9 grams). A continuous weighing system is preferred.

(8) *Timing Device.* A stopwatch or other device (calibrated to ±1 second) must be used to measure the time of application of the burner flame and self-extinguishing time or test duration.

(e) *Preparation of Apparatus.* Before calibration, all equipment must be turned on and the burner fuel must be adjusted as specified in paragraph (d)(2).

(f) *Calibration.* To ensure the proper thermal output of the burner, the following test must be made:

(1) Place the calorimeter on the test stand as shown in Figure 4 at a distance of 4± ⅛ inches (102±3 mm) from the exit of the burner cone.

(2) Turn on the burner, allow it to run for 2 minutes for warmup, and adjust the burner air intake damper to produce a reading of 10.5±0.5 BTU/ft²-sec. (11.9±0.6 w/cm²) on the calorimeter to ensure steady state conditions have been achieved. Turn off the burner.

(3) Replace the calorimeter with the thermocouple rake (Figure 5).

(4) Turn on the burner and ensure that the thermocouples are reading 1900±100 °F (1038±38 °C) to ensure steady state conditions have been achieved.

(5) If the calorimeter and thermocouples do not read within range, repeat steps in paragraphs 1 through 4 and adjust the burner air intake damper until the proper readings are obtained. The thermocouple rake and the calorimeter should be used frequently to maintain and record calibrated test parameters. Until the specific apparatus has demonstrated consistency, each test should be calibrated. After consistency has been confirmed, several tests may be conducted with the pre-test calibration before and a calibration check after the series.

(g) *Test Procedure.* The flammability of each set of specimens must be tested as follows:

(1) Record the weight of each set of seat bottom and seat back cushion specimens to be tested to the nearest 0.02 pound (9 grams).

(2) Mount the seat bottom and seat back cushion test specimens on the test stand as shown in Figure 2, securing the seat back cushion specimen to the test stand at the top.

(3) Swing the burner into position and ensure that the distance from the exit of the burner cone to the side of the seat bottom cushion specimen is $4 \pm \frac{1}{8}$ inches ($102 \pm 3$ mm).

(4) Swing the burner away from the test position. Turn on the burner and allow it to run for 2 minutes to provide adequate warmup of the burner cone and flame stabilization.

(5) To begin the test, swing the burner into the test position and simultaneously start the timing device.

(6) Expose the seat bottom cushion specimen to the burner flame for 2 minutes and then turn off the burner. Immediately swing the burner away from the test position. Terminate test 7 minutes after initiating cushion exposure to the flame by use of a gaseous extinguishing agent (i.e., Halon or $CO_2$).

(7) Determine the weight of the remains of the seat cushion specimen set left on the mounting stand to the nearest 0.02 pound (9 grams) excluding all droppings.

(h) *Test Report.* With respect to all specimen sets tested for a particular seat cushion for which testing of compliance is performed, the following information must be recorded:

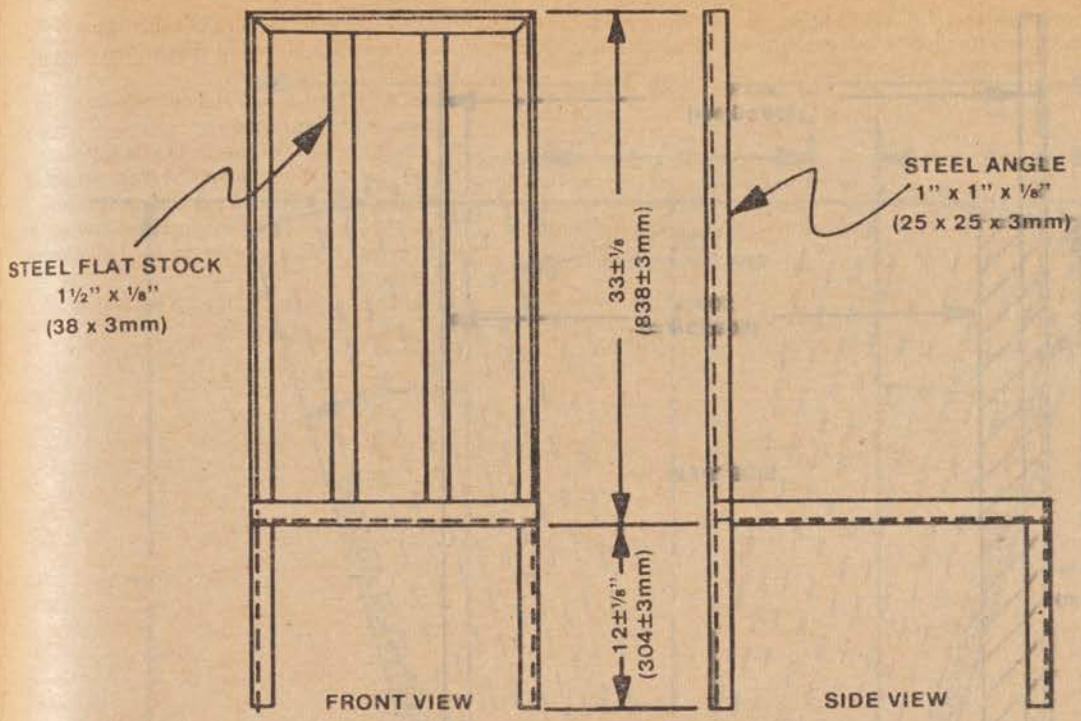
(1) An identification and description of the specimens being tested.

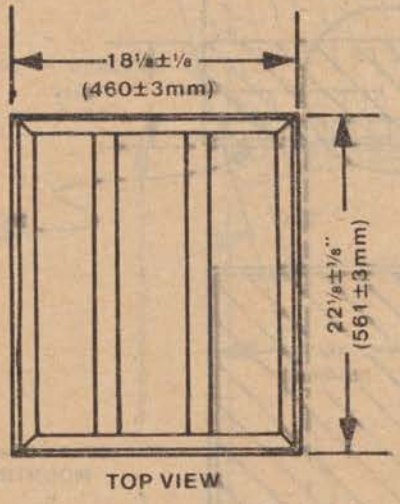
(2) The number of specimen sets tested.

(3) The initial weight and residual weight of each set, the calculated percentage weight loss of each set, and the calculated average percentage weight loss for the total number of sets tested.

(4) The burn length for each set tested.

BILLING CODE 4910-13-M




NOTE:

ALL JOINTS WELDED
FLAT STOCK BUTT WELDED
ALL MEASUREMENTS INSIDE

FIGURE 1

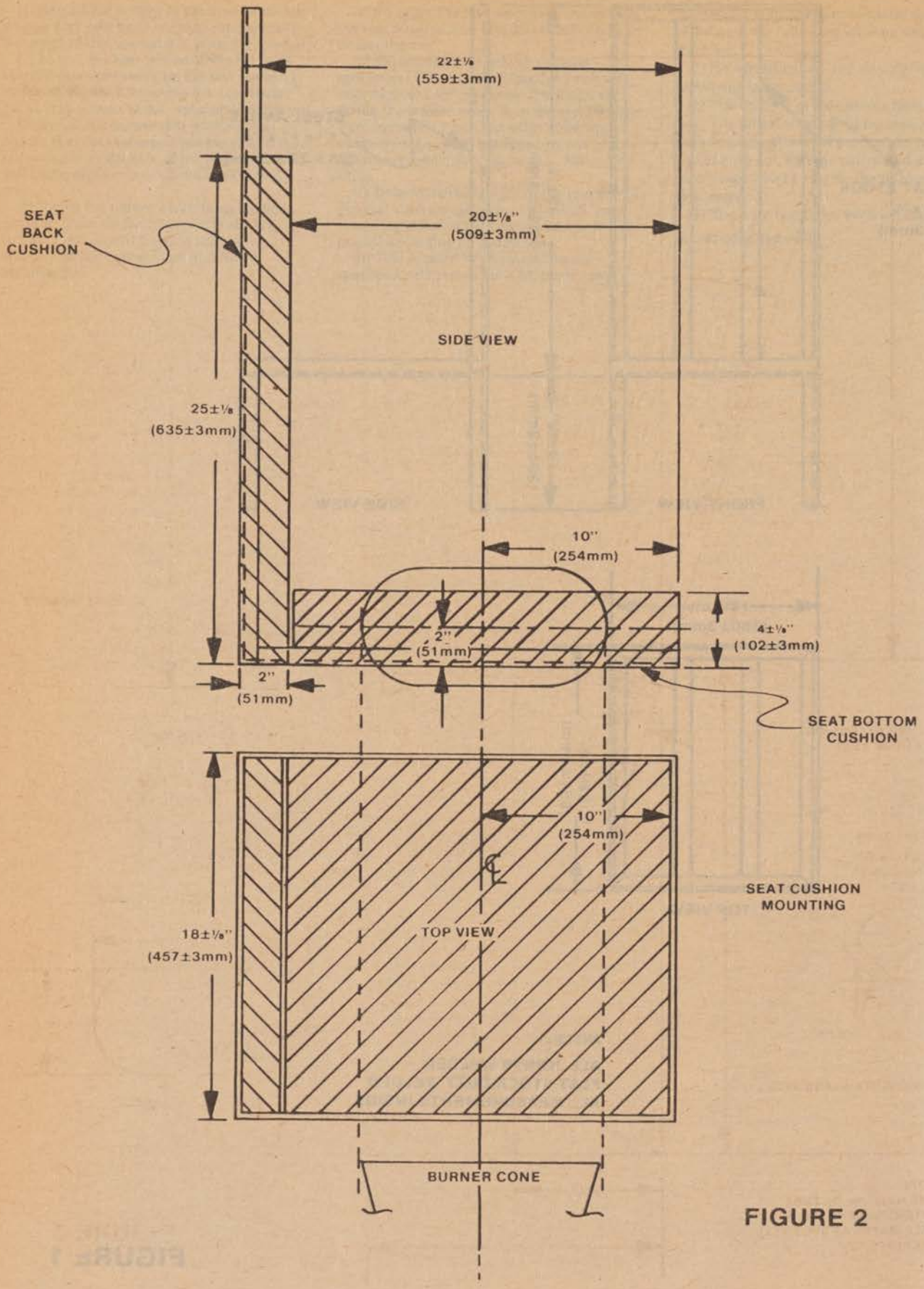

22±⅛
(559±3mm)
SEAT
BACK
CUSHION
20±⅛"
(509±3mm)
SIDE VIEW
25±⅛
(635±3mm)
10"
(254mm)
2"
(51mm)
4±⅛"
(102±3mm)
2"
(51mm)
SEAT BOTTOM
CUSHION
10"
(254mm)
SEAT CUSHION
MOUNTING
18±⅛"
(457±3mm)
TOP VIEW
BURNER CONE

FIGURE 2

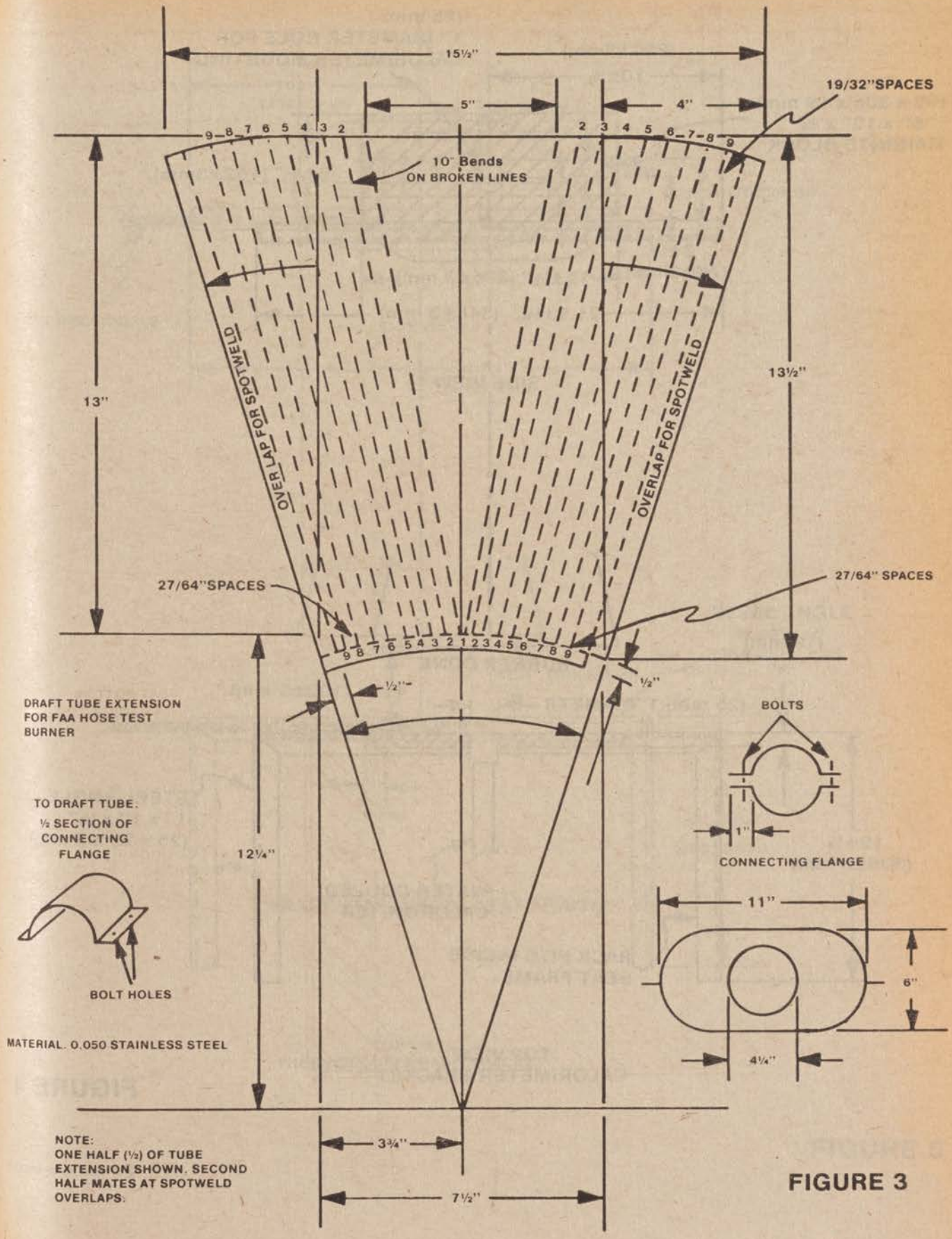

15½"
5"
4"
19/32"SPACES
9 8 7 6 5 4 3 2
2 3 4 5 6 7 8 9
10° Bends
ON BROKEN LINES
OVER LAP FOR SPOTWELD
OVERLAP FOR SPOTWELD
13"
13½"
27/64"SPACES
27/64" SPACES
9 8 7 6 5 4 3 2 1 2 3 4 5 6 7 8 9
½"
½"
DRAFT TUBE EXTENSION
FOR FAA HOSE TEST
BURNER
TO DRAFT TUBE.
½ SECTION OF
CONNECTING
FLANGE
12¼"
BOLTS
1"
CONNECTING FLANGE
11"
6"
4¼"
BOLT HOLES
MATERIAL. 0.050 STAINLESS STEEL
NOTE:
ONE HALF (½) OF TUBE
EXTENSION SHOWN. SECOND
HALF MATES AT SPOTWELD
OVERLAPS.
3¾"
7½"

FIGURE 3

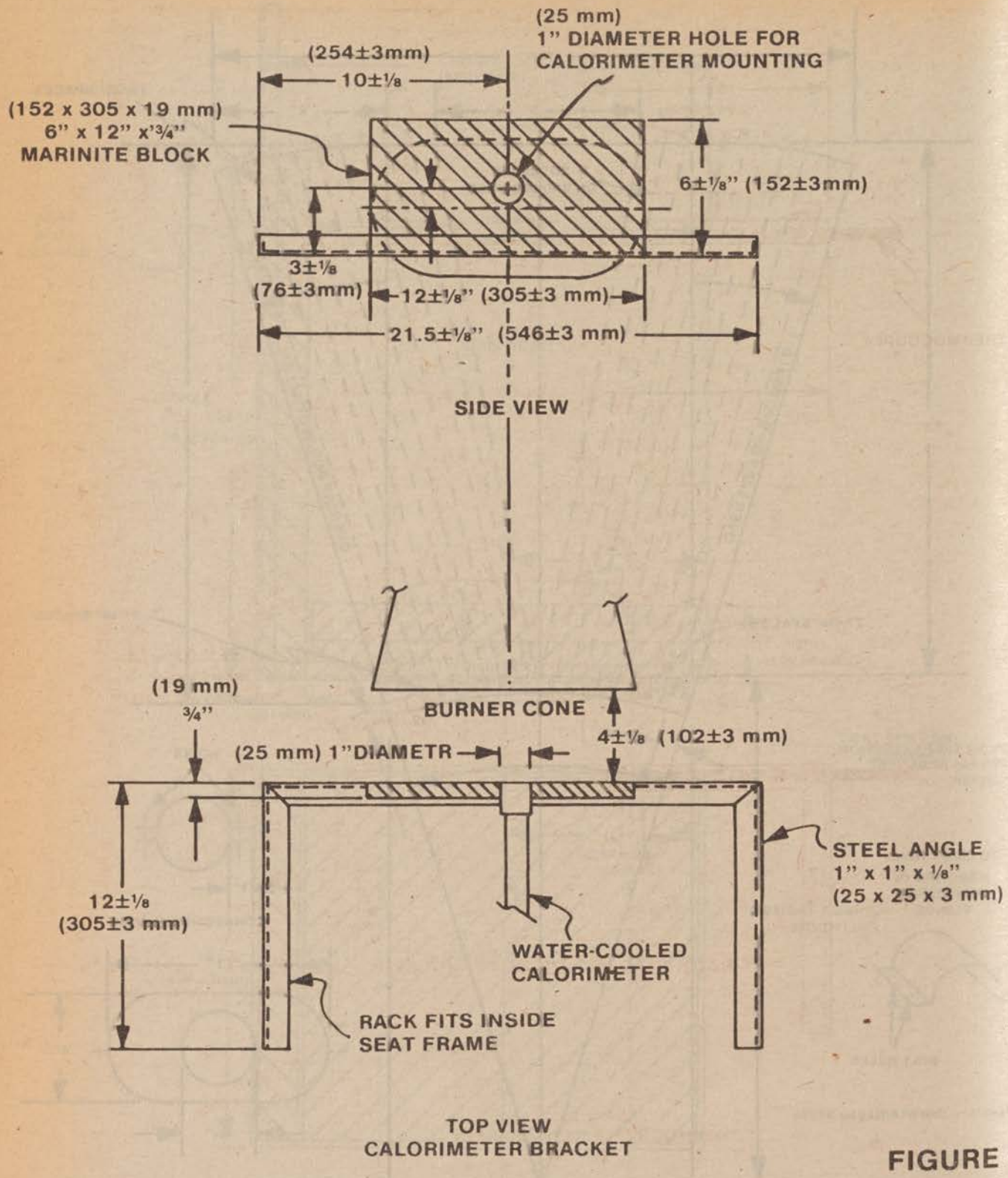
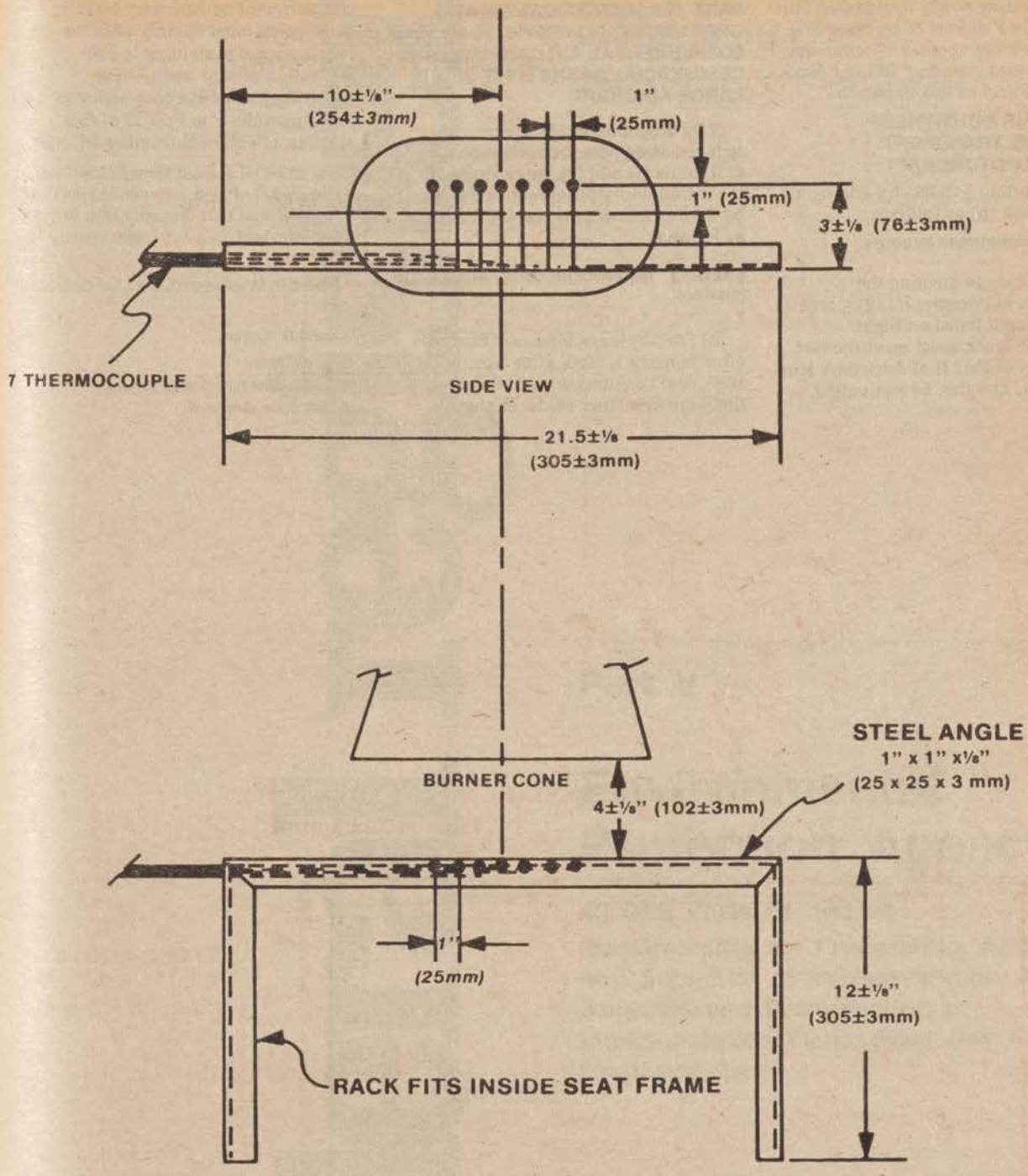

(25 mm)
1" DIAMETER HOLE FOR
CALORIMETER MOUNTING
(254±3mm)
10±⅛
(152 x 305 x 19 mm)
6" x 12" x ¾"
MARINITE BLOCK
6±⅛" (152±3mm)
3±⅛
(76±3mm)
12±⅛" (305±3 mm)
21.5±⅛" (546±3 mm)
SIDE VIEW
BURNER CONE
(19 mm)
¾"
(25 mm) 1"DIAMETR
4±⅛ (102±3 mm)
12±⅛
(305±3 mm)
STEEL ANGLE
1" x 1" x ⅛"
(25 x 25 x 3 mm)
WATER-COOLED
CALORIMETER
RACK FITS INSIDE
SEAT FRAME
TOP VIEW
CALORIMETER BRACKET

FIGURE 4




TOP VIEW
THERMOCOUPLE RAKE BRACKET

FIGURE 5

BILLING CODE 4910-13-C

4. By amending newly designated Part I of Appendix F of Part 25 by removing the words "of this appendix" wherever they appear and inserting, in their place, the words "Part I of this appendix".

## PART 29—AIRWORTHINESS STANDARDS: TRANSPORT CATEGORY ROTORCRAFT

5. By amending § 29.853 by adding a new paragraph (b) as follows:

**§ 29.853 Compartment interiors.**

* * * * *

(b) In addition to meeting the requirements of paragraph (a)(2), seat cushions, except those on flight crewmember seats, must meet the test requirements of Part II of Appendix F of Part 25 of this chapter, or equivalent.

* * * * *

## PART 121—CERTIFICATION AND OPERATIONS: DOMESTIC, FLAG, AND SUPPLEMENTAL AIR CARRIERS AND COMMERCIAL OPERATORS OF LARGE AIRCRAFT

6. By amending § 121.312 by redesignating present paragraphs (a) and (b) as (1) and (2), by redesignating the introductory paragraph as (a), and by adding a new paragraph (b) to read as follows:

**§ 121.312 Materials for compartment interiors.**

* * * * *

(b) For airplanes type certificated after January 1, 1958, after November 26, 1987, seat cushions, except those on flight crewmember seats, in any compartment occupied by crew or passengers must comply with the requirements pertaining to fire protection of seat cushions in § 25.853(c), effective November 26, 1984, and Appendix F to Part 25 of this chapter, effective November 26, 1984.

(Secs. 313, 314, and 601 through 610, Federal Aviation Act of 1958, as amended (49 U.S.C. 1354, 1355, and 1421 through 1430); 49 U.S.C. 106(g) (Revised, Pub. L. 97–449, January 12, 1983))

Issued in Washington, D.C., on October 23, 1984.

**Donald D. Engen,**

*Administrator.*

[FR Doc. 84–28294 Filed 10–23–84; 2:10 pm]

BILLING CODE 4910–13–M

federal register

Friday
October 26, 1984

Part V

# Environmental Protection Agency

40 CFR Parts 51 and 52

**Requirements for Preparation, Adoption and Submittal of Implementation Plans; Approval and Promulgation of Implementation Plans; Final and Proposed Rules**

**ENVIRONMENTAL PROTECTION AGENCY**

**40 CFR Parts 51 and 52**

**[AD-FRL 2683-1]**

# Requirements for Preparation, Adoption and Submittal of Implementation Plans; Approval and Promulgation of Implementation Plans

**AGENCY:** Environmental Protection Agency (EPA).

**ACTION:** Final rule.

**SUMMARY:** EPA here reaffirms its current requirements for the inclusion of fugitive emissions in calculations of whether a source is "major" for purposes of new source review under the Clean Air Act (the "Act"). EPA proposed to delete those requirements at 48 FR 38742 (August 25, 1983). In a companion notice in the **Federal Register**, EPA is proposing to extend the requirements for inclusion of fugitive emissions to surface coal mines, is reopening the comment period on the current list of sources to which these requirements apply, and is soliciting comments on an interpretive ruling regarding the underlying statutory provisions as they relate to consideration of fugitive emissions in the modification of existing sources.

**DATES:** This reaffirmation takes effect on November 26, 1984. Under section 307(b)(1) of the Act, 42 U.S.C. 7607(b)(1), petitions for judicial review must be filed on or before December 26, 1984, in the United States Court of Appeals for the District of Columbia Circuit.

**FOR FURTHER INFORMATION CONTACT:** Kirt Q. Cox, New Source Review Section, Office of Air Quality Planning and Standards, Research Triangle Park, North Carolina 27711 (919-541-5591, FTS-629-5591).

**SUPPLEMENTARY INFORMATION:**

## I. Introduction

EPA has various rules that regulate the construction of new stationary sources of air pollution and modifications to existing sources. In general, these rules apply only to sources and modifications that are "major." The rules define "major" in terms of annual rates of emissions, for example, 100 tons per year ("tpy"). They require the inclusion of "fugitive emissions" in quantifying emissions rate, but only for 30 listed source categories. "Fugitive emissions" are emissions that could not reasonably pass through a stack or other functionally equivalent opening.

On August 25, 1983, EPA proposed to delete entirely this requirement for the inclusion of fugitive emissions on the grounds that it rests on an incorrect interpretation of the Act (48 FR 38742). After reviewing the comments on the proposal, however, EPA has concluded that the interpretation in question is correct. EPA, therefore, has decided to retain the requirement, but to provide a further opportunity for comment on whether the requirement should not apply to one or more of the 30 categories already listed. In a companion notice in the **Federal Register**, EPA is soliciting comment on that question. In that notice, EPA is also proposing to add surface coal mines to the list and is soliciting comment on a new interpretation of the underlying statutory provisions as they apply to modifications involving fugitive emissions.

This preamble describes the statutory and regulatory background, the comments on the August 1983 proposal, and EPA's responses.

## II. Background

### *A. Statute*

Section 110 of the Clean Air Act, 42 U.S.C. 7410, requires each State to have a plan for (1) attaining and maintaining national ambient air quality standards (NAAQS) in all areas of the country, especially in areas that have yet to meet the standards ("nonattainment areas"); and (2) preventing significant deterioration in areas not classified as nonattainment ("PSD areas"). Section 110(a)(2)(D) requires each plan ("State implementation plan" or "SIP") to contain, among other measures, a program for regulating the construction of new stationary sources and modifications, including "a permit program as required in parts C and D * * * and a permit or equivalent program for any major emitting facility * * *." Section 110(a)(2)(I) requires each SIP to contain a ban on the construction or modification of any "major stationary source" in nonattainment areas that lack a SIP that meets the requirements of Part D.

Part C of the Act, 42 U.S.C. 7470-91, specifies a permit program that applies to the construction or modification of any "major emitting facility" in any PSD area. It defines "major emitting facility" as any stationary source that either emits 100 tpy of a pollutant and belongs to one of 28 listed industrial categories or else emits 250 tpy of a pollutant.

Part D of the Act, 42 U.S.C. 7501-08, specifies a permit program that applies to the construction or modification of any "major stationary source" in any nonattainment area. It contains no definition of "major stationary source."

Section 302(j) of the Act, 42 U.S.C. 7602(j), defines both "major stationary source" and "major emitting facility" as follows:

> Except as otherwise expressly provided, the terms "major stationary source" and "major emitting facility" mean any stationary facility or source of air pollutants which directly emits, or has the potential to emit, one hundred tons per year or more of any air pollutant (including any major emitting facility or source of fugitive emissions of any such pollutant, *as determined by rule by the Administrator*). [Emphasis added.]

### *B. Initial EPA Interpretation*

Following the enactment of the bulk of these statutory provisions in 1977, EPA issued various regulations and guidelines that interpreted and elaborated upon them. In June 1978, EPA promulgated PSD regulations to implement Part C. The regulations appear now at 40 CFR 51.24 (1984) (the "Part 51 PSD regulations") and 40 CFR 52.21 (1984) (the "Part 52 PSD regulations"). In January 1979, EPA revised its Emissions Offset Interpretative Ruling (the "Offset Ruling"), which now appears at 40 CFR Part 51, Appendix S (1984), to conform it to Part D. Then, in April 1979, EPA issued a guideline entitled "General Preamble for Proposed Rulemaking on Approval of Revisions for Nonattainment Areas" that included guidance on the content of Part D permitting programs (*see* 44 FR 20372). Finally, in July 1979, EPA issued an interpretive rule on construction bans for nonattainment areas, which now appears at 40 CFR 52.24 (1984).

In each of these pronouncements, EPA assumed without discussion that the fugitive emissions of a source of modification were to be included in quantifying its emissions rate in order to determine whether it is "major" [*see, e.g.*, 43 FR 26 382-83, 26403-04 (June 19, 1978)]. (EPA refers to these quantifications of emissions rate as "threshold applicability determinations," since they determine whether a project is subject to all the PSD or nonattainment permit requirements or the construction ban.)[1]

[1] A threshold applicability determination is distinct from a pollutant applicability determination, which is a determination of which pollutant streams from a "major" source or "major" modification are subject to the substantive requirements of the regulations in question. The PSD requirements, for instance, apply to each regulated pollutant that a "major" source emits in "significant" amounts. *E.g.*, 40 CFR 52.21(j) (1984).

EPA regarded the Part C definition of "major emitting facility" as exclusively governing the meaning of that term for PSD purposes. Since that definition does not distinguish between fugitive and nonfugitive emissions, EPA concluded that fugitive emissions are as eligible for inclusion in the threshold determinations of PSD applicability as nonfugitive emissions.

One of the consequences of this assumption was that sources of predominately fugitive emissions, such as surface coal mines, could be "major" and hence subject to new source review permit requirements or the construction ban.

*C. D.C. Circuit Interpretation*

In December 1979, the U.S. Court of Appeals for the District of Columbia Circuit held that EPA may require the inclusion of fugitive emissions in threshold applicability determinations for projects in a particular category only if it has first satisfied the rulemaking requirement of section 302(j) as to that category (*see Alabama Power Company* v. *Costle*, 636 F.2d 325, 369). Unfortunately, the court did not specify what it thought EPA had to consider in such a rulemaking. It did say, however, that:

> EPA's regulation of fugitive emissions has been of special concern to the mining and forestry industries which contend, without serious opposition, that they are incapable of meeting the strict limitations on the emission of particulate matter set by the PSD provisions * * *.
>
> * * * * *
>
> The legislative history of this rulemaking provision [Section 302(j)] is sparse, but *it may well define a legislative response to the policy considerations presented by the regulation of sources where the predominant emissions are fugitive in origin, particularly fugitive dust.* Whatever the motivation of the "rule" provision of 302(j), its existence is unmistakable. Even if the origin of this provision is fortuitous, the provision may well be welcomed as serendipitous, *for it gives EPA flexibility to provide industry-by-industry consideration and appropriate tailoring of coverage.* [*Id.* (emphasis added).]

*D. Revisions in Response to Alabama Power*

In response to this holding, EPA proposed amendments to both the PSD and nonattainment regulations that would exclude fugitive emissions from threshold applicability determinations except as to 30 listed categories of sources [*e.g.*, 44 FR 51924, 51948 (September 5, 1979)]. Twenty-eight of the categories correspond generally to the categories in the Part C definition of "major emitting facility"; the remaining two categories encompassed any source subject on August 7, 1980, to an emission standard under either Section 111 or 112 of the Act, 42 U.S.C. 7411 or 7412. Surface coal mines were not among the 30 categories (*Id.* at 51931).[2] EPA explained that it was proposing to require the inclusion of fugitive emissions as to those categories because (1) emissions from sources in those categories deteriorate air quality regardless of how they emanate, and (2) the Agency's experience in quantifying fugitive emissions from such sources was in general greater than its experience in quantifying fugitive emissions from other sources (*Id.*).

During the comment period, various industry representatives attacked this test as too undemanding to satisfy the court's opinion. They contended that section 302(j) obliges EPA to determine with some precision and through rulemaking that reasonably satisfactory methods for the measurement, modeling, and control of fugitive emissions[3] from a particular category of sources exist before EPA requires those emissions to be included in threshold applicability determinations [45 FR 52676, 52692 (col. 2) (August 7, 1980)]. Indeed, some contended that EPA had to promulgate such methods in the form of regulations [*Id.* at 52690 (col. 3)].

In its response to comments, EPA pointed out that, according to the D.C. Circuit, Congress intended the substantive PSD requirements to be applied "with equal force" to the fugitive and nonfugitive emissions of any facility that would be "major" by virtue of its nonfugitive emissions, even if EPA has yet to determine that there are reasonably satisfactory measurement, modeling, or control methods for the fugitive emissions [*Id.* at 52691 (quoting 636 F.2d at 369)].[4] Thus, Congress consigned any problems of measurement, modeling, and control in those cases to each individual permit proceeding for resolution by the permitting authority. EPA reasoned that if Congress were willing to accept case-by-case resolution of such problems by the permitting authority in those circumstances, it must have been willing to do the same for projects that would be "major" only if their fugitive emissions were counted (*Id.* at 52691, 52692). Hence, the Agency took the position that section 303(j) obliges it simply to afford the public with an opportunity to oppose the inclusion of fugitive emissions as to particular category, once EPA has determined at the proposal stage that sources in the category could degrade air quality significantly:

> EPA * * * believes that the purpose of the rulemaking under section 302(j) is to afford members of affected categories of sources an opportunity to comment on the Administrator's determination to include fugitive emissions in the threshold calculation and to allow them to present factual or policy arguments in support of claims that it would *not* be appropriate to do so. [*Id.* at 52690 (col. 3) (emphasis in original).]

EPA did not specify the grounds on which it thought a commenter could oppose inclusion of fugitive emissions. But the possible candidates include adverse economic or social impacts relative to the benefits associated with the listing of the applicable category. EPA in harmony with its basic reasoning said that it thought that the adequacy of measurement and modeling methods is not by itself "critical in determining whether, as a general policy matter, it is appropriate to include fugitive emissions in the threshold calculations" [*Id.* at 52692 (col. 2)].

In sum, EPA's position was that section 302(j) requires it to determine only that (1) the sources in category could degrade air quality significantly and (2) there were no unreasonable costs compared to the benefits associated with listing the category that commenters raised during the comment period. If no commenter raised on objection, then EPA would have to make only the first determination.

EPA concluded finally that the rulemaking it was conducting had afforded sources the opportunity to comment on the proposed inclusion of fugitive emissions in their threshold calculations (*Id.* a 52961). Hence, in August 1980, it promulgated the substance of the amendments it had proposed (*e.g.*, 45 FR 52739).[5]

---

[2] EPA said it would consider later whether to add strip mines and other sources to the list [*Id.* (col. 2)].

[3] The phrase "measurement of fugitive emissions" refers in this notice to the quantification of the rate at which pollutants emanate "fugitively" from a particular activity at a source, for instance, the rate at which particulate matter emanates from an unpaved road at a surface mine due to truck traffic. The phrase "modeling of fugitive emissions" refers to the prediction through mathematical models of the concentrations of a pollutant in the ambient air that would result from fugitive emissions of the pollutant.

[4] The relevant language from the court's opinion is as follows: The term of section 165, which detail the preconstruction review and permit requirements for each new or modified "major emitting facility" *apply with equal force* to fugitive emissions and emissions from industrial point sources * * *.

EPA is correct that a major emitting facility is subject to the requirements of section 165 for each pollutant it emits irrespective of the manner in which it is emitted. However, a source emitting large quantities of fugitive emissions may remain outside the definition of major emitting facility and thus may not be subject to the requirements of section 165. [Emphasis added.]

[5] EPA simultaneously promulgated a wide array of other changes to the various new source review

Continued

EPA, however, put the changes into a different form. The new provisions on their face require fugitive emissions to be included in threshold applicability determinations for any project, but then exempt from the relevant PSD or nonattainment requirements any project that (1) would be "major" only if fugitive emissions were included and (2) does not belong to one of the 30 categories [*e.g.*, 40 CFR 52.21 (b)(4), (i)(4)(vii) (1981)].

*E. Industry Challenges*

In December 1980, the American Mining Congress and various individual mining companies (collectively, "AMC") petitioned EPA for reconsideration of the new PSD provisions. AMC pointed out that, even though the provisions would exempt a mining operation that would be "major" only if fugitive emissions were taken into account for the PSD permit requirements, nevertheless they could affect such an operation adversely in other ways.[6] AMC also observed that the preamble to the regulations strongly indicates that EPA did not intend these results [*see* Petition for Reconsideration of Regulations Relating to the Prevention of Significant Deterioration of Air Quality, Part I (December 1, 1980) hereinafter, "AMC Petition for Reconsideration")].

In a letter dated January 19, 1981, EPA granted the AMC petition. The Agency confirmed that it intended to establish that any project that would be "major" only if fugitive emissions were taken into account is not to be considered "major" for any PSD purpose, unless the project belongs to one of the 30 listed categories. EPA agreed to amend the regulations to conform then to that intention.

In late 1980, AMC and other industry organizations (collectively, the "industry petitioners") petitioned the D.C. Circuit to review the provisions that require the fugitive emissions of projects in the 30 listed categories to be taken into account in threshold applicability determinations. These challenges were subsequently consolidated into *Chemical Manufacturers Association ("CMA")* v. *EPA* (No. 79-1112).[7]

The industry petitioners argued that EPA, before it established those provisions, should have considered the problems of measuring, modeling, and controlling fugitive emissions that are peculiar to each category and then provided—in the words of the *Alabama Power* opinion—"appropriate tailoring of coverage." They also contended that the Act required the Agency to consider, on an industry-by-industry basis, the social, economic, health, and welfare impacts of including fugitive emissions in threshold applicability determinations. They suggested that EPA could decline to require the inclusion of fugitive emissions as to a particular category on the ground that growth in that industry was important to the economy and that the emissions posed low risks to human health and welfare. Finally, the industry petitioners asserted that EPA entirely failed to meet those requirements of the Act [*see* Petitioners Brief on Fugitive Emissions and Certain other Issues, at 12–19 (February 11, 1981) (hereinafter, "Fugitive Emissions Brief")].

In June 1981, EPA began negotiations with the industry petitioners to settle the issues relating to fugitive emissions in the *CMA* case. In February 1982, EPA entered into a settlement agreement with these petitioners in which it agreed to propose to delete the requirement for including fugitive emissions and to take final action on that proposal. Subsequently, the court granted a stay of the case pending implementation of the agreement.

*F. D.C. Circuit Decision in Duquesne Light*

In January 1983, the D.C. Circuit issued an opinion that is relevant here on EPA's noncompliance penalty regulations under section 120 of the Act (*Duquesne Light Company* v. *EPA*, 698 F.2d 456). The extent of liability for those penalties depends in large measure on whether a source qualifies as a "major stationary source," which is also defined by Section 302(j). The noncompliance penalty regulations simply required the inclusion of fugitive emissions in threshold applicability determinations to the extent they were "regulated by the applicable state implementation plan" [40 CFR 66.3(j)(1981)]. Industry challenged this requirement as failing to satisfy the rulemaking requirement in section 302(j). They asserted:

> To properly notify sources how emissions will be calculated requires the formal promulgation of measurement, modeling, or control techniques, or guidelines for determining the categories to which fugitive emissions will apply and the basis for quantifying such emissions.
>
> * * * * *
>
> EPA has not complied with this Court's command to "provide industry-by-industry consideration and the appropriate tailoring of coverage" [Joint Brief for Industry Petitioners on Applicability of the Regulations, at 48–49 (April 26, 1982)].

The court responded as follows:

> EPA's treatment of fugitive emissions is * * * reasonable. Section 120 penalties against major stationary sources are to be assessed for violations of state SIP's. The use of SIP's to determine whether fugitive emissions should be included in calculating a source's potential to emit reasonably links the noncompliance penalties against major sources with the SIP's that give rise to their assessment.
>
> Moreover, EPA has engaged in the rulemaking required for inclusion of fugitive emissions in the calculation of whether a source is major. [*See* § 302(j)]. In promulgating the PSD regulations, EPA assumed that the rulemaking requirement was inapposite. Here, by contrast, EPA followed SIP regulations in determining whether to include fugitive emissions. As we emphasized in *Alabama Power*, the purpose of the rulemaking requirement may have been to enable EPA to tailor the inclusion of fugitive emissions to particular industrial conditions. Adoption of a SIP involves an exploration of whether industrial conditions in the state warrant limiting fugitive emissions from a particular source. EPA's reliance on the SIP itself to determine whether to include fugitive emissions in the calculation of a source's potential to emit thus met the statutory rulemaking requirement, and we affirm that action. [698 F.2d at 474–75].

*G. EPA Proposal*

To meet the commitments on fugitive emissions that EPA made to AMC in January 1981 and to industry petitioners in February 1982, EPA proposed certain amendments to its regulations on August 25, 1983 (48 FR 38742). The main effect of these amendments would be (1) to delete the current requirements for including fugitive emissions in threshold applicability determinations, and (2) expressly to exempt from all substantive requirements applicable to "major" projects any project that would be "major" only if its fugitive emissions were included.

In the preamble to the proposal, EPA stated its "preliminary conclusion" that it had misinterpreted section 302(j) when it promulgated the current requirements

---

regulations in effect at the time: not only the Part 51 and 52 PSD regulations, the Offset Ruling, and the construction ban, but also 40 CFR 51.18(j), which set forth the requirements of the Part D permit program and which EPA had first promulgated in May 1980 (45 FR 31307).

EPA did not include strip mines on the list of 30 categories, although the Sierra Club in its comments had argued for their inclusion.

[6] For example, such an operation would consume increment even before the baseline date, if construction on it commenced after January 6, 1975 [*see* 40 CFR 52.21(b)(13)(ii)(a) (1981)].

[7] The Sierra Club challenged the apparent decision to postpone action on whether to list strip mines. The court considered this challenge separately from the challenges in *CMA*.

and hence had failed to go through the necessary rulemaking. EPA identified two new interpretations of how the rulemaking requirement of section 302(j) was to be conducted. One was that:

> [t]he parenthetical [in section 302(j)] obliges EPA, before it may require the inclusion of fugitive emissions in threshold applicability determinations for a particular Clean Air Act program and a particular category of sources, only to (1) identify those problems the sources would encounter in that program that are specifically due to the fugitive nature of their emissions, and (2) determine that reasonable solutions to those problems exist. For the PSD and nonattainment new source review programs and some source categories, those problems may include problems of measurement, modeling, and control (48 FR 38744–45).

The second interpretation was that:

> EPA, before it may require the inclusion of fugitive emissions in threshold applicability determinations [for a particular category of sources], need determine only that reasonable solutions exist for the problems of measurement that are endemic to the fugitive emissions from those sources [48 FR 38745 (col. 1)].

EPA said that it viewed these interpretations as stronger than the one it had adopted in 1980, the one industry had espoused in its brief, or the one that the Natural Resources Defense Council ("NRDC") raised in a September 1982 letter. The NRDC interpretation was that the parenthetical merely requires EPA to identify those sources that are substantial emitters of fugitive emissions [48 FR 38745 (col. 2–3)]. EPA asked commenters, in commenting on these various interpretations, to take the D.C. Circuit decision in *Duquesne Light* into account [*Id.* (col. 3)].

Another effect of the amendments EPA proposed would be to allow credit for net decreases in fugitive emissions in netting calculations under the definition of "major modification," even if EPA had yet to go through the necessary rulemaking for the source in question.[8]

EPA observed that disallowance of credit for net decreases "could result in a company having to obtain a permit [for a source alteration], but not having to satisfy any substantive requirements," and concluded that Congress could not have intended that (48 FR 38746).

In the preamble, EPA recognized that some environmentally significant projects that now would be subject to PSD or nonattainment requirements would escape those requirements if EPA were to make the proposed amendments final before completing the necessary section 302(j) rulemaking. In view of this, EPA said that, if it made the amendments final, it would probably not do so until it had completed the necessary rulemaking as to those projects [48 FR 38747 (col. 1)].[9]

## III. Comments

The commenters on the proposal addressed mainly the various interpretations of section 302(j) that EPA had outlined. They also gave views on *Duquesne Light*, the crediting of net decreases in fugitive emissions under the definition of "major modification," the postponement of deletion pending the necessary rulemaking, and the inclusion of fugitive emissions in pollutant applicability determinations. This section summarizes the material comments in each of those areas.

### *A. Comments Favoring the Industry Interpretation*

Many commenters supported roughly the interpretation industry had advanced in its briefs in the *CMA* case. They agreed that section 302(j) requires EPA to conduct a rulemaking to ascertain that reasonable techniques exist for measuring, modeling, and controlling fugitive emissions for each source category. Many commenters further contended that even if emissions from a source can be measured and modeled, there is little point in subjecting the source to PSD review if there is no economically and technically reasonable control methods.

Central to these arguments was *Alabama Power's* observation that section 302(j) contemplates a "legislative response to policy considerations" and gives "flexibility to provide industry-by-industry consideration and the appropriate tailoring of coverage." These commenters viewed such language as requiring EPA to engage in a broad rulemaking, not one limited to consideration of a single technical factor, such as measurement, in disregard of other considerations, such as modeling and control techniques, and important social and economic factors.

Finally, representatives of the mining industry submitted extensive comments on why fugitive dust emissions from mines should not be included in threshold applicability determinations. AMC said that, under the current definition of particulate matter, it would be impossible to permit any but the smallest new or modified mines if mines were brought under the PSD regulations, even if mining operations applied BACT, and even though their emissions present no substantial health or welfare concerns. It would be impossible, AMC asserted, because the mines would cause violations of the PSD increments for particulate matter.

### *B. Comments Favoring EPA's First Interpretation in the Proposal*

EPA's first interpretation of section 302(j) was that it obliges the Agency, before it includes fugitive emissions in threshold applicability determinations, only to (1) identify those problems that sources would encounter in the program in question that are specifically due to the fugitive nature of emissions, and (2) determine that reasonable solutions to those problems exist. One commenter expressly agreed with this interpretation, stating that "emissions must be quantifiable and reasonable solutions must exist." Another commenter stated that its support of EPA's proposal was contingent on EPA's ability to actually resolve within some reasonable time the problems arising from the inclusion of fugitive emissions. This commenter noted that health and welfare effects occur regardless of whether pollutants are emitted from stacks or are fugitive.

### *C. Comments Favoring EPA's Alternative Interpretation*

As noted above, an alternative interpretation that EPA proposed was that section 302(j) requires EPA to determine only that reasonable solutions exist for problems of measurement that are endemic to the fugitive emissions from a source category before it may require inclusion

[8] Specifically, EPA proposed to delete the current exclusion for projects that fall outside the 30 categories and to add a new paragraph to the definitions of "major stationary source" and a similar paragraph to the definition of "major modification." This first paragraph would provide that the "fugitive emissions of a stationary source shall not be included in determining for any of the purposes of [the regulations in question] whether it is a major stationary source, unless the source belongs to one of the following categories of stationary sources: [Reserved]" [*See, e.g.*, 48 FR 38753 (col. 2) (Section A(1))]. The second paragraph would provide that "[a]ny net increase in fugitive emissions from a change at a stationary source shall not be included in determining for any of the purposes of [the regulations in question] whether the change is a major modification, unless the source belongs to one of the following categories of stationary sources: [Reserved]" [*See, e.g.*, 48 FR 38753 (col. 3) (Section A(4))].

[9] The day after the publication of this proposal, August 26, 1983, the D.C. circuit issued its decision in *Sierra Club* v. *EPA*, the challenge to EPA's postponement of a decision on whether to add strip mines to the list of 30 categories. The court remanded the postponement, ruling that EPA under the logic of its 1980 action appeared to have no good reason for listing the 30 categories but not strip mines (715 F.2d 653). EPA subsequently agreed, and committed to propose to list or not to list strip mines. The court then ordered EPA to propose one way or the other by a specific date. In a companion notice in the **Federal Register**, EPA is proposing to list strip mines.

of fugitive emissions in threshold applicability determinations.

A State agency strongly supported this interpretation, noting that "the only real difference between fugitive emissions is in the degree of difficulty in measuring emissions rates." This commenter stated that once emission levels are known, there is no important difference in either controlling or modeling fugitive emissions. Modeling fugitive emissions, this commenter stated, is often simply a matter of including deposition factors in the models in order to account for the settling of larger particles.

*D. Comments Favoring EPA's 1980 Interpretation*

While no commenter directly focused on EPA's 1980 interpretation, several did offer general support for it. For example, the San Diego Air Pollution Control District concluded that Congress did not have in mind the "cumbersome administrative process" that the industry and the proposed EPA interpretations would entail. The District concluded that "Congress * * * provided EPA with the flexibility to subject fugitive emissions to the same requirements as those for nonfugitive emissions if that is deemed appropriate." In addition, the California Air Resources Board expressed support for the existing regulations and recommended that EPA begin work to add to the current list of 30 categories for which fugitive emissions are known to be significant. Other commenters, while not expressly supporting the approach taken in the 1980 rulemaking, strongly supported retention of the coverage of fugitive emissions that it provided. These commenters, such as the Association of Local Air Pollution Control Officials, also generally stressed that "[f]ugitive emissions represent a very significant component of total air pollution emissions for many industrial categories * * *," and that these fugitive emissions have as great an impact on health and welfare as stack emissions.

*E. Comments Favoring the NRDC Interpretation*

Many commenters favored the NRDC interpretation, which is that section 302(j) requires EPA merely to identify those sources that are substantial emitters of fugitive emissions. They contended that to exclude figitive emissions from threshold applicability determinations would have substantial adverse air quality effects. According to these commenters, section 302(j) performs a sorting function, telling permitting agencies on a category-by-category basis which sources must count fugitive emissions and which need not. The commenters noted that there is nothing in the Act's legislative history that would require a more complicated test. Since Congress was very specific in detailing permit processes, as in sections 165 and 173, it is highly unlikely that Congress, by use of the unadorned word "rule" in section 302(j), intended to establish difficult hurdles for EPA to jump before requiring sources to account for great amounts of fugitive emissions. The only legislative comment on the parenthetical in section 302(j) is the House Report which says simply that "the major stationary source definition is clarified to indicate inclusion of major sources of fugitive emissions. Last year's bill was unclear in this respect." This reflects Congress' appreciation that fugitive emissions, no less than stack emissions, are real pollution.

NRDC added that interpretations that would require EPA to do more than conclude that fugitive emissions can be measured for a source category have absolutely no support in the Act, since section 302(j) relates only to one issue, whether a source emits more than 100 tons per year. Modeling, control technology, and economic and social impacts have nothing to do with this, the commenter noted, and indeed EPA has properly left room for handling the measurement issue in allowing sources to make hardship determinations at the applicability stage.

In rebuttal comments, the American Petroleum Institute (API) criticized NRDC's interpretation on the grounds that it (1) makes the rulemaking requirement of section 302(j) meaningless; (2) violates *Alabama Power's* construction of section 302(j); (3) fails to recognize that the Act distinguishes between fugitive and stack emissions; (4) would unreasonably preclude consideration of measurement, modeling, and control issues which are necessary to apply the PSD requirements and can best be dealt within in the context of national rulemakings rather than individual permit proceedings where individual source owners cannot marshall the same resources and broad-based unput; and (5) would effectively eliminate the "impossibility of compliance" criterion sanctioned by *Alabama Power.*

*F. Interpretation of Duquesne Light*

Commenters who considered *Duquesne Light* differed in their view of the significance of that court's holding according to the interpretation of section 302(j) that then endorsed.

AMC argued that *Duquesne Light* is not controlling here, despite its "superficial similarity," because the fugitive emissions issue in that case was minor and peripheral. The purpose of including fugitive dust emissions in applicability determinations under Section 120 was simply to impose penalties on companies that it did not comply with State fugitive emissions requirements. But the consequences of including fugitive emissions in PSD determinations, AMC argued, are far more significant, amounting in some cases to a bar on new sources.

Another commenter stated that *Duquesne Light* reaffirms that section 302(j) rulemaking allows EPA to tailor inclusion of fugitive emissions to particular industry conditions, but stated that the same outcome should not necessarily follow for PSD as for the very different section 120 program.

Another commenter stated that *Duquesne Light* supports a view of section 302(j) that requires EPA to determine that "reasonable controls" exist for fugitive source categories. The court limited its holding to sources subject to SIP requirements, which, according to the commenter, generally entail only reasonable controls.

On the other hand, one commenter stated that EPA's proposal goes far beyond any requirement imposed under *Duquesne Light* by placing a heavy burden on EPA both to identify industry fugitive emissions and to come up with reasonable solutions to the problems of modeling and measurement before requiring inclusion of fugitive emissions in threshold applicability determinations. Similarly, NRDC stated that *Duquesne Light* rejected precisely the contentions industry makes in this rulemaking, and upheld the same type of simple sorting determinations that NRDC says are all that section 302(j) requires.

*G. Crediting of Decreases in Fugitive Emissions*

Most commenters opposed EPA's proposal to credit net decreases in fugitive emissions, but not net increases. These commenters agreed that the proposal in this respect is "anomalous," and stated that increases and decreases should be treated similarly.

One State agency stated that both increases and decreases in fugitive emissions should be taken into account and that it was feasible to quantify most fugitive emissions. Another State agency, in noting that logic would require equal treatment of fugitive emissions increases and decreases, stated that the aim of section 302(j) is not to relax requirements with respect to nonfugitive emissions.

Several commenters stated that EPA should follow the terms of the settlement agreement, which provided for equal treatment of increases and decreases.

The Department of the Interior (DOI) commented that:

> EPA's rationale for allowing credit for decreases in fugitive emissions while excluding increases in determining whether a source is major is flawed. EPA states that if both increases and decreases in fugitive emissions are excluded in determining if a source is a major modification, the result could be that a company might be required to obtain a permit, but not have to satisfy any substantive requirements, if the sum of contemporaneous increases and decreases are less than the values considered "significant" under 40 CFR 52.21(b)(23)(i) * * * Although this could occur in some instances, it is not necessarily true for all situations. For example, this is not true in the case when the sum of contemporaneous fugitive increases and nonfugitive increases exceed fugitive decreases by more than the specific significant pollutant levels. The following example illustrates this point:

| | Nonfugitive VOC (TPY) | Fugitive VOC (TPY) |
|---|---|---|
| First modification | +39 | +2,000 |
| Second modification | +2,039 | −2,000 |
| (Contemporaneous with first modification) | +2,078 | 0 |

> If increases in fugitive emissions are not included in the PSD applicability determination, the first modification, although increasing emissions by 2039 TPY would be exempt from PSD review (significant VOC level is 40 TPY). The second modification would also be exempt from review if the fugitive decreases are credited. [Note that under current EPA policy, if the sum of increases and decreases resulting directly from a proposed alteration is not "significant," then the alteration cannot be a "major modification," regardless of other contemporaneous emission increases.] However, if fugitive decreases could not be credited, the second modification would trigger the substantive PSD requirements by virtue of a net 2078 TPY increase in contemporaneous emissions. Suppose further that a different source proposes a modification resulting in a 40 TPY increase in nonfugitive VOC emissions. This modification, assuming no contemporaneous decreases, would require PSD review, whereas the first modification emitting 2039 TPY would require no PSD review whatsoever under the proposed revision. These examples illustrate the inconsistency and inequity of crediting decreases while ignoring fugitive emission increases. [Footnote omitted.]

DOI agreed that creditable decreases in fugitive emissions should be allowed, but only if increases are also counted and the decreases result from an enforcement control strategy. It follows logically, DOI stated, that any source category using quantifiable decreases in fugitive emissions should be held liable for any fugitive emissions increase quantified in the same way. DOI noted that past State permitting has produced much information in estimating fugitive emissions from various source categories. Finally, DOI noted that the EPA proposal would encourage piecemeal project development since a source, by breaking a project in two, could get the fugitive sources exempt under the new provisions and then apply for its nonfugitive sources, thereby eliminating review of the fugitive emissions.

In supporting DOI's comments, another commenter noted that it is important that the permit continue to be the authoritative record of the current obligations of each major source because permits are the only reference point for (1) enforcement by EPA and the public; and (2) modeling by subsequent permit applicants in areas to assess the cumulative impact of all PSD sources on increments, NAAQS, and air quality related values of Class I areas. Moreover, this commenter stated, the EPA scenario illustrates why its proposal not to count increases in fugitive emissions is faulty; since Congress did not intend meaningless permits, then it must have intended fugitives to be counted, both increases and decreases.

The American Iron and Steel Institute (AISI) commented that EPA's proposal, though perhaps lacking in symmetrical logic, is justifiable as a practical, realistic interim approach to a short-term problem and, as such, is a reasonable exercise of EPA's discretionary authority. AISI noted that the superficially inconsistent treatment of fugitive emissions increases and decreases is temporary, and will last only until EPA undertakes category-by-category 302(j) rulemaking. In addition, AISI stated that by allowing credit for decreases, EPA provided a strong incentive for companies to develop fugitive emissions measuring, modeling, and control techniques, and this added experience gained during the interim period may aid EPA in making section 302(j) category-by-category determinations.

*H. Future Rulemaking on Fugitive Emissions*

EPA stated that it planned, if it were still inclined after reviewing comments to delete the current requirements, to withhold final deletion until it completed the necessary rulemaking to reestablish the requirement as to at least some of the 30 categories presently listed.

Many commenters disagreed with this approach and stated that EPA should delete the 30 source categories in accordance with the *CMA* settlement agreement. Some stated that EPA needed to delete the 30 categories now because, under the holding in *Alabama Power*, it simply had no authority to keep them in effect until after it had instituted industry-by-industry rulemaking. API stated that NRDC grossly overestimated the impact of delisting fugitive emissions until completion of an adequate rulemaking.

NRDC stated that while this proposal was an improvement, it had two concerns: (1) That EPA had not indicated how many or which categories are encompassed by the phrase "at least some," and (2) that the entire exercise would be a waste of time since it is clear that EPA will be able to make determinations that fugitive emissions should be included for nearly every source category.

DOI supported withholding immediate deletion because it would avoid the permitting loophole for large sources of fugitive emissions that could adversely affect Class I areas such as national parks.

Other commenters also supported the EPA proposal to avoid confusion, to avoid a rush of applications for permits during the time between this amendment and promulgation of the section 302(j) list, and to avoid the incongruity of reviewing sources subject to PSD under different sets of rules before and after section 302(j) rulemaking.

*I. Inclusion of Fugitive Emissions in Pollutant Applicability Determinations*

Two commenters stated that, as with threshold applicability determinations, EPA should not include fugitive emissions in pollutant applicability determinations before acting under section 302(j). API stated that applicability determinations, whether for an entire source or an individual pollutant, involve the same function; each examines emissions at a particular stage in the process to determine whether PSD review will be triggered. Any problems relating to quantification, control, and increment compliance which arise in such an inquiry—and are cognizable in section 302(j) rulemakings—will exist irrespective of the context in which fugitives are being considered.

## IV. Response to Comments

### *A. Interpretation of Section 302(j)*

EPA has concluded that its 1980 interpretation regarding the nature of the section 302(j) rulemaking requirement was correct. Congress intended that EPA make only two determinations before it required fugitive emissions to be included in threshold applicability determinations for sources in a particular category: (1) That the sources have the potential to degrade air quality significantly and (2) that no unreasonable socioeconomic impacts relative to the benefits would result from subjecting the sources to the relevant PSD or nonattainment programs. Thus, a finding that the sources in a category pose a threat of significant air quality degradation is enough to propose listing, though EPA must consider broader-based objections raised by commenters during the rulemaking before taking final action.[10]

The intention behind section 302(j) is difficult to discern. On the one hand, the "by rule" requirement plainly evidences an intention (1) to shift the policy decision on whether new sources of predominantly fugitive emissions should be subject to PSD and nonattainment review to EPA, and (2) to ensure that EPA makes that decision only after it hears from the general public and the target industries. Contrary to the contentions of NRDC, it is unlikely that Congress intended EPA to determine merely that the sources in a category are substantial emitters, since to determine that requires little expertise and public participation.

On the other hand, the statute and the legislative history strongly suggest that Congress did not intend to require EPA to shoulder the burden of intense, industry-by-industry rulemaking. First, as EPA observed in 1980, the statute does not expressly require EPA to go through rulemaking to require the inclusion of fugitive emissions in pollutant applicability determinations. In fact, section 165(a), 42 U.S.C. 7575(a), by its own terms requires the inclusion of fugitives in such determinations. In addition, the committee report on the bill that first contained the "by rule" requirement completely ignores it, saying only that "the 'major stationary source' definition is clarified to indicate the inclusion of major surces of fugitive emissons (last year's bill was unclear in this respect)." H.R. Rep. No. 95–294, 95th Cong., 1st Sess. at 4 (1977). Similarly, the conference report completely fails to mention the requirement, even though it purports to summarize the definition of "major stationary source" in the House bill. H.R. Report No. 95–564, 95th Cong., 1st Sess. at 192 (1977). If Congress had intended to require the sort of rulemaking effort that industry contends it did, it would have shown at least some recognition of the intensity and massiveness of that effort.

The interpretation of section 302(j) that EPA espoused in 1980 reasonably harmonizes these discordant Congressional signals. A determination by EPA that the sources in a category pose a threat of significant air quality degradation in effect establishes a presumption that the sources should be subject to PSD and nonattainment review. This is because the primary purpose of that review is to prevent the construction of new projects that would interfere materially with timely attainment and maintenance of NAAQS and PSD increments. Commenters then may seek to rebut this presumption by producing a record that unreasonable social or economic costs relative to the anticipated benefits would occur if PSD or nonattainment review were applied to a particular category of sources.[11] In the end, it is EPA's role to resolve any clash of views. Thus, EPA engages in a deliberative process that can go far beyond the virtually ministerial decision-making that NRDC advocates, but that need go beyond it only if and to the extent that there are legitimate, cost/benefit concerns. Under this interpretation, section 302(j) functions as a useful "safety valve," while at the same time minimizing the expenditure of Agency resources.

This "safety valve" interpretation not only harmonizes the signals from Congress, it also is consistent with the relevant case law. It affords EPA precisely the "flexibility to provide industry-by-industry consideration and appropriate tailoring of coverage" that Judge Leventhal had in mind in *Alabama Power*. In addition, it assures that EPA will establish as to each category of sources that rational nexus between the listing and the purposes of the program in question that the D.C. Circuit found essential in *Duquesne Light*. Moreover, the rulemaking potentially "involves an exploration of whether industrial conditions * * * warrant limiting fugitive emissions from a particular source." *Duquesne Light Company* v. *EPA*, 698 F.2d at 475.[12] Finally, the D.C. Circuit has offered a similar approach in an analogous setting. *See Portland Cement Assoc.* v. *Ruckelshaus*, 486 F.2d 375, 387 (col. 2) (1973).[13]

### *B. Crediting of Decreases in Fugitive Emissions*

EPA agrees that its rationale for allowing credit for net decreases in fugitive emissions was flawed primarily for the reasons DOI gave in the comments quoted above.

### *C. Proposal to Postpone Deletion of the Current Listing Pending Rulemaking*

Since EPA has decided to reaffirm the current listing, its proposal to postpone deletion of the listing, and the comments on it, are moot.

### *D. Inclusion of Fugitive Emissions in Pollutant Applicability Determinations*

As EPA indicated above in sections II.D. and IV.A., it does not agree that Congress must have intended the rulemaking requirement of section 302(j) to apply to pollutant applicability determinations.

## V. Final Action

In light of its conclusion that its 1980 interpretation was correct, EPA has decided to retain its current requirements for the inclusion of fugitive emissions and, to reconfigure those requirements as they relate to major stationary sources in a form that as to those sources will satisfy its commitment to AMC in January 1981.

---

[10] In adopting this "safety valve" interpretation, EPA does not intend to suggest that it views section 302(j) as prohibiting it from gathering and analyzing cost/benefit data prior to a proposal or a final action under that section. To the contrary, while EPA has concluded that Congress did not intend to require a cost/benefit analysis before proposal, it has also concluded that Congress did not intend to prohibit it, either. Consequently, EPA intends to adhere in the future to applicable requriements for cost/benefit analyses under E.O. 12291 not only with respect to proposals but also with respect to final actions. 46 FR 13193 (1981).

This reaffirmation of the 1980 interpretation goes primarily to the content of the rulemaking requirement in section 302(j) as opposed to the *applicability* of the requirement. As indicated in the companion notice, EPA is inclined to view the requirement as not applying to modifications. For this reason, EPA is withholding final action with respect to that portion of the rulemaking regarding the definition of "major modification" pending a final decision on the new applicability interpretation. EPA is taking final action on the change in the definition of "major stationary source," but is retaining current provisions concerning the treatment of fugitive emissions from modifications.

[11] EPA will consider all available information regarding socioeconomic impacts and associated benefits in arriving at a final decision whether to list or not to list the given source category. EPA will consider a broad range of cost/benefit concerns, including economic efficiency, societal costs and benefits, and distributive costs such as changes in price, employment, and balance in trade.

[12] Further, the court in *Duquesne Light* rejected industry arguments, identical to those made in this proceeding, that EPA should make a much more searching inquiry into the availability of measurement, modeling, and control techniques.

[13] See Footnote, 10.

The amendments EPA is promulgating today are intended to implement this decision. As mentioned, the Agency, elsewhere in today's **Federal Register**, is proposing an interpretive ruling regarding the applicability of the section 302(j) rulemaking requirement to modifications. EPA anticipates final action on the interpretive ruling, after the public comment period, in the form of promulgating, as proposed in the August 25, 1983 **Federal Register**, the amendments to 40 CFR 51.24(i)(4)(ii); 52.21(i)(4)(vii); 51.18(j)(4); Part 51, Appendix S, II.G.; 52.24(h). This will also take the form of not promulgating the changes identified as 40 CFR 51.24(b)(2)(iv); 52.21(b)(2)(iv); 51.18(j)(1)(v)(d); Part 51, Appendix S, II.A.5(iv); 52.24(f)(5)(iv) in the August 25, 1983, proposal.

## VI. Parallel Actions

EPA's review of the 1980 rulemaking record has made it aware that that rulemaking could have given fuller notice of EPA's interpretation of section 302(j). Though EPA believes its 1980 rulemaking met the applicable legal standards, section 307(d)(8), 42 U.S.C. 7607(d)(8), as a matter of policy it is soliciting further comment on the listing of the 30 categories in a parallel notice in the "Proposed Rules" section of the **Federal Register**. If EPA receives significant comment on any source category, it will reconsider the listing of that category.

In the same parallel notice, EPA is also proposing to add strip mines to the list of 30 categories and to adopt a new interpretation of Section 302(j) as it relates to modifications.

## VII. Miscellaneous

The final actions here are nationally applicable and based on determinations of nationwide scope and effect. Therefore, under section 307(b)(1) of the Act, 42 U.S.C. 7607(b)(1), judicial review may be sought only in the United States Court of Appeals for the District of Columbia Circuit. Petitions for judicial rule must be filed on or before December 26, 1984.

## VIII. Regulatory Flexibility Act

As required by section 3(a) of the Regulatory Flexibility Act, 5 U.S.C. 605(b), I certify that this regulation will not have a significant impact on a substantial number of small entities. This action reaffirms the current requirements and imposes no new costs on any entities, small or large.

## IX. E.O. 12291

Under Executive Order 12291, this action is not considered "major." This action reaffirms the current requirements and therefore, does not have an annual effect on the economy of $100 million or more. This action has been submitted to the Office of Management and Budget for review under Executive Order 12291.

## List of Subjects

*40 CFR Part 51*

Administrative practice and procedures, Air pollution control, Intergovernmental relations, Reporting and recordkeeping requirements, Ozone, Sulfur oxides, Nitrogen dioxide, Lead, Particulate matter, Hydrocarbon, Carbon monoxide.

*40 CFR Part 52*

Air pollution control, Ozone, Sulfur oxides, Nitrogen dioxide, Lead, Particulate matter, Carbon monoxide, Hydrocarbons.

**Authority:** Section 101(b)(1), 110, 160–169, 171–178, and 301(a) of the Clean Air Act as amended [42 U.S.C. 7401(b)(1), 7410, 7470–79, 7501–08 and 7601(a); Section 129(a) of the Clean Air Act Amendments of 1977 (Pub. L. No. 95–95, 91 Stat. 685 (August 7, 1977))].

Dated: October 19, 1984.

**Alvin L. Alm,**

*Deputy Administrator.*

*A. Requirements for State PSD Plans*

Section 51.24 of Title 40 of the Code of Federal Regulations is amended as follows:

1. By adding a new paragraph (b)(1)(iii) to read as follows: "(iii) The fugitive emissions of a stationary source shall not be included in determining for any of the purposes of this section whether it is a major stationary source, unless the source belongs to one of the following categories of stationary sources:

(*a*) Coal cleaning plants (with thermal dryers);
(*b*) Kraft pulp mills;
(*c*) Portland cement plants;
(*d*) Primary zinc smelters;
(*e*) Iron and steel mills;
(*f*) Primary aluminum ore reduction plants;
(*g*) Primary copper smelters;
(*h*) Municipal incinerators capable of charging more than 250 tons of refuse per day;
(*i*) Hydrofluoric, sulfuric, or nitric acid plants;
(*j*) Petroleum refineries;
(*k*) Lime plants;
(*l*) Phosphate rock processing plants;
(*m*) Coke oven batteries;
(*n*) Sulfur recovery plants;
(*o*) Carbon black plants (furnace process);
(*p*) Primary lead smelters;
(*q*) Fuel conversion plants;
(*r*) Sintering plants;
(*s*) Secondary metal production plants;
(*t*) Chemical process plants;
(*u*) Fossil-fuel boilers (or combination thereof) totaling more than 250 million British thermal units per hour heat input;
(*v*) Petroleum storage and transfer units with a total storage capacity exceeding 300,000 barrels;
(*w*) Taconite ore processing plants;
(*x*) Glass fiber processing plants;
(*y*) Charcoal production plants;
(*z*) Fossil fuel-fired steam electric plants of more that 250 million British thermal units per hour heat input;
(*aa*) Any other stationary source category which, as of August 7, 1980, is being regulated under Section 111 or 112 of the Act."

*B. New Source Review for PSD Purposes*

Section 52.21 of Title 40 of the Code of Federal Regulations is amended as follows:

1. By adding a new paragraph (b)(1)(iii) to read as follows: "(iii) The fugitive emissions of a stationary source shall not be included in determining for any of the purposes of this section whether it is a major stationary source, unless the source belongs to one of the following categories of stationary sources:

(*a*) Coal cleaning plants (with thermal dryers);
(*b*) Kraft pulp mills;
(*c*) Portland cement plants;
(*d*) Primary zinc smelters;
(*e*) Iron and steel mills;
(*f*) Primary aluminum ore reduction plants;
(*g*) Primary copper smelters;
(*h*) Municipal incinerators capable of charging more than 250 tons of refuse per day;
(*i*) Hydrofluoric, sulfuric, or nitric acid plants;
(*j*) Petroleum refineries;
(*k*) Lime plants;
(*l*) Phosphate rock processing plants;
(*m*) Coke oven batteries;
(*n*) Sulfur recovery plants;
(*o*) Carbon black plants (furnace process);
(*p*) Primary lead smelters;
(*q*) Fuel conversion plants;
(*r*) Sintering plants;
(*s*) Secondary metal production plants;
(*t*) Chemical process plants;
(*u*) Fossil-fuel boilers (or combination thereof) totaling more than 250 million British thermal units per hour heat input;
(*v*) Petroleum storage and transfer units with a total storage capacity exceeding 300,000 barrels;
(*w*) Taconite ore processing plants;
(*x*) Glass fiber processing plants;
(*y*) Charcoal production plants;
(*z*) Fossil fuel-fired steam electric plants of more that 250 million British thermal units per hour heat input." and
(*aa*) Any other stationary source category which, as of August 7, 1980, is being regulated under Section 111 or 112 of the Act."

*C. State Plans for New Source Review for Nonattainment Purposes*

Section 51.18 of Title 40 of the Code of Federal Regulations is amended as follows:

1. By adding a new paragraph (j)(l)(iv)(*c*) to read as follows: "(*c*) The fugitive emissions of a stationary source shall not be included in determining for any of the purposes of this subsection whether it is a major stationary source, unless the source belongs to one of the following categories of stationary sources:

(*1*) Coal cleaning plants (with thermal dryers);
(*2*) Kraft pulp mills;
(*3*) Portland cement plants;
(*4*) Primary zinc smelters;
(*5*) Iron and steel mills;
(*6*) Primary aluminum ore reduction plants;
(*7*) Primary copper smelters;
(*8*) Municipal incinerators capable of charging more than 250 tons of refuse per day;
(*9*) Hydrofluoric, sulfuric, or nitric acid plants;
(*10*) Petroleum refineries;
(*11*) Lime plants;
(*12*) Phosphate rock processing plants;
(*13*) Coke oven batteries;
(*14*) Sulfur recovery plants;
(*15*) Carbon black plants (furnace process);
(*16*) Primary lead smelters;
(*17*) Fuel conversion plants;
(*18*) Sintering plants;
(*19*) Secondary metal production plants;
(*20*) Chemical process plants;
(*21*) Fossil-fuel boilers (or combination thereof) totaling more than 250 million British thermal units per hour heat input;
(*22*) Petroleum storage and transfer units with a total storage capacity exceeding 300,000 barrels;
(*23*) Taconite ore processing plants;
(*24*) Glass fiber processing plants;
(*25*) Charcoal production plants;
(*26*) Fossil fuel-fired steam electric plants of more than 250 million British thermal units per hour heat input;
(*27*) Any other stationary source category which, as of August 7, 1980, is being regulated under Section 111 or 112 of the Act."

*D. Emission Offset Interpretative Ruling*

Appendix S of Part 51 of Title 40 of the Code of Federal Regulations is amended as follows:

1. By adding a new paragraph II.A.4(iii) to read as follows: "(iii) The fugitive emissions of a stationary source shall not be included in determining for any of the purposes of this ruling whether it is a major stationary source, unless the source belongs to one of the following categories of stationary sources:

(*a*) Coal cleaning plants (with thermal dryers);
(*b*) Kraft pulp mills;
(*c*) Portland cement plants;
(*d*) Primary zinc smelters;
(*e*) Iron and steel mills;
(*f*) Primary aluminum ore reduction plants;
(*g*) Primary copper smelters;
(*h*) Municipal incinerators capable of charging more than 250 tons of refuse per day;
(*i*) Hydrofluoric, sulfuric, or nitric acid plants;
(*j*) Petroleum refineries;
(*k*) Lime plants;
(*l*) Phosphate rock processing plants;
(*m*) Coke oven batteries;
(*n*) Sulfur recovery plants;
(*o*) Carbon black plants (furnace process);
(*p*) Primary lead smelters;
(*q*) Fuel conversion plants;
(*r*) Sintering plants;
(*s*) Secondary metal production plants;
(*t*) Chemical process plants;
(*u*) Fossil-fuel boilers (or combination thereof) totaling more than 250 million British thermal units per hour heat input;
(*v*) Petroleum storage and transfer units with a total storage capacity exceeding 300,000 barrels;
(*w*) Taconite ore processing plants;
(*x*) Glass fiber processing plants;
(*y*) Charcoal production plants;
(*z*) Fossil fuel-fired steam electric plants of more than 250 million British thermal units per hour heat input;
(*aa*) Any other stationary source category which, as of August 7, 1980, is being regulated under Section 111 or 112 of the Act."

*E. Restrictions on Construction for Nonattainment Areas*

Section 52.24 of Title 40 of the Code of Federal Regulations is amended as follows:

1. By adding a new paragraph (f)(4)(iii) to read as follows: "(iii) The fugitive emissions of a stationary source shall not be included in determining for any of the purposes of this section whether it is a major stationary source, unless the source belongs to one of the following categories of stationary sources:

(*a*) Coal cleaning plants (with thermal dryers);
(*b*) Kraft pulp mills;
(*c*) Portland cement plants;
(*d*) Primary zinc smelters;
(*e*) Iron and steel mills;
(*f*) Primary aluminum ore reduction plants;
(*g*) Primary copper smelters;
(*h*) Municipal incinerators capable of charging more than 250 tons of refuse per day;
(*i*) Hydrofluoric, sulfuric, or nitric acid plants;
(*j*) Petroleum refineries;
(*k*) Lime plants;
(*l*) Phosphate rock processing plants;
(*m*) Coke oven batteries;
(*n*) Sulfur recovery plants;
(*o*) Carbon black plants (furnace process);
(*p*) Primary lead smelters;
(*q*) Fuel conversion plants;
(*r*) Sintering plants;
(*s*) Secondary metal production plants;
(*t*) Chemical process plants;
(*u*) Fossil-fuel boilers (or combination thereof) totaling more than 250 million British thermal units per hour heat input;
(*v*) Petroleum storage and transfer units with a total storage capacity exceeding 300,000 barrels;
(*w*) Taconite ore processing plants;
(*x*) Glass fiber processing plants;
(*y*) Charcoal production plants;
(*z*) Fossil fuel-fired steam electric plants of more than 250 million British thermal units per hour heat input;
(*aa*) Any other stationary source category which, as of August 7, 1980, is being regulated under Section 111 or 112 of the Act."

[FR Doc. 84-28197 Filed 10-25-84; 8:45 am]
BILLING CODE 6560-50-M

**ENVIRONMENTAL PROTECTION AGENCY**

**40 CFR Parts 51 and 52**

**[AD-FRL 2683-1a]**

## Requirements for Preparation, Adoption, and Submittal of Implementation Plans; Approval and Promulgation of Implementation Plans

**AGENCY:** Environmental Protection Agency (EPA).

**ACTION:** Proposal of amendments to regulations.

**SUMMARY:** The various EPA regulations that govern new construction under the Clean Air Act (the "Act") generally apply to "major" new stationary sources of air pollution and "major" modifications of existing sources. They require fugitive emissions to be included in threshold applicability determinations of whether a new project is "major," but only for 30 categories of sources. EPA here proposes to add surface coal mines to the current list of 30 categories and reopens the comment period on the current list. EPA is also soliciting public comment on an interpretive ruling regarding section 302(j) of the Act, 42 U.S.C. 7602(j), as it relates to the review of modifications involving fugitive emissions. In a parallel notice, EPA is announcing its reaffirmation of the current list and the legal interpretation behind it.

**DATES:** The period for initial comment on the addition of surface coal mines to the current list, on the list itself, and on EPA's legal interpretation of Section 302(j) with respect to modification closes on December 26, 1984. A public hearing on these items will be held in Washington, D.C., on December 3, 1984, and in Denver, Colorado, on December 7, 1984. EPA will hold the public docket for this rulemaking open for 30 days after the close of the initial comment period for the submission of written rebuttal and supplementary information.

**ADDRESSES:** Comments should be submitted (in triplicate, if possible) to: Central Docket Section (LE-131A), Environmental Protection Agency, 401 M Street, S.W., Washington, D.C. 20460, Attention: Docket No. A-84-33.

Public Hearing: Room 2126/2409, Waterside Mall, 401 M Street, S.W., Washington, D.C. and EPA Regional Administrator's Conference Room, 9th Floor, 1860 Lincoln Street, Denver, Colorado.

Docket: EPA has established a docket for this rulemaking, Docket No. A-84-33, in accordance with section 307(d) of the Clean Air Act, 42 U.S.C. 7607(d). The docket is available for public inspection and copying between 8:00 a.m. and 4:00 p.m., Monday through Friday, at EPA's Central Docket Section, West Tower Lobby, Gallery I, 401 M Street, S.W., Washington, D.C. A reasonable fee may be charged for copying.

**FOR FURTHER INFORMATION CONTACT:** Kirt Q. Cox, New Source Review Section, Office of Air Quality Planning and Standards, Research Triangle Park, North Carolina 27711, (919-541-5591, FTS-629-5591).

**SUPPLEMENTARY INFORMATION:**

### I. Background

In 1980, EPA overhauled its regulations governing the construction of new stationary sources of air pollution and modifications to existing sources in response to *Alabama Power Company* v. *Costle*, 636 F.2d 323 (D.C. Cir. 1979) [*see* 45 FR 52676 (August 7, 1980)]. The various regulations [1] that emerged from the overhaul apply generally to "major stationary sources" and "major modifications." In addition, they require "fugitive emissions" [2] to be included in threshold applicability determinations of whether a project is "major," but only for 30 source categories.[3] Those categories do not include surface coal mines. EPA said that it would decide at a future time whether to add such mines to the list of 30 categories. The effect of this postponement was to exempt at least temporarily most surface coal mines from the substantive requirements of the relevant PSD and nonattainment regulations.

In requiring the inclusion of fugitive emissions for 30 source categories, EPA took account of section 302(j) of the Clean Air Act, 42 U.S.C. 7602(j), which provides:

> Except as otherwise expressly provided, the terms "major stationary source" and "major emitting facility" mean any stationary facility or source of air pollutants which directly emits, or has the potential (including any major emitting facility or source of fugitive emissions of any such pollutant, as determined by rule by the Administrator).

The D.C. Circuit, in *Alabama Power*, held in light of this section that EPA may require the inclusions of fugitive emissions in threshold applicability determinations for a particular source only if it has first established through rulemaking that fugitives are to be included for that class of sources. The court did not specifically list what factors it thought EPA had to consider in such a rulemaking. EPA took the position that the section obliges it, in proposing to require inclusion for a particular category of sources, to determine at least that the sources pose a significant threat of air quality degradation and then, in taking final action, to consider, if commenters objected to the proposal, whether inclusion for the category would produce unreasonable social and economic impacts relative to the anticipated benefits. EPA added that the adequacy of methods of measuring and modeling the fugitive emissions was not by itself a decisive factor in determining whether to require inclusion, suggesting that the potential of social or economic disruption and damage to national security would have much greater weight (*see generally* 44 FR 52690-92).

Various industry and environmental groups, including the Sierra Club, petitioned the D.C. Circuit to review the 1980 regulations, including the provisions relating to fugitive emissions. Industry contended that EPA, in requiring the inclusion of fugitive emissions for the 30 categories, had failed to conduct the rulemaking the court had contemplated. The Sierra Club contended that EPA, in postponing a decision on strip mines, had acted arbitrarily and capriciously. The court consolidated the industry challenges under *Chemical Manufacturers Association (CMA)* v. *EPA*, No. 79-1112, and kept the Sierra Club challenge on its own track.

In 1982, EPA entered into a settlement agreement with the industry petitioners in the *CMA* case. EPA committed to propose, among other things, to delete the requirement for including fugitive emissions for the 30 categories and to take final action on the proposal. The court stayed the litigation pending implementation of the agreement.

On August 25, 1983, 48 FR 38742, EPA proposed to delete that requirement. EPA stated that it had concluded preliminarily that its original

---

[1] One set, 40 CFR 51.24, specifies the minimum requirements that a prevention of significant deterioration (PSD) permit program must contain to warrant approval by EPA as a revision to a State implementation plan (SIP). Another set, 40 CFR 52.21, delineates the Federal PSD permit program, which currently applies in a majority of the States as part of their SIP's. Another set, 40 CFR 51.18(j), specifies the elements of an approvable state permit program for reconstruction review for nonattainment purposes. A fourth set, 40 CFR Part 51, Appendix S, embodies the "Emissions Offset Interpretative Ruling" for certain nonattainment situations. A fifth set, 40 CFR 52.24, embodies the construction moratorium for certain nonattainment areas. A sixth set, 40 CFR 51.18(k), embodies the basic requirement of section 110(a)(2)(D) that each SIP contain a permit program for "major" projects to assure they will not interfere with timely attainment and maintenance of the national ambient air quality standards (NAAQS).

[2] "Fugitive emissions" are emissions that would not pass through a stack or other functionally equivalent opening [*e.g.*, 40 CFR 52.21(b)(20) (1984)].

[3] *See, e.g.*, 40 CFR 52.21 (b)(4), (i)(4)(vii) (1984).

interpretation of section 302(j) was wrong.

In a **Federal Register** notice that parallels this one, EPA is announcing a final action on that proposal which reaffirms the current requirements. EPA states that it has concluded that its original interpretation was correct after all. Thus, EPA continues to view section 302(j) as requiring it to determine only that (1) the sources in a category could degrade air quality significantly, and (2) no factors raised by commenters during the comment period indicate that listing the category would produce unreasonable social and economic impacts when compared to the benefits of listing. In performing rulemaking for a particular source catagory, the Agency regards the first determination as establishing a presumption in favor of listing the category that could be rebutted only by an analysis which proves that unreasonable costs would result taking into account all positive aspects of the listing. The reader should see the companion notice for a more detailed explanation of this "safety valve" interpretation of Section 302(j) and the reaffirmation of the current listing.

Meanwile, on August 26, 1983, in the *Sierra Club* case, the D.C. Circuit remanded EPA's postponement of action on strip mines, ruling that EPA under the logic of its 1980 action appeared to have no good reason for listing the 30 categories but not strip mines (715 F. 2d 653). In EPA's response to the remand, it agreed with that ruling and committed to propose to list or not list strip mines. The court then ordered EPA to propose one way or the other by October 22, 1984.

## II. Proposal to List Surface Coal Mines

EPA has concluded on the basis of various studies [4] that surface coal mines do pose a threat of significant air quality degradation. Most such mines of an economically viable size emit in excess of 250 tons per year, the volume of emissions that Congress found significant in section 169(1) of the Act, 42 U.S.C. 7479(1), for sources outside the 28 categories listed in that section. EPA therefore proposed to add surface coal mines to the current list of 30 categories of sources whose fugitive emissions are to be included in threshold applicability determinations. EPA solicits comments on this proposal.[5]

EPA fully expects that weighty objections to the listing of surface coal mines will be raised. EPA expects these to include claims that such mines simply cannot meet PSD requirements if they are listed, and that to prevent the opening of new surface mines by subjecting them to PSD requirements would not serve either the cause of a sound energy policy or the broader national interest. Moreover, surface mines differ from many other sources of pollution in that they must be located where the mineral deposit on which they depend is found, and therefore have limited geographical mobility.

The fact that EPA is proposing to list surface coal mines does not mean that it has determined that these factors lack merit, or that it is in some way unlikely to give weight to them at the time of final promulgation. Rather, as today's companion notice makes clear, EPA believes that the "safety valve" function of section 302(j) is best served if these broader issues are first raised in public comments and thereafter considered by EPA in making its final decision.

Because of particular concern with longstanding problems arising from the regulation of surface mining and the requirements of Executive Order 12291, EPA is independently preparing an analysis of the effects of adding strip mines to the current list.[6] EPA intends to make the results of the study available to the public for comment before the Agency takes final action on today's proposal.

EPA is analyzing the nature of the surface coal mining industry and is assembling information regarding the size, number, location, and lifespan of mines, and the rate, size and location of industry growth. After addressing the population of mines that could be subject to PSD permitting requirements, an analysis will be prepared of the costs of PSD review to them. This includes the cost of preparing a permit application and of applying best available control technology to mine operations. Most significantly, the study is addressing the costs that would accrue to the mining industry as a result of the constraints from the PSD increments. Analysis is also being prepared on the degree of emissions reductions that the increments and NAAQS might require and the estimated benefits to public health and welfare that would arise from it.

This study will also address potential impact on Class I areas. Protection of Class I areas, such as national parks, has long been a special concern and may present a rationale for regulation of mines which would affect them that is considerably stronger than it is for mines locating in order areas. EPA therefore solicits comments on the possibility of restricting the applicability of the relevant regulations to those surface coal mines that would affect Class I areas and possibly the mandatory Class II areas [7] as well. One issue is whether section 302(j) provides authority to list only a subclass of surface coal mines. EPA is inclined to conclude that it does. Another issue is whether the balance of benefits and costs to society justify a distinction along those lines.

In addition, EPA also solicits comment on whether the existing strip mines that the proposed listing would render "major" should consume PSD increment. Section 169(4) of the Act provides that all major emitting facilities on which construction commenced after January 6, 1975, are not to be included in the baseline concentration and instead consume the available PSD increment. A listing of surface coal mines pursuant to section 302(j) would cause many of them to be classified as major sources. This raises the issue whether section 169(4) would require all large mines that commenced construction (even though not then as "major" stationary sources) since January 6, 1975, but prior to today, to retroactively consume increment instead of continuing to contribute to baseline concentration. EPA does not believe that the Congress intended such a disruption to occur. Instead, the Agency believes that it should "grandfather" existing mines from retroactive consumption of increment but seeks public comment on this matter. The effects of not doing so will be addressed to a limited degree in EPA's study.

---

[4] Characterization of $PM_{10}$ and TSP Air Quality Around Western Surface Coal Mines, PEDCo Environmental, Inc., and TRC Environmental Consultants [EPA Contract No. 68-02-3512 (August 1982)].

[5] EPA is obligated to propose visibility new source review SIP's for numerous States under a statement agreement in EDF v. EPA, N.D. Calif. No. C82-6850 RPA. EPA anticipates that such SIP's will contain similar lists of source categories whose fugitive emissions would be included in threshold applicability determinations. EPA also proposed to add surface coal mines to those lists if and when these are promulgated. EPA solicits comment on such an action at this time.

[6] As EPA stated in the companion notice, it does not view section 302(j) as barring the preparation of a cost/benefit analysis prior to a proposal or final action under the section, but rather as allowing EPA not to prepare one. Under normal circumstances, EPA would have prepared such an analysis under E.O. 12291 for the strip mines proposal here. EPA is constrained by the court ordered deadline of October 22, 1984, to propose action on strip mines without having completed such an analysis. Nevertheless, that analysis will be completed prior to final action. Moreover, for future proposals as to other source categories, if any, EPA intends to adhere to the applicable requirements for cost benefit analysis under the Executive Order.

[7] Such areas are described in section 164(a) of the Act, 42 U.S.C. 7474(a).

Any grandfathering from increment consumption granted must be reasonably bounded. The Agency proposes that this exemption be granted to all mines which have commenced construction prior to the date of final action on this notice. "Commencement of construction" is the key term. For the pruposes of this action, EPA suggests that the term includes the receipt of all necessary permits plus certain other activities evidencing a commitment to the project. These may include on-site activity or site specific contracts. Because of the location specific nature of mining permits and possibility that factors other than those applicable to a conventional industrial source may be germaine, EPA solicits comment on an appropriate policy for addressing the potential grandfathering of mines within the presently applicable definitions of commencement of construction. EPA also proposes to extend an exemption from PSD permitting requirements to these same sources. Today's notice contains proposed regulations to accomplish both of these grandfathering actions. Public comment is also solicited on these proposed regulations.

### III. Reopening of Comment Period on the List of Thirty Categories

As explained in detail in the companion notice, although EPA has reaffirmed the list of 30 source categories and the underlying statutory interpretation, it recognizes it could have provided a fuller opportunity to comment on the original listing. Therefore, as a policy matter, EPA is reopening the comment period for the current list of 30 categories, and solicits comments on that list.

Consistent with its "safety valve" interpretation of Section 302(j), however, EPA is inclined not to remove a category from the list unless a commenter shows adverse consequences to the board national interest in continuing the listing.

### IV. Nonapplicability of Section 302(j) to Modifications

In overhauling its new source review regulations in 1980 and in proposing further amendments in 1983, EPA assumed that the rulemaking requirement in section 302(j) applies to modifications as well as to sources. The litigants and commenters have all carried that assumption into their communications, without evidencing any examination of it. Drawn by the latest round of proposal and comment into examining the assumption, EPA has concluded that it appears to be incorrect. This section of this notice outlines the reasons for that conclusion and solicits comment on it. After evaluating any comments, EPA may translate the conclusion directly without further comment into regulatory amendments.[8]

The literal language of the Act strongly suggests that Congress did not intend the rulemaking requirement in section 302(j) to apply to modifications. First, section 302(j) on its face applies only to new and existing emissions units and, hence, not to modifications. It provides that:

> the terms "major stationary source" and "major emitting facility" mean any *stationary facility or source* [that has a certain pollution potential] (including any major emitting facility or source of fugitive emission * * *, as determined by rule by the Administrator). [42 U.S.C. 7602(j) (emphasis added)].

Plainly, this language explicitly attached the rulemaking requirement only to proposed or actual units; it says nothing about modifications to such units.

In contrast, Parts C and D, the provisions that detail the applicability and content of the PSD and nonattainment new source review program, expressly define the term "modification" by reference to a parallel definition which appears to require the inclusion of fugitive emissions in threshold applicability determinations for modifications. Section 169(2)(C), 42 U.S.C. 7479(2)(C), states, for purposes of Part C, that:

> [t]he term "construction" when used in connection with any source or facility, includes the modification (*as defined in section 111(a) of this title*) of any source or facility. [Emphasis added.]

Similarly, section 171(4), 42 U.S.C. 7501(4), provides, for purposes of both Part D and the construction ban in section 100(a)(2)(I), that:

> [t]he terms "modification" and "modified" mean the same as the term "modification" *as used in section 111(a)(4) of this title.* [Emphasis added.]

Section 111(a)(4), 42 U.S.C. 7411(a)(4), in turn provides that:

> [t]he term "modification" means any physical change in, or change in the method of operation of, a stationary source *which increases the amount of any air pollutant emitted by such source or which results in the emissions of any air pollutant not previously emitted.* [Emphasis added.]

In defining "modification" solely in terms of the total amount of pollution that a source change would produce, section 111(a)(4) suggests that Congress intended to establish here no qualitative distinction between emissions, and, hence, to require the inclusion of fugitive emissions for modifications, without any intermediate rulemaking step.

The legislative history strongly supports this view that Congress intended the section 302(j) rulemaking requirement not to apply to modifications. First, the passages in the relevant House and conference reports that focus on section 302(j) refer, as does section 302(j), only to proposed and existing emissions units, and not at all to modifications [*see* H.R. Report No. 95–294, 95th Cong., 1st Sess. 4, 9, 144 (1977); H.S. Rep. No. 95–564, 95th Cong., 1st Sess. 172 (1977)]. In addition, the explanation by the conference committee of section 169(2)(C) states that it:

> [i]mplements conference agreement to cover "modification" as well as "construction" by defining "construction" in Part C *to conform to usage in other parts of the Act.* [123 Cong. Rec. H 11957, col. 3 (daily ed.) (November 1, 1977) (emphasis added).]

The phrase "usage in other parts of the Act" most probably refers, not only to section 111(a)(4), but also to the EPA regulations implementing section 111 that were in effect at the time. Like section 111(a)(4), those regulations on their face require the inclusion of fugitive emissions in section 111 applicability determination, inasmuch as they concern themselves only with the quantity of the emissions in question [*see* 40 CFR 60.14(a) (1977)]. Finally, EPA and the States prior to enactment of section 302(j) in 1977 made no distinction between fugitive and nonfugitive emissions in threshold applicability determinations [*see, e.g.*, 40 CFR 51.18 (1977); 41 FR 55528 (December 21, 1976) (the original Offset Ruling), 40 CFR 51.21(d)(1) (1977) (original PSD regulations)]. Section 302(j), therefore, ran against the grain of longstanding practice. If Congress had intended a change as to modifications, it probably would have said so explicitly, yet it said nothing.

EPA's new interpretation as to the applicability of the section 302(j) rulemaking requirement is consistent with Congress' purposes. Requiring fugitive emissions to be included in threshold applicability determinations for modifications would not frustrate the protections from the burdens of new source review that the rulemaking requirement affords. Under the Act and EPA regulations, a modification is

[8] Specifically, EPA would not promulgate the amendment to the definition of "major modification" that was proposed on August 25, 1983, namely those proposed as §§ 51.24(b)(2)(iv), 52.21(b)(2)(iv), 51.18(j)(1)(v)(d), II.A.5(iv) of Appendix S to Part 51, and 52.24(f)(5)(iv). EPA would also add a provision to the relevant regulations that would "grandfather" any modifications that (1) would not have been subject to review but for the deletion of those amendments and (2) commenced construction before the final deletion.

"major" and subject to review only if the source at which it would occur is also "major" [*see, e.g.*, 40 CFR 52.21(b)[2](i) (1984)]. Hence, a modification to a source of predominantly fugitive emissions that does not belong to a currently listed category, for instance a surface coal mine, could not be subject to review, even if its fugitive emissions were taken into account, because the source would not be "major". In addition, requiring fugitive emissions to be included in threshold applicability determinations would promote the Congressional goal of minimizing unnecessary burdens. As discussed at length in the preamble to the 1983 proposal and in the comments (*see, e.g.*, 48 FR 38746), the exclusion of fugitive emissions decreases, as well as increases, in threshold applicability determinations for modifications can result in a company having to obtain a permit, but not having to meet any substantive requirements to obtain it. The universal inclusion of fugitive emissions increases and decreases would cure this problem of an empty proceeding. Finally, universal inclusion removes any incentive a company would otherwise have to artifically sequence the construction of a group of modifications so that the modifications that produce only fugitive emissions might escape review on the theory that they are separate and not "major."

## V. Miscellaneous

### *A. Docket*

EPA has established a docket for this rulemaking, Docket No. A–84–33. The docket is an organized and complete file of all significant information submitted to or otherwise considered by EPA during this proceeding. The contents of the docket will serve as the record in the case of judicial review under section 307(b) of the Act, 42 U.S.C. 7607(b). The docket is available for public inspection and copying between 8:00 a.m. and 4:00 p.m., Monday through Friday, at EPA's Central Docket Section, West Tower Lobby, Gallery I, 401 M Street, S.W., Washington, D.C. A reasonable fee may be charged for copying.

### *B. Public Hearing*

EPA will hold a public hearing on December 4, 1984, at 10:00 a.m., in Room 2126/2409, Waterside Mall, 401 M Street, S.W., Washington, D.C., and on December 6, 1984, at 10:00 a.m., in EPA Regional Administrator's Conference Room, 9th Floor, 1860 Lincoln Street, Denver, Colorado. The hearings will be informal. A panel of EPA staff will hear the oral presentations. There will be no cross-examination and no requirement that any person be under oath. Each member of the panel may seek clarification or amplification of any presentation. The presiding officer of the panel may set a time limit for each presentation and may restrict any presentation that would be irrelevant or repetitious. A transcript of each hearing will be made and placed in the rulemaking docket.

Any person who wishes to speak at a hearing should as soon as possible send written notice of this to EPA, give name, address, telephone number, and the length of the presentation. Anyone stating that his or her presentation would be longer than 20 minutes should also state why it need be longer. Each notice should be sent to Kirt Q. Cox, at the address given at the beginning of this notice. EPA will develop a schedule for presentations based on the notices it receives. Anyone who fails to submit a notice, but wishes nevertheless to speak at the hearing should so notify the presiding officer immediately before the hearing. The presiding officer will decide whether, when, and for how long the person may speak. Each speaker should bring extra copies of his or her presentation for the convenience of the hearing panel, the hearing reporter, the press, and other participants. The hearings will be open to the public.

### *C. Executive Order 12291*

Under Executive Order 12291, EPA must prepare a regulatory impact analysis for any regulation that qualifies as a "major rule." A major rule is defined as any regulation that is likely to result in an annual effect on the economy of $100 million or more. EPA has determined that the proposal to list strip mines might have an annual effect on the economy of $100 million or more, and EPA is therefore preparing the economic analysis described above in section II. EPA has determined, on the other hand, that the proposed new interpretation of section 302(j) as it relates to modifications will not have sufficient economic effect to be considered a major rule. The new interpretation will not require large numbers of additional modifications to obtain permits because there are relatively few modifications involving fugitive emissions at major sources other than those sources already on the current list of 30 source categories. Those modifications must presently obtain permits under the existing regulations; therefore, EPA is not preparing a regulatory impact analysis for this portion of the rulemaking.

These regulations have been submitted to the Office of Management and Budget for review under Executive Order 12291. Any comments from the office, and EPA's responses to any such comments, will be placed in the docket for this proceeding and are available for public inspection at the times and place described earlier in this preamble.

### *D. Economic Impact Assessment*

Section 317 of the Clean Air Act, 42 U.S.C. 7617, requires EPA to prepare an economic impact assessment in connection with proposal of any substantial revisions to existing regulations. The economic analysis concerning strip mines that EPA is preparing in order to comply with Executive Order 12291 will also satisfy the requirement for preparation of an economic impact assessment on the proposal to list strip mines. This analysis will be available to the public within the next few months. The comment period on these proposed regulations will be provided to allow the public sufficient time to comment on the analysis. EPA will not be preparing an economic analysis on the new interpretation of section 302(j) as it relates to modifications because this is not a substantial revision. As discussed above, this interpretation will not bring many additional modifications under review.

### *E. Regulatory Flexibility Act Certification*

As required by section 3(a) of the Regulatory Flexibility Act, 5 U.S.C. 605(b), I certify that this regulation will not have a significant adverse impact on a substantial number of small entities. The proposal to list strip mines will primarily effect large mining operations. The proposed new interpretation of section 302(j) as it relates to modifications will not bring a large number of additional modifications into the permitting system.

### *F. Paperwork Reduction Act*

The Office of Management and Budget (OMB) has approved some of the information collection requirements contained in this proposed rule under the provisions of the Paperwork Reduction Act of 1980, 44 U.S.C. 3501 *et seq.* and has assigned OMB control number 2060–0003. This proposed rule also introduces additional requirements, and these have been transmitted to OMB. Comments on these requirements should be submitted to the office of Information and Regulatory Affairs of OMB, marked "Attention: Desk Officer for EPA." The final rule will respond to any OMB or public comments on the information collection requirements.

List of Subjects

*40 CFR Part 51*

Administrative practice and procedures, Air pollution control, Intergovernmental relations, Reporting and recordkeeping requirements, Ozone, Sulfur Oxides, Nitrogen dioxide, Lead, particulate matter, Hydrocarbon, Carbon monoxide.

*40 CFR Part 52*

Air pollution control, Ozone, Sulfur oxides, Nitrogen dioxide, Lead, Particulate matter, Carbon monoxide, Hydrocarbons.

Authority: Sections 101(b)(1), 160–169, 171–178, and 301(a) of the Clean Air Act as amended [42 U.S.C. 7401(b)(1), 7410, 7470–79, 7501–08 and 7601(a)]; Section 129(a) of the Clean Air Act Amendments of 1977 [Pub. L. No. 95–95, 91 Stat. 685 (August 7, 1977)].

Dated: October 19, 1984.

Alvin L. Alm,

*Deputy Administrator.*

Title 40 of the Code of Federal Regulations, as amended, is proposed to be amended, as follows:

## PARTS 51 AND 52—[AMENDED]

### A. Requirements for State PSD Plans

**§ 51.24 [Amended]**

Section 51.24 of Title 40 of the Code of Federal Regulations is proposed to be amended as follows:

1. By redesignating paragraph (aa) of § 51.24(b)(1)(iii), as (bb) to and adding a new paragraph (aa) to read as follows: "(aa) Surface coal mines,";

2. By adding a new paragraph (b)(13)(iii) to read as follows: "(iii) The actual emissions of coal surface mines on which construction commenced prior to [the date that final action for the listing of surface coal mines is published in the **Federal Register**] will not affect the applicable maximum allowable increase, unless such emissions occurred after an applicable baseline date has been established for the affected area."; and

3. By adding a new paragraph (i)(4)(iv) to read as follows: "(iv) The source or modification is a surface coal mine otherwise qualifying as a new or modified major stationary source under this section if the owner or operator:

(*a*) Obtained all final federal, state, and local preconstruction approvals or permits necessary under the applicable State implementation plan before [the date that final action for the listing of surface coal mines is published in the **Federal Register**];

(*b*) Commenced construction within 18 months from [the date that final action for the listing of surface coal mines is published in the **Federal Register**], or any earlier time required under the State implementation plan; and

(*c*) Did not discontinue construction for a period of 18 months or more and completed construction within a reasonable time."

### B. New Source Review for PSD Purposes

**§ 52.21 [Amended]**

Section 52.21 of Title 40 of the Code of Federal Regulations is proposed to be amended as follows:

1. By redesignating paragraph (aa) of § 52.21(b)(1)(iii), as (bb) and adding a new paragraph (aa) to read as follows: "(aa) Surface coal mines,";

2. By adding a new paragraph (b)(13)(iii) to read as follows: "(iii) The actual emissions of coal surface mines on which construction commenced prior to [the date that final action for the listing of surface coal mines is published in the **Federal Register**] will not affect the applicable maximum allowable increase, unless such emissions occurred after an applicable baseline date has been established for the affected area."; and

3. By adding a new paragraph (i)(4)(ix) to read as follows: "(ix) The source or modification is a surface coal mine otherwise qualifying as a new or modified major stationary source under this section if the owner or operator:

(*a*) Obtained all final federal, state, and local preconstruction approvals or permits necessary under the applicable State implementation plan before [the date that final action for the listing of surface coal mines is published in the **Federal Register**];

(*b*) Commenced construction within 18 months from [the date that final action for the listing of surface coal mines is published in the **Federal Register**], or any earlier time required under the State implementation plan; and

(*c*) Did not discontinue construction for a period of 18 months or more and completed construction within a reasonable time.

### C. State Plans for New Source Review for Nonattainment Purposes

**§ 51.18 [Amended]**

Section 51.18 of Title 40 of the Code of Federal Regulations is proposed to be amended as follows:

1. By redesignating paragraph "(27) of § 51.18(j)(1)(iv)(*c*) as (28) and adding a new paragraph (27) to read as follows: "(27) Surface Coal Mines"; and

2. By adding a new paragraph (j)(6) to read as follows: "(6) Each plan may provide that the requirements of this subsection do not apply to the construction of a surface coal mine otherwise qualifying as a new or modified major stationary source under this section if the owner or operator:

(i) Obtained all final federal, state, and local preconstruction approvals or permits necessary under the applicable State implementation plan before [the date that final action for the listing of surface coal mines is published in the **Federal Register**];

(ii) Commenced construction within 18 months from [the date that final action for the listing of surface coal mines is published in the **Federal Register**], or any earlier time required under the State implementation plan; and

(iii) Did not discontinue construction for a period of 18 months or more and completed construction within a reasonable time.

### D. Emission Offset Interpretative Ruling

**Appendix S [Amended]**

Appendix S of Part 51 of Title 40 of the Code of Federal Regulations is proposed to be amended as follows:

1. By redesignating paragraph (aa) of II.A.4(iii) as (bb) and adding a new paragraph (aa) to read as follows: "(aa) Surface Coal Mines,"; and

2. By adding a new paragraph II.E. to read as follows: "II.E. Section IV.A. of this Ruling shall not apply to the construction of a coal surface mine otherwise qualifying as a new or modified major stationary source under this section if the owner or operator:

(a) Obtained all final federal, state, and local preconstruction approvals or permits necessary under the applicable State implementation plan before [the date that final action for the listing of surface coal mines is published in the **Federal Register**];

(b) Commenced construction within 18 months from [the date that final action for the listing of surface coal mines is published in the **Federal Register**], or any earlier time required under the State implementation plan; and

(c) Did not discontinue construction for a period of 18 months or more and completed construction within a reasonable time.

### E. Restrictions on Construction for Nonattainment Areas

**§ 52.24 [Amended]**

Section 52.24 of Title 40 of the Code of Federal Regulations is proposed to be amended as follows:

1. By redesignating paragraph (aa) of § 52.24(f)(4)(iii) as (bb) and adding a new paragraph (aa) to read as follows: "(aa) Surface Coal Mines,"; and

2. By adding a new paragraph (e) to read as follows: "(e) This section shall not apply to the construction of a surface coal mine otherwise qualifying as a new or modified major stationary source under this section if the owner or operator:

(1) Obtained all final federal, state, and local preconstruction approvals or permits necessary under the applicable State implementation plan before [the data that final action for the listing of surface coal mines is published in the **Federal Register**];

(2) Commenced construction within 18 months from [the date that final action for the listing of surface coal mines is published in the **Federal Register**], or any earlier time required under the State implementation plan; and

(3) Did not discontinue construction for a period of 18 months or more and completed construction within a reasonable time.

[FR Doc. 84-28200 Filed 10-25-84; 8:45 am]
BILLING CODE 6560-50-M

federal register

Friday
October 26, 1984

Part VI

# Department of Transportation

## Urban Mass Transportation Administration

## Fiscal Year 1985 UMTA Formula Grant Apportionments; Notice

## DEPARTMENT OF TRANSPORTATION

### Urban Mass Transportation Administration

### Fiscal Year 1985 UMTA Formula Grant Apportionments

**AGENCY:** Urban Mass Transportation Administration (UMTA), DOT.

**ACTION:** Notice.

**SUMMARY:** The Federal Public Transportation Act of 1982 (Pub. L. 97–424) established a formula grant program for Fiscal Years 1984, 1985 and 1986 under Section 9 and Section 18 of The Urban Mass Transportation Act of 1964, as amended. This Notice provides the Fiscal Year 1985 apportionment of funds to each urbanized area over 200,000 in population, and to State Governors for apportionment to urbanized areas under 200,000 in population and to rural areas.

**FOR FURTHER INFORMATION CONTACT:** Edward R. Fleischman, Chief, Resource Management Division, (202) 426–2053, 400 Seventh Street, SW., Washington, DC. 20590.

**SUPPLEMENTARY INFORMATION:** A program of federal assistance to urban mass transportation systems by means of formula grants for capital and operating assistance was enacted January 6, 1983, under the Federal Public Transportation Act of 1982 (Pub. L. 97–424). The legislation is authorized for Fiscal Years 1984, 1985, and 1986. Funds for Fiscal Year 1985 were appropriated by Pub. L. 98–473.

This Notice, based on Census information and operating and financial data contained in the section 15 Report, provides the Fiscal Year 1985 apportionment of section 9 and section 18 funds for urbanized and nonurbanized areas. Applications for section 9 funds should be submitted in conformance with UMTA Circular 9030.1, published June 27, 1983. Applications for section 18 funds should be submitted in conformance with UMTA Circular 9040.1, published September 26, 1983.

The funds shown in this Notice apportioned to urbanized areas will remain available to be obligated by UMTA to recipients for three (3) fiscal years following Fiscal Year 1985, after which time any unobligated funds will become available for reapportionment under section 9. Funds apportioned to nonurbanized areas will remain available to be obligated by UMTA to recipients for two (2) years following Fiscal Year 1985, after which time any unobligated funds will become available for reapportionment under section 18.

Corrections have been made to the data from certain urbanized areas that were used to compute the Fiscal Year 1984 formula grant apportionments published in the **Federal Register** of November 2, 1983 (48 FR 50656). Differences between corrected apportionments and previously published apportionments have been determined, and necessary adjustments have been made by adding to or subtracting from, as appropriate, the apportionments calculated for Fiscal Year 1985.

For urbanized areas over 200,000 in population, funds apportioned under section 9 are based, in part, on data collected through the section 15 reporting system. In the process of collecing these data, there have been repeated problems with late reports, missing certifications and the inclusion of possible erroneous data in vehicle revenue miles (see UMTA Circular 9030.1 for definitions). The issues are summarized below:

*Late reports.* UMTA regulations require transit agencies to submit section 15 reports within 120 calendar days of the end of their fiscal year. Many agencies have been delinquent in submitting their section 15 report or have submitted incomplete reports. This makes it difficult for UMTA to validate the data and increases the risk of erroneous data being used in the apportionment.

*Independent certification.* All reporters in urbanized areas of 200,000 or more are required to submit with their section 15 reports a certification by an independent auditor of the recordkeeping system and definitions used to collect data used in the Section 9 apportionment formula. When reporters fail to comply with this requirement, it calls into question the validity of the data submitted.

*Deadhead mileage.* Certain agencies have not made the appropriate adjustment between total vehicle mileage and vehicle revenue mileage as required. The difference between these two figures represents deadhead mileage, which is not included in the data used for the apportionment. Those urbanized areas that include deadhead mileage in the vehicle revenue mileage reported receive a greater apportionment than they are entitled to while reducing the apportionments of those reporters who follow the definition of revenue vehicle mileage.

These issues have resulted in problems in the timeliness of UMTA's publication of apportionment data, and in some cases raise questions about the validity of certain submitted data. In this connection, UMTA is planning to issue a notice of proposed rulemaking that would propose penalties to address this problem. These penalties could affect an area's ability to receive apportioned funds for failure to report in a timely manner, provide certifications, or submit data which are in accordance with UMTA definitions.

Issued on: October 17, 1984.

**Ralph L. Stanley,**

*Administrator.*

BILLING CODE 84-28322-M

## FISCAL YEAR 1985 UMTA SECTION 9 FORMULA APPORTIONMENTS
## AMOUNTS APPORTIONED TO URBANIZED AREAS OVER 200,000 POPULATION

| URBANIZED AREA | APPORTIONMENT |
|---|---|
| Akron, Ohio | $ 4,929,697 |
| Albany-Schenectady-Troy, New York | 8,374,270 |
| Albuquerque, New Mexico | 4,312,685 |
| Allentown-Bethlehem-Easton, Pa.-N.J. | 3,537,492 |
| Ann Arbor, Michigan | 2,323,863 |
| Atlanta, Georgia | 25,488,902 |
| Augusta, Georgia-South Carolina | 1,613,077 |
| Austin, Texas | 3,827,942 |
| Bakersfield, California | 2,247,405 |
| Baltimore, Maryland | 27,077,092 |
| Baton Rouge, Louisiana | 2,919,220 |
| Birmingham, Alabama | 4,533,656 |
| Boston, Massachusetts | 67,359,107 |
| Bridgeport, Connecticut | 5,642,655 |
| Buffalo, New York | 10,658,219 |
| Canton, Ohio | 1,917,281 |
| Charleston, South Carolina | 1,349,742 |
| Charlotte, North Carolina | 3,403,943 |
| Chattanooga, Tennessee-Georgia | 2,225,477 |
| Chicago, Illinois-Northwestern Indiana | 181,648,252 |
| Cincinnati, Ohio-Kentucky | 12,524,229 |
| Cleveland, Ohio | 25,322,268 |
| Colorado Springs, Colorado | 2,510,731 |
| Columbia, South Carolina | 1,361,130 |
| Columbus, Georgia-Alabama | 1,846,178 |
| Columbus, Ohio | 10,713,064 |
| Corpus Christi, Texas | 1,846,770 |
| Dallas-Fort Worth, Texas | 21,231,745 |
| Davenport-Rock Island-Moline, Iowa-Illinois | 2,662,927 |
| Dayton, Ohio | 12,563,622 |
| Denver, Colorado | 16,865,486 |
| Des Moines, Iowa | 2,615,319 |
| Detroit, Michigan | 46,240,969 |
| El Paso, Texas | 5,691,747 |
| Fayetteville, North Carolina | 1,257,625 |
| Flint, Michigan | 2,796,941 |
| Fort Lauderdale-Hollywood, Florida | 9,082,408 |
| Fort Wayne, Indiana | 2,331,787 |
| Fresno, California | 3,796,942 |
| Grand Rapids, Michigan | 3,794,004 |
| Greenville, South Carolina | 1,255,915 |
| Harrisburg, Pennsylvania | 2,732,046 |
| Hartford, Connecticut | 7,449,961 |
| Honolulu, Hawaii | 18,311,649 |
| Houston, Texas | 27,272,483 |
| Indianapolis, Indiana | 8,029,277 |
| Jackson, Mississippi | 1,727,578 |
| Jacksonville, Florida | 5,969,070 |
| Kansas City, Missouri-Kansas | 8,523,728 |
| Knoxville, Tennessee | 2,743,631 |
| Lansing, Michigan | 2,631,203 |
| Las Vegas, Nevada | 3,164,311 |
| Lawrence-Haverhill, Mass.-New Hampshire | 2,765,605 |
| Little Rock-North Little Rock, Arkansas | 2,406,903 |
| Lorain-Elyria, Ohio | 994,410 |
| Los Angeles-Long Beach, California | 142,139,674 |
| Louisville, Kentucky-Indiana | 9,456,354 |
| Madison, Wisconsin | $ 4,346,370 |
| Melbourne-Cocoa, Florida | 1,128,920 |
| Memphis, Tennessee-Arkansas-Mississippi | 7,829,691 |
| Miami, Florida | 22,087,236 |
| Milwaukee, Wisconsin | 17,224,234 |
| Minneapolis-St. Paul, Minnesota | 19,784,348 |
| Mobile, Alabama | 1,901,700 |
| Nashville-Davidson, Tennessee | 4,608,676 |
| New Haven, Connecticut | 6,440,623 |
| New Orleans, Louisiana | 14,990,274 |
| Newport News-Hampton, Virginia | 3,164,708 |
| New York, N.Y.-Northeastern New Jersey | 605,064,321 |
| Norfolk-Portsmouth, Virginia | 8,879,475 |
| Ogden, Utah | 2,536,128 |
| Oklahoma City, Oklahoma | 4,148,307 |
| Omaha, Nebraska-Iowa | 6,274,493 |
| Orlando, Florida | 4,271,798 |
| Oxnard-Ventura-Thousand Oaks, California | 3,692,403 |
| Pensacola, Florida | 1,391,021 |
| Peoria, Illinois | 2,285,278 |
| Philadelphia, Pennsylvania-New Jersey | 108,609,372 |
| Phoenix, Arizona | 11,356,875 |
| Pittsburgh, Pennsylvania | 33,979,415 |
| Portland, Oregon-Washington | 17,405,100 |
| Providence-Pawtucket-Warwick, R.I.-Mass. | 13,875,854 |
| Raleigh, North Carolina | 1,871,219 |
| Richmond, Virginia | 5,557,753 |
| Rochester, New York | 7,323,171 |
| Rockford, Illinois | 1,995,552 |
| Sacramento, California | 8,995,978 |
| St. Louis, Missouri-Illinois | 19,985,924 |
| St. Petersburg, Florida | 7,537,603 |
| Salt Lake City, Utah | 8,889,233 |
| San Antonio, Texas | 13,749,546 |
| San Bernardino-Riverside, California | 7,792,108 |
| San Diego, California | 28,129,504 |
| San Francisco-Oakland, California | 103,691,323 |
| San Jose, California | 20,909,604 |
| San Juan, Puerto Rico | 15,507,178 |
| Sarasota-Bradenton, Florida | 2,450,187 |
| Scranton-Wilkes-Barre, Pennsylvania | 3,708,253 |
| Seattle-Everett, Washington | 34,531,593 |
| Shreveport, Louisiana | 2,553,883 |
| South Bend, Indiana-Michigan | 2,366,123 |
| Spokane, Washington | 4,334,020 |
| Springfield-Chicopee-Holyoke, Mass.-Conn. | 5,305,326 |
| Syracuse, New York | 5,231,074 |
| Tacoma, Washington | 6,002,007 |
| Tampa, Florida | 5,540,712 |
| Toledo, Ohio-Michigan | 6,501,565 |
| Trenton, New Jersey-Pennsylvania | 6,854,298 |
| Tucson, Arizona | 5,647,661 |
| Tulsa, Oklahoma | 3,730,245 |
| Washington, D.C.-Maryland-Virginia | 68,252,428 |
| West Palm Beach, Florida | 3,826,276 |
| Wichita, Kansas | 2,739,869 |
| Wilmington, Delaware-New Jersey-Maryland | 4,147,679 |
| Worcester, Massachusetts | 2,795,342 |
| Youngstown-Warren, Ohio | 2,574,154 |

## FISCAL YEAR 1985 UMTA SECTION 9 FORMULA APPORTIONMENTS AMOUNTS APPORTIONED TO AREAS UNDER 200,000 POPULATION

| STATE/URBANIZED AREA | APPORTIONMENT |
|---|---|
| **ALABAMA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | $ 5,779,318 |
| Anniston | 501,775 |
| Auburn-Opelika | 311,952 |
| Decatur | 365,425 |
| Dothan | 320,267 |
| Florence | 490,772 |
| Gadsden | 462,476 |
| Huntsville | 1,031,991 |
| Montgomery | 1,539,604 |
| Tuscaloosa | 755,056 |
| ***Non-urbanized*** | ***1,714,383*** |
| **ALASKA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***1,171,880*** |
| Anchorage | 1,171,880 |
| ***Non-urbanized*** | ***186,550*** |
| **ARIZONA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***504,708*** |
| Yuma, Ariz.-Calif. | 504,708 |
| ***Non-urbanized*** | ***648,105*** |
| **ARKANSAS:** | |
| ***Governor's apportionment for areas 50,000 to 200,00 population*** | ***1,702,972*** |
| Fayetteville-Springdale | 405,109 |
| Fort Smith, Ark.-Okla. | 605,737 |
| Pine Bluff | 555,429 |
| Texarkana, Tex.-Ark. | 136,697 |
| ***Non-urbanized*** | ***1,387,894*** |
| **CALIFORNIA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***17,287,920*** |
| Antioch-Pittsburg | 970,617 |
| Chico | 449,498 |
| Fairfield | 612,068 |
| Hemet | 468,180 |
| Lancaster | 391,909 |
| Modesto | 1,838,887 |
| Napa | 640,717 |
| Palm Springs | 435,084 |
| Redding | 359,436 |
| Salinas | 1,188,955 |
| Santa Barbara | 1,690,384 |
| Santa Cruz | 962,285 |
| Santa Maria | 544,206 |
| Santa Rosa | 1,344,011 |
| Seaside-Monterey | 1,247,299 |
| Simi Valley | 837,491 |
| Stockton | 2,196,533 |
| Visalia | 544,636 |
| Yuba City | 563,490 |
| Yuma, Ariz.-Calif. | 2,234 |
| ***Non-urbanized*** | ***3,138,108*** |
| **COLORADO:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | $ 3,820,739 |
| Boulder | 947,106 |
| Fort Collins | 703,674 |
| Grand Junction | 457,107 |
| Greeley | 672,717 |
| Pueblo | 1,040,135 |
| ***Non-urbanized*** | ***703,095*** |
| **CONNECTICUT:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***16,187,091*** |
| Bristol | 666,121 |
| Danbury, Conn.-N.Y. | 2,602,453 |
| Meriden | 542,368 |
| New Britain | 1,340,061 |
| New London-Norwich | 1,099,013 |
| Norwalk | 2,909,631 |
| Stamford | 3,627,840 |
| Waterbury | 3,399,604 |
| ***Non-urbanized*** | ***638,541*** |
| **DELAWARE:** | |
| ***Non-urbanized*** | ***182,661*** |
| **FLORIDA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***7,987,616*** |
| Daytona Beach | 1,319,668 |
| Fort Myers | 1,048,156 |
| Fort Pierce | 494,377 |
| Fort Walton Beach | 619,468 |
| Gainesville | 881,149 |
| Lakeland | 850,610 |
| Naples | 353,753 |
| Ocala | 352,607 |
| Panama City | 566,425 |
| Tallahassee | 947,939 |
| Winter Haven | 553,464 |
| ***Non-urbanized*** | ***1,866,939*** |
| **GEORGIA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***4,448,850*** |
| Albany | 641,762 |
| Athens | 474,175 |
| Macon | 1,142,323 |
| Rome | 361,518 |
| Savannah | 1,381,376 |
| Warner Robins | 447,696 |
| ***Non-urbanized*** | ***2,303,529*** |
| **HAWAII:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***1,140,229*** |
| Kailua-Kaneohe | 1,140,229 |
| ***Non-urbanized*** | ***222,725*** |

## FISCAL YEAR 1985 UMTA SECTION 9 AND SECTION 18 FORMULA APPORTIONMENTS AMOUNTS APPORTIONED TO AREAS UNDER 200,000 POPULATION—(*Continued*)

| STATE/URBANIZED AREA | APPORTIONMENT |
|---|---|
| **IDAHO:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | $ ***1,654,671*** |
| Boise City | 1,194,830 |
| Pocatello | 459,841 |
| ***Non-urbanized*** | ***608,682*** |
| **ILLINOIS:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***10,975,638*** |
| Alton | 722,669 |
| Aurora | 1,449,977 |
| Beloit, Wis.-Ill. | 51,766 |
| Bloomington-Normal | 943,789 |
| Champaign-Urbana | 1,357,578 |
| Danville | 473,243 |
| Decatur | 971,748 |
| Dubuque, Iowa-Ill. | 19,745 |
| Elgin | 1,082,924 |
| Joliet | 1,559,194 |
| Kankakee | 639,637 |
| Round Lake Beach | 504,341 |
| Springfield | 1,199,027 |
| ***Non-urbanized*** | ***2,523,246*** |
| **INDIANA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***6,760,551*** |
| Anderson | 614,782 |
| Bloomington | 693,836 |
| Elkhart-Goshen | 697,210 |
| Evansville, Ind.-Ky. | 1,566,186 |
| Kokomo | 635,885 |
| Lafayette-West Lafayette | 993,554 |
| Muncie | 893,351 |
| Terre Haute | 665,747 |
| ***Non-urbanized*** | ***2,327,051*** |
| **IOWA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***3,770,914*** |
| Cedar Rapids | 1,179,170 |
| Dubuque, Iowa-Ill. | 633,129 |
| Iowa City | 501,182 |
| Sioux City, Iowa-Nebr.-S.Dak. | 626,699 |
| Waterloo | 830,734 |
| ***Non-urbanized*** | ***1,596,402*** |
| **KANSAS:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***1,620,996*** |
| Lawrence | 533,689 |
| St. Joseph, Mo.-Kans. | 8,831 |
| Topeka | 1,078,476 |
| ***Non-urbanized*** | ***1,215,555*** |

| STATE/URBANIZED AREA | APPORTIONMENT |
|---|---|
| **KENTUCKY:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | $ ***3,429,414*** |
| Clarksville, Tenn.-Ky. | 190,232 |
| Evansville, Ind.-Ky. | 205,123 |
| Huntington-Ashland, W.Va.-Ky.-Ohio | 483,991 |
| Lexington-Fayette | 1,871,434 |
| Owensboro | 678,634 |
| ***Non-urbanized*** | ***1,955,756*** |
| **LOUISIANA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***4,005,619*** |
| Alexandria | 711,613 |
| Houma | 462,826 |
| Lafayette | 1,057,102 |
| Lake Charles | 913,826 |
| Monroe | 860,252 |
| ***Non-urbanized*** | ***1,614,353*** |
| **MAINE:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***1,729,170*** |
| Bangor | 367,130 |
| Lewiston-Auburn | 435,028 |
| Portland | 844,076 |
| Portsmouth-Dover-Rochester, N.H.-Maine | 82,936 |
| ***Non-urbanized*** | ***705,181*** |
| **MARYLAND:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***1,517,943*** |
| Annapolis | 549,645 |
| Cumberland, Md.-W.Va. | 440,300 |
| Hagerstown, Md.-Pa. | 527,998 |
| ***Non-urbanized*** | ***866,367*** |
| **MASSACHUSETTS:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***7,565,099*** |
| Brockton | 1,796,287 |
| Fall River, Mass.-R.I. | 1,429,016 |
| Fitchburg-Leominster | 527,785 |
| Lowell, Mass.-N.H. | 1,535,136 |
| New Bedford | 1,546,875 |
| Pittsfield | 403,643 |
| Taunton | 326,357 |
| ***Non-urbanized*** | ***1,040,942*** |
| **MICHIGAN:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***6,731,839*** |
| Battle Creek | 605,859 |
| Bay City | 695,318 |
| Benton Harbor | 509,917 |
| Jackson | 722,463 |
| Kalamazoo | 1,313,226 |
| Muskegon-Muskegon Heights | 865,851 |
| Port Huron | 522,972 |
| Saginaw | 1,496,233 |
| ***Non-urbanized*** | ***2,803,290*** |

## FISCAL YEAR 1985 UMTA SECTION 9 AND SECTION 18 FORMULA APPORTIONMENTS AMOUNTS APPORTIONED TO AREAS UNDER 200,000 POPULATION—(*Continued*)

| STATE/URBANIZED AREA | APPORTIONMENT |
|---|---|
| **MINNESOTA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | $ 2,286,805 |
| Duluth-Superior, Minn.-Wis. | 682,133 |
| Fargo-Moorhead, N.Dak.-Minn. | 323,287 |
| Grand Forks, N.Dak.-Minn. | 76,704 |
| La Crosse, Wis.-Minn. | 32,793 |
| Rochester | 623,722 |
| St. Cloud | 548,166 |
| ***Non-urbanized*** | 1,628,684 |
| **MISSISSIPPI:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | 2,168,968 |
| Biloxi-Gulfport | 1,314,635 |
| Hattiesburg | 400,742 |
| Pascagoula-Moss Point | 453,591 |
| ***Non-urbanized*** | 1,556,077 |
| **MISSOURI:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | 2,673,238 |
| Columbia | 486,432 |
| Joplin | 381,668 |
| St. Joseph, Mo.-Kans. | 627,428 |
| Springfield | 1,177,710 |
| ***Non-urbanized*** | 1,849,814 |
| **MONTANA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | 1,934,407 |
| Billings | 765,275 |
| Great Falls | 667,113 |
| Missoula | 502,019 |
| ***Non-urbanized*** | 465,618 |
| **NEBRASKA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | 1,820,643 |
| Lincoln | 1,729,893 |
| Sioux City, Iowa-Nebr.-S.Dak. | 90,750 |
| ***Non-urbanized*** | 753,658 |
| **NEVADA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | 1,464,378 |
| Reno | 1,464,378 |
| ***Non-urbanized*** | 165,389 |
| **NEW HAMPSHIRE:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | 2,189,420 |
| Lowell, Mass.-N.H. | 5,339 |
| Manchester | 969,353 |
| Nashua | 663,466 |
| Portsmouth-Dover-Rochester, N.H.-Maine | 551,262 |
| ***Non-urbanized*** | 505,466 |

| STATE/URBANIZED AREA | APPORTIONMENT |
|---|---|
| **NEW JERSEY:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | $ 1,716,512 |
| Atlantic City | 1,202,718 |
| Vineland-Millville | 513,794 |
| ***Non-urbanized*** | 864,448 |
| **NEW MEXICO:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | 830,083 |
| Las Cruces | 442,780 |
| Santa Fe | 387,303 |
| ***Non-urbanized*** | 626,312 |
| **NEW YORK:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | 5,472,688 |
| Binghamton | 1,531,416 |
| Danbury, Conn.-N.Y. | 17,500 |
| Elmira | 667,687 |
| Glens Falls | 394,997 |
| Newburgh | 489,842 |
| Poughkeepsie | 1,078,323 |
| Utica-Rome | 1,292,923 |
| ***Non-urbanized*** | 3,033,154 |
| **NORTH CAROLINA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | 9,378,989 |
| Asheville | 708,661 |
| Burlington | 503,870 |
| Concord | 502,540 |
| Durham | 1,387,166 |
| Gastonia | 773,629 |
| Goldsboro | 392,329 |
| Greensboro | 1,555,328 |
| Hickory | 418,370 |
| High Point | 737,446 |
| Jacksonville | 492,872 |
| Wilmington | 599,463 |
| Winston-Salem | 1,307,315 |
| ***Non-urbanized*** | 3,125,013 |
| **NORTH DAKOTA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | 1,644,380 |
| Bismarck-Mandan | 524,040 |
| Fargo-Moorhead, N.Dak.-Minn. | 636,270 |
| Grand Forks, N.Dak.-Minn. | 484,070 |
| ***Non-urbanized*** | 383,301 |
| **OHIO:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | 5,326,321 |
| Hamilton | 965,018 |
| Huntington-Ashland, W.Va.-Ky.-Ohio | 279,732 |

## FISCAL YEAR 1985 UMTA SECTION 9 AND SECTION 18 FORMULA APPORTIONMENTS AMOUNTS APPORTIONED TO AREAS UNDER 200,000 POPULATION—(*Continued*)

| STATE/URBANIZED AREA | APPORTIONMENT |
|---|---|
| **OHIO—*Continued*:** | |
| Lima | $ 613,790 |
| Mansfield | 607,724 |
| Middletown | 685,881 |
| Newark | 412,834 |
| Parkersburg, W.Va.-Ohio | 68,176 |
| Sharon, Pa.-Ohio | 40,935 |
| Springfield | 954,214 |
| Steubenville-Weirton, Ohio-W.Va.-Pa. | 376,243 |
| Wheeling, W.Va.-Ohio | 321,774 |
| ***Non-urbanized*** | ***3,430,836*** |
| **OKLAHOMA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***1,159,915*** |
| Enid | 372,302 |
| Fort Smith, Ark.-Okla. | 14,100 |
| Lawton | 773,513 |
| ***Non-urbanized*** | ***1,416,362*** |
| **OREGON:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***3,706,638*** |
| Eugene | 1,905,967 |
| Longview, Wash.-Oreg. | 9,759 |
| Medford | 466,095 |
| Salem | 1,324,817 |
| ***Non-urbanized*** | ***1,099,368*** |
| **PENNSYLVANIA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***10,613,630*** |
| Altoona | 819,104 |
| Erie | 2,091,982 |
| Hagerstown, Md.-Pa. | 6,783 |
| Johnstown | 861,479 |
| Lancaster | 1,462,661 |
| Monessen | 506,617 |
| Reading | 1,952,038 |
| Sharon, Pa.-Ohio | 449,423 |
| State College | 612,006 |
| Steubenville-Weirton, Ohio-W.Va.-Pa. | 1,873 |
| Williamsport | 565,016 |
| York | 1,284,648 |
| ***Non-urbanized*** | ***3,782,198*** |
| **PUERTO RICO:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***7,415,680*** |
| Aguadilla | 593,324 |
| Arecibo | 679,573 |
| Caguas | 1,591,575 |
| Mayaguez | 1,114,570 |
| Ponce | 2,619,528 |
| Vega Baja-Manati | 817,110 |
| ***Non-urbanized*** | ***1,189,688*** |
| **RHODE ISLAND:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***590,068*** |
| Fall River, Mass.-R.I. | 128,553 |
| Newport | 461,515 |
| ***Non-urbanized*** | ***125,931*** |
| **SOUTH CAROLINA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***$ 1,853,926*** |
| Anderson | 381,020 |
| Florence | 401,385 |
| Rock Hill | 359,219 |
| Spartanburg | 712,302 |
| ***Non-urbanized*** | ***1,556,284*** |
| **SOUTH DAKOTA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***1,177,652*** |
| Rapid City | 427,975 |
| Sioux City, Iowa-Nebr.-S.Dak. | 12,321 |
| Sioux Falls | 737,356 |
| ***Non-urbanized*** | ***444,667*** |
| **TENNESSEE:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***2,017,425*** |
| Bristol, Tenn.-Bristol, Va. | 195,679 |
| Clarksville, Tenn.-Ky. | 370,488 |
| Jackson | 357,809 |
| Johnson City | 551,487 |
| Kingsport, Tenn.-Va. | 541,962 |
| ***Non-urbanized*** | ***2,017,400*** |
| **TEXAS:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***18,556,838*** |
| Abilene | 690,001 |
| Amarillo | 1,226,886 |
| Beaumont | 931,266 |
| Brownsville | 959,046 |
| Bryan-College Station | 659,565 |
| Galveston | 536,539 |
| Harlingen-San Benito | 515,657 |
| Killeen | 786,285 |
| Laredo | 1,260,572 |
| Longview | 494,548 |
| Lubbock | 1,443,414 |
| McAllen-Pharr-Edinburg | 1,500,013 |
| Midland | 607,011 |
| Odessa | 916,460 |
| Port Arthur | 827,111 |
| San Angelo | 620,847 |
| Sherman-Denison | 383,852 |
| Temple | 354,292 |
| Texarkana, Tex.-Ark. | 338,825 |
| Texas City-La Marque | 682,733 |
| Tyler | 623,772 |
| Victoria | 487,205 |
| Waco | 933,798 |
| Wichita Falls | 777,140 |
| ***Non-urbanized*** | ***4,108,567*** |

## FISCAL YEAR 1985 UMTA SECTION 9 AND SECTION 18 FORMULA APPORTIONMENTS AMOUNTS APPORTIONED TO AREAS UNDER 200,000 POPULATION—(*Continued*)

| STATE/URBANIZED AREA | APPORTIONMENT |
|---|---|
| **UTAH:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | $ ***1,503,979*** |
| Provo-Orem | 1,503,979 |
| ***Non-urbanized*** | ***331,364*** |
| **VERMONT:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***586,413*** |
| Burlington | 586,413 |
| ***Non-urbanized*** | ***350,321*** |
| **VIRGINIA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***4,203,228*** |
| Bristol, Tenn.-Bristol, Va. | 152,308 |
| Charlottesville | 624,030 |
| Danville | 439,696 |
| Kingsport, Tenn.-Va. | 29,444 |
| Lynchburg | 615,322 |
| Petersburg-Colonial Heights | 844,176 |
| Roanoke | 1,498,252 |
| ***Non-urbanized*** | ***1,854,473*** |
| **WASHINGTON:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***3,542,365*** |
| Bellingham | 429,809 |
| Bremerton | 527,785 |
| Longview, Wash.-Oreg. | 420,790 |
| Olympia | 531,444 |
| Richland-Kennewick | 866,936 |
| Yakima | 765,601 |
| ***Non-urbanized*** | ***1,216,820*** |
| **WEST VIRGINIA:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***3,870,711*** |
| Charleston | 1,409,599 |
| Cumberland, Md.-W.Va. | 20,943 |
| Huntington-Ashland, W.Va.-Ky.-Ohio | $ 912,444 |
| Parkersburg, W.Va.-Ohio | 600,280 |
| Steubenville-Weirton, Ohio-W.Va.-Pa. | 238,036 |
| Wheeling, W.Va.-Ohio | 689,409 |
| ***Non-urbanized*** | ***1,245,470*** |
| **WISCONSIN:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***8,801,229*** |
| Appleton | 1,472,307 |
| Beloit, Wis.-Ill. | 383,770 |
| Duluth-Superior, Minn.-Wis. | 172,554 |
| Eau Claire | 573,230 |
| Green Bay | 1,113,255 |
| Janesville | 465,039 |
| Kenosha | 1,064,126 |
| La Crosse, Wis.-Minn. | 617,347 |
| Oshkosh | 552,981 |
| Racine | 1,354,801 |
| Sheboygan | 577,265 |
| Wausau | 454,554 |
| ***Non-urbanized*** | ***1,908,239*** |
| **WYOMING:** | |
| ***Governor's apportionment for areas 50,000 to 200,000 population*** | ***1,105,266*** |
| Casper | 588,877 |
| Cheyenne | 516,389 |
| ***Non-urbanized*** | ***283,397*** |
| **AMERICAN SAMOA:** | |
| ***Non-urbanized*** | ***26,014*** |
| **GUAM:** | |
| ***Non-urbanized*** | ***85,363*** |
| **NORTHERN MARIANA ISLANDS:** | |
| ***Non-urbanized*** | ***13,516*** |
| **VIRGIN ISLANDS:** | |
| ***Non-urbanized*** | ***77,783*** |

[FR Doc. 84-28322 Filed 10-25-84; 8:45 am]
BILLING CODE 4910-57-C

federal register

Friday
October 26, 1984

Part VII

# Department of Education

34 CFR Part 760

Secretary's Discretionary Program; Final Regulations

**DEPARTMENT OF EDUCATION**

**34 CFR Part 760**

## Secretary's Discretionary Program

**AGENCY:** Department of Education.

**ACTION:** Final regulations.

**SUMMARY:** The Secretary issues regulations for the implementation of the Secretary's Discretionary Program. The Secretary's Discretionary Program supports research, demonstration, dissemination, training, and technical assistance projects designed to meet special educational needs of educationally deprived children or to improve elementary and secondary education for children consistent with the purposes of the Education Consolidation and Improvement Act of 1981.

**DATE:** These regulations will take effect either 45 days after they are published in the **Federal Register** or later if Congress takes certain adjournments. If you want to know the effective date of these regulations, write or call the Department of Education contact person.

**FOR FURTHER INFORMATION CONTACT:** Dr. Thomas E. Enderlein, 400 Maryland Avenue, SW., Room 4181, Washington, D.C. 20202. Telephone: (202) 472–1762.

**SUPPLEMENTARY INFORMATION:**

### Background

The Education Consolidation and Improvement Act of 1981 (ECIA) (20 U.S.C. 3801 *et seq.*) was enacted as Title V of the Omnibus Budget Reconciliation Act of 1981 (Pub. L. 97–35). The ECIA has two principal purposes: Chapter 1 provides financial assistance to State and local educational agencies (SEA's and LEA's) to meet the special needs of educationally deprived children, and Chapter 2 consolidates 28 elementary and secondary level education grant programs into a single authorization of grants to States for the same purposes contained in the consolidated programs.

Section 583(a) of Chapter 2 authorizes the Secretary to carry out directly, or through grants or contracts, programs and projects that: (1) Provide a national source for gathering and disseminating information on the effectiveness of programs designed to meet the special educational needs of educationally deprived children and others served by the ECIA, and for assessing the needs of such individuals; (2) carry out research and demonstrations related to the purposes of the ECIA; (3) are designed to improve the training of teachers and other instructional personnel needed to carry out the purposes of the ECIA; or (4) are designed to assist SEA's and LEA's in the implementation of programs under the ECIA.

### Public Comments

The Secretary published in the **Federal Register** on February 29, 1984 (49 FR 7546), a Notice of Proposed Rulemaking (NPRM) and invited public comment on the proposed regulations. A summary of all substantive comments received on the NPRM and the Secretary's response can be found in the Appendix to these final regulations.

### Summary of Changes From the NPRM

Only one change was made in the final regulations after the review of the NPRM. Section 760.11(e) has been revised to clarify that the Secretary may decline to fund a project that is eligible for funding under another "specific Department of Education grant competition." This provision implements the Secretary's policy to use limited resources available under the Discretionary Program for activities that cannot be assisted under other grant competitions.

### Summary of Major Provisions

*(1) Projects Eligible for Funding*

Section 760.10 implements the statutory restrictions on the types of projects that may be funded under the Secretary's Discretionary Program. To be eligible for funding under this program, a project must relate to the purposes of the ECIA, and must be limited to one or more of the following: gathering and disseminating information, assessing the effectiveness of programs, research, demonstration, training of teachers or other instructional personnel, or technical assistance to SEA's or LEA's.

Section 583 of the ECIA does not authorize the Secretary to provide general operating revenue to any applicant, including a local school district, an institution of higher education, or other agencies that need or wish additional resources to meet local needs.

*(2) Establishing Priorities*

Section 760.11 of the regulations permits the Secretary to announce funding priorities for the Discretionary Program in one or more notices published in the **Federal Register** each year. The regulations incorporate the statutorily broad discretion of the Secretary in order to permit the Secretary to exercise leadership in education by focusing national attention on unmet national needs within the educational areas addressed by the ECIA.

Under the final regulations, the Secretary would select funding priorities by taking into consideration any unmet national needs within the scope of the ECIA. For example, in fiscal year 1983, the Secretary established as funding priorities for this program: (1) Expanding parental choice in education, (2) improving teacher quality through incentives, (3) strengthening local school boards, and (4) improving education through the application of technology. For fiscal year 1984, the Secretary has made planning teacher incentive structures a funding priority for this program. See "Notice of Final Annual Funding Priorities, Required Activities, and Geographical Distribution for Fiscal Year 1984" published in 48 FR 56257 (December 20, 1983).

In addition to or instead of establishing funding priorities, the Secretary could invite applicants to propose projects in any area of education within the purposes of the ECIA. For example, in fiscal year 1983, the Secretary funded unsolicited applications on topics that included follow-up activities to the National Commission on Excellence in Education, literacy in the school-age population, school violence, and training school board members in the area of special education.

The ECIA generally addresses meeting the special educational needs of educationally deprived children and improving elementary and secondary education for children. However, also included within the scope of the ECIA are the purposes of the programs consolidated in section 561 of the ECIA. Accordingly, subject to other requirements of the ECIA and the final regulations, the Secretary could fund any project that would involve the same activities as formerly authorized by one of the programs consolidated into the ECIA, some of which included specific activities beyond the elementary and secondary level.

For example, the Secretary could fund a demonstration project on consumer education that benefitted the public as a whole because consumer education was authorized to be provided to the public under Title III, Part E of the Elementary and Secondary Education Act (ESEA), one of the authorities consolidated into the ECIA.

The only exception is that although Title II of the ESEA authorized activities beyond the elementary and secondary education level, Chapter 2 of the ECIA limits basic skills to activities relating to elementary and secondary school instruction.

*(3) Selection Criteria*

Section 760.31 of the regulations establishes selection criteria for this program and indicates the maximum number of points that an application could score for each criterion. Additionally, under 760.30, the Secretary will distribute a reserved 15 points among the criteria for each grant competition. The Secretary will announce, in a notice published in the Federal Register, how the reserved points would be distributed for each competition.

*(4) Geographical Distribution*

Under § 760.32 of the regulations, the Secretary could fund applications other than the most highly rated applications if doing so would improve the geographical distribution of projects receiving funding in a particular competition.

*(5) Private School Children Participation*

Section 760.20 requires an LEA to provide an assurance that it will comply with the provisions of section 586 of the ECIA, governing the equitable participation of private school children, if the LEA submits an application for funding to provide services, materials, or equipment for the benefit of public school students. Specific requirements are established in EDGAR, 34 CFR 75.650. Applicants other than LEA's as described above are not subject to the equitable participation requirements in section 586 of the ECIA or in EDGAR.

*(6) Scope of the Final Regulations*

Under § 760.3, the regulations will apply to grants awarded under the Secretary's Discretionary Program, except for grants awarded under any grant program, such as the National Diffusion Network, for which the Secretary issues separate regulations. The regulations do not apply to contracts awarded under the Secretary's Discretionary Program.

**Executive Order 12291**

These regulations have been reviewed in accordance with Executive Order 12291.

They are classified as non-major because they do not meet the criteria for major regulations established in the Order.

**Paperwork Reduction Act of 1980**

Information collection requirements contained in these regulations (Sections 760.20; 760.30–760.32) have been approved by the Office of Management and Budget under the provisions of the Paperwork Reduction Act of 1980 (Pub. L. 96–511) and have been assigned OMB Control No. 1880–0505. This control number appears as a citation at the end of each applicable section.

**Assessment of Educational Impact**

In the notice of proposed rulemaking, the Secretary requested comments on whether the proposed regulations would require transmission of information that is being gathered by or is available from any other agency or authority of the United States.

Based on the comments on the proposed rules and the Department's own review, it has been determined that the regulations in this document do not require information that is being gathered by or is available from any other agency or authority of the United States.

**List of Subjects in 34 CFR Part 760**

Education, Education of disadvantaged, Elementary and secondary education, Grant programs—education, Reporting and recordkeeping requirements.

**Citation of Legal Authority**

A citation of statutory or other legal authority is placed in parentheses on the line following each substantive provision of these final regulations.

Dated: October 22, 1984.

(Catalog of Federal Domestic Assistance Number 84.122, Secretary's Discretionary Program)

**T.H. Bell,**

*Secretary of Education.*

The Secretary amends Title 34 of the Code of Federal Regulations by adding a new Part 760 to read as follows:

## PART 760—SECRETARY'S DISCRETIONARY PROGRAM

**Subpart A—General**

Sec.

760.1 What is the Secretary's Discretionary Program?
760.2 Who is eligible for a grant under the Secretary's Discretionary Program?
760.3 What regulations apply to the Secretary's Discretionary Program?
760.4 What definitions apply to this program?

**Subpart B—What Types of Projects Does the Secretary Assist Under This Program?**

760.10 What types of projects does the Secretary assist under this program?
760.11 Does the Secretary establish priorities for this program?

**Subpart C—How Does One Apply for a Grant?**

760.20 What assurance must an applicant make?

**Subpart D—How Does the Secretary Make a Grant?**

Sec.

760.30 How does the Secretary evaluate an application?
760.31 What selection criteria does the Secretary use?
760.32 How does the Secretary select an application for funding?

**Subpart E—What Conditions Must Be Met by the Grantee?**

760.40 May the Secretary restrict the use of funds under this program?
760.41 May program funds be used for construction purposes?

**Authority:** Sec. 583 of the Education Consolidation and Improvement Act of 1981 (20 U.S.C. 3851), unless otherwise noted.

### Subpart A—General

**§ 760.1 What is the Secretary's Discretionary Program?**

The Secretary's Discretionary Program supports projects designed to meet the special educational needs of educationally deprived children or to improve elementary and secondary education for children consistent with the purposes of the Education Consolidation and Improvement Act of 1981.

(20 U.S.C. 3851)

**§ 760.2 Who is eligible for a grant under the Secretary's Discretionary Program?**

State educational agencies (SEA's), local educational agencies (LEA's), institutions of higher education, and other public and private agencies, organizations, and institutions are eligible for a grant under the Secretary's Discretionary Program.

(20 U.S.C. 3851)

**§ 760.3 What regulations apply to the Secretary's Discretionary Program?**

(a) The following regulations apply to grants made under the Secretary's Discretionary Program:

(1) The Education Department General Administrative Regulations (EDGAR) established in Title 34 of the Code of Federal Regulations in Part 74 (Administration of Grants), Part 75 (Direct Grant Programs), Part 77 (Definitions), and Part 78 (Education Appeal Board).

(2) The regulations in this Part 760.

(b) The regulations in this Part 760 do not apply to—

(1) Contracts awarded under the Secretary's Discretionary Program; and

(2) Grants and contracts awarded under any grant program, such as the National Diffusion Network, for which the Secretary issues separate regulations.

Note.—Grants awarded under the National Diffusion Network are subject to the regulations in 34 CFR Part 796.

(20 U.S.C. 3851; 3474)

**§ 760.4 What definitions apply to this program?**

(a) *Definitions in EDGAR.* The following terms used in these regulations are defined in 34 CFR Part 77:

Applicant
Application
Award
Budget
Department
EDGAR
Local educational agency
Minor remodeling
Nonprofit
Private
Project
Public
Secretary
State education agency

(b) *Definitions that apply to this Part.* The following definitions apply to this Part:

"Children" means persons—

(1) Up to age 21 who are entitled to a free public education not above grade 12; or

(2) Who are of preschool age.

"Construction" means the preparation of drawings and specifications for school facilities; erecting, building, acquiring, altering, remodeling, improving, or extending school facilities; and the inspection and supervision of the construction of school facilities.

(20 U.S.C. 3875)

"ECIA" means Education Consolidation and Improvement Act of 1981.

"Educationally deprived children" means children whose educational attainment is below the level that is appropriate for children of their age.

(20 U.S.C. 3851; 3474)

## Subpart B—What Types of Projects Does the Secretary Assist Under This Program?

**§ 760.10 What types of projects does the Secretary assist under this program?**

(a) The Secretary may fund projects that—

(1) Provide a national source for gathering and disseminating information on the effectiveness of programs designed to meet the special educational needs of educationally deprived children and others served by the ECIA, and for assessing the needs of such individuals;

(2) Carry out research and demonstrations related to the purposes of the ECIA;

(3) Are designed to improve the training of teachers and other instructional personnel needed to carry out the purposes of the ECIA; or

(4) Provide technical assistance to SEA's and LEA's in the implementation of programs under the ECIA.

(b) The Secretary may not provide general operating revenue to meet local needs to any applicant under this program.

(20 U.S.C. 3851)

**§760.11 Does the Secretary establish priorities for this program?**

(a) Each year, through one or more notices published in the **Federal Register**, the Secretary announces the priorities for this program, if any, and the manner in which those priorities will be implemented.

(b) The Secretary selects priorities by taking into consideration unmet national needs within the scope of the ECIA.

(c) The Secretary may also fund any project that does not meet an established priority if the project—

(1) Is limited to activities—

(i) That are at the elementary or secondary education level; or

(ii) That were specifically authorized by one of the programs consolidated in the ECIA as listed in section 561 of the ECIA except as limited by the ECIA; and

(2) Satisfies all other requirements for funding under this program.

(d) If the Secretary selects priorities, the Secretary may establish a separate competition for each priority selected. If a separate competition is established for a priority, the Secretary may reserve all applications that relate to that priority for review under the separate competition.

(e) The Secretary may decline to fund a project that is eligible for funding under a specific Department of Education grant competition.

Note.—EDGAR establishes the method for applying priorities. See 34 CFR 75.105 (Annual priorities).

(20 U.S.C. 3851; 3474)

## Subpart C—How Does One Apply for a Grant?

**§ 760.20 What assurance must an applicant make?**

An applicant must make an assurance in its application that it will comply with the provisions of section 586 of the ECIA, governing equitable participation of private school children in the purposes and benefits of Chapter 2, if the applicant—

(a) Is an LEA; and

(b) Proposes to use grant funds to provide—for the benefit of public school students—services, materials, or equipment (including training for teachers or other personnel serving the school children).

Note.—EDGAR establishes requirements for private school children participation. See 34 CFR 75.650.

(20 U.S.C. 3862)

(Approved by the Office of Management and Budget under control number 1880–0505)

## Subpart D—How Does The Secretary Make a Grant?

**§ 760.30 How does the Secretary evaluate an application?**

(a) The Secretary evaluates an application submitted under this program on the basis of the criteria in § 760.31 of this part.

(b) The Secretary may award up to 100 points, including a reserved 15 points to be distributed in accordance with paragraph (d) of this section, based on the criteria in § 760.31.

(c) Subject to paragraph (d) of this section, the maximum possible points for each criterion is indicated in parentheses after the heading for each criterion.

(d) For each competition, as announced through a notice published in the **Federal Register**, The Secretary may distribute the reserved 15 points among the criteria listed in § 760.31.

(20 U.S.C. 3851; 3774)

(Approved by the Office of Management and Budget under control number 1880–0505)

**§ 760.31 What selection criteria does the Secretary use?**

The Secretary uses the following criteria in evaluating each application:

(a) *Plan of operation.* (20 points)

(1) The Secretary reviews each application for information that shows the quality of the plan of operation for the project.

(2) The Secretary looks for information that shows—

(i) High quality in the design of the project;

(ii) An effective plan of management that insures proper and efficient administration of the project;

(iii) A clear description of how the objectives of the project relate to the purposes of the program;

(iv) The way the applicant plans to use its resources and personnel to achieve each objective;

(v) A clear description of how the applicant will provide equal access and treatment for eligible project participants who are members of groups that have been traditionally underrepresented, such as—

(A) Members of racial or ethnic minority groups;

(B) Women;
(C) Handicapped persons; and
(D) The elderly; and
(vi) For applicants required to provide an opportunity for participation of students enrolled in private schools—a clear description of how the applicant will provide that opportunity.
(b) *Quality of key personnel.* (15 Points)
(1) The Secretary reviews each application for information that shows the quality of the key personnel the applicant plans to use on the project.
(2) The Secretary looks for information that shows—
(i) The qualifications of the project director (if one is to be used);
(ii) The qualifications of each of the other key personnel to be used in the project;
(iii) The time that each person referred to in paragraphs (b)(2) (i) and (ii) of this section plans to commit to the project;
(iv) The extent to which the applicant, as a part of its nondiscriminatory employment practices, encourages applications for employment from persons who are members of groups that have been traditionally underrepresented, such as—
(A) Members of racial or ethnic minority groups;
(B) Women;
(C) Handicapped persons; and
(D) The elderly.
(3) To determine the qualifications of a person, the Secretary considers evidence of past experience and training, in fields related to the objectives of the project, as well as other information that the applicant provides.
(c) *Budget and cost effectiveness.* (5 Points)
(1) The Secretary reviews each application for information that shows that the project has an adequate budget and is cost effective.
(2) The Secretary looks for information that shows—
(i) The budget for the project is adequate to support the project activities; and
(ii) Costs are reasonable in relation to the objectives of the project.
(d) *Evaluation plan.* (5 Points)
(1) The Secretary reviews each application for information that shows the quality of the evaluation plan for the project. Cross-reference—See EDGAR 34 CFR 75.590 (Evaluation by the grantee).
(2) The Secretary looks for information that shows methods of evaluation that are appropriate for the project and, to the extent possible, are objective and produce data that are quantifiable.
(e) *Adequacy of resources.* (5 Points)
(1) The Secretary reviews each application for information that shows that the applicant plans to devote adequate resources to the project.
(2) The Secretary looks for information that shows—
(i) The facilities that the applicant plans to use are adequate; and
(ii) The equipment and supplies that the applicant plans to use are adequate.
(f) *Improving elementary and secondary education.* (10 Points)
(1) The Secretary reviews each application for information that shows the extent to which the project contributes to the improvement of elementary and secondary education.
(2) The Secretary looks for information such as—
(i) The objectives of the project; and
(ii) The manner in which the objectives of the project further the purpose of improving elementary and secondary education.
(g) *National significance.* (15 Points)
(1) The Secretary reviews each application for information that shows the national significance of the project.
(2) The Secretary looks for information that shows the extent to which the project makes a contribution of national significance, as measured by factors such as—
(i) The extent to which the project addesses educational problems of national significance;
(ii) The extent to which the project meets specific national needs as shown by—
(A) The needs addressed by the project;
(B) The manner in which the applicant identified those needs;
(C) The manner in which those needs are met by the project; and
(D) The benefits to be gained by meeting those needs;
(iii) The extent to which the project involves techniques that are innovative;
(iv) The extent to which the project builds upon and adds to current educational information and research; and
(v) The extent to which the project provides a model or other information that could be used by others to solve educational problems.
(h) *Applicant's commitment and capacity.* (10 Points)
The Secretary looks for information that shows the extent of the applicant's commitment to the project, its capacity to continue the project, and the likelihood that it will build upon the project when Federal assistance ends.

(20 U.S.C. 3851; 3474)

(Approved by the Office of Management and Budget under control number 1880–0505)

**§ 760.32 How does the Secretary select an application for funding?**

(a) After evaluating the applications according to the criteria contained in § 760.31, the Secretary may determine whether or not the most highly rated applications are broadly and equitably distributed throughout the Nation for each competition.
(b) The Secretary may select other applications for funding if doing so would improve the geographical distribution of projects funded under a particular competition.

(20 U.S.C. 3851; 3474)

(Approved by the Office of Management and Budget under control number 1880–0505)

## Subpart E—What Conditions Must Be Met by the Grantee?

**§ 760.40 May the Secretary restrict the use of funds under this program?**

The Secretary may restrict the amount of funds made available through a grant under this program that may be used to purchase equipment.

(20 U.S.C. 3851)

**§ 760.41 May program funds be used for construction purposes?**

(a) No recipient may use grant funds for repairs, minor remodeling, or construction of private school facilities.
(b) To the extent necessary for an LEA to carry out its responsibility under section 596 of the ECIA to provide an opportunity for the equitable participation of private school children, an LEA may use grant funds for the construction of public facilities.
(c) Subject to the restrictions in paragraph (a) of this section, a recipient may use grant funds for repairs or minor remodeling only to the extent necessary to carry out an approved project under the regulations in this part.

(20 U.S.C. 3851; 3474)

**Note.**—This Appendix will not appear in the Code of Federal Regulations.

## Appendix—Comments and Responses from NPRM

The following is a summary of the public comments received on the proposed regulations published on February 29, 1984 (49 FR 7546), and the Secretary's responses to those comments. Generally, the comments are presented according to the numbered order of the regulations.

During the 45 days allowed for comment, two letters and one informal response were received. The commenters generally requested

clarification of specific provisions of the regulations. However, one commenter, the Chicago Board of Education (Chicago)—in addition to making many of the specific comments summarized below—characterized the proposed rules as "a blatant misuse, if not abuse, of the administrative rulemaking powers delegated to the Department."

Chicago contended in its letter that one of the main "purposes of the proposed rules, if not the sole purpose, appears to be preventing the Chicago Board of Education from securing funding from the Discretionary Fund for implementation of its desegregation plan pursuant to the obligations of the Board and the United States under the Consent Decree of September 24, 1980." Chicago further stated, "The Department is attempting to accomplish through its rulemaking process what it cannot accomplish before a neutral judicial forum. . . . This is certainly a morally questionable and quite possibly a constitutionally questionable use of the power of the United States against a local Board of Education." Finally, after making many other comments (which are summarized below) on specific sections of the regulations, Chicago recommended "that this entire set of flawed regulations be redrafted pursuant to these comments and issued again in proposed form."

The rules of general applicability established by these final regulations—including the rules for selecting priorities and the selection criteria—are needed to govern competitive awards of grants with Discretionary Program funds. These regulations have been promulgated in accordance with the rulemaking requirements of section 431 of the General Education Provisions Act, 20 U.S.C. 1232, and of the Administrative Procedure Act, 5 U.S.C. 553, and are consistent with established statutory interpretations and practices under the Discretionary Program. See, for example, "The Secretary's Discretionary Program—Planning Grants to Develop Teacher Incentive Structures," 48 FR 56257 (December 20, 1983).

These regulations are not motivated by any attempt to deprive Chicago or any other potential applicant of its right to compete for funds under the statute. On the contrary, the Secretary is issuing these final regulations in accordance with the statute and its legislative history, as consistently interpreted by the Department since the beginning of this program.

**General**

*Comment.* One commenter questioned the Secretary's authority to issue regulations under the Secretary's Discretionary Program. The commenter cited section 591 of the ECIA, which authorizes the Secretary to issue regulations "relating to the discharge of duties specifically assigned to the Secretary under [the ECIA]," and which prohibits the Secretary from issuing regulations "in all other matters relating to the details of planning, developing, implementing, and evaluating programs and projects by State and local educational agencies * * *."

*Response.* No change has been made. In establishing the Secretary's Discretionary Program, section 583 of the ECIA specifically assigns to the Secretary the duty of carrying out the authorized activities directly or through grants or contracts. Beyond indicating in broad terms the types of activities that are eligible for funding, the statute does not prescribe the priorities, evaluation criteria, or procedures to be used by the Secretary in making these funding decisions. These are matters for the Secretary to determine under section 583, as opposed to details of State and local administration of programs. These regulations clearly are authorized by section 591.

**Subpart A—General**

*Section 760.3(a)(1) What regulations apply to the Secretary's Discretionary Program?*

*Comment.* One commenter questioned why the Secretary's Discretionary Program is not subject to 34 CFR Part 79, the implementing regulations for Executive Order 12372 (Intergovernmental Review of Federal Programs). The commenter contended that, in some cases, projects assisted under the Secretary's Discretionary Program may be of special interest to States.

*Response.* No change has been made. The Secretary's Discretionary Program is excluded from coverage under 34 CFR Part 79 because its purpose is not to support services to particular State or local jurisdictions, nor is it directly relevant to the governmental responsibilities of a State or local government. Nevertheless, as a courtesy in a case such as that described by the commenter, the Secretary may choose to notify the State single points of contact of competitions that may be of special interest to States.

*Comment.* One commenter noted that the regulations in 34 CFR Part 760 do not apply to contracts awarded under the Secretary's Discretionary Program and asked what regulations do apply.

*Response.* No change has been made. Contracts awarded after April 1, 1984 are subject to the government-wide Federal Acquisition Regulations, 48 CFR Chapter 1. Additionally, the Department will issue its own implementing regulations in the near future.

**Subpart B—What Types of Projects Does the Secretary Assist under This Program?**

*Section 760.10 What types of projects does the Secretary assist under this program?*

*Comment.* One commenter recommended that the language in proposed §§ 760.10(a) and 760.11(c) be changed to include "programs" as well as "projects" as being eligible for funding.

*Response.* No change has been made. Only the term "project" is used in the above-cited provisions of the final regulations to ensure consistency of terminology throughout the Department's regulations. In 34 CFR 77.1, "project" is defined as "the activity described in an application." In essence, a "project" consists of the specific activities that the Department assists. Nevertheless, those activities—if otherwise eligible—may be part of one or more larger programs conducted by a grantee.

*Comment.* One commenter questioned the use of the term "technical assistance" in § 760.10(a)(4), in describing the types of projects that may be funded under the Secretary's Discretionary Program. The commenter pointed out that section 583 of the ECIA authorizes the Secretary to carry out programs and projects which "are designed to assist State and local educational agencies in the implementation of programs under [the ECIA]."

*Response.* No change has been made. The term "technical assistance" reflects an interpretation of the activities Congress intended to authoriaze in section 583(a)(4) of the ECIA. The specific language of section 583(a)(4) supports this interpretation in that it authorizes projects which " * * * assist State and local educational agencies in the implementation of programs under [the ECIA]." Under section 583(a)(4), projects are eligible only if they assist programs funded under the ECIA. The only logical reading of this authority is as a technical assistance provision to help improve State and local programs receiving funds under other parts of the ECIA. The legislative history of the ECIA also supports the Department's conclusion that Congress, in section 583(a)(4), intended to assist activities in the nature of technical assistance to State and local educational agencies,

not direct funding of State and local programs. The Senate committee report on the language in the Senate Bill, which was enacted as the ECIA, provided: "Up to five percent of the funds under subpart 2 may be used at the discretion of the Secretary for national dissemination activities, research and demonstration programs, and technical assistance programs." S. Rep. 97-139, 97th Cong., 1st Sess. 897 (1981).

*Comment.* One commenter objected to the provision prohibiting the Secretary from providing operating revenue to meet local needs to any applicant under this program. The commenter argued that (1) rarely, if ever, does an applicant for a Federal grant propose a project for support which does not address a local need, (2) the purpose of the ECIA is to meet the special needs of certain categories of children, and (3) the grant assistance either adds to, or provides all of, the operating revenue to implement a grant project.

*Response.* No change has been made. The Secretary has consistently interpreted section 583 of the ECIA as not authorizing the Department to make a grant to an LEA or other applicant for general operating support. This is expressly authorized only by sections 561-582 of Chapter 2 of the ECIA (20 U.S.C. 3811-3842), governing funds allocated by formula to LEA's or reserved for State use. It would have been illogical for Congress to have established an authority for very limited direct funding by the Secretary at the national level—with a maximum of 6 percent of Chapter 2 funds—if that authority were to be used for direct funding to meet the general operating needs of a particular school district, thereby duplicating the authorized uses for the 94 percent of Chapter 2 funds allocated to the States. In fact, Congress did not intend to do so. The nature of the activities specifically authorized by section 583, including the mandated programs in section 583(b); the level of funds authorized; the overall structure of Chapter 2 of the ECIA; the consistent practice under section 583 and its antecedent authority, section 303(a) of the Elementary and Secondary Education Act of 1965 (20 U.S.C. 2943); and the legislative history of section 583 all lead to this conclusion.

Section 760.10(b) is consistent with previous notices for competitions under the Secretary's Discretionary Program which the Department has issued. *See* Application Notice for the Secretary's Discretionary Program Planning Grants to Develop Teacher Incentive Structures (48 FR 56257, December 20, 1983 at 56258, "Funding for projects under these grants will be limited to the cost of developing a workable plan."); Application Notice for the Secretary's Discretionary Program Grants—Improving Education through the Application of Technology (48 FR 30080, June 29, 1983 at 30081, "Funding under these awards will cover the cost of planning, administering, evaluating, and disseminating information about the project."); Application Notice for the Secretary's Discretionary Program Grants (48 FR 13220, March 30, 1983 at 13221, "Funding for demonstration projects under these awards will be limited to the cost of administering and evaluating the project," and at 13222, "Funding for projects under these awards will be limited to the cost of collecting and analyzing information to describe and assess the effects of the demonstration," and "Funding of projects under this award will not include stipends or travel costs for trainees.")

Moreover, the Catalog of Federal Domestic Assistance (CFDA) lists the Secretary's Discretionary Program, beginning with the 1983 update, and indicates in the description of "uses and use restrictions" that "[n]o awards can be made to provide general operating support to a local educational agency (LEA) or other eligible applicants." *See* CFDA No. 84.122. The CFDA—which is published by the Executive Office of the President, Office of Management and Budget—is a basic source of information on Federal programs and has as its primary purpose assisting potential applicants to identify Federal programs for which they may apply, and providing general information on these programs. See similar provisions, making operating costs ineligible for support under other research and demonstration programs in 34 CFR 709.12, 710.6, 716.11, 718.6, and 720.6.

*Section 760.11(b) Does the Secretary establish priorities for this program?*

*Comment.* One commenter objected to the provision which provides for the Secretary to select priorities by taking into consideration unmet national needs and asserted that the Secretary may not disable himself from the discretion to fund any project authorized by the statute. The commenter noted that the ECIA makes the Secretary's Discretionary Program funds available to support projects which relate to the purposes of the ECIA.

*Response.* No change has been made. The Secretary has the authority to issue regulations "relating to the discharge of the Secretary's duties specifically assigned under [the ECIA]." 20 U.S.C. 3871(a). One of those duties is to determine how monies appropriated by Congress for the Discretionary Program are spent. In carrying out this duty, consistently with the purposes of the Discretionary Program, the Secretary sets priorities for distribution of monies "taking into consideration unmet national needs within the scope of the ECIA." Selecting priorities does not preclude the Secretary from funding a project that does not meet an established priority provided that the project is within the scope of the ECIA and satisfies the requirements for funding described in the statute; namely, a project which relates to the purposes of the ECIA and the Discretionary Program's national perspective, and which involves dissemination of information, research, demonstration, training of instructional personnel, or technical assistance to State and local educational agencies.

*Comment.* One commenter questioned whether proposed § 760.11(c)(1)(ii) limits funding under the Secretary's Discretionary Program to those activities which are allowable under programs consolidated into the ECIA, and argued that the purposes and authorized activities contained in the ECIA itself are the controlling authority for permissible activities under the Secretary's Discretionary Program.

*Response.* No change has been made. Each of the activities specifically authorized by section 583(a)—namely, dissemination of information, research, demonstration, the training of instructional personnel, or technical assistance for State or local educational agencies—is tied by the language of section 583 to the purposes of the subtitle, children served by the subtitle, or programs under the subtitle. The subtitle is Subtitle D of the Omnibus Education Reconciliation Act of 1981 and includes Chapters 1 and 2 of the ECIA. As interpreted by the Secretary, the purposes and activities of the subtitle include—in addition to Chapter 1 programs—all of the purposes and activities that previously could have been funded under the antecedent programs consolidated by Chapter 2, except to the extent inconsistent with specific language in Chapter 2.

The only such inconsistency is that although the former basic skills program under Title II of the Elementary and Secondary Education Act authorized activities beyond the elementary and secondary level, Chapter 2 of the ECIA specifically limits basic skills activities to those relating to elementary and secondary school instruction. Of course, as indicated above, projects may be funded under the Secretary's

Discretionary Program only if they meet the specific authorizations in section 583.

*Comment.* One commenter questioned whether § 760.11(e) should enable the Secretary to decline to fund a project that is eligible for funding under another Department of Education program. The commenter noted that every activity eligible for support under the Secretary's Discretionary Program, by definition, can be funded under the Chapter 1 or Chapter 2 (block grant programs) of the ECIA.

*Response.* A change has been made. Section 760.11(e) has been revised to permit the Secretary to decline to fund a project that is eligible for funding under another "specific Department of Education grant competition." This change should eliminate the problem raised by the commenter while still carrying out the Secretary's policy of using limited resources available under the Discretionary Program for worthwhile activities that cannot be assisted under other grant competitions.

**Subpart D—How Does the Secretary Make a Grant?**

*Section 760.31 What selection criteria does the Secretary use?*

*Comment.* One commenter recommended that the Secretary delete the selection criterion related to the national significance of a project in proposed § 760.31(g). The commenter contended that any project which relates to the purposes of the ECIA may be funded. The commenter suggested that in an instance where national significance is an appropriate criterion, the Secretary could assign part of the reserved 15 points to that criterion.

*Response.* No change has been made. As noted above in response to a comment on § 760.11(b), § 760.11(c) permits the Secretary to fund any project that is within the scope of the ECIA and that involves dissemination of information, research, demonstration, training of instructional personnel, or technical assistance to State or local educational agencies related to programs implemented under the ECIA.

Chapter 2 of the ECIA essentially converted the categorical antecedent programs into State and local programs and reserved a small amount of appropriated funds for the Secretary to use under the Discretionary Program in identifying significant programs from a national perspective. Use of the criterion of "national significance" in making awards is designed to identify those projects which best carry out the purposes of the Discretionary Program. This criterion has been consistently used in selecting applications for funding under this program. The Catalog of Federal Domestic Assistance (CFDA) describes the objectives of the Secretary's Discretionary Program "to assist in research, dissemination, demonstration, improvement of training, and technical assistance activities, which address some national education priority as authorized by Section 583 of the [ECIA]." *See* CFDA No. 84.122.

*Comment.* One commenter questioned the usefulness of the capacity to continue a project as a criterion for evaluating an application for funds in view of the fact that there may be no need to continue some types of projects, for example, a teacher training project.

*Response.* No change has been made. Section 760.32(h) provides for the Secretary, in selecting applications for funding, to consider the extent of the applicant's commitment to, and capacity to continue, a project, as well as the likelihood that the applicant will build upon the project when Federal assistance ends. While the training phase of a project, for example, may end, the continued use of the training by those individuals that received it is a measure of the impact and usefulness of the project.

*Section 760.32 How does the Secretary select an application for funding?*

*Comment.* One commenter questioned the authority of the Secretary to consider geographic distribution of the most highly rated applications as a final factor in selecting applications for funding, because the ECIA does not mention geographic location.

*Response.* No change has been made. Section 760.32 of the final regulations enables the Secretary to select for funding those projects that are best located to serve as resources for solving educational problems nationwide. Because the ECIA does not contain any selection criteria for the Secretary to consider in making awards, it is necessary for the Secretary to issue regulations establishing selection criteria to be used in making competitive awards under the statute. The Secretary has the authority and discretion to "issue regulations * * * relating to the discharge of duties specifically assigned to the Secretary under [the ECIA]." 20 U.S.C. 3871(a).

With respect to demonstration projects, for example, the potential impact of a project may be greatest in the area in closest proximity to the location of the project. Therefore, in order to ensure nationwide impact of a competition limited to, for example, demonstration projects related to a particular educational priority, the Secretary believes it is appropriate to base the final selection of applications for funding, in part, on the geographic location of the applicant in order to ensure a broad and equitable distribution of projects throughout the Nation.

[FR Doc. 84-28319 Filed 10-25-84; 8:45 am]

BILLING CODE 4000-01-M

federal register

Friday
October 26, 1984

## Part VIII

# Environmental Protection Agency

**40 CFR Part 136**

**Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule**

**ENVIRONMENTAL PROTECTION AGENCY**

**40 CFR Part 136**

**[FRL-2636-6]**

# Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act

**AGENCY:** Environmental Protection Agency (EPA).

**ACTION:** Final Rule and Interim Final Rule with Request for Comments.

**SUMMARY:** EPA is amending its *Guidelines Establishing Test Procedures for the Analysis of Pollutants*, as proposed on December 3, 1979. EPA is also reprinting existing test procedures for the convenience of readers. However, only those test procedures which are new or revised are being promulgated for purposes of judicial review. The purpose of this amendment is to establish:

- New test procedures (including quality control requirements) for the analysis of priority toxic organic pollutants;
- A new test procedure for the measure of carbonaceous biochemical oxygen demand (CBOD);
- A new test procedure based upon inductively coupled plasma optical emission spectroscopy for the analysis of most of the regulated trace metal pollutants; and,
- Mandatory container materials, preservations, and holding times for samples of the parameters covered by this regulation.

The quality control requirements establish control limits for acceptable analytical performance. However, the specific control limits in the test procedures for the priority toxic organic pollutants are being promulgated as an interim rule with a request for comments. Comments should be limited to the calculation of the numerical warning limits for the revised quality control sections.

In accordance with the Clean Water Act (CWA), these procedures will be required for filing applications for National Pollution Discharge Elimination System (NPDES) permits and for State certifications. These test procedures will also be used for compliance monitoring and to express pollutant quantities, characteristics, or properties in effluent limitations guidelines and standards and in pretreatment standards set forth at 40 CFR Parts 402 through 699 (unless otherwise specifically noted or defined in those parts).

**DATES:** In accordance with 40 CFR 100.01 (45 FR 26048), this regulation shall be considered issued for purposes of judicial review at 1:00 p.m. eastern time, November 9, 1984. These regulations shall become effective for all methods except $CBOD_5$ on January 24, 1985. The regulation relating to $CBOD_5$ [40 CFR § 136.3(a) Table IB, parameter 14] will be effective November 26, 1984. Comments on the interim final rule for specific control limits [40 CFR § 136.3(a) Table IC, footnote 7, and Table ID, footnote 7] will be accepted until December 26, 1984.

Under section 509(b)(1) of the Clean Water Act, judicial review of this regulation can be obtained only by filing a petition for review in the United States Court of Appeals within 90 days after these regulations are considered issued for purposes of judicial review (see *NRDC* v. *EPA*, 673 F.2d 402, D.C. Cir., 1982). Under Section 509(b)(2) of the Clean Water Act, the requirements of this regulation may not be challenged later in civil or criminal proceedings brought by EPA to enforce these requirements.

The information collection requirements contained in 40 CFR 136.3(e), has not been approved by the Office of Management and Budget (OMB) and is not effective until OMB approves it. The incorporation by reference of certain publications listed in the regulation is approved by the Director of the Federal Register as of January 24, 1985.

**ADDRESSES:** Comments on the calculation of control limits should be labelled as "Section 304(h): Control Limit Calculations" and submitted to: Dr. Robert B. Medz, Water and Waste Management Monitoring Research Division, Office of Research and Development (RD–680), U.S. Environmental Protection Agency, 401 M Street, SW., Washington, D.C. 20460. Telephone Number: (202) 382–5788.

Most of the public record for this rulemaking will be available for inspection from 8:00 A.M. to 4:00 P.M. in EPA's Public Information Reference Unit, Room 2404 (rear of the EPA Library), 401 M Street, SW., Washington, D.C. 20460. The remainder of the record (primarily materials describing interlaboratory studies) will be available at the Environmental Monitoring and Support Laboratory at the Andrew W. Breidenbach Environmental Research Center, 26 West St. Clair Street, Cincinnati, Ohio 45268, from 8:00 a.m. to 4:00 p.m., Monday through Friday.

The EPA information regulation (40 CFR Part 2) allows the Agency to charge a reasonable fee for copying.

**FOR FURTHER INFORMATION CONTACT:** Dr. Robert B. Medz, at the address listed above or call (202) 382–5788.

**SUPPLEMENTARY INFORMATION:**

**Outline of Preamble Discussion**

I. Authority
II. History and Background
  A. Structure and History of 40 CFR Part 136
  B. Consent Decree and the Priority Pollutants
III. Summary of the Proposed Amendment
  A. Purposes
  B. GC, HPLC, and GC/MS Test Procedures
  C. Quality Control and Quality Assurance
  D. Other Table I Organic Parameters
  E. ICP Test Procedure
  F. $CBOD_5$ Test Procedure
IV. Highlights of Final Test Procedures
  A. Restructuring of Table I
  B. GC, HPLC, and GC/MS Test Procedures
  C. ICP Test Procedure
  D. $CBOD_5$ Test Procedure
  E. Table II: Required Containers, Preservation Techniques, and Holding Times
  F. Incorporation by Reference
V. Public Participation and Response to Most Significant Comments
  A. GC, HPLC, and GC/MS Test Procedures
  1. Policy on Applicability of Test Procedures
  2. Flexibility and Analysts' Professional Judgment
  3. Quality Control and Quality Assurance
  B. ICP Method
  C. $CBOD_5$ Method
  D. Table II: Required Containers, Preservation Techniques, and Holding Times
  E. Cost Estimates
  F. Publication of Full Texts of Test Procedures
  G. Consistency of Analytical Methods Approved Under Different Acts
VI. Economic Analyses
VII. Effective Dates

## I. Authority

Today's amendment was proposed on December 3, 1979 (44 FR 69464). It is being promulgated under the authority of sections 301, 304(h) and 501(a) of the CWA, 33 U.S.C. 1251 et seq. Section 301 forbids anyone to discharge any pollutant into navigable waters except pursuant to an NPDES permit issued under the CWA. Permits are issued under § 402, which is referenced in Section 304(h). Subsection 304(h) requires the Administrator to "promulgate guidelines establishing test procedures for the analysis of pollutants that shall include the factors which must be provided in any certification pursuant to section 401 of the Act or permit application pursuant to section 402 of the Act." Section 501(a) authorizes the Administrator "to prescribe such regulations as are necessary to carry out his functions under the Act."

The Administrator has also made these test methods applicable to monitoring and reporting of NPDES permits (40 CFR Part 122, Subsections 122.21, 122.41, 122.44, and 123.25), and implementation of the pretreatment standards issued under section 307 of the CWA (40 CFR Part 403, Subsections 403.10, and 403.12).

## II. History and Background

### *A. Structure and History of 40 CFR Part 136*

The Administrator first approved test procedures for the analysis of wastewater pollutants on October 16, 1973, and first amended the list of approved test procedures on December 1, 1976 (See 38 FR 28758, October 16, 1973 and 41 FR 52780, December 1, 1976).

The full texts of the approved test procedures incorporated by reference in the regulation are considered to be part of the regulatory language. Most of the test procedures were cited from the following compilations of consensus test procedures:

- "EPA Manual of Methods for the Chemical Analysis of Water and Wastes,"
- "Standard Methods for the Examination of Water and Wastewater,"
- "American Society for Testing and Materials (ASTM) Annual Book of Standards, Part 31, Water,"
- "Official Methods of Analysis of the Association of Official Analytical Chemists (AOAC),"
- "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments of the U.S. Geological Survey."

Additional test procedures were incorporated from other standards groups, such as the American National Standards Institute (ANSI), or from the open literature. Several test procedures, such as those for the analysis of benzidine, were incorporated from specific EPA sources.

Test procedures have previously been approved for about 115 different parameters. Those procedures apply to the analysis of inorganic (metal, non-metal, mineral), nutrient, demand, residue, radiological, organic, bacteriological, and physical parameters. For any given parameter, the regulations generally approved several different analytical methods. The December 1, 1976 amendments to 40 CFR Part 136 approved certain test procedures which were identified in tabular form (Table I). The discharge parameters to be measured were presented alphabetically. Each parameter was followed by a brief test procedure description and by page numbers of the incorporated references. This unambiguously identified the approved test procedure.

An equivalency program is provided in 40 CFR Part 136. Under this program the Administrator may approve alternate test procedures developed and proposed by dischargers or other persons. If dischargers or other persons wish to use such alternate test procedures, they must apply to the State or Regional EPA permitting office (for limited approval) and to the Director of the Environmental Monitoring and Support Laboratory in Cincinnati (for nationwide approval). The equivalency provisions are included in these guidelines to encourage the development of new analytical methods, and to give analysts a number of options for resolving analytical problems that may be unique to specific wastewaters.

Finally, there may be discharges from some particular industries which need to be regulated on the basis of parameters or test procedures which have not been proposed and approved within the scope of the test procedures guidelines under 40 CFR Part 136. EPA may include such parameters and alternate test procedures within the rule-making for these industries in accordance with the provisions prescribed at 40 CFR 401.13, "Test Procedures for Measurements." Such test procedures may be integrated into the text of future amendments and revisions of 40 CFR Part 136.

The following provides a brief regulatory history of 40 CFR Part 136 prior to the current amendment.

- First proposal: 38 FR 17318 (at 40 CFR Part 130, later redesignated as Part 136), June 29, 1973.
- First promulgation: 38 FR 28750 (at 40 CFR Part 136), October 16, 1973.
- First amendment proposal: 40 FR 24535, June 9, 1975.
- First amendment promulgation: 40 FR 52780, December 1, 1976.
- Second amendment proposal: 44 FR 69464, December 3, 1979.
- Correction Document, second amendment proposal: 44 FR 75031, December 18, 1979.
- Comment period reopened, second amendment proposal: 46 FR 3033, January 13, 1981.
- Equivalent Alternate Test Procedure Approvals:

Chemical Oxygen Demand: 43 FR 9341, March 7, 1978.
pH and Ammonia: 43 FR 38618, August 29, 1978.
Nitrite Nitrogen: 44 FR 25505, May 1, 1979.
Manganese: 44 FR 34193, June 14, 1979.
Chemical Oxygen Demand: 45 FR 26811, April 21, 1980.
Copper and Zinc: 45 FR 36166, May 29, 1980.
Iron: 45 FR 43459, June 27, 1980.
Residual Chlorine: 46 FR 58489, December 2, 1981.

Many reviewers of the proposed amendments requested that certain documents upon which the procedures were based be made available for review. In response, the Administrator sent 38 supporting documents to EPA's Regional Offices and to the EPA Headquarters Library in Washington, D.C., for inspection by the public. The closing date for comments was also extended from February 1, 1980, to April 28, 1980, to permit adequate time for public inspection of the record.

The Agency also started a series of formal inter-laboratory collaborative validation studies (each comprising of 15 to 20 laboratories) for the trace organic priority pollutant test procedures and the trace metal ICP test procedure. These were designed to establish expected inter-labaoratory precision and accuracy characteristics of the test procedures.

Late in 1980, representatives of the Chemical Manufacturers Association (CMA), the American Petroleum Institute (API), and EPA met informally to discuss the reliability of some of the proposed test procedures. On January 5, 1981, these representatives met again to more formally discuss these concerns. CMA and API felt the test procedures for the toxic organic priority pollutants should not be promulgated as final until the inter-laboratory validation studies had been completed. The comment period was reopened on January 12, 1981, to allow all interested persons to inspect the official transcript of the January 5, 1981 meeting, and if needed, to amend their earlier comments on the proposed regulation. The extended comment period closed on February 2, 1981.

### *B. Consent Decree and the Priority Pollutants*

In 1976, the Agency entered into a consent decree in the District Court of the District of Columbia (*Natural Resources Defense Council, Inc., et al.* v. *Train*, 8 ERC 2120 (D.D.C. 1976), as modified 12 ERC 1833 (D.D.C. 1979), and by the Court's Orders of October 26, 1982, August 2, 1983, January 6, 1984, and July 5, 1984), and the decree requires EPA to study the occurrence of 65 categories of pollutants in industrial wastewaters, and to limit the discharge of those pollutants in effluent by issuing effluent guidelines based on the "best

available control technology economically achievable" (BAT), pretreatment standards for new and existing sources, and new source performance standards. (**Note:** Within those 65 categories, 114 specific organic toxic pollutants and 15 inorganic pollutants were identified for a total of 129 specific toxic pollutants studied by EPA. Bis-chloromethyl ether (46 FR 10723, Feb. 4, 1981) and dichlorofluoromethane and trichlorofluoromethane (46 FR 46103, Jan. 8, 1981) have since been removed from the list, leaving 126 toxic pollutants now listed as "priority" toxic pollutants). The Agency began development of test procedures for measuring these pollutants in complex industrial wastewater matrices.

Paragraph 4(c) of the Consent Decree also required the EPA to establish and implement a program to identify and study pollutants other than the priority pollutants. At a minimum, EPA was to consider those additional pollutants listed in Appendix C of the Settlement Agreement. Those additional pollutants were not included in the December 3, 1979 proposal of the regulation. In studying Methods 1624 and 1625, EPA has evaluated applying the methods to those additional pollutants. A separate notice in today's **Federal Register** proposes to extend the scope of those methods to include the paragraph 4(c) pollutants.

The 1976 Test Procedures Guidelines (41 FR 56780, December 1, 1976), provided approved test procedures, selected from the various consensus standards, for 14 of the 15 inorganic priority toxic pollutants. The exception was asbestos, for which no adequate procedure was then available. The 1976 Guidelines also provided approved test procedures, similarly selected, for several chlorinated organic compounds (including PCBs, pesticides, benzidine, and pentachlorophenol). However, neither those procedures nor existing consensus standards were adequate to meet the testing requirements for all of the 114 priority toxic organic pollutants.

To fill this gap, the Agency embarked on an extensive program to develop additional test procedures to implement sections 301, 304(h) and 402. By 1979, these test procedures had been developed to a stage that represented the state-of-the-art analysis of the trace organic priority pollutants in industrial wastewater discharges. On December 3, 1979 the Agency proposed these methods, together with a test procedure for analysis of trace metals by inductively coupled plasma optical emission (ICP) and a test procedure for determining the carbonaceous biochemical oxygen demand of municipal wastewaters, as amendments to 40 CFR Part 136.

## III. Summary of the Proposed Regulation

### *A. Purposes*

On December 3, 1979, the Agency proposed to revise the "Guidelines Establishing Test Procedures for the Analysis of Pollutants." The primary purposes of this proposal were:

(1) To amend Table I, List of Approved Test Procedures, by adding the priority toxic organic pollutant parameters and approved alternate test procedures for their analysis;

(2) To add an approved test procedure for a new parameter, "carbonaceous biochemical oxygen demand" (CBOD), which is important to secondary biological treatment technology for municipal wastewaters;

(3) To approve an additional state-of-the-art test procedure based on the inductively coupled plasma optical emission (ICP) principle for the analysis of most of the trace metal parameters which were already covered in Table I; and,

(4) To remove Table I, footnote 1, which recommended sample container materials, preservation procedures, and maximum holding times, and to specifically list those elements as mandatory requirements in a new Table II, "Required Containers, Preservation Techniques, and Holding Times."

Several test procedures were included for each of the priority toxic organic pollutants. This allowed several analytical options for most analyses. The CBOD parameter was included for analysis of a new specific measure of oxygen demand. The ICP test procedure was included to provide an additional and more rapid tool for trace metal analysis. Mandatory sample container materials, preservation techniques, and maximum holding times were included because these have been found to be critical to assure NPDES data quality.

The test procedures for the toxic organic priority pollutants were developed by the Agency in response to the mandates of the Consent Decree. At the time of proposal, the test procedures had been subjected to intensive single laboratory developmental testing. They were considered to be the best state-of-the-art test procedures available for the routine analysis of treated wastewaters for organic pollutants. They also appeared to be applicable to the analysis of untreated wastewaters. Multi-laboratory validations of these test procedures had been planned but had not yet been started. The Agency decided to propose the test procedures for priority toxic organic pollutants before completion of the inter-laboratory validation studies because:

- Even without inter-laboratory validation these were (and are) the most tested and intensively validated test procedures available for the analysis of the toxic organic priority pollutants in industrial and municipal wastewater discharges,
- Many permits were expiring and permit renewals would require some provision for priority pollutant analysis,
- The new round of BAT effluent guidelines regulations would include limits on priority toxic organic pollutant discharges, and,
- The priority toxic organic pollutants would need to be reported under the permits regulations at 40 CFR Parts 122 and 123, and by pretreatment regulations at 40 CFR Part 403.

The following discussion covers the provisions of the proposed regulation in more detail.

### *B. GC, HPLC, and GC/MS Test Procedures*

Since 1976 the text of the regulation has listed pollutant parameters alphabetically in Table I, "List of Approved Test Procedures,"—either as specific compounds or entities such as "Benzidine" or as classes of compounds or entities such as "Pesticides." The individual parameters within such classes, when identified, were entered alphabetically within the class. Approved test procedures were then identified by test procedure descriptors and by page numbers in specifically identified references. In the case of "Pesticides" and several other organic chemical classes, the Table refers to the full text of the approved test procedures in order to clearly identify the scope of the test procedures.

In 1979 the Agency proposed to consolidate all of these organic chemical parameters under a new class entry, "Organic Compounds", and to identify alphabetically all of the specific organic compounds which were included within the scope of the various approved test procedures. All of the test procedures and organic compounds which were approved in 1976 continued to be approved, and were re-printed (unmodified) in the proposal only because Table I was being restructured.

The new organic chemical entries in Table I were the 114 (now 111) priority toxic organic pollutants and the proposed test procedures for their analysis. Twelve of the proposed test procedures were based on gas chromatography (GC) and/or high-

pressure liquid chromatography (HPLC). Three of the proposed alternate test procedures were based on gas chromatography with mass spectrometric detection (GC/MS).

Chromatography is the technique by which compounds in mixtures are separated by interaction between a mobile and a stationary phase. The stationary phase is placed in a tube called a column, and the mixture is moved through the column by the mobile phase under the influence of a pressure differential across the tube. Different compounds are carried through the column at different speeds by the mobile phase. The order in which the compounds exit the column (elute) is determined by the chemical and physical properties of the compounds and of the mobile and stationary phases. In each method the stationary and mobile phases are selected to give the most effective separation of the compounds of interest.

In gas chromatography (GC), the mobile phase is a gas, and the stationary phase is usually a high molecular weight liquid, coated on an inert support or on the column wall of a very small diameter tube (an open tubular column). In liquid chromatography (LC) the mobile phase is as solvent, and the stationary phase is usually a selective adsorbant. As compounds in the mixture elute (exit) from the column, their presence is indicated by various detectors. One detector is the mass spectrometer, hence the term GC/MS. Following detection, the compounds can be identified and then quantified by a computer or by the analyst.

Separations by chromatographic techniques are not always complete. Several compounds within a mixture which have similar chemical and/or physical properties may simultaneously elute from the column, along with the compounds of interest. These are known as interfering compounds or interferents.

Where compounds are easily identified, conventional detectors can often make better quantitative assessments than GC/MS can. However, in many cases, the conventional detectors used in chromatography are not able to discriminate between the compounds of interest and the interferents. In such instances, a mass spectrometer usually is able to discriminate between the compounds of interest and the interferents. Thus, it would be the preferred detection system where interferents are expected or many compounds must be identified. An alternative solution is to use a second column containing a different stationary phase. This aids in the identification of the compounds of interest by providing additional qualitative identification when conventional chromatography detectors are used.

The low cost of the conventional GC detectors, relative to MS, makes the GC option particularly attractive for routine monitoring of small numbers of pollutants. On the other hand, the GC/MS test procedures allow for the simultaneous or rapid sequential measurement of large numbers of different organic pollutants. They also provide certain structural information that can be used to minimize interferences that would mask compound identification by the less specific conventional GC detectors.

EPA divided priority toxic organic pollutants into 12 categories, based on their physical and chemical properties and chemical structures. A GC or HPLC test procedure was then developed for each category, with the expectation that the pollutants within each category could be measured by a single procedure. These procedures were to be routinely used where the pollutants to be measured were known to have a high probability of occurrence. GC and HPLC *could* also be used for *qualitative* identifications of unknown materials, although the proposed GC/MS test procedures were more suitable for this purpose. In most cases, several GC or HPLC configuations of inlet, column, operating conditions, and detectors were recommended with each procedure. Each test procedure stipulated that, if it were used to screen samples for priority pollutants, an analyst needed to verify any compound identified with an independent analytical protocol. The GC/MS was suggested as such a protocol.

In the proposed tests of the organic toxic priority pollutant test procedures, it was not EPA's intent to require separate samples for each test procedure. Subsequent comments have indicated that this was not clear from the test procedure texts or in the proposed sample preservation and holding time requirements in Table II.

*C. Quality Control and Quality Assurance*

Quality control (QC) includes all of the means taken by an analyst or an analytical laboratory to make certain that the total measurement system, including the analyst's performance and matrix problems, are calibrated correctly or accounted for, and remain in calibration or accounted for in all ensuing analyses. Quality assurance (QA) includes all the means taken within or beyond the laboratory to make certain that the measurement systems in different laboratories in a monitoring network remain in calibration with a common external standard, and hence with each other. QA/QC seeks to assure that analyses of the same substances taken by different analysts at different times and places are of the same quality and are comparable within known statistical confidence limits. EPA proposed that the QC within the GC and HPLC test procedures require the use of field replicates to validate the precision of sampling techniques. Laboratory replicates and fortified samples were also proposed in 1979 to validate the precision and accuracy of analyses. Since EPA's studies in this area were not yet finalized at the time of the proposal, additional quality control guidance was described in general terms and proposed as necessary to enable evaluation of the performance of test procedures.

Similar GC configuration and quality control guidance was proposed for the GC/MS test procedures, except that the GC/MS could be used in a screening application without a mandatory confirmation protocol. However, a separate, more intensive quality control procedure was proposed and described in detail, as it might be applicable to the GC/MS test procedures.

*D. Other Table I Organic Parameters*

Proposed Table I was restructured to include the previously designated organic parameters benzidine, pentachlorophenol, Aldrin, δ-BHC, γ-BHC, chlordane, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Dieldrin, Endosulfan, Endrin, and Heptachlor as specific entries in the organic compounds subsection. The previously approved test procedures (41 FR 52780, Dec. 1, 1976) for most of the organic pollutants incorporated within Table I had been entered without changes under the broad parameters "chlorinated organic compounds (except pesticides)" and "pesticides." These broad parameters included test procedures for chlorinated organic solvents, chlorinated hydrocarbon pesticides, carbamate pesticides, triazine pesticides, phosphate pesticides, and chlorinated phenoxy carboxylic acid pesticides. Approved test procedures for these parameters have been available on request from EPA's Environmental Monitoring and Support Laboratory in Cincinnati, Ohio (EMSL–CI).

*E. ICP Test Procedure (ICP)*

The ICP test procedure was proposed as an additional option for the analysis of 23 trace elements: aluminum, arsenic, barium, beryllium, boron, cadmium,

calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, potassium, selenium, silicon, silver, sodium, vanadium, and zinc. It was listed in Table I, under Metals. Lithium and strontium were also included within the scope of the ICP method although the consent decree had not listed them as high priority toxic parameters. This proposal did not affect the previously approved atomic absorption (AA), voltametric, gravimetric, flame photometric, and colorimetric test procedures referenced in Table I for these elements.

The ICP test is a rapid, multielement procedure, representing state-of-the-art. It was proposed because of its time- and cost-effectiveness, and because available data showed it to be equivalent to the other approved procedures for analyzing the designated metals in wastewater discharges.

*F. $CBOD_5$ Test Procedure*

The $CBOD_5$ test procedure was a new parameter proposed in Table I. It responded to many requests for an approved procedure to measure the carbonaceous component of oxygen demand. It was a draft version of the consensus method now included as Method 507 paragraph e(6) in the 15th Edition of "Standard Methods for the Analysis of Water and Wastewater."

The $CBOD_5$ is a different measure of oxygen demand than the total $BOD_5$ (Standard Method 507). Thus it cannot be used to analyze oxygen demand when an NPDES permit calls for $BOD_5$ to be measured. The $CBOD_5$ procedure uses a nitrogen biochemical oxygen demand inhibitor. This inhibitor allows oxygen to be consumed only by organisms that require carbon as their nutrient source. In the presence of such an inhibitor, the nitrogen compounds remain refractory to biochemical degradation, since the activity of nitrifying organisms is suppressed.

## IV. Highlights of Final Test Procedures

*A. Restructuring of Table I*

Users familiar with the former text of section 136 will first notice the reorganization of Table I, which lists pollutant parameters for which approved analytical methods exist, and indicates the approved method(s) available for each parameter. In the 1976 regulations, the parameters in Table I of § 136.3(a) were organized around broad categories, such as bacteriological test procedures and test procedures for metallic or residue parameters. These broader categories were then entered alphabetically into Table I.

Analyses for an additional 111 organic parameters have now become essential. However, entering all those parameters into a single list of approved methods became unwieldy, especially with the proliferation of footnotes to the table. Therefore, to make Table I easier to use, it has been restructured for this final rulemaking into five sub-tables:

- Table IA. List of Approved Biological Test Procedures.
- Table IB. List of Approved Inorganic Test Procedures.
- Table IC. List of Approved Test Procedures for Non-Pesticide Organic Compounds.
- Table ID. List of Approved Test Procedures for Pesticides.
- Table IE. List of Approved Radiological Test Procedures.

Throughout Table I, EPA has updated numerous references to consensus methods, e.g., from the 14th to the 15th edition of "Standard Methods." These changes are technical amendments without substantive effect and are promulgated as final amendments. EPA has determined that notice and public procedure on these updates are unnecessary and contrary to public interest. See 5 U.S.C. 553(b)(3)(B).

These technical amendments should not affect any on-going enforcement actions or other regulatory actions on analyses performed with earlier methods. Today's amendments do not approve consensus methods adopted since the proposal when they contain substantive revisions to the previously approved methods. Instead, the Agency has retained its approval of the prior method. Examples discussed below include the retained approval of the 14th edition Standard Method for phenols (4AAP), and the limited approval of the U.S.G.S. method for fecal streptococci which, as approved, is identical to the previous U.S.G.S. method.

Table IA includes bacteriological test parameters which were approved in the 1976 Guidelines. Approved methods for their analysis are now listed in a new format; they are not substantively changed. Previously cited references have been updated. With the exception of the U.S.G.S. test procedures for fecal streptococci, no changes have been made in the test procedures. As noted only editorial changes have been made in the texts of the other test procedures in these updated references.

A new EPA reference is now approved for several bacteriological test parameters. The updated USGS fecal streptococci test procedure is approved *only* if the dissolution of the nutrient medium is conducted in a boiling water bath. This is because dissolution on a hot plate or over a open flame (which appears to be permitted in the updated reference) can lead to scorching or to other alterations in the nutrient medium.

Table IB includes all of the inorganic and physical parameters that were in the 1976 Guidelines. The previously cited references have been updated.

The ICP test procedure is now approved as an additional alternate test procedure for the analysis of 25 Table IB trace element parameters. Antimony and thallium are now included within the scope of the ICP test procedure in response to information made available in comments which were received. The only new parameter which has been added to Table IB is the Carbonaceous Biochemical Oxygen Demand ($CBOD_5$) parameter.

Table IC includes 97 organic, non-pesticide chemical parameters. Test procedures for 21 of these parameters were approved in the 1976 Guidelines and continue to be approved, unchanged, in Table IC. Ninety-five of the Table IC parameters (including 19 parameters approved since 1976) are priority toxic organic pollutants for which new test procedures were proposed. The new test procedures are essentially the same as those proposed, with the exception that, (1) where possible, they have been made more flexible in response to comments and (2) quality assurance and quality control standards have been defined. Two newly-modified GC/MS test procedures, Methods 1624 and 1625, which are variants of Methods 624 and 625, have been added to Table IC. They differ from Methods 624 and 625 by utilizing stable, isotopically labeled analogs of the analytes as internal standards for GC/MS analysis. This allows the analyst to accurately correct for variability in analyte recovery efficiency, responding to a criticism raised by commenters.

With the exception of the test procedures for benzyl chloride and epichlorohydrin, all test procedures in Table IC prescribe quality control limits. The actual specific control limits are the sole elements of this regulation which are promulgated as an "interim final" action. This is because the data base and calculations of these limits have been developed since proposal. However, the idea of specific mandatory, acceptability criteria was fully proposed, favorably commented on, and finally accepted. Thus the regulatory framework for the specific limits is being promulgated as final.

Table ID contains the 67 parameters included under the general "pesticides" parameter in the 1976 Guidelines. Although most pesticides are organic

compounds, they have been listed separately in Table ID rather than with the other organic parameters in Table IC because of the wide association between this subset of organic compounds and their end use. Sixteen of the 67 parameters are priority pollutants. Three additional pesticides were identified as priority pollutants under the consent decree. Table ID therefore now identifies 70 specific pesticides, of which 19 are priority pollutants. Methods 608 and 625, which were proposed for the priority organic toxic pollutants, were revised to incorporate substantive comments. All other references in Table ID have been updated, but the updated references do not require any substantive changes from previously approved test procedures.

Table IE now includes the five radiological test procedures approved in the 1976 Guidelines. All references have been updated, and an EPA reference has been added. There are no substantive textual changes in these updated test procedures.

*B. GC, HPLC, and GC/MS Test Procedures*

Analyses for organics depend upon a variety of chromatographic techniques. See subsection III–B above. EPA proposed and is approving two HPLC methods (605 and 610), 10 GC methods, and three GC/MS methods (613, 624, and 625). In addition, EPA has responded to critiques of Methods 624 and 625 by approving two GC/MS/isotope dilution variants (1624 and 1625). Each method is accompanied by a specific set of quality assurance (QA) procedures. The QA process relies on specific control limits calculated for each parameter for which the method can be used. The control limits indicate the outer range of precision and accuracy found in an extensive inter-laboratory study. The limits represent the minimum threshold of quality expected of competent laboratories: 95 percent confidence level per compound for the 600 series and the 99 percent confidence level across the set of compounds for the 1624 and 1625 methods. Most analyses should have far better precision and accuracy. The calculations of specific numerical control limits for the calibration and quality control sections of the GC, HPLC, and GC/MS test procedures is interim final. This means that they are legally effective, but that EPA will accept comments on their calculation. All other parts of these test procedures are finally approved for the analysis of the parameters which are indicated in Table IC and ID.

Each method is approved for specific organic compounds. In general, GC Methods 601–603 and GC/MS Methods 624 and 1624 are approved for the analyses of the purgeable priority pollutants. GC Methods 604 and 606–612 and GC/MS Methods 625 and 1625 are approved for the analysis of the non-purgeable, volatile priority pollutants, including, for Method 625 only, the priority pesticide pollutants. Method 625 is also approved for screening samples for 2,3,7,8-TCDD (2,3,7,8-tetrachlorodibenzo-p-dioxin), but only GC/MS Method 613 is approved for final qualitative confirmation or quantification of 2,3,7,8-TCDD in samples. HPLC Methods 605 and 610 are also approved for the analysis of the nonpurgeable volatiles (the benzidines and polynuclear aromatic hydrocarbons). Methods 1624 and 1625 are approved for use interchangeably with the other test procedures which are being approved for the analysis of the priority toxic organic pollutants. Their most significant difference from Methods 624 and 625 is the requirement that, where available, stable, isotopically-labeled analogs of the priority pollutants are to be used as method internal standards. Since Methods 624 and 625 do permit flexibility in the selection of internal calibration standards and surrogate standards, Methods 1624 and 1625 are, in essence, acceptable variants permitted by Methods 624 and 625. They improve on Methods 624 and 625 and are generally preferable. However, Methods 624 and 625 are also being approved because they are widely available, slightly less expensive, and they are of use when interference and recovery efficiency are not expected to be problems.

In general, both GC/MS and non-MS test procedures have been approved for each of the priority toxic pollutants. Most of the revisions of the proposed test procedures were made either for clarification or to give the analyst more flexibility to practice professional judgment. These procedures now contain a section on safety, cautioning analysts of the potential hazards associated with exposure to the chemical reagents required by the test procedures, or to the toxic chemicals being analyzed. Recommended and mandatory quality assurance practices are also given in each of the test procedures.

Methods 601–604, 606–609, 611–613, 624, 625, 1624, and 1625 include specifications for performing the tests. These specifications are based on a required primary GC column and specified detector. A primary HPLC column and specified detector are required for Methods 605 and 610 and specifications are provided. The primary column is also used to identify the pollutant. A secondary column and detector are also defined, but not required, for non-MS Methods 601–604 and 606–611. The secondary column and detector can be used for confirmation of priority pollutants identified by the primary column for unfamiliar (non-routine) samples (see sections 1.2 of the methods). The GC/MS test procedures are suggested as the confirmatory test for identifications made by Methods 605 and 612, and may also be used as the confirmatory test for identifications made by Methods 601–604 and 606–611. For example, an unfamiliar sample which would be likely to need confirmation would be a single sample taken for an NPDES application. See 40 CFR 122.21. In contrast, routine monitoring, such as that for discharge monitoring reports, would be less likely to require a secondary column for confirmation since the sample is more likely to be familiar to the analyst.

Methods 606, 609, 611 and 612 all use essentially the same procedure for sampling, sample extraction, and concentration. Thus a single sample may be used to measure the parameters within the scope of these methods.

Sample container materials, preservation techniques, and holding times are critical to the procedures and are specifically defined (Methods 601–613, 624, 625, 1624 and 1625). The design and operation of the purge-and-trap device in Methods 601–603, 624 and 1624, and the sample extraction procedures of Methods 604–613, 625 and 1625 are precisely defined as well.

In response to public comments, substantive revisions were made to allow more flexibility in the remaining parts of Methods 601–613, 624, 625, 1624 and 1625. In Methods 604–613, after the sample has been extracted, the analysts are now free to choose a technique to concentrate the extract. The same flexibility is provided for selecting the GC or HPLC configurations (column packings, operating conditions, and detectors). When analysts use concentration techniques or chromatographic configurations other than those described in the test procedures, their approaches must meet the performance criteria defined in the section of the procedures dealing with calibration and quality control.

The most difficult task in finalizing the methods for organic analyses was defining the relationship between desirable flexibility in the methods and

necessary quality control. The proposal specifically solicited comments on both issues and many comments were received on each. The final methods resolve the issue by allowing far greater method flexibility, but by establishing specific control limits as a mandatory part of the quality control procedure.

The proposal noted, and comments confirmed, that method flexibility should be inherent in the methods. Historically, rigid protocols have been a problem in organics analyses. For example, an analyst may be using a method, other than mass spectrometry, to identify a few specific components out of the several million known to exist. This requires that interferences be overcome and "canned" approaches may not effectively address interferences, particularly where matrices are variable or diverse. Thus, the Food and Drug Administration (FDA) and AOAC and other method standardization organizations have usually provided optional "clean-up" procedures for organics, for example, permitting analysts to use Florisil clean-up for pesticides. Further, the analyst may be interested in measuring only a few compounds, while the proposed method may be designed to measure large categories of compounds. For example, a particular industry may be regulated only for the compound that elutes from the gas chromatography after a long program temperature run. An inflexibile method might require the analyst to go through the entire temperature run to look for a single peak that elutes late in the chromatogram. This may be needlessly inefficient. For such reasons, EPA has decided to permit flexibility in chromatographic conditions.

Commentors also raised concerns about inflexibility in sample preparation. They objected to the Kuderna-Danish glassware concentration technique being the only approved approach for concentrating extracts. In fact, if the analysts are measuring only the less volatile compounds in a method category, it may not be necessary to require a rigid procedure for concentration. In this case, it may be appropriate to allow other procedures for concentrating extracts.

After considering these issues, the Agency has decided to allow limited flexibility within the methods. Specifically, chromatographic conditions, including column packings and detectors can be varied. This approach allows continued technical development of the methods. Thus EPA avoided a rigid prescription of technology that would soon be obsolete due to the rapid advances occurring in chromatography. However, the primary objective underlying this flexibility is to enhance precision and accuracy for each analysis. Flexibility should not be permitted if the altered technique would be *less* precise or *less* accurate than the standard approved analytical method. Thus, a corollary of increased flexibility was an increased need for a rigorous and unambiguous quality control procedure.

These basic decisions had become clear by the time of the second, reopened comment period. The comments received in the second comment period again supported the issue of quality control and requested that the criteria be specified more clearly. Another general comment was that the criteria should wait for the results of the inter-laboratory method validation studies and be based upon those results. Today's rulemaking reflects these comments, while specifying that EPA will accept further comments, limited specifically to the calculation of control limits from that new data base.

The quality control procedures now take two different forms. First, there is a "start-up test" to establish the laboratory's basic ability to set up and operate the analytical equipment and procedure. The purpose of the start-up test is two-fold; it establishes that analytical equipment has been properly set up, and it demonstrates the basic ability of the analyst to recognize the compounds of interest. It is required every time the method is changed. It requires the analysis of four spiked distilled water samples. The analyst compares his measures of precision and accuracy to establish criteria developed from the inter-laboratory method validation studies. Because of the basic threshold nature of the start-up test, the methods allow the test to be performed with reagent water.

If the analyst fails the criteria for accuracy or precision in the start-up test, the analyst is to repeat the test for any compound that fails a criterion. If the analyst is measuring, for example, eight compounds at once using Method 601, and fails the criteria for three of them, the analyst is required only to repeat the three that failed provided the method is not changed. It is not very difficult to meet the criteria for any individual compound. However, when one is analyzing for numerous compounds there is an accumulation of failure probabilities; that is, an increased likelihood that one of several parameters will fail for "statistical" reasons. Thus EPA allows a "second pass" opportunity to meet the criteria, as long as the method is not changed. Exhibit 1, below, offers some guidance as to when analysts may want to skip the "second pass" opportunity based on an excessive number of test criteria failures occurring on the first pass. An excessive number of failures should not occur if the system is operating properly. Thus, such a number of failed criteria may suggest poor operation to the analyst. In this case, the first pass criteria failures suggested the compound(s) tested would fail a second round. The analyst may wish to simply adjust the system and reinitiate the start-up test.

If the method is changed as a result of the initial test, the startup begins again. For example, if the start-up test indicates zero recovery of vinly chloride and a check reveals that the instrument trap was installed backwards, the operator must correct the problem and reinitiate the test for all compounds, since the method was just modified.

The second form of quality control is contained in the ongoing quality control program. Laboratories are required to analyze blank samples (e.g., reagent water) daily, and to analyze spiked wastewater samples periodically. Ten percent of all samples are to be spiked (five percent for Methods 624 and 625). The resulting accuracy of recovery must be compared to the established accuracy criteria for the method developed from the results of the inter-laboratory method studies.

If an analyst fails one or more accuracy criteria with the spiked wastewater, the analyst must analyze a check sample (e.g., spiked reagent water). The purpose of analyzing the check sample is to establish whether the inaccuracy is caused by matrix effects or by the laboratory operating improperly (i.e., out of control). Again, accuracy results are compared to the established accuracy criteria. The criteria for acceptable accuracy in these methods are based upon accuracy derived from testing reagent water. Use of check samples rather than spiked wastewater to verify the accuracy criteria for a laboratory is consistent with the fact that one set of regression equations in the inter-laboratory method study is derived from reagent water. That set of regression equations is the basis quality control criteria.

The decision to rely on spiked wastewater samples for the initial test is an alternative to requiring that analyses be conducted on ten percent spiked reagent water samples (to verify laboratory control) and ten percent of

spiked wastewater samples (to verify matrix effects). Accordingly, the need to also analyze a check sample is reduced to a second-tier requirement which is only mandated if accuracy criteria are not met with spiked wastewater.

The limits that are in the methods have been derived on a compound-by-compound and method-by-method basis. They are derived directly from the inter-laboratory method validation studies. The formal inter-laboratory validation studies for Methods 601–602, 604–613, 624, 625, and 1625 have been completed with 15 to 20 laboratories. These fifteen methods have been revised to include methods performance results derived from these studies.

Two methods (603 and 1624) have not been subject to an inter-laboratory validation study. A formal inter-laboratory validation study for Method 603 has not been completed due to an error in the draft method. Although the error was corrected, EPA was not able to perform an inter-laboratory validation study on the same scale as performed for the other methods. However, one commercial laboratory did validate the method and that validation was verified by EPA's laboratory. In addition, the method is similar to Methods 601 and 602 and the results from the validation are similar. EPA believes that the validation of 603 is adequate to establish that the method is appropriate. Therefore, Method 603 is being promulgated with warning limits based upon the best data now available.

Method 1624 was not formally validated through an inter-laboratory study. The specifications for Method 1624 were developed from Method 624 which was formally validated. In informal multi-laboratory and single-laboratory studies, Method 1624 has been shown to yield slightly better performance on treated effluents than Method 624, but this improvement is insufficient to warrant a separate inter-laboratory validation study.

The multi-laboratory validation studies were designed according to the method of W.J. Youden (Youden, W.J., "Statistical Technique for Collaborative Tests," Statistical Manual of the Association of Official Analytical Chemists, 1975) in which pairs of samples having slightly different spiked concentrations of the compound of interest are analyzed. Each collaborating analyst analyzes a sample only once and reports a single value. By having the analyst perform the analysis as he would have done for a normal routine sample, the Youden design helps to avoid accidental manipulation of data that can sometimes occur in a laboratory doing replicate determinations.

Each Youden sample pair for a given parameter is prepared so that the concentration of the pollutant of interest in one-half of the pair is similar to, but measureably different from, the concentration of the pollutant in the other half. Three Youden pairs were analyzed for each of the parameters. The mean values of each of the three pairs were designed to spread over a usable and realistic range of concentrations. The lowest concentration pair was prepared so that the concentration would be above the minimum detection concentration for the method.

The Youden pairs, prepared as concentrates, were spiked into six different water matrices: distilled water, municipal drinking water, a surface water vulnerable to synthetic chemical contaminants, and usually, three different industrial wastewaters from industries that normally would be regulated for the priority pollutants under study. The data were reduced to four statistical relationships related to the overall study: (1) Multi-laboratory mean recovery for each sample, (2) accuracy expressed as relative error or bias (the difference between the multi-laboratory mean recovery and the true value divided by the true value), (3) the multi-laboratory standard deviation of the spike recovery for each sample, and (4) the multi-laboratory relative standard deviation. In addition, two statistics were reduced from the raw data relating to the single-analyst performance: (1) Single-analyst standard deviation, and (2) single-analyst relative standard deviation.

The single-analyst standard deviations were calculated for each of the sample pairs according to the method of Youden by (1) calculating the difference for recoveries from each sample pair reported by each analyst, (2) calculating the average value of these differences across the entire study, (3) calculating a "sum-of-the-squares" by adding the square of the differences between each difference and mean difference, (4) dividing the "sum-of-the-squares" by the degrees of freedom to give the single-analyst variance, and (5) taking the square root of the variance to give the single-analyst standard deviation.

Fifteen to twenty-five percent of the data generated in the multi-laboratory validation studies were discarded as outliers, i.e., data too far from the vast majority of data to be acceptable. Outliers were determined based on widely accepted statistical tests prescribed by ASTM and AOAC.

There is an apparent linear relationship between the mean recovered spike values and the true spike values, overall standard deviation, and single-analyst standard deviation. These linear relationships have been expressed as regression equations over the concentration ranges studied in each matrix. Six different regression equations are derived for each of the six matrices for any given compound. In most cases the variations of the six lines do not appear to be statistically significant at the 5% significance level. The conclusions were reached for each water type by using the F-distribution to compare variance statistics of waste waters with those of distilled water. Mean recoveries were compared between wastewater and distilled water using paired t-test statistics.

EPA is aware that there are limits to the strength of these analyses. These comparisons assume independence among the observations and this was not exactly the case since the "spike" was made up of mixtures of all of the compounds under consideration in each method and hence there was an interdependence among compounds. Despite these limitations, the tests still provide strong evidence that water type generally had no statistically significant effect on the method's performance.

The multi-laboratory tests support an important conclusion. If a laboratory performs well with the methods using distilled water, it should be able to obtain good results with surface waters and industrial wastewaters. Based upon this conclusion, the multi-laboratory regression equations for accuracy and single-analyst overall precision for distilled or reagent water have been incorporated into the quality assurance and quality control provisions of the final texts of Methods 601, 602, 604–613, 624, and 625 to define method performance. The regression equations for the other matrices are also included in the texts of the methods.

The multi-laboratory validation of Method 1625 was performed at a single concentration in a reagent water matrix. Specifications were derived for linearity of calibration, for calibration verification, for retention time precision, for compound recovery from a reagent water matrix, and for precision and accuracy of analysis by isotope dilution and internal standard techniques. All specifications derived from the study are applied at the same level at which they were tested, and sample matrices which show labeled compound recoveries significantly different from

recoveries of these compounds from reagent water are diluted with reagent water to bring these recoveries into the expected range.

It is also important to note that the studies provide a strong basis for setting control limits which represent a range of acceptability. The studies show that most laboratories will do far better, especially on a single-operator, single-laboratory basis. Other performance studies, completed since the inter-laboratory analyses, incorporate too much flexibility to be directly analogous to EPA's collaborative test of the methods. However, they appear to confirm the assumption that most laboratories will exceed the minimum standards and indicate that method variability will be well within the range of the control limits.

The final specifications derived for all of the organics methods (except 603) were the result of a statistical analysis of the data from the multi-laboratory studies. These specifications adopt initial precision and accuracy for all methods. For start-up calibration verification, they specify control limits for Methods 601, 602, 624, 1624, 625 and 1625. For on-going accuracy, they specify control limits for recovery of pollutant spikes for Methods 601–613, 624, and 625, and for recovery of labeled compound spikes for Methods 1624 and 1625. The methods allow for simultaneous testing of all the parameters listed in each method.

In theory, a problem could arise from simultaneous tests for numerous compounds. The control limits have been calculated to allow only a 5% likelihood that a result that exceeds the limits for each compound is merely a statistical fluctuation (rather than actual error). However, the chance of "statistical error" rises with the number of compounds being tested.

EPA has corrected for this possibility in several ways. First, most users will not apply each analysis to all parameters simultaneously; thus they will have a greater chance of passing all test criteria. Second, in order to allow for simultaneous testing of all parameters in a given method, the specifications for accuracy and precision have either been broadened, or a re-test has been allowed, or both. The technique of using a re-test was chosen because a one-test-only specification which allowed for simultaneous testing of a large number of parameters would be so broad as to have little meaning. The provision for a re-test preserved a meaningful specification while allowing for simultaneous testing of all parameters. If a laboratory fails the re-test as well as the initial test, the likelihood of "statistical error" is extremely low (5% times 5%, i.e., .0025 for a given compound). Third, when a re-test is required, it need only be performed on the particular compounds which failed the initial test. Finally, the control criteria for Methods 1624 and 1625—those most likely to be simultaneously used on many compounds—were determined based on the 99% confidenee level.

As a voluntary guide to laboratories practicing a given method, the following Exhibit 1 gives suggested numbers of first pass test criteria failures which are unlikely if the laboratory is satisfying the probability based quality control specifications. It assumes all parameters in a given method are tested simultaneously. The Exhibit indicates the maximum number of parameters for which each method can be used simultaneously. The two right-hand columns indicate a certain number of unacceptable results. If the analyst finds that number, or a greater number, of unacceptable results, he may conclude that the entire analysis is flawed. If so, it may be more efficient to repeat the entire analysis than to re-examine only the compounds which exceed the control limits.

EXHIBIT 1.—SUGGESTED MAXIMUM NUMBER OF TEST CRITERIA FAILURES WHICH JUSTIFY REPEATING ENTIRE ANALYSIS

| Method | Number of simultaneous parameters | Number of test startup [1] | Criteria failures on-going [2] |
|---|---|---|---|
| 601 | 29 | 7 | 4 |
| 602 | 7 | 3 | 2 |
| 603/605 | 2 | 2 | 2 |
| 604 | 11 | 4 | 3 |
| 606 | 6 | 3 | 2 |
| 607 | 3 | 2 | 2 |
| 608 | 25 | 6 | 4 |
| 609 | 4 | 3 | 2 |
| 610 | 16 | 5 | 3 |
| 611 | 5 | 3 | 2 |
| 612 | 9 | 4 | 3 |
| 613 | 1 | 2 | 1 |
| 624 | 31 | 7 | 5 |
| 625 | 61 | 11 | 7 |
| 1624 | 66 | 12 | 7 |
| 1625 | 151 | 7 | 5 |

[1] Based on twice the number of parameters being tested since both accuracy and precision are being evaluated.
[2] Based on the number of parameters being tested.

Section 8 of each method defines acceptable analytical performance limits for the GC, HPLC, and GC/MS test procedures (Methods 601–613, 624, 625, 1624, and 1625). These acceptable performance limits are also specified in Footnote 7 to Table IC, "List of Approved Test Procedures for Non-Pesticide Organic Compounds," and Footnote 7 to Table ID, "List of Approved Test Procedures for Pesticides." System performance is acceptable only when the average recoveries and standard deviations of spikes of the pollutants of interest into reagent water meet these performance standards. Where large numbers of parameters are being analyzed (see Exhibit 1 above), there is an increased chance that at least one parameter will fail for either average recovery or standard deviation limits based purely on chance. Where such failure occurs, the spiking and recoveries must be repeated, but only for the failed parameters. Repeated failure confirms a general problem with the analytical measurement system. When such failed recoveries are experienced the system is judged to be out-of-control for the failed parameter. Thus, the results for the failed parameters in unspiked samples are suspect and cannot be reported to show regulatory compliance.

The acceptance criteria for spikes into samples for each parameter were calculated to include both an allowance for error in prior measurement of the background and another allowance for error in prior measurement of spike concentrations. The calculation assumed a spike-to-background ratio of 5 to 1. Thus such error will be accounted for to the extent the analysts' spike-to-background ratio approaches 5 to 1. In many cases this allows analysts a greater margin of error than should actually be expected. This is because the calculation assumes that two prior errors are cumulative, ignoring the degree to which they actually cancel each other out.

Today's final test procedures represent an effort to provide the maximum uniformity that is practical for a wide cross-section of classes of chemical compounds. They will be continually reevaluated for their general applicability to complex wastewater matrices.

The substantive revisions made in the GC, HPLC, and GC/MS methods in response to comments are discussed in the public participation section of this preamble. Three of the most significant changes include: (1) Addition of a confirmatory column to Method 602; (2) deletion (from 613) of the gas chromatographic/electron capture (GC/EC) test procedure for screening for 2,3,7,8-TCDD, and (3) revision of Methods 613 and 625 to show that Method 625 may be used whenever screening for 2,3,7,8-TCDD is required. The full text of the approved GC, HPLC, and GC/MS test procedures are being printed in Appendix A of this regulation.

The GC, HPLC, and GC/MS test procedures are now cited in the regulations in the new Table IC, "List of

Approved Test Procedures for Non-Pesticide Organic Compounds," and Table ID, "List of Approved Test Procedures for Pesticides."

*C. ICP Test Procedure*

The ICP test procedure is cited in the regulation as an additional analytical option for trace metal analysis in the new Table IB, "List of Approved Inorganic Test Procedures."

The ICP test procedure, Method 200.7, has been changed only slightly from the version proposed on December 3, 1979. EPA proposed that lithium and strontium be analyzed using the ICP test procedure, since these parameters could be analyzed using this method. Because EPA did not propose or develop accuracy or precision criteria for these parameters, EPA is unable to approve the ICP test procedure for them. EPA is considering the ICP and other alternative test procedures in a separate rulemaking. In light of additional information received in the public comments showing good recoveries for antimony and adequate recoveries for thallium by the proposed test procedure, both of these metals have been added to the scope of the ICP test procedure. Also in response to public comments the detection limit for silica has been doubled and the wavelengths of the metal are now given to the third decimal. In section 3 of the ICP test procedure a new definition for "Quality Control Sample" has been provided for clarification, and a new section on safety has been added to alert the analyst to the hazards of the toxic reagents and pollutants involved. Other revisions made in response to comments are discussed in the public participation section of this preamble. The full text of the ICP procedure is printed as Appendix C to this regulation.

*D. $CBOD_5$ Test Procedure*

The final test procedure for $CBOD_5$ is essentially the same as that proposed. See Section III–D, above. EPA's proposed test procedure was taken from a draft *Standard Methods* test procedure for $CBOD_5$.

The final method language is the same as the language now included in the 15th edition of *Standard Methods*. This has required minor changes from the wording of the proposal, but no substantive changes were required.

*E. Table II: Required Containers, Preservation Techniques, and Holding Times*

Table II in Section 136.3(e) now restricts the materials of which sample containers can be made, and specifies the procedures by which samples are to be preserved. Table II also limits the maximum time for which samples may be held from the the time of sampling until they are analyzed. Table II has been restructured in this final regulation to correlate with the parameters in the new Tables IA, IB, IC, ID, and IE in Section 136.3(a). Table II allows cross-reference between the container, preservative, and holding times and the individual parameters in Tables IA, to IE.

In response to comments, several changes were made in Table II of the final regulations for prescribed container materials, preservation requirements, and holding times of wastewater samples. Where supported by comments, changes were made primarily in holding times. In response to comments, EPA has adopted the requirement that some samples be analyzed immediately, to avoid sample degradation. This would be as soon as the sample is collected and labelled, generally within 15 minutes. Longer holding times are generally not appropriate where the sample may quickly degrade. However, a longer time period may be justified under the variance procedure. Exhibits 3 and 4, below, show that for organic compounds and pesticides, the holding times were generally extended from 30 days after extraction to 40 days after extraction. Changes were also made to enable a single sample to be used for analyses of extractable organics and of pesticides. This was a step towards the goal of uniformity, sought by EPA and by the commenters.

Table II as promulgated also allows a variance to holding times under § 136.3(e). Analysts may exceed the holding times if they have data on file to show that the specific types of samples are stable for a longer time and if they receive a variance from the Regional Administrator.

No changes were made for container materials, preservation requirements, or holding times in final Table II from the proposed requirements for the biological parameters listed in Table IA, or the radiological parameters listed in Table IE. Changes which were made in Table II for inorganic parameters listed in Table IB, organic parameters listed in Table IC, and pesticide parameters listed in Table ID are summarized in the following Exhibits 2, 3, and 4, of this preamble. Proposed and final container materials, preservation requirements, and holding times in Exhibits in 2, 3, and 4 are given only for the affected pollutant parameters in Tables IB, IC and ID of the regulation.

EXHIBIT 2.—CHANGES MADE IN TABLE II FOR TABLE IB PARAMETERS

| Parameter | Requirement | Change | |
|---|---|---|---|
| | | From (proposed) | To (final) |
| Chlorine residual | Holding time | 2 hours | Analyze immediately. |
| Cyanide | Preservative | $Na_2S_2O_3$ | Ascorbic acid. |
| | | | Add: Remove sulfide as cadmium sulfide. |
| pH | Holding time | 2 hours | Analyze immediately. |
| Chromium VI | Holding time | 48 hours | 24 hours. |
| Mercury | Preservative | 0.05% $K_2Cr_2O_7$ | Delete. |
| Organic carbon | Preservative | None | Add: HCl or $H_2SO_4$ to pH<2. |
| Dissolved oxygen probe | Holding time | 1 hour | Analyze immediately. |
| Winkler | Preservative | | Add: Store in dark. |
| Phenols | Container | P and G | G only. |
| Residue, total | Holding time | 14 days | 7 days. |
| Residue, filterable | Holding time | 14 days | 7 days. |
| Residue, settleable | Holding time | 7 days | 48 hours. |
| Sulfide | Preservative | | Add: NaOH to pH>9. |
| | Holding time | 28 days | 7 days. |
| Sulfite | Holding time | 48 hours | Analyze immediately. |
| | Preservative | Cool to 4 °C | None required. |

EXHIBIT 3.—CHANGES MADE IN TABLE II FOR TABLE IC PARAMETERS

| Parameter | Requirement | Change | |
|---|---|---|---|
| | | From (proposed) | To (final) |
| Chlorinated organic compounds | (2) | (2) | (2) |
| Purgeable aromatics | Preservative | | Add: HCl to pH<2. Samples not receiving pH treatment must be analyzed within 7 days. |
| Acrolein and acrylonitrile | Holding time | 3 days | 14 days. |
| Phenols [1] | Preservative | $H_2SO_4$ to | Deleted. |
| | Holding time | pH<2 30 days after extraction | 40 days after extraction. |
| Benzidine [1] | Preservative | | Add: pH 2-7, if 1,2-diphenylhydrazine is present, adjust pH to 4.0±0.2. |
| Phthalate esters [1] | Preservative | $Na_2S_2O_3$ | Deleted. |
| | Holding time | 30 days after extraction | 40 days after extraction. |
| Nitrosamines [1] | Preservative | | Add: Store in dark. |
| | Holding time | 30 days after extraction | 40 days after extraction, adjust pH 7-10 for diphenylnitrosamine. |
| PCBs [1] | Preservative | $Na_2S_2O_3$ | Deleted. |
| | Holding time | 30 days after extraction | 40 days after extraction. |
| Nitroaromatics and isophorone [1] | Preservative | $Na_2S_2O_3$ | Deleted. |
| | Holding time | 30 days after extraction | 40 days after extraction. |
| Polynuclear aromatic hydrocarbons [1] | Preservative | | Add: Store in dark. |
| | Holding time | 30 days after extraction | 40 days after extraction. |
| Haloethers [1] | Holding time | 30 days after extraction | 40 days after extraction. |
| Chlorinated hydrocarbons [1] | Preservative | $Na_2S_2O_3$ | Deleted. |
| | Holding time | 30 days after extraction | 40 days after extraction. |
| TCDD [1] | Holding time | 30 days after extraction | 40 days after extraction. |

[1] The following optional preservation may be used when the asterisked categories are to be analyzed in a single sample: cool to 4 °C, add 0.008% $Na_2S_2O_3$, store in dark, adjust pH 6.0-9.0.
[2] Dropped as a parameter—see Purgeable Halocarbons and Chlorinated Hydrocarbons.

EXHIBIT 4.—CHANGES MADE IN TABLE II FOR TABLE ID PARAMETERS

| Parameter | Requirement | Change | |
|---|---|---|---|
| | | From (proposed) | To (final) |
| Pesticides [1] | Preservative | $Na_2S_2O_3$ | Deleted. |
| | Holding time | 30 days after extraction | Add: pH 5-9. 40 days after extraction. |

[1] The following optional preservation may be used when the asterisked categories are to be analyzed in a single sample: cool to 4 °C, add 0.008% $Na_2S_2O_3$, store in dark, adjust pH 6.0-9.0.

*F. Incorporation by Reference*

The analytical methods approved are lengthy and detailed. Many are readily available to the public. Thus, § 136.3(a) has been revised to show that the full texts of the test procedures taken from the various references in Tables IA, IB, IC, ID and IE are "incorporated by reference" into the regulatory language of these Guidelines in accordance with the regulations of the Office of the Federal Register, and with the approval of the Director of the Office of the Federal Register. Methods which are not readily available are printed in full as appendices to this notice.

As a convenience to the users, § 136.3(b) has also been added. This cross-references Tables IA, IB, IC, ID, and IE and the parameters therein. It cites specific references, the sources from which they may be readily acquired, and indicates, where available, approximate costs of the references.

The full texts of the test procedures cited are available for inspection only at the Office of the Federal Register Information Center, Room 8301, 1100 L Street, NW., Washington, D.C. Full texts of non-copyrighted and copyrighted test procedures are available from the sources indicated in § 136.3(b).

To accommodate this new paragraph (b), paragraphs (b) and (c) in the existing regulation have been redesignated as paragraphs (c) and (d), respectively.

**V. Public Participation and Response to Most Significant Comments**

Two hundred and twenty letters, many with attachments of data and other information, were received in response to the Administrator's December 1979 request for comments. Ten additional letters were received in response to the reopened comment period in January 1981. Letters were received from industries, Federal and State agencies, industrial and trade associations, universities, testing laboratories, research institutes, engineering and consulting firms, local government agencies, standards and professional organizations, and from one private citizen.

To facilitate analysis, comments within the letters were classified into three categories—policy, technical, and general, and then into thirteen subcategories. The comments were then reviewed by a technical group of EPA analytical experts and by an EPA policy review group. The record contains a response to the comments, organized on the basis of these subcategories. The final rulemaking today incorporates changes based upon this review. This section highlights significant and generic comments and EPA responses.

In the following paragraphs of this section only issues of major concern will be discussed. Many lesser technical questions or minor issues which were raised are discussed in the "Response to Comments" document contained in the record.

*A. GC, HPLC, and GC/MS Test Procedures*

1. Policy on Applicability of Test Procedures

EPA requested comments on the general applicability of the proposed methods. To present the best state-of-the-art test procedures for analyzing trace organic pollutants in industrial wastewaters, EPA specifically requested commenters to share their experiences and data relative to sensitivity, precision, accuracy, and detection limits of the proposed GC, HPLC, and GC/MS test procedures.

*Comment:* Several commenters felt that the EPA should not publish some of these test procedures (specifically, the GC, HPLC, and the GC/MS) as regulations until they had been fully validated by multi-laboratory studies.

*Response:* No data or information were provided to the EPA in the comments that would permit revisions of the texts of the proposed test procedures to better represent the state-of-the-art, to enhance general applicability, or to preclude interference problems. Consequently, EPA believes these GC, HPLC, and GC/MS test procedures to be the best state-of-the-art test procedures currently available for the analysis of wastewater discharges for the priority toxic organic pollutants.

The multi-laboratory validation studies for Methods 601–602, 604–613, 624, and 625 have been completed and support the general applicability of these test procedures to the analysis of treated wastewater effluents. Since the front-end chemistry of the GC/MS test procedures, i.e., sample preservation, holding times, and purge and trap or methylene chloride extraction, are similar for the GC and the GC/MS test procedures, it would be expected that the precision and accuracy of these test procedures will be substantially the same over the concentration ranges which give linear detector responses. This has been substantiated by the validation studies.

*Comment:* Several commenters felt that GC, HPLC, and GC/MS test procedures did not meet rigorous criteria, such as those of the consensus standards organizations, had not been precisely written, and did not meet the state-of-the-art.

*Response:* The Agency recognizes that these test procedures include some state-of-the-art techniques which have not been as widely used as the consensus methods which were approved and promulgated in 1973 and 1976. But for Methods 601–602, 604–613, 624, 625, 1624, and 1625, method performance is now supported by the multi-laboratory validation studies. These 16 test procedures are now backed by data which meet criteria at least as rigorous as that employed by consensus standards organizations.

Only single-laboratory data are available for 4 parameters (2-chloroethylvinyl ether, 1,2-Dichloropropane, cis-1,3-Dichloropropene, and trans-1,3-Dichloropropene) in Method 601, 2 parameters (acrolein and acrylonitrile) in Method 603, 1 parameter (Hexachlorocyclopentadiene) in Method 612, and 5 parameters (Bromomethane, 2-chloroethylvinyl ether, 1,2-Dichloropropane, cis-1,3-Dichloropropene, and trans-1,3-dichloropropene) in Method 624. It was necessary to resort to single-laboratory testing due to the long-term instability of these parameters in the Youden pair samples used in the multi-laboratory validation studies. In many instances these single-laboratory evaluated test procedures have been more exhaustively tested for their applicability to industrial wastewaters than the currently approved test procedures in these guidelines which are cited from consensus sources.

The procedures promulgated today, as written, most precisely define the current state-of-the-art for the analysis of trace organic compounds in wastewater discharges. No better methods are now available for general use.

*Comment:* Several commenters said that these test procedures had not been demonstrated by the Agency to be generally applicable to all wastewater effluents.

*Response:* These test procedures were developed by single laboratories to be applicable under various conditions of preservation and holding times, to samples taken from wastewaters which were shown in various surveys to have the compounds of interest. Their general applicability to multiple industrial wastewater effluents is now corroborated by the multi-laboratory validation studies, which specifically searched for matrix effects. In specific cases of interference, analysts may seek leave to use alternative procedures under 40 CFR 136.4.

*Comment:* Several commenters suggested that these test procedures be issued as "interim test procedures" without regulatory impact within the limits of their analytical uncertainties.

*Response:* This comment was made prior to the completion of the multi-laboratory validation studies. Acceptable performance for these methods is now verified for analysis of industrial wastewater effluents. Mandatory quality control within the text of the test procedures further strengthens their credible applicability to effluent analysis. When spike or surrogate recoveries are poor (do not fall within the acceptable performance levels for the parameters of concern), the method will not have produced data acceptable for NPDES reporting purposes such as permit applications and discharge monitoring reports demonstrating compliance.

*Comment:* Several commenters felt that the test procedures should be continually reevaluated to ensure they are kept abreast of the state-of-the-art. They requested that the test procedures be re-submitted for comment and peer review after the 20-laboratory validation tests were completed.

*Response:* The Agency decision to conduct the 20-laboratory validation studies was made in the spirit expressed by these commenters. The studies have been completed, and their results are incorporated in the revised texts of these test procedures. These results corroborate the general applicability of the validated test procedures to multiple wastewater effluents. EPA may amend 40 CFR Part 136, if comments or future results of these studies suggest that this is necessary. EPA is accepting comment on the actual calculation of specific control limits.

2. Flexibility and Analysts' Professional Judgment

EPA requested commenters' experiences and opinions on the flexibility that should be incorporated within the GC, GC/MS, and HPLC procedures. Optimum flexibility would render the test procedures most generally applicable to industrial discharges, without seriously compromising data quality.

After careful study, EPA has found most of the comments received in response to this request to have merit and has reacted positively to them.

*Comment:* Several commenters requested that test procedures be revised to allow the analyst maximum leeway to exercise professional judgment to adapt the test procedures to the sample at hand. They felt that defining test procedures in terms of "good laboratory practices" and analytical criteria would allow the incorporation of technological advances not permitted in rigidly structured procedures. The commenters specifically requested flexibility in GC temperature programming and extraction solvent stripping procedures.

*Response:* Where technically justified, the proposed test procedures have been relaxed to allow flexibility for analysts to exercise professional judgment. This flexibility is being incorporated within the text of the GC and HPLC procedures, in recognition of the rapid advances occuring in the state-of-the-art for packed and open tubular columns. A primary configuration of GC or HPLC components is described within the test procedure. However, to optimize the applicability of the chromatographic technique to analytical requirements unique to specific discharges, the analyst is allowed to use professional judgment in selecting packed or open tubular columns, operating temperature programs, carrier gas or solvent flow rates, and detectors. Analysts may also use their discretion in selecting cleanup procedures. EPA has also relaxed the strict protocol for sample extract concentration. In the proposed test

procedures, sample extracts were concentrated by means of a Kuderna-Danish concentrator, which consists of a concentrator tube, an evaporative flask, and a Snyder column. Many studies comparing the Kuderna-Danish concentration technique to a variety of others have provided no statistically conclusive support for the rigid protocol for most materials tested. Accordingly, within the scope of the GC, HPLC, and GC/MS test procedures, analysts are permitted some discretion in selection of concentration techniques.

*Comment:* Other commenters said these analytical criteria for the test procedures should be in terms of mandatory verification and quality control procedures.

*Response:* The EPA agrees. With the added flexibility in test procedures for analyst discretion, an added responsibility is borne by the analyst for mandatory quality control and validation procedures. These requirements have long been recognized as elements of good analytical laboratory practices. Accordingly, Methods 601–613, 624, and 625 now incorporate verification and quality control and the criteria for reportable analytical results for regulatory purposes.

*Comment:* Many of the commenters were concerned with the costs associated with rigidly prescribed methods, especially where required use of several such methods would call for significant capital expenditures in highly specialized equipment.

*Response:* The flexibility which has been incorporated into the test procedures is expected to encourage rapid incorporation of technical advances, and to allow the dischargers to select the most cost-effective analytical options when acquiring major analytical equipment. However, any modifications of Methods 601–613, 624, 625, 1624, and 1625, which are not expressly permitted in the text of the methods as given in Appendix A to this part, will be considered major modifications and must be approved as alternate test procedures under the provisions of §§ 136.4 and 136.5.

Sample preservation and holding times have also been standardized to allow the same sample to be used for more than one method. This flexibility allows monitoring costs to be minimized and existing equipment to be fully utilized. Additionally, contract laboratory services are available when the analytical load of a discharger does not justify purchase of specialized analytical equipment (See cost discussion below).

*General Response to Comments on Flexibility and Analysts' Professional Judgment:* Flexibility is permitted only in discretionary elements of the chromatographic test procedures. Changes in non-discretionary elements of the test procedures are outside the scope of the flexibility provision. Change may be made only when conditions of analytical equivalency are demonstrated within the provisions of §§ 136.4 and 136.5 of this regulation. For all discretionary changes which analysts may make, their laboratory records should corroborate that the quality of the data generated meets all stated performance criteria of precision and accuracy. (See 40 CFR 122.21, 122.41, 122.44 and 403.12).

3. Quality Control and Quality Assurance

This issue is integrally related to the preceding discussion of allowable flexibility. The availability of rigorous quality control criteria is what allows the flexibility now built into the methods. Conversely, the allowance for modification based on professional judgment makes it essential that there be a procedure for assuring that those modifications do not undermine the quality of reported data.

EPA proposed, within the test procedures, that method blanks should be processed each time a set of samples was to be extracted, or whenever there was a change in reagents. This was to safeguard against chronic laboratory contamination. EPA also proposed that standard quality assurance practices be used. The practices proposed included field replicates to validate the precision of the sampling technique, laboratory replicates to validate the precision of the analysis, and special (or fortified) samples to validate the accuracy of the analysis. If doubt existed in the identification of any peak in a gas chromatogram, the proposed quality assurance required a confirmation by use of a technique such as MS. In addition, Appendix III of the proposal required an intensive quality control plan for the GC/MS test procedures. The Agency requested comments on the proposed levels of quality assurance/quality control (QA/QC) and on any additional levels that should be specified in the test procedures or left to the discretion of the analyst.

*Comments:* Most commenters, including several large industrial associations, restricted their comments to the operational details of the suggested programs, thus implying acceptance of the concept of the mandatory quality control procedures. Others specifically urged adoption of such procedures.

Some commenters addressed the issue of appropriate costs and number of samples that should be analyzed for continuing QA/QC. One corporation suggested that 10%–20% of cost was reasonable, with one control sample for 10 samples analyzed. A trade association stated the QA/QC should not exceed 20% of the analytical costs. Another corporation suggested 10% spiked samples and duplicate analysis of all purgeable samples. A permitting authority suggested that 15%–20% QA/QC was reasonable.

Some commenters specified that mandatory QC should be an integral part of all of the proposed organic test procedures. There were no objections to the required analysis of four spiked reagent water samples, as proposed in Appendix III.

*Response:* The comments have confirmed EPA's belief that a reasonable level of mandatory quality assurance should be incorporated in all of the test procedures for organic priority pollutants to assure that data of known and acceptable quality are produced.

The analyst must demonstrate correct use of the method, reagents, and equipment before analyzing samples. Users of Methods 601–613, 624, 625, 1624, and 1625 must demonstrate their ability to generate data of acceptable quality by analyzing four spiked aliquots of reagent water containing each parameter of interest, and meeting performance specifications based upon the multiple-laboratory methods studies completed by the Agency. For a limited number of parameters discussed earlier, the performance specifications are based upon other laboratory test data. Reagent water is used to ensure that there are no matrix effects in this preliminary test.

On-going quality assurance for Methods 601–612 consists of mandatory spiking of 10% of all samples (spiking with the compounds being measured) and comparing the degree and precision of spike recovery with the test procedure criteria established in the multiple-laboratory methods studies. This will lead to a QA/QC cost in the range of 10% of the analytical costs. For Method 613, samples must be spiked with isotopically labeled 2,3,7,8-TCDD internal standards. For Methods 624 and 625 the requirement is for a 5% mandatory spiking of samples. See Section IV B above.

Users of these methods are required to spike all samples with surrogates. These QA/QC procedures are

essentially the same as those proposed except that criteria are now specified in more detail and specific control limits are based upon multiple-laboratory studies. If the recovery of any parameter falls outside the control limits, the laboratory performance for that parameter is inconsistent with control, and the analytical results for that parameter in the unspiked sample are suspect and may not be reported for regulatory compliance purposes.

If the performance criteria for the GC, HPLC, and GC/MS test procedures which have been reprinted in full text in Appendix A of this part cannot be attained in a specific wastewater matrix, the analyst may consult the Director of EPA's Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, for technical assistance. If the method must be modified beyond its allowable flexibility to meet the performance criteria, the procedures of § 136.4(b) are available.

Although these requirements for quality assurance and control are minimum EPA standards, analysts are expected to perform significantly better in almost all cases. They are also encouraged to participate in additional performance testing to assure continued analytical proficiency.

*B. ICP Method*

EPA proposed the ICP instrumental test procedure as an alternative to the approved test procedures for elemental analysis (such as the AA and colorimetric test procedures). In particular, comments were solicited on the general applicability of the ICP test procedure to industrial discharges.

*Comment:* Some commenters thought that the detection limits given in the test procedures were unrealistic. They suggested that industrial samples would need to be concentrated before such limits could be achieved.

*Response:* The detection limits given in Table 1 of the ICP test procedure were taken from an EPA publication (EPA–600/4–79–017, "Inductively Coupled Plasma—Atomic Emission Spectroscopy—Prominent Lines"). These detection limits are intended as guides for instrumental limits and are published for informational purposes. The method performance in terms of precision and accuracy were determined at different concentrations of analytes in distilled water in a multi-laboratory test. These are reported in Table 4 in the ICP test procedure and are the performance standards which are required by section 12 of the test procedure for instrument quality assurance.

*Comment:* Some commenters thought that the ICP test procedure was not equivalent to the AA test procedure, and especially to the furnace AA test procedure.

*Response:* The approved furnace AA test procedure can be used to measure concentrations lower than the ICP technique can measure. Data filed with the EPA show the equivalency of the AA and ICP test procedures across the concentration ranges that are common to the two procedures. The common ranges include the concentrations which are of regulatory concern. These comparisons were made over a number of industrial discharges and a broad concentration range.

*Comment:* Several commenters asked for the approval of the direct current plasma (DCP) atomic emission spectrometric method as equivalent to the ICP and AA test procedures, and requested its inclusion in Table IB as an approved test procedure for elemental analysis.

*Response:* EPA did not have data to show the applicability of the DCP technique to wastewater analysis and accordingly did not propose it as an alternate test procedure for elemental analysis. Data showing applicability to wastewaters were not made available by the commenters, although data were presented to show the applicability of DCP to sample matrices other than wastewater. Because of this incomplete data base, EPA was unable to include DCP in the list of approved test procedures.

Recently, a DCP manufacturer has provided such data to EPA for approval thorugh the equivalency provisions of §§ 136.4 and 136.5, and EPA is currently considering approval of the DCP test procedure for the analysis of trace elements in wastewater.

*Comment:* Several commenters had difficulty understanding the guidance given for background corrections and for high dissolved solids (salt buildup) interferences.

*Response:* When using the ICP procedure it is important to make background corrections. The analyst must determine and incorporate into the analysis the valid correction factors for spectral interferences caused by high dissolved solids concentrations. Section 2.1 of the ICP test procedure has been revised to make this clear. Additional guidance has also been given to correct for salt buildup at the tip of the nebulizer.

*Comment:* Some commenters thought that the suggested combinations of elements in stock solutions were ambiguous.

*Response:* The mixed standard solutions suggested by the EPA in the test procedure were found to be compatible mixtures. Other combinations may be used within the scope of the test procedure, as long as precipitations and other chemical reactions do not occur. The test procedure text has been revised to make this clear.

*Comment:* Some commenters felt that other wavelengths of light which were also emitted in the characteristic spectrum of the element should be permitted.

*Response:* Other wavelengths may be used when those given in the test procedure are obscured. In using them, the analyst must demonstrate that they are free from interfering wavelengths or that interference results can be corrected.

*Comment:* Several commenters presented data and suggested that antimony and thallium should be included within the scope of the ICP test procedure.

*Response:* Antimony and thallium are priority pollutants and the EPA agrees they should be included within the scope of the ICP test procedure since the submitted data show these metals can be recovered in excess of 90% from spiked samples using the ICP test procedure.

*Comment:* Several commenters requested sources from which verified outside samples could be obtained.

*Response:* Verified outside check samples are necessary for testing the analytical system. Upon request, the Director of EPA's Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, will either supply such samples or provide information on where quality control check samples can be obtained.

*Comment:* Several commenters found errors in the tables of reference wavelengths.

*Response:* Suggested corrections of errors in the tables and various other parts of the ICP test procedure text have been made.

*Comment:* Several commenters questioned the estimated costs for analyses made by the ICP test procedure, relative to other approved test procedures.

*Response:* Cost estimates for analysis by the ICP test procedure were given only as general comparisons with the costs of the other alternate test procedures in Table IB, which are still approved for trace metal analyses. The analyst may exercise professional judgment in selection of the approved alternate test procedure from Table IB which best meets his analytical needs. If the ICP instrument system is available to the analyst, it may well be the most

cost-effective approved alternate test procedure, especially if large numbers of samples containing several of the ICP-approved metals are to be analyzed. Otherwise, the AA or one of the other approved alternate test procedures may prove to be more cost-effective. The estimates given for the ICP analyses are average contract charges experienced by the EPA for multi-element analyses of large lots of samples.

*C. ($CBOD_5$) Method*

The Agency proposed the $CBOD_5$ test procedure in response to requests from environmental analysis laboratories to measure the carbonaceous BOD of wastewater without the complications caused by the nitrogenous oxygen demand.

*Comments:* EPA requested comments and additional data on the control of nitrification in BOD measurements. Many letters were received, several accompanied by extensive test data. A majority of the letters expressed confusion over the use of the $CBOD_5$ versus the traditional 5-day BOD test. None of the commenters questioned the test procedure.

*Response:* The $CBOD_5$ method (Parameter No. 14, Table IB) is being added to the list of approved measurements. This method is new, and should not to be confused with the traditional 5-day BOD test which is listed in Table IB as Parameter No. 9. The nitrogen inhibitor is not a procedural option, rather $CBOD_5$ must be designated as the pollutant or effluent limitation that is measured to report the $CBOD_5$ parameter.

A discharger whose permit requires reporting the traditional 5-day biochemical oxygen demand (Parameter No. 9, Table IB) may not use a nitrogen inhibitor in the procedure for reporting results. Only when a discharger's permit explicitly states that $CBOD_5$ is the parameter whose monitoring is required may the permittee report data using the $CBOD_5$ nitrogen inhibited analytical method.

*D. Table II: Required Containers, Preservation, and Holding Times*

EPA proposed mandatory preservation techniques, container materials, and holding times for industrial wastewater samples. It also proposed a mechanism by which a permit holder or analytical laboratory could obtain a variance from these requirements if justified by data. The permittee or analyst was also limited to shorter holding periods, if this was necessary to maintain sample stability. In the proposed guidelines, these were paragraph 136.3(d) and footnote 4 to Table II. These provisions are now contained in paragraph 136.3(e) and footnote 3 of Table II.

*Comment:* Some commenters thought that Table II was too detailed and should not be included in the text of the regulation. They suggested that EPA make it available under a separate cover.

*Response:* The EPA disagrees. EPA continues to believe that the integrity of the sample is critical to the quality of monitoring data. It is best presented and publicized as a requirement through inclusion in the regulation.

*Comment:* Several commenters felt that preservation techniques, holding times, and container materials should be presented as recommendations only. They thought the diversity of wastewater samples and sampling requirements could best be judged by the professional taking the sample.

*Response:* EPA agrees that the judgment of the professional taking the sample is critical to the integrity of the sample. However, after the sample is taken, sample integrity is maintained and verified only by control of container materials, preservation procedures, and holding times. Only with verifiable sample integrity can analytical data be correlated to the sample source. EPA's evaluation of NPDES monitoring practices has confirmed that inadequate sample preservation and failure to observe recommended holding times are major analytical problem areas. For these reasons EPA has included Table II in the mandatory text of this regulation.

*Comment:* Other commenters said that there should be more standardization of the container, holding time, and preservation requirements between parameters in order to minimize the number of samples that needed to be taken.

*Response:* EPA agrees that this is desirable. It is, however, secondary to quality assurance. Table II requirements have now been standardized to a significant degree. This minimizes the number of preserved samples that need to be taken.

*Comment:* Several commenters felt that there were stable wastewater sample types that would not require adherence to the mandatory requirements of Table II.

*Response:* When a permit applicant knows that Table II requirements do not apply to his wastewater—and he has data to substantiate this conclusion—the variance provisions of paragraph 136.3(e) and footnote 3 of Table II allow less stringent requirements to prevail. Such variances must be obtained from the Regional Administrator under these provisions.

*General Response:* Many commenters provided data or information relating to specific requirements of Table II. The changes made in Table II in response to these comments were summarized earlier in Exhibits 2, 3, and 4 in section 4(e) of this preamble.

*E. Cost Estimates for Methods*

EPA is concerned with the costs of analyses required by the new regulations, because of their overall importance to programs under the Clean Water Act. In the December 3, 1979, **Federal Register**, the Agency solicited comments on these costs and listed EPA's typical costs for Carbonaceous BOD, ICP, GC, HPLC, and the GC/MS analyses.

*Comments:* Several comments from the public addressed issues such as the high cost of routine analysis, the appropriateness of the figures presented by EPA, and the variations in cost experienced, depending upon lot size, throughput, and complexity of the analyses performed. Nearly all of the comments received were directed toward organic analyses and the related requirements for capital equipment. Several comments also seemed to rely on the erroneous assumption that the proposed methods would require separate samples for each procedure.

*Response:* EPA noted a wide range of unit costs cited, based largely on volume. These regulations do not require that each permittee perform its own analyses. When the small volume of analytical loads makes unit costs less economical, the permittee may use commercial laboratories, where analytical work loads can be maintained at levels which optimize costs.

EXHIBIT 5.—EPA CURRENT EXPENDITURES FOR WATER AND WASTEWATER ANALYSIS FOR PRIORITY POLLUTANTS

| | Cost |
|---|---|
| 113 Organic Compounds by GC/MS (includes field & laboratory blanks) | [1] $600.00 |
| Each Replicate Sample for 114 Organics by GC/MS | [1] 600.00 |
| Each Priority Pollutant Spiked Sample for 114 Organics by GC/MS (with data calculations) | [1] 660.00 |
| 13 Elemental Compounds by AA-Flame or Flameless | [2] 130.00 |
| ICP—Up to 21 Selected Elements | [2] 30.00 |
| Cyanide, EPA Standard Method—Cyanide Amendable to Chlorination or Free Cyanide | [1] 4.50 |
| Asbestos (transmission electron microscope analysis) | [3] 332.00 |
| GC (Average of costs for methods 601-612) | [4] 100.00 |

[1] Based upon actual prices per sample offered to the Agency for 200-sample bid lots.
[2] Based upon actual prices offered to the Agency for 750 ICP and 6500 AA sample bid lots.
[3] Based upon actual prices offered per sample to the Agency for 250-sample bid lots.
[4] Based upon actual prices per sample for 5400 samples (900 volatile, 4500 semi-volatile), averaged for three contracts with the Agency.

The Agency has compiled the data presented in Exhibit 5 from actual (lowest) contract bids, obtained through a competitive bidding process. As shown in the footnotes, they were based upon lots of specific sizes and on sample throughputs of approximately 200 samples per month for GC/MS and transmission electron microscope (for asbestos), and 100 per month for elemental analyses. The table costs were constructed on the basis of survey analysis. Permittees may expect somewhat lower costs if they are only analyzing for certain specific parameters.

A commenter making a commercial quotation places the cost of a quality assured analysis of the 111 currently designated organic toxic pollutants at $500 per sample. However, comparison of the Agency's and other commenters' cost data has generally been impossible, since the Agency has not known the factors on which the various commenters' cost estimates were based.

The Agency has estimated analytical costs in other regulations which implement various NPDES monitoring requirements of the Clean Water Act. Analytical costs were estimated in the preambles of the Consolidated Permits Regulations (45 FR 33290, May 19, 1980) and NPDES Regulations (44 FR 34408, June 14, 1979), which are consistent with the estimates given in Exhibit 5. Analytical cost estimates consistent with those given above have also been included in promulgations of effluent limitations guidelines and pretreatment standards for individual industrial categories.

While EPA agrees with many commenters that less costly procedures would be highly desirable, the proposed methods appear to be the most cost-effective currently available. These cost estimates include 4 spiked reagent water analyses and 10% spiked sample analyses. Ongoing research by this Agency and others should produce less expensive approaches that, with testing, will prove equivalent or better, especially for analyzing trace organic toxic pollutants in industrial and municipal wastewater.

To encourage development of new, improved, and less costly analytical technologies, the Agency, by establishing the Equivalency Program (see § 136.4 and 136.5), has provided a mechanism for their rapid approval and use of new procedures.

*F. Publication of Full Texts of Test Procedures*

*Comments:* Several commenters thought that it was inappropriate for the EPA to publish the full texts of test procedures within these guidelines. They felt that the Agency had reversed its past policy to "incorporate test procedures by reference" into these regulations.

*Response:* When test procedures are readily available from other sources, EPA will incorporate them by reference. However, test procedures for routine analysis of trace organic pollutants, with the exception of a few pesticidal organic chemicals, were unavailable to meet the analytical requirements for controlling priority organic pollutants. EPA developed the proposed test procedures through a high-priority research effort. For the convenience of the public and to facilitate timely comment, EPA published the full texts of the draft procedures in the preamble of the proposed regulation. In order to continue the convenience, and to ensure that the final methods are as widely distributed as the proposed methods, EPA is including, as an appendix to this amendment, the full text of the approved, Agency developed, test procedures for analysis of the priority pollutants. It is possible that in the future full text of these test procedures will become available from other sources.

In general, the text of approved test procedures will not be published in the **Federal Register** when they are readily available from other sources. EPA will continue to "incorporate approved test procedures by reference" in these guidelines as it has in the past. Methods are clearly incorporated by reference in § 136.3(a) and a new § 136.3(b) has been added to clearly identify the sources of these references. This should resolve the commenter's concerns.

*G. Consistency of Analytical Methods Approved Under Different Acts*

*Comments:* Several commenters were concerned that EPA was requiring the regulated community to use two different test procedures for analyzing similar or identical compounds, one set under the Safe Drinking Water Act, and another in these Guidelines under the Clean Water Act. They concluded that the cost of compliance with these various requirements unduly burdened the "rate payer" by what appeared to be duplication.

*Response:* EPA makes every effort to ensure that the analytical methods prescribed under its regulations are proliferated only when necessary. Regulations issued under different Acts are applicable to the matrices defined by those Acts. Where analytical methods are prescribed in these regulations, consideration must be given to the concentration ranges that must be measured and the interferents to be expected.

Interferents may be unique to a matrix or may be common to several matrices covered by these Acts and regulations. Whenever possible, within the information and data available to the Agency, the same methods are approved for measuring the same parameters in the different matrices. There are many instances where this is not possible. For example, for drinking water analyses both a purge and trap procedure (Method 501.1) and an alternate solvent extraction procedure (Method 501.2) are approved for the analysis of the trihalomethanes (THM). In drinking water, interferents for THM analysis are minimal. If the drinking water extraction procedure were applied to wastewater samples for the purgeable compounds, extracts which result would present much more formidable analytical problems than would have been the case if the purge and trap procedure had been used. Therefore, it is not possible to approve the drinking water THM test procedures for the analysis of THMs and related purgeable pollutants in wastewater samples. On the other hand, Method 601 can easily be used for analysis of THMs in drinking water.

**VI. Regulatory Analyses**

(a) Under Executive Order 12291, EPA must judge whether a regulation is "major" and therefore subject to the requirement of a "Regulatory Impact Analysis." This regulation is not major for the following reasons: (1) It only prescribes analytical methods and sample handling requirements that ensure a uniform measure of pollutants across all wastewater discharges within minimum acceptance criteria. It does not require that analyses actually be made. The purpose is to ensure that the quality of environmental monitoring data meets certain minimum standards.

(2) The impact of this regulation will be far less than $100 million.

(a) The regulation affects unit monitoring costs for other regulatory programs, e.g., effluent guidelines regulations and the implementation regulations of the National Pollutant Discharge Elimination System (NPDES), and the pretreatment programs. However, it does not impose those costs. In fact, the monitoring costs for other programs are considered in each other rule-making. This is appropriate because total (rather than unit) monitoring costs are determined by the monitoring provisions of those other regulations.

(b) This regulation has deliberately provided approval of several analytical options for most compounds. This often

allows NPDES permittees to select the option that is best suited to their particular monitoring requirements and that will minimize their monitoring costs. In addition, the approval of the $CBOD_5$ analysis will facilitate modification of CWA permits to allow treatment works and their control authorities to focus on the best measure of oxygen demand, thereby achieving treatment economies that will reduce costs of treatment significantly.

(c) Further, through the equivalency provisions, these test procedure guidelines have been designed to encourage the development of innovative analytical methods by the private sector and to encourage the competitive viability of the instrument manufacturing industry. The equivalency provision also allows individual dischargers to gain approval of analytical systems of their own design that may futher reduce their total monitoring costs.

(3) The impact of compliance with these regulations will not be concentrated on any particular sectors of American industry.

This regulation was submitted to the Office of Management and Budget (OMB) for review as required by Executive Order 12291. Any comments from OMB to EPA and any EPA response to those comments will be available for public inspection at the Public Information Reference Unit, Room M2904 (EPA Library—Rear), TM–213, Environmental Protection Agency, 401 M Street, SW., Washington, D.C. 20460, Phone: (202) 382–5926, Office Hours 8:00 a.m. to 4:30 p.m.

(b) Under the Regulatory Flexibility Act, 5 U.S.C. 601 et seq., EPA is required to determine whether a regulation will significantly affect a substantial number of small entities so as to require a regulatory analysis. The regulation requires no new reports beyond those already now required. The analytical techniques approved here either can be handled by small facilities, or are widely available by contract at a reasonable price. Therefore, in accordance with 5 U.S.C. 605(b), I hereby certify that this rule will not have a significant adverse economic impact on a substantial number of small facilities.

(c) The equivalency information provision in this rule has been submitted for approval to the Office of Management and Budget (OMB) under the Paperwork Reduction Act of 1980, 44 U.S.C. 3501 *et seq.* It is not effective until OMB approves it and a technical amendment to that effect will be published in the **Federal Register.**

## VII. Effective Dates

The effective date of this rule is January 22, 1985 for all methods except the method for $CBOD_5$. This date, 90 days hence, was chosen to allow analysts sufficient time to learn of the new methods and to implement the necessary changes in laboratory practices. After January 24, 1985, data reported to EPA must be generated using the methods approved under 40 CFR Part 136, including these methods added by today's amendment.

An effective date of 30 days, November 26, 1984, was chosen for using the method for $CBOD_5$. This is the only method for a new parameter. Treatment works and permitting authorities are eager to begin measurement and analysis for this new parameter because it may result in achieving treatment economies and reduce costs significantly. Although data reported to permitting authorities or EPA on this parameter must be generated using this method after 30 days, there is no requirement that any treatment works monitor for this parameter or use the new method until a treatment works applies for and receives a permit modification. Permits may be modified after EPA's amendment to the secondary treatment rule is effective. The amended secondary treatment rule should be effective before or on the same day that today's rule approving the $CBOD_5$ method is effective. There is no reason why EPA should postpone the effective date of this rule for more than 30 days and possibly delay use of this method and parameter for persons whose permit can be changed quickly.

The Director of the **Federal Register** has approved all materials which are "incorporated by reference" in the text of the regulation to be incorporated. They are incorporated by reference on the effective dates given above.

## List of Subjects in 40 CFR Part 136

Water Pollution Control; Incorporation by reference.

Dated: September 26, 1984.

**William D. Ruckelshaus,**

*Administrator.*

## Regulation

For the reasons set out in the Preamble, Part 136, Chapter 1, Subchapter D of Title 40, Code of Federal Regulations, is amended as set forth below. Only new provisions are being promulgated at this time; unchanged provisions are reprinted for the purpose of clarity.

1. The authority citation for Part 136 is revised to read as follows:

**Authority:** Sections 301, 304(h), 307 and 501(a), Pub. L. 95–217, 91 Stat. 1566, et seq. (33 U.S.C. 1251, et seq.) (the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

2. In paragraphs 136.1 (a) and (c), reference to the Federal Water Pollution Control Act Amendments of 1972 (FWPCA) is replaced by reference to the Clean Water Act of 1977, and applicability under paragraph 136.1(a) is extended to Parts 122–125 of Title 40. As revised, paragraphs (a) and (c) of § 136.1 read as follows:

**§ 136.1 Applicability.**

* * * * *

(a) An application submitted to the Administrator, or to a State having an approved NPDES program for a permit under § 402 of the Clean Water Act of 1977, as amended (CWA), and/or to reports required to be submitted under NPDES permits or other requests for quantitative or qualitative effluent data under Parts 122 to 125 of Title 40, and,

* * * * *

(c) Certifications issued by States pursuant to § 401 of the CWA, as amended.

3. In § 136.2, paragraph (a) is revised to reference the Clean Water Act of 1977, and paragraphs (f), (g), and (h) are removed, and a new paragraph (f) is added defining "Detection limit," and paragraphs (g) and (h) are reserved, as follows:

**§ 136.2 Definitions.**

As used in this part, the term:

(a) "Act" means the Clean Water Act of 1977, Pub. L. 95–217, 91 Stat. 1566, et seq. (33 U.S.C. 1251 et seq.) (The Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

* * * * *

(f) "Detection limit" means the minimum concentration of an analyte (substance) that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero as determined by the procedure set forth at Appendix B of this Part.

(g) [Reserved.]

(h) [Reserved.]

4. In § 136.3, Table I is restructured into five new tables by transferring the biological parameters formerly designated as parameters 4, 5, 6, 7 and 8 to new Table IA, entitled "List of Approved Biological Test Procedures," by adding an additional EPA reference to the approved test procedures, and by updating the references to Standard Methods and USGS: by transfering the inorganic parameters formerly designated as parameters 1–3, 10–13, 15–

93, 96–98, and 104–115 to new Table IB, entitled "List of Approved Inorganic Test Procedures", adding two new inorganic parameters, Carbonaceous Biochemical Oxygen Demand ($CBOD_5$) and Nitrate-Nitrite, including an additional test procedure based upon the inductively coupled plasma technique in Table IB for 25 of the metal parameter designations, by including 10 methods approved under the equivalency provisions of §§ 136.4(d) and 136.5(e), and updating references to EPA, Standard Methods, ASTM, AOAC and USGS test procedures; by deleting former parameter 14 (Chlorinated organic compounds) and by entering the individual chlorinated organic compounds into new Table IC, entitled, "List of Approved Test Procedures for Non-Pesticide Organic Compounds", transferring old parameters 9 (Benzidine) and 94 (Pentachlorophenol) to Table IC, by including the 78 additional proposed non-pesticidal organic parameters and by adding 17 new test procedures in Table IC; by deleting former parameter 95 (Pesticides) and by entering the 68 individual pesticides into new Table ID, entitled "List of Approved Test Procedures for Pesticides", by including the 2 additional proposed pesticide parameters, and the two new test procedures in Table ID; and by transferring the former radiological parameters 99–103 to new Table IE, entitled "Approved Radiological Test Procedures", adding an EPA reference to the approved test procedures, and updating the Standard Methods, ASTM and USGS references. As revised, Table I reads as follows:

**§ 136.3 Identification of Test Procedures.**

* * * * *

TABLE IA.—LIST OF APPROVED BIOLOGICAL TEST PROCEDURES

| Parameter and units | Method [1] | EPA [2] | Reference (Method Number or Page) | | |
|---|---|---|---|---|---|
| | | | Standard Methods 15th Ed. | ASTM | USGS |
| Bacteria: | | | | | |
| 1. Coliform (fecal) number per 100 ml | MPN, 5 tube, 3 dilution; or, membrane filter (MF) [4], single step | p. 132 | 908C | | |
| | | p. 124 | 909C | | B–0050–77 |
| 2. Coliform (fecal) in presence of chlorine number per 100 ml | MPN, 5 tube, 3 dilution | p.132 | 908C | | |
| 3. Coliform (total, number per 100 ml | MPN, 5 tube, 3 dilution; or, MF [4] single step or two step | p. 114 | 908A | | |
| | | p. 108 | 909A | | B–0025–77. |
| 4. Coliform (total) in presence of chlorine, number per 100 ml | MPN, 5 tube, dilution; or MF [4] with enrichment | p. 114 | 908A | | |
| | | p. 111 | 909 (A + A.5c) | | |
| 5. Fecal streptococci, number per 100 ml | MPN, 5 tube, 3 dilution; MF [4]; or, plate count | p. 139 | 910A | | |
| | | p. 136 | 910B | | B0055–77. [5] |
| | | p. 143 | 910C | | |

Table IA Notes

[1] The method must be specified when results are reported.

[2] "Microbiological Methods for Monitoring the Environment, Water and Wates, 1978", EPA–600/8–78–017, U.S. Environmental Protection Agency.

[3] Greeson, P.E., *et al.*, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, "U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A4, Laboratory Analysis, 1977.

[4] 0.45 um membrane filter or other pore size certified by the manufacturer to fully retain organisms to be cultivated, and free of extractables which could interfere with their growth and development.

[5] Approved only if dissolution of the KF Streptococcus Agar (Section 5.1, USGS Method B–0055–77) is made in a boiling water bath to avoid scorching of the medium.

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES

| Parameter, units, and method | Reference (method No. or page) | | | | |
|---|---|---|---|---|---|
| | EPA 1979 | Standard methods 15th Ed. | ASTM | USGS [1] | Other |
| 1. Acidity, as $CaCO_3$, mg/L: Electrometric end point or phenolphthalein end point. | 305.1 | 402(4.d) | D1067–70(E) | | |
| 2. Alkalinity, as $CaCO_3$, mg/L: Electrometric or colorimetric: | | | | | |
| Titration to pH 4.5, manual | 310.1 | 403 | D1067(B) | I–1030–78 | P. 548. [2] |
| Or automated | 310.2 | | | I–2030–78 | |
| 3. Aluminum—Total [3] mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration | 202.1 | 303C | | I–3051–78 | |
| AA furnace | 202.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7. [4] |
| Or colorimetric (Eriochrome cyanine R) | | 306B | | | |
| 4. Ammonia (as N), mg/L: Manual distillation [5] (at pH 9.5): | | | | | |
| Followed by | 350.2 | 417A | | | |
| Nesslerization | 350.2 | 417B | D1426–79(A) | I–3520–78 | P. 553. [2] |
| Titration | 350.2 | 417D | | | |
| Electrode | 350.3 | | D1426–79(D) | | |
| Automated phenate, or | 350.1 | 417F | D1426–79(C) | I–4523–78 | |
| Automated electrode | | | | | [6] |
| 5. Antimony—Total [3], mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration | 204.1 | 303A | | | |
| AA furnace, or | 204.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7. [4] |
| 6. Arsenic—Total [3], mg/L: | | | | | |
| Digestion [3] followed by | 206.5 | | | | |
| Hydride | 206.3 | 303E | D2972–78(B) | I–3062–78 | |
| AA furnace | 206.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7. [4] |
| Or, colorimetric (SDDC) | 206.4 | 307B | D2972–78(A) | I–3060–78 | |
| 7. Barium—Total [3], mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration | 208.1 | 303C | | I–3084–78 | |
| AA furnace, or | 208.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7. [4] |
| 8. Beryllium—Total [3], mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration | 210.1 | 303C | D3645–78 | I–3095–78 | |
| AA furnace | 210.2 | 304 | | | |

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

| Parameter, units, and method | Reference (method No. or page) | | | | |
|---|---|---|---|---|---|
| | EPA 1979 | Standard methods 15th Ed. | ASTM | USGS [1] | Other |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Or colorimetric (aluminon) | | 309B | | | |
| 9. Biochemical oxygen demand ($BOD_5$), mg/L: | | | | | |
| Winkler (Azide modification) | 405.1 | 507 | | I-1578-78 | P. 17.[6] |
| Or electrode method | | | | | P. 548.[3] |
| 10. Boron—Total, mg/L: | | | | | |
| Colorimatic (curcumin) or | 212.3 | 404A | | I-3112-78 | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| 11. Bromide, mg/L: Titrimetric | 320.1 | | D1246-77(C) | I-1125-78 | P. 544.[3][6] |
| 12. Cadmium—Total [3], mg/L: Digestion[5] followed by: | | | | | |
| AA direct aspiration | 213.2 | 303A or 303B | D3557-78 (A or B) | I-3135-78 or I-3136-78 | Pg. 557.[3] |
| AA furnace | 213.2 | 304 | | | P. 37.[6] |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Voltametry[8] or | | | D3557-78(C) | | |
| Colorimetric (Dithizone) | | 310B | | | |
| 13. Calcium—Total [3] mg/L: Digestion[5] followed by: | | | | | |
| Atomic absorption | 215.1 | 303A | D511-77(C) | I3152-78 | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Or EDTA titration | 215.2 | 311C | D511-77(B) | | |
| 14. Carbonaceous Biochemical oxygen demand ($CBOD_5$), mg/L: Winkler (Azide modification) or electrode method with nitrification inhibitor. | | 507(5,e,6) | | | |
| 15. Chemical oxygen demand (COD), mg/L: | | | | | |
| Titrimetric colorimetric | 410.1 | 508A | D1252-78 | I-3560-78 | P. 550[3] and |
| Manual or | 410.2 | | | I-3562-78 | P. 17[6] and |
| | 410.3 | | | I-3561-78 | ([10].) |
| Automated | 410.4 | | | | |
| Spectrophotometric | | | | | ([11]) |
| 16. Chloride, mg/L: | | | | | |
| Titrimetric (silver nitrate) or | | 407A | D512-67(B) | I-1183-78 | |
| Mercuric nitrate | 325.3 | 407B | D512-67(A) | I-1184-78 | P. 554.[3] |
| Colorimetric (ferricyanide) manual or | | | D512-67(C) | I-1187-78 | |
| Automated | 325.1 or 325.2 | 407D | | I-2187-78 | |
| 17. Chlorine—Total residual, mg/L: | | | | | |
| Titrimetric-amperometric [12] | 330.1 | 408C | D1253-76(A) | | |
| Starch end point | 330.2 | 408B | | | |
| Iodometric or | 330.3 | 408A | D1253-76(B) | | |
| DPD-FAS | 330.4 | 408D | | | |
| Spectrophotometric, DPD; or | 330.5 | 408E | | | |
| Electrode | | | | | ([87].) |
| 18. Chromium VI dissolved, mg/L: 0.45 micron filtration with: | | | | | |
| Extraction and atomic absorption, or | 218.4 | 303B | | I-1232-78 | |
| Colorimetric (Diphenylcarbazide) | | | | I-1230-78 | |
| 19. Chromium—Total [3], mg/L: | | | | | |
| Digestion [3] (optional extraction) followed by | 218.3 | | | | |
| AA direct aspiration | 218.1 | 303A or 303B | D1687-77(D) | I-3236-78 | P. 557.[3] |
| AA furnace | 218.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Or colorimetric (Diphenylcarbazide) | | 312A | D1687-77(A) | | |
| 20. Cobalt—Total [3], mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration | 219.1 | 303A or 303B | D3558-77 (A or B) | I-3240-78 or I-3239-78 | P. 37.[6] |
| AA furnace, or | 219.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| 21. Color, platinum Cobalt units or dominant wavelength hue, luminance, purity: | | | | | |
| Colorimetric, ADMI | 110.1 | 204D | | | ([13].) |
| Platinum cobalt; or | 110.2 | 204A | | I-1250-78 | |
| Spectrophotometric | 110.3 | 204B | | | |
| 22. Copper—Total [3], mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration | 220.1 | 303A or 303B | D1688-77 (D or E) | I-3271-78 or I-3270-78 | P. 557[3] and P. 37.[6] |
| AA furnace | 220.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Colorimetric (Neocuproine) | | 313B | D1688-77(A) | | |
| Bicinchoninate | | | | | ([14].) |
| 23. Cyanide—Total mg/L: | | | | | |
| Manual distillation with $MgCl_2$ | 335.2 | 412D | | | |
| Followed by titrimetric | 335.2 | 412B | | | P. 22.[6] |
| Manual or | 335.2 | 412C | D2036-75(A) | | |
| Automated [15] spectrophotometric | 335.3 | 412D | D2036-75(A) | I-3300-78 | |
| 24. Cyanide amenable to chlorination, mg/L: Manual distillation with $MgCl_2$: Followed by titrimetric, manual or automated [15] spectrophotometric. | 335.1 | 412F | D2036-75(B) | | |
| 25. Fluoride—Total, mg/L: | | | | | |
| Manual distillation [5] | | 413A | | | |
| Followed by manual or | 340.2 | 413B | D1179-72(B) | | |
| Automated electrode | | | | I-4327-78 | |
| SPADNS | 340.1 | 413C | D1179-72(A) | | |
| Or automated complexone | 340.3 | 413E | | | |
| 26. Gold—Total [3], mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration | 231.1 | 303A | | | |
| Or AA furnace | 231.2 | 304 | | | |
| 27. Hardness—Total as $CaCo_3$, mg/L: | | | | | |
| Automated colorimetric | 130.1 | | | | |
| EDTA titration | 130.2 | 314B | D1126-67(B) | I-1338-78 | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Or atomic absorption (sum of Ca and Mg as their respective carbonates) | 215.1+ 242.1 | 303A | | I-3153-78+ I-3448-78 | |

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

| Parameter, units, and method | Reference (method No. or page) | | | | |
|---|---|---|---|---|---|
| | EPA 1979 | Standard methods 15th Ed. | ASTM | USGS [1] | Other |
| 28. Hydrogen ion (pH), pH units: | | | | | |
| Electrometric | 150.1 | 423 | D1293-78(A) or D1293-78(B). | I-1586-78 | |
| Measurements; or automated electrode | | | | | ([14]) |
| 29. Iridium—Total [3], mg/L: Digestion [5] followed by: | | | | | |
| AA direct aspiration | 235.1 | 303A | | | |
| Or AA furnace | 235.2 | 304 | | | |
| 30. Iron—Total [3], mg/L: | | | | | |
| Digestion [5] followed by | | 303A or 303B | D1068-77 | | |
| AA direct aspiration | 236.1 | 303B | (C or D) | I-3381-78 | P. 557.[8] |
| AA furnace | 236.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Or colorimetric (Phenanthroline) | | 315B | D1068-77(A) | | ([17]) |
| 31. Kjeldahl nitrogen—Total (as N), mg/L: | | | | | |
| Digestion and distillation | 351.3 | 420A or B | | | P. 552.[8] |
| Followed by titration | 351.3 | 417D | D3590-77 | | |
| Nesslerization or | 351.3 | 417B | | | |
| Electrode | 351.3 | 417E | | I-4551-78 | |
| Automated phenate | 351.1 | | | I-4552-78 | |
| Semi-automated block digestor | 351.2 | | | | |
| Or potentiometric | 351.4 | | | | |
| 32. Lead—Total[18], mg/L: Digestion [5] followed by: | | | | | |
| AA direct aspiration | 239.1 | 303A or 303B | D3559-78 (A or B) | I-3399-78 | P. 557.[8] |
| AA furnace | 239.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Voltametry [9] or | | | D3559-78(C) | | |
| Colorimetric (Dithizone) | | 316B | | | |
| 33. Magnesium—Total [3], mg/L: Digestion [5] followed by: | | | | | |
| Atomic absorption | 242.1 | 303A | D511-77(B) | I-3447-78 | P. 557.[8] |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Or gravimetric | | 318B | D511-77(A) | | |
| 34. Maganese—Total [3], mg/L: Digestion [5] followed by: | | | | | |
| AA direct aspiration | 243.1 | 303A or 303B | D858-77 (B or C) | I-3454-78 | P. 557.[8] |
| AA furnace | 243.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Or colorimetric (Persulfate) | | 319B | D858-77(A) | | P. 564.[8] |
| Periodate | | | | | 18. |
| 35. Mercury—Total [3], mg/L: | | | | | |
| Cold vapor, manual or | 245.1 | 303F | D3223-79 | I-3462-78 | P. 559.[8] |
| Automated | 245.2 | | | | |
| 36. Molybdenum—Total [3], mg/L: Digestion [5] followed by: | | | | | |
| AA direct aspiration | 246.1 | 303C | | I-3490-78 | |
| AA furnace, or | 246.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| 37. Nickel—Total [3], mg/L: Digestion [5] followed by: | | | | | |
| AA direct aspiration | 249.1 | 303A or 303B | D1886-77 (C or D) | I-3499-78 | |
| AA furnace | 249.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Or colorimetric (Heptoxime) | | 321B | | | |
| 38. Nitrate (as N), mg/L: | | | | | |
| Brucine sulfate, or | 352.1 | | D092-71 | | P. 554.[8] |
| Nitrate-nitrite N minus Nitrite N | See parameters 39 and 40. | See parameters 39 and 40. | See parameters 39 and 40. | See parameters 39 and 40. | P. 28.[8] |
| 39. Nitrate-nitrite (as N), mg/L: | | | | | |
| Cadmium reduction, manual | 353.3 | 418C | D3867-79(B) | | |
| Or automated; or | 353.2 | 418F | D3867-79(A) | I-4545-78 | |
| Automated hydrazine | 353.1 | | | | |
| 40. Nitrite (as N), mg/L: | | | | | |
| Spectrophotometric, manual or | 354.1 | 419 | D1254-67 | | 19. |
| Automated (Diazotization) | | | | I-4540-78 | |
| 41. Oil and grease—Total recoverable, mg/L: Gravimetric (extraction). | 413.1 | 503A | | | |
| 42. Organic carbon—Total (TOC), mg/L: Combustion or oxidation. | 415.1 | 505 | D2579-78(A) or D2579-78(B). | | P. 551 [8] and P. 4.[20] |
| 43. Organic nitrogen (as N), mg/L: Total Kjeldahl N minus ammonia N. | See parameters 31 and 4. | 420A | D3590-77 minus D1426-79(A). | See parameters 31 and 4. | PP. 552-53.[8] |
| 44. Orthophosphate (as P), mg/L: Ascorbic acid method, automated | 365.1 | 424G | | I-4601-78 | P. 561.[8] |
| Or manual single reagent or | 365.2 | 424F | D515-78(A) | | |
| Manual two reagent | 365.3 | | | | |
| 45. Osmium—Total [3], mg/L: Digestion [5] followed by: | | | | | |
| AA direct aspiration, or | 252.1 | 303C | | | |
| AA furnace | 252.2 | 304 | | | |
| 46. Oxygen, dissolved, mg/L: | | | | | |
| Winkler (Azide modification) | 360.2 | 421B | D1589-60(A) | I-1575-78 | P. 550.[8] |
| Or electrode | 360.1 | 421F | | I-1576-78 | |
| 47. Palladium—Total [3], mg/L: Digestion [5] followed by: | | | | | |
| AA direct aspiration | 253.1 | | | | P. S27.[25] |
| Or AA furnace | 253.2 | | | | P. S28.[25] |
| 48. Phenols, mg/L: | | | | | |
| Manual distillation | 420.1 | | D1783-70 (A or B) | | 26. |
| Followed by manual | 420.1 | | | | 26. |
| Or automated [15] colorimetric (4AAP) | 420.2 | | | | |
| 49. Phosphorus (elemental), mg/L: Gas-liquid chromatography. | | | | | 21. |

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

| Parameter, units, and method | Reference (method No. or page) | | | | |
|---|---|---|---|---|---|
| | EPA 1979 | Standard methods 15th Ed. | ASTM | USGS [1] | Other |
| 50. Phosphorus—Total, mg/L: | | | | | |
| Persulfate digestion | 365.2 | 424C (III) | | | P. 561.[2] |
| Followed by manual or | 365.2 or 365.3 | 424F | D515-78(A) | | |
| Automated ascorbic acid | 365.1 | 424G | | I-4600-78 | |
| Reduction; or semi-automated block digestor | 365.4 | | | I-4603-78 | |
| 51. Platinum—Total [3], mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration | 255.1 | 303A | | | |
| Or AA furnace | 255.2 | 304 | | | |
| 52. Potassium—Total [3], mg/L: Digestion [3] followed by: | | | | | |
| Atomic absorption | 258.1 | 303A | | I-3630-78 | P. 560.[2] |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Or flame photometric | | 322B | D1428-64(A) | | |
| 53. Residue—total, mg/L: Gravimetric, 103–105°C | 160.3 | 209A | | I-3750-78 | |
| 54. Residue—filterable, mg/L: Gravimetric, 180°C | 160.1 | 209B | | I-1750-78 | |
| 55. Residue—nonfilterable, (TSS), mg/L: Gravimetric, 103–105°C post washing of residue. | 160.2 | 209D | | I-3765-78 | |
| 56. Residue—settleable, mg/L: Volumetric (Imhoff cone) or gravimetric. | 160.5 | 209F | | | |
| 57. Residue—volatile, mg/L: Gravimetric, 550°C | 160.4 | 209E | | I-3753-78 | |
| 58. Rhodium—Total [3], mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration | 265.1 | 303A | | | |
| Or AA furnace | 267.2 | 304 | | | |
| 59. Ruthenium—Total [3], mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration | 267.1 | 303A | | | |
| Or AA furnace | 267.2 | 304 | | | |
| 60. Selenium—Total [3] mg/L: Digestion [3] followed by: | | | | | |
| AA furnance | 270.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Or hydride | 270.3 | 303E | D3859-79 | I-3667-78 | |
| 61. Silica—Dissolved, mg/L: 0.45 micron filtration: | | | | | |
| Followed by manual or | 370.1 | 425C | D859-68(B) | I-1700-78 | |
| Automated colorimetric (Molybdosilicate), or | | | | I-2700-78 | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| 62. Silver—Total [3,5] mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration | 272.1 | 303A or 303B | | I-3720-78 | P. 557 [3] and p. 37.[6] |
| AA furnace, or | 272.1 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| 63. Sodium—Total [3], mg/L: Digestion [3] followed by: | | | | | |
| Atomic absorption | 273.1 | 303A | | I-3735-78 | P. 561.[2] |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Or flame photometric | | | D1428-64(A) | | |
| 64. Specific conductance, mhos/cm: Wheatstone bridge. | 120.1 | 205 | D1125-77(A) | I-1780-78 | P. 547.[2] |
| 65. Sulfate (as $SO_4$), mg/L: | | | | | |
| Automated methylthymol blue | 375.2 | | | I-2822-78 | |
| Gravimetric, or | 375.3 | 426A or 426B | D516-68(A) | | PP. 562-63.[3] |
| Turbidimetric | 375.4 | 426C | D516-68(B) | | |
| 66. Sulfide (as S), mg/L: | | | | | |
| Titrimetric (iodine) or | 376.1 | 427D | | I-3840-78 | |
| Colorimetric (methylene blue) | 376.2 | 427C | | | |
| 67. Sulfite (as $SO_4$), mg/L: Titrimetric (iodine iodate) | 377.1 | 428F | D1339-78(C) | | |
| 68. Surfactants, mg/L: Colorimetric (methylene blue) | 425.1 | 512A | D2330-68(A) | | |
| 69. Temperature, °C.: Thermometric | 170.1 | 212 | | | [2,3]. |
| 70. Thallium—Total,[5] mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration | 279.1 | 303A | | | |
| AA furnace, or | 279.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| 71. Tin—Total,[5] mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration or | 282.1 | 303A | | I-3850-78 | |
| AA furnace | 282.2 | 304 | | | |
| 72. Titanium—Total,[5] mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration or | 283.1 | 303C | | | |
| AA furnace | 283.2 | 304 | | | |
| 73. Turbidity, NTU: Nephelometric | 180.1 | 214A | D1889-71 | I-3860-78 | |
| 74. Vanadium—Total,[5] mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspiration | 286.1 | 303C | | | |
| AA furnace | 286.2 | 304 | | | |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Or colorimetric (Gallic acid) | | | D3373-75 | | |
| 75. Zinc—Total,[5] mg/L: Digestion [3] followed by: | | | | | |
| AA direct aspirationb | 289.1 | 303A or 303B | D1691-77(D) | I-3900-78 | P. 557.[2] |
| AA furnace | 289.2 | 304 | D1691-77(C) | | P. 37.[6] |
| Inductively coupled plasma | | | | | Method 200.7.[4] |
| Or colorimetric (Zincon) | | | | | [3,4]. |

**Table IB Notes**

[1] "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, U.S. Geological Survey, Open-File Report 78-679, or "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments," N.W. Skougstad, *et al*; U.S. Geological Survey, Techniques of Water-Resources Investigation, Book 5, Chapter A1, 1979.

[2] "Official Methods of Analysis of the Association of Official Analytical Chemists" methods manual, 13th ed. (1980).

[3] For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic-metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979." One (§ 4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (§ 4.1.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all sample types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic bonds be broken so that the metal is in a reactive state. In those situations, the vigorous digestion is to be preferred making certain that at no time does the sample go to dryness. Samples containing large amounts of organic materials would also benefit by this vigorous digestion. Use of the graphite furnace technique, inductively coupled plasma, as well as determinations for certain elements such as arsenic, the noble metals, mercury, selenium, and titanium require a modified digestion and in all cases the method write-up should be consulted for specific instructions and/or cautions.

**Note:** If the digestion procedure for direct aspiration or graphite furnace atomic absorption analysis included in one of the other approved references is different than the above, the EPA procedure must be used.

Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals must be followed. Sample digestion of the filtrate for dissolved metals, or digestion of the original sample solution for total metals may be omitted for AA (direct aspiration or graphite furnace) and ICP analyses provided the sample has a low COD and the filtrate meets the following criteria:

(a) Is visibly transparent

(b) Has no perceptible odor, and

(c) Is free of particulate or suspended matter following acidification.

[4] The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," is given at Appendix C of this Part 136.

[5] Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary; however, manual distillation will be required to resolve any controversies.

[6] Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 19, 1976, Technicon AutoAnalyzer II, Technicon Industrial Systems, Tarrytown, New York 10591.

[7] Carbonaceous biochemical oxygen demand ($CBOD_5$) must not be confused with the traditional $BOD_5$ test which measures "total BOD". The addition of the nitrification inhibitor is not a procedural option, but must be included to report the $CBOD_5$ parameter. A discharger whose permit requires reporting the traditional $CBOD_5$ may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states $CBOD_5$ is required can the permittee report data obtained using the nitrification inhibitor.

[8] American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.

[9] The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

[10] Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

[11] COD Method, Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840.

[12] The back titration method will be used to resolve controversy.

[13] National Council of the Paper Industry for Air and Stream Improvement, Inc., Technical Bulletin 253, December 1971.

[14] Copper, Bicinchoninate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

[15] After the manual distillation is completed, the auto-analyzer manifolds in EPA Methods 335.03 (Cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the manifold setup shown in Method 335, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.

[16] Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon Auto-Analyzer II, Technicon Industrial Systems, Tarrytown, New York 10591.

[17] Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

[18] Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, Loveland, Colorado 80537.

[19] Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537.

[20] Goerlitz, D., Brown, E., "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A3, p.4 (1972).

[21] R.F. Addison and R.G. Ackman, "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography," Journal of Chromatography, Vol. 47, No. 3, pp. 421-426, 1970.

[22] Recommended methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to a pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M $Na_2S_2O_3$ and 2M NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the recommended method is satisfactory.

[23] Stevens, H.H., Ficke, J.F., and Smoot, G.F., "Water Temperature-Influential Factors, Field Measurement and Data Presentation," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 1, Chapter D1, 1975.

[24] Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2-231 and 2-333, Hach Chemical Company, Loveland, Colorado 80537.

[25] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater (1981)*.

[26] The approved method is that cited in *Standard Methods for the Examination of Water and Wastewater*, 14th Edition. The colorimetric reaction is conducted at a pH of 10.0 ± 0.2. The approved methods are given on pp. 576-81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectrophotometric procedure.

[27] ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, Massachusetts 02138.

## TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

| Parameter [1] | EPA Method Number [5, 7] | | | Other |
|---|---|---|---|---|
| | GC | GC/MS | HPLC | |
| 1. Acenaphthene | 610 | 625, 1625 | 610 | |
| 2. Acenaphthylene | 610 | 625, 1625 | 610 | |
| 3. Acrolein | 603 | [4]624, 1624 | | |
| 4. Acrylonitrile | 603 | [4]624, 1624 | | |
| 5. Anthracene | 610 | 625, 1625 | 610 | |
| 6. Benzene | 602 | 624, 1624 | | |
| 7. Benzidine | | [6]625, 1625 | 605 | Note 3, p. 1; |
| 8. Benzo(a)anthracene | 610 | 625, 1625 | 610 | |
| 9. Benzo(a)pyrene | 610 | 625, 1625 | 610 | |
| 10. Benzo(b)fluoranthene | 610 | 625, 1625 | 610 | |
| 11. Benzo(ghi)perylene | 610 | 625, 1625 | 610 | |
| 12. Benzo(k)fluoranthene | 610 | 625, 1625 | 610 | |
| 13. Benzyl Chloride | | | | Note 3, p. 130; Note 6, p. S102. |
| 14. Benzyl Butyl Phthalate | 606 | 625, 1625 | | |
| 15. Bis(2-chloroethoxy) methane | 611 | 625, 1625 | | |
| 16. Bis(2-chloroethyl) ether | 611 | 625, 1625 | | |
| 17. Bis(2-ethylhexyl) phthalate | 606 | 625, 1625 | | |
| 18. Bromodichloromethane | 601 | 624, 1624 | | |
| 19. Bromoform | 601 | 624, 1624 | | |
| 20. Bromomethane | 601 | 624, 1624 | | |
| 21. 4-Bromophenylphenyl ether | 611 | 625, 1625 | | |
| 22. Carbon tetrachloride | 601 | 624, 1624 | | Note 3, p. 130; |
| 23. 4-Chloro-3-methylphenol | 604 | 625, 1625 | | |
| 24. Chlorobenzene | 601, 602 | 624, 1624 | | Note 3, p. 130; |
| 25. Chloroethane | 601 | 624, 1624 | | |
| 26. 2-Chloroethylvinyl ether | 601 | 624, 1624 | | |
| 27. Chloroform | 601 | 624, 1624 | | Note 3, p. 130; |
| 28. Chloromethane | 601 | 624, 1624 | | |
| 29. 2-Chloronaphthalene | 612 | 625, 1625 | | |
| 30. 2-Chlorophenol | 604 | 625, 1625 | | |
| 31. 4-Chlorophenylphenyl ether | 611 | 625, 1625 | | |
| 32. Chrysene | 610 | 625, 1625 | 610 | |
| 33. Dibenzo(a,h)anthracene | 610 | 625, 1625 | 610 | |
| 34. Dibromochloromethane | 601 | 624, 1624 | | |
| 35. 1,2-Dichlorobenzene | 601, 602, 612 | 624, 625, 1625 | | |
| 36. 1,3-Dichlorobenzene | 601, 602, 612 | 624, 625, 1625 | | |
| 37. 1,4-Dichlorobenzene | 601, 602, 612 | 625, 1624, 1625 | | |
| 38. 3,3'-Dichlorobenzidine | | 625, 1625 | 605 | |
| 39. Dichlorodifluoromethane | 601 | | | |
| 40. 1,1-Dichloroethane | 601 | 624, 1624 | | |
| 41. 1,2-Dichloroethane | 601 | 624, 1624 | | |
| 42. 1,1-Dichloroethene | 601 | 624, 1624 | | |
| 43. trans-1,2-Dichloroethene | 601 | 624, 1624 | | |
| 44. 2,4-Dichlorophenol | 604 | 625, 1625 | | |
| 45. 1,2-Dichloropropane | 601 | 624, 1624 | | |
| 46. cis-1,3-Dichloropropene | 601 | 624, 1624 | | |
| 47. trans-1,3-Dichloropropene | 601 | 624, 1624 | | |
| 48. Diethyl phthalate | 606 | 625, 1625 | | |

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

| Parameter [1] | EPA Method Number [2, 7] | | | Other |
|---|---|---|---|---|
| | GC | GC/MS | HPLC | |
| 49. 2,4-Dimethylphenol | 604 | 625, 1625 | | |
| 50. Dimethyl phthalate | 606 | 625, 1625 | | |
| 51. Di-n-butyl phthalate | 606 | 625, 1625 | | |
| 52. Di-n-octyl phthalate | 606 | 625, 1625 | | |
| 53. 2,4-Dinitrophenol | 604 | 625, 1625 | | |
| 54. 2,4-Dinitrotoluene | 609 | 625, 1625 | | |
| 55. 2,6-Dinitrotoluene | 609 | 625, 1625 | | |
| 56. Epichlorohydrin | | | | Note 3, p. 130; Note 6, p. S102. |
| 57. Ethylbenzene | 602 | 624, 1624 | | |
| 58. Fluoranthene | 610 | 625, 1625 | 610 | |
| 59. Fluorene | 610 | 625, 1625 | 610 | |
| 60. Hexachlorobenzene | 612 | 625, 1625 | | |
| 61. Hexachlorobutadiene | 612 | 625, 1625 | | |
| 62. Hexachlorocyclopentadiene | 612 | [5] 625, 1625 | | |
| 63. Hexachloroethane | 612 | 625, 1625 | | |
| 64. Ideno(1,2,3-cd)pyrene | 610 | 625, 1625 | 610 | |
| 65. Isophorone | 609 | 625, 1625 | | |
| 66. Methylene Chloride | 601 | 624, 1624 | | Note 3, p. 130; |
| 67. 2-Methyl-4,6-Dinitrophenol | 604 | 625, 1625 | | |
| 68. Naphthalene | 610 | 625, 1625 | | |
| 69. Nitrobenezene | 609 | 625, 1625 | | |
| 70. 2-Nitrophenol | 604 | 625, 1625 | | |
| 71. 4-Nitrophenol | 604 | 625, 1625 | | |
| 72. N-Nitrosodimethylamine | 607 | 625, 1625 | | |
| 73. N-Nitrosodi-n-propylamine | 607 | [5] 625, 1625 | | |
| 74. N-Nitrosodiphenylamine | 607 | [5] 625, 1625 | | |
| 75. 2,2-oxybis(1-chloropropane) | 611 | 625, 1625 | | |
| 76. PCB-1016 | 608 | 625 | | Note 3, p. 43; |
| 77. PCB-1221 | 608 | 625 | | Note 3, p. 43; |
| 78. PCB-1232 | 608 | 625 | | Note 3, p. 43; |
| 79. PCB-1242 | 608 | 625 | | Note 3, p. 43; |
| 80. PCB-1248 | 608 | 625 | | Note 3, p. 43; |
| 81. PCB-1254 | 608 | 625 | | Note 3, p. 43; |
| 82. PCB-1260 | 608 | 625 | | Note 3, p. 43; |
| 83. Pentachlorophenol | 604 | 625, 1625 | | Note 3, p. 140; |
| 84. Phenanthrene | 610 | 625, 1625 | 610 | |
| 85. Phenol | 604 | 625, 1625 | | |
| 86. Pyrene | 610 | 625, 1625 | 610 | |
| 87. 2,3,7,8-Tetrachlorodibenzo-p-dioxin | | [8] 613 | | |
| 88. 1,1,2,2-Tetrachloroethane | 601 | 624, 1624 | | Note 3, p. 130; |
| 89. Tetrachloroethene | 601 | 624, 1624 | | Note 3, p. 130; |
| 90. Toluene | 602 | 624, 1624 | | |
| 91. 1,2,4-Trichlorobenzene | 612 | 625, 1625 | | Note 3, p. 130; |
| 92. 1,1,1-Trichloroethane | 601 | 624, 1624 | | |
| 93. 1,1,2-Trichloroethane | 601 | 624, 1624 | | Note 3, p. 130; |
| 94. Trichloroethene | 601 | 624, 1624 | | |
| 95. Trichlorofluoromethane | 601 | 624 | | |
| 96. 2,4,6-Trichlorophenol | 604 | 625, 1625 | | |
| 97. Vinyl Chloride | 601 | 624, 1624 | | |

**Table IC Notes**

[1] All parameters are expressed in micrograms per liter (μg/L).

[2] The full text of Methods 601–613, 624, 625, 1624, and 1625, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 136.

[3] "Methods for Benzidine; Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978.

[4] Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 603 or Method 1624.

[5] Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodiumethyamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625, are preferred methods for these compounds.

[6] 625, Screening only.

[8] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).

[7] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 601–613, 624, 625, 1624, and 1625 (See Appendix A of this Part 136) in accordance with procedures each in section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going bases must spike and analyze 10% (5% for Methods 624 and 625 and 100% for methods 1624, and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance.

**Note.**—These warning limits are promulgated as an "interim final action with a request for comments."

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES [1]

| Parameter μg/L) | Method | EPA [2, 7] | Standard Methods 15th Ed | ASTM | Other |
|---|---|---|---|---|---|
| 1. Aldrin | GC | 608 | 509A | D3086 | Note 3, p. 7; Note 4, p. 30. |
| | GC/MS | 625 | | | |
| 2. Ametryn | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 3. Aminocarb | TLC | | | | Note 3, p. 94; Note 6, p. S16. |
| 4. Atraton | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 5. Atrazine | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 6. Azinphos methyl | GC | | | | Note 3, p. 25; Note 6, p. S51. |
| 7. Barban | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 8. α-BHC | GC | 608 | 509A | D3086 | Note 3, p. 7. |
| | GC/MS | [5] 625 | | | |
| 9. β-BHC | GC | 608 | | D3086 | |
| | GC/MS | 625 | | | |
| 10. δ-BHC | GC | 608 | | D3086 | |

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES [1]—Continued

| Parameter µg/L) | Method | EPA [2,7] | Standard Methods 15th Ed | ASTM | Other |
|---|---|---|---|---|---|
| 11. γ-BHC (Lindane) | GC/MS | [5] 625 | | | |
| | GC | 608 | 509A | D3086 | Note 3, p. 7; Note 4, p. 30. |
| | GC/MS | 625 | | | |
| 12. Captan | GC | | 509A | | Note 3, p. 7. |
| 13. Carbaryl | TLC | | | | Note 3, p. 94; Note 6, p. S60. |
| 14. Carbophenothion | GC | | | | Note 4, p. 30; Note 6, p. S73. |
| 15. Chlordane | GC | 608 | 509A | D3086 | Note 3, p. 7. |
| | GC-MS | 625 | | | |
| 16. Chloropropham | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 17. 2,4–D | GC | | 509B | | Note 3, p. 115; Note 4, p. 35. |
| 18. 4,4'–DDD | GC | 608 | 509A | D3086 | Note 3, p. 7; Note 4, p. 30. |
| | GC-MS | 625 | | | |
| 19. 4,4'–DDE | GC | 608 | 509A | D3086 | Note 3, p. 7; Note 4, p. 30. |
| | GC/MS | 625 | | | |
| 20. 4,4'–DDT | GC | 608 | 509A | D3086 | Note 3, p. 7; Note 4, p. 30. |
| | GC/MS | 625 | | | |
| 21. Demeton-O | GC | | | | Note 3, p. 25; Note 6, p. S51. |
| 22. Dementon-S | GC | | | | Note 3, p. 25; Note 6, p. S51. |
| 23. Diazinon | GC | | | | Note 3, p. 25; Note 4, p. 30; Note 6, p. S51. |
| 24. Dicamba | GC | | | | Note 3, p. 115. |
| 25. Dichlofenthion | GC | | | | Note 4, p. 30; Note 6, p. S73. |
| 26. Dichloran | GC | | 509A | | Note 3, p. 7. |
| 27. Dicofol | GC | | | D3086 | |
| 28. Dieldrin | GC | 608 | 509A | | Note 3, p. 7; Note 4, p. 30. |
| | GC/MS | 625 | | | |
| 29. Dioxathion | GC | | | | Note 4, p. 30; Note 6, p. S73. |
| 30. Disulfoton | GC | | | | Note 3, p. ; Note 6, p. S51. |
| 31. Diuron | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 32. Endosulfan I | GC | 608 | 509A | D3086 | Note 3, p. 7. |
| | GC/MS | [5] 625 | | | |
| 33. Endosulfan II | GC | 608 | 509A | D3086 | Note 3, p. 7. |
| | GC/MS | [5] 625 | | | |
| 34. Endosulfan sulfate | GC | 608 | | | |
| | GC/MS | 625 | | | |
| 35. Endrin | GC | 608 | 509A | D3086 | Note 3, p. 7; Note 4, p. 30. |
| | GC/MS | [5] 625 | | | |
| 36. Endrin aldehyde | GC | 608 | | | |
| | GC/MS | 625 | | | |
| 37. Ethion | GC | | | | Note 4, p. 30; Note 6, p. S73. |
| 38. Fenuron | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 39. Fenuron-TCA | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 40. Heptachlor | GC | 608 | 509A | D3086 | Note 3, p. 7; Note 4, p. 30. |
| | GC/MS | 625 | | | |
| 41. Heptachlor epoxide | GC | 608 | 509A | D3086 | Note 3, p. 7; Note 4, p. 30; Note 6, p. S73. |
| | GC/MS | 625 | | | |
| 42. Isodrin | GC | | | | Note 4, p. 30; Note 6, p. S73. |
| 43. Linuron | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 44. Malathion | GC | | 509A | | Note 3, p. 25; Note 4, p. 30; Note 6, p. S51. |
| 45. Methiocarb | TLC | | | | Note 3, p. 94; Note 6, p. S60. |
| 46. Methoxychlor | GC | | 509A | D3086 | Note 3, p. 7; Note 4, p. 30. |
| 47. Mexacarbate | TLC | | | | Note 3, p. 94; Note 6, p. S60. |
| 48. Mirex | GC | | 509A | | Note 3, p. 7. |
| 49. Monuron | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 50. Monuron-TCA | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 51. Neburon | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 52. Parathion methyl | GC | | 509A | | Note 3, p. 25; Note 4, p. 30. |
| 53. Parathion ethyl | GC | | 509A | | Note 3, p. 25. |
| 54. PCNB | GC | | 509A | | Note 3, p. 7. |
| 55. Perthane | GC | | | D3086 | |
| 56. Prometon | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 57. Prometryn | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 58. Propazine | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 59. Propham | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 60. Propoxur | TLC | | | | Note 3, p. 94; Note 6, p. S60. |
| 61. Secbumeton | TLC | | | | Note 3, p. 83; Note 6, p. S68. |
| 62. Siduron | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 63. Simazine | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 64. Strobane | GC | | 509A | | Note 3, p. 7. |
| 65. Swep | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 66. 2,4,5–T | GC | | 509B | | Note 3, p. 115; Note 4, p. 35. |
| 67. 2,4,5–TP (Silvex) | GC | | 509B | | Note 3, p. 115. |
| 68. Terbuthylazine | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 69. Toxaphene | GC | 608 | 509A | D3086 | Note 3, p. 7; Note 4, p. 30. |
| | GC/MS | 625 | | | |
| 70. Trifluralin | GC | | 509A | | Note 3, p. 7 |

**Table ID Notes**

[1] Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table IC, where entries are listed by chemical name.

[2] The full text of methods 608 and 625 are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit", of this Part 136.

[3] "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods.

[4] "Methods for Analysis of Organic Substances in Water," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A3 (1972).

[5] The method may be extended to include α-BHC, δ-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

[6] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).

[7] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A of this Part 136) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with

Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance.

**Note.**—These warning limits are promulgated as an "interim final action with a request for comments."

TABLE IE.—LIST OF APPROVED RADIOLOGICAL TEST PROCEDURES

| Parameter and units | Methods | EPA [1] | Reference (method No. or page) | | |
|---|---|---|---|---|---|
| | | | Standard Methods 15th Ed. | ASTM | USGS [2] |
| 1. Alpha-Total, $pC^{1}$ per liter | Proportional or scintillation counter | 900.0 | 703 | D1943-66 | pp. 75 and 78.[3] |
| 2. Alpha-Counting error, $pC^{1}$ per liter | Proportional or scintillation counter | Appendix B | 703 | D1943-66 | p. 79. |
| 3. Alpha-Counting error, $pC^{1}$ per liter | Proportional counter | 900.0 | 703 | D1890-66 | pp. 75 and 78.[3] |
| 4. Beta-Counting error, $pC^{1}$ per liter | Proportional counter | Appendix B | 703 | D1890-66 | p. 79. |
| 5. (a) Radium-Total, $pC^{1}$ per liter | Proportional counter | 903.0 | 705 | D2460-70 | |
| (b) $^{226}Ra$, $pC^{1}$ per liter | Scintillation counter | 903.1 | 706 | D3454-79 | p. 81. |

**Table IE Notes**

[1] "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA-600/4-80-032 (1980 update), U.S. Environmental Protection Agency, August 1980.
[2] Fishman, M.J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 76-177 (1976).
[3] The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total."

5. In § 136.3, paragraph (a) is revised to show that the full text of approved test procedures have been incorporated by reference, into the regulation to read as follows:

**§ 136.3 Identification of test procedures.**

(a) Parameters or pollutants, for which methods are approved, are listed together with test procedure descriptions and references in Tables IA, IB, IC, ID, and IE. The full text of the referenced test procedures are incorporated by reference into Tables IA, IB, IC, ID, and IE. The references and the sources from which they are available are given in paragraph (b) of this section. These test procedures are incorporated as they exist on the day of approval and a notice of any change in these test procedures will be published in the **Federal Register.** The discharge parameter values for which reports are required must be determined by one of the standard analytical test procedures incorporated by reference and described in Tables IA, IB, IC, ID, and IE, or by any alternate test procedure which has been approved by the Administrator under the provisions of paragraph (d) of this section and sections 136.4 and 136.5 of this Part 136. Under certain circumstances (§§ 136.3 (b) or (c) or 40 CFR Part 401.13) other test procedures may be used that may be more advantageous when such other test procedures have been previously approved by the Regional Administrator of the Region in which the discharge will occur, and providing the Director of the State in which such discharge will occur does not object to the use of such alternate test procedure.

6. In § 136.3, paragraphs (b) and (c) are redesignated as (c) and (d) and a new paragraph (b) is added to itemize the references which are "incorporated by reference" and to identify the sources from which they may be obtained. As added, the new paragraph (b) reads as follows:

**§ 136.3 Identification of test procedures.**

* * * * *

(b) The full texts of the methods from the following references which are cited in Tables IA, IB, IC, ID, and IE are incorporated by reference into this regulation and may be obtained from the sources identified. All costs cited are subject to change and must be verified from the indicated sources.

REFERENCES, SOURCES, AND COSTS

| Table | Parameters | Reference, source and cost |
|---|---|---|
| IA—EPA | 1–5 | "Microbiological Methods for Monitoring the Environment, Water and Wastes," United States Environmental Protection Agency, EPA-600/8-78-017, 1978. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268. |
| IA—Standard Methods | 1–5 | *Standard Methods for the Examination of Water and Wastewater,* Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation 15th Edition, 1981. Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20036. Cost: $50.00 including the Supplement to the Fifteenth Edition. |
| IB—Standard | 1–10, 12–46, 50–75 | |
| ID—Standard Methods | 1, 8, 11, 12, 15, 17–20, 26, 28, 32, 33, 35, 40, 41, 44, 46, 48, 52–54, 64, 66, 67, 69, 70 | |
| IE—Standard Methods | 1–5 | |
| IB—Standard Methods | 48 | Ibid, 14th Edition. |
| IB—Other (Standard Methods Supplement) | 11, 47 | "Selected Analytical Methods approved and Cited by the United States Environmental Protection Agency," Supplement to the 15th Edition of *Standard Methods for the Examination of Water and Wastewater* (1981). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20036. Cost: Included with the 15th Edition of *Standard Methods for the examination of Water and Wastewater.* |
| IC—Other (Standard Methods Supplement) | 13, 56 | |
| ID—Other (Standard Methods Supplement) | 2–7, 13, 14, 16, 21–23, 25, 29–31, 37, 38, 39, 41–45, 47, 49, 50, 51, 56–63, 65, 68 | |
| IA—U.S. Geological Survey (USGS) | 1, 3, 5 | "Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples," edited by P.E. Greeson, T.A. Ehlke, G.A. Irwin, B.W. Lium, and K.V. Slack: U.S. Geological Survey, Techniques of Water-Resources Investigation (USGS TWRI), Book 5, Chapter A4 (1977). Revised edition, 332 pages. Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. (Authorized agent of the Superintendent of Documents, Government Printing Office.) Cost: $9.25. Prices are subject to change. |
| IB—EPA | 1–13, 15–48, 50–75 | "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020 United States Environmental Protection Agency, March, 1979. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency Cincinnati, Ohio 45268. |
| IB—ASTM | 1, 2, 4, 6, 8, 11–13, 15–17, 19, 20, 22–25, 27, 28, 30–35, 37–40, 42–44, 46, 48, 50, 52, 60, 61, 63–65, 67, 68, 73–75 | "Annual Book of Standards, Part 31, Water", American Society for Testing and Materials, 1980. Available from: American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103. Cost available from publisher. |
| ID—ASTM | 1, 8–11, 15, 18–20, 27, 32, 33, 35, 40, 41, 46, 55, 69 | |
| IE—ASTM | 1–5 | |

REFERENCES, SOURCES, AND COSTS—Continued

| Table | Parameters | Reference, source and cost |
|---|---|---|
| IB—USGS | 2, 3, 4, 6–13, 15, 16, 18–23, 25, 27, 28, 30–40, 43, 44, 46, 50, 52–55, 57, 60–66, 71, 73, 75. | "Methods for determination of inorganic substances in water and fluvial sediments," N.W. Skougstad and others, editors: USGS—TWRI Book 5, Chapter A1, 1979. $10.00. Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. (Authorized agent of the Superintendent of Documents, Government Printing Office). Prices are subject to change. |
| IB—Other (AOAC) | 2, 4, 9, 12, 15, 16, 19, 22, 30–35, 38, 42–44, 46, 50, 52, 62–65, 75. | Official Methods of Analysis of the Association of Official Analytical Chemists, methods manual, 13th Edition (1980). Price: $78.00. Available from: The Association of Official Analytical Chemists, 1111 N. 19th St., Suite 210, Arlington, VA 22209. |
| IB—Other (ANSI) | 9, 12, 15, 20, 22, 23, 38, 62, 75 | "American National Standard on Photographic Processing Effluents," April 2, 1975. Available from: American National Standards Institute, 1430 Broadway, New York, New York 10018. |
| IB—Other (EPA) | 3, 5–8, 10, 12, 13, 19, 20, 22, 27, 30, 32–34, 36, 37, 52, 60–63, 70, 74, 75. | The full text of the inductively coupled plasma optical emission spectroscopic test procedure, Method 200.7, is printed in Appendix C of this Part 136. |
| IB—Other | 21 | "An Investigation of Improved Procedures for Measurement of Mill Effluent and Receiving Water Color," NCASI Technical Bulletin No. 253, December, 1971. Available from: National Council of the Paper Industry for Air and Stream Improvements, Inc., 260 Madison Avenue. Cost available from publisher. |
| IB—Other | 4 | Ammonia, Automated Electrode Method, Industrial Method Number 379–75WE, dated February 19, 1976, Technicon AutoAnalyzer II. Method and price available from Technicon Industrial Systems, Tarrytown, New York 10591. |
| IB—Other | 15 | Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. |
| IB—Other | 15 | OIC Chemical Oxygen Demand Method. Method and price available from Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840. |
| IB—Other | 17 | ORION Research Instruction Manual, Residual Chlorine Electrode Model 97–70, 1977. Method and price available from Orion Research Incorporated, 840 Memorial Drive, Cambridge, Massachusetts 02138. |
| IB—Other | 22 | Bicinchoninate Method for Copper, Method 8506, Hach Handbook of Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. |
| IB—Other | 28 | Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378–75WA, October 1976, Technicon AutoAnalyzer II. Method and Price available from Technicon Industrial Systems, Tarrytown, New York 10591. |
| IB—Other | 30 | 1, 10-Phenanthroline Method for Iron, Hach Method 8008. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. |
| IB—Other | 34 | Periodate Oxidation Method for Manganese, Method 8034, Hach Handbook for Water Analysis, 1979. Method and Price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. |
| IB—Other | 40 | Nitrite Nitrogen, Hach Method 8507. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. |
| IB—Other | 75 | Zincon Method for Zinc, Method 8009, Hach Handbook for Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. |
| IB—Other | 49 | "Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography", by R.F. Addison and R.G. Ackman, Journal of Chromatography, Volume 47, No. 3, pp. 421–426, 1970. Available in most public libraries. Back volumes of the Journal of Chromatography are available from Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164. Cost available from publisher. |
| IB—Other (USGS) | 69 | "Water temperature-influential factors, field measurement, and data presentation," by H.H. Stevens, Jr., J. Ficke, and G.F. Smoot: USGS–TWRI Book 1, Chapter D1. 1975. 65 pages, $1.60, Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Prices are subject to change. |
| IB—Other (USGS) | 42 | |
| ID—Other (USGS) | 1, 11, 14, 17–20, 23, 25, 28, 29, 35, 37, 40–42, 44, 46, 52, 66, 69 | "Methods for analysis of organic substances in water," by D. F. Goerlitz and Eugene Brown: USGS–TWRI. Book 5, Chapter A3, 1972, 40 pages, $.90. Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Prices are subject to change. |
| IC—EPA | 1–12, 14–55, 57–97 | The full texts of Methods 601–613, 624, 625, 1624, and 1625 are printed in appendix A of this Part 136. The full text for determining the method detection limit when using the test procedures is given in Appendix B of this Part 136. |
| ID—EPA | 1, 8–11, 15, 18, 19, 20, 28, 32–36, 40, 41, 69 | |
| IC—Other (EPA) | 7, 13, 22, 24, 27, 56, 66, 76–83, 88, 89, 91, 93 | "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," Environmental Monitoring and Support Laboratory, United States Environmental Protection Agency, Cincinnati, Ohio 1978. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268. |
| ID—Other | 1–8, 11–13, 15–24, 26, 28, 30–33, 35, 38–41, 43–54, 56–70 | |
| IE—EPA | 1–5 | "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," EPA–600/4–80–032 (1980 Update), United States Environmental Protection Agency, 1980. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268. |
| IE—USGS | 1–5 | "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," by M.J. Fishman and Eugene Brown; U.S. Geological Survey Open File Report 76–77 (1976). $13.50. Available from: U.S. Geological Survey, Branch Distribution, 1200 South Eads Street, Arlington, VA 22202. |

The full texts of all the test procedures cited are available for inspection at the Office of the Federal Register Information Center, Room 8301, 1110 L Street, N.W., Washington, D.C. 20408.

7. In section 136.3 a new paragraph (e) is added together with a new Table II entitled, "Table II, Required Containers, Preservation Techniques, and Holding Times," to read as follows:

**§ 136.3 Identification of test procedures.**

* * * * *

(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters cited in Tables IA, IB, IC, ID, and IE are

prescribed in Table II. Any person may apply for a variance from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the Regional Administrator in the Region in which the discharge will occur. Sufficient data should be provided to assure such variance does not adversely affect the integrity of the sample. Such data will be forwarded by the Regional Administrator to the Director of the Environmental Monitoring and Support Laboratory in Cincinnati, Ohio for technical review and recommendations for action on the variance application. Upon receipt of the recommendations from the Director of the Environmental Monitoring and Support Laboratory, the Regional Administrator may grant a variance applicable to the specific discharge to the applicant. A decision to approve or deny a variance will be made within 90 days of receipt of the application by the Regional Administrator.

* * * * *

TABLE II.—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

| Parameter No./name | Container [1] | Preservation [2,3] | Maximum holding time [4] |
|---|---|---|---|
| Table IA—Bacterial Tests: | | | |
| 1–4. Coliform, fecal and total | P, G | Cool, 4°C, 0.008% $Na_2S_2O_3$ [5] | 6 hours. |
| 5. Fecal streptococci | P, G | do | Do. |
| Table IB—Inorganic Tests: | | | |
| 1. Acidity | P, G | Cool, 4°C | 14 days. |
| 2. Alkalinity | P, G | do | Do. |
| 4. Ammonia | P, G | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days. |
| 9. Biochemical oxygen demand | P, G | Cool, 4°C | 48 hours. |
| 11. Bromide | P, G | None required | 28 days. |
| 14. Biochemical oxygen demand, carbonaceous | P, G | Cool, 4°C | 48 hours. |
| 15. Chemical oxygen demand | P, G | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days. |
| 16. Chloride | P, G | None required | Do. |
| 17. Chlorine, total residual | P, G | do | Analyze immediately. |
| 21. Color | P, G | Cool, 4°C | 48 hours. |
| 23–24. Cyanide, total and amenable to chlorination | P, G | Cool, 4°C, NaOH to pH>12, 0.6g ascorbic acid [6] | 14 days.[6] |
| 25. Fluoride | P | None required | 28 days. |
| 27. Hardness | P, G | $HNO_3$ to pH<2, $H_2SO_4$ to pH<2 | 6 months. |
| 28. Hydrogen ion (pH) | P, G | None required | Analyze immediately. |
| 31, 43. Kjeldahl and organic nitrogen | P, G | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days. |
| Metals: [7] | | | |
| 18. Chromium VI | P, G | Cool, 4°C | 24 hours. |
| 35. Mercury | P, G | $HNO_3$ to pH<2 | 28 days. |
| 3, 5–8, 10, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals, except chromium VI and mercury. | P, G | do | 6 months. |
| 38. Nitrate | P, G | Cool, 4°C | 48 hours. |
| 39. Nitrate-nitrite | P, G | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days. |
| 40. Nitrite | P, G | Cool, 4°C | 48 hours. |
| 41. Oil and grease | G | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days. |
| 42. Organic carbon | P, G | Cool, 4°C, HCl or $H_2SO_4$ to pH<2 | Do. |
| 44. Orthophosphate | P, G | Filter immediately, Cool, 4°C | 48 hours. |
| 46. Oxygen, Dissolved Probe | G Bottle and top | None required | Analyze immediately. |
| 47. Winkler | do | Fix on site and store in dark | 8 hours. |
| 48. Phenols | G only | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days. |
| 49. Phosphorus (elemental) | G | Cool, 4°C | 48 hours. |
| 50. Phosphorus, total | P, G | Cool, 4°C, $H_2SO_4$ to pH<2 | 28 days. |
| 53. Residue, total | P, G | Cool, 4°C | 7 days. |
| 54. Residue, Filterable | P, G | do | 48 hours. |
| 55. Residue, Nonfilterable (TSS) | P, G | do | 7 days. |
| 56. Residue, Settleable | P, G | do | 48 hours. |
| 57. Residue, volatile | P, G | do | 7 days. |
| 61. Silica | P | do | 28 days. |
| 64. Specific conductance | P, G | do | Do. |
| 65. Sulfate | P, G | do | Do. |
| 66. Sulfide | P, G | Cool, 4°C add zinc acetate plus sodium hydroxide to pH>9. | 7 days. |
| 67. Sulfite | P, G | None required | Analyze immediately. |
| 68. Surfactants | P, G | Cool, 4°C | 48 hours. |
| 69. Temperature | P, G | None required | Analyze. |
| 73. Turbidity | P, G | Cool, 4°C | 48 hours. |
| Table IC—Organic Tests.[8] | | | |
| 13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 66, 68, 69, 92–95, 97. Purgeable Halocarbons. | G, Teflon-lined septum | Cool, 4°C, 0.008% $Na_2S_2O_3$ [9] | 14 days. |
| 6, 57, 90. Purgeable aromatic hydrocarbons | do | Cool, 4°C, 0.008% $Na_2S_2O_3$ [9], HC1 to pH2 [9] | Do. |
| 3, 4, Acrolein and acrylonitrile | do | Cool, 4°C, 0.008% $Na_2S_2O_3$ [9]; Adjust pH to 4–5 [10] | Do. |
| 23, 30, 44, 49, 53, 67, 70, 71, 83, 85, 96. Phenols [11] | G, Teflon-lined cap | Cool, 4°C, 0.008% $Na_2S_2O_3$ [9] | 7 days until extraction, 40 days after extraction. |
| 7, 38. Benzidines [11] | do | do | 7 days until extraction.[12] |
| 14, 17, 48, 50–52. Phthalate esters [11] | do | Cool, 4°C | 7 days until extraction; 40 days after extraction. |
| 72–74. Nitrosamines [11, 14] | do | Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3$ [5] | Do. |
| 76–82. PCBs [11] acrylonitrile | do | Cool, 4°C | Do. |
| 54, 55, 65, 69. Nitroaromatics and isophorone [11] | do | Cool, 4°C, 0.008% $Na_2S_2O_3$ [9] store in dark | Do. |
| 1, 2, 5, 8–12, 32, 33, 58, 59, 64, 68, 84, 86. Polynuclear aromatic hydrocarbons. [11] | do | do | Do. |
| 15, 16, 21, 31, 75. Haloethers [11] | do | Cool, 4°C, 0.008% $Na_2S_2O_3$ [5] | Do. |
| 29, 35–37, 60–63, 91. Chlorinated hydrocarbons [11] | do | Cool, 4°C | Do. |
| 87. TCDD [11] | do | Cool, 4°C, 0.008% $Na_2S_2O_3$ [9] | Do. |
| Table ID—Pesticides Tests: | | | |
| 1–70. Pesticides [11] | do | Cool, 4°C, pH 5–9 [15] | Do. |
| Table IE—Radiological Tests: | | | |
| 1–5. Alpha, beta and radium | P, G | $HNO_3$ to pH<2 | 6 months. |

**Table II Notes**

[1] Polyethylene (P) or Glass (G).

[2] Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

[3] When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid ($HNO_3$) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid ($H_2SO_4$) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See § 136.3(e) for details.

[5] Should only be used in the presence of residual chlorine.

[6] Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[7] Samples should be filtered immediately on-site before adding preservative for dissolved metals.

[8] Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[9] Sample receiving no pH adjustment must be analyzed within seven days of sampling.

[10] The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

[12] If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

[13] Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

[15] The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

8. Appendices A, B, and C are added to Part 136 to read as follows:

## APPENDIX A TO PART 136—METHODS FOR ORGANIC CHEMICAL ANALYSIS OF MUNICIPAL AND INDUSTRIAL WASTEWATER.

### Method 601—Purgeable Halocarbons

*1. Scope and Application*

1.1 This method covers the determination of 29 purgeable halocarbons.

The following parameters may be determined by this method:

| Parameter | STORET No. | CAS No. |
|---|---|---|
| Bromodichloromethane | 32101 | 75-27-4 |
| Bromoform | 32104 | 75-25-2 |
| Bromomethane | 34413 | 74-83-9 |
| Carbon tetrachloride | 32102 | 56-23-5 |
| Chlorobenzene | 34301 | 108-90-7 |
| Chloroethane | 34311 | 75-00-3 |
| 2-Chloroethylvinyl ether | 34576 | 100-75-8 |
| Chloroform | 32106 | 67-66-3 |
| Chloromethane | 34418 | 74-87-3 |
| Dibromochloromethane | 32105 | 124-48-1 |
| 1,2-Dichlorobenzene | 34536 | 95-50-1 |
| 1,3-Dichlorobenzene | 34566 | 541-73-1 |
| 1,4-Dichlorobenzene | 34571 | 106-46-7 |
| Dichlorodifluoromethane | 34668 | 75-71-8 |
| 1,1-Dichloroethane | 34496 | 75-34-3 |
| 1,2-Dichloroethane | 34531 | 107-06-2 |
| 1,1-Dichloroethene | 34501 | 75-35-4 |
| trans-1,2-Dichloroethene | 34546 | 156-60-5 |
| 1,2-Dichloropropane | 34541 | 78-87-5 |
| cis-1,3-Dichloropropene | 34704 | 10061-01-5 |
| trans-1,3-Dichloropropene | 34699 | 10061-02-6 |
| Methylene chloride | 34423 | 75-09-2 |
| 1,1,2,2-Tetrachloroethane | 34516 | 79-34-5 |
| Tetrachloroethene | 34475 | 127-18-4 |
| 1,1,1-Tetrachloroethane | 34506 | 71-55-6 |
| 1,1,2-Tetrachloroethane | 34511 | 79-00-5 |
| Tetrachloroethene | 39180 | 79-01-6 |
| Terichlorofluoromethane | 34488 | 75-69-4 |
| Vinyl chloride | 39715 | 75-01-4 |

1.2 This is a purge and trap gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 624 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for most of the parameters listed above.

1.3 The method detection limit (MDL, defined in Section 12.1) [1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The halocarbons are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the halocarbons are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the halocarbons onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the halocarbons which are then detected with a halide-specific detector.[2,3]

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

*3. Interferences*

3.1 Impurities in the purge gas and organic compounds outgassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

*4. Safety*

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [4-6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with a hole in the center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for 1 h before use.

5.2 Purge and trap system—The purge and trap system consists of three separate pieces of equipment: a purging device, trap, and desorber. Several complete systems are now commercially available.

5.2.1 The purging device must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging device illustrated in Figure 1 meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. The trap must be packed to contain the following minimum lengths of adsorbents: 1.0 cm of methyl silicone coated packing (Section 6.3.3), 7.7 cm of 2,6-diphenylene oxide polymer (Section 6.3.2), 7.7 cm of silica gel (Section 6.3.4), 7.7 cm of coconut charcoal (Section 6.3.1). If it is not necessary to analyze for dichlorodifluoromethane, the charcoal can be eliminated, and the polymer section lengthened to 15 cm. The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 200 °C. The desorber illustrated in Figure 2 meets these design criteria.

5.2.4 The purge and trap system may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

5.3 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.3.1 Column 1—8 ft long x 0.1 in. ID stainless steel or glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent. This column was used to develop the method performance statements in Section 12. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.3.2 Column 2—6 ft long x 0.1 in. ID stainless steel or glass, packed with chemically bonded n-octane on Porasil-C (100/120 mesh) or equivalent.

5.3.3 Detector—Electrolytic conductivity or microcoulometric detector. These types of detectors have proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1). The electrolytic conductivity detector was used to develop the method performance statements in Section 12. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25-μL, 0.006 in. ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Syringe—5-mL, gas-tight with shut-off valve.

5.8 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighing 0.0001 g.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can ge generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrasorb-300, Calgon Corp., or equivalent).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for 1 h. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Trap Materials:

6.3.1 Coconut charcoal—6/10 mesh sieved to 26 mesh, Barnebey Cheney, CA-580-26 lot # M-2649 or equivalent.

6.3.2 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.3.3 Methyl silicone packing—3% OV-1 on Chromosorb-W (60/80 mesh) or equivalent.

6.3.4 Silica gel—35/60 mesh, Davison, grade-15 or equivalent.

6.4 Methanol—Pesticide quality or equivalent.

6.5 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.5.1 Place about 9.8 mL of methanol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.5.2 Add the assayed reference material:

6.5.2.1 Liquid—Using a 100 μL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.5.2.2 Gases—To prepare standards for any of the six halocarbons that boil below 30 °C (bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid (the heavy gas will rapidly dissolve into the methanol).

6.5.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in μg/μL from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.5.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at −10 to −20 °C and protect from light.

6.5.5 Prepare fresh standards weekly for the six gases and 2-chloroethylvinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.

6.6 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Sections 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Assemble a purge and trap system that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Condition the trap for 10 min once daily prior to use.

7.2 Connect the purge and trap system to a gas chromatograph. The gas chromatograph must be operated using temperature and flow

rate conditions equivalent to those given in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a miminum of three concentration levels for each parameter by carefully adding 20.0 μL of one or more secondary dilution standards to 100, 500, or 1000 mL of reagent water. A 25-μL syringe with a 0.006 in. ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These aqueous standards can be stored up to 24 h, if held in sealed vials with zero headspace as described in Section 9.2. If not so stored, they must be discarded after 1 h.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.4 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compounds recommended for use as surrogate spikes in Section 8.7 have been used successfully as internal standards, because of their generally unique retention times.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.5 and 6.6. It is recommended that the secondary dilution standard be prepared at a concentration of 15 μg/mL of each internal standard compound. The addition of 10 μL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 μg/L.

7.4.3 Analyze each calibration standard according to Section 10, adding 10 μL of internal standard spiking solution directly to the syringe (Section 10.4). Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard.

$C_s$ = Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.5 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of a QC check sample.

7.5.1 Prepare the QC check sample as described in Section 8.2.2.

7.5.2 Analyze the QC check sample according to Section 10.

7.5.3 For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in Table 2. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual Q falls outside the range, proceed according to Section 7.5.4.

**Note:** The large number of parameters in Table 2 present a substantial probability that one or more will not meet the calibration acceptance criteria when all parameters are analyzed.

7.5.4 Repeat the test only for those parameters that failed to meet the calibration acceptance criteria. If the response for a parameter does not fall within the range in this second test, a new calibration curve, calibration factor, or RF must be prepared for that parameter according to Section 7.3 or 7.4.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 10.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Each day, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system are under control.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 10 μg/mL in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Prepare a QC check sample to contain 20 μg/L of each parameter by adding 200 μL of QC check sample concentrate to 100 mL of reagent water.

8.2.3 Analyze four 5-mL aliquots of the well-mixed QC check sample according to Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter of interest using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, then the system performance is unacceptable for that parameter.

**Note:** The large number of parameters in Table 2 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance

criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.3.

8.2.6.2 Beginning with Section 8.2.3, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.3.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 20 μg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.2 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second 5-mL sample aliquot with 10 μL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than 20 μg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X') using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 3, substituting X' for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X'/T)±2.44(100 S'/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

Note: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of parameters in Table 2 must be measured in the sample in Section 8.3, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spiked sample.

8.4.1 Prepare the QC check standard by adding 10 μL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 5 mL of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{p}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

8.7 The analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with surrogate halocarbons. A combination of bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane is recommended to encompass the range of the temperature program used in this method. From stock standard solutions prepared as in Section 6.5, add a volume to give 750 μg of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix and dilute to volume for a concentration of 15 ng/μL. Add 10 μL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. Prepare a fresh surrogate spiking solution on a weekly basis. If the internal standard calibration procedure is being used, the surrogate compounds may be added directly to the internal standard spiking solution (Section 7.4.2).

*9. Sample Collection, Preservation, and Handling*

9.1 All samples must be iced or refrigerated from the time of collection until analysis. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm $Cl_2$) to the empty sample bottle just prior to shipping to the sampling site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[8] Field test kits are available for this purpose.

9.2 Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for 1 min. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 All samples must be analyzed within 14 days of collection.[3]

*10. Procedure*

10.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are estimated retention times and MDL that can be achieved under these conditions. An example of the separations achieved by Column 1 is shown in Figure 5. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

10.2 Calibrate the system daily as described in Section 7.

10.3 Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the purge and trap system to purge (Figure 3). Open the syringe valve located on the purging device sample introduction needle.

10.4 Allow the sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual

air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 μL of the surrogate spiking solution (Section 8.7) and 10.0 μL of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore, then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for 11.0±0.1 min at ambient temperature.

10.7 After the 11-min purge time, attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode (Figure 4), and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for 4 min. If rapid heating of the trap cannot be achieved, the GC column must be used as a secondary trap by cooling it to 30 °C (subambient temperature, if poor peak geometry or random retention time problems persist) instead of the initial program temperature of 45 °C

10.8 While the trap is being desorbed into the gas chromatograph, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for 4 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

10.10 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.11 If the response for a peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

*11. Calculations*

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of the parameter being measured from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and Equation 2

Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})}{(A_{is})(RF)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard.

11.2 Report results in μg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

*12. Method Performance*

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence tha. the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[9] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.2 This method is recommended for use in the concentration range from the MDL to 1000×MDL. Direct aqueous injection techniques should be used to measure concentration levels above 1000×MDL.

12.3 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 8.0 to 500 μg/L.[9] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

*References*

1. 40 CFR Part 136, Appendix B.

2. Bellar, T.A., and Lichtenberg, J.J. "Determining Volatile Organics at Microgram-per-Litre-Levels by Gas Chromatography," *Journal of the American Water Works Association, 66,* 739 (1974).

3. Bellar, T.A., and Lichtenberg, J.J. "Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds," Proceedings from Symposium on Measurement of Organic Pollutants in Water and Wastewater, American Society for Testing and Materials, STP 686, C.E. Van Hall, editor, 1978.

4. "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, August 1977.

5. "OSHA Safety and Health Standards, General Industry" (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58-63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

8. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

9. "EPA Method Validation Study 23, Method 601 (Purgeable Halocarbons)," Report for EPA Contract 68-03-2856 (in preparation).

10. "Method Validation Data for EPA Method 601," Memorandum from B. Potter, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, November 10, 1983.

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

| Parameter | Retention time (min) | | Method detection limit (μg/L) |
|---|---|---|---|
| | Column 1 | Column 2 | |
| Chloromethane | 1.50 | 5.28 | 0.08 |
| Bromomethane | 2.17 | 7.05 | 1.18 |
| Dichlorodifluoromethane | 2.62 | nd | 1.81 |
| Vinyl chloride | 2.67 | 5.28 | 0.18 |
| Chloroethane | 3.33 | 8.68 | 0.52 |
| Methylene chloride | 5.25 | 10.1 | 0.25 |
| Trichlorofluoromethane | 7.18 | nd | nd |
| 1,1-Dichloroethene | 7.93 | 7.72 | 0.13 |
| 1,1-Dichloroethane | 9.30 | 12.6 | 0.07 |
| trans-1,2-Dichloroethene | 10.1 | 9.38 | 0.10 |

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS—Continued

| Parameter | Retention time (min) | | Method detection limit (µg/L) |
|---|---|---|---|
| | Column 1 | Column 2 | |
| Chloroform | 10.7 | 12.1 | 0.05 |
| 1,2-Dichloroethane | 11.4 | 15.4 | 0.03 |
| 1,1,1-Trichloroethane | 12.6 | 13.1 | 0.03 |
| Carbon tetrachloride | 13.0 | 14.4 | 0.12 |
| Bromodichloromethane | 13.7 | 14.6 | 0.10 |
| 1,2-Dichloropropane | 14.9 | 16.6 | 0.04 |
| cis-1,3-Dichloropropene | 15.2 | 16.6 | 0.34 |
| Trichloroethene | 15.8 | 13.1 | 0.12 |
| Dibromochloromethane | 16.5 | 16.6 | 0.09 |
| 1,1,2-Trichloroethane | 16.5 | 18.1 | 0.02 |
| trans-1,3-Dichloropropene | 16.5 | 18.0 | 0.20 |
| 2-Chloroethylvinyl ether | 18.0 | nd | 0.13 |
| Bromoform | 19.2 | 19.2 | 0.20 |
| 1,1,2,2-Tetrachloroethane | 21.6 | nd | 0.03 |
| Tetrachloroethene | 21.7 | 15.0 | 0.03 |
| Chlorobenzene | 24.2 | 18.8 | 0.25 |
| 1,3-Dichlorobenzene | 34.0 | 22.4 | 0.32 |
| 1,2-Dichlorobenzene | 34.9 | 23.5 | 0.15 |
| 1,4-Dichlorobenzene | 35.4 | 22.3 | 0.24 |

Column 1 conditions: Carbopack B (60/80 mesh) coated with 1% SP-1000 packed in an 8 ft x 0.1 in. ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 45 °C for 3 min then programmed at 8 °C/min to 220 °C and held for 15 min.

Column 2 conditions: Porisil-C (100/120 mesh) coated with n-octane packed in a 6 ft x 0.1 in. ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 50 °C for 3 min then programmed at 6 °C/min to 170 °C and held for 4 min.

nd = not determined.

TABLE 2.—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 601 *

| Parameter | Range for Q (µg/L) | Limit for s (µg/L) | Range for $\bar{X}$ (µg/L) | Range P, $P_s$ (%) |
|---|---|---|---|---|
| Bromodichloromethane | 15.2–24.8 | 4.3 | 10.7–32.0 | 42–172 |
| Bromoform | 14.7–25.3 | 4.7 | 5.0–29.3 | 13–159 |
| Bromomethane | 11.7–28.3 | 7.6 | 3.4–24.5 | D–144 |
| Carbon tetrachloride | 13.7–26.3 | 5.6 | 11.8–25.3 | 43–143 |
| Chlorobenzene | 14.4–25.6 | 5.0 | 10.2–27.4 | 38–150 |
| Chloroethane | 15.4–24.6 | 4.4 | 11.3–25.2 | 46–137 |
| 2-Chloroethylvinyl ether | 12.0–28.0 | 8.3 | 4.5–35.5 | 14–186 |
| Chloroform | 15.0–25.0 | 4.5 | 12.4–24.0 | 49–133 |
| Chloromethane | 11.9–28.1 | 7.4 | D–34.9 | D–193 |
| Dibromochloromethane | 13.1–26.9 | 6.3 | 7.9–35.1 | 24–191 |
| 1,2-Dichlorobenzene | 14.0–26.0 | 5.5 | 1.7–38.9 | D–208 |
| 1,3-Dichlorobenzene | 9.9–30.1 | 9.1 | 6.2–32.6 | 7–187 |
| 1,4-Dichlorobenzene | 13.9–26.1 | 5.5 | 11.5–25.5 | 42–143 |
| 1,1-Dichloroethane | 16.8–23.2 | 3.2 | 11.2–24.6 | 47–132 |
| 1,2-Dichloroethane | 14.3–25.7 | 5.2 | 13.0–26.5 | 51–147 |
| 1,1-Dichloroethene | 12.6–27.4 | 6.6 | 10.2–27.3 | 28–167 |
| trans-1,2-Dichloroethene | 12.8–27.2 | 6.4 | 11.4–27.1 | 38–155 |
| 1,2-Dichloropropane | 14.8–25.2 | 5.2 | 10.1–29.9 | 44–156 |
| cis-1,3-Dichloropropene | 12.8–27.2 | 7.3 | 6.2–33.8 | 22–178 |
| trans-1,3-Dichloropropene | 12.8–27.2 | 7.3 | 6.2–33.8 | 22–178 |
| Methylene chloride | 15.5–24.5 | 4.0 | 7.0–27.6 | 25–162 |
| 1,1,2,2-Tetrachloroethane | 9.8–30.2 | 9.2 | 6.6–31.8 | 8–184 |
| Tetrachloroethene | 14.0–26.0 | 5.4 | 8.1–29.6 | 26–162 |
| 1,1,1-Trichloroethane | 14.2–25.8 | 4.9 | 10.8–24.8 | 41–138 |
| 1,1,2-Trichloroethane | 15.7–24.3 | 3.9 | 9.6–25.4 | 39–136 |
| Trichloroethene | 15.4–24.6 | 4.2 | 9.2–26.6 | 35–146 |
| Trichlorofluoromethane | 13.3–26.7 | 6.0 | 7.4–28.1 | 21–156 |
| Vinyl chloride | 13.7–26.3 | 5.7 | 8.2–29.9 | 28–163 |

Q = Concentration measured in QC check sample, in µg/L (Section 7.5.3).
s = Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.
* Criteria were calculated assuming a QC check sample concentration of 20 µg/L.

**Note:** These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 601

| Parameter | Accuracy, as recovery, $\bar{X}'$ (µg/L) | Single analyst precision, $s_r'$ (µg/L) | Overall precision, $S'$ (µg/L) |
|---|---|---|---|
| Bromodichloromethane | 1.12C − 1.02 | $0.11\bar{X} + 0.04$ | $0.20\bar{X} + 1.00$ |
| Bromoform | 0.96C − 2.05 | $0.12\bar{X} + 0.58$ | $0.21\bar{X} + 2.41$ |
| Bromomethane | 0.76C − 1.27 | $0.28\bar{X} + 0.27$ | $0.36\bar{X} + 0.94$ |
| Carbon tetrachloride | 0.98C − 1.04 | $0.15\bar{X} + 0.38$ | $0.20\bar{X} + 0.39$ |
| Chlorobenzene | 1.00C − 1.23 | $0.15\bar{X} - 0.02$ | $0.18\bar{X} + 1.21$ |
| Chloroethane | 0.99C − 1.53 | $0.14\bar{X} - 0.13$ | $0.17\bar{X} + 0.63$ |
| 2-Chloroethylvinyl ether * | 1.00C | $0.20\bar{X}$ | $0.35\bar{X}$ |
| Chloroform | 0.93C − 0.39 | $0.13\bar{X} + 0.15$ | $0.19\bar{X} - 0.02$ |
| Chloromethane | 0.77C + 0.18 | $0.28\bar{X} - 0.31$ | $0.52\bar{X} + 1.31$ |
| Dibromochloromethane | 0.94C + 2.72 | $0.11\bar{X} + 1.10$ | $0.24\bar{X} + 1.68$ |
| 1,2-Dichlorobenzene | 0.93C + 1.70 | $0.20\bar{X} + 0.97$ | $0.13\bar{X} + 6.13$ |

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 601—Continued

| Parameter | Accuracy, as recovery, $\bar{X}'$ (μg/L) | Single analyst precision, $s_r'$ (μg/L) | Overall precision, $S'$ (μg/L) |
|---|---|---|---|
| 1,3-Dichlorobenzene | 0.95C+0.43 | 0.14$\bar{X}$+2.33 | 0.26$\bar{X}$+2.34 |
| 1,4-Dichlorobenzene | 0.93C−0.09 | 0.15$\bar{X}$+0.29 | 0.20$\bar{X}$+0.41 |
| 1,1-Dichloroethane | 0.95C−1.08 | 0.08$\bar{X}$+0.17 | 0.14$\bar{X}$+0.94 |
| 1,2-Dichloroethane | 1.04C−1.06 | 0.11$\bar{X}$+0.70 | 0.15$\bar{X}$+0.94 |
| 1,1-Dichloroethene | 0.98C−0.87 | 0.21$\bar{X}$−0.23 | 0.29$\bar{X}$−0.40 |
| trans-1,2-Dichloroethene | 0.97C−0.16 | 0.11$\bar{X}$+1.46 | 0.17$\bar{X}$+1.46 |
| 1,2-Dichloropropane * | 1.00C | 0.13$\bar{X}$ | 0.23$\bar{X}$ |
| cis-1,3-Dichloropropene * | 1.00C | 0.18$\bar{X}$ | 0.32$\bar{X}$ |
| trans-1,3 Dichloropropene * | 1.00C | 0.18$\bar{X}$ | 0.32$\bar{X}$ |
| Methylene chloride | 0.91C−0.93 | 0.11$\bar{X}$+0.33 | 0.21$\bar{X}$+1.43 |
| 1,1,2,2-Tetrachloroethene | 0.95C+0.19 | 0.14$\bar{X}$+2.41 | 0.23$\bar{X}$+2.79 |
| Tetrachloroethene | 0.94C+0.06 | 0.14$\bar{X}$+0.38 | 0.18$\bar{X}$+2.21 |
| 1,1,1-Trichloroethane | 0.90C−0.16 | 0.15$\bar{X}$+0.04 | 0.20$\bar{X}$+0.37 |
| 1,1,2-Trichloroethane | 0.86C+0.30 | 0.13$\bar{X}$−0.14 | 0.19$\bar{X}$+0.67 |
| Trichloroethene | 0.87C+0.48 | 0.13$\bar{X}$−0.03 | 0.23$\bar{X}$+0.30 |
| Trichlorofluoromethane | 0.89C−0.07 | 0.15$\bar{X}$+0.67 | 0.26$\bar{X}$+0.91 |
| Vinyl chloride | 0.97C−0.36 | 0.13$\bar{X}$+0.65 | 0.27$\bar{X}$+0.40 |

$\bar{X}'$ = Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
$s_r'$ = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
$S'$ = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C = True value for the concentration, in μg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in μg/L.
* Estimates based upon the performance in a single laboratory.[10]

BILLING CODE 6560-50-M

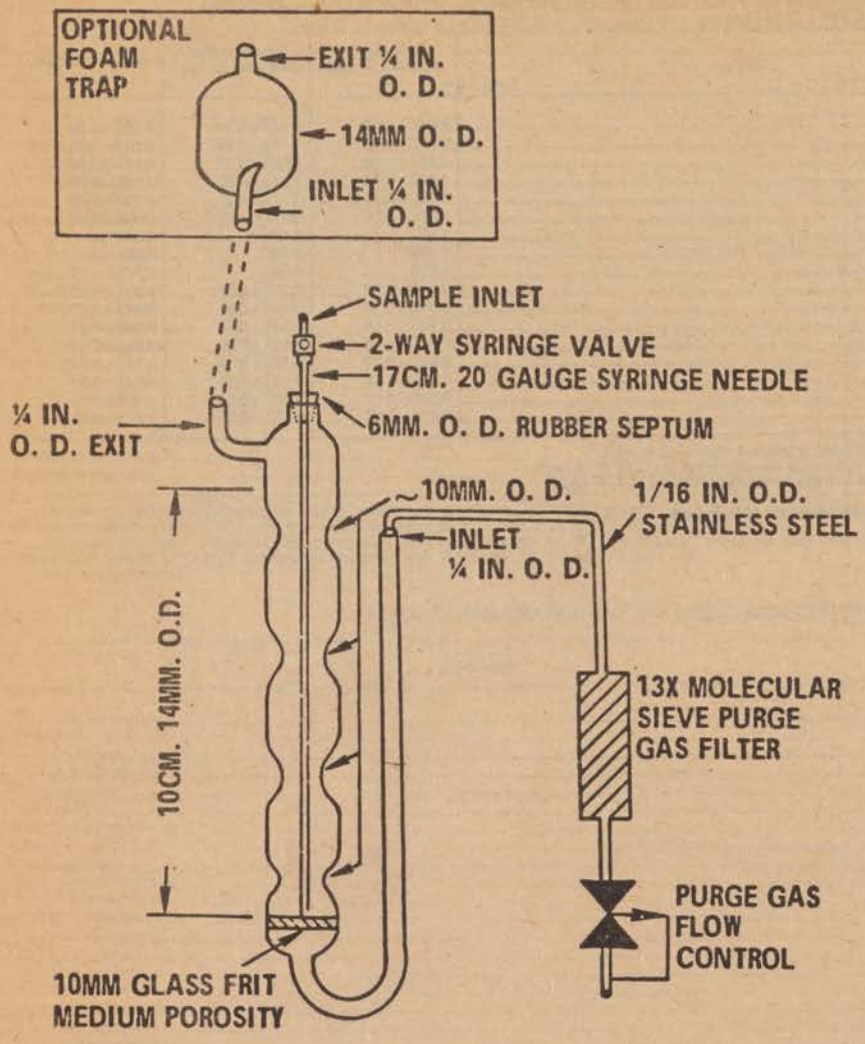



**Figure 1. Purging device.**

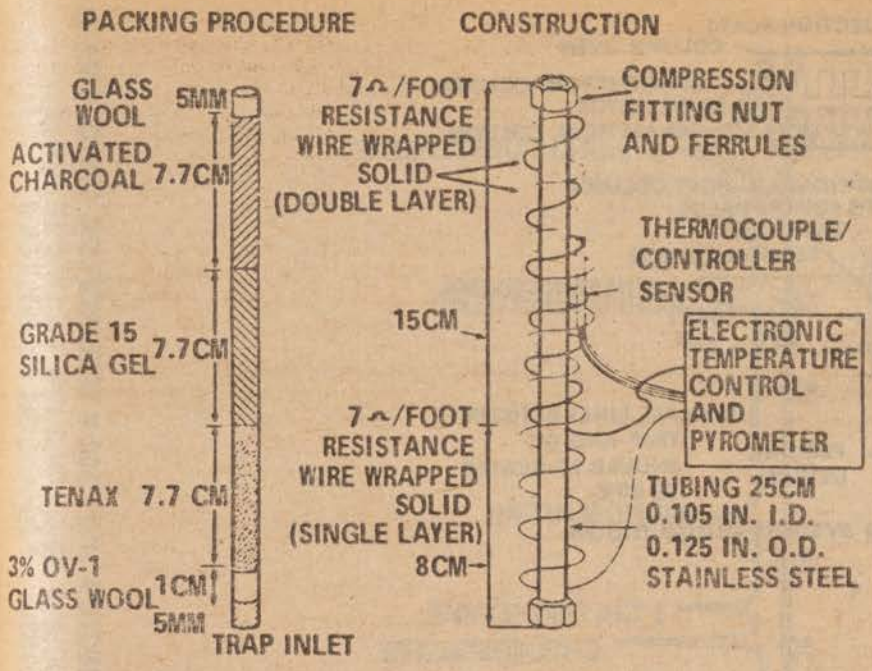



**Figure 2. Trap packings and construction to include desorb capability**

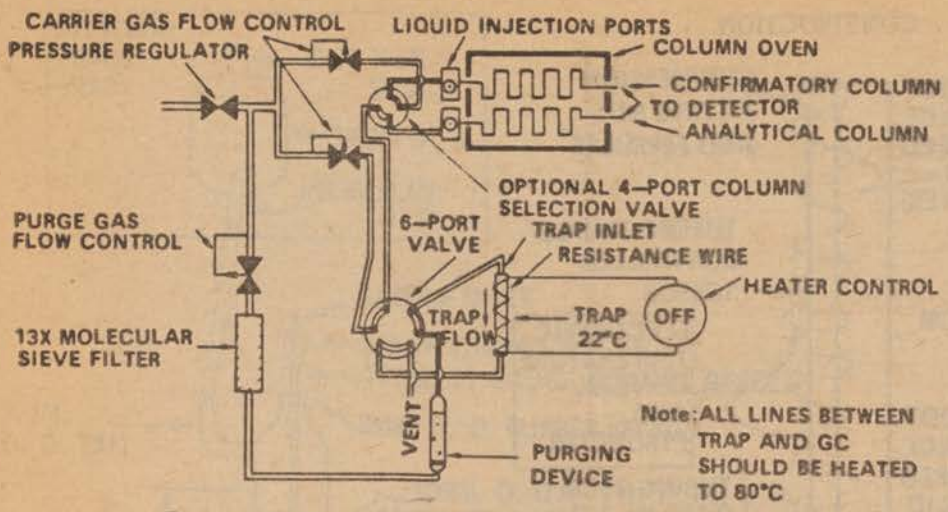



Figure 3. Purge and trap system-purge mode.

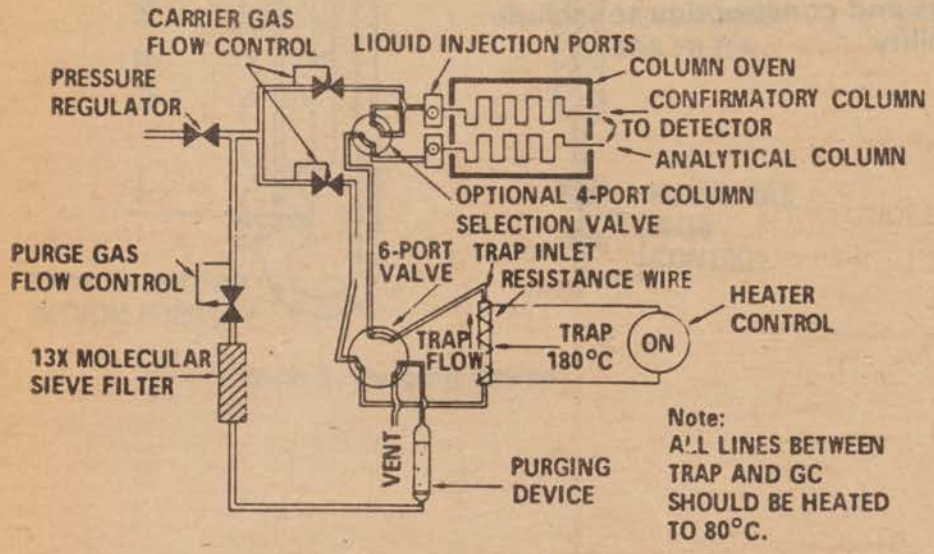



Figure 4. Purge and trap system - desorb mode.

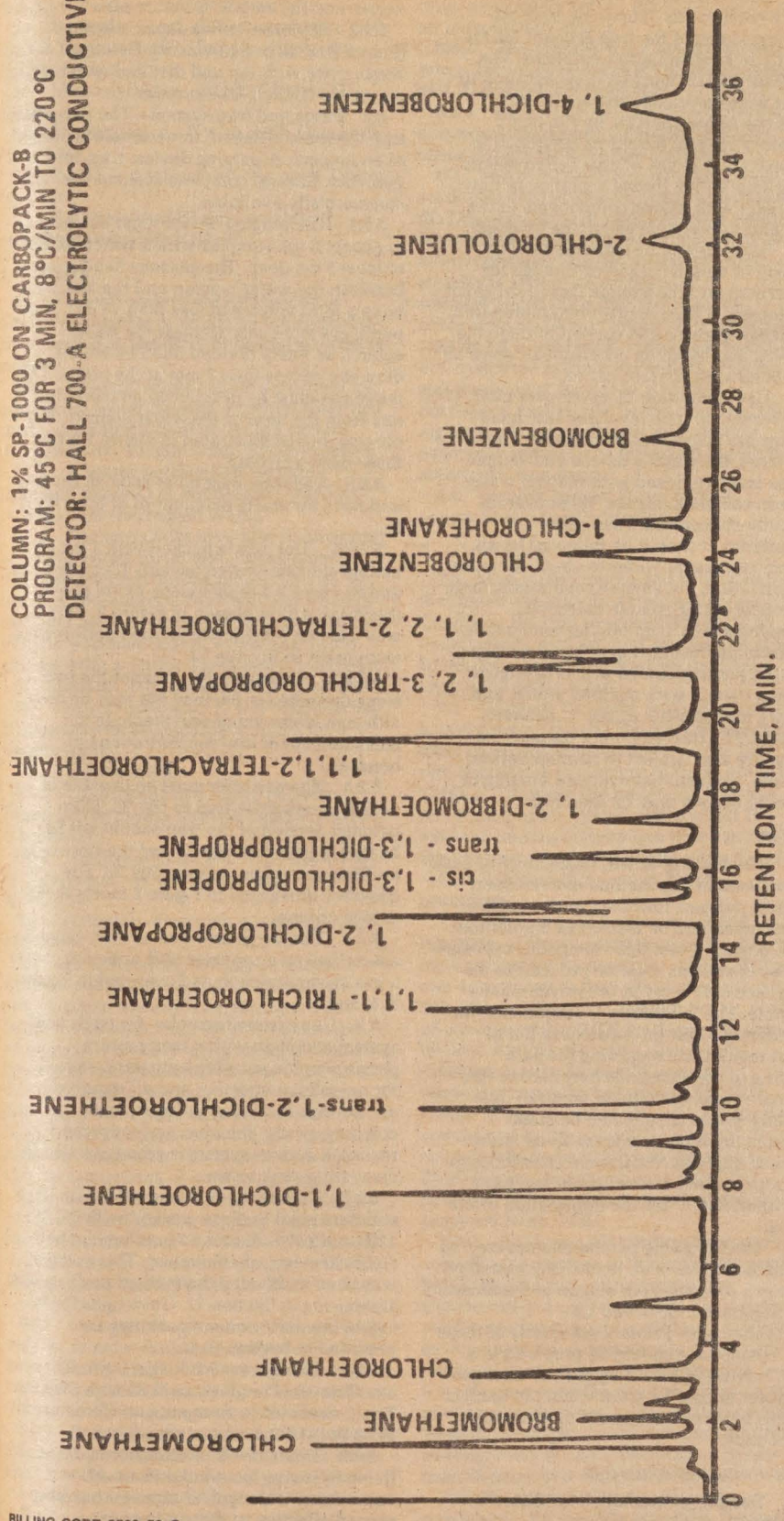



Figure 5. Gas chromatogram of purgeable halocarbons.

BILLING CODE 6560-50-C

**Method 602–Purgeable Aromatics**

*1. Scope and Application*

1.1 This method covers the determination of various purgeable aromatics. The following parameters may be determined by this method:

| Parameter | STORET No. | CAS No. |
|---|---|---|
| Benzene | 34030 | 71-43-2 |
| Chlorobenzene | 34301 | 108-90-7 |
| 1,2-Dichlorobenzene | 34536 | 95-50-1 |
| 1,3-Dichlorobenzene | 34566 | 541-73-1 |
| 1,4-Dichlorobenzene | 34571 | 106-46-7 |
| Ethylbenzene | 34371 | 100-41-4 |
| Toluene | 34010 | 108-88-3 |

1.2 This is a purge and trap gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 624 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above.

1.3 The method detection limit (MDL, defined in Section 12.1) [1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The aromatics are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the aromatics are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the aromatics onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the aromatics which are then detected with a photoionization detector.[2,3]

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

*3. Interferences*

3.1 Impurities in the purge gas and organic compounds outgassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high aromatic levels, it may be necessary to wash the purging device with a detergent solution, rinse it with distilled water, and then dry it in an oven at 105 °C between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

*4. Safety*

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [4-6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene and 1,4-dichlorobenzene. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with a hole in the center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for 1 h before use.

5.2 Purge and trap system—The purge and trap system consists of three separate pieces of equipment: A purging device, trap, and desorber. Several complete systems are now commercially available.

5.2.1 The purging device must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging device illustrated in Figure 1 meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in.

5.2.2.1 The trap is packed with 1 cm of methyl silicone coated packing (Section 6.4.2) and 23 cm of 2,6-diphenylene oxide polymer (Section 6.4.1) as shown in Figure 2. This trap was used to develop the method performance statements in Section 12.

5.2.2.2 Alternatively, either of the two traps described in Method 601 may be used, although water vapor will preclude the measurement of low concentrations of benzene.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 200 °C. The desorber illustrated in Figure 2 meets these design criteria.

5.2.4 The purge and trap system may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3, 4, and 5.

5.3 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.3.1 Column 1—6 ft long x 0.082 in. ID stainless steel or glass, packed with 5% SP-1200 and 1.75% Bentone-34 on Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statements in Section 12. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.3.2 Column 2—8 ft long x 0.1 in ID stainless steel or glass, packed with 5% 1,2,3-Tris(2-cyanoethoxy)propane on Chromosorb W-AW (60/80 mesh) or equivalent.

5.3.3 Detector—Photoionization detector (h-Nu Systems, Inc. Model PI-51-02 or equivalent). This type of detector has been proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), and was used to develop

the method performance statements in Section 12. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25-μL, 0.006 in. ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.8 Balance—Analytical, capable of accurately weighing 0.0001 g.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrasorb-300, Calgon Corp., or equivalent).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for 1 h. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Hydrochloric acid (1+1)—Add 50 mL of concentrated HCl (ACS) to 50 mL of reagent water.

6.4 Trap Materials:

6.4.1 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.4.2 Methyl silicone packing—3% OV-1 on Chromosorb-W (60/80 mesh) or equivalent.

6.5 Methanol—Pesticide quality or equivalent.

6.6 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids. Because of the toxicity of benzene and 1,4-dichlorobenzene, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.6.1 Place about 9.8 mL of methanol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.6.2 Using a 100-μL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.6.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in μg/μL from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.6.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store at 4 °C and protect from light.

6.6.5 All standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.

6.7 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Sections 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary solution standards must be stored with zero headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.8 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Assemble a purge and trap system that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Condition the trap for 10 min once daily prior to use.

7.2 Connect the purge and trap system to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate conditions equivalent to those given in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 μL of one or more secondary dilution standards to 100, 500, or 1000 mL of reagent water. A 25-μL syringe with a 0.006 in. ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These aqueous standards must be prepared fresh daily.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.4 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compound, α,α,α,-trifluorotoluene, recommended as a surrogate spiking compound in Section 8.7 has been used successfully as an internal standard.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Section 6.6 and 6.7. It is recommended that the secondary dilution standard be prepared at a concentration of 15 μg/mL of each internal standard compound. The addition of 10 μl of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 μg/L.

7.4.3 Analyze each calibration standard according to Section 10, adding 10 μL of internal standard spiking solution directly to the syringe (Section 10.4). Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard

$C_s$ = Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.5 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of a QC check sample.

7.5.1 Prepare the QC check sample as described in Section 8.2.2.

7.5.2 Analyze the QC check sample according to Section 10.

7.5.3 For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in Table 2. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual Q falls outside the range, a new calibration curve, calibration factor, or RF must be prepared for that parameter according to Section 7.3 or 7.4.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The mimimum requirements of this program consist of an initial demonstration of

laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 10.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Each day, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system are under control.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 10 μg/mL in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Prepare a QC check sample to contain 20 μg/L of each parameter by adding 200 μL of QC check sample concentrate to 100 mL of reagant water.

8.2.3 Analyze four 5-mL aliquots of the well-mixed QC check sample according to Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter of interest using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter.

**Note.**—The large number of parameters in Table 2 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.3.

8.2.6.2 Beginning with Section 8.2.3, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.3.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 20 μg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.2 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second 5-mL sample aliquot with 10 μL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than 20 μg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X') using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 3, substituting X' for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X'/T) ± 2.44(100 S'/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

**Note:** The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 10 μL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 5 mL of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

8.7 The analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with surrogate compounds (e.g. α, α, α,-trifluorotoluene) recommended to encompass the range of the temperature program used in this method. From stock standard solutions prepared as in Section 6.6, add a volume to give 750 μg of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix and dilute to volume for a concentration of 15 mg/μL. Add 10 μL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. Prepare a fresh surrogate spiking solution on a weekly basis. If the internal standard calibration procedure is being used, the surrogate compounds may be added directly to the internal standard spiking solution (Section 7.4.2).

*9. Sample Collection, Preservation, and Handling*

9.1 The samples must be iced or refrigerated from the time of collection until analysis. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm $Cl_2$) to the empty sample bottle just prior to shipping to the sampling site. EPA Method 330.4 or 330.5 may be used for measurement of residual chlorine.[8] Field test kits are available for this purpose.

9.2 Collect about 500 mL of sample in a clean container. Adjust the pH of the sample to about 2 by adding 1+1 HCl while stirring. Fill the sample bottle in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 All samples must be analyzed within 14 days of collection.[3]

*10. Procedure*

10.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are estimated retention times and MDL that can be achieved under these conditions. An example of the separations achieved by Column 1 is shown in Figure 6. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

10.2 Calibrate the system daily as described in Section 7.

10.3 Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the purge and trap system to purge (Figure 3). Open the syringe valve located on the purging device sample introduction needle.

10.4 Allow the sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 μL of the surrogate spiking solution (Section 8.7) and 10.0 μL of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore, then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for 12.0±0.1 min at ambient temperature.

10.7 After the 12-min purge time, disconnect the purging device from the trap. Dry the trap by maintaining a flow of 40 mL/min of dry purge gas through it for 6 min (Figure 4). If the purging device has no provision for bypassing the purger for this step, a dry purger should be inserted into the device to minimize moisture in the gas. Attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode (Figure 5), and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for 4 min. If rapid heating of the trap cannot be achieved, the GC column must be used as a secondary trap by cooling it to 30 °C (subambient temperature, if poor peak geometry and random retention time problems persist) instead of the initial program temperature of 50 °C.

10.8 While the trap is being desorbed into the gas chromatograph column, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for 4 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s, then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C. After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

10.10 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.11 If the response for a peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

*11. Calculations*

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of the parameter being measured from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and Equation 2.

Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})}{(A_{is})(RF)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard.

11.2 Report results in μg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

*12. Method Performance*

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[9] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.2 This method has been demonstrated to be applicable for the concentration range from the MDL to 1000 X MDL.[9] Direct aqueous injection techniques should be used to measure concentration levels above 1000 x MDL.

12.3 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 2.1 to 550 μg/L.[9] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample

matrix. Linear equations to describe these relationships are presented in Table 3.

*References*

1. 40 CFR Part 136, Appendix B.

2. Bellar, T.A., and Lichtenberg, J.J. *Journal American Water Works Association, 66,* 739 (1974).

3. Bellar, T.A., and Lichtenberg, J.J. "Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds," Proceedings of Symposium on Measurement of Organic Pollutants in Water and Wastewater. American Society for Testing and Materials, STP 686, C.E. Van Hall, editor, 1978.

4. "Carcinogens—Working with Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.

5. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58-63 (1983). (The value 2.44 used in the equation in Section 8.3.3. is two times the value 1.22 derived in this report.)

8. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual." Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. March 1979.

9. "EPA Method Validation Study 24, Method 602 (Purgeable Aromatics)," Report for EPA Contract 68-03-2856 (In preparation).

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

| Parameter | Retention time (min) | | Method detection limit (μg/L) |
|---|---|---|---|
| | Column 1 | Column 2 | |
| Benzene | 3.33 | 2.75 | 0.2 |
| Toluene | 5.75 | 4.25 | 0.2 |
| Ethylbenzene | 8.25 | 6.25 | 0.2 |
| Chlorobenzene | 9.17 | 8.02 | 0.2 |
| 1,4-Dichlorobenzene | 16.8 | 16.2 | 0.3 |
| 1,3-Dichlorobenzene | 18.2 | 15.0 | 0.4 |
| 1,2-Dichlorobenzene | 25.9 | 19.4 | 0.4 |

Column 1 conditions: Supelcoport (100/120 mesh) coated with 5% SP-1200/1.75% Bentone-34 packed in a 6 ft x 0.085 in. ID stainless steel column with helium carrier gas at 36 mL/min flow rate. Column temperature held at 50 °C for 2 min then programmed at 6 °C/min to 90 °C for a final hold.

Column 2 conditions: Chromosorb W-AW (60/80 mesh) coated with 5% 1,2,3-Tris(2-cyanoethyoxy)propane packed in a 6 ft x 0.085 in. ID stainless steel column with helium carrier gas at 30 mL/min flow rate. Column temperature held at 40 °C for 2 min then programmed at 2 °C/min to 100 °C for a final hold.

TABLE 2.—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 602 *

| Parameter | Range for Q (μg/L) | Limit for s (μg/L) | Range for $\bar{X}$ (μg/L) | Range for $P_s$, $P_s$ (%) |
|---|---|---|---|---|
| Benzene | 15.4-24.6 | 4.1 | 10.0-27.9 | 39-150 |
| Chlorobenzene | 16.1-23.9 | 3.5 | 12.7-25.4 | 55-135 |
| 1,2-Dichlorobenzene | 13.6-26.4 | 5.8 | 10.6-27.6 | 37-154 |
| 1,3-Dichlorobenzene | 14.5-25.5 | 5.0 | 12.8-25.5 | 50-141 |
| 1,4-Dichlorobenzene | 13.9-26.1 | 5.5 | 11.6-25.5 | 42-143 |
| Ethylbenzene | 12.6-27.4 | 6.7 | 10.0-28.2 | 32-160 |
| Toluene | 15.5-24.5 | 4.0 | 11.2-27.7 | 46-148 |

Q = Concentration measured in QC check sample, in μg/L (Section 7.5.3).
s = Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
$P_s$, P = Percent recovery measured (Section 8.3.2, Section 8.4.2).
* Criteria were calculated assuming a QC check sample concentration of 20 μg/L.

Note: These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 602

| Parameter | Accuracy, as recovery, X' (μg/L) | Single analyst precision, s' (μg/L) | Overall precision, S' (μg/L) |
|---|---|---|---|
| Benzene | 0.92C + 0.57 | 0.09$\bar{X}$ + 0.59 | 0.21$\bar{X}$ + 0.56 |
| Chlorobenzene | 0.95C + 0.02 | 0.09$\bar{X}$ + 0.23 | 0.17$\bar{X}$ + 0.10 |
| 1,2-Dichlorobenzene | 0.93C + 0.52 | 0.17$\bar{X}$ − 0.04 | 0.22$\bar{X}$ + 0.53 |
| 1,3-Dichlorobenzene | 0.96C − 0.04 | 0.15$\bar{X}$ − 0.10 | 0.19$\bar{X}$ + 0.09 |
| 1,4-Dichlorobenzene | 0.93C + 0.09 | 0.15$\bar{X}$ + 0.28 | 0.20$\bar{X}$ + 0.41 |
| Ethylbenzene | 0.94C + 0.31 | 0.17$\bar{X}$ + 0.46 | 0.26$\bar{X}$ + 0.23 |
| Toluene | 0.94C + 0.65 | 0.09$\bar{X}$ + 0.48 | 0.18$\bar{X}$ + 0.71 |

X' = Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
s' = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C = True value for the Concentration, in μg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in μg/L.

BILLING CODE 6560-50-M

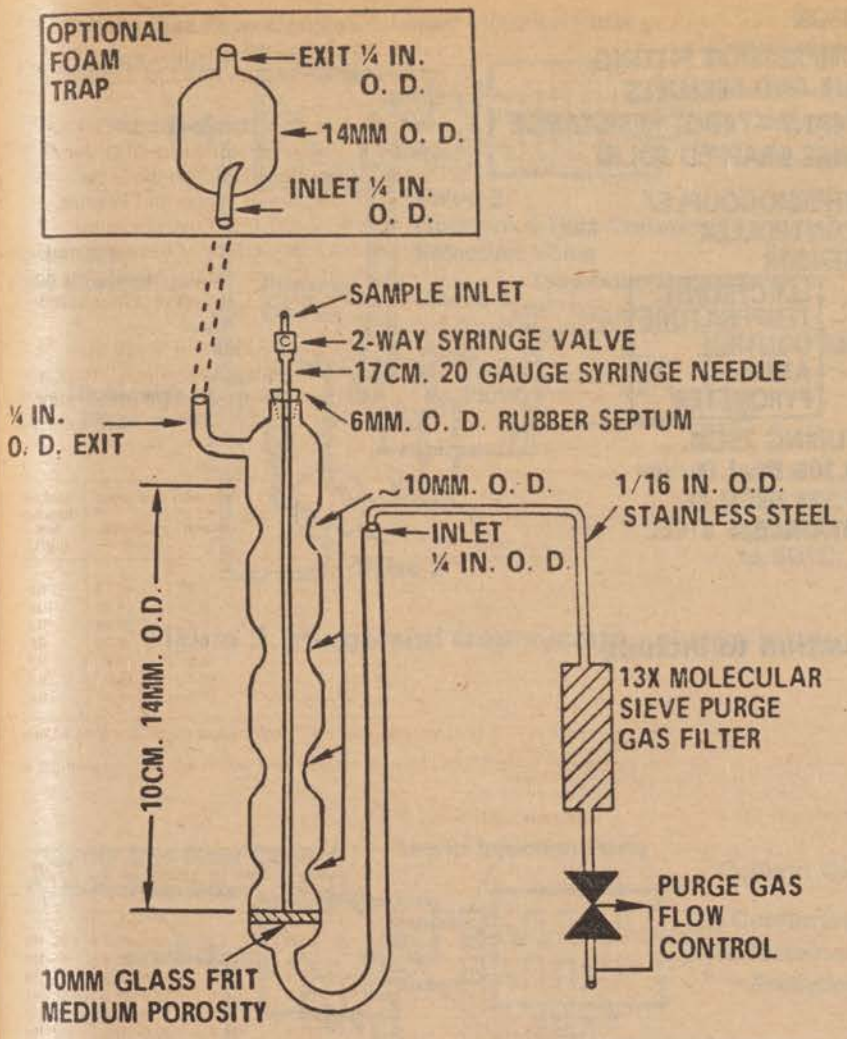



**Figure 1. Purging device.**

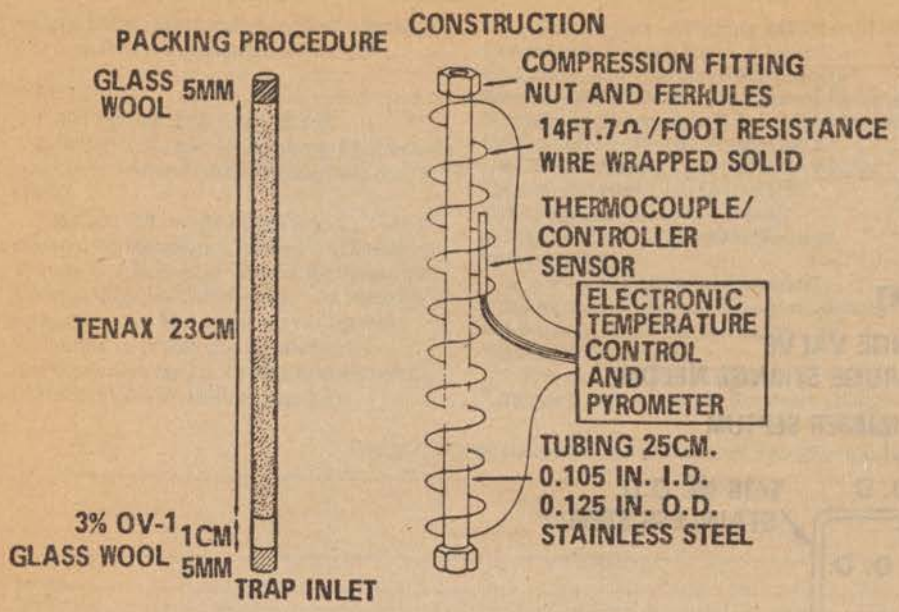



**Figure 2. Trap packings and construction to include desorb capability.**

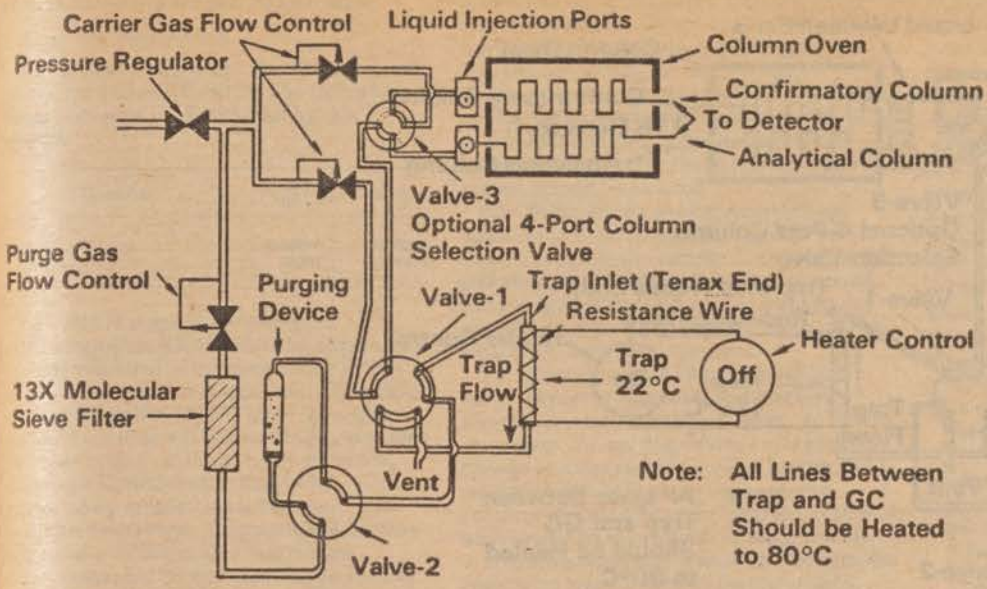



**Figure 3. Purge and trap system - purge mode.**

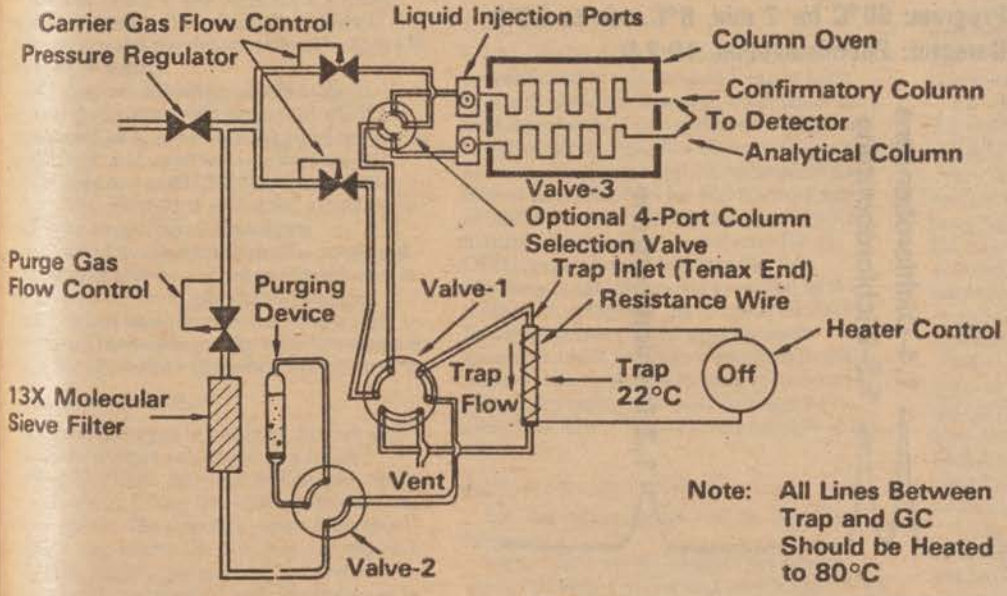



**Figure 4. Purge and trap system-dry mode.**

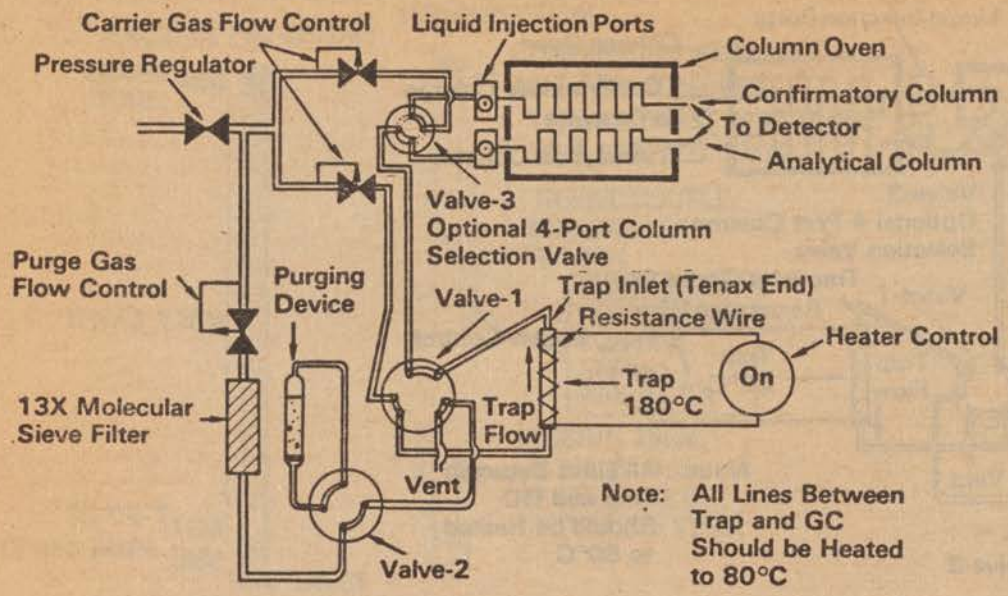



**Figure 5. Purge and trap system-desorb mode.**

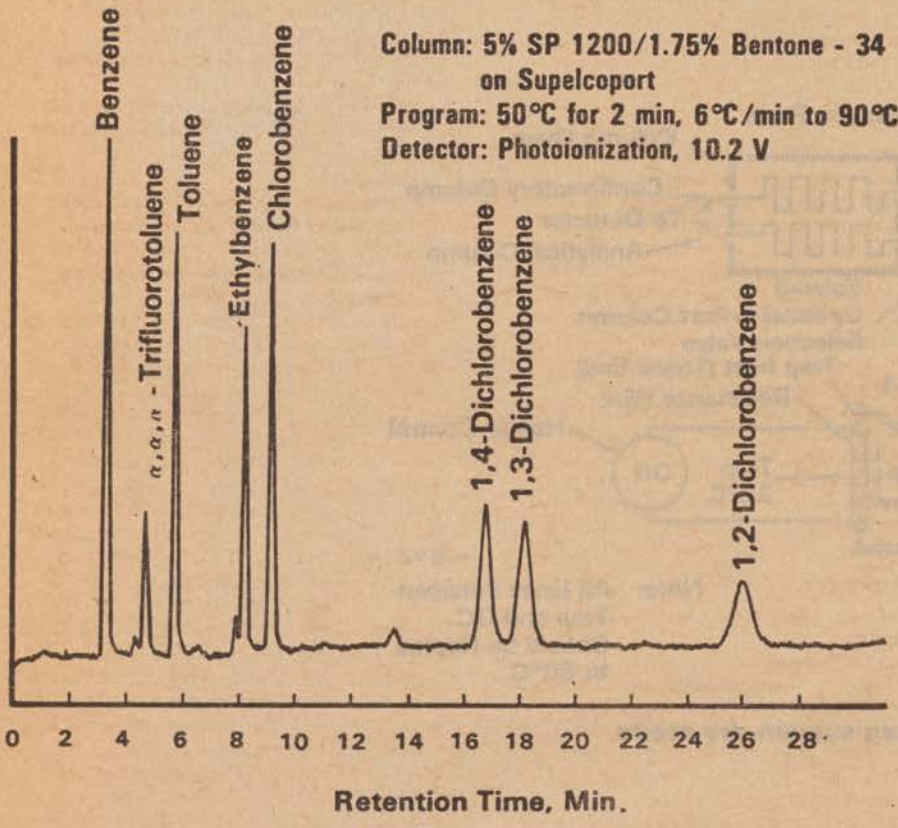



**Figure 6. Gas chromatogram of purgeable aromatics.**



BILLING CODE 6560-50-C

## Method 603—Acrolein and Acrylonitrile

*1. Scope and Application*

1.1 This method covers the determination of acrolein and acrylonitrile. The following parameters may be determined by this method:

| Parameter | STORET No. | CAS No. |
|---|---|---|
| Acrolein | 34210 | 107-02-8 |
| Acrylonitrile | 34215 | 107-13-1 |

1.2 This is a purge and trap gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for either or both of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 624 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for the parameters listed above, if used with the purge and trap conditions described in this method.

1.3 The method detection limit (MDL, defined in Section 12.1) [1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 An inert gas is bubbled through a 5-ml water sample contained in a heated purging chamber. Acrolein and acrylonitrile are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the analytes are trapped. After the purge is completed, the trap is heated and backflushed with the inert gas to desorb the compound onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the analytes which are then detected with a flame ionization detector.[2,3]

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from the interferences that may occur.

*3. Interferences*

3.1 Impurities in the purge gas and organic compound outgassing from the plumbing of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed between samples with reagent water. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high analyte levels, it may be necessary to wash the purging device with a detergent solution, rinse it with distilled water, and then dry it in an oven at 105 °C between analyses. The trap and other parts of the system are also subject to contamination, therefore, frequent bakeout and purging of the entire system may be required.

*4. Safety*

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this view point, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [4-6] for the information of the analyst.

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with a hole in the center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water and dry at 105 °C for 1 h before use.

5.2 Purge and trap system—The purge and trap system consists of three separate pieces of equipment: a purging device, trap, and desorber. Several complete systems are now commercially available.

5.2.1 The purging device must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging device must be capable of being heated to 85 °C within 3.0 min after transfer of the sample to the purging device and being held at 85 ±2 °C during the purge cycle. The entire water column in the purging device must be heated. Design of this modification to the standard purging device is optional, however, use of a water bath is suggested.

5.2.1.1 Heating mantle—To be used to heat water bath.

5.2.1.2 Temperature controller—Equipped with thermocouple/sensor to accurately control water bath temperature to ±2 °C. The purging device illustrated in Figure 1 meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. The trap must be packed to contain 1.0 cm of methyl silicone coated packing (Section 6.5.2) and 23 cm of 2,6-diphenylene oxide polymer (Section 6.5.1). The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The desorber illustrated in Figure 2 meets these design criteria.

5.2.4 The purge and trap system may be assembled as a separate unit as illustrated in Figure 3 or be coupled to a gas chromatograph.

5.3 pH paper—Narrow pH range, about 3.5 to 5.5 (Fisher Scientific Short Range Alkacid No. 2, #14-837-2 or equivalent).

5.4 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.4.1 Column 1—10 ft long x 2 mm ID glass or stainless steel, packed with Porapak-QS (80/100 mesh) or equivalent. This column was used to develop the method performance statements in Section 12. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.4.2 Column 2—6 ft long x 0.1 in. ID glass or stainless steel, packed with Chromosorb 101 (60/80 mesh) or equivalent.

5.4.3 Detector—Flame ionization detector. This type of detector has proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 12. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.5 Syringes—5-mL, glass hypodermic with Luerlok tip (two each).

5.6 Micro syringes—25-μL, 0.006 in. ID needle.

5.7 Syringe valve—2-way, with Luer ends (three each).

5.8 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighint 0.0001 g.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrasorb-300, Calgon Corp., or equivalent).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Regent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for 1 h. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.4 Hydrochloric acid (1+1)—Slowly, add 50 mL of concentrated HCl (ACS) to 50 mL of reagent water.

6.5 Trap Materials:

6.5.1 2,6-Diphenylene oxide polymer—Tenax (60/80 mesh), chromatographic grade or equivalent.

6.5.2 Methyl silicone packing—3% OV-1 on Chromosorb-W (60/80 mesh) or equivalent.

6.6 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in reagent water using assayed liquids. Since acrolein and acrylonitrile are lachrymators, primary dilutions of these compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.6.1 Place about 9.8 mL of reagent water into a 10-mL ground glass stoppered volumetric flask. For acrolein standards the reagent water must be adjusted to pH 4 to 5. Weight the flask to the nearest 0.1 mg.

6.6.2 Using a 100-μL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the water without contacting the neck of the flask.

6.6.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in μg/μL from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Optionally, stock standard solutions may be prepared using the pure standard material by volumetrically measuring the appropriate amounts and determining the weight of the material using the density of the material. Commercially prepared stock standards may be used at any concentration if they are certified by the manufactaurer or by an independent source.

6.6.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store at 4 °C and protect from light.

6.6.5 Prepare fresh standards daily.

6.7 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in reagent water that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Section 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary dilution standards should be prepared daily and stored at 4 °C.

6.8 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Assemble a purge and trap system that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Condition the trap for 10 min once daily prior to use.

7.2 Connect the purge and trap system to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate conditions equivalent to those given in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 μL of one or more secondary dilution standards to 100, 500, or 1000 mL of reagent water. A 25-μL syringe with a 0.006 in. ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These standards must be prepared fresh daily.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration of the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (< 10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.4 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.6 and 6.7. It is recommended that the secondary dilution standard be prepared at a concentration of 15 μg/mL of each internal standard compound. The addition of 10 μL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 μg/L.

7.4.3 Analyze each calibration standard according to Section 10, adding 10 μL of internal standard spiking solution directly to the syringe (Section 10.4). Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard.

$C_s$ = Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.5 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of a QC check sample.

7.5.1 Prepare the QC check sample as described in Section 8.2.2.

7.5.2 Analyze the QC check sample according to Section 10.

7.5.3 For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in Table 2. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual Q falls outside the range, a new calibration curve, calibration factor, or RF must be prepared for that parameter according to Section 7.3 or 7.4.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check

standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an inital, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 10.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Each day, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system are under control.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 25 μg/mL in reagent water. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Prepare a QC check sample to contain 50 μg/L of each parameter by adding 200 μL of QC check sample concentrate to 100 mL of reagent water.

8.2.3 Analyze four 5-mL aliquots of the well-mixed QC check sample according to Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 3. If s and $\bar{X}$ for all prameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If either s exceeds the precision limit or either $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter. Locate and correct the source of the problem and repeat the test for each compound of interest.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 50 μg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.2 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second 5-mL sample aliquot with 10 μL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 3. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed. NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 10 μL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 5 mL of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 3. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outsied the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($S_p$). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

*9. Sample Collection, Preservation, and Handling*

9.1 All samples must be iced or refrigerated from the time of collection until analysis. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm $Cl_2$) to the empty sample bottle just prior to shipping to the sampling site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[8] Field test kits are available for this purpose.

9.2 If acrolein is to be analyzed, collect about 500 mL of sample in a clean glass container. Adjust the pH of the sample to 4 to 5 using acid or base, measuring with narrow range pH paper. Samples for acrolein analysis receiving no pH adjustment must be analyzed within 3 days of sampling.

9.3 Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative

has been added, shake vigorously for 1 min. Maintain the hermetic seal on the sample bottle until time of analysis.

9.4 All samples must be analyzed within 14 days of collection.[3]

*10. Procedure*

10.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are estimated retention times and MDL that can be achieved under these conditions. An example of the separations achieved by Columnm 1 is shown in Figure 5. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

10.2 Calibrate the system daily as described in Section 7.

10.3 Adjust the purge gas (nitrogen or helium) flow rate to 20 mL/min. Attach the trap inlet to the purging device, and set the purge and trap system to purge (Figure 3). Open the syringe valve located on the purging device sample introduction needle.

10.4 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 $\mu$L of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for 15.0 $\pm$ 0.1 min while heating at 85 $\pm$ 2 °C.

10.7 After the 15-min purge time, attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode (Figure 4), and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for 1.5 min.

10.8 While the trap is being desorbed into the gas chromatograph, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for 1.5 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 210 °C. After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

10.10 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

*11. Calculations*

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of the parameter being measured from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and Equation 2.

Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})}{(A_{is})(RF)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard.

11.2 Report results in $\mu$g/L without correction for recovery data. All QC data obtained should be reported with the sample results.

*12. Method Performance*

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[9] The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.2 This method is recommended for the concentration range from the MDL to 1,000 $\times$ MDL. Direct aqueous injection techniques should be used to measure concentration levels above 1,000 $\times$ MDL.

12.3 In a single laboratory (Battelle-Columbus), the average recoveries and standard deviations presented in Table 2 were obtained.[9] Seven replicate samples were analyzed at each spike level.

*References*

1. 40 CFR Part 136, Appendix B.

2. Bellar, T.A., and Lichtenberg, J.J. "Determining Volatile Organics at Microgram-per-Litre-Levels by Gas Chromatography," *Journal American Water Works Association, 66,* 739 (1974).

3. Kerns, E.H., et al. "Determination of Acrolein and Acrylonitrile in Water by Heated Purge and Trap Technique," 1980, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

4. "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58–63 (1983).

8. "Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

9. "Evaluation of Method 603," Final report for EPA Contract 68–03–1760 (In preparation).

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

| Parameter | Retention time (min) | | Method detection limit ($\mu$g/L) |
|---|---|---|---|
| | Column 1 | Column 2 | |
| Acrolein | 10.6 | 8.2 | 0.7 |
| Acrylonitrile | 12.7 | 9.8 | 0.5 |

Column 1 conditions: Porapak-QS (80/100 mesh) packed in a 10 ft $\times$ 2 mm ID glass or stainless steel column with helium carrier gas at 30 mL/min flow rate. Column temperature held isothermal at 110 °C for 1.5 min (during desorption), then heated as rapidly as possible to 150 °C and held for 20 min; column bakeout at 190 °C for 10 min.[9]

Column 2 conditions: Chromosorb 101 (60/80 mesh) packed in a 6 ft. x 0.1 in. ID glass or stainless steel column with helium carrier gas at 40 mL/min flow rate. Column temperature held isothermal at 80°C for 4 min, then programmed at 50°C/min to 120°C and held for 12 min.

TABLE 2.—SINGLE LABORATORY ACCURACY AND PRECISION—METHOD 603

| Parameter | Sample matrix | Spike conc. (μg/L) | Average recovery (μg/L) | Standard deviation (μg/L) | Average percent recovery |
|---|---|---|---|---|---|
| Acrolein | RW | 5.0 | 5.2 | 0.2 | 104 |
| | RW | 50.0 | 51.4 | 0.7 | 103 |
| | POTW | 5.0 | 4.0 | 0.2 | 80 |
| | POTW | 50.0 | 44.4 | 0.8 | 89 |
| | IW | 5.0 | 0.1 | 0.1 | 2 |
| | IW | 100.0 | 9.3 | 1.1 | 9 |
| Acrylonitrile | RW | 5.0 | 4.2 | 0.2 | 84 |
| | RW | 50.0 | 51.4 | 1.5 | 103 |
| | POTW | 20.0 | 20.1 | 0.8 | 100 |
| | POTW | 100.0 | 101.3 | 1.5 | 101 |
| | IW | 10.0 | 9.1 | 0.8 | 91 |
| | IW | 100.0 | 104.0 | 3.2 | 104 |

RW = Reagent water.
POTW = Prechlorination secondary effluent from a municipal sewage treatment plant.
IW = Industrial wastewater containing an unidentified acrolein reactant.

TABLE 3.—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 603 *

| Parameter | Range for Q (μg/L) | Limit for S (μg/L) | Range for X (μg/L) | Range for P, $P_s$ (%) |
|---|---|---|---|---|
| Acrolein | 45.9–54.1 | 4.6 | 42.9–60.1 | 88–118 |
| Acrylonitrile | 41.2–58.8 | 9.9 | 33.1–69.9 | 71–135 |

Q = Concentration measured in QC check sample, in μg/L (Section 7.5.3).
s = Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).
X = Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
P, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2).
* = Criteria were calculated assuming a QC check sample concentration of 50 μg/L.*

BILLING CODE 6560-50-M

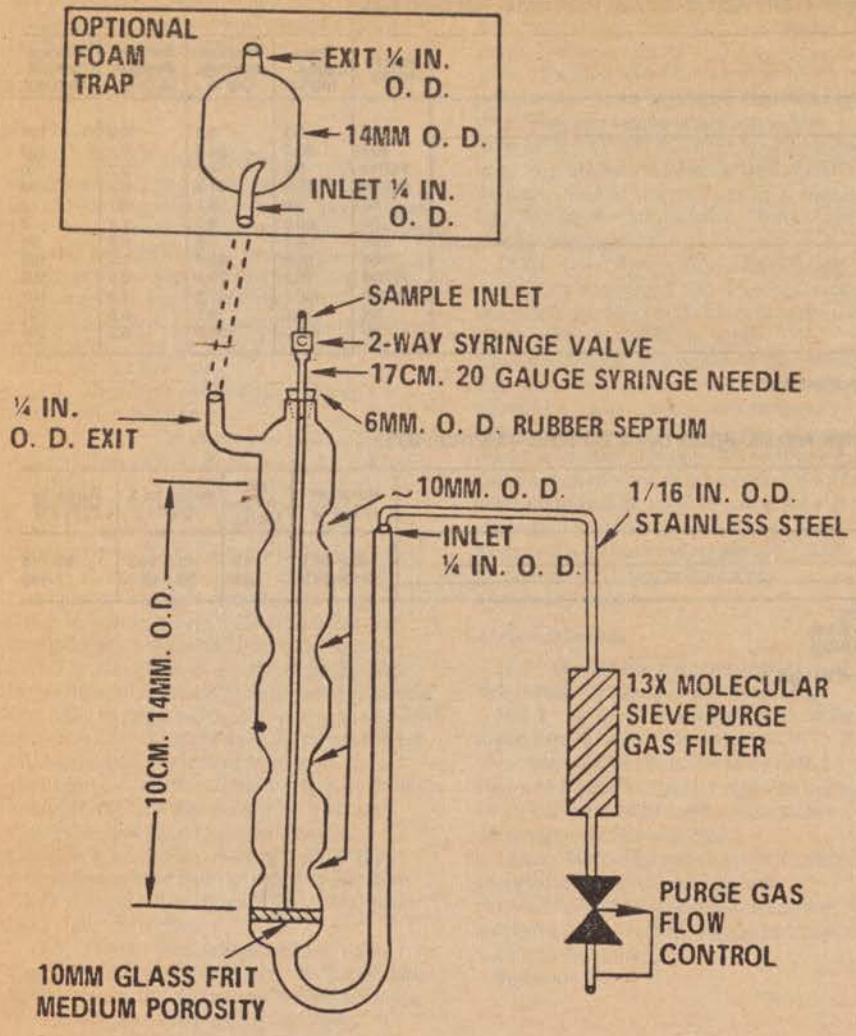



**Figure 1. Purging device.**

-243-

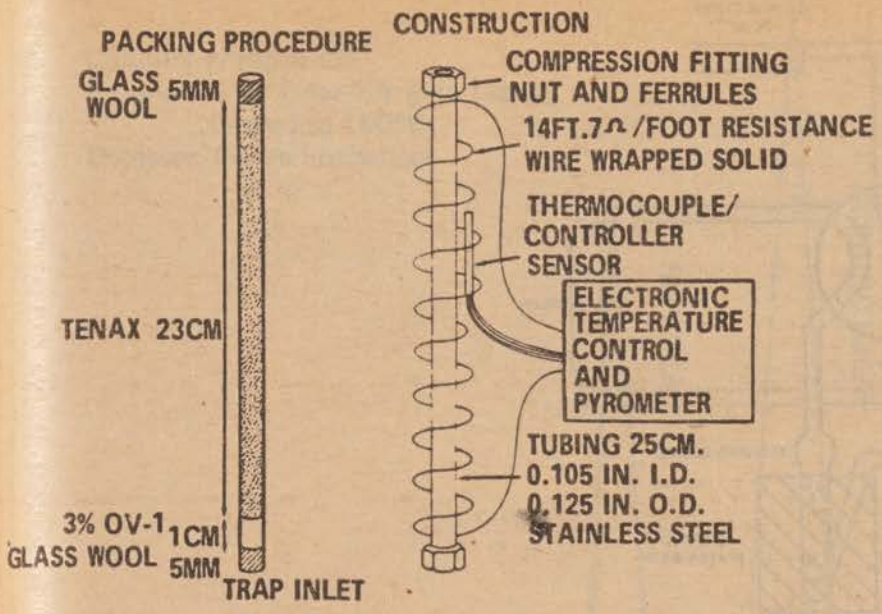



**Figure 2. Trap packings and construction to include desorb capability.**

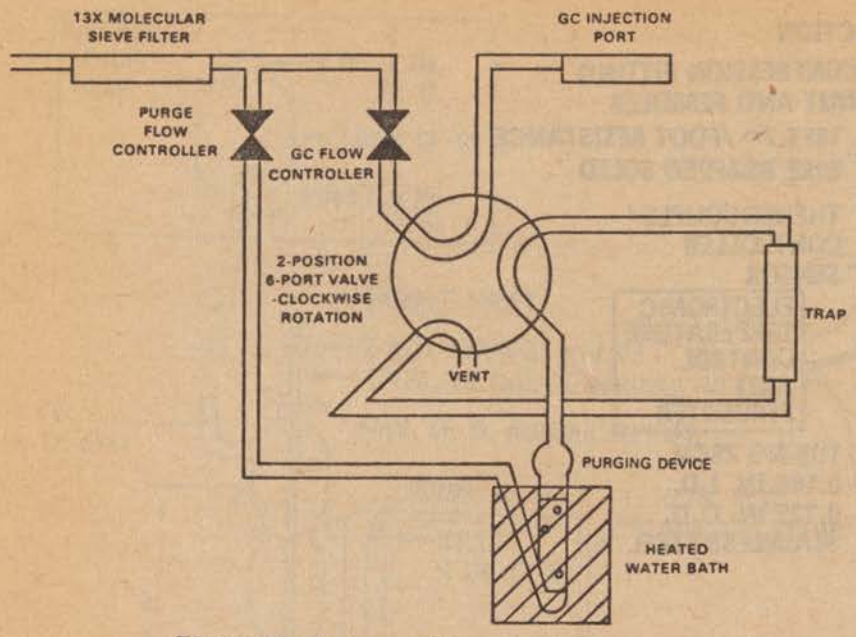



**Figure 3. Purge and trap system-purge mode.**

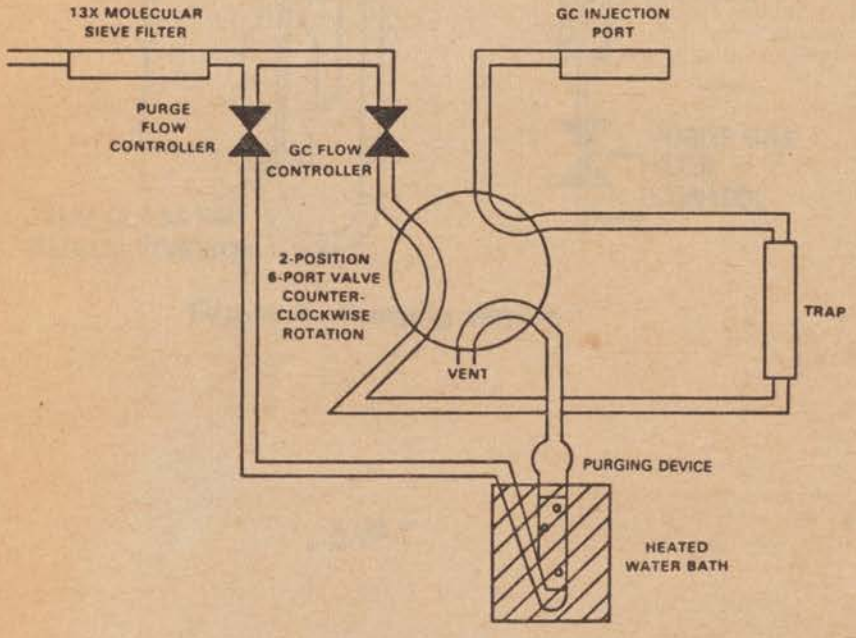



**Figure 4. Purge and trap system-desorb mode.**

-245-

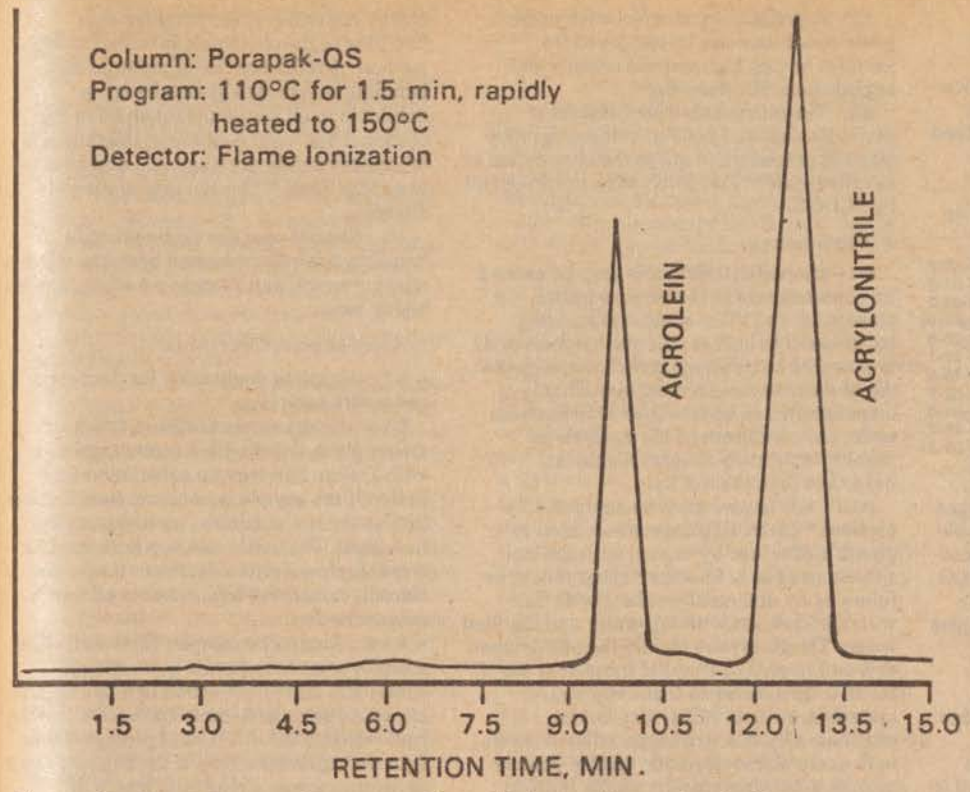



**Figure 5. Gas chromatogram of acrolein and acrylonitrile.**

BILLING CODE 6560-50-C

**Method 604—Phenols**

*1. Scope and Application*

1.1 This method covers the determination of phenol and certain substituted phenols. The following parameters may be determined by this method:

| Parameter | STORET No. | CAS No. |
|---|---|---|
| 4-Chloro-3-methylphenol | 34452 | 59-50-7 |
| 2-Chlorophenol | 34586 | 95-57-8 |
| 2,4-Dichlorophenol | 34601 | 120-83-2 |
| 2,4-Dimethylphenol | 34606 | 105-67-9 |
| 2,4-Dinitrophenol | 34616 | 51-28-5 |
| 2-Methyl-4,6-dinitrophenol | 34657 | 534-52-1 |
| 2-Nitrophenol | 34591 | 88-75-5 |
| 4-Nitrophenol | 34646 | 100-02-7 |
| Pentachlorophenol | 39032 | 87-86-5 |
| Phenol | 34694 | 108-95-2 |
| 2,4,6-Trichlorophenol | 34621 | 88-06-2 |

1.2 This is a flame ionization detector gas chromatographic (FIDGC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for derivatization, cleanup, and electron capture detector gas chromatography (ECDGC) that can be used to confirm measurements made by FIDGC. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix. The MDL listed in Table 1 for each parameter was achieved with a flame ionization detector (FID). Comparable results were achieved when the derivatization cleanup and electron capture detector (ECD) were employed (Table 2).

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 A measured volume of sample, approximately 1-L, is acidified and extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to 2-propanol during concentration to a volume of 10 mL or less. The extract is separated by gas chromatography and the phenols are then measured with an FID.[2]

2.2 A preliminary sample wash under basic conditions can be employed for samples having high general organic and organic base interferences.

2.3 The method also provides for a derivatization and column chromatography cleanup procedure to aid in the elimination of interferences.[2,3] The derivatives are analyzed by ECDGC.

*3. Interferences*

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[4] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The derivatization cleanup procedure in Section 12 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Tables 1 and 2.

3.3 The basic sample wash (Section 10.2) may cause significantly reduced recovery of phenol and 2,4-dimethylphenol. The analyst must recognize that results obtained under these conditions are minimum concentrations.

*4. Safety*

4.1 The toxicity or carcinogenicity of each reagent used in this mothod has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [5-7] for the information of analyst.

4.2 Special care should be taken in handling pentafluorobenzyl bromide, which is a lachrymator, and 18-crown-6-ether, which is highly toxic.

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1-L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2-L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, 400 mm long x 19 mm ID, with coarse frit filter disc.

5.2.3 Chromatographic column—100 mm long x 10 mm ID, with Teflon stopcock.

5.2.4 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.5 Evaporative flask, Kuderna-Danish—500-mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

5.2.6 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K-503000-0121 or equivalent).

5.2.7 Snyder column, Kuderna-Danish—Two-ball micro (Kontes K-569001-0219 or equivalent).

5.2.8 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.2.9 Reaction flask—15 to 25-mL round bottom flask, with standard tapered joint, fitted with a water-cooled condenser and U-shaped drying tube containing granular calcium chloride.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2°C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighting 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column for underivatized phenols—1.8 m long x 2 mm ID glass, packed with 1% SP-1240DA on Supelcoport (80/100 mesh) or equivalent. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 11.1.

5.6.2 Column for derivatized phenols—1.8 m long x 2 mm ID glass, packed with 5% OV-17 on Chromosorb W-AW-DMCS (80/100 mesh) or equivalent. This column has proven effective in the analysis of wastewaters for derivatization products of the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 11.1.

5.6.3 Detectors—Flame ionization and electron capture detectors. The FID issued when determining the parent phenols. The ECD is used when determining the derivatized phenols. Guidelines for the use of alternatve detectors are provided in Section 11.1.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.3 Sodium hydroxide solution (1 N)—Dissolve 4 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.4 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400°C for 4 h in a shallow tray.

6.5 Sodium thiosulfate—(ACS) Granular.

6.6 Sulfuric acid (1+1)—Slowly, add 50 mL of $H_2SO_4$ (ACS, sp. gr. 1.84) to 50 mL of reagent water.

6.7 Sulfuric acid (1 N)—Slowly, add 58 mL of $H_2SO_4$ (ACS, sp. gr. 1.84) to reagent water and dilute to 1 L.

6.8 Potassium carbonate—(ACS) Powdered.

6.9 Pentafluorobenzyl bromide (α-Bromopentafluorotoluene)—97% minimum purity. **Note:** This chemical is a lachrymator. (See Section 4.2.)

6.10 18-crown-6-ether (1,4,7,10,13,16-Hexaoxacyclooctadecane)—98% minimum purity. **Note:** This chemical is highly toxic.

6.11 Derivatization reagent—Add 1 mL of pentafluorobenzyl bromide and 1 g of 18-crown-6-ether to a 50-mL volumetric flask and dilute to volume with 2-propanol. Prepare fresh weekly. This operation should be carried out in a hood. Store at 4 °C and protect from light.

6.12 Acetone, hexane, methanol, methylene chloride, 2-propanol, toluene—Pesticide quality or equivalent.

6.13 Silica gel—100/200 mesh, Davison, grade-923 or equivalent. Activate at 130 °C overnight and store in a desiccator.

6.14 Stock standard solutions (1.00 μg/μL)—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions.

6.14.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in 2-propanol and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.14.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.14.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.15 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 To calibrate the FIDGC for the anaylsis of underivatized phenols, establish gas chromatographic operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure for FIDGC:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with 2-propanol. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 μl, analyze each calibration standard according to Section 11 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure for FIDGC—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with 2-propanol. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 μL, analyze each calibration standard according to Section 11 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard (μg/L).

$C_s$ = Concentration of the parameter to be measured (μg/L).

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, a new calibration curve must be prepared for that compound.

7.5 To calibrate the ECDGC for the analysis of phenol derivatives, establish gas chromatographic operating conditions equivalent to those given in Table 2.

7.5.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with 2-propanol. One of the external standards should be at a concentration near, but above, the MDL (Table 2) and the other concentrations should correspond to the expected range of concentrations found in

real samples or should define the working range of the detector.

7.5.2 Each time samples are to be derivatized, simultaneously treat a 1-mL aliquot of each calibration standard as described in Section 12.

7.5.3 After derivatization, analyze 2 to 5 μL of each column eluate collected according to the method beginning in Section 12.8 and tabulate peak height or area responses against the calculated equivalent mass of underivatized phenol injected. The results can be used to prepare a calibration curve for each compound.

7.6 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.6 and 11.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 100 μg/mL in 2-propanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at a concentration of 100 μg/L by adding 1.00 mL of QC check sample concentrate to each of four 1-L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 3. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter.

**Note.**—The large number of parameters in Talbe 3 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.2.6.2 Beginning with Section 8.2.2, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 100 μg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any, or, if none, (2) the larger of either 5 times higher than the expected background concentration or 100 μg/L.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 3. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[8] If spiking was performed at a concentration lower than 100 μg/L, the analyst must use either the QC acceptance criteria in Table 3, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X') using the equation in Table 4, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 4, substituting X' for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X'/T)±2.44(100 S'/T)%.[8]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

**Note.**—The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample

concentrate (Sections 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 3. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6. It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

*9. Sample Collection, Preservation, and Handling*

9.1 Grab samples must be collected in glass containers. Conventional sampling practices [9] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C from the time of collection until extraction. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[10] Field test kits are available for this purpose.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

*10. Sample Extraction*

10.1 Mark the water meniscus on the side of sample bottle for later determination of sample volume. Pour the entire sample into a 2-L separatory funnel.

10.2 For samples high in organic content, the analyst may solvent wash the sample at basic pH as prescribed in Sections 10.2.1 and 10.2.2 to remove potential method interferences. Prolonged or exhaustive contact with solvent during the wash may result in low recovery of some of the phenols, notably phenol and 2,4-dimethylphenol. For relatively clean samples, the wash should be omitted and the extraction, beginning with Section 10.3, should be followed.

10.2.1 Adjust the pH of the sample to 12.0 or greater with sodium hydroxide solution.

10.2.2 Add 60 mL of methylene chloride to the sample by shaking the funnel for 1 min with periodic venting to release vapor pressure. Discard the solvent layer. The wash can be repeated up to two additional times if significant color is being removed.

10.3 Adjust the sample to a pH of 1 to 2 with sulfuric acid.

10.4 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.5 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.6 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.7 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.8 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.9 Increase the temperature of the hot water bath to 95 to 100 °C. Remove the Synder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of 2-propanol. A 5-mL syringe is recommended for this operation. Attach a two-ball micro-Snyder column to the concentrator tube and prewet the column by adding about 0.5 mL of 2-propanol to the top. Place the micro-K–D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches 2.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min. Add an additional 2 mL of 2-propanol through the top of the micro-Snyder column and resume concentrating as before. When the apparent volume of liquid reaches 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.10 Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a minimum amount of 2-propanol. Adjust the extract volume to 1.0 mL. Stopper the concentrator tube and store refrigerated at 4 °C if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with FIDGC analysis (Section 11). If the sample requires further cleanup, proceed to Section 12.

10.11 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

*11. Flame Ionization Detector Gas Chromatography*

11.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. An example of the separations achieved by this column is shown in Figure 1. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

11.2 Calibrate the system daily as described in Section 7.

11.3 If the internal standard calibration procedure is used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the gas chromatograph.

11.4 Inject 2 to 5 μL of the sample extract or standard into the gas chromatograph using the solvent-flush technique.[11] Smaller (1.0 μL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 μL, and the resulting peak size in area or peak height units.

11.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound may be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

11.6 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

11.7 If the measurement of the peak response is prevented by the presence of interferences, an alternative gas chromatographic procedure is required. Section 12 describes a derivatization and column chromatographic procedure which has been tested and found to be a practical means of analyzing phenols in complex extracts.

*12. Derivatization and Electron Capture Detector Gas Chromatography*

12.1 Pipet a 1.0-mL aliquot of the 2-propanol solution of standard or sample extract into a glass reaction vial. Add 1.0 mL of derivatizing reagent (Section 6.11). This amount of reagent is sufficient to derivatize a solution whose total phenolic content does not exceed 0.3 mg/mL.

12.2 Add about 3 mg of potassium carbonate to the solution and shake gently.

12.3 Cap the mixture and heat it for 4 h at 80 °C in a hot water bath.

12.4 Remove the solution from the hot water bath and allow it to cool.

12.5 Add 10 mL of hexane to the reaction flask and shake vigorously for 1 min. Add 3.0 mL of distilled, deionized water to the reaction flask and shake for 2 min. Decant a portion of the organic layer into a concentrator tube and cap with a glass stopper.

12.6 Place 4.0 g of silica gel into a chromatographic column. Tap the column to settle the silica gel and add about 2 g of anhydrous sodium sulfate to the top.

12.7 Preelute the column with 6 mL of hexane. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, pipet onto the column 2.0 mL of the hexane solution (Section 12.5) that contains the derivatized sample or standard. Elute the column with 10.0 mL of hexane and discard the eluate. Elute the column, in order, with: 10.0 mL of 15% toluene in hexane (Fraction 1); 10.0 mL of 40% toluene in hexane (Fraction 2); 10.0 mL of 75% toluene in hexane (Fraction 3); and 10.0 mL of 15% 2-propanol in toluene (Fraction 4). All elution mixtures are prepared on a volume:Volume basis. Elution patterns for the phenolic derivatives are shown in Table 2. Fractions may be combined as desired, depending upon the specific phenols of interest or level of interferences.

12.8 Analyze the fractions by ECDGC. Table 2 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. An example of the separations achieved by this column is shown in Figure 2.

12.9 Calibrate the system daily with a minimum of three aliquots of calibration standards, containing each of the phenols of interest that are derivatized according to Section 7.5.

12.10 Inject 2 to 5 μL of the column fractions into the gas chromatograph using the solvent-flush technique. Smaller (1.0 μL) volumes can be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 μL, and the resulting peak size in area or peak height units. If the peak response exceeds the linear range of the system, dilute the extract and reanalyze.

*13. Calculations*

13.1 Determine the concentration of individual compounds in the sample analyzed by FIDGC (without derivatization) as indicated below.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

where:

A = Amount of material injected (ng).
$V_i$ = Volume of extract injected (μL).
$V_t$ = Volume of total extract (μL).
$V_s$ = Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

$A_s$ = Response for the parameter to be measured.
$A_{is}$ = Response for the internal standard.
$I_s$ = Amount of internal standard added to each extract (μg).
$V_o$ = Volume of water extracted (L).

13.2 Determine the concentration of individual compounds in the sample analyzed by derivatization and ECDGC according to Equation 4.

Equation 4.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)(B)(D)}{(V_i)(V_s)(C)(E)}$$

where:

A = Mass of underivatized phenol represented by area of peak in sample chromatogram, determined from calibration curve in Section 7.5.3 (ng).
$V_i$ = Volume of eluate injected (μL).
$V_t$ = Total volume of column eluate or combined fractions from which $V_i$ was taken (μL).
$V_s$ = Volume of water extracted in Section 10.10 (mL).
B = Total volume of hexane added in Section 12.5 (mL).
C = Volume of hexane sample solution added to cleanup column in Section 12.7 (mL).
D = Total volume of 2-propanol extract prior to derivatization (mL).
E = Volume of 2-propanol extract carried through derivatization in Section 12.1 (mL).

13.3 Report results in μg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

*14. Method Performance*

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Tables 1 and 2 were obtained using reagent water.[12] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked as six concentrations over the range 12 to 450 μg/L.[13] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these xc relationships for a flame ionization detector are presented in Table 4.

*References*

1. 40 CFR Part 136, Appendix B.

2. "Determination of Phenols in Industrial and Municipal Wastewaters," Report for EPA Contract 68-03-2625 (In preparation).

3. Kawahara, F. K. "Microdetermination of Derivatives of Phenols and Mercaptans by Means of Electron Capture Gas Chromatography," *Analytical Chemistry, 40,* 1009 (1968).

4. ASTM Annual Book of Standards, Part 31, D3694-78. "Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents," American Society for Testing and Materials, Philadelphia.

5. "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health

Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, August 1977.

6. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

7. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

8. Provost, L. P., and Elder, R. S. "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58-63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

9. ASTM Annual Book of Standards, Part 31, D3370-76. "Standard Practices for Sampling Water," American Society for Testing and Materials, Philadelphia.

10. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

11. Burke, J. A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

12. "Development of Detection Limits, EPA Method 604, Phenols," Special letter report for EPA Contract 68-03-2625, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

13. "EPA Method Validation Study 14, Method 604 (Phenols)," Report for EPA Contract 68-03-2625 (In preparation).

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

| Parameter | Retention time (min) | Method detection limit (μg/L) |
|---|---|---|
| 2-Chlorophenol | 1.70 | 0.31 |
| 2-Nitrophenol | 2.00 | 0.45 |
| Phenol | 3.01 | 0.14 |
| 2,4-Dimethylphenol | 4.03 | 0.32 |
| 2,4-Dichlorophenol | 4.30 | 0.39 |
| 2,4,6-Trichlorophenol | 6.05 | 0.64 |
| 4-Chloro-3-methylphenol | 7.50 | 0.36 |
| 2,4-Dinitrophenol | 10.00 | 13.0 |
| 2-Methyl-4,6-dinitrophenol | 10.24 | 16.0 |
| Pentachlorophenol | 12.42 | 7.4 |
| 4-Nitrophenol | 24.25 | 2.8 |

Column conditions: Supelcoport (80/100 mesh) coated with 1% SP-1240DA packed in a 1.8 m long x 2 mm ID glass column with nitrogen carrier gas at 30 mL/min flow rate. Column temperature was 80°C at injection, programmed immediately at 8°C/min to 150°C final temperature. MDL were determined with an FID.

TABLE 2.— SILICA GEL FRACTIONATION AND ELECTRON CAPTURE GAS CHROMATOGRAPHY OF PFBB DERIVATIVES

| Parent compound | Percent recovery by fraction * | | | | Retention time (min) | Method detection limit (μg/L) |
|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | | |
| 2-Chlorophenol | | 90 | 1 | | 3.3 | 0.58 |
| 2-Nitrophenol | | | 9 | 90 | 9.1 | 0.77 |
| Phenol | | 90 | 10 | | 1.8 | 2.2 |
| 2,4-Dimethylphenol | | 95 | 7 | | 2.9 | 0.63 |
| 2,4-Dichlorophenol | | 95 | 1 | | 5.8 | 0.68 |
| 2,4,6-Trichlorophenol | 50 | 50 | | | 7.0 | 0.58 |
| 4-Chloro-3-methylphenol | | 84 | 14 | | 4.8 | 1.8 |
| Pentachlorophenol | 75 | 20 | | | 28.8 | 0.59 |
| 4-Nitrophenol | | | 1 | 90 | 14.0 | 0.70 |

Column conditions: Chromosorb W-AW-DMCS (80/100 mesh) coated with 5% OV-17 packed in a 1.8 m long x 2.0 mm ID glass column with 5% methane/95% argon carrier gas at 30 mL/min flow rate. Column temperature held isothermal at 200 °C. MDL were determined with an ECD.

* Eluant composition:
Fraction 1—15% toluene in hexane.
Fraction 2—40% toluene in hexane.
Fraction 3—75% toluene in hexane.
Fraction 4—15% 2-propanol in toluene.

TABLE 3.—QC ACCEPTANCE CRITERIA—METHOD 604

| Parameter | Test conc. (μg/L) | Limit for s (μg/L) | Range for $\bar{X}$ (μg/L) | Range for P, $P_s$ (percent) |
|---|---|---|---|---|
| 4-Chloro-3-methylphenol | 100 | 16.6 | 56.7-113.4 | 99-122 |
| 2-Chlorophenol | 100 | 27.0 | 54.1-110.2 | 38-126 |
| 2,4-Dichlorophenol | 100 | 25.1 | 59.7-103.3 | 44-119 |
| 2,4-Dimethylphenol | 100 | 33.3 | 50.4-100.0 | 24-118 |
| 4,6-Dinitro-2-methylphenol | 100 | 25.0 | 42.4-123.6 | 30-136 |
| 2,4-Dinitrophenol | 100 | 36.0 | 31.7-125.1 | 12-145 |
| 2-Nitrophenol | 100 | 22.5 | 56.6-103.8 | 43-117 |
| 4-Nitrophenol | 100 | 19.0 | 22.7-100.0 | 13-110 |
| Pentachlorophenol | 100 | 32.4 | 56.7-113.5 | 36-134 |
| Phenol | 100 | 14.1 | 32.4-100.0 | 23-108 |
| 2,4,6-Trichlorophenol | 100 | 16.6 | 60.8-110.4 | 53-119 |

s—Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).
$\bar{X}$—Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
P, $P_s$—Percent recovery measured (Section 8.3.2, Section 8.4.2).

NOTE.—These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

TABLE 4.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 604

| Parameter | Accuracy, as recovery, X' (μg/L) | Single Analyst precision, $s_r'$ (μg/L) | Overall precision, S' (μg/L) |
|---|---|---|---|
| 4-Chloro-3-methylphenol | 0.87C–1.97 | 0.11$\bar{X}$–0.21 | 0.16$\bar{X}$+1.41 |
| 2-Chlorophenol | 0.83C–0.84 | 0.18$\bar{X}$+0.20 | 0.21$\bar{X}$+0.75 |
| 2,4-Dichlorophenol | 0.81C+0.48 | 0.17$\bar{X}$–0.02 | 0.18$\bar{X}$+0.62 |
| 2,4-Dimethylphenol | 0.62C–1.64 | 0.30$\bar{X}$–0.89 | 0.25$\bar{X}$+0.48 |
| 4,6-Dinitro-2-methylphenol | 0.84C–1.01 | 0.15$\bar{X}$+1.25 | 0.19$\bar{X}$+5.85 |
| 2,4-Dinitrophenol | 0.80C–1.58 | 0.27$\bar{X}$–1.15 | 0.29$\bar{X}$+4.51 |
| 2-Nitrophenol | 0.81C–0.76 | 0.15$\bar{X}$+0.44 | 0.14$\bar{X}$+3.84 |
| 4-Nitrophenol | 0.46C+0.18 | 0.17$\bar{X}$+2.43 | 0.19$\bar{X}$+4.79 |
| Pentachlorophenol | 0.83C+2.07 | 0.22$\bar{X}$–0.58 | 0.23$\bar{X}$+0.57 |
| Phenol | 0.43C+0.11 | 0.20$\bar{X}$–0.88 | 0.17$\bar{X}$+0.77 |
| 2,4,6-Trichlorophenol | 0.86C–0.40 | 0.10$\bar{X}$+0.53 | 0.13$\bar{X}$+2.40 |

X' = Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
$s_r'$ = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C = True value for the concentration, in μg/L.
$\bar{X}$ = Average rscovery found for measurements of samples containing a concentration of C, in μg/L.

BILLING CODE 6560–50–M

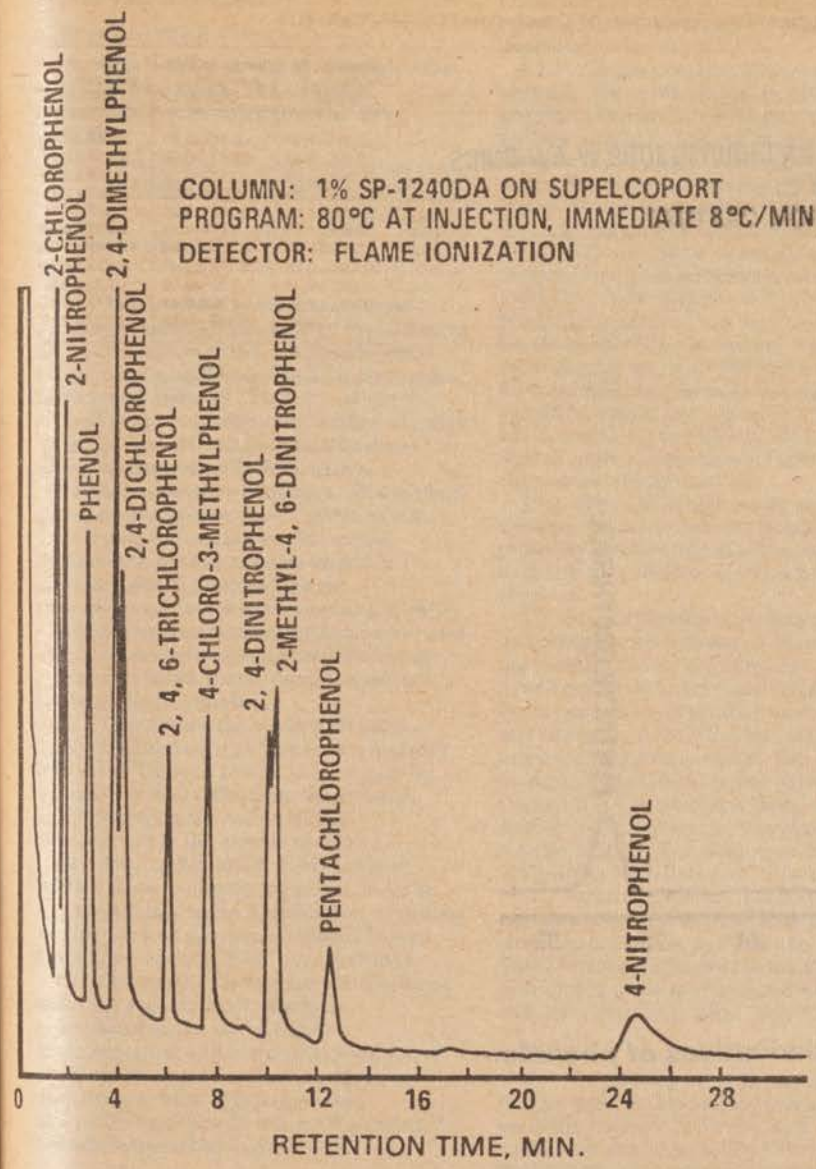



Figure 1. Gas chromatogram of phenols.

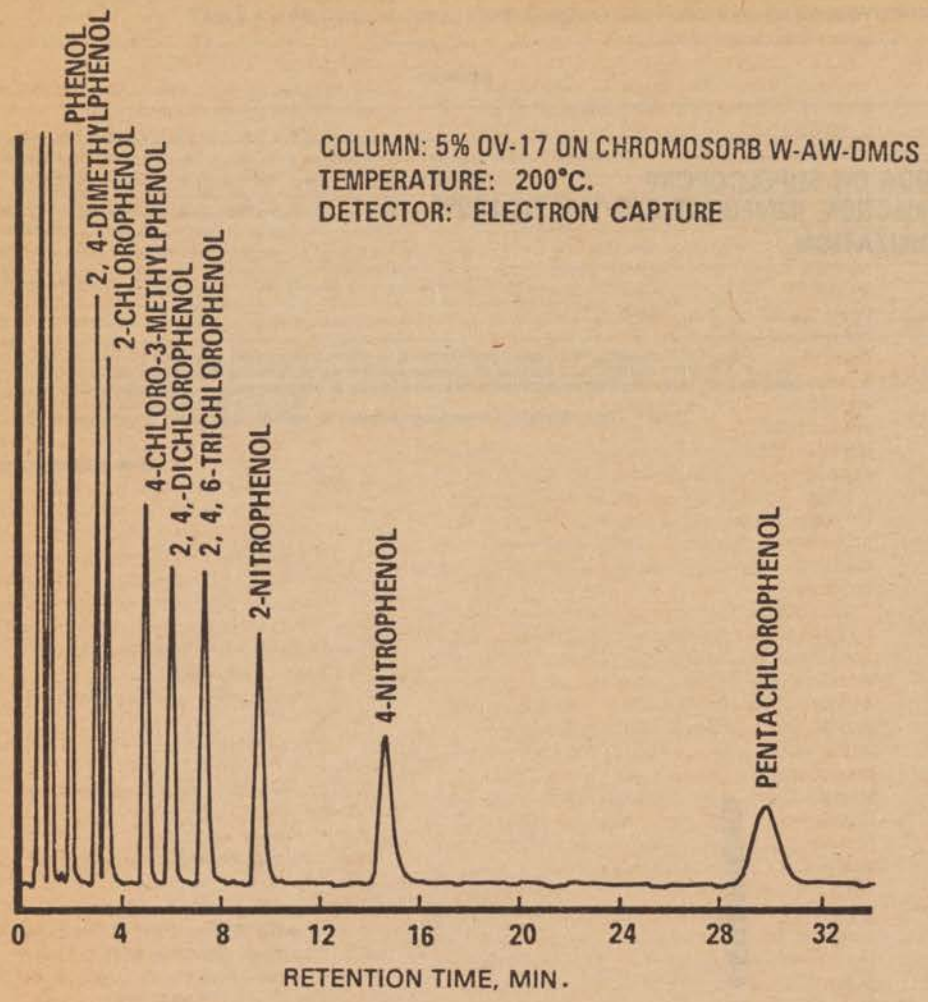



**Figure 2. Gas chromatogram of PFB derivatives of phenols.**

BILLING CODE 6560-50-C

## Method 605—Benzidines

*1. Scope and Application*

1.1 This method covers the determination of certain benzidines. The following parameters can be determined by this method:

| Parameter | Storet No. | CAS No. |
|---|---|---|
| Benzidine | 39120 | 92-87-5 |
| 3,3'-Dichlorobenzidine | 34631 | 91-94-1 |

1.2 This is a high performance liquid chromatography (HPLC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for the compounds above, identifications should be supported by at least one additional qualitative technique. This method describes electrochemical conditions at a second potential which can be used to confirm measurements made with this method. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1) [1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of the interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC instrumentation and in the interpretation of liquid chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 A measured volume of sample, approximately 1-L, is extracted with chloroform using liquid-liquid extractions in a separatory funnel. The chloroform extract is extracted with acid. The acid extract is then neutralized and extracted with chloroform. The final chloroform extract is exchanged to methanol while being concentrated using a rotary evaporator. The extract is mixed with buffer and separated by HPLC. The benzidine compounds are measured with an electrochemical detector.[2]

2.2 The acid back-extraction acts as a general purpose cleanup to aid in the elimination of interferences.

*3. Interferences*

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedures that are inherent in the extraction step are used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

3.3 Some dye plant effluents contain large amounts of components with retention times closed to benzidine. In these cases, it has been found useful to reduce the electrode potential in order to eliminate interferences and still detect benzidine. (See Section 12.7.)

*4. Safety*

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health harzard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [4-6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzidine and 3,3'-dichlorobenzidine. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

4.3 Exposure to chloroform should be minimized by performing all extractions and extract concentrations in a hood or other well-ventiliated area.

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1-L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4°C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested):

5.2.1 Separatory funnels—2000, 1000, and 250-mL, with Teflon stopcock.

5.2.2 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.2.3 Rotary evaporator.

5.2.4 Flasks—Round bottom, 100-mL, with 24/40 joints.

5.2.5 Centrifuge tubes—Conical, graduated, with Teflon-lined screw caps.

5.2.6 Pipettes—Pasteur, with bulbs.

5.3 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.4 High performance liquid chromatograph (HPLC)—An analytical system complete with column supplies, high pressure syringes, detector, and compatible recorder. A data system is recommended for measuring peak areas and retention times.

5.4.1 Solvent delivery system—With pulse damper, Altex 110A or equivalent.

5.4.2 Injection valve (optional)—Waters U6K or equivalent.

5.4.3 Electrochemical detector—Bioanalytical Systems LC-2A with glassy carbon electrode, or equivalent. This detector has proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 14. Guidelines for the use of alternate detectors are provided in Section 12.1.

5.4.4 Electrode polishing kit—Princeton Applied Research Model 9320 or equivalent.

5.4.5 Column—Lichrosorb RP-2, 5 micron particle diameter, in a 25 cm × 4.6 mm ID stainless steel column. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 12.1.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is

not observed at the MDL of the parameters of interest.

6.2 Sodium hydroxide solution (5 N)—Dissolve 20 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.3 Sodium hydroxide solution (1 M)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 1 L.

6.4 Sodium thiosulfate—(ACS) Granular.

6.5 Sodium tribasic phosphate (0.4 M)—Dissolve 160 g of trisodium phosphate decahydrate (ACS) in reagent water and dilute to 1 L.

6.6 Sulfuric acid (1+1)—Slowly, add 50 mL of $H_2SO_4$ (ACS, sp. gr. 1.84) to 50 mL of reagent water.

6.7 Sulfuric acid (1 M)—Slowly, add 58 mL of $H_2SO_4$ (ACS, sp. gr. 1.84) to reagent water and dilute to 1 L.

6.8 Acetate buffer (0.1 M, pH 4.7)—Dissolve 5.8 mL of glacial acetic acid (ACS) and 13.6 g of sodium acetate trihydrate (ACS) in reagent water which has been purified by filtration through a RO–4 Millipore System or equivalent and dilute to 1 L.

6.9 Acetonitrile, chloroform (preserved with 1% ethanol), methanol—Pesticide quality or equivalent.

6.10 Mobile phase—Place equal volumes of filtered acetonitrile (Millipore type FH filter or equivalent) and filtered acetate buffer (Millipore type GS filter or equivalent) in a narrow-mouth, glass container and mix thoroughly. Prepare fresh weekly. Degas daily by sonicating under vacuum, by heating an stirring, or by purging with helium.

6.11 Stock standard solutions (1.00 μg/μL)—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions.

6.11.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in methanol and dilute to volume in a 10–mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.11.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.11.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.12 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Establish chromatographic operating conditions equivalent to those given in Table 1. The HPLC system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with mobile phase. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using syringe injections of 5 to 25 μL or a constant volume injection loop, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with mobile phase. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using syringe injections of 5 to 25 μL or a constant volume injection loop, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard (μg/L).

$C_s$ = Concentration of the parameter to be measured (μg/L).

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, a new calibration curve must be prepared for that compound. If serious loss of response occurs, polish the electrode and recalibrate.

7.5 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.9, 11.1, and 12.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality

of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing benzidine and/or 3,3'-dichlorobenzidine at a concentration of 50 μg/mL each in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at a concentration of 50 μg/L by adding 1.00 mL of QC check sample concentrate to each of four 1-L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter. Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 50 μg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or 50 μg/L.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than 50 μg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) calculate accuracy (X') using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 3, substituting X' for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X'/T) ± 2.44(100 S'/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

**Note.**—The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent recovery interval from $\bar{P} - 2s_p$ to $\bar{P} + 2s_p$. If $\bar{P} = 90\%$ and $s_p = 10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as HPLC with a dissimilar column, gas chromatography, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

*9. Sample Collection, Preservation, and Handling*

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[8] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4°C and stored in the dark from the time of collection until extraction. Both benzidine and 3,3'-dichlorobenzidine are easily oxidized. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[9] Field test kits are available for this purpose. After mixing, adjust the pH of the sample to a range of 2 to 7 with sulfuric acid.

9.3 If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

9.4 All samples must be extracted within 7 days of collection. Extracts may be held up to 7 days before analysis, if stored under an inert (oxidant free) atmosphere.[2] The extract should be protected from light.

*10. Sample Extraction*

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2-L separatory funnel. Check the pH of the sample with wide-range pH paper and adjust

to within the range of 6.5 to 7.5 with sodium hydroxide solution or sulfuric acid.

10.2 Add 100 mL of chloroform to the sample bottle, seal, and shake 30 s to rinse the inner surface. (Caution: Handle chloroform in a well ventilated area.) Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the chloroform extract in a 250-mL separatory funnel.

10.3 Add a 50-mL volume of chloroform to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the separatory funnel. Perform a third extraction in the same manner.

10.4 Separate and discard any aqueous layer remaining in the 250-mL separatory funnel after combining the organic extracts. Add 25 mL of 1 M sulfuric acid and extract the sample by shaking the funnel for 2 min. Transfer the aqueous layer to a 250-mL beaker. Extract with two additional 25-mL portions of 1 M sulfuric acid and combine the acid extracts in the beaker.

10.5 Place a stirbar in the 250-mL beaker and stir the acid extract while carefully adding 5 mL of 0.4 M sodium tribasic phosphate. While monitoring with a pH meter, neutralize the extract to a pH between 6 and 7 by dropwise addition of 5 N sodium hydroxide solution while stirring the solution vigorously. Approximately 25 to 30 mL of 5 N sodium hydroxide solution will be required and it should be added over at least a 2-min period. Do not allow the sample pH to exceed 8.

10.6 Transfer the neutralized extract into a 250-mL separatory funnel. Add 30 mL of chloroform and shake the funnel for 2 min. Allow the phases to separate, and transfer the organic layer to a second 250-mL separatory funnel.

10.7 Extract the aqueous layer with two additional 20-mL aliquots of chloroform as before. Combine the extracts in the 250-mL separatory funnel.

10.8 Add 20 mL of reagent water to the combined organic layers and shake for 30 s.

10.9 Transfer the organic extract into a 100-mL round bottom flask. Add 20 mL of methanol and concentrate to 5 mL with a rotary evaporator at reduced pressure and 35 °C. An aspirator is recommended for use as the source of vacuum. Chill the receiver with ice. This operation requires approximately 10 min. Other concentration techniques may be used if the requirements of Section 8.2 are met.

10.10 Using a 9-in. Pasteur pipette, transfer the extract to a 15-mL, conical, screw-cap centrifuge tube. Rinse the flask, including the entire side wall, with 2-mL portions of methanol and combine with the original extract.

10.11 Carefully concentrate the extract to 0.5 mL using a gentle stream of nitrogen while heating in a 30 °C water bath. Dilute to 2 mL with methanol, reconcentrate to 1 mL, and dilute to 5 mL with acetate buffer. Mix the extract thoroughly. Cap the centrifuge tube and store refrigerated and protected from light if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with HPLC analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.12 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1,000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

*11. Cleanup and Separation*

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure.

*12. High Performance Liquid Chromatography*

12.1 Table 1 summarizes the recommended operating conditions for the HPLC. Included in this table are retention times, capacity factors, and MDL that can be achieved under these conditions. An example of the separations achieved by this HPLC column is shown in Figure 1. Other HPLC columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met. When the HPLC is idle, it is advisable to maintain a 0.1 mL/min flow through the column to prolong column life.

12.2 Calibrate the system daily as described in Section 7.

12.3 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the instrument.

12.4 Inject 5 to 25 μL of the sample extract or standard into the HPLC. If constant volume injection loops are not used, record the volume injected to the nearest 0.05 μL, and the resulting peak size in area or peak height units.

12.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.6 If the response for a peak exceeds the working range of the system, dilute the extract with mobile phase and reanalyze.

12.7 If the measurement of the peak response for benzidine is prevented by the presence of interferences, reduce the electrode potential to +0.6 V and reanalyze. If the benzidine peak is still obscured by interferences, further cleanup is required.

*13. Calculations*

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

where:

$A$ = Amount of material injected (ng).
$V_i$ = Volume of extract injected (μL).
$V_t$ = Volume of total extract (μL).
$V_s$ = Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

$A_s$ = Response for the parameter to be measured.
$A_{is}$ = Response for the internal standard.
$I_s$ = Amount of internal standard added to each extract (μg).
$V_o$ = Volume of water extracted (L).

13.2 Report results in μg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

*14. Method Performance*

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[10] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 7×MDL to 3000×MDL.[10]

14.3 This method was tested by 17 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 1.0 to 70 μg/L.[11] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample

matrix. Linear equations to describe these relationships are presented in Table 3.

*References*

1. 40 CFR Part 136, Appendix B.

2. "Determination of Benzidines in Industrial and Municipal Wastewaters," EPA–600/4–82–022, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, May 1982.

3. ASTM Annual Book of Standards, Part 31, D3694–78. "Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents," American Society for Testing and Materials, Philadelphia.

4. "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

8. ASTM Annual Book of Standards, Part 31, D3370–76. "Standard Practices for Sampling Water," American Society for Testing and Materials, Philadelphia.

9. "Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

10. "Determination of Method Detection Limit and Analytical Curve for EPA Method 605—Benzidines," Speical letter report for EPA Contract 68–03–2624, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

11. "EPA Method Validation Study 15, Method 605 (Benzidines)," Report for EPA Contract 68–03–2624 (In preparation).

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

| Parameter | Retention time (min) | Column capacity factor (k') | Method detection limit (μg/L) |
|---|---|---|---|
| Benzidine | 6.1 | 1.44 | 0.08 |
| 3,3'-Dichlorobenzidine | 12.1 | 3.84 | 0.13 |

HPLC Column conditions: Lichrosorb RP–2, 5 micron particle size, in a 25 cm × 4.6 mm ID stainless steel column. Mobile Phase: 0.8 mL/min of 50% acetonitrile/50% 0.1M pH 4.7 acetate buffer. The MDL were determined using an electrochemical detector operated at +0.8 V.

TABLE 2.—QC ACCEPTANCE CRITERIA—METHOD 605

| Parameter | Test conc. (μg/L) | Limit for s (μg/L) | Range for $\bar{X}$ (μg/L) | Range for P, $P_s$ (percent) |
|---|---|---|---|---|
| Benzidine | 50 | 18.7 | 9.1–61.0 | D–140 |
| 3,3'-Dichlorobenzidine | 50 | 23.6 | 18.7–50.0 | 5–128 |

s = Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
P, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.

**Note.**—These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 605

| Parameter | Accuracy, as recovery, X'(μg/L) | Single analyst precision, $s_r'$ (μg/L) | Overall precision, S' (μg/L) |
|---|---|---|---|
| Benzidine | 0.70C + 0.06 | 0.28$\bar{X}$ + 0.19 | 0.40$\bar{X}$ + 0.18 |
| 3,3'-Dichlorobenzidine | 0.66C + 0.23 | 0.39$\bar{X}$ − 0.05 | 0.38$\bar{X}$ + 0.02 |

X' = Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
$s_r'$ = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C = True value for the concentration, in μg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in μg/L.

BILLING CODE 6560–50–M

COLUMN: LICHROSORB RP-2
MOBILE PHASE: 50% ACETONITRILE IN ACETATE BUFFER
DETECTOR: ELECTROCHEMICAL AT + 0.8 V

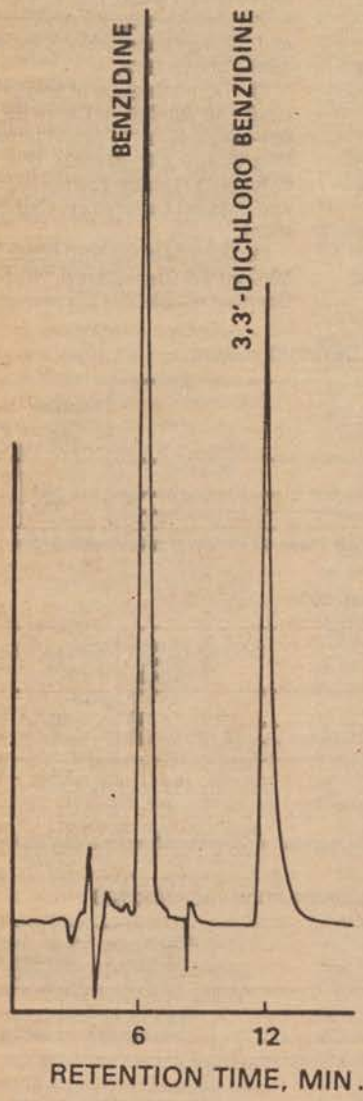



**Figure 1. Liquid chromatogram of benzidines.**

BILLING CODE 6560-50-C

## Method 606—Phthalate Esters

### 1. Scope and Application

1.1 This method covers the determination of certain phthalate esters. The following parameters can be determined by this method:

| Parameter | STORET No. | CAS No. |
|---|---|---|
| Bis(2-ethylhexyl) phthalate | 39100 | 117-81-7 |
| Butyl benzyl phthalate | 34292 | 85-68-7 |
| Di-n-butyl phthalate | 39110 | 84-74-2 |
| Diethyl phthalate | 34336 | 84-66-2 |
| Dimethyl phthalate | 34341 | 131-11-3 |
| Di-n-octyl phthalate | 34596 | 117-84-0 |

1.2 This is a gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 The sample extraction and concentration steps in this method are essentially the same as in Methods 608, 609, 611, and 612. Thus, a single sample may be extracted to measure the parameters included in the scope of each of these methods. When cleanup is required, the concentration levels must be high enough to permit selecting aliquots, as necessary, to apply appropriate cleanup procedures. The analyst is allowed the latitude, under Section 12, to select chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.5 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

### 2. Summary of Method

2.1 A measured volume of sample, approximately 1-L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to hexane during concentration to a volume of 10 mL or less. The extract is separated by gas chromatography and the phthalate esters are then measured with an electron capture detector.[2]

2.2 Analysis for phthalates is especially complicated by their ubiquitous occurrence in the environment. The method provides Florisil and alumina column cleanup procedures to aid in the elimination of interferences that may be encountered.

### 3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Phthalate esters are contaminants in many products commonly found in the laboratory. It is particularly important to avoid the use of plastics because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination can result at any time, if consistent quality control is not practiced. Great care must be experienced to prevent such contamination. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.[4,5]

3.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedures in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

### 4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [6-8] for the information of the analyst.

### 5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1-L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only).

5.2.1 Separatory funnel—2-L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long × 19 mm ID, with coarse frit filter disc.

5.2.3 Chromatographic column—300 mm long × 10 mm ID, with Teflon stopcock and coarse frit filter disc at bottom (Kontes K-420540-0213 or equivalent).

5.2.4 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.5 Evaporative flask, Kuderna-Danish—500-mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

5.2.6 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K-503000-0121 or equivalent).

5.2.7 Snyder column, Kuderna-Danish—Two-ball micro (Kontes K-569001-0219 or equivalent).

5.2.8 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2 °C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column 1—1.8 m long × 4 mm ID glass, packed with 1.5% SP–2250/1.95% SP–2401 Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 12.1.

5.6.2 Column 2—1.8 m long × 4 mm ID glass, packed with 3% OV–1 on Supelcoport (100/120 mesh) or equivalent.

5.6.3 Detector—Electron capture detector. This detector has proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 14. Guidelines for the use of alternate detectors are provided in Section 12.1.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Acetone, hexane, isooctane, methylene chloride, methanol—Pesticide quality or equivalent.

6.3 Ethyl ether—nanograde, redistilled in glass if necessary.

6.3.1 Ethyl ether must be shown to be free of peroxides before it is used as indicated by EM Laboratories Quant test strips. (Available from Scientific Products Co., Cat. No. P1126–8, and other suppliers.)

6.3.2 Procedures recommended for removal of peroxides are provided with the test strips. After cleanup, 20 mL of ethyl alcohol preservative must be added to each liter of ether.

6.4 Sodium sulfate—(ACS) Granular, anhydrous. Several levels of purification may be required in order to reduce background phthalate levels to an acceptable level: 1) Heat 4 h at 400 °C in a shallow tray, 2) Heat 16 h at 450 to 500 °C in a shallow tray, 3) Soxhlet extract with methylene chloride for 48 h.

6.5 Florisil—PR grade (60/100 mesh). Purchase activated at 1250 °F and store in the dark in glass containers with ground glass stoppers or foil-lined screw caps. To prepare for use, place 100 g of Florisil into a 500-mL beaker and heat for approximately 16 h at 40 °C. After heating transfer to a 500-mL reagent bottle. Tightly seal and cool to room temperature. When cool add 3 mL of reagent water. Mix thoroughly by shaking or rolling for 10 min and let it stand for at least 2 h. Keep the bottle sealed tightly.

6.6 Alumina—Neutral activity Super I, W200 series (ICN Life Sciences Group, No. 404583). To prepare for use, place 100 g of alumina into a 500-mL beaker and heat for approximately 16 h at 400 °C. After heating transfer to a 500-mL reagent bottle. Tightly seal and cool to room temperature. When cool add 3 mL of reagent water. Mix thoroughly by shaking or rolling for 10 min and let it stand for at least 2 h. Keep the bottle sealed tightly.

6.7 Stock standard solutions (1.00 μg/μL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.7.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in isooctane and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.7.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.8 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Establish gas chromatograph operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepared calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with isooctane. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 μL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (< 10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flash. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with isooctane. One of the standards should be at a concentraton near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 μL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard (μg/L).

$C_s$ = Concentration of the parameter to be measured (μg/L).

If the RF value over the working range is a constant (< 10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, a new calibration curve must be prepared for that compound.

7.5 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality

checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.4, 11.1, and 12.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at the following concentrations in acetone: butyl benzyl phthalate, 10 μg/mL; bis(2-ethylhexyl)phthalate, 50 μg/mL; di-n-octyl phthalate, 50 μg/mL; any other phthlate, 25 μg/mL. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at the test concentrations shown in Table 2 by adding 1.00 mL of QC check sample concentrate to each of four 1-L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter. Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at the test concentration in Section 8.2.2 or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or the test concentration in Section 8.2.2.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A-B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[9] If spiking was performed at a concentration lower than the test concentration in Section 8.2.2, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) calculate accuracy (X') using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 3, substituting X' for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X'/T)±2.44(100 S'/T)%.[9]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

**Note.**—The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most

productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

*9. Sample Collection, Preservation, and Handling*

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[10] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C from the time of collection until extraction.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

*10. Sample Extraction*

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2–L separatory funnel.

10.2 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min. with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phrase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentrator devices or techniques may be used in place of the K-D concentrator if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K-D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K-D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min.

10.7 Increase the temperature of the hot water bath to about 80 °C. Momentarily remove the Snyder column, add 50 mL of hexane and a new boiling chip, and reattach the Snyder column. Concentrate the extract as in Section 10.6, except use hexane to prewet the column. The elapsed time of concentration should be 5 to 10 min.

10.8 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of hexane. A 5-mL syringe is recommended for this operation. Adjust the extract volume to 10 mL. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with gas chromatographic analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.9 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

*11. Cleanup and Separation*

11. Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use either procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure.

11.2 If the entire extract is to be cleaned up by one of the following procedures, it must be concentrated to 2.0 mL. To the concentrator tube in Section 10.8, add a clean boiling chip and attach a two-ball micro-Snyder column. Prewet the column by adding about 0.5 mL of hexane to the top. Place the micro-K-D apparatus on a hot water bath (80 °C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches about 0.5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with 0.2 mL of hexane. Adjust the final volume to 2.0 mL and proceed with one of the following cleanup procedures.

11.3 Florisil column cleanup for phthalate esters:

11.3.1 Place 10 g of Florisil into a chromatographic column. Tap the column to settle the Florisil and add 1 cm of anhydrous sodium sulfate to the top.

11.3.2 Preelute the column with 40 mL of hexane. The rate for all elutions should be about 2 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 2-mL sample extract onto the column using an additional 2 mL of hexane to complete the transfer. Just prior to exposure of the sodium sulfate layer to the air, add 40 mL of hexane and continue the elution of the column. Discard this hexane eluate.

11.3.3 Next, elute the column with 100 mL of 20% ethyl ether in hexane (V/V) into a 500-mL K-D flask equipped with a 10-mL concentrator tube. Concentrate the collected fraction as in Section 10.6. No solvent exchange is necessary. Adjust the volume of the cleaned up extract to 10 mL in the concentrator tube and analyze by gas chromatography (Section 12).

11.4 Alumina column cleanup for phthalate esters:

11.4.1 Place 10 g of alumina into a chromatographic column. Tap the column to settle the alumina and add 1 cm of anhydrous sodium sulfate to the top.

11.4.2 Preelute the column with 40 mL of hexane. The rate for all elutions should be about 2 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 2-mL sample extract onto the column using an additional 2 mL of hexane to complete the transfer. Just prior to exposure of the sodium sulfate layer to the air, add 35 mL of hexane and continue the elution of the column. Discard this hexane eluate.

11.4.3 Next, elute the column with 140 mL of 20% ethyl ether in hexane (V/V) into a 500-mL K-D flask equipped with a 10–mL concentrator type. Concentrate the collected fraction as in Section 10.6. No solvent exchange is necessary. Adjust the volume of the cleaned up extract to 10 mL in the concentrator tube and analyze by gas chromatography (Section 12).

*12. Gas Chromatography*

12.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by Column 1 are shown in Figures 1 and 2. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 6.2 are met.

12.2 Calibrate the system daily as described in Section 7.

12.3 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and

mixed thoroughly immediately before injection into the gas chromatograph.

12.4 Inject 2 to 5 μL of the sample extract or standard into the gas-chromatograph using the solvent-flush technique.[11] Smaller (1.0 μL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 μL, and the resulting peak size in area or peak height units.

12.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.6 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

12.7 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

*13. Calculations*

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

where:

$A$ = Amount of material injected (ng).
$V_i$ = Volume of extract injected (μL).
$V_t$ = Volume of total extract (μL).
$V_s$ = Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

$A_s$ = Response for the parameter to be measured.
$A_{is}$ = Response for the internal standard.
$I_s$ = Amount of internal standard added to each extract (μg).
$V_o$ = Volume of water extracted (L).

13.2 Report results in μg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

*14. Method Performance*

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[12] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 5 × MDL to 1000 × MDL with the following exceptions: dimethyl and diethyl phthalate recoveries at 1000 × MDL were low (70%); bis-2-ethylhexyl and di-n-octyl phthalate recoveries at 5 × MDL were low (60%).[12]

14.3 This method was tested by 16 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 0.7 to 106 μg/L.[13] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

*References*

1. 40 CFR Part 136, Appendix B.

2. "Determination of Phthalates in Industrial and Municipal Wastewaters," EPA-600/4-81-063, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, October 1981.

3. ASTM Annual Book of Standards, Part 31, D3694-78, "Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents," American Society for Testing and Materials, Philadelphia.

4. Giam, C.S., Chan, H.S., and Nef, G.S. "Sensitive Method for Determination of Phthalate Ester Plasticizers in Open-Ocean Biota Samples," *Analytical Chemistry, 47,* 2225 (1975).

5. Giam, C.S., and Chan, H.S. "Control of Blanks in the Analysis of Phthalates in Air and Ocean Biota Samples," U.S. National Bureau of Standards, Special Publication 442, pp. 701-708, 1976.

6. "Carcinogens—Working with Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, August 1977.

7. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

8. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

9. Provost L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58-63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

10. ASTM Annual Book of Standards, Part 31, D3370-76, "Standard Practices for Sampling Water," American Society for Testing and Materials, Philadelphia.

11. Burke, J.A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

12. "Method Detection Limit and Analytical Curve Studies, EPA Methods 606, 607, and 608," Special letter report for EPA Contract 68-03-2606, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

13. "EPA Method Validation Study 16, Method 606 (Phthlate Esters)," Report for EPA Contract 68-03-2606 (In preparation).

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

| Parameter | Retention time (min) | | Method detection limit (μg/L) |
|---|---|---|---|
| | Column 1 | Column 2 | |
| Dimethyl phthalate | 2.03 | 0.95 | 0.29 |
| Diethyl phthalate | 2.82 | 1.27 | 0.49 |
| Di-n-butyl phthalate | 8.65 | 3.50 | 0.36 |
| Butyl benzyl phthalate | *6.94 | *5.11 | 0.34 |
| Bis(2-ethylhexyl) phthalate | *8.92 | *10.5 | 2.0 |
| Di-n-octyl phthalate | *16.2 | *18.0 | 3.0 |

Column 1 conditions: Supelcoport (100/120 mesh) coated with 1.5% SP-2250/1.95% SP-2401 packed in a 1.8 m long × 4 mm ID glass column with 5% methane/95% argon carrier gas at 60 mL/min flow rate. Column temperature held isothermal at 180°C, except where otherwise indicated.

Column 2 conditions: Supelcoport (100/120 mesh) coated with 3% OV-1 packed in a 1.8 m long × 4 mm ID glass column with 5% methane/95% argon carrier gas at 60 mL/min flow rate. Column temperature held isothermal at 200 °C, except where otherwise indicated.

*220 °C column temperature.

TABLE 2.—QC ACCEPTANCE CRITERIA—METHOD 606

| Parameter | Test conc. (μg/L) | Limit for s (μg/L) | Range for $\bar{X}$ (μg/L) | Range for P, $P_s$ (percent) |
|---|---|---|---|---|
| Bis(2-ethylhexyl) phthalate | 50 | 38.4 | 1.2–55.9 | D–158 |
| Butyl benzyl phthalate | 10 | 4.2 | 5.7–11.0 | 30–136 |
| Di-n-butyl phthalate | 25 | 8.9 | 10.3–29.6 | 23–136 |
| Diethyl phthalate | 25 | 9.0 | 1.9–33.4 | D–149 |
| Dimethyl phathalate | 25 | 9.5 | 1.3–35.5 | D–156 |
| Di-n-octyl phthalate | 50 | 13.4 | D–50.0 | D–114 |

s = Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
P, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.

Note.—These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 606

| Parameter | Accuracy, as recovery, X' (μg/L) | Single analyst precision, $s_r'$ (μg/L) | Overall precision, S' (μg/L) |
|---|---|---|---|
| Bis(2-ethylhexyl) phthalate | $0.53C + 2.02$ | $0.80\bar{X} - 2.56$ | $0.73\bar{X} - 0.17$ |
| Butyl benzyl phthalate | $0.82C + 0.13$ | $0.26\bar{X} + 0.04$ | $0.25\bar{X} + 0.07$ |
| Di-n-butyl phthalate | $0.79C + 0.17$ | $0.23\bar{X} + 0.20$ | $0.29\bar{X} + 0.06$ |
| Diethyl phthalate | $0.70C + 0.13$ | $0.27\bar{X} + 0.05$ | $0.45\bar{X} + 0.11$ |
| Dimethyl phthalate | $0.73C + 0.17$ | $0.26\bar{X} + 0.14$ | $0.44\bar{X} + 0.31$ |
| Di-n-octyl phthalate | $0.35C - 0.71$ | $0.38\bar{X} + 0.71$ | $0.62\bar{X} + 0.34$ |

$\bar{X}'$ = Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
$s_r'$ = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C = True value for the concentration, in μg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in μg/L.

BILLING CODE 6560-50-M

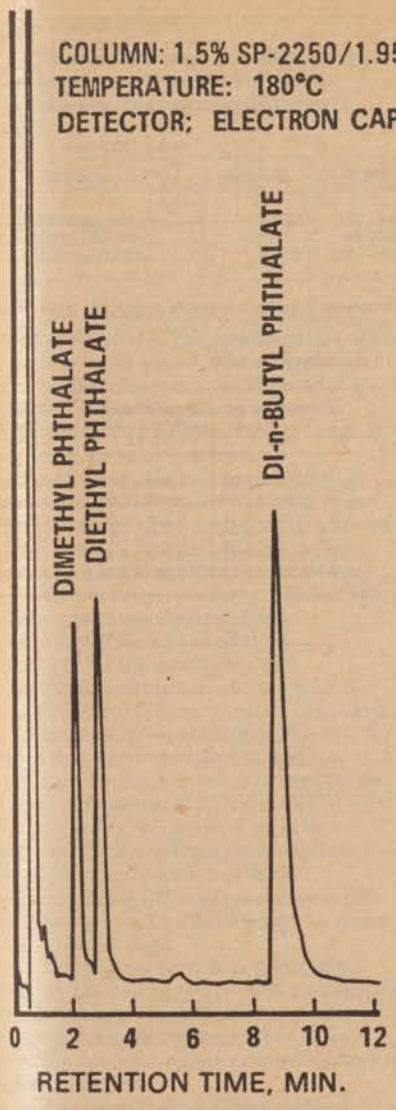



Figure 1. Gas chromatogram of phthalates.

COLUMN: 1.5% SP-2250/1.95% SP-2401 ON SUPELCOPORT
TEMPERATURE: 220°C
DETECTOR: ELECTRON CAPTURE

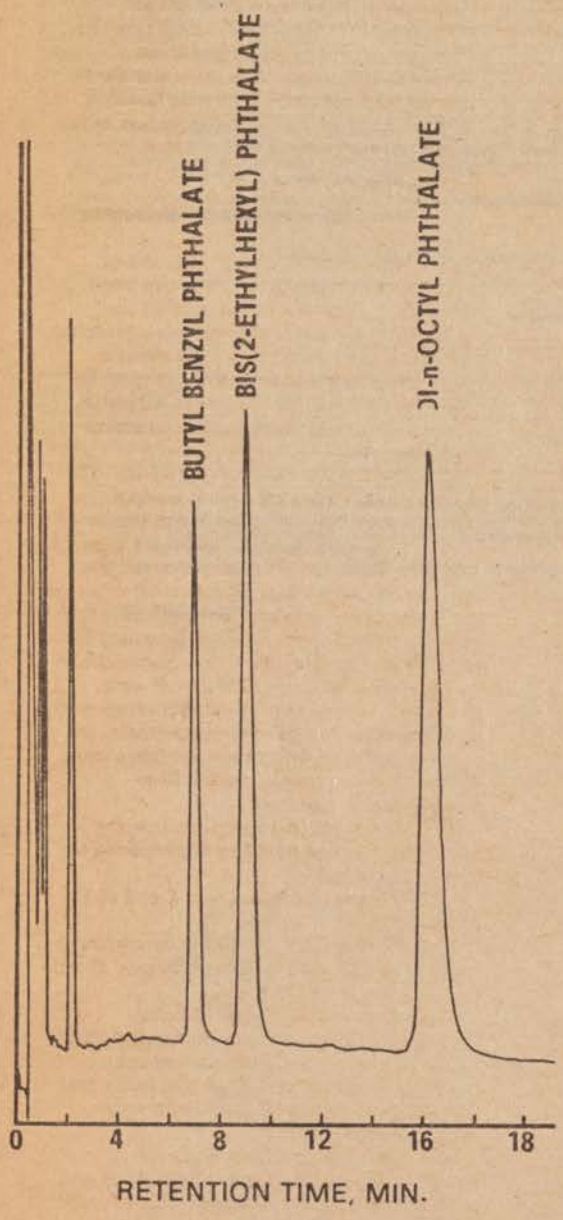



Figure 2. Gas chromatogram of phthalates.

BILLING CODE 6560-50-C

## Method 607—Nitrosamines

*1. Scope and Application*

1.1 This method covers the determination of certain nitrosamines. The following parameters can be determined by this method:

| Parameter | Storet No. | CAS No. |
|---|---|---|
| N-Nitrosodimethylamine | 34438 | 62-75-9 |
| N-Nitrosodiphenylamine | 34433 | 86-30-6 |
| N-Nitrosodi-n-propylamine | 34428 | 621-64-7 |

1.2 This is a gas chromatographic (GC) method applicable to the determination of the parameters listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for N-nitrosodi-n-propylamine. In order to confirm the presence of N-nitrosodiphenylamine, the cleanup procedure specified in Sections 11.3 or 11.4 must be used. In order to confirm the presence of N-nitrosodimethylamine by GC/MS, Column 1 of this method must be substituted for the column recommended in Method 625. Confirmation of these parameters using GC-high resolution mass spectrometry or a Thermal Energy Analyzer is also recommended. [1,2]

1.3 The method detection limit (MDL, defined in Section 14.1)[3] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 A measured volume of sample, approximately 1-L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is washed with dilute hydrochloric acid to remove free amines, dried, and concentrated to a volume of 10 mL or less. After the extract has been exchanged to methanol, it is separated by gas chromatography and the parameters are then measured with a nitrogen-phosphorus detector.[4]

2.2 The method provides Florisil and alumina column cleanup procedures to separate diphenylamine from the nitrosamines and to aid in the elimination of interferences that may be encountered.

*3. Interferences*

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[5] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedures in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

3.3 N-Nitrosodiphenylamine is reported[6,9] to undergo transnitrosation reactions. Care must be exercised in the heating or concentrating of solutions containing this compound in the presence of reactive amines.

3.4 The sensitive and selective Thermal Energy Analyzer and the reductive Hall detector may be used in place of the nitrogen-phosphorus detector when interferences are encountered. The Thermal Energy Analyzer offers the highest selectivity of the non-MS detectors.

*4. Safety*

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [10-12] for the information of the analyst.

4.2 These nitrosamines are known carcinogens [13-17], therefore, utmost care must be exercised in the handling of these materials. Nitrosamine reference standards and standard solutions should be handled and prepared in a ventilated glove box within a properly ventilated room.

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1-L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flowmeter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnels—2-L and 250-mL, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long × 19 mm ID, with coarse frit filter disc.

5.2.3 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.4 Evaporative flask, Kuderna-Danish—500-mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

5.2.5 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K-503000-0121 or equivalent).

5.2.6 Snyder column, Kuderna-Danish—Two-ball micro (Kontes K-569001-0219 or equivalent).

5.2.7 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.2.8 Chromatographic column—Approximately 400 mm long × 22 mm ID, with Teflon stopcock and coarse frit filter disc at bottom (Kontes K-420540-0234 or equivalent), for use in Florisil column cleanup procedure.

5.2.9 Chromatographic column—Approximately 300 mm long × 10 mm ID, with Teflon stopcock and coarse frit filter

disc at bottom (Kontes K-420540-0213 or equivalent), for use in alumina column cleanup procedure.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2 °C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column 1—1.8 m long × 4 mm ID glass, packed with 10% Carbowax 20 M/2% KOH on Chromosorb W-AW (80/100 mesh) or equivalent. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 12.2.

5.6.2 Column 2—1.8 m long x 4 mm ID glass, packed with 10% SP-2250 on Supel coport (100/120 mesh) or equivalent.

5.6.3 Detector—Nitrogen-phosphorus, reductive Hall, or Thermal Energy Analyzer detector.[1,2] These detectors have proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1). A nitrogen-phosphorus detector was used to develop the method performance statements in Section 14. Guidelines for the use of alternate detectors are provided in Section 12.2.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 ml.

6.3 Sodium thiosulfate—(ACS) Granular.

6.4 Sulfuric acid (1+1)—Slowly, add 50 mL of $H_2SO_4$ (ACS, sp. gr. 1.84) to 50 mL of reagent water.

6.5 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray.

6.6 Hydrochloric acid (1+9)—Add one volume of concentrated HCl (ACS) to nine volumes of reagent water.

6.7 Acetone, methanol, methylene chloride, pentane—Pesticide quality or equivalent.

6.8 Ethyl ether—Nanograde, redistilled in glass if necessary.

6.8.1 Ethyl ether must be shown to be free of peroxides before it is used as indicated by EM Laboratories Quant test strips. (Available from Scientific Products Co., Cat No. P1126-8, and other suppliers.)

6.8.2 Procedures recommended for removal of peroxides are provided with the test strips. After cleanup, 20 mL of ethyl alcohol preservative must be added to each liter of ether.

6.9 Florisil—PR grade (60/100 mesh). Purchase activated at 1250 °F and store in the dark in glass containers with ground glass stoppers or foil-lined screw caps. Before use, activate each batch at least 16 h at 130 °C in a foil-covered glass container and allow to cool.

6.10 Alumina—Basic activity Super I, W200 series (ICN Life Sciences Group, No. 404571, or equivalent). To prepare for use, place 100 g of alumina into a 500-mL reagent bottle and add 2 mL of reagent water. Mix the alumina preparation thoroughly by shaking or rolling for 10 min and let it stand for at least 2 h. The preparation should be homogeneous before use. Keep the bottle sealed tightly to ensure proper activity.

6.11 Stock standard solutions (1.00 μg/μL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.11.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in methanol and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.11.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.11.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.12 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Establish gas chromatographic operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with methanol. One of the external standards should be at a concentraton near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 μL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (< 10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with methanol. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 μL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard (μg/L).

$C_s$ = Concentration of the parameter to be measured (μg/L).

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, a new calibration curve must be prepared for that compound.

7.5 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and

document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 10.4, 11.1, and 12.2) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 20 μg/mL in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at a concentration of 20 μg/L by adding 1.00 mL of QC check sample concentrate to each of four 1-L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter. Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 20 μg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or 20 μg/L.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[18] If spiking was performed at a concentration lower than 20 μg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) calculate accuracy (X') using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 3, substituting X' for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X'/T) ± 2.44(100 S'/T)%.[18]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

**Note:** The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the

environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

*9. Sample Collection, Preservation, and Handling*

9.1 Grab samples must be collected in glass containers. Conventional sampling practices [19] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C from the time of collection until extraction. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[20] Field test kits are available for this purpose. If N-nitrosodiphenylamine is to be determined, adjust the sample pH to 7 to 10 with sodium hydroxide solution or sulfuric acid.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[4]

9.4 Nitrosamines are known to be light sensitive.[7] Samples should be stored in amber or foil-wrapped bottles in order to minimize photolytic decomposition.

*10. Sample Extraction*

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2-L separatory funnel. Check the pH of the sample with wide-range pH paper and adjust to within the range of 5 to 9 with sodium hydroxide solution or sulfuric acid.

10.2 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.5 Add 10 mL of hydrochloric acid to the combined extracts and shake for 2 min. Allow the layers to separate. Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.7 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of methylene chloride. A 5-mL syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If N-nitrosodiphenylamine is to be measured by gas chromatography, the analyst must first use a cleanup column to eliminate diphenylamine interference (Section 11). If N-nitrosodiphenylamine is of no interest, the analyst may proceed directly with gas chromatographic analysis (Section 12).

10.8 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

*11. Cleanup and Separation*

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use either procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure. Diphenylamine, if present in the original sample extract, must be separated from the nitrosamines if N-nitrosodiphenylamine is to be determined by this method.

11.2 If the entire extract is to be cleaned up by one of the following procedures, it must be concentrated to 2.0 mL. To the concentrator tube in Section 10.7, add a clean boiling chip and attach a two-ball micro-Snyder column. Prewet the column by adding about 0.5 mL of methylene chloride to the top. Place the micr-K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches about 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with 0.2 mL of methylene chloride. Adjust the final volume to 2.0 mL and proceed with one of the following cleanup procedures.

11.3 Florisil column cleanup for nitrosamines:

11.3.1 Place 22 g of activated Florisil into a 22-mm ID chromatographic column. Tap the column to settle the Florisil and add about 5 mm of anhydrous sodium sulfate to the top.

11.3.2 Preelute the column with 40 mL of ethyl ether/pentane (15+85)(V/V). Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 2-mL sample extract onto the column using an additional 2 mL of pentane to complete the transfer.

11.3.3 Elute the column with 90 mL of ethyl ether/pentane (15+85)(V/V) and discard the eluate. This fraction will contain the diphenylamine, if it is present in the extract.

11.3.4 Next, elute the column with 100 mL of acetone/ethyl ether (5+95)(V/V) into a 500-mL K–D flask equipped with a 10-mL concentrator tube. This fraction will contain all of the nitrosamines listed in the scope of the method.

11.3.5 Add 15 mL of methanol to the collected fraction and concentrate as in Section 10.6, except use pentane to prewet the column and set the water bath at 70 to 75 °C. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of pentane. Analyze by gas chromatography (Section 12).

11.4 Alumina column cleanup for nitrosamines:

11.4.1 Place 12 g of the alumina preparation (Section 6.10) into a 10-mm ID chromatographic column. Tap the column to settle the alumina and add 1 to 2 cm of anhydrous sodium sulfate to the top.

11.4.2 Preelute the column with 10 mL of ethyl ether/pentane (3+7)(V/V). Discard the eluate (about 2 mL) and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 2 mL sample extract onto the column using an additional 2 mL of pentane to complete the transfer.

11.4.3 Just prior to exposure of the sodium sulfate layer to the air, add 70 mL of ethyl ether/pentane (3+7)(V/V). Discard the first 10 mL of eluate. Collect the remainder of the eluate in a 500-mL K–D flask equipped with a 10 mL concentrator tube. This fraction contains N-nitrosodiphenylamine and probably a small amount of N-nitrosodi-n-propylamine.

11.4.4 Next, elute the column with 60 mL of ethyl ether/pentane (1+1)(V/V), collecting the eluate in a second K–D flask equipped with a 10–mL concentrator tube. Add 15 mL of methanol to the K–D flask. This fraction will contain N-nitrosodimethylamine, most of the N-nitrosodi-n-propylamine and any diphenylamine that is present.

11.4.5 Concentrate both fractions as in Section 10.6, except use pentane to prewet the column. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of pentane. Analyze the fractions by gas chromatography (Section 12).

*12. Gas Chromatography*

12.1 N-nitrosodiphenylamine completely reacts to form diphenylamine at the normal operating temperatures of a GC injection port (200 to 250 °C). Thus, N-nitrosodiphenylamine is chromatographed and detected as diphenylamine. Accurate determination depends on removal of diphenylamine that may be present in the original extract prior to GC analysis (See Section 11).

12.2 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by Column 1 are shown in Figures 1 and 2. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

12.3 Calibrate the system daily as described in Section 7.

12.4 If the extract has not been subjected to one of the cleanup procedures in Section 11, it is necessary to exchange the solvent from methylene chloride to methanol before the thermionic detector can be used. To a 1 to 10-mL volume of methylene chloride extract in a concentrator tube, add 2 mL of methanol and a clean boiling chip. Attach a two-ball micro-Snyder column to the concentrator tube. Prewet the column by adding about 0.5 mL of methylene chloride to the top. Place the micro-K–D apparatus on a boiling (100 °C) water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches about 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with 0.2 mL of methanol. Adjust the final volume to 2.0 mL.

12.5 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the gas chromatograph.

12.6 Inject 2 to 5 μL of the sample extract or standard into the gas chromatograph using the solent-flush technique.[21] Smaller (1.0 μL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 μL, and the resulting peak size in area or peak height units.

12.7 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.8 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

12.9 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

*13. Calculations*

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

Equation 2.

$$\text{Concentration } (\mu/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

where:

$A$ = Amount of material injected (ng).
$V_i$ = Volume of extract injected (μL).
$V_t$ = Volume of total extract (μL).
$V_s$ = Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

$A_s$ = Response for the parameter to be measured.
$A_{is}$ = Response for the internal standard.
$I_s$ = Amount of internal standard added to each extract (μg).
$V_o$ = Volume of water extracted (L).

13.2 Report results in μg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

*14. Method Performance*

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[3] The MDL concentrations listed in Table 1 were obtained using reagent water.[22] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 4 x MDL to 1000 x MDL.[22]

14.3 This method was tested by 17 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 0.8 to 55 μg/L.[23] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

*References*

1. Fine, D.H., Lieb, D., and Rufeh, R. "Principle of Operation of the Thermal Energy Analyzer for the Trace Analysis of Volatile and Non-volatile N-nitroso Compounds," *Journal of Chromatography, 107,* 351 (1975).

2. Fine, D.H., Hoffman, F., Rounbehler, D.P., and Belcher, N.M. "Analysis of N-nitroso Compounds by Combined High Performance Liquid Chromatography and Thermal Energy Analysis," Walker, E.A., Bogovski, P. and Griciute, L., Editors, N-nitroso Compounds—Analysis and Formation, Lyon, International Agency for Research on Cancer (IARC Scientific Publications No. 14), pp. 43–50 (1976).

3. 40 CFR Part 136, Appendix B.

4. "Determination of Nitrosamines in Industrial and Municipal Wastewaters," EPA–600/4–82–016, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, May 1982.

5. ASTM Annual Book of Standards, Part 31, D3694–78. "Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents," American Society for Testing and Materials, Philadelphia.

6. Buglass, A.J., Challis, B.C., and Osborn, M.R. "Transnitrosation and Decomposition of Nitrosamines," Bogovski, P. and Walker, E.A., Editors, N-nitroso Compounds in the Environment, Lyon, International Agency for Research on Cancer (IARC Scientific Publication No. 9), pp. 94–100 (1974).

7. Burgess, E.M., and Lavanish, J.M. "Photochemical Decomposition of N-nitrosamines," *Tetrahedon Letters,* 1221 (1964)

8. Druckrey, H., Preussmann, R., Ivankovic, S., and Schmahl, D. "Organotrope Carcinogene Wirkungen bei 65 Verschiedenen N-NitrosoVerbindungen an BD-Ratten," *Z. Krebsforsch., 69,* 103 (1967).

9. Fiddler, W. "The Occurrence and Determination of N-nitroso Compounds," *Toxicol. Appl. Pharmacol., 31,* 352 (1975).

10. "Carcinogens—Working With Carcinogens," Department of Health,

Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, August 1977.

11. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

12. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

13. Lijinsky, W. "How Nitrosamines Cause Cancer," *New Scientist, 73,* 216 (1977).

14. Mirvish, S.S. "N-Nitroso compounds: Their Chemical and in vivo Formation and Possible Importance as Environmental Carcinogens," *J. Toxicol. Environ. Health, 3,* 1267 (1977).

15. "Reconnaissance of Environmental Levels of Nitrosamines in the Central United States," EPA-330/1-77-001, National Enforcement Investigations Center, U.S. Environmental Protection Agency (1977).

16. "Atmospheric Nitrosamine Assessment Report," Office of Air Quality Planning and Standards, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina (1976).

17. "Scientific and Technical Assessment Report on Nitrosamines," EPA-660/6-7-001, Office of Research and Development, U.S. Environmental Protection Agency (1976).

18. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58-63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value of 1.22 derived in this report.)

19. ASTM Annual Book of Standards, Part 31, D3370-76. "Standard Practices for Sampling Water," American Society for Testing and Materials, Philadelphia.

20. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

21. Burke, J. A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

22. "Method Detection Limit and Analytical Curve Studies EPA Methods 606, 607, and 608," Special letter report for EPA Contract 68-03-2606, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

23. "EPA Method Validation Study 17, Method 607 (Nitrosamines)," Report for EPA Contract 68-03-2606 (In preparation).

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

| Parameter | Retention time (min) | | Method detection limit (μg/L) |
|---|---|---|---|
| | Column 1 | Column 2 | |
| N-Nitrosodimethylamine | 4.1 | 0.68 | 0.15 |
| N-Nitrosodi-n-propylamine | 12.1 | 4.2 | .46 |
| N-Nitrosodiphenylamine * | [b] 12.8 | [c] 6.4 | .81 |

Column 1 conditions: Chromosorb W-AW (80/100 mesh) coated with 10% Carbowax 20 M/2% KOH packed in a 1.8 m long x 4mm ID glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held isothermal at 110 °C, except where otherwise indicated.

Column 2 conditions: Supelcoport (100/120 mesh) coated with 10% SP-2250 packed in a 1.8 m long x 4 mm ID glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held isothermal at 120 °C, except where otherwise indicated.

* Measured as diphenylamine.
[b] 220 °C column temperature.
[c] 210 °C column temperature.

TABLE 2.—QC ACCEPTANCE CRITERIA—METHOD 607

| Parameter | Test conc. (μg/L) | Limit for s (μg/L) | Range for $\bar{X}$ (μg/L) | Range for P, $P_s$ (percent) |
|---|---|---|---|---|
| N-Nitrosodimethylamine | 20 | 3.4 | 4.6-20.0 | 13-109 |
| N-Nitrosodiphenyl | 20 | 6.1 | 2.1-24.5 | D-139 |
| N-Nitrosodi-n-propylamine | 20 | 5.7 | 11.5-26.8 | 45-146 |

s = Standard deviation for four recovery measurements, in μg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
P, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.

**Note.**—These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 607

| Parameter | Accuracy, as recovery, X' (μg/L) | Single analyst precision, $s_r'$ (μg/L) | Overall precision, S' (μg/L) |
|---|---|---|---|
| N-Nitrosodimethylamine | $0.37C+0.06$ | $0.25\bar{X}-0.04$ | $0.25\bar{X}+0.11$ |
| N-Nitrosodiphenylamine | $0.64C+0.52$ | $0.36\bar{X}-1.53$ | $0.46\bar{X}-0.47$ |
| N-Nitrosodi-n-propylamine | $0.96C-0.07$ | $0.15\bar{X}+0.13$ | $0.21\bar{X}+0.15$ |

X' = Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
$s_r'$ = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C = True value for the concentration, in μg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in μg/L.

BILLING CODE 6560-50-M

COLUMN: 10% CARBOWAX 20M / 2% KOH ON CHROMOSORB W-AW
TEMPERATURE: 110°C
DETECTOR: PHOSPHORUS/NITROGEN

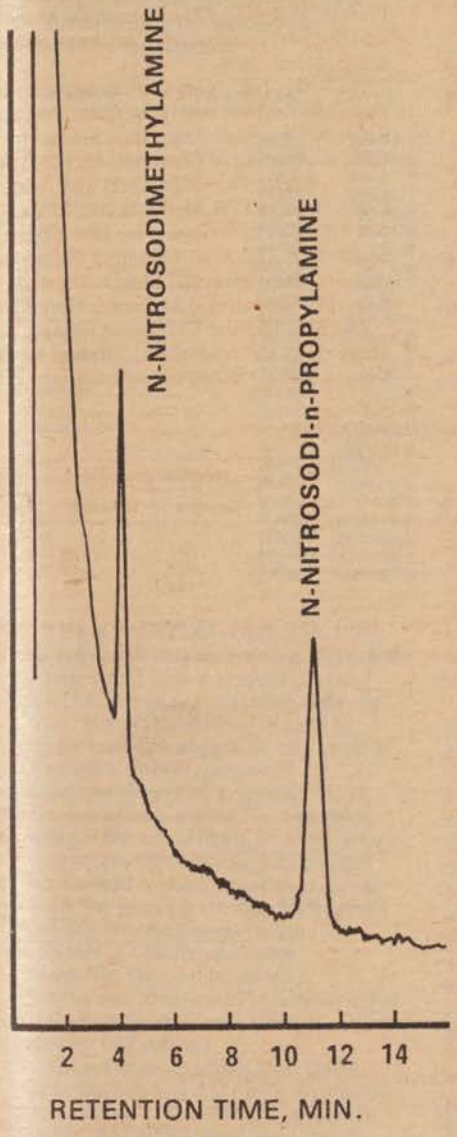



Figure 1. Gas chromatogram of nitrosamines.

COLUMN: 10% CARBOWAX 20M / 2% KOH ON CHROMOSORB W-AW
TEMPERATURE: 220°C
DETECTOR: PHOSPHORUS/NITROGEN

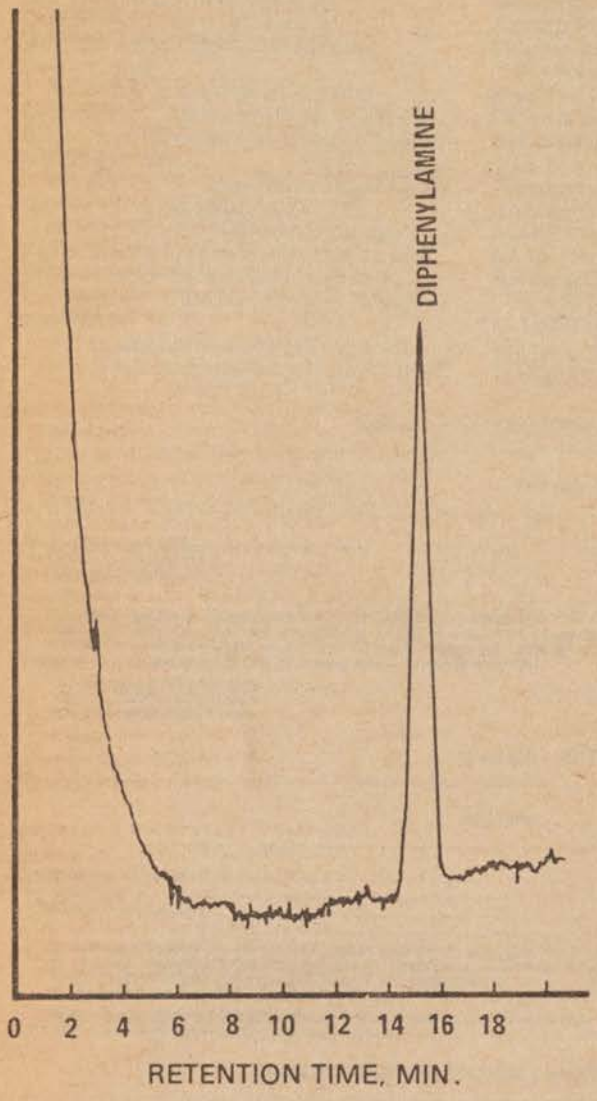



Figure 2. Gas chromatogram of N-nitrosodiphenylamine as diphenylamine.

BILLING CODE 6560-50-C

## Method 608—Organochlorine Pesticides and PCBs

*1. Scope and Application*

1.1 This method covers the determination of certain organochlorine pesticides and PCBs. The following parameters can be determined by this method:

| Parameter | STORET No. | CAS No. |
|---|---|---|
| Aldrin | 39330 | 309-00-2 |
| α-BHC | 39337 | 319-84-6 |
| β-BHC | 39338 | 319-85-7 |
| δ-BHC | 34259 | 319-86-8 |
| γ-BHC | 39340 | 58-89-9 |
| Chlordane | 39350 | 57-74-9 |
| 4,4'-DDD | 39310 | 72-54-8 |
| 4,4'-DDE | 39320 | 72-55-9 |
| 4,4'-DDT | 39300 | 50-29-3 |
| Dieldrin | 39380 | 60-57-1 |
| Endosulfan I | 34361 | 959-98-8 |
| Endosulfan II | 34356 | 33212-65-9 |
| Endosulfan sulfate | 34351 | 1031-07-8 |
| Endrin | 39390 | 72-20-8 |
| Endrin aldehyde | 34366 | 7421-93-4 |
| Heptachlor | 39410 | 76-44-8 |
| Heptachlor epoxide | 39420 | 1024-57-3 |
| Toxaphene | 39400 | 8001-35-2 |
| PCB-1016 | 34671 | 12674-11-2 |
| PCB-1221 | 39488 | 1104-28-2 |
| PCB-1232 | 39492 | 11141-16-5 |
| PCB-1242 | 39496 | 53469-21-9 |
| PCB-1248 | 39500 | 12672-29-6 |
| PCB-1254 | 39504 | 11097-69-1 |
| PCB-1260 | 39508 | 11096-82-5 |

1.2 This is a gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 The sample extraction and concentration steps in this method are essentially the same as in Methods 606, 609, 611, and 612. Thus, a single sample may be extracted to measure the parameters included in the scope of each of these methods. When cleanup is required, the concentration levels must be high enough to permit selecting aliquots, as necessary, to apply appropriate cleanup procedures. The analyst is allowed the latitude, under Section 12, to select chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.5 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 A measured volume of sample, approximately 1-L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchagned to hexane during concentration to a volume of 10 mL or less. The extract is separated by gas chromatography and the parameters are then measured with an electron capture detector.[2]

2.2 The method provides a Florisil column cleanup procedure and an elemental sulfur removal procedure to aid in the elimination of interferences that may be encountered.

*3. Interferences*

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Interferences by phthalate esters can pose a major problem in pesticide analysis when using the electron capture detector. These compounds generally appear in the chromatogram as large late eluting peaks, especially in the 15 and 50% fractions from Florisil. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding the use of plastics in the laboratory. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.[4,5] The interferences from phthalate esters can be avoided by using a microcoulometric or electrolytic conductivity detector.

3.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedures in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

*4. Safety*

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [6-8] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, the BHCs, and the PCBs. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1-L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4°C and protected from light during composting. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating

flow meter is required to collect flow proportional composites.

5.2. Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2-L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long × 19 mm ID, with coarse frit filter disc.

5.2.3 Chromatographic column—400 mm long × 22 mm ID, with Teflon stopcock and coarse frit filter disc (Kontes K-42054 or equivalent).

5.2.4 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.5 Evaporative flask, Kuderna-Danish—500-mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

5.2.6 Snyder column, Kuderna/Danish—Three-ball macro (Kontes K-503000-0121 or equivalent).

5.2.7 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.3. Boiling chips—Approximately 10/40 mesh. Heat to 400°C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2°C). The bath should be used in a hood.

5.5. Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6. Gas chromatograph—An analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column 1—1.8 m long × 4 mm ID glass, packed with 1.5% SP-2250/1.95% SP-2401 on Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 12.1.

5.6.2 Column 2—1.8 m long × 4 mm ID glass, packed with 3% OV-1 on Supelcoport (100/120 mesh) or equivalent.

5.6.3 Detector—Electron capture detector. This detector has proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 14. Guidelines for the use of alternate detectors are provided in Section 12.1.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.3 Sodium thiosulfate—(ACS) Granular.

6.4 Sulfuric acid (1+1)—Slowly, add 50 mL to $H_2SO_4$ (ACS, sp. gr. 1.84) to 50 mL of reagent water.

6.5 Acetone, hexane, isooctane, methylene chloride—Pesticide quality or equivalent.

6.6 Ethyl ether—Nanograde, redistilled in glass if necessary.

6.6.1 Ethyl ether must be shown to be free of peroxides before it is used as indicated by EM Laboratories Quant test strips. (Available from Scientific Products Co., Cat. No. P1126-8, and other suppliers.)

6.6.2 Procedures recommended for removal of peroxides are provided with the test strips. After cleanup, 20 mL of ethyl alcohol preservative must be added to each liter of ether.

6.7 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray.

6.8 Florisil—PR grade (60/100 mesh). Purchase activated at 1250 °F and store in the dark in glass containers with ground glass stoppers or foil-lined screw caps. Before use, activate each batch at least 16 h at 130 °C in a foil-covered glass container and allow to cool.

6.9 Mercury—Triple distilled.

6.10 Copper powder—Activated.

6.11 Stock standard solutions (1.00 μg/μL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.11.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in isooctane and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.11.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.11.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.12 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Establish gas chromatographic operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with isooctane. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 μL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with isooctane. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 μL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard (μg/L).

$C_s$ = Concentraton of the parameter to be measured (μg/L).

If the RF value over the working range is a constant (< 10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, the test must be repeated using a fresh calibration

standard. Alternatively, a new calibration curve must be prepared for that compound.

7.5 The cleanup procedure in Section 11 utilizes Florisil column chromatography. Florisil from different batches or sources may vary in adsorptive capacity. To standardize the amount of Florisil which is used, the use of lauric acid value [9] is suggested. The referenced procedure determines the adsorption from hexane solution of lauric acid (mg) per g of Florisil. The amount of Florisil to be used for each column is calculated by dividing 110 by this ratio and multiplying by 20 g.

7.6 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.4, 11.1, and 12.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each single-component parameter of interest at the following concentrations in acetone: 4,4'-DDD, 10 μg/mL; 4,4'-DDT, 10 μg/mL; endosulfan II, 10 μg/mL; endosulfan sulfate, 10 μg/mL; endrin, 10 μg/mL; any other single-component pesticide, 2 μg/mL. If this method is only to be used to analyze for PCBs, chlordane, or toxaphene, the QC check sample concentrate should contain the most representative multicomponent parameter at a concentration of 50 μg/mL in acetone. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at the test concentrations shown in Table 3 by adding 1.00 mL of QC check sample concentrate to each of four 1-L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/mL; and the standard deviation of the recovery (s) in μg/mL, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 3. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter.

**Note.**—The large number of parameters in Table 3 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.2.6.2 Beginning with Section 8.2.2, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at the test concentration in Section 8.2.2 or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or the test concentration in Section 8.2.2.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 3. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[10] If spiking was performed at a concentration lower than the test concentration in Section 8.2.2, the analyst must use either the QC acceptance criteria in Table 3, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X') using the equation in Table 4, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 4, substituting X' for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as $(100\ X'/T) \pm 2.44(100\ S'/T)\%$.[10]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC

check standard containing each parameter that failed must be prepared and analyzed.

**Note.**—The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of parameters in Table 3 must be measured in the sample in Section 8.3, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spike sample.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standards to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 3. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2\,s_p$ to $\bar{P}+2\,s_p$. If $\bar{P}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

*9. Sample Collection, Preservation, and Handling*

9.1 Grab samples must be collected in glass containers. Conventional sampling practices [11] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C from the time of collection until extraction. If the samples will not be extracted within 72 h of collection, the sample should be adjusted to a pH range of 5.0 to 9.0 with sodium hydroxide solution or sulfuric acid. Record the volume of acid or base used. If aldrin is to be determined, add sodium thiosulfate when residual chlorine is present. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[12] Field test kits are available for this purpose.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

*10. Sample Extraction*

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2–L separatory funnel.

10.2 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optium technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250–mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.7 Increase the temperature of the hot water bath to about 80 °C. Momentarily remove the Snyder column, add 50 mL of hexane and a new boiling chip, and reattach the Snyder column. Concentrate the extract as in Section 10.6, except use hexane to prewet the column. The elapsed time of concentration should be 5 to 10 min.

10.8 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of hexane. A 5-mL syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with gas chromatographic analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.9 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

*11. Cleanup and Separation*

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use either procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure. The Florisil column allows for a select fractionation of the compounds and will eliminate polar interferences. Elemental sulfur, which interferes with the electron capture gas chromatography of certain pesticides, can be removed by the technique described in Section 11.3.

11.2 Florisil column cleanup:

11.2.1 Place a weight of Florisil (nominally 20 g) predetermined by calibration (Section 7.5), into a chromatographic column. Tap the column to settle the Florisil and add 1 to 2 cm of anhydrous sodium sulfate to the top.

11.2.2 Add 60 mL of hexane to wet and rinse the sodium sulfate and Florisil. Just prior to exposure of the sodium sulfate layer to the air, stop the elution of the hexane by closing the stopcock on the chromatographic column. Discard the eluate.

11.2.3 Adjust the sample extract volume to 10 mL with hexane and transfer it from the K–D concentrator tube onto the column. Rinse the tube twice with 1 to 2 mL of hexane, adding each rinse to the column.

11.2.4 Place a 500-mL K-D flask and clean concentrator tube under the chromatographic column. Drain the column into the flask until the sodium sulfate layer is nearly exposed. Elute the column with 200 mL of 6% ethyl ether in hexane (V/V) (Fraction 1) at a rate of

about 5 mL/min. Remove the K-D flask and set it aside for later concentration. Elute the column again, using 200 mL of 15% ethyl ether in hexane (V/V) (Fraction 2), into a second K-D flask. Perform the third elution using 200 mL of 50% ethyl ether in hexane (V/V) (Fraction 3). The elution patterns for the pesticides and PCBs are shown in Table 2.

11.2.5 Concentrate the fractions as in Section 10.6, except use hexane to prewet the column and set the water bath at about 85 °C. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with hexane. Adjust the volume of each fraction to 10 mL with hexane and analyze by gas chromatography (Section 12).

11.3 Elemental sulfur will usually elute entirely in Fraction 1 of the Florisil column cleanup. To remove sulfur interference from this fraction or the original extract, pipet 1.00 mL of the concentrated extract into a clean concentrator tube or Teflon-sealed vial. Add one to three drops of mercury and seal.[13] Agitate the contents of the vial for 15 to 30 s. Prolonged shaking (2 h) may be required. If so, this may be accomplished with a reciprocal shaker. Alternatively, activated copper powder may be used for sulfur removal.[14] Analyze by gas chromatography.

*12. Gas Chromatography*

12.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by Column 1 are shown in Figures 1 to 10. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

12.2 Calibrate the system daily as described in Section 7.

12.3 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the gas chromatograph.

12.4 Inject 2 to 5 μL of the sample extract or standard into the gas chromatograph using the solvent-flush technique.[15] Smaller (1.0 uL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 μL, the total extract volume, and the resulting peak size in area or peak height units.

12.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.6 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

12.7 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

*13. Calculations*

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

where:

$A$ = Amount of material injected (ng).
$V_i$ = Volume of extract injected (μL).
$V_t$ = Volume of total extract (μL).
$V_s$ = Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)\ (I_s)}{(A_{is})(RF)(V_o)}$$

where:

$A_s$ = Response for the parameter to be measured.
$A_{is}$ = Response for the internal standard.
$I_s$ = Amount of internal standard added to each extract (μg).
$V_o$ = Volume of water extracted (L).

13.2 When it is apparent that two or more PCB (Aroclor) mixtures are present, the Webb and McCall procedure [16] may be used to identify and quantify the Aroclors.

13.3 For multicomponent mixtures (chlordane, toxaphene, and PCBs) match retention times of peaks in the standards with peaks in the sample. Quantitate every identifiable peak unless interference with individual peaks persist after cleanup. Add peak height or peak area of each identified peak in the chromatogram. Calculate as total response in the sample versus total response in the standard.

13.4 Report results in μg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

*14. Method Performance*

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[17] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 4×MDL to 1000×MDL with the following exceptions: Chlordane recovery at 4×MDL was low (60%); Toxaphene recovery was demonstrated linear over the range of 10×MDL to 1000×MDL.[17]

14.3 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations.[18] Concentrations used in the study ranged from 0.5 to 30 μg/L for single-component pesticides and from 8.5 to 400 μg/L for multicomponent parameters. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 4.

**References**

1. 40 CFR Part 136, Appendix B.

2. "Determination of Pesticides and PCBs in Industrial and Municipal Wastewaters," EPA–600/4–82–023, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, June 1982.

3. ASTM Annual Book of Standards, Part 31, D3694–78. "Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents," American Society for Testing and Materials, Philadelphia.

4. Giam, C.S., Chan, H.S., and Nef, G.S., "Sensitive Method for Determination of Phthalate Ester Plasticizers in Open-Ocean Biota Samples," *Analytical Chemistry, 47,* 2225 (1975).

5. Giam, C.S., Chan, H.S. "Control of Blanks in the Analysis of Phthalates in Air and Ocean Biota Samples," U.S. National Bureau of Standards, Special Publication 442, pp. 701–708, 1976.

6. "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

7. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

8. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

9. Mills, P.A. "Variation of Florisil Activity: Simple Method for Measuring Absorbent Capacity and Its Use in Standardizing Florisil Columns," *Journal of the Association of Official Analytical Chemists, 51,* 29, (1968).

10. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58–63 (1983). (The

value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

11. ASTM Annual Book of Standards, Part 31, D3370–76, "Standard Practices for Sampling Water," American Society for Testing and Materials, Philadelphia.

12. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

13. Goerlitz, D.F., and Law, L.M. *Bulletin for Environmental Contamination and Toxicology, 6,* 9 (1971).

14. "Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples," EPA–600/8–80–038, U.S. Environmental Protection Agency, Health Effects Research Laboratory, Research Triangle Park, North Carolina.

15. Burke, J.A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

16. Webb, R.G., and McCall, A.C. "Quantitative PCB Standards for Election Capture Gas Chromatography," *Journal of Chromatographic Science, 11,* 366 (1973).

17. "Method Detection Limit and Analytical Curve Studies, EPA Methods 606, 607, and 608," Special letter report for EPA Contract 68–03–2606, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

18. "EPA Method Validation Study 18, Method 608 (Organochlorine Pesticides and PCBs)," Report for EPA Contract 68–03–2606 (In preparation).

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

| Parameter | Retention time (min) | | Method detection limit (µg/L) |
|---|---|---|---|
| | Col. 1 | Col. 2 | |
| α-BHC | 1.35 | 1.82 | 0.003 |
| γ-BHC | 1.70 | 2.13 | 0.00 |
| β-BHC | 1.90 | 1.97 | 0.00 |
| Heptachlor | 2.00 | 3.35 | 0.003 |
| δ-BHC | 2.15 | 2.20 | 0.009 |
| Aldrin | 2.40 | 4.10 | 0.004 |
| Heptachlor epoxide | 3.50 | 5.00 | 0.083 |
| Endosulfan I | 4.50 | 6.20 | 0.014 |
| 4,4'-DDE | 5.13 | 7.15 | 0.004 |
| Dieldrin | 5.45 | 7.23 | 0.002 |
| Endrin | 6.55 | 8.10 | 0.006 |
| 4,4'-DDD | 7.83 | 9.08 | 0.011 |
| Endosulfan II | 8.00 | 8.28 | 0.004 |
| 4,4'-DDT | 9.40 | 11.75 | 0.012 |
| Endrin aldehyde | 11.82 | 9.30 | 0.023 |
| Endosulfan sulfate | 14.22 | 10.70 | 0.066 |
| Chlordane | mr | mr | 0.014 |
| Toxaphene | mr | mr | 0.24 |
| PCB–1016 | mr | mr | nd |
| PCB–1221 | mr | mr | nd |
| PCB–1232 | mt | mr | nd |
| PCB–1242 | mr | mr | 0.065 |
| PCB–1248 | mr | mr | nd |
| PCB–1254 | mr | mr | nd |
| PCB–1260 | mr | mr | nd |

Column 1 conditions: Supelcoport (100/120 mesh) coated with 1.5% SP–2250/1.95% SP–2401 packed in a 1.8 m long × 4 mm ID glass column with 5% methane/95% argon carrier gas at 60 mL/min flow rate. Column temperature held isothermal at 200 °C, except for PCB–1016 through PCB–1248, should be measured at 160 °C.

Column 2 conditions: Supelcoport (100/120 mesh) coated with 3% OV–1 packed in a 1.8 m long × 4 mm ID glass column with 5% methane/95% argon carrier gas at 60 mL/min flow rate. Column temperature held isothermal at 200 °C for the pesticides; at 140 °C for PCB–1221 and 1232; and at 170 °C for PCB–1016 and 1242 to 1268.

mr = Multiple peak response. See Figures 2 thru 10.
nd = Not determined.

TABLE 2.—DISTRIBUTION OF CHLORINATED PESTICIDES AND PCBs INTO FLORISIL COLUMN FRACTIONS [2]

| Parameter | Percent recovery by fraction * | | |
|---|---|---|---|
| | 1 | 2 | 3 |
| Aldrin | 100 | | |
| α-BHC | 100 | | |
| β-BHC | 97 | | |
| δ-BHC | 98 | | |
| γ-BHC | 100 | | |
| Chlordane | 100 | | |
| 4,4'-DDD | 99 | | |
| 4,4'-DDE | 98 | | |
| 4,4'-DDT | 100 | | |
| Dieldrin | 0 | 100 | |
| Endosulfan I | 37 | 64 | |
| Endosulfan II | 0 | 7 | 91 |
| Endosulfan sulfate | 0 | 0 | 106 |
| Endrin | 4 | 96 | |
| Endrin aldehyde | 0 | 68 | 26 |
| Heptachlor | 100 | | |
| Heptachlor epoxide | 100 | | |
| Toxaphene | J6 | | |
| PCB–1016 | 97 | | |
| PCB–1221 | 97 | | |
| PCB–1232 | 95 | 4 | |
| PCB–1242 | 97 | | |
| PCB–1248 | 103 | | |
| PCB–1254 | 90 | | |
| PCB–1260 | 95 | | |

* Eluant composition:
Fraction 1–6% ethyl ether in hexane.
Fraction 2–15% ethyl ether in hexane.
Fraction 3–50% ethyl ether in hexane.

TABLE 3.—QC ACCEPTANCE CRITERIA—METHOD 608

| Parameter | Test conc. (µg/L) | Limit for s (µg/L) | Range for $\bar{X}$ (µg/L) | Range for P, $P_s$(%) |
|---|---|---|---|---|
| Aldrin | 2.0 | 0.42 | 1.08–2.24 | 42–122 |
| α-BHC | 2.0 | 0.48 | .98–2.44 | 37–134 |
| β-BHC | 2.0 | 0.64 | 0.78–2.60 | 17–147 |
| δ-BHC | 2.0 | 0.72 | 1.01–2.37 | 19–140 |
| γ-BHC | 2.0 | 0.46 | 0.86–2.32 | 32–127 |
| Chlordane | 50 | 10.0 | 27.6–54.3 | 45–119 |
| 4,4'-DDD | 10 | 2.8 | 4.8–12.6 | 31–141 |
| 4,4'-DDE | 2.0 | 0.55 | 1.08–2.60 | 30–145 |
| 4,4'-DDT | 10 | 3.6 | 4.6–13.7 | 25–160 |
| Dieldrin | 2.0 | 0.76 | 1.15–2.49 | 36–146 |
| Endosulfan I | 2.0 | 0.49 | 1.14–2.82 | 45–153 |
| Endosulfan II | 10 | 6.1 | 2.2–17.1 | D–202 |
| Endosulfan Sulfate | 10 | 2.7 | 3.8–13.2 | 26–144 |
| Endrin | 10 | 3.7 | 5.1–12.6 | 30–147 |
| Heptachlor | 2.0 | 0.40 | 0.86–2.00 | 34–111 |
| Heptachlor epoxide | 2.0 | 0.41 | 1.13–2.63 | 37–142 |
| Toxaphene | 50 | 12.7 | 27.8–55.6 | 41–126 |
| PCB–1016 | 50 | 10.0 | 30.5–51.5 | 50–114 |
| PCB–1221 | 50 | 24.4 | 22.1–75.2 | 15–178 |
| PCB–1232 | 50 | 17.9 | 14.0–98.5 | 10–215 |
| PCB–1242 | 50 | 12.2 | 24.8–89.6 | 39–150 |
| PCB–1248 | 50 | 15.9 | 29.0–70.2 | 38–158 |
| PCB–1254 | 50 | 13.8 | 22.2–57.9 | 29–131 |
| PCB–1260 | 50 | 10.4 | 18.7–54.9 | 8–127 |

$s$ = Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.

**Note.**—These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

TABLE 4. METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 608

| Parameter | Accuracy, as recovery, $X'$ (µg/L) | Single analyst precision, $s_r'$ (µg/L) | Overall precision, $S'$ (µg/L) |
|---|---|---|---|
| Aldrin | 0.81C+0.04 | 0.16$\bar{X}$−0.04 | 0.20$\bar{X}$−0.01 |
| α-BHC | 0.84C+0.03 | 0.13$\bar{X}$+0.04 | 0.23$\bar{X}$−0.00 |
| β-BHC | 0.81C+0.07 | 0.22$\bar{X}$−0.02 | 0.33$\bar{X}$−0.95 |
| δ-BHC | 0.81C+0.07 | 0.18$\bar{X}$+0.09 | 0.25$\bar{X}$+0.03 |
| γ-BHC | 0.82C−0.05 | 0.12$\bar{X}$+0.06 | 0.22$\bar{X}$+0.04 |
| Chlordane | 0.82C−0.04 | 0.13$\bar{X}$+0.13 | 0.18$\bar{X}$+0.18 |
| 4,4'-DDD | 0.84C+0.30 | 0.20$\bar{X}$−0.18 | 0.27$\bar{X}$−0.14 |
| 4,4'-DDE | 0.85C+0.14 | 0.13$\bar{X}$+0.06 | 0.28$\bar{X}$−0.09 |
| 4,4'-DDT | 0.93C−0.13 | 0.17$\bar{X}$+0.39 | 0.31$\bar{X}$−0.21 |
| Dieldrin | 0.90C+0.02 | 0.12$\bar{X}$+0.19 | 0.16$\bar{X}$+0.16 |
| Endosulfan I | 0.97C+0.04 | 0.10$\bar{X}$+0.07 | 0.18$\bar{X}$+0.08 |
| Endosulfan II | 0.93C+0.34 | 0.41$\bar{X}$+0.65 | 0.47$\bar{X}$−0.20 |
| Endosulfan Sulfate | 0.89C−0.37 | 0.13$\bar{X}$+0.33 | 0.24$\bar{X}$+0.35 |
| Endrin | 0.89C−0.04 | 0.20$\bar{X}$+0.25 | 0.24$\bar{X}$+0.25 |
| Heptachlor | 0.69C+0.04 | 0.06$\bar{X}$+0.13 | 0.16$\bar{X}$+0.08 |
| Heptachlor epoxide | 0.89C+0.10 | 0.18$\bar{X}$−0.11 | 0.25$\bar{X}$−0.08 |
| Toxaphene | 0.80C+1.74 | 0.09$\bar{X}$+3.20 | 0.20$\bar{X}$+0.22 |
| PCB–1016 | 0.81C+0.50 | 0.13$\bar{X}$+0.15 | 0.15$\bar{X}$+0.45 |
| PCB–1221 | 0.96C+0.65 | 0.29$\bar{X}$−0.76 | 0.35$\bar{X}$−0.62 |
| PCB–1232 | 0.91C+10.79 | 0.21$\bar{X}$−1.93 | 0.31$\bar{X}$+3.50 |
| PCB–1242 | 0.93C+0.70 | 0.11$\bar{X}$+1.40 | 0.21$\bar{X}$+1.52 |
| PCB–1248 | 0.97C+1.06 | 0.17$\bar{X}$+0.41 | 0.25$\bar{X}$−0.37 |
| PCB–1254 | 0.76C+2.07 | 0.15$\bar{X}$+1.66 | 0.17$\bar{X}$+3.62 |
| PCB–1260 | 0.66C+3.76 | 0.22$\bar{X}$−2.37 | 0.39$\bar{X}$−4.86 |

$\bar{X}'$ = Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.
$s_r'$ = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
$S'$ = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
C = True value for the concentration, in µg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in µg/L.

BILLING CODE 6560–50–M

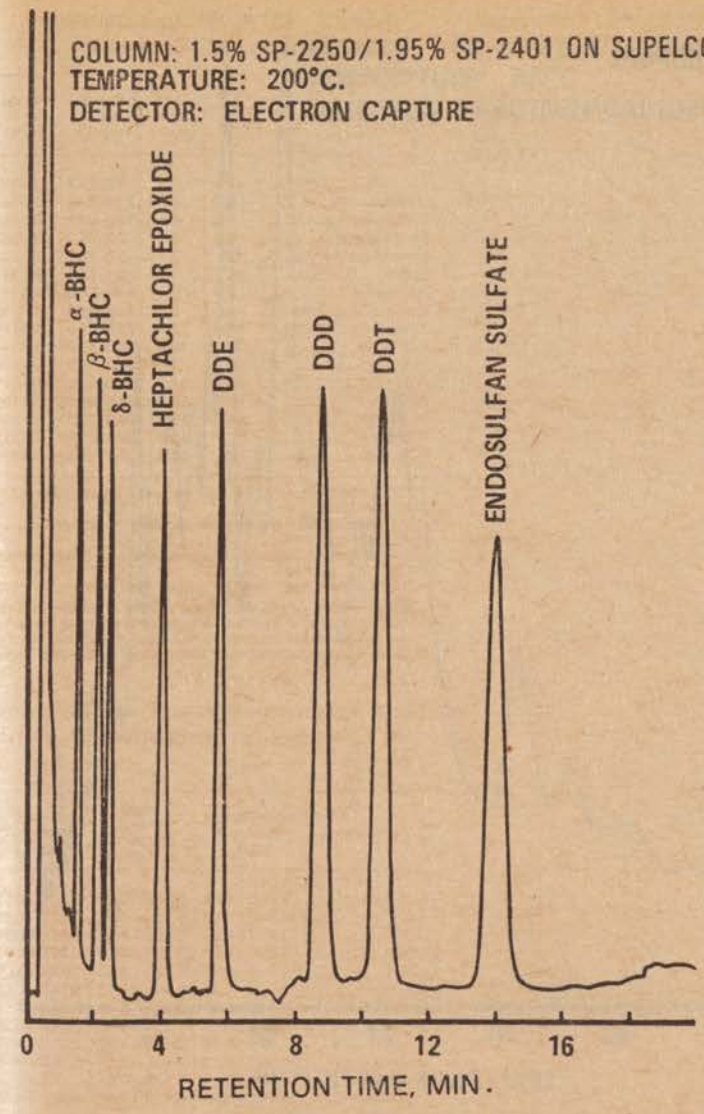



**Figure 1. Gas chromatogram of pesticides.**

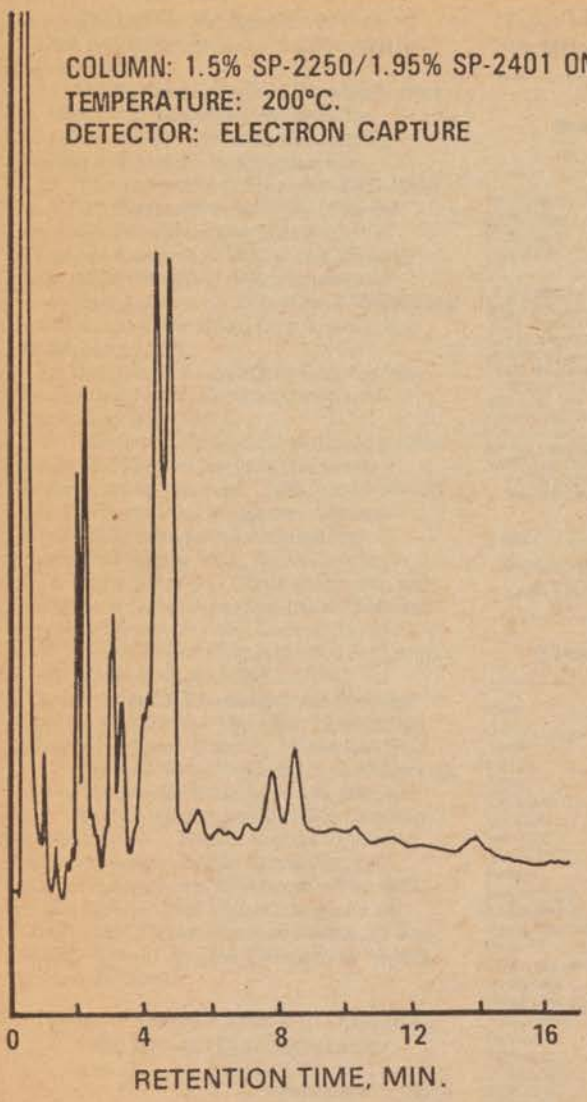



Figure 2. Gas chromatogram of chlordane.

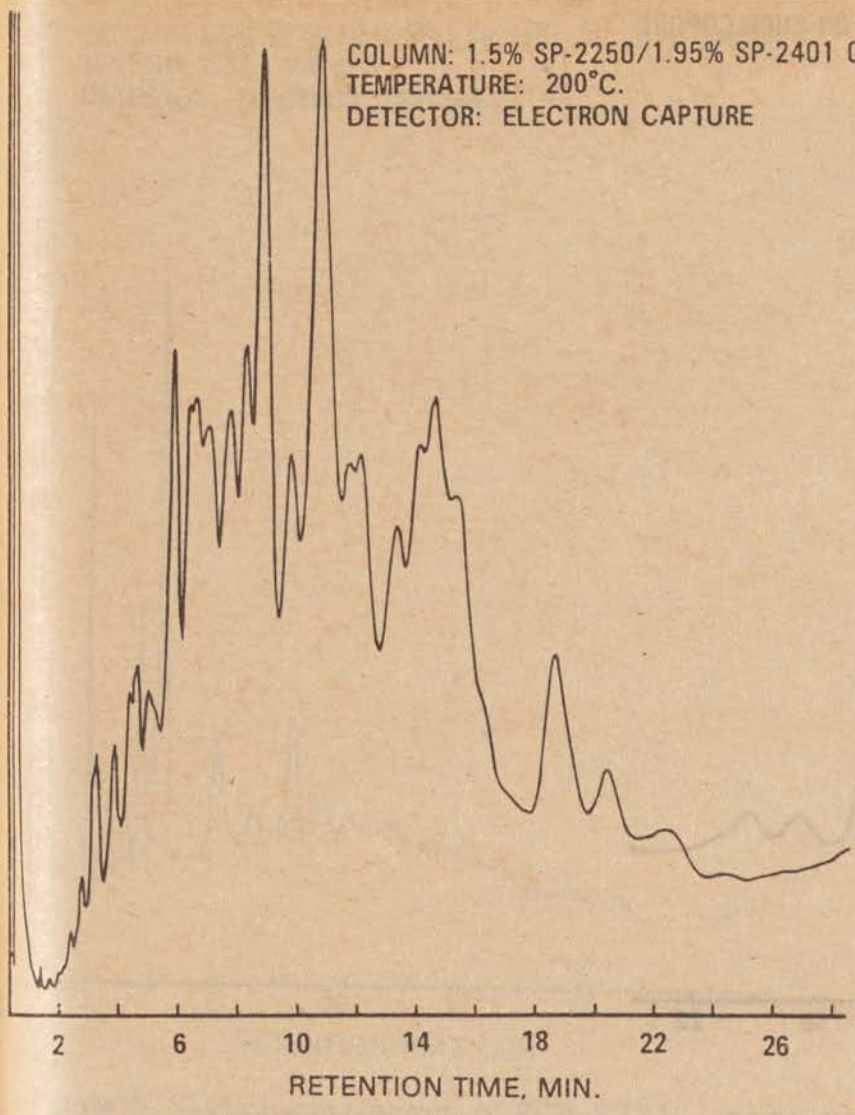



Figure 3. Gas chromatogram of toxaphene.

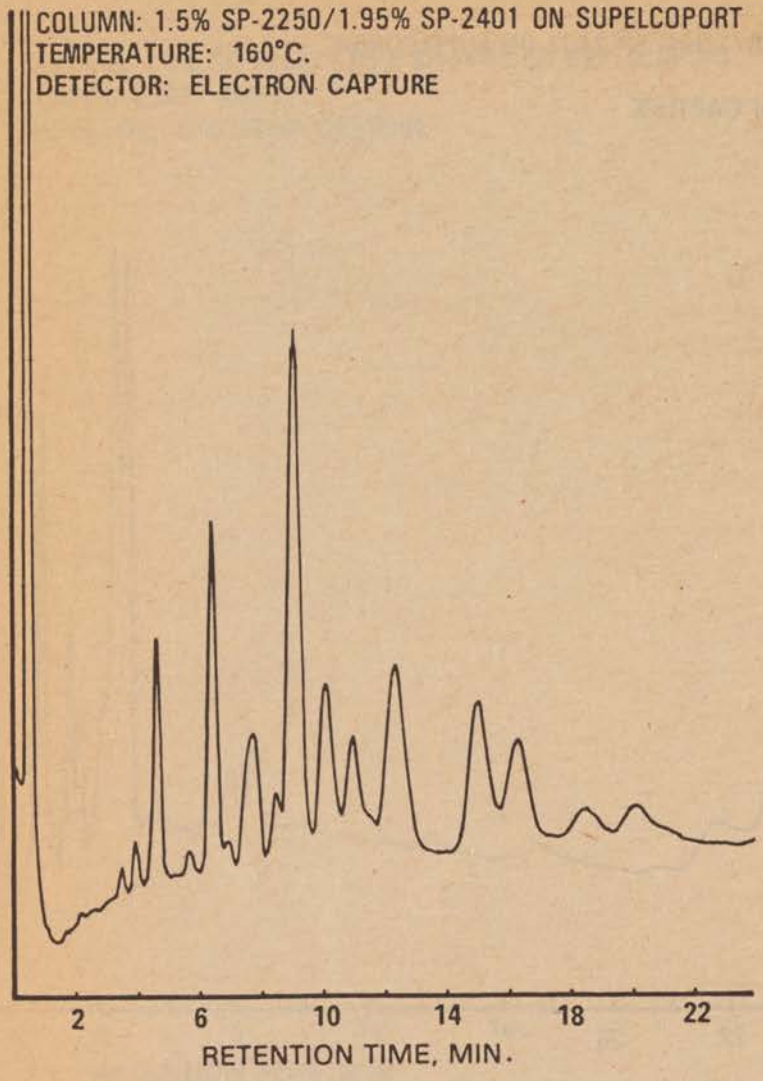



**Figure 4. Gas chromatogram of PCB-1016.**

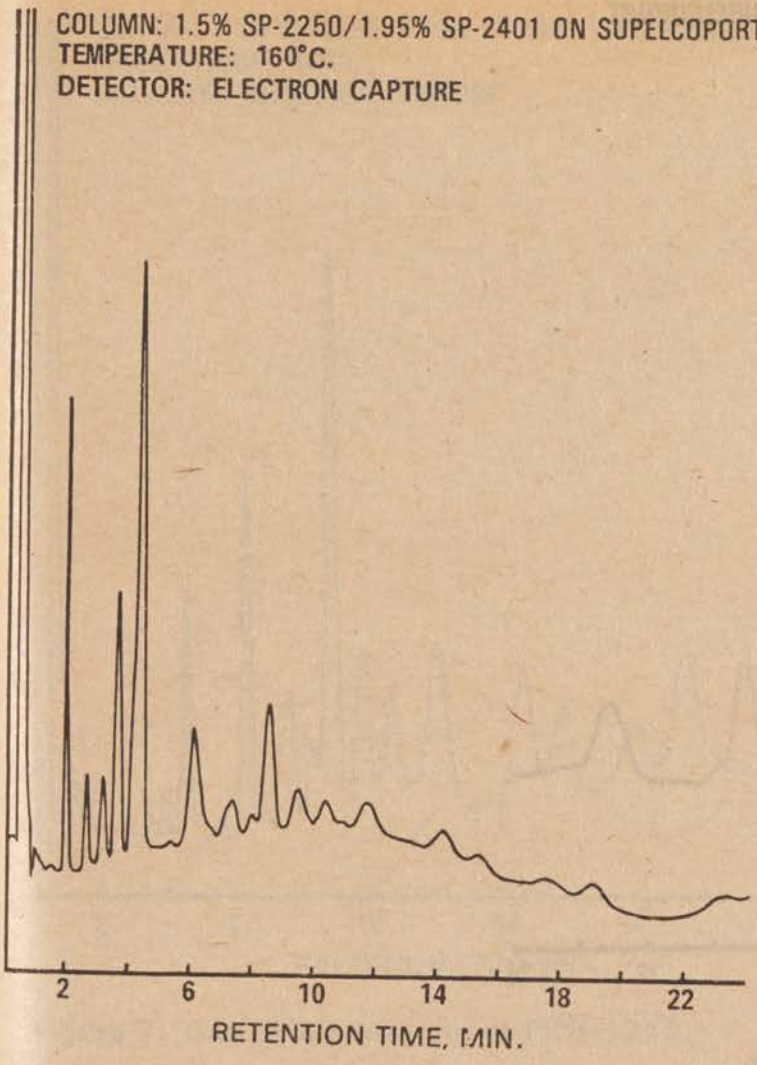



Figure 5. Gas chromatogram of PCB-1221.

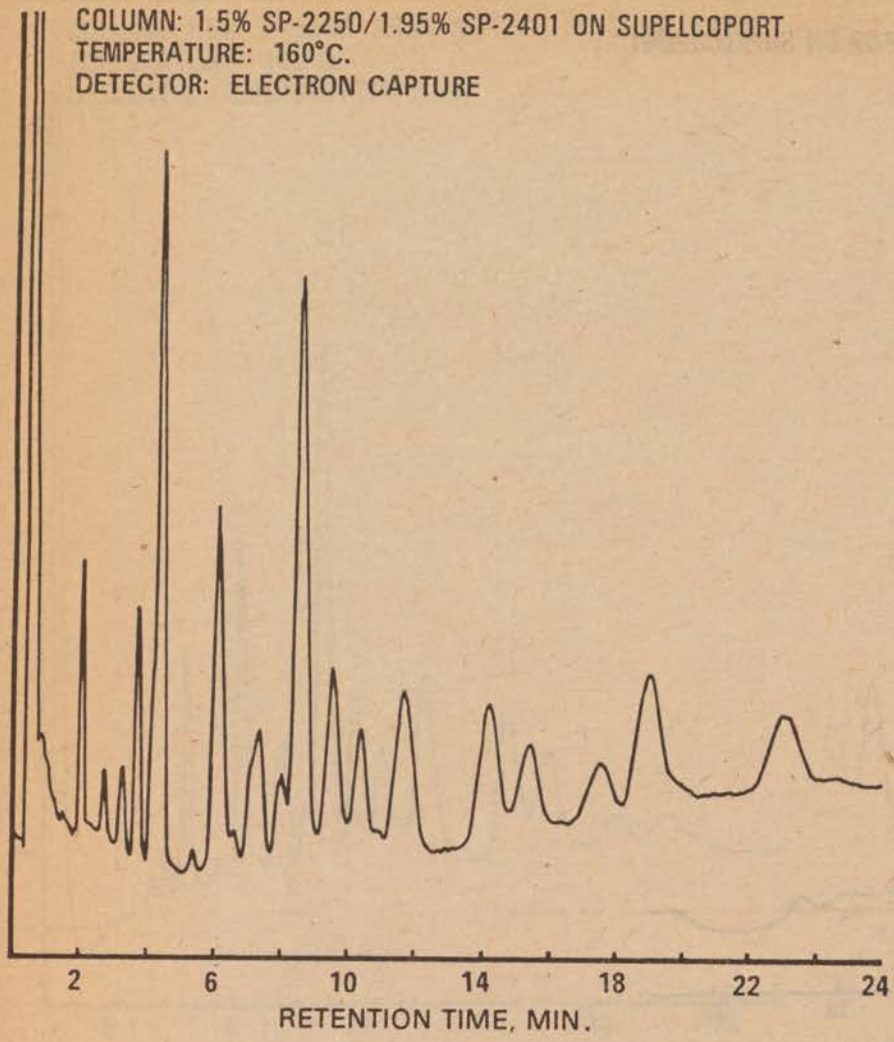



Figure 6. Gas chromatogram of PCB-1232.

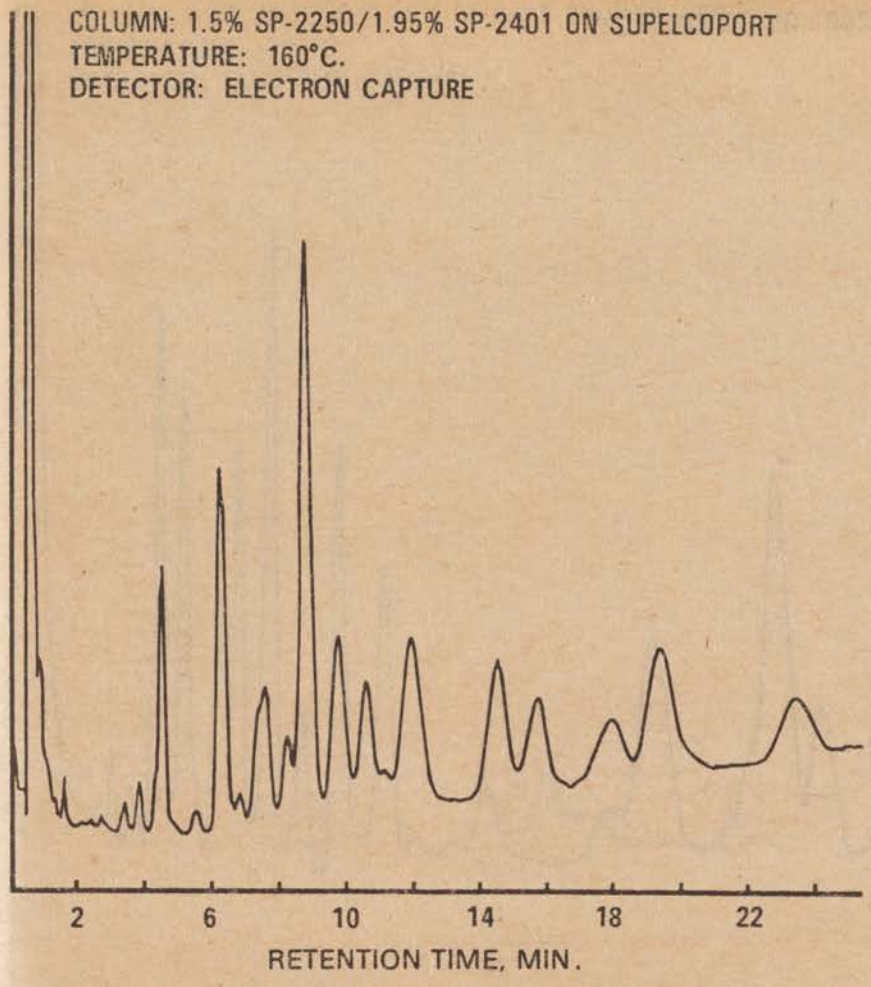



Figure 7. Gas chromatogram of PCB-1242.

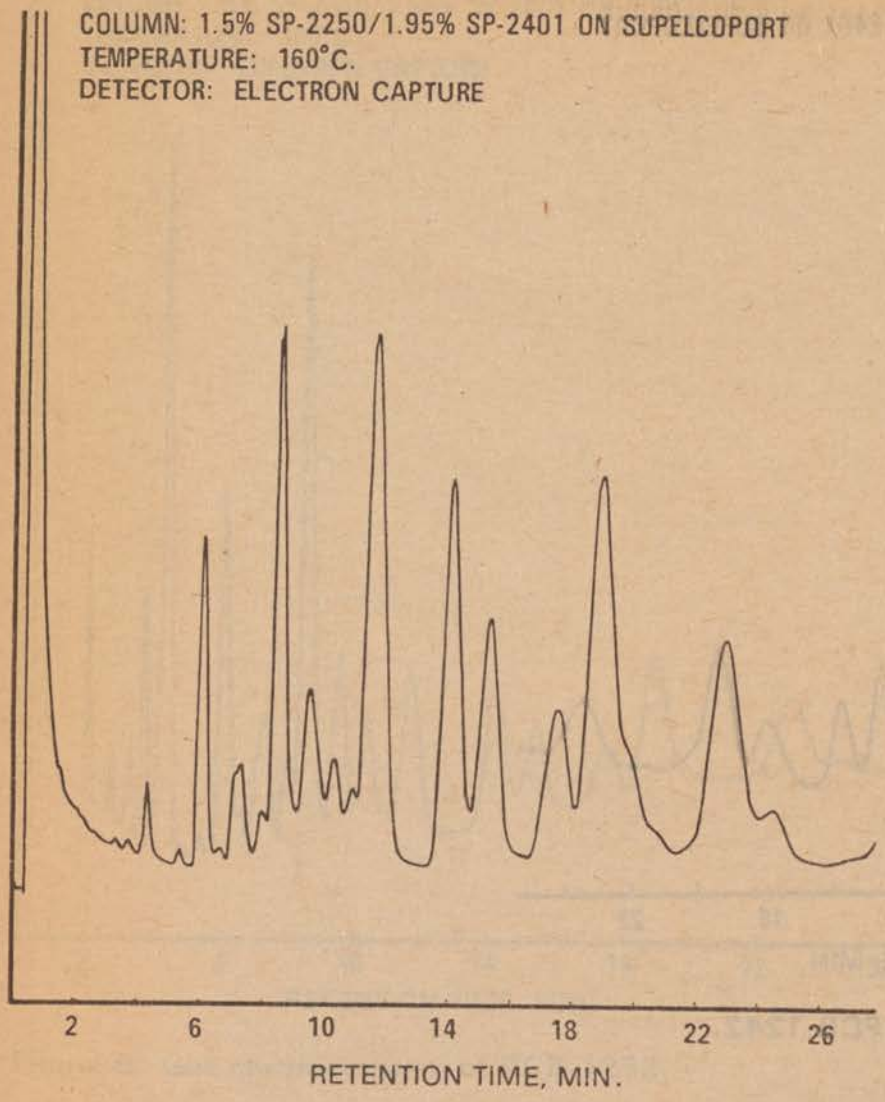



Figure 8. Gas chromatogram of PCB-1248.

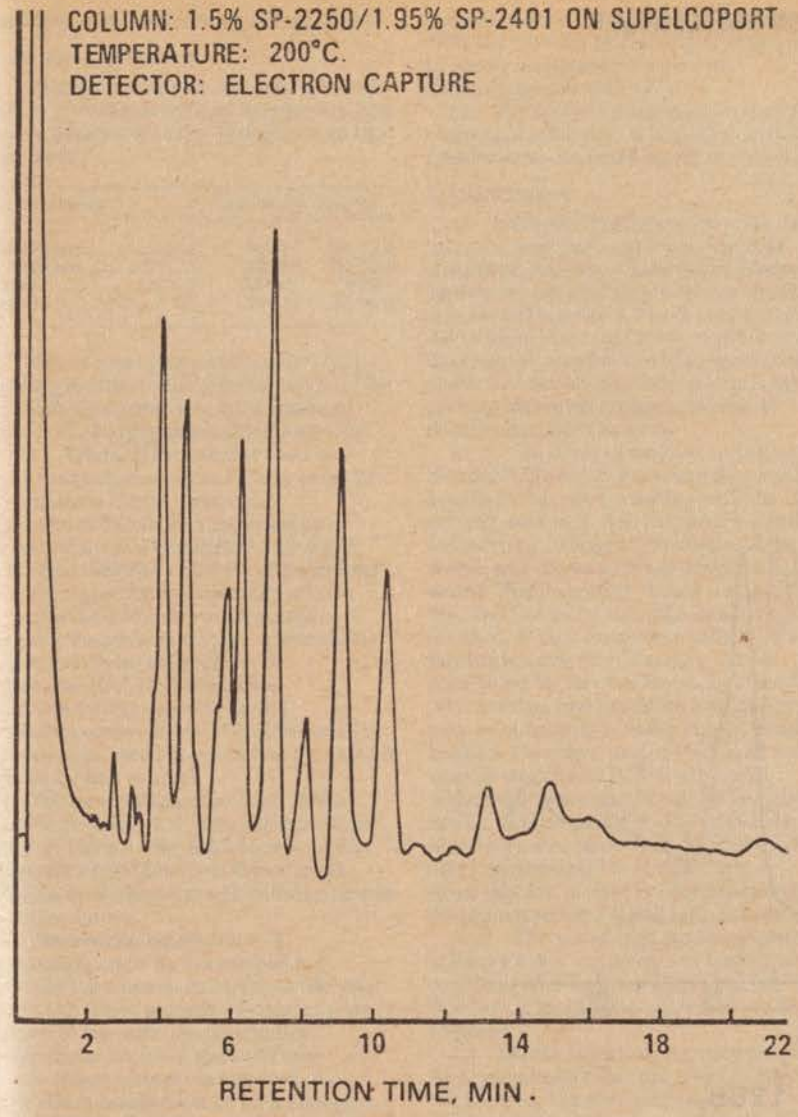



Figure 9. Gas chromatogram of PCB-1254.

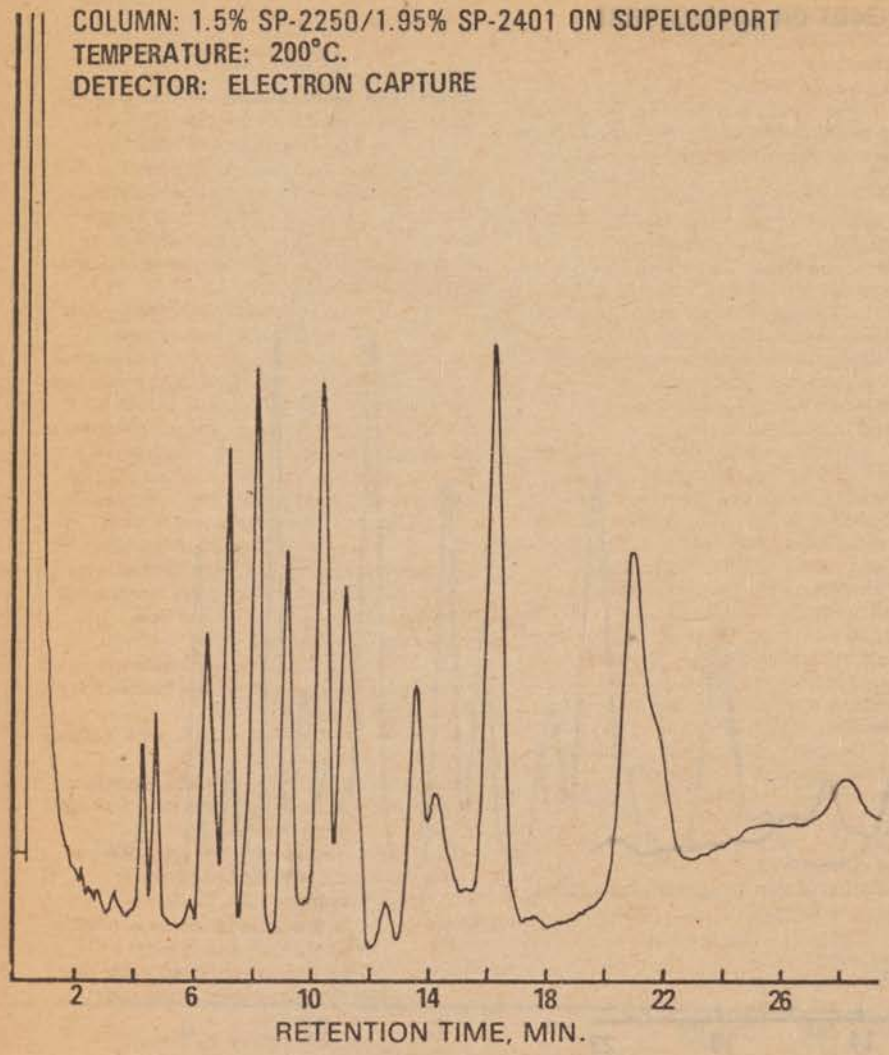



Figure 10. Gas chromatogram of PCB-1260.



BILLING CODE 6560-50-C

## Method 609—Nitroaromatics and Isophorone

*1. Scope and Application*

1.1 This method covers the determination of certain nitroaromatics and isophorone. The following parameters may be determined by this method:

| Parameter | Storet No. | Cas No. |
|---|---|---|
| 2,4-Dinitrotoluene | 34611 | 121-14-2 |
| 2,6-Dinitrotoluene | 34626 | 606-20-2 |
| Isophorone | 34408 | 78-59-1 |
| Nitrobenzene | 34447 | 98-95-3 |

1.2 This is a gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 The sample extraction and concentration steps in this method are essentially the same as in Methods 606, 608, 611, and 612. Thus, a single sample may be extracted to measure the parameters included in the scope of each of these methods. When cleanup is required, the concentration levels must be high enough to permit selecting aliquots, as necessary, to apply appropriate cleanup procedures. The analyst is allowed the latitude, under Section 12, to select chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.5 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 A measured volume of sample, approximately 1-L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to hexane during concentration to a volume of 10 mL or less. Isophorone and nitrobenzene are measured by flame ionization detector gas chromatography (FIDGC). The dinitrotoluenes are measured by electron capture detector gas chromatography (ECDGC).[2]

2.2 The method provides a Florisil column cleanup procedure to aid in the elimination of interferences that may be encountered.

*3. Interferences*

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedure in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

*4. Safety*

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [4-6] for the information of the analyst.

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1-L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2-L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long x 19 mm ID, with coarse frit filter disc.

5.2.3 Chromatographic column—100 mm long x 10 mm ID, with Teflon stopcock.

5.2.4 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.5 Evaporative flask, Kuderna-Danish—500-mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

5.2.6 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K-503000-0121 or equivalent).

5.2.7 Snyder column, Kuderna-Danish—Two-ball micro (Kontes K-569001-0219 or equivalent).

5.2.8 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (± 2 °C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column 1—1.2 m long x 2 or 4 mm ID glass, packed with 1.95% QF-1/1.5% OV-17 on Gas-Chrom Q (80/100 mesh) or equivalent. This column was used to develop the method performance statements given in Section 14.

Guidelines for the use of alternate column packings are provided in Section 12.1.

5.6.2 Column 2—3.0 m long x 2 or 4 mm ID glass, packed with 3% OV-101 on Gas-Chrom Q (80/100 mesh) or equivalent.

5.6.3 Detectors—Flame ionization and electron capture detectors. The flame ionization detector (FID) is used when determining isophorone and nitrobenzene. The electron capture detector (ECD) is used when determining the dinitrotoluenes. Both detectors have proven effective in the analysis of wastewaters and were used in develop the method performance statements in Section 14. Guidelines for the use to alternate detectors are provided in Section 12.1.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.3 Sulfuric acid (1+1)—Slowly, add 50 mL of $H_2SO_4$ (ACS, sp. gr. 1.84) to 50 mL of reagent water.

6.4 Acetone, hexane, methanol, methylene chloride—Pesticide quality or equivalent.

6.5 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray.

6.6 Florisil—PR grade (60/100 mesh). Purchase activated at 1250 °F and store in dark in glass containers with ground glass stoppers or foil-lined screw caps. Before use, activate each batch at least 16 h at 200 °C in a foil-covered glass container and allow to cool.

6.7 Stock standard solutions (1.00 μg/μL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.7.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in hexane and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.7.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.8 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Establish gas chromatographic operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with hexane. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 μL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (< 10% relative standard deviation, RSD) linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flash. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with hexane. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 μL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard (μg/L).

$C_s$ = Concentration of the parameter to be measured (μg/L).

If the RF value over the working range is a constant (< 10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ± 15%, a new calibration curve must be prepared for that compound.

7.5 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.4, 11.1, and 12.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest in acetone at a concentration of 20 μg/mL for each dinitrotoluene and 100 μg/mL for isophorone and nitrobenzene. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at the test concentrations shown in Table 2 by adding 1.00 mL of QC check sample concentrate to each of four 1-L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter. Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at the test concentration in Section 8.2.2 or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or the test concentration in Section 8.2.2.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100 (A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than the test concentration in Section 8.2.2, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X') using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 3, substituting X' for $\bar{X}$8; (3) calculate the range for recovery at the spike concentration as (100 X'/T) ± 2.44 (100 S'/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4. If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

Note.—The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}O=90\%$ and $s_p$ = 10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

*9. Sample Collection, Preservation, and Handling.*

9.1 Grab samples must be collected in glass containers. Conventional sampling practices [8] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C from the time of collection until extraction.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

*10. Sample Extraction*

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2-L separatory funnel. Check the pH of the sample with wide-range pH paper and adjust to within the range of 5 to 9 with sodium hydroxide solution or sulfuric acid.

10.2 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the

emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Sections 10.7 and 10.8 describe a procedure for exchanging the methylene chloride solvent to hexane while concentrating the extract volume to 1.0 mL. When it is not necessary to achieve the MDL in Table 2, the solvent exchange may be made by the addition of 50 mL of hexane and concentration to 10 mL as described in Method 606, Sections 10.7 and 10.8.

10.7 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.8 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of methylene chloride. A 5-mL syringe is recommended for this operation. Add 1 to 2 mL of hexane and a clean boiling chip to the concentrator tube and attach a two-ball micro-Snyder column. Prewet the column by adding about 0.5 mL of hexane to the top. Place the micro-K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.9 Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a minimum amount of hexane. Adjust the extract volume to 1.0 mL. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with gas chromatographic analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.10 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

*11. Cleanup and Separation*

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use the procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure.

11.2 Florisil column cleanup:

11.2.1 Prepare a slurry of 10 g of activated Florisil in methylene chloride/hexane (1+9)(V/V) and place the Florisil into a chromatographic column. Tap the column to settle the Florisil and add 1 cm of anhydrous sodium sulfate to the top. Adjust the elution rate to about 2 mL/min.

11.2.2 Just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the sample extract onto the column using an additional 2 mL of hexane to complete the transfer. Just prior to exposure of the sodium sulfate layer to the air, add 30 mL of methylene chloride/hexane (1 + 9)(V/V) and continue the elution of the column. Discard the eluate.

11.2.3 Next, elute the column with 30 mL of acetone/methylene chloride (1 + 9)(V/V) into a 500-mL K–D flask equipped with a 10-mL concentrator tube. Concentrate the collected fraction as in Sections 10.6, 10.7, 10.8, and 10.9 including the solvent exchange to 1 mL of hexane. This fraction should contain the nitroaromatics and isophorone. Analyze by gas chromatography (Section 12).

*12. Gas Chromatography*

12.1 Isophorone and nitrobenzene are analyzed by injection of a portion of the extract into an FIDGC. The dinitrotoluenes are analyzed by a separate injection into an ECDGC. Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by Column 1 are shown in Figures 1 and 2. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

12.2 Calibrate the system daily as described in Section 7.

12.3 If the internal standard calibration procedure is being used, the internal standard must be added to the same extract and mixed thoroughly immediately before injection into the gas chromatograph.

12.4 Inject 2 to 5 μL of the sample extract or standard into the gas chromatograph using the solvent-flush technique.[9] Smaller (1.0 μL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 μL, the total extract volume, and the resulting peak size in area or peak height units.

12.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.6 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

12.7 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

*13. Calculations*

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in section 7.2.2. The concentration in the sample can be calculated from Equation 2.

Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

where:

$A$ = Amount of material injected (ng).
$V_i$ = Volume of extract injected (μL).
$V_t$ = Volume of total extract (μL).
$V_s$ = Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the samle using the response factor (RF) determined in Section 7.3.2 and Equation 3.

Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.
$I_s$ = Amount of internal standard added to each extract (μg).
$V_o$ = Volume of water extracted (L).

13.2 Report results in μg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

*14. Method Performance*

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[10] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 7×MDL to 1000×MDL.[10]

14.3 This method was tested by 18 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 1.0 to 515 μg/L.[11] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

References

1. 40 CFR Part 136, Appendix B.
2. "Determination of Nitroaromatics and Isophorone in Industrial and Municipal Wastewaters," EPA-600/4-82-024, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, June 1982.
3. ASTM Annual Book of Standards, Part 31, D3694-78, "Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents," American Society for Testing and Materials, Philadelphia.
4. "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, August 1977.
5. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).
6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.
7. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58-63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)
8. ASTM Annual Book of Standards, Part 31, D3370-76. "Standard Practices for Sampling Water," American Society for Testing and Materials, Philadelphia.
9. Burke, J.A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).
10. "Determination of Method Detection Limit and Analytical Curve for EPA Method 609—Nitroaromatics and Isophorone," Special letter report for EPA Contract 68-03-2624, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.
11. "EPA Method Validation Study 19, Method 609 (Nitroaromatics and Isophorone)," Report for EPA Contract 68-03-2624 (In preparation).

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

| Parameter | Retention time (min) | | Method detection limit (μg/L) | |
|---|---|---|---|---|
| | Col. 1 | Col. 2 | ECDGC | FIDGC |
| Nitrobenzene | 3.31 | 4.31 | 13.7 | 3.6 |
| 2,6-Dinitrotoluene | 3.52 | 4.75 | 0.01 | — |
| Isophorone | 4.49 | 5.72 | 15.7 | 5.7 |
| 2,4-Dinitrotoluene | 5.35 | 6.54 | 0.02 | — |

Column 1 conditions: Gas-Chrom Q (80/100 mesh) coated with 1.95% OF-1/1.5% OV-17 packed in a 1.2 m long × 2 mm or 4 mm ID glass column. A 2 mm ID column and nitrogen carrier gas at 44 mL/min flow rate were used when determining isophorone and nitrobenzene by FIDGC. The column temperature was held isothermal at 85 °C. A 4 mm ID column and 10% methane/90% argon carrier gas at 44 mL/min flow rate were used when determining the dinitrotoluenes by ECDGC. The column temperature was held isothermal at 145 °C.

Column 2 conditions: Gas-Chrom Q (80/100 mesh) coated with 3% OV-101 packed in a 3.0 m long × 2 mm or 4 mm ID glass column. A 2 mm ID column and nitrogen carrier gas at 44 mL/min flow rate were used when determining isophorone and nitrobenzene by FIDGC. The column temperature was held isothermal at 100 °C. A 4 mm ID column and 10% methane/90% argon carrier gas at 44 mL/min flow rate were used when determining the dinitrotoluenes by ECDGC. The column temperature was held isothermal at 150 °C.

TABLE 2.—QC ACCEPTANCE CRITERIA—METHOD 609

| Parameter | Test Conc. (μg/L) | Limit for s (μg/L) | Range for $\bar{X}$ (μg/L) | Range for P, $P_s$ (%) |
|---|---|---|---|---|
| 2,4-Dinitrotoluene | 20 | 5.1 | 3.6-22.8 | 6-125 |
| 2,6-Dinitrotoluene | 20 | 4.8 | 3.8-23.0 | 8-126 |
| Isophorone | 100 | 32.3 | 8.0-100.0 | D-117 |
| Nitrobenzene | 100 | 33.3 | 25.7-100.0 | 6-118 |

s = Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
P, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.

**Note.**—These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 609

| Parameter | Accuracy, as recovery, X' (μg/L) | Single analyst precision, $s_r'$ (μg/L) | Overall precision, S' (μg/L) |
|---|---|---|---|
| 2,4-Dinitrotoluene | 0.65C+0.22 | 0.20$\bar{X}$+0.08 | 0.37$\bar{X}$−0.07 |
| 2,6-Dinitrotoluene | 0.66C+0.20 | 0.19$\bar{X}$+0.06 | 0.36$\bar{X}$−0.00 |
| Isophorone | 0.49C+2.93 | 0.28$\bar{X}$+2.77 | 0.46$\bar{X}$+0.31 |
| Nitrobenzene | 0.60C+2.00 | 0.25$\bar{X}$+2.53 | 0.37$\bar{X}$−0.78 |

X' = Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
$s_r'$ = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C = True value for the concentration, in μg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in μg/L.

BILLING CODE 6560-50-M

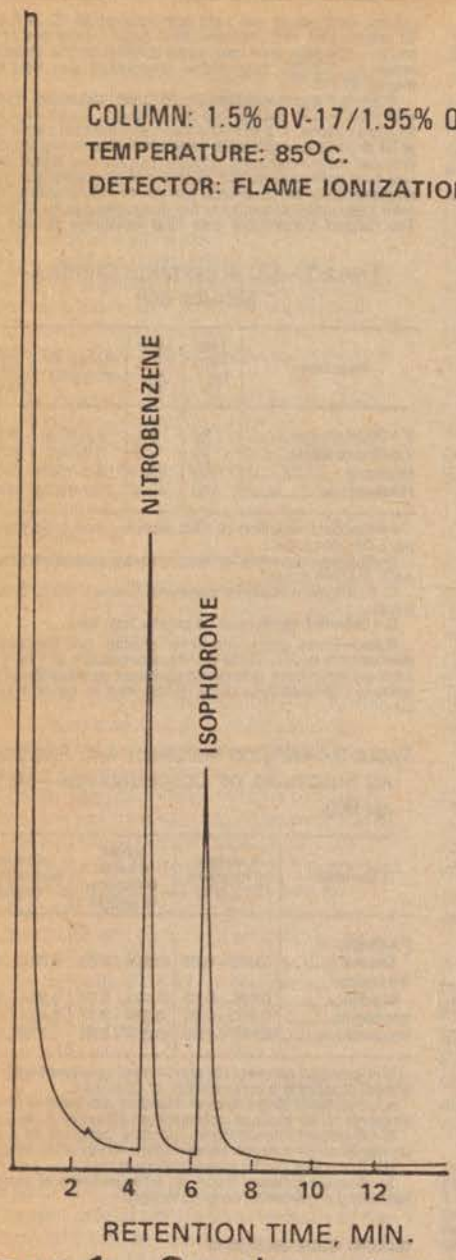



**Figure 1. Gas chromatogram of nitrobenzene and isophorone.**

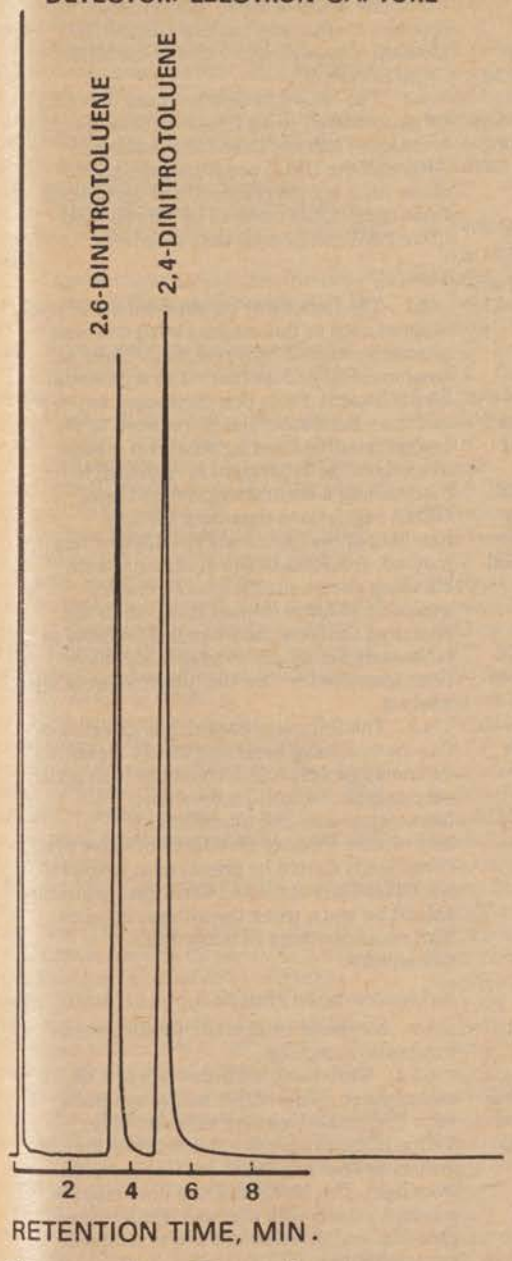



**Figure 2. Gas chromatogram of dinitrotoluenes.**

BILLING CODE 6560-50-C

## Method 610—Polynuclear Aromatic Hydrocarbons

*1. Scope and Application*

1.1 This method covers the determination of certain polynuclear aromatic hydrocarbons (PAH). The following parameters can be determined by this method:

| Parameter | Storet No. | CAS No. |
|---|---|---|
| Acenaphthene | 34205 | 83-32-9 |
| Acenaphthylene | 34200 | 208-96-8 |
| Anthracene | 34220 | 120-12-7 |
| Benzo(a)anthracene | 34526 | 56-55-3 |
| Benzo(a)pyrene | 34247 | 50-32-8 |
| Benzo(b)fluoranthene | 34230 | 205-99-2 |
| Benzo(ghi)perylene | 34521 | 191-24-2 |
| Benzo(k)fluoranthene | 34242 | 207-08-9 |
| Chrysene | 34320 | 218-01-9 |
| Dibenzo(a,h)anthracene | 34556 | 53-70-3 |
| Fluoranthene | 34376 | 206-44-0 |
| Fluorene | 34381 | 86-73-7 |
| Indeno(1,2,3-cd)pyrene | 34403 | 193-39-5 |
| Naphthalene | 34696 | 91-20-3 |
| Phenanthrene | 34461 | 85-01-8 |
| Pyrene | 34469 | 129-00-0 |

1.2 This is a chromatographic method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for many of the parameters listed above, using the extract produced by this method.

1.3 This method provides for both high performance liquid chromatographic (HPLC) and gas chromatographic (GC) approaches for the determination of PAHs. The gas chromatographic procedure does not adequately resolve the following four pairs of compounds: anthracene and phenanthrene; chrysene and benzo(a)anthracene; benzo(b)fluoranthene and benzo(k)fluoranthene; and dibenzo(a,h) anthracene and indeno (1,2,3-cd)pyrene. Unless the purpose for the analysis can be served by reporting the sum of an unresolved pair, the liquid chromatographic approach must be used for these compounds. The liquid chromatographic method does resolve all 16 of the PAHs listed.

1.4 The method detection limit (MDL, defined in Section 15.1) [1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.5 The sample extraction and concentration steps in this method are essentially the same as in Methods 606, 608, 609, 611, and 612. Thus, a single sample may be extracted to measure the parameters included in the scope of each of these methods. When cleanup is required, the concentration levels must be high enough to permit selecting aliquots, as necessary, to apply appropriate cleanup procedures. Selection of the aliquots must be made prior to the solvent exchange steps of this method. The analyst is allowed the latitude, under Sections 12 and 13, to select chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.6 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.7 This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC and GC systems and in the interpretation of liquid and gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 A measured volume of sample, approximately 1–L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and concentrated to a volume of 10 mL or less. The extract is then separated by HPLC or GC. Ultraviolet (UV) and fluorescence detectors are used with HPLC to identify and measure the PAHs. A flame ionization detector is used with GC.[2]

2.2 The method provides a silica gel column cleanup procedure to aid in the elimination of interferences that may be encountered.

*3. Interferences*

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardward that lead to discrete artifacts and/or elevated baselines in the chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedure in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in table 1.

3.3 The extent of interferences that may be encountered using liquid chromatographic techniques has not been fully assessed. Although the HPLC conditions described allow for a unique resolution of the specific PAH compounds covered by this method, other PAH compounds may interfere.

*4. Safety*

4.1 The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [4–6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzo(a)pyrene, and dibenzo(a,h)-anthracene. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1-L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2-L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long × 19 mm ID, with coarse frit filter disc.

5.2.3 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K–570050–1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.4 Evaporative flask, Kuderna-Danish—500-mL (Kontes K–570001–0500 or equivalent). Attach to concentrator tube with springs.

5.2.5 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K–503000–0121 or equivalent).

5.2.6 Snyder column, Kuderna-Danish—Two-ball micro (Kontes K–569001–0219 or equivalent).

5.2.7 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.2.8 Chromatographic column—250 mm long x 10 mm ID, with coarse frit filter disc at bottom and Teflon stopcock.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2 °C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 High performance liquid chromatograph (HPLC)—An analytical system complete with column supplies, high pressure syringes, detectors, and compatible strip-chart recorder. A data system is recommended for measuring peak areas and retention times.

5.6.1 Gradient pumping system—Constant flow.

5.6.2 Reverse phase column—HC-ODS Sil-X, 5 micron particle diameter, in a 25 cm x 2.6 mm ID stainless steel column (Perkin Elmer No. 089–0716 or equivalent). This column was used to develop the method performance statements in Section 15. Guidelines for the use of alternate column packings are provided in Section 12.2.

5.6.3 Detectors—Fluorescence and/or UV detectors. The fluorescence detector is used for excitation at 280 nm and emission greater than 389 nm cutoff (Corning 3–75 or equivalent). Fluorometers should have dispersive optics for excitation and can utilize either filter or dispersive optics at the emission detector. The UV detector is used at 254 nm and should be coupled to the fluorescence detector. These detectors were used to develop the method performance statements in Section 15. Guidelines for the use of alternate detectors are provided in Section 12.2.

5.7 Gas chromatograph—An analytical system complete with temperature programmable gas chromatograph suitable for on-column or splitless injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.7.1 Column—1.8 m long x 2 mm ID glass, packed with 3% OV–17 on Chromosorb W-AW-DCMS (100/120 mesh) or equivalent. This column was used to develop the retention time data in Table 2. Guidelines for the use of alternate column packings are provided in Section 13.3.

5.7.2 Detector—Flame ionization detector. This detector has proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), excluding the four pairs of unresolved compounds listed in Section 1.3. Guidelines for the use of alternate detectors are provided in Section 13.3.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Cyclohexane, methanol, acetone, methylene chloride, pentane—Pesticide quality or equivalent.

6.4 Acetonitrile—HPLC quality, distilled in glass.

6.5 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray.

6.6 Silica gel—100/200 mesh, desiccant, Davison, grade-923 or equivalent. Before use, activate for at least 16 h at 130 °C in a shallow glass tray, loosely covered with foil.

6.7 Stock standard solutions (1.00 μg/μL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.7.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in acetonitrile and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.7.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.8 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Establish liquid or gas chromatographic operating conditions equivalent to those given in Table 1 or 2. The chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with acetonitrile. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 5 to 25 μL for HPLC and 2 to 5 μL for GC, analyze each calibration standard according to Section 12 or 13, as appropriate. Tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with acetonitrile. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 5 to 25 μL for HPLC and 2 to 5 μL for GC, analyze each calibration standard according to Section 12 or 13, as appropriate. Tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard (μg/L).

$C_s$ = Concentration of the parameter to be measured (μg/L).

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

7.5 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagants.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.4, 11.1, 12.2, and 13.3) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at the following concentrations in acetonitrile: 100 μg/mL of any of the six early-eluting PAHs (naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, and anthracene); 5 μg/mL of benzo(k)fluoranthene; and 10 μg/mL of any of the other PAHs. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at the test concentrations shown in Table 3 by adding 1.00 mL of QC check sample concentrate to each of four 1-L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 3. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter.

**Note.**—The large number of parameters in Table 3 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.2.6.2 Beginning with Section 8.2.2, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at the test concentration in Section 8.2.2 or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none, (2) the larger of either 5 times higher than the expected background concentration or the test concentration in Section 8.2.2.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100 (A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 3. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than the test concentration in Section 8.2.2, the analyst must use either the QC acceptance criteria in Table 3, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) calculate accuracy (X') using the equation in Table 4, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 4, substituting X' for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as $(100\ X'/T) \pm 2.44(100\ S'/T)\%$.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the critiera must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

**Note.**—The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the

laboratory. If the entire list of parameters in Table 3 must be measured in the sample in Section 8.3, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spike sample.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 3. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

*9. Sample Collection, Preservation, and Handling*

9.1 Grab samples must be collected in glass containers. Conventional sampling practices [8] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C from the time of collection until extraction. PAHs are known to be light sensitive; therefore, samples, extracts, and standards should be stored in amber or foil-wrapped bottles in order to minimize photolytic decomposition. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[9] Field test kits are available for this purpose.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

*10. Sample Extraction*

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2-L separatory funnel.

10.2 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min. with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250–mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.7 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of methylene chloride. A 5-mL syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial and protected from light. If the sample extract requires no further cleanup, proceed with gas or liquid chromatographic analysis (Section 12 or 13). If the sample requires further cleanup, proceed to Section 11.

10.8 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

*11. Cleanup and Separation*

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use the procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the methods as revised to incorporate the cleanup procedure.

11.2 Before the silica gel cleanup technique can be utilized, the extract solvent must be exchanged to cyclohexane. Add 1 to 10 mL of the sample extract (in methylene chloride) and a boiling chip to a clean K–D concentrator tube. Add 4 mL of cyclohexane and attach a two-ball micro-Snyder column. Prewet the column by adding 0.5 mL of methylene chloride to the top. Place the micro-K–D apparatus on a boiling (100 °C) water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of the liquid reaches 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a minimum amount of cyclohexane. Adjust the extract volume to about 2 mL.

11.3 Silica gel column cleanup for PAHs:

11.3.1 Prepare a slurry of 10 g of activiated silica gel in methylene chloride and place this into a 10-mm ID chromatographic column. Tap the column to settle the silica gel and elute the methylene chloride. Add 1 to 2 cm of anhydrous sodium sulfate to the top of the silica gel.

11.3.2 Preelute the column with 40 mL of pentane. The rate for all elutions should be about 2 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, transfer the 2-mL cyclohexane sample extract onto the column using an additional 2 mL cyclohexane to complete the transfer. Just prior to exposure of the sodium

sulfate layer to the air, add 25 mL of pentane and continue the elution of the column. Discard this pentane eluate.

11.3.3 Next, elute the column with 25 mL of methylene chloride/pentane (4+6)(V/V) into a 500-mL K–D flask equipped with a 10-mL concentrator tube. Concentrate the collected fraction to less than 10 mL as in Section 10.6. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint with pentane. Proceed with HPLC or GC analysis.

*12. High Performance Liquid Chromatography*

12.1 To the extract in the concentrator tube, add 4 mL of acetonitrile and a new boiling chip, then attach a two-ball micro-Snyder column. Concentrate the solvent as in Section 10.6, except set the water bath at 95 to 100 °C. When the apparatus is cool, remove the micro-Snyder column and rinse its lower joint into the concentrator tube with about 0.2 mL of acetonitrile. Adjust the extract volume to 1.0 mL.

12.2 Table 1 summarizes the recommended operating conditions for the HPLC. Included in this table are retention times, capacity factors, and MDL that can be achieved under these conditions. The UV detector is recommended for the determination of naphthalene, acenaphthylene, acenapthene, and fluorene and the fluorescence detector is recommended for the remaining PAHs. Examples of the separations achieved by this HPLC column are shown in Figures 1 and 2. Other HPLC columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

12.3 Calibrate the system daily as described in Section 7.

12.4 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the instrument.

12.5 Inject 5 to 25 μL of the sample extract or standard into the HPLC using a high pressure syringe or a constant volume sample injection loop. Record the volume injected to the nearest 0.1 μL, and the resulting peak size in area or peak height units. Re-equilibrate the HPLC column at the initial gradient conditions for at least 10 min between injections.

12.6 Identify the parameters in the sample by comparing the retention time of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.7 If the response for a peak exceeds the working range of the system, dilute the extract with acetonitrile and reanalyze.

12.8 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

*13. Gas Chromatography*

13.1 The packed column GC procedure will not resolve certain isomeric pairs as indicated in Section 1.3 and Table 2. The liquid chromatographic procedure (Section 12) must be used for these parameters.

13.2 To achieve maximum sensitivity with this method, the extract must be concentrated to 1.0 mL. Add a clean boiling chip to the methylene chloride extract in the concentrator tube. Attach a two-ball micro-Snyder column. Prewet the micro-Snyder column by adding about 0.5 mL of methylene chloride to the top. Place the micro-K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a minimum amount of methylene chloride. Adjust the final volume to 1.0 mL and stopper the concentrator tube.

13.3 Table 2 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times that were obtained under these conditions. An example of the separations achieved by this column is shown in Figure 3. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

13.4 Calibrate the gas chromatographic system daily as described in Section 7.

13.5 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the gas chromatograph.

13.6 Inject 2 to 5 μL of the sample extract or standard into the gas chromatograph using the solvent-flush technique.[10] Smaller (1.0 μL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 μL, and the resulting peak size in area or peak height units.

13.7 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

13.8 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

13.9 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

*14. Calculations*

14.1 Determine the concentration of individual compounds in the sample.

14.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

where:

$A$ = Amount of material injected (ng).
$V_i$ = Volume of extract injected (μL).
$V_t$ = Volume of total extract (μL).
$V_s$ = Volume of water extracted (mL).

14.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2. and Equation 3.

Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

$A_s$ = Response for the parameter to be measured.
$A_{is}$ = Response for the internal standard.
$I_s$ = Amount of internal standard added to each extract (μg).
$V_o$ = Volume of water extracted (L).

14.2 Report results in μg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

*15. Method Performance*

15.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[11] Similar results were achieved using representative wastewaters. MDL for the GC approach were not determined. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

15.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 8 × MDL to 800 × MDL[11] with the following exception: benzo(ghi)perylene recovery at 80 × and 800 × MDL were low (35% and 45%, respectively).

15.3 This method was tested by 16 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations

over the range 0.1 to 425 μg/L.[12] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 4.

### References


1. 40 CFR Part 136, Appendix B.
2. "Determination of Polynuclear Aromatic Hydrocarbons in Industrial and Municipal Wastewaters," EPA-600/4-82-025, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, September 1982.
3. ASTM Annual Book of Standards, Part 31, D3694-78. "Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents," American Society for Testing and Materials, Philadelphia.
4. "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, August 1977.
5. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).
6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.
7. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58-63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)
8. ASTM Annual Book of Standards, Part 31, D3370-76. "Standard Practices for Sampling Water," American Society for Testing and Materials, Philadelphia.
9. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.
10. Burke, J.A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).
11. Cole, T., Riggin, R., and Glaser, J. "Evaluation of Method Detection Limits and Analytical Curve for EPA Method 610—PNAs," International Symposium on Polynuclear Aromatic Hydrocarbons, 5th, Battelle's Columbus Laboratories, Columbus, Ohio (1980).
12. "EPA Method Validation Study 20, Method 610 (Polynuclear Aromatic Hydrocarbons)," Report for EPA Contract 68-03-2624 (In preparation).


TABLE 1.—HIGH PERFORMANCE LIQUID CHROMATOGRAPHY CONDITIONS AND METHOD DETECTION LIMITS

| Parameter | Retention time (min) | Column capacity factor (k') | Method detection limit (μg/L)* |
|---|---|---|---|
| Naphthalene | 16.6 | 12.2 | 1.8 |
| Acenaphthylene | 18.5 | 13.7 | 2.3 |
| Acenaphthene | 20.5 | 15.2 | 1.8 |
| Fluorene | 21.2 | 15.8 | 0.21 |
| Phenanthrene | 22.1 | 16.6 | 0.64 |
| Anthracene | 23.4 | 17.6 | 0.66 |
| Fluoranthene | 24.5 | 18.5 | 0.21 |
| Pyrene | 25.4 | 19.1 | 0.27 |
| Benzo(a)anthracene | 28.5 | 21.6 | 0.013 |
| Chrysene | 29.3 | 22.2 | 0.15 |
| Benzo(b)fluoranthene | 31.6 | 24.0 | 0.018 |
| Benzo(k)fluoranthene | 32.9 | 25.1 | 0.017 |
| Benzo(a)pyrene | 33.9 | 25.9 | 0.023 |
| Dibenzo(a,h)anthracene | 35.7 | 27.4 | 0.030 |
| Benzo(ghi)perylene | 36.3 | 27.8 | 0.076 |
| Indeno(1,2,3-cd)pyrene | 37.4 | 28.7 | 0.043 |

HPLC column conditions: Reverse phase HC-ODS Sil-X, 5 micron particle size, in a 25 cm × 2.6 mm ID stainless steel column. Isocratic elution for 5 min using acetonitrile/water (4+6), then linear gradient elution to 100% acetonitrile over 25 min at 0.5 mL/min flow rate. If columns having other internal diameters are used, the flow rate should be adjusted to maintain a linear velocity of 2 mm/sec.

*The MDL for naphthalene, acenaphthylene, acenaphthene, and fluorene were determined using a UV detector. All others were determined using a fluorescence detector.

TABLE 2.—GAS CHROMATOGRAPHIC CONDITIONS AND RETENTION TIMES

| Parameter | Retention time (min) |
|---|---|
| Naphthalene | 4.5 |
| Acenaphthylene | 10.4 |
| Acenaphthene | 10.8 |
| Fluorene | 12.6 |
| Phenanthrene | 15.9 |
| Anthracene | 15.9 |
| Fluoranthene | 19.8 |
| Pyrene | 20.6 |
| Benzo(a)anthracene | 24.7 |
| Chrysene | 24.7 |
| Benzo(b)fluoranthene | 28.0 |
| Benzo(k)fluoranthene | 28.0 |
| Benzo(a)pyrene | 29.4 |
| Dibenzo(a,h)anthracene | 36.2 |
| Indeno(1,2,3-cd)pyrene | 36.2 |
| Benzo(ghi)perylene | 38.6 |

GC Column conditions: Chromosorb W-AW-DCMS (100/120 mesh) coated with 3% OV-17 packed in a 1.8 x 2 mm ID glass column with nitrogen carrier gas at 40 mL/min flow rate. Column temperature was held at 100 °C for 4 min, then programmed at 8 °C/min to a final hold at 280 °C.

TABLE 3.—QC ACCEPTANCE CRITERIA—METHOD 610

| Parameter | Test conc. (μg/L) | Limit for s (μg/L) | Range for $\bar{X}$ (μg/L) | Range for $P$, $P_s$ (%) |
|---|---|---|---|---|
| Acenaphthene | 100 | 40.3 | D-105.7 | D-124 |
| Acenaphthylene | 100 | 45.1 | 22.1-112.1 | D-139 |
| Anthracene | 100 | 28.7 | 11.2-112.3 | D-126 |
| Benzo(a)anthracene | 10 | 4.0 | 3.1-11.6 | 12-135 |
| Benzo(a)pyrene | 10 | 4.0 | 0.2-11.0 | D-128 |
| Benzo(b)fluoranthene | 10 | 3.1 | 1.8-13.8 | 6-150 |
| Benzo(ghi)perylene | 10 | 2.3 | D-10.7 | D-116 |
| Benzo(k)fluoranthene | 5 | 2.5 | D-7.0 | D-159 |
| Chrysene | 10 | 4.2 | D-17.5 | D-199 |
| Dibenzo(a,h)anthracene | 10 | 2.0 | 0.3-10.0 | D-110 |
| Fluoranthene | 10 | 3.0 | 2.7-11.1 | 14-123 |
| Fluorene | 100 | 43.0 | D-119 | D-142 |
| Indeno(1,2,3-cd)pyrene | 10 | 3.0 | 1.2-10.0 | D-116 |
| Naphthalene | 100 | 40.7 | 21.5-100.0 | D-122 |
| Phenanthrene | 100 | 37.7 | 8.4-133.7 | D-155 |
| Pyrene | 10 | 3.4 | 1.4-12.1 | D-140 |

s = Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
$P$, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.

NOTE.—These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

TABLE 4.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 610

| Parameter | Accuracy, as recovery, $X'$ (μg/L) | Single analyst precision, $s_r'$ (μg/L) | Overall precision, $S'$ (μg/L) |
|---|---|---|---|
| Acenaphthene | $0.52C + 0.54$ | $0.39\bar{X} + 0.76$ | $0.53\bar{X} + 1.32$ |
| Acenaphthylene | $0.69C - 1.89$ | $0.36\bar{X} + 0.29$ | $0.42\bar{X} + 0.52$ |
| Anthracene | $0.63C - 1.26$ | $0.23\bar{X} + 1.16$ | $0.41\bar{X} + 0.45$ |
| Benzo(a)anthracene | $0.73C + 0.05$ | $0.28\bar{X} + 0.04$ | $0.34\bar{X} + 0.02$ |
| Benzo(a)pyrene | $0.56C + 0.01$ | $0.38\bar{X} - 0.01$ | $0.53\bar{X} - 0.01$ |
| Benzo(b)fluoranthene | $0.78C + 0.01$ | $0.21\bar{X} + 0.01$ | $0.38\bar{X} - 0.00$ |
| Benzo(ghi)perylene | $0.44C + 0.30$ | $0.25\bar{X} + 0.04$ | $0.58\bar{X} + 0.10$ |
| Benzo(k)fluoranthene | $0.59C + 0.00$ | $0.44\bar{X} - 0.00$ | $0.69\bar{X} + 0.01$ |
| Chrysene | $0.77C - 0.18$ | $0.32\bar{X} - 0.18$ | $0.66\bar{X} - 0.22$ |
| Dibenzo(a,h)anthracene | $0.41C + 0.11$ | $0.24\bar{X} + 0.02$ | $0.45\bar{X} + 0.03$ |
| Fluoranthene | $0.68C + 0.07$ | $0.22\bar{X} + 0.06$ | $0.32\bar{X} + 0.03$ |
| Fluorene | $0.56C - 0.52$ | $0.44\bar{X} - 1.12$ | $0.63\bar{X} - 0.65$ |
| Indeno(1,2,3-cd)pyrene | $0.54C + 0.06$ | $0.29\bar{X} + 0.02$ | $0.42\bar{X} + 0.01$ |
| Naphthalene | $0.57C - 0.70$ | $0.39\bar{X} - 0.18$ | $0.41\bar{X} - 0.74$ |
| Phenanthrene | $0.72C - 0.95$ | $0.29\bar{X} + 0.05$ | $0.47\bar{X} - 0.25$ |
| Pyrene | $0.69C - 0.12$ | $0.25\bar{X} + 0.14$ | $0.42\bar{X} - 0.00$ |

$X'$ = Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
$s_r'$ = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
$S'$ = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C = True value for the concentration, in μg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in μg/L.

BILLING CODE 6560-50-M

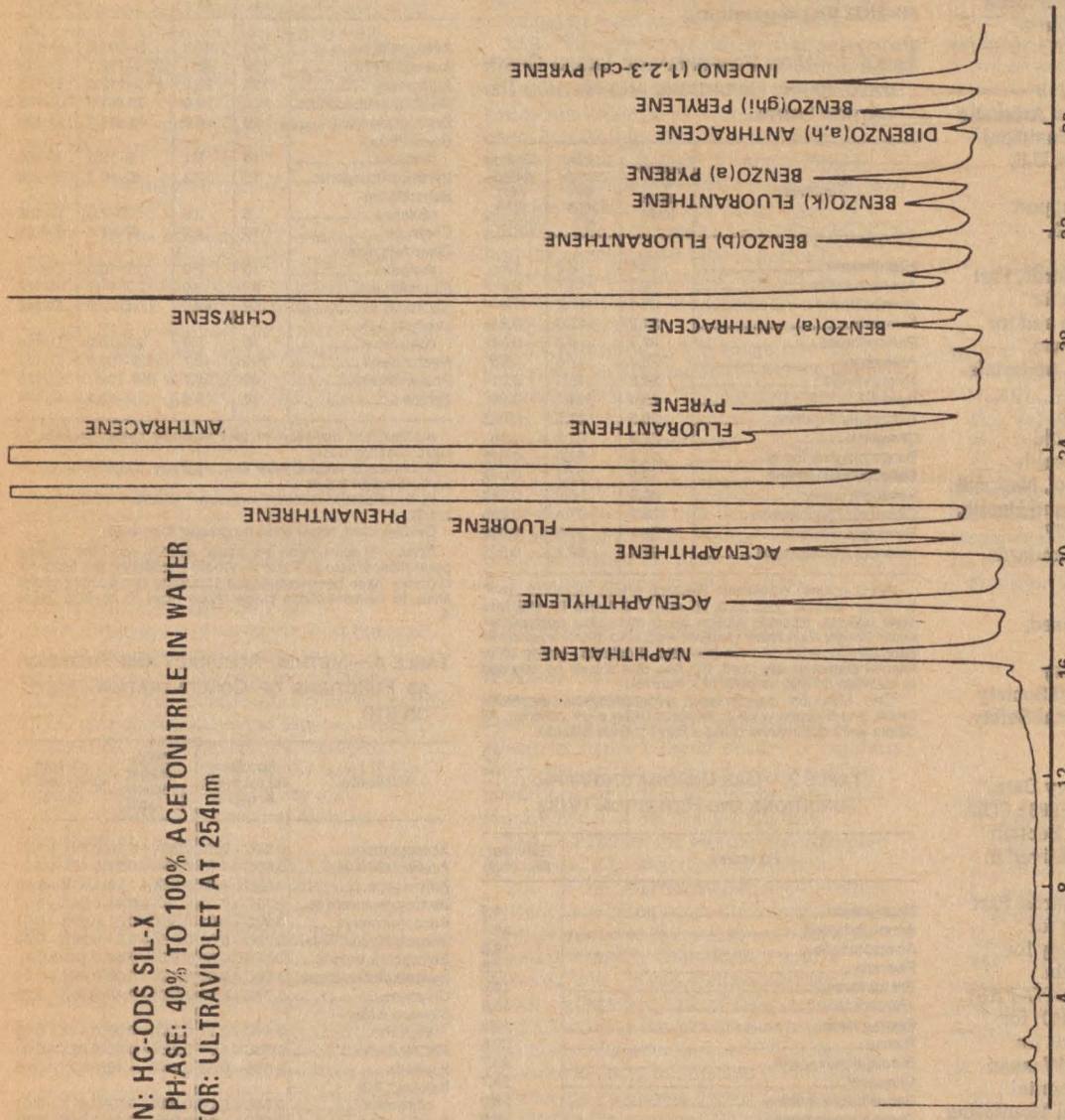



Figure 1. Liquid chromatogram of polynuclear aromatic hydrocarbons.

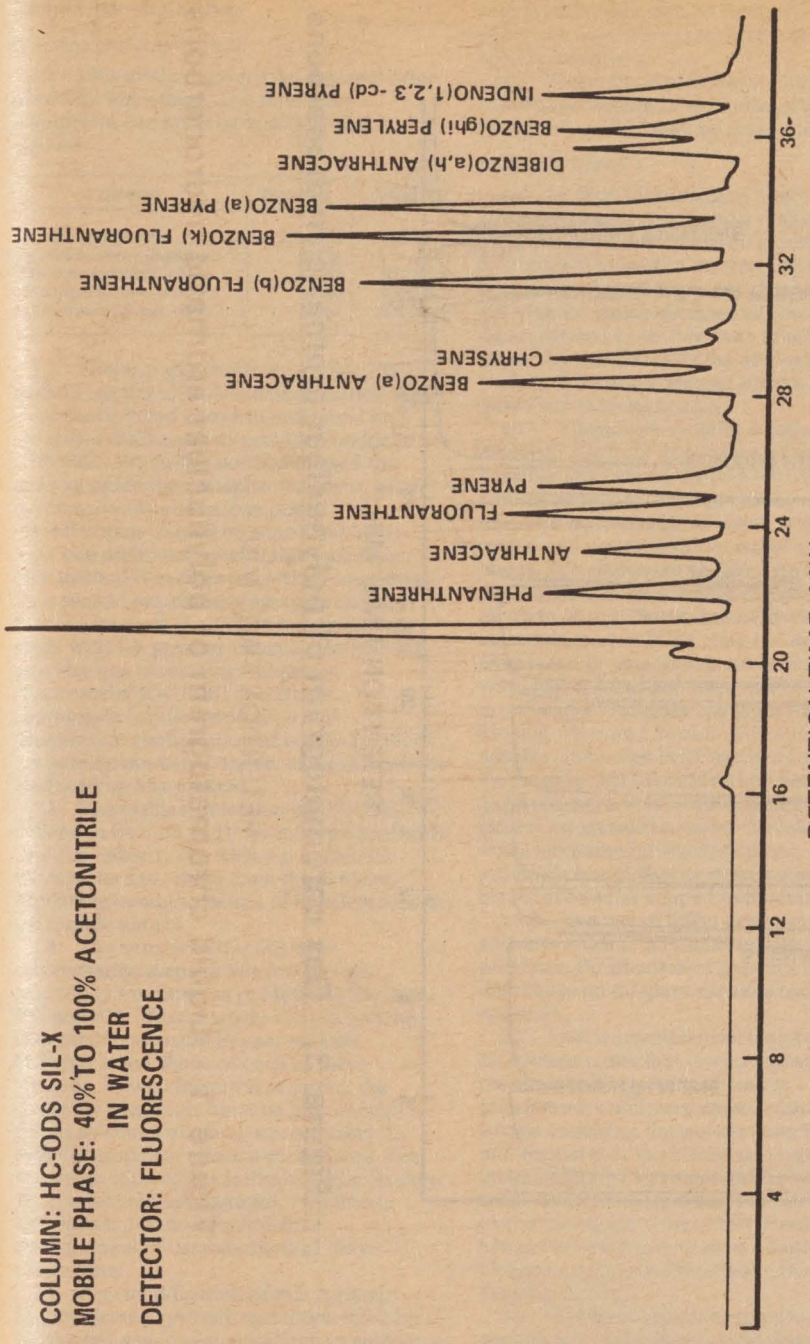



Figure 2. Liquid chromatogram of polynuclear aromatic hydrocarbons.

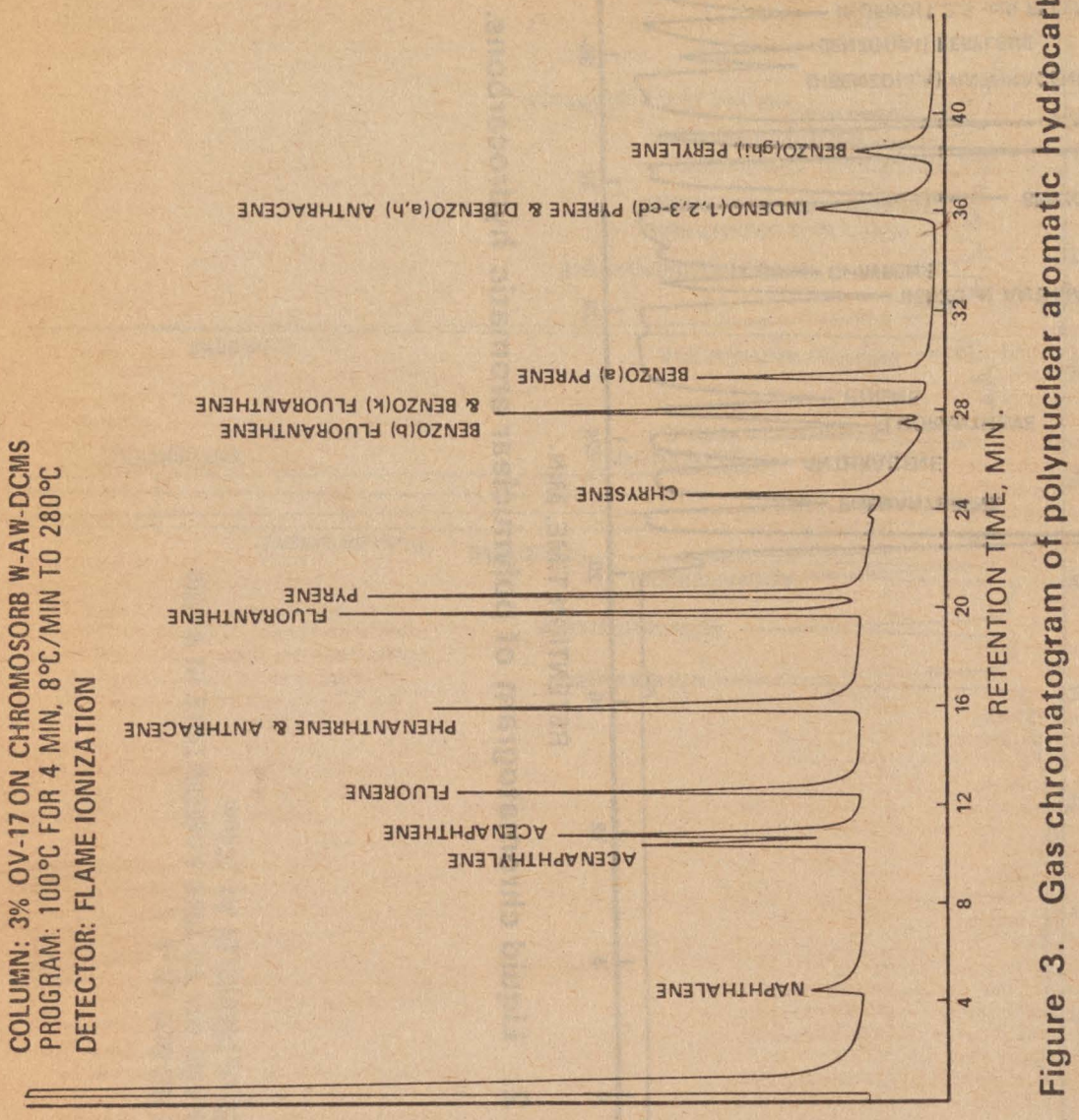



Figure 3. Gas chromatogram of polynuclear aromatic hydrocarbons.

BILLING CODE 6560-50-C

**Method 611—Haloethers**

*1. Scope and Application*

1.1 This method covers the determination of certain haloethers. The following parameters can be determined by this method:

| Parameter | Storet No. | CAS No. |
|---|---|---|
| Bis(2-chloroethyl) ether | 34273 | 111-44-4 |
| Bis(2-chloroethoxy) methane | 34278 | 111-91-1 |
| Bis(2-chloroisopropyl) ether | 34283 | 108-60-1 |
| 4-Bromophenyl phenyl ether | 34636 | 101-55-3 |
| 4-Chlorophenyl phenyl either | 34641 | 7005-72-3 |

1.2 This is a gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 The sample extraction and concentration steps in this method are essentially the same as in Methods 606, 608, 609, and 612. Thus, a single sample may be extracted to measure the parameters included in the scope of each of these methods. When cleanup is required, the concentration levels must be high enough to permit selecting aliquots, as necessary, to apply appropriate cleanup procedures. The analyst is allowed the latitude, under Section 12, to select chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.5 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 A measured volume of sample, approximately 1-L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to hexane during concentration to a volume of 10 mL or less. The extract is separated by gas chromatography and the parameters are then measured with a halide specific detector.[2]

2.2 The method provides a Florisil column cleanup procedure to aid in the elimination of interferences that may be encountered.

*3. Interferences*

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed be detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such a PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedure in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

3.3 Dichlorobenzenes are known to coelute with haloethers under some gas chromatographic conditions. If these materials are present together in a sample, it may be necessary to analyze the extract with two different column packings to completely resolve all of the compounds.

*4. Safety*

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [4-6] for the information of the analyst.

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1-L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2-L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long x 19 mm ID, with coarse frit filter disc.

5.2.3 Chromatographic column—400 mm long x 19 mm ID, with Teflon stopcock and coarse frit filter disc at bottom (Kontes K-420540-0224 or equivalent).

5.2.4 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.5 Evaporative flask, Kuderna-Danish—500-mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

5.2.6 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K-503000-0121 or equivalent).

5.2.7 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2°C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical

columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column 1—1.8 m long x 2 mm ID glass, packed with 3% SP-1000 on Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 12.1.

5.6.2 Column 2—1.8 m long x 2 mm ID glass, packed with 2,6-diphenylene oxide polymer (60/80 mesh), Tenax, or equivalent.

5.6.3 Detector—Halide specific detector: electrolytic conductivity or microcoulometric. These detectors have proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1). The Hall conductivity detector was used to develop the method performance statements in Section 14. Guidelines for the use of alternate detectors are provided in Section 12.1. Although less selective, an electron capture detector is an acceptable alternative.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Acetone, hexane, methanol, methylene chloride, petroleum ether (boiling range 30–60 °C)—Pesticide quality or equivalent.

6.4 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray.

6.5 Florisil—PR Grade (60/100 mesh). Purchase activated at 1250 °F and store in the dark in glass containers with ground glass stoppers or foil-lined screw caps. Before use, activate each batch at least 16 h at 130 °C in a foil-covered glass container and allow to cool.

6.6 Ethyl ether—Nanograde, redistilled in glass if necessary.

6.6.1 Ethyl ether must be shown to be free of peroxides before it is used as indicated by EM Laboratories Quant test strips. (Available from Scientific Products Co., Cat. No. P1126-8, and other suppliers.)

6.6.2 Procedures recommended for removal of peroxides are provided with the test strips. After cleanup, 20 mL of ethyl alcohol preservative must be added to each liter of ether.

6.7 Stock standard solutions (1.00 µg/µL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.7.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in acetone and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.7.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.8 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Establish gas chromatographic operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with hexane. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with hexane. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard (µg/L).

$C_s$ = Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (< 10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, a new calibration curve must be prepared for that compound.

7.5 The cleanup procedure in Section 11 utilizes Florisil column chromatography. Florisil from different batches or sources may vary in adsorptive capacity. To standardize the amount of Florisil which is used, the use of lauric acid value [7] is suggested. The referenced procedure determines the adsorption from hexane solution of lauric acid (mg) per g of Florisil. The amount of Florisil to be used for each column is calculated by dividing 110 by this ratio and multiplying by 20 g.

7.6. Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.4, 11.1, and 12.1) to improve the separations or lower the cost of

measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 100 μg/mL in acetone. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at a concentration of 100 μg/L by adding 1.00 mL of QC check sample concentrate to each of four 1-L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter. Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1. The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 100 μg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or 100 μg/L.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[8] If spiking was performed at a concentration lower than 100 μg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) calculate accuracy (X') using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 3, substituting X' for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X'/T)±2.44(100 S'/T)%.[8]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

**NOTE.**—The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

*9. Sample Collection, Preservation, and Handling*

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[9] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4°C from the time of collection until extraction. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[10] Field test kits are available for this purpose.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

*10. Sample Extraction*

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2-L separatory funnel.

10.2 Add 60 mL methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

**Note.**—Some of the haloethers are very volatile and significant losses will occur in concentration steps if care is not exercised. It is important to maintain a constant gentle evaporation rate and not to allow the liquid volume to fall below 1 to 2 mL before removing the K–D apparatus from the hot water bath.

10.7 Momentarily remove the Snyder column, add 50 mL of hexane and a new boiling chip, and reattach the Snyder column. Raise the temperature of the water bath to 85 to 90 °C. Concentrate the extract as in Section 10.6, except use hexane to prewet the column. The elapsed time of concentration should be 5 to 10 min.

10.8 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of hexane. A 5-mL syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with gas chromatographic analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.9 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

*11. Cleanup and Separation*

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use the procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure.

11.2 Florisil column cleanup for haloethers:

11.2.1 Adjust the sample extract volume to 10 mL.

11.2.2. Place a weight of Florisil (nominally 20 g) predetermined by calibration (Section 7.5), into a chromatographic column. Tap the column to settle the Florisil and add 1 to 2 cm of anhydrous sodium sulfate to the top.

11.2.3 Preelute the column with 50 to 60 mL of petroleum ether. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the sample extract onto the column by decantation and subsequent petroleum ether washings. Discard the eluate. Just prior to exposure of the sodium sulfate layer to the air, begin eluting the column with 300 mL of ethyl ether/petroleum ether (6+94) (V/V). Adjust the elution rate to approximately 5 mL/min and collect the eluate in a 500-mL K–D flask equipped with a 10-mL concentrator tube. This fraction should contain all of the haloethers.

11.2.4 Concentrate the fraction as in Section 10.6, except use hexane to prewet the column. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with hexane. Adjust the volume of the cleaned up extract to 10 mL with hexane and analyze by gas chromatography (Section 12).

*12. Gas Chromatography*

12.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by Columns 1 and 2 are shown in Figures 1 and 2, respectively. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

12.2 Calibrate the system daily as described in Section 7.

12.3 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the gas chromatrograph.

12.4 Inject 2 to 5 μL of the sample extract or standard into the gas chromatograph using the solvent-flush technique.[11] Smaller (1.0 μL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 μL, the total extract volume, and the resulting peak size in area or peak height units.

12.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weight heavily in the interpretation of chromatograms.

12.6 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

12.7 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

*13. Calculations*

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

Equation 2.

$$\text{Concentration, } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

where:

$A$ = Amount of material injected (ng).
$V_i$ = Volume of extract injected (μL).
$V_t$ = Volume of total extract (μL).
$V_s$ = Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$I_s$ = Amount of internal standard added to each extract ($\mu g$).

$V_o$ = Volume of water extracted (L).

13.2 Report results in $\mu g/L$ without correction for recovery data. All QC data obtained should be reported with the sample results.

*14. Method Performance*

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[12] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 4 × MDL to 1000 × MDL.[12]

14.3 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 1.0 to 626 $\mu$/L.[12] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

**References**


1. 40 CFR Part 136, Appendix B.

2. "Determination of Haloethers in Industrial and Municipal Wastewaters," EPA–600/4–81–062, U.S. Environmental Protection Agency , Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, December 1981.

3. ASTM Annual Book of Standards, Part 31, D3694–78. "Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constitutents," American Society for Testing and Materials, Philadelphia.

4. "Carcinogens—Working Carcinogens," Department of Health, Education, and Welfare, Public Health Services, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Mills., P.A. "Variation of Florisil Activity: Simple Method for Measuring Absorbent Capacity and Its Use in Standardizing Florisil Columns," *Journal of the Association of Official Analytical Chemists, 51,* 29 (1968).

8. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

9. ASTM Annual Book of Standards, Part 31, D3370–76. "Standard Practices for Sampling Water," American Society for Testing and Materials, Philadelphia.

10. "Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

11. Burke, J.A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

12. "EPA Method Validation Study 21, Method 611 (Haloethers)," Report for EPA Contract 68–03–2633 (In preparation).


TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHODS DETECTION LIMITS

| Parameters | Retention time (min) | | Method detection limit ($\mu$/L) |
|---|---|---|---|
| | Column 1 | Column 2 | |
| Bis (2-chloroisopropyl) ether | 8.4 | 9.7 | 0.8 |
| Bis (2-chloroethyl) ether | 9.3 | 9.1 | 0.3 |
| Bis (2-chloroethoxy) methane | 13.1 | 10.0 | 0.5 |
| 4-Chlorophenyl ether | 19.4 | 15.0 | 3.9 |
| 4-Bromophenyl phenyl ether | 21.2 | 16.2 | 2.3 |

Column 1 conditions: Supelcoport (100/120 mesh) coated with 3% SP–1000 packed in a 1.8 m long x 2 mm ID glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 60 °C for 2 min after injection then programmed at 8 ° C/min to 230 °C and held for 4 min. Under these conditions the retention time for Aldrin is 22.6 min.

Column 2 conditions: Tenax–GC (60/80 mesh) packed in a 1.8 m long x 2mm ID glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 150 °C for 4 min after injection then programmed at 16 °C/min to 310 °C. Under these conditions the retention time for Aldrin is 18.4 min.

TABLE 2.—QC ACCEPTANCE CRITERIA—METHOD 611

| Parameter | Test conc. ($\mu$g/L) | Limit for s ($\mu$g/L) | Range for $\bar{X}$ ($\mu$g/L) | Range for P, $P_s$ percent |
|---|---|---|---|---|
| Bis(2-chloroethyl) ether | 100 | 26.3 | 26.3–136.8 | 11–152 |
| Bis(2-chloroethoxy) methane | 100 | 25.7 | 27.3–115.0 | 12–128 |
| Bis(2-chloroisopropyl) ether | 100 | 32.7 | 26.4–147.0 | 9–165 |
| 4–Bromophenyl phenyl ether | 100 | 39.3 | 7.6 –167.5 | D–189 |
| 4–Chlorophenyl phenyl ether | 100 | 30.7 | 15.4–152.5 | D–170 |

s = Standard deviation of four recovery measurements, in $\mu$g/L (Section 8.2.4).

$\bar{X}$ = Average recovery for four recovery measurements, in $\mu$g/L (Section 8.2.4).

P, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2).

D = Detected; result must be greater than zero.

**Note.**—These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 611

| Parameter | Accuracy, as recovery, X' ($\mu$g/L) | Single analyst precision, $s_r$' ($\mu$g/L) | Overall precision, S' ($\mu$g/L) |
|---|---|---|---|
| Bis(2-chloroethyl) ether | 0.81C+0.54 | 0.19$\bar{X}$+0.28 | 0.35$\bar{X}$+0.36 |
| Bis(2-chloroethoxy) methane | 0.71C+0.13 | 0.20$\bar{X}$+0.15 | 0.33$\bar{X}$+0.11 |
| Bis(2-chloroisopropyl) ether | 0.85C+1.67 | 0.20$\bar{X}$+1.05 | 0.36$\bar{X}$+0.79 |
| 4–Bromophenyl phenyl ether | 0.84C+2.55 | 0.25$\bar{X}$+0.21 | 0.47$\bar{X}$+0.37 |
| 4–Chlorophenyl phenyl ether | 0.82C+1.97 | 0.18$\bar{X}$+2.13 | 0.41$\bar{X}$+0.55 |

X' = Expected recovery for one or more measurements of a sample containing a concentration of C, in $\mu$g/L.

$s_r$' = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in $\mu$g/L.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in $\mu$g/L.

C = True value for the concentration, in $\mu$g/L.

$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in $\mu$g/L.

BILLING CODE 6560–50–M

COLUMN: 3% SP-1000 ON SUPELCOPORT
PROGRAM 60°C FOR 2 MIN, 8°C/MIN TO 230°C
DETECTOR: HALL ELECTROLYTIC CONDUCTIVITY

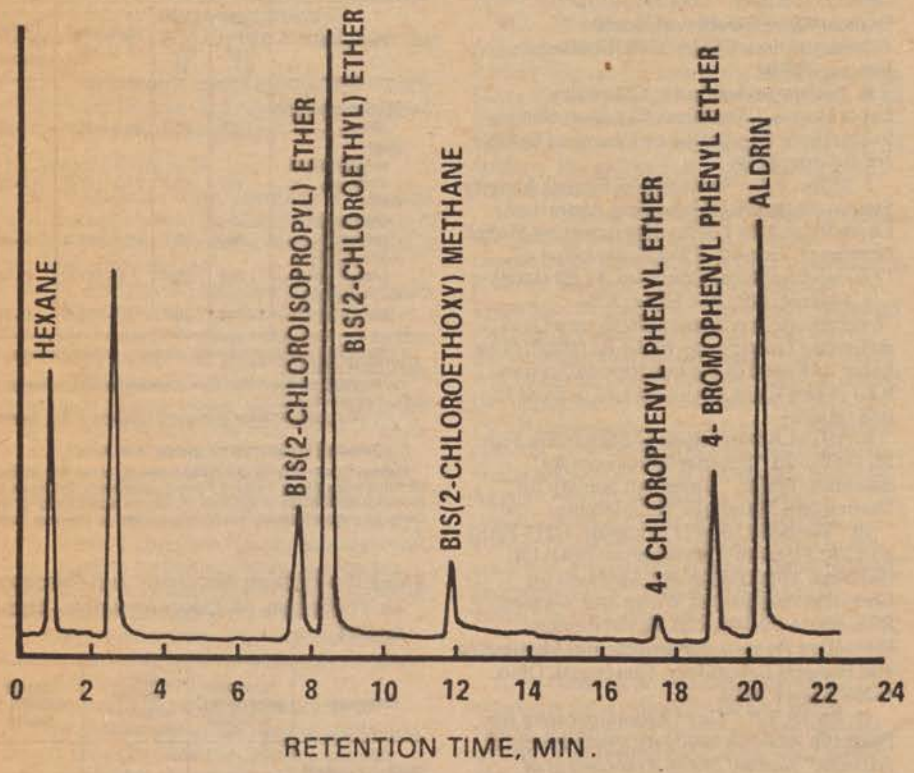



**Figure 1. Gas chromatogram of haloethers.**

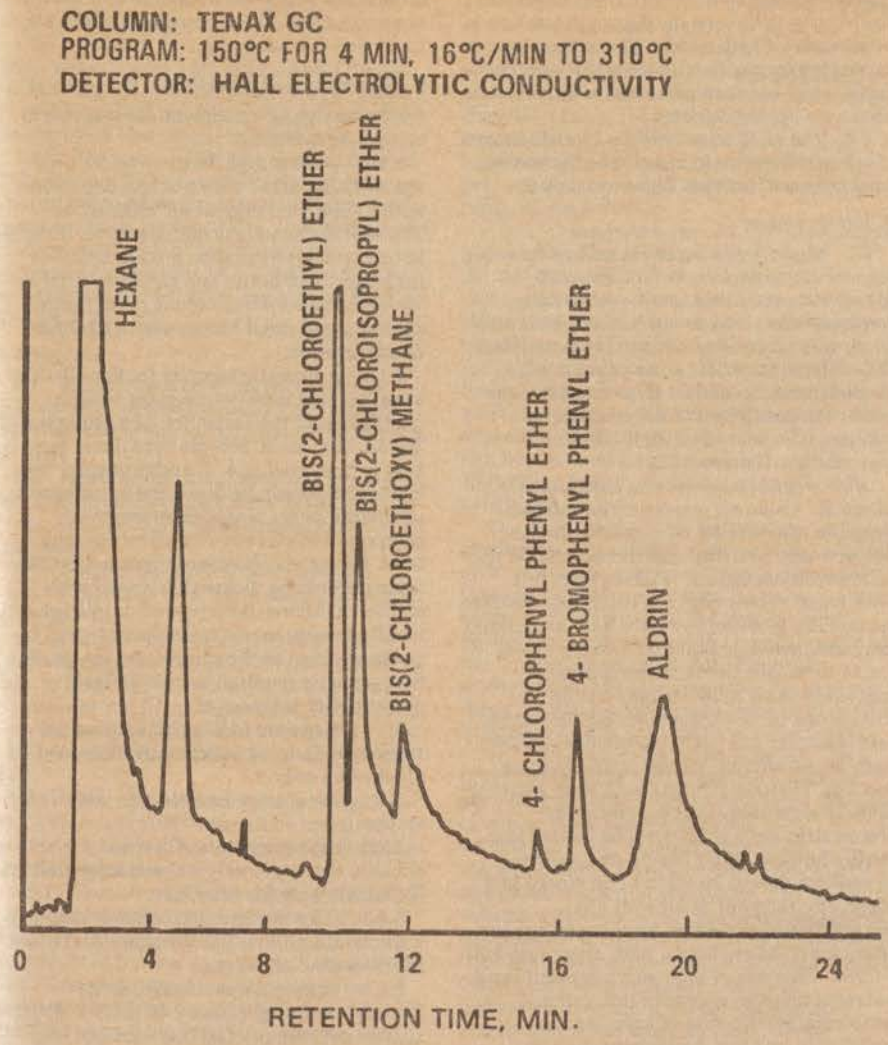



**Figure 2. Gas chromatogram of haloethers.**

BILLING CODE 6560-50-C

## Method 612—Chlorinated Hydrocarbons

*1. Scope and Application*

1.1 This method covers the determination of certain chlorinated hydrocarbons. The following parameters can be determined by this method:

| Parameter | STORET No. | CAS No. |
|---|---|---|
| 2-Chloronaphthalene | 34581 | 91-58-7 |
| 1,2-Dichlorobenzene | 34536 | 95-50-1 |
| 1,3-Dichlorobenzene | 34566 | 541-73-1 |
| 1,4-Dichlorobenzene | 34571 | 106-46-7 |
| Hexachlorobenzene | 39700 | 118-74-1 |
| Hexachlorobutadiene | 34391 | 87-68-3 |
| Hexachlorocyclopentadiene | 34386 | 77-47-4 |
| Hexachloroethane | 34396 | 67-72-1 |
| 1,2,4-Trichlorobenzene | 34551 | 120-82-1 |

1.2 This is a gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 The sample extraction and concentration steps in this method are essentially the same as in Methods 606, 608, 609, and 611. Thus, a single sample may be extracted to measure the parameters included in the scope of each of these methods. When cleanup is required, the concentration levels must be high enough to permit selecting aliquots, as necessary, to apply appropriate cleanup procedures. The analyst is allowed the latitude, under Section 12, to select chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.5 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 A measured volume of sample, approximately 1-L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to hexane during concentration to a volume of 10 mL or less. The extract is separated by gas chromatography and the parameters are then measured with an electron capture detector.[2]

2.2 The method provides a Florisil column cleanup procedure to aid in the elimination of interferences that may be encountered.

*3. Interferences*

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedure in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

*4. Safety*

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [4-6] for the information of the analyst.

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1-L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2-L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long x 19 mm ID, with coarse frit filter disc.

5.2.3 Chromatographic column—300 long x 10 mm ID, with Teflon stopcock and coarse frit filter disc at bottom.

5.2.4 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.5 Evaporative flask, Kuderna-Danish—500-mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

5.2.6 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K-503000-0121 or equivalent).

5.2.7 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (± 2 °C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column 1—1.8 m long x 2 mm ID glass, packed with 1% SP-1000 on Supelcoport (100/120 mesh) or equivalent.

Guidelines for the use of alternate column packings are provide in Section 12.1.

5.6.2 Column 2—1.8 m long × 2 mm ID glass, packed with 1.5% OV-1/2.4% OV-225 on Supelcoport (80/100 mesh) or equivalent. This column was used to develop the method performance statements in Section 14.

5.6.3 Detector— Electron capture detector. This detector has proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 14. Guidelines for the use of alternate detectors are provided in Section 12.1.

*6. Reagents*

6.1 Reagent water— Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Acetone, hexane, isooctane, methanol, methylene chloride, petroleum ether (boiling range 30 to 60 °C)—Pesticide quality or equivalent.

6.3 Sodium sulfate—(ACS) Granular, anhydrous. Purify heating at 400 °C for 4 h in a shallow tray.

6.4 Florisil—PR grade (60/100 mesh). Purchase activated at 1250 °F and store in the dark in glass containers with ground glass stoppers or foil-lined screw caps. Before use, activate each batch at least 16 h at 130 ° C in a foil-covered glass container and allow to cool.

6.5 Stock standard solution (1.00 $_{u}g/_{u}L$)— Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.5.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in isooctane and dilute to volume in a 120-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.5.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.5.3 Stock standard solutions must be replaced after six months, or sooner if comparision with check standards indicates a problem.

6.6 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Establish gas chromatographic operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with isooctane. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with isooctane. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Response for the parameter to be measured.

$A_{is}$ = Response for the internal standard.

$C_{is}$ = Concentration of the internal standard (µg/L).

$C_s$ = Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, a new calibration curve must be prepared for that compound.

7.5 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When the results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.4, 11.1, and 12.1) to improve the separations or lower the cost of measurements. Each time such modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at the following concentrations in acetone: Hexachloro-substituted parameters, 109 μg/mL; any other chlorinated hydrocarbon, 100 μg/mL. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at the test concentrations shown in Table 2 by adding 1.00 mL of QC check sample concentrate to each of four 1-L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μ g/L, and the standard deviation of the recovery (s) in μ g/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter.

**Note.**—The large number of parameters in Table 2 persent a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.2.6.2 Beginning with Section 8.2.2, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spike sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at the test concentration in Section 8.2.2 or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none by (2) the larger of either 5 times higher than the expected background concentration or the test concentration in Section 8.2.2.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. In necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100 (A-B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than the test concentration in Section 8.2.2, the analyst must use either the QC acceptance criteria in Tab'e 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) calculate accuracy (X') using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 3, substituting X' for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X'/T) ± 2.44 (100 S'/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4. If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

**Note.**—The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent recovery interval from $P-2s_p$ to $P+2s_p$. If P=90% and $s_p$=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevent performance evaluation studies.

*9. Sample Collection, Preservation, and Handling*

9.1 Grab samples must be collected in glass containers. Conventional sampling practices [8] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C from the time of collection until extraction.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[3]

*10. Sample Extraction*

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2-L separatory funnel.

10.2 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 to 2 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

**Note.**—The dichloribenzenes have a sufficiently high volatility that significant losses may occur in concentration steps if care is not exercised. It is important to maintain a constant gentle evaporation rate and not to allow the liquid volume to fall below 1 to 2 mL before removing the K–D apparatus from the hot water bath.

10.7 Momentarily remove the Snyder column, add 50 mL of hexane and a new boiling chip, and reattach the Snyder column. Raise the tempeature of the water bath to 85 to 90 °C. Concentrate the extract as in Section 10.6, except use hexane to prewet the column. The elapsed time of concentration should be 5 to 10 min.

10.8 Romove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of hexane. A 5-mL syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with gas chromatographic analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.9 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

*11. Cleanup and Separation*

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use the procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure.

11.2 Florisil column cleanup for chlorinated hydrocarbons:

11.2.1 Adjust the sample extract to 10 mL with hexane.

11.2.2 Place 12 g of Florisil into a chromatographic column. Tap the column to settle the Florisil and add 1 to 2 cm of anhydrous sodium sulfate to the top.

11.2.3 Preelute the column with 100 mL of petroleum ether. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the sample extract onto the column by decantation and subsequent petroleum ether washings. Discard the eluate. Just prior to exposure of the sodium sulfate layer to the air, begin eluting the column with 200 mL of petroleum ether and collect the eluate in a 500-mL K–D flask equipped with a 10-mL concentrator tube. This fraction should contain all of the chlorinated hydrocarbons.

11.2.4 Concentrate the fraction as in Section 10.6, except use hexane to prewet the column. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with hexane. Analyze by gas chromatography (Section 12).

*12. Gas Chromatography*

12.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by Column 2 are shown in Figures 1 and 2. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

12.2 Calibrate the system daily as described in Section 7.

12.3 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed throughly immediately before injection into the gas chromatograph.

12.4 Inject 2 to 5 μL of the sample extract or standard into the gas chromatograph using the solvent-flush technique.[9] Smaller (1.0 μL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 μL, the total extract volume, and the resulting peak size in area or peak height units.

12.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.6 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

12.7 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

*13. Calculations*

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

where:

$A$ = Amount of material injected (ng).
$V_i$ = Volume of extract injected (μL).
$V_t$ = Volume of total extract (μL).
$V_s$ = Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

$A_s$ = Response for the parameter to be measured.
$A_{is}$ = Response for the internal standard.
$I_s$ = Amount of internal standard added to each extract (μg).
$V_o$ = Volume of water extracted (L).

13.2 Report results in μg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

*14. Method Performance*

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[10] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 4×MDL to 1000×MDL.[10]

14.3 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 1.0 to 356 μg/L.[11] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

**References**


1. 40 CFR Part 136, Appendix B.
2. "Determination of Chlorinated Hydrocarbons In Industrial and Municipal Wastewaters," Report for EPA Contract 68-03-2625 (In preparation).
3. ASTM Annual Book of Standards, Part 31, D3694-78. "Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents," American Society for Testing and Materials, Philadelphia.
4. "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, August 1977.
5. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).
6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.
7. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58-63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)
8. ASTM Annual Book of Standards, Part 31, D3370-76. "Standard Practices for Sampling Water," American Society for Testing and Materials, Philadelphia.
9. Burke, J.A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).
10. "Development of Detection Limits, EPA Method 612, Chlorinated Hydrocarbons," Special letter report for EPA Contract 68-03-2625, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.
11. "EPA Method Validation Study 22, Method 612 (Chlorinated Hydrocarbons)," Report for EPA Contract 68-03-2625 (In preparation).
12. "Method Performance for Hexachlorocyclopentadiene by Method 612," Memorandum from R. Slater, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, December 7, 1983.


TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

| Parameter | Retention time (min) | | Method detection limit (μg/L) |
|---|---|---|---|
| | Column 1 | Column 2 | |
| 1,3-Dichlorobenzene | 4.5 | 6.8 | 1.19 |
| Hexachloroethane | 4.9 | 8.3 | 0.03 |
| 1,4-Dichlorobenzene | 5.2 | 7.6 | 1.34 |
| 1,2-Dichlorobenzene | 6.6 | 9.3 | 1.14 |
| Hexachlorobutadiene | 7.7 | 20.0 | 0.34 |
| 1,2,4-Trichlorobenzene | 15.5 | 22.3 | 0.05 |
| Hexachlorocyclopentadiene | nd | [c] 16.5 | 0.40 |
| 2-Chloronaphthalene | [a] 2.7 | [b] 3.6 | 0.94 |
| Hexachlorobenzene | [a] 5.6 | [b] 10.1 | 0.05 |

Column 1 conditions: Supelcoport (100/120 mesh) coated with 1% SP-1000 packed in a 1.8 m x 2 mm ID glass column with 5% methane/95% argon carrier gas at 25 mL/min flow rate. Column temperature held isothermal at 65 °C, except where otherwise indicated.
Column 2 conditions: Supelcoport (80/100 mesh) coated with 1.5% OV-1/2.4% OV-225 packed in a 1.8 m x 2 mm ID glass column with 5% methane/95% argon carrier gas at 25 mL/min flow rate. Column temperature held isothermal at 75 °C, except where otherwise indicated.

nd = Not determined.
[a] 150 °C column temperature.
[b] 165 °C column temperature.
[c] 100 °C column temperature.

TABLE 2.—QC ACCEPTANCE CRITERIA—METHOD 612

| Parameter | Test conc. (μg/L) | Limit for s (μg/L) | Range for $\bar{X}$ (μg/L) | Range for P, $P_s$ (percent) |
|---|---|---|---|---|
| 2-Chloronaphthalene | 100 | 37.3 | 29.5-126.9 | 9-148 |
| 1,2-Dichlorobenzene | 100 | 28.3 | 23.5-145.1 | 9-160 |
| 1,3-Dichlorobenzene | 100 | 26.4 | 7.2-138.6 | D-150 |
| 1,4-Dichlorobenzene | 100 | 20.6 | 22.7-126.9 | 13-137 |
| Hexachlorobenzene | 10 | 2.4 | 2.6-14.8 | 15-159 |
| Hexachlorobutadiene | 10 | 2.2 | D-12.7 | D-139 |
| Hexachlorocyclopentadiene | 10 | 2.5 | D-10.4 | D-111 |
| Hexachloroethane | 10 | 3.3 | 2.4-12.3 | 8-139 |
| 1,2,4-Trichlorobenzene | 100 | 31.6 | 20.2-133.7 | 5-149 |

s = Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
P, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.

NOTE.—These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 612

| Parameter | Accuracy, as recovery, X' (μg/L) | Single analyst precision, $s_r'$ (μg/L) | Overall precision, S' (μg/L) |
|---|---|---|---|
| 2-Chloronaphthalene | 0.75C+3.21 | $0.28\bar{X}-1.17$ | $0.38\bar{X}-1.39$ |
| 1,2-Dichlorobenzene | 0.85C-0.70 | $0.22\bar{X}-2.95$ | $0.41\bar{X}-3.92$ |
| 1,3-Dichlorobenzene | 0.72C+0.87 | $0.21\bar{X}-1.03$ | $0.49\bar{X}-3.98$ |

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 612—Continued

| Parameter | Acccuracy, as recovery, X' (μg/L) | Single analyst precision, $s_r$' (μg/L) | Overall precision, S' (μg/L) |
|---|---|---|---|
| 1,4-Dichlorobenzene | 0.72C+2.80 | 0.16$\bar{X}$−0.48 | 0.35$\bar{X}$−0.57 |
| Hexachlorobenzene | 0.87C−0.02 | 0.14$\bar{X}$+0.07 | 0.36$\bar{X}$−0.19 |
| Hexachlorobutadiene | 0.61C+0.03 | 0.18$\bar{X}$+0.08 | 0.53$\bar{X}$−0.12 |
| Hexachlorocyclopentadiene * | 0.47C | 0.24$\bar{X}$ | 0.50$\bar{X}$ |
| Hexachloroethane | 0.74C−0.02 | 0.23$\bar{X}$+0.07 | 0.36$\bar{X}$−0.00 |
| 1,2,4-Trichlorobenzene | 0.76C+0.98 | 0.23$\bar{X}$−0.44 | 0.40$\bar{X}$−1.37 |

X' = Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
$s_r$' = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C = True value for the concentration, in μg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in μg/L.
* Estimates based upon the performance in a single laboratory.[18]

BILLING CODE 6560-50-M

COLUMN: 1.5% OV-1/2.4% OV-225 ON SUPELCOPORT
TEMPERATURE: 75°C
DETECTOR: ELECTRON CAPTURE

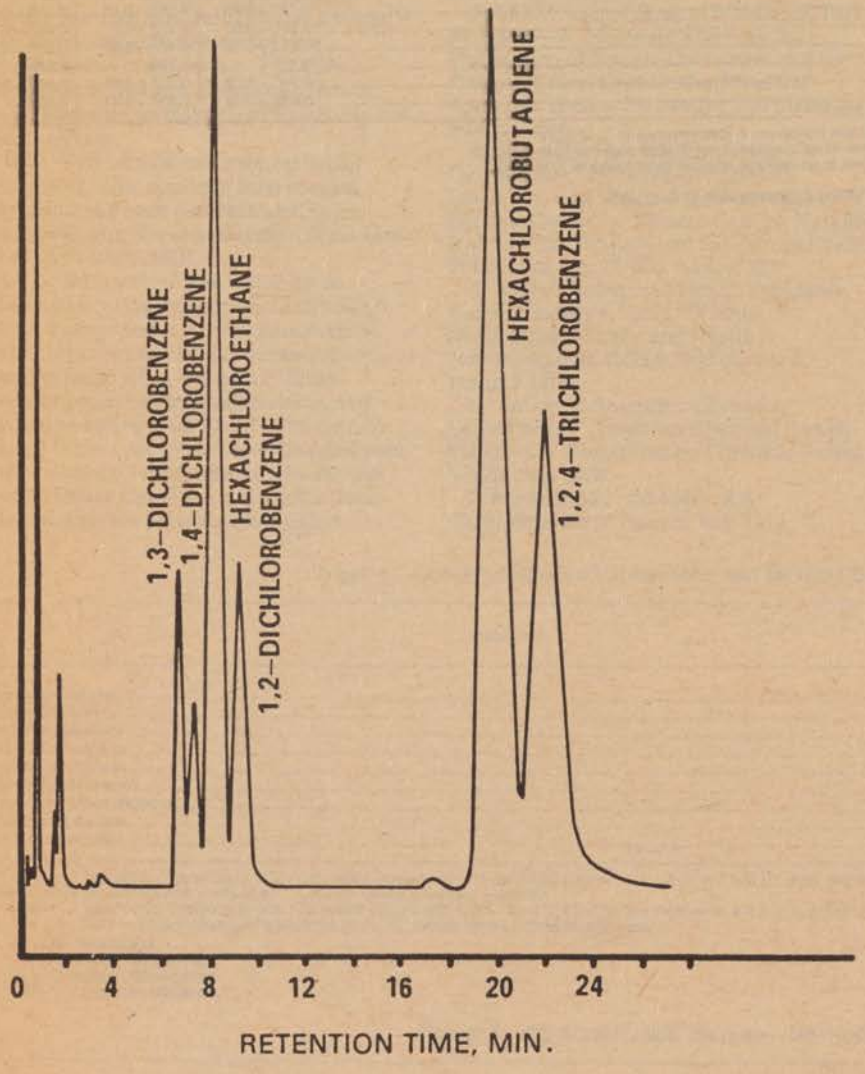



**Figure 1. Gas chromatogram of chlorinated hydrocarbons.**

COLUMN: 1.5% OV-1/2.4% OV-225 ON SUPELCOPORT
TEMPERATURE: 165°C
DETECTOR: ELECTRON CAPTURE

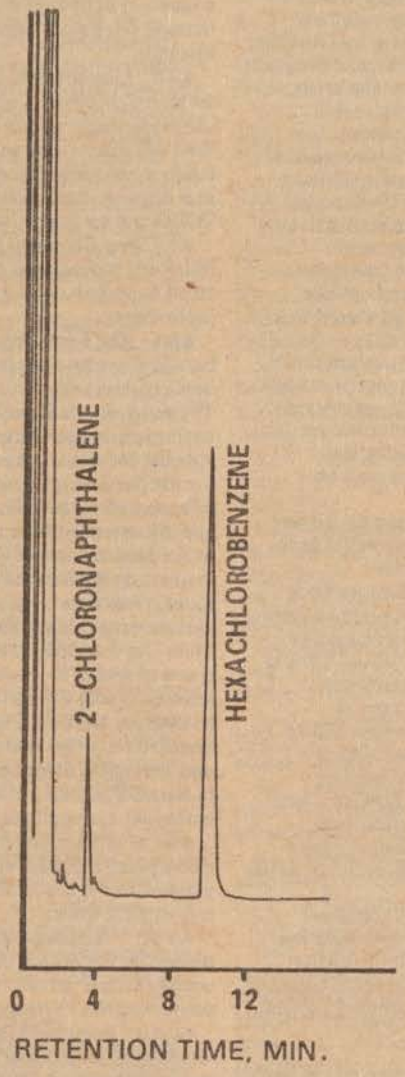



**Figure 2. Gas chromatogram of chlorinated hydrocarbons.**

BILLING CODE 6560-50-C

**Method 613—2,3,7,8-Tetrachlorodibenzo-p-Dioxin**

*1. Scope and Application*

1.1 This method covers the determination of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD). The following parameter may be determined by this method:

| Parameter | STORET No. | GAS No. |
|---|---|---|
| 2,3,7,8-TCDD | 34675 | 1746-01-6 |

1.2 This is a gas chromatographic/mass spectrometer (GC/MS) method applicable to the determination of 2,3,7,8-TCDD in municipal and industrial discharges as provided under 40 CFR 136.1. Method 625 may be used to screen samples for 2,3,7,8-TCDD. When the screening test is positive, the final qualitative confirmation and quantification mlust be made using Method 613.

1.3 The method detection limit (MDL, defined in Section 14.1) [1] for 2,3,7,8-TCDD is listed in Table 1. The MDL for a specific wastewater may be different from that listed, depending upon the nature of interferences in the sample matrix.

1.4 Because of the extreme toxicity of this compound, the analyst must prevent exposure to himself, of to others, by materials knows or believed to contain 2,3,7,8-TCDD. Section 4 of this method contains guidelines and protocols that serve as minimum safe-handling standards in a limited-access laboratory.

1.5 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 A measured volume of sample, approximately 1–L, is spiked with an internal standard of labeled 2,3,7,8-TCDD and extracted with methylene chloride using a separatory funnel. The methylene chloride extract is exchanged to hexane during concentration to a volume of 1.0 mL or less. The extract is then analyzed by capillary column GC/MS to separate and measure 2,3,7,8-TCDD.[2,3]

2.2 The method provides selected column chromatographic cleanup proceudres to aid in the elimination of interferences that may be encountered.

*3. Interferences*

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated backgrounds at the masses (m/z) monitored. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleand.[4] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by the treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to mininmize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. 2,3,7,8-TCDD is often associated with other interfering chlorinated compounds which are at concentrations several magnitudes higher than that of 2,3,7,8-TCDD. The cleanup producers in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches [1,5-7] to eliminate false positives and achieve the MDL listed in Table 1.

3.3 The primary column, SP–2330 or equivalent, resolves 2,3,7,8-TCDD from the other 21 TCDD insomers. Positive results using any other gas chromatographic column must be confirmed using the primary column.

*4. Safety*

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [8-10] for the information of the analyst. Benzene and 2,3,7,8-TCDD have been identified as suspected human or mammalian carcinogens.

4.2 Each laboratory must develop a strict safety program for handling 2,3,7,8-TCDD. The following laboratory practices are recommended:

4.2.1 Contamination of the laboratory will be minimized by conducting all manipulations in a hood.

4.2.2 The effluents of sample splitters for the gas chromatograph and roughing pumps on the GC/MS should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.

4.2.3 Liquid waste should be dissolved in methanol or ethanol and irradiated with ultraviolet light with a wavelength greater than 290 nm for several days. (Use F 40 BL lamps or equivalent). Analyze liquid wastes and dispose of the solutions when 2,3,7,8–TCDD can no longer be detected.

4.3 Dow Chemical U.S.A. has issued the following precautions (revised November 1978) for safe handling of 2,3,7,8-TCDD in the laboratory:

4.3.1 The following statements on safe handling are as complete as possible on the basis of available toxicological information. The precautions for safe handling and use are necessarily general in nature since detailed, specific recommendations can be made only for the particular exposure and circumstances of each individual use. Inquiries about specific operations or uses may be addressed to the Dow Chemical Company. Assistance in evaluating the health hazards of particular plant conditions may be obtained from certain consulting laboratories and from State Departments of Health or of Labor, many of which have an industrial health service. 2,3,7,8-TCDD is extremely toxic to laboratory animals. However, it has been handled for years without injury in analytical and biological laboratories. Techniques used in handling radioactive and infectious materials are applicable to 2,3,7,8,-TCDD.

4.3.1.1 Protective equipment—Throw-away plastic gloves, apron or lab coat, safety glasses, and a lab hood adequate for radioactive work.

4.3.1.2 Training—Workers must be trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces.

4.3.1.3 Personal hygiene—Thorough washing of hands and forearms after each manipulation and before breaks (coffee, lunch, and shift).

4.3.1.4 Confinement—Isolated work area, posted with signs, segregated glassware and tools, plastic-backed absorbent paper on benchtops.

4.3.1.5 Waste—Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans. Janitors must be trained in the safe handling of waste.

4.3.1.6 Disposal of wastes—2,3,7,8-TCDD decomposes above 800 °C. Low-level waste such as absorbent paper, tissues, animal remains, and plastic gloves may be burned in a good incinerator. Gross quantities (milligrams) should be packaged securely and disposed through commercial or governmental channels which are capable of handling high-level radioactive wastes or extremely toxic wastes. Liquids should be allowed to evaporate in a good hood and in a disposable container. Residues may then be handled as above.

4.3.1.7 Decontamination—For personal decontamination, use any mild soap with plenty of scrubbing action. For decontamination of glassware, tools, and surfaces, Chlorothene NU Solvent (Trademark of the Dow Chemical Company) is the least toxic solvent shown to be effective. Satisfactory cleaning may be accomplished by rinsing with Chlorothene, then washing with any detergent and water. Dishwater may be disposed to the sewer. It is prudent to minimize solvent wastes because they may require special disposal through commercial sources which are expensive.

4.3.1.8 Laundry—Clothing known to be contaminated should be disposed with the precautions described under Section 4.3.1.6. Lab coats or other clothing worn in 2,3,7,8-TCDD work areas may be laundered.

Clothing should be collected in plastic bags. Persons who convey the bags and launder the clothing should be advised of the hazard and trained in proper handling. The clothing may be put into a washer without contact if the launderer knows the problem. The washer should be run through a cycle before being used again for other clothing.

4.3.1.9 Wipe tests—A useful method of determining cleanliness of work surfaces and tools is to wipe the surface with a piece of filter paper. Extraction and analysis by gas chromatography can achieve a limit of sensitivity of 0.1 μg per wipe. Less than 1 μg of 2,3,7,8-TCDD per sample indicates acceptable cleanliness; anything higher warrants further cleaning. More than 10 μg on a wipe sample constitutes an acute hazard and requires prompt cleaning before further use of the equipment or work space. A high (>10 μg) 2,3,7,8-TCDD level indicates that unacceptable work practices have been employed in the past.

4.3.1.10 Inhalation—Any procedure that may produce airborne contamination must be done with good ventilation. Gross losses to a ventilation system must not be allowed. Handling of the dilute solutions normally used in analytical and animal work presents no inhalation hazards except in the case of an accident.

4.3.1.11 Accidents—Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1-L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.1.3 Clearly label all samples as "POISON" and ship according to U.S. Department of Transportation regulations.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnels—2-L and 125-mL, with Teflon stopcock.

5.2.2 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K–570050–1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.3 Evaporative flask, Kuderna-Danish—500-mL (Kontes K–570001–0500 or equivalent). Attach to concentrator tube with springs.

5.2.4 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K–503000–0121 or equivalent).

5.2.5 Snyder column, Kuderna-Danish—Two-ball micro (Kontes K–569001–0219 or equivalent).

5.2.6 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.2.7 Chromatographic column—300 mm long × 10 mm ID, with Teflon stopcock and coarse frit filter disc at bottom.

5.2.8 Chromatographic column—400 mm long × 11 mm ID, with Teflon stopcock and coarse frit filter disc at bottom.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2 °C). The bath should be used in a hood.

5.5 GC/MS system:

5.5.1 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port must be designed for capillary columns. Either split, splitless, or on-column injection techniques may be employed, as long as the requirements of Section 7.1.1 are achieved.

5.5.2 Column—60 m long × 0.25 mm ID glass or fused silica, coated with SP–2330 (or equivalent) with a film thickness of 0.2 μm. Any equivalent column must resolve 2, 3, 7, 8–TCDD from the other 21 TCDD isomers.[16]

5.5.3 Mass spectrometer—Either a low resolution mass spectrometer (LRMS) or a high resolution mass spectrometer (HRMS) may be used. The mass spectrometer must be equipped with a 70 V (nominal) ion source and be capable of aquiring m/z abundance data in real time selected ion monitoring (SIM) for groups of four or more masses.

5.5.4 GC/MS interface—Any GC to MS interface can be used that achieves the requirements of Section 7.1.1. GC to MS interfaces constructed of all glass or glass-lined materials are recommended. Glass surfaces can be deactivated by silanizing with dichlorodimethylsilane. To achieve maximum sensitivity, the exit end of the capillary column should be placed in the ion source. A short piece of fused silica capillary can be used as the interface to overcome problems associated with straightening the exit end of glass capillary columns.

5.5.5 The SIM data acquired during the chromatographic program is defined as the Selected Ion Current Profile (SICP). The SICP can be acquired under computer control or as a real time analog output. If computer control is used, there must be software available to plot the SICP and report peak height or area data for any m/z in the SICP between specified time or scan number limits.

5.6 Balance—Analytical, capable of accurately weighing 0.0001 g.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of 2, 3, 7, 8–TCDD.

6.2 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 mL. Wash the solution with methylene chloride and hexane before use.

6.3 Sodium thiosulfate—(ACS) Granular.

6.4 Sulfuric acid—Concentrated (ACS, sp. gr. 1.84).

6.5 Acetone, methylene chloride, hexane, benzene, ortho-xylene, tetradecane—Pesticide quality or equivalent.

6.6 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray.

6.7 Alumina—Neutral, 80/200 mesh (Fisher Scientific Co., No. A–540 or equivalent). Before use, activate for 24 h at 130 °C in a foil-covered glass container.

6.8 Silica gel—High purity grade, 100/120 mesh (Fisher Scientific Co., No. S–679 or equivalent).

6.9 Stock standard solutions (1.00 μg/μL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions. Acetone should be used as the solvent for spiking solutions; ortho-xylene is recommended for calibration standards for split injectors; and tetradecane is recommended for splitless or on-colum injectors. Analyze stock internal standards to verify the absence of native 2,3,7,8-TCDD.

6.9.1 Prepare stock standard solutions of 2,3,7,8–TCDD (mol wt 320) and either $^{37}Cl_4$ 2,3,7,8-TCDD (mol wt 328) or $^{13}C_{12}$ 2,3,7,8-TCDD (mol wt 332) in an isolated area by accurately weighing about 0.0100 g of pure material. Dissolve the material in pesticide quality solvent and dilute to volume in a 10-mL volumetric flask. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.9.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store in an isolated refrigerator protected from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just

prior to preparing calibration standards or spiking solutions from them.

6.9.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.10 Internal standard spiking solution (25 ng/mL)—Using stock standard solution, prepare a spiking solution in acetone of either $^{13}C_{12}$ or $^{37}Cl_4$ 2,3,7,8-TCDD at a concentration of 25 ng/ML. (See Section 10.2)

6.11 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Establish gas chromatograhic operating conditions equivalent to those given in Table 1 and SIM conditions for the mass spectrometer as described in Section 12.2 The GC/MS system must be calibrated using the internal standard technique.

7.1.1 Using stock standards, prepare calibration standards that will allow measurement of relative response factors of at least three concentration ratios of 2,3,7,8-TCDD to internal standard. Each calibration standard must be prepared to contain the internal standard at a concentration of 25 ng/mL. If any interferences are contributed by the internal standard at n/z 320 and 322, its concentration may be reduced in the calibration standards and in the internal standard spiking solution (Section 6.10). One of the calibration standards should contain 2,3,7,8-TCDD at a concentration near, but above, the MDL and the other 2,3,7,8-TCDD concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC/MS system.

7.1.2 Using injections of 2 to 5 μL, analyze each calibration standard according to Section 12 and tabulate peak height or area response against the concentration of 2,3,7,8-TCDD and internal standard. Calculate response factors (RF) for 2,3,7,8-TCDD using Equation 1.

Equation 1.

$$RF = \frac{(A_s)\,(C_{is})}{(A_{is})\,(C_s)}$$

where:

$A_s$ = SIM response for 2,3,7,8-TCDD m/z 320.

$A_{is}$ = SIM response for the internal standard, m/z 332 for $^{13}C_{12}$ 2,3,7,8-TCDD m/z 328 for $^{37}Cl_4$ 2,3,7,8-TCDD.

$C_{is}$ = Concentration of the internal standard (μg/L).

$C_s$ = Concentration of 2,3,7,8-TCDD (μg/L).

If the RF value over the working range is a constant (< 10% relative standard deviation, RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.1.3 The working calibration curve or RF must be verified on each working day by the measurement of one or more 2,3,7,8-TCDD calibration standards. If the response for 2,3,7,8-TCDD varies from the predicted response by more than ±15%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared.

7.2 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.5, 11.1, and 12.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples with native 2,3,7,8-TCDD to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing 2,3,7,8-TCDD at a concentration of 0.100 μg/mL in acetone. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at a concentration of 0.100 μg/L (100 ng/L) by adding 1.00 mL of QC check sample concentrate to each of four 1-L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for 2,3,7,8-TCDD using the four results.

8.2.5 Compare s and ($\bar{X}$) with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If s exceeds the precision limit or $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for 2,3,7,8-TCDD. Locate and correct the source of the problem and repeat the test beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of 2,3,7,8-TCDD in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of 2,3,7,8-TCDD in the sample is not being checked against a limit specific to that parameter, the spike should be at 0.100 μg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or 0.100 μg/L.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of 2,3,7,8-TCDD. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentration in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the

concentration after spiking (A) of 2,3,7,8-TCDD. Calculate percent recovery (P) as 100(A–B)%T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for 2,3,7,8-TCDD with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[11] If spiking was performed at a concentration lower than 0.100 μg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of 2,3,7,8-TCDD: (1) calculate accuracy (X') using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 3, substituting X' for X; (3) calculate the range for recovery at the spike concentration as (100 X'/T)±2.44(100 S'/T)%.[11]

8.3.4 If the recovery of 2,3,7,8-TCDD falls outside the designated range for recovery, a check standard must be analyzed as described in Section 8.4.

8.4 If the recovery of 2,3,7,8-TCDD fails the acceptance criteria for recovery in Section 8.3, a QC check standard must be prepared and analyzed.

Note.—The frequency for the required analysis of a QC check standard will depend upon the complexity of the sample matrix and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 1 L of reagent water.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of 2,3,7,8-TCDD. Calculate the percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) with the corresponding QC acceptance criteria found in Table 2. If the recovery of 2,3,7,8-TCDD falls outside the designated range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for 2,3,7,8-TCDD in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the spandard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratoary adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

*9. Sample Collection, Preservation, and Handling*

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[12] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C and protected from light from the time of collection until extraction. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[13] Field test kits are available for this purpose.

9.3 Label all samples and containers "POISON" and ship according to applicable U.S. Department of Transportation regulations.

9.4 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

*10. Sample Extraction*

**Caution:** When using this method to analyze for 2,3,7,8-TCDD, all of the following operations must be performed in a limited-access laboratory with the analyst wearing full protective covering for all exposed skin surfaces. See Section 4.2.

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2–L separatory funnel.

10.2 Add 1.00 mL of internal standard spiking solution to the sample in the separatory funnel. If the final extract will be concentrated to a fixed volume below 1.00 mL (Section 12.3), only that volume of spiking solution should be added to the sample so that the final extract will contain 25 ng/mL of internal standard at the time of analysis.

10.3 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.4 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.5 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.6 Pour the combined extract into the K–D concentrator. Rinse the Erlenmeyer flask with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.7 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.8 Momentarily remove the Snyder column, add 50 mL of hexane and a new boiling chip, and reattach the Snyder column. Raise the temperature of the water bath to 85 to 90°C. Concentrate the extract as in Section 10.7, except use hexane to prewet the column. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of hexane. A 5-mL syringe is recommended for this operation. Set aside the K–D glassware for reuse in Section 10.14.

10.9 Pour the hexane extract from the concentrator tube into a 125-mL separatory funnel. Rinse the concentrator tube four times with 10-mL aliquots of hexane. Combine all rinses in the 125-mL separatory funnel.

10.10 Add 50 mL of sodium hydroxide solution to the funnel and shake for 30 to 60 s. Discard the aqueous phase.

10.11 Perform a second wash of the organic layer with 50 mL of reagent water. Discard the aqueous phase.

10.12 Wash the hexane layer with a least two 50-mL aliquots of concentrated sulfuric acid. Continue washing the hexane layer with 50-mL aliquots of concentrated sulfuric acid until the acid layer remains colorless. Discard all acid fractions.

10.13 Wash the hexane layer with two 50-mL aliquots of reagent water. Discard the aqueous phases.

10.14 Transfer the hexane extract into a 125-mL Erlenmeyer flask containing 1 to 2 g of anhydrous sodium sulfate. Swirl the flask for 30 s and decant the hexane extract into the reassembled K–D apparatus. Complete the quantitative transfer with two 10-mL hexane rinses of the Erlenmeyer flask.

10.15 Replace the one or two clean boiling chips and concentrate the extract to 6 to 10 mL as in Section 10.8.

10.16 Add a clean boiling chip to the concentrator tube and attach a two-ball micro-Snyder column. Prewet the column by adding about 1 mL of hexane to the top. Place the micro-K–D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches about 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with 0.2 mL of hexane.

Adjust the extract volume to 1.0 mL with hexane. Stopper the concentrator tube and store refrigerated and protected from light if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with GC/MS analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.17 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

*11. Cleanup and Separation*

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use either procedure below or any other appropriate procedure.[1,5-7] However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure. Two cleanup column options are offered to the analyst in this section. The alumina column should be used first to overcome interferences. If background problems are still encountered, the silica gel column may be helpful.

11.2 Alumina column cleanup for 2,3,7,8-TCDD:

11.2.1 Fill a 300 mm long x 10 mm ID chromatographic column with activated alumina to the 150 mm level. Tap the column gently to settle the alumina and add 10 mm of anhydrous sodium sulfate to the top.

11.2.2 Preelute the column with 50 mL of hexane. Adjust the elution rate to 1 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 1.0-mL sample extract onto the column using two 2-mL portions of hexane to complete the transfer.

11.2.3 Just prior to exposure of the sodium sulfate layer to the air, add 50 mL of 3% methylene chloride/97% hexane (V/V) and continue the elution of the column. Discard the eluate.

11.2.4 Next, elute the column with 50 mL of 20% methylene chloride/80% hexane (V/V) into a 500-mL K–D flask equipped with a 10-mL concentrator tube. Concentrate the collected fraction to 1.0 mL as in Section 10.16 and analyze by GC/MS (Section 12).

11.3 Silica gel column cleanup for 2,3,7,8-TCDD:

11.3.1 Fill a 400 mm long x 11 mm ID chromatographic column with silica gel to the 300 mm level. Tap the column gently to settle the silica gel and add 10 mm of anhydrous sodium sulfate to the top.

11.3.2 Preelute the column with 50 mL of 20% benzene/80% hexane (V/V). Adjust the elution rate to 1 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 1.0-mL sample extract onto the column using two 2-mL portions of 20% benzene/80% hexane to complete the transfer.

11.3.3 Just prior to exposure of the sodium sulfate layer to the air, add 40 mL of 20% benzene/80% hexane to the column. Collect the eluate in a clean 500-mL K-D flask equipped with a 10-mL concentrator tube. Concentrate the collected fraction to 1.0 mL as in Section 10.16 and analyze by GC/MS.

*12. GC/MS Analysis*

12.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Other capillary columns or chromatographic conditions may be used if the requirements of Sections 5.5.2 and 8.2 are met.

12.2 Analyze standards and samples with the mass spectrometer operating in the selected ion monitoring (SIM) mode using a dwell time to give at least seven points per peak. For LRMS, use masses at m/z 320, 322, and 257 for 2,3,7,8-TCDD and either m/z 328 for $^{37}Cl_4$ 2,3,7,8-TCDD or m/z 332 for $^{13}C_{12}$ 2,3,7,8-TCDD. For HRMS, use masses at m/z 319.8965 and 321.8936 for 2,3,7,8-TCDD and either m/z 327.8847 for $^{37}Cl_4$ 2,3,7,8-TCDD or m/z 331.9367 for $^{13}C_{12}$ 2,3,7,8-TCDD.

12.3 If lower detection limits are required, the extract may be carefully evaporated to dryness under a gentle stream of nitrogen with the concentrator tube in a water bath at about 40 °C. Conduct this operation immediately before GC/MS analysis. Redissolve the extract in the desired final volume of ortho-xylene or tetradecane.

12.4 Calibrate the system daily as described in Section 7.

12.5 Inject 2 to 5 μL of the sample extract into the gas chromatograph. The volume of calibration standard injected must be measured, or be the same as all sample injection volumes.

12.6 The presence of 2,3,7,8-TCDD is qualitatively confirmed if all of the following criteria are achieved:

12.6.1 The gas chromatographic column must resolve 2,3,7,8-TCDD from the other 21 TCDD isomers.

12.6.2 The masses for native 2,3,7,8-TCDD (LRMS-m/z 320, 322, and 257 and HRMS-m/z 320 and 322) and labeled 2,3,7,8-TCDD (m/z 328 or 332) must exhibit a simultaneous maximum at a retention time that matches that of native 2,3,7,8-TCDD in the calibration standard, with the performance specifications of the analytical system.

12.6.3 The chlorine isotope ratio at m/z 320 and m/z 322 must agree to within ±10% of that in the calibration standard.

12.6.4 The signal of all peaks must be greater than 2.5 times the noise level.

12.7 For quantitation, measure the response of the m/z 320 peak for 2,3,7,8-TCDD and the m/z 332 peak for $^{13}C_{12}$ 2,3,7,8-TCDD or the m/z 328 peak for $^{37}Cl_4$ 2,3,7,8-TCDD.

12.8 Co-eluting impurities are suspected if all criteria are achieved except those in Section 12.6.3. In this case, another SIM analysis using masses at m/z 257, 259, 320 and either m/a 328 or m/z 322 can be performed. The masses at m/z 257 and m/z 259 are indicative of the loss of one chlorine and one carbonyl group from 2,3,7,8-TCDD. If masses m/z 257 and m/z 259 give a chlorine isotope ratio that agrees to within ±10% of the same cluster in the calibration standards, then the presence of TCDD can be confirmed. Co-eluting DDD, DDE, and PCB residues can be confirmed, but will require another injection using the appropriate SIM masses or full repetitive mass scans. If the response for $^{37}Cl_4$ 2,3,7,8-TCDD at m/z 328 is too large, PCB contamination is suspected and can be confirmed by examining the response at both m/z 326 and m/z 328: The $^{37}Cl_4$ 2,3,7,8-TCDD internal standard gives negligible response at m/z 326. These pesticide residues can be removed using the alumina column cleanup procedure.

12.9 If broad background interference restricts the sensitivity of the GC/MS analysis, the analyst should employ additional cleanup procedures and reanalyze by GC/MS.

12.10 In those circumstances where these procedures do not yield a definitive conclusion, the use of high resolution mass spectrometry is suggested.[5]

*13. Calculations*

13.1 Calculate the concentration of 2,3,7,8-TCDD in the sample using the response factor (RF) determined in Section 7.1.2 and Equation 2.

Equation 2:

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

$A_s$ = SIM response for 2,3,7,8-TCDD at m/z 320.

$A_{is}$ = SIM response for the internal standard at m/z 328 or 332.

$I_s$ = Amount of internal standard added to each extract (μg).

$V_o$ = Volume of water extracted (L).

13.2 For each sample, calculate the percent recovery of the internal standard by comparing the area of the m/z peak measured in the sample to the area of the same peak in the calibration standard. If the recovery is below 50%, the analyst should review all aspects of his analytical technique.

13.3 Report results in μg/L without correction for recovery data. All QC data

obtained should be reported with the sample results.

*14. Method Performance*

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentration listed in Table 1 was obtained using reagent water.[14] The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method was tested by 11 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 0.02 to 0.20 μg/L.[15] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

**References**


1. 40 CFR Part 136, Appendix B.
2. "Determination of 2,3,7,8-TCDD in Industrial and Municipal Wastewaters," EPA-600/4-82-028, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, June 1982.
3. Buser, H.R., and Rappe, C. "High Resolution Gas Chromatography of the 22 Tetrachlorodibenzo-p-dioxin Isomers," *Analytical Chemistry, 52,* 2257 (1980).
4. ASTM Annual Book of Standards, Part 31, D3694-78. "Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents," American Society for Testing and Materials, Philadelphia.
5. Harless, R. L., Oswald, E. O., and Wilkinson, M. K. "Sample Preparation and Gas Chromatography/Mass Spectrometry Determination of 2,3,7,8-Tetrachlorodibenzo-p-dioxin," *Analytical Chemistry, 52,* 1239 (1980).
6. Lamparski, L. L., and Nestrick, T. J. "Determination of Tetra-, Hepta-, and Octachlorodibenzo-p-dioxin Isomers in Particulate Samples at Parts per Trillion Levels," *Analytical Chemistry, 52,* 2045 (1980).
7. Longhorst, M. L., and Shadoff, L. A. "Determination of Parts-per-Trillion Concentrations of Tetra-, Hexa-, and Octachlorodibenzo-p-dioxins in Human Milk," *Analytical Chemistry, 52,* 2037 (1980).
8. "Carcinogens—Working with Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77-206, August 1977.
9. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occuptional Safety and Health Administration, OSHA 2206 (Revised, January 1976).
10. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.
11. Provost, L. P., and Elder, R. S., "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58-63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)
12. ASTM Annual Book of Standards, Part 31, D3370-76, "Standard Practices for Sampling Water," American Society for Testing and Materials, Philadelphia.
13. "Methods, 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.
14. Wong, A.S. et al. "The Determination of 2,3,7,8-TCDD in Industrial and Municipal Wastewaters, Method 613, Part 1—Development and Detection Limits," G. Choudhay, L. Keith, and C. Ruppe, ed., Butterworth Inc., (1983).
15. "EPA Method Validation Study 23, Method 613 (2,3,7,8-Tetrachlorodibenzo-p-dioxin)," Report for EPA Contract 68-03-2863 (In preparation).


TABLE 1.—Chromatographic Conditions and Method Detection Limit

| Parameter | Retention time (min) | Method detection limit (μg/L) |
|---|---|---|
| 2,3,7,8,-TCDD | 13.1 | 0.002 |

Column conditions: SP-2330 coated on a 60 m long × 0.25 mm ID glass column with hydrogen carrier gas at 40 cm/sec linear velocity, splitless injection using tetradecane. Column temperature held isothermal at 200°C for 1 min, then programmed at 8°C/min to 250 °C and held. Use of helium carrier gas will approximately double the retention time.

TABLE 2.—QC Acceptance Criteria—Method 613

| Parameter | Test conc. (μg/L) | Limit for s (μg/L) | Range for X (μg/L) | Range for P, $P_s$ (%) |
|---|---|---|---|---|
| 2,3,7,8-TCDD | 0.100 | 0.0276 | 0.0523-0.1226 | 45-129 |

s = Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
P, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2).

**Note.**—These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE. 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 613

| Parameter | Accuracy, as recovery, $X'$ (μg/L) | Single analyst, precision, $s_r'$ (μ/L) | Overall precision, S (μ/g/L) |
|---|---|---|---|
| 2,3,7,8-TCDD | 0.86C+0.00145 | 0.13$\bar{X}$+0.00129 | 0.19$\bar{X}$+0.00028 |

$X'$ = Expected recovery for one or more measurements, of a sample containing a concentration of C, in μg/L.
$s_r'$ = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
$S'$ = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C = True value for the concentration, in μg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in μg/L.

**Method 624—Purgeables**

*1. Scope and Application*

1.1 This method covers the determination of a number of purgeable organics. The following parameters may be determined by this method:

| Parameter | STORET No. | CAS No. |
|---|---|---|
| Benzene | 34030 | 71-43-2 |
| Bromodichloromethane | 32101 | 75-27-4 |
| Bromoform | 32104 | 75-25-2 |
| Bromomethane | 34413 | 74-83-9 |
| Carbon tetrachloride | 32102 | 56-23-5 |
| Chlorobenzene | 34301 | 108-90-7 |
| Chloroethane | 34311 | 75-00-3 |
| 2-Chloroethylvinyl ether | 34576 | 110-75-8 |
| Chloroform | 32106 | 67-66-3 |
| Chloromethane | 34418 | 74-87-3 |
| Dibromochloromethane | 32105 | 124-48-1 |
| 1,2-Dichlorobenzene | 34536 | 95-50-1 |
| 1,3-Dichlorobenzene | 34566 | 541-73-1 |
| 1,4-Dichlorobenzene | 34571 | 106-46-7 |
| 1,1-Dichloroethane | 34496 | 75-34-3 |
| 1,2-Dichloroethane | 34531 | 107-06-2 |
| 1,1-Dichloroethane | 34501 | 75-35-4 |
| trans-1,2-Dichloroethane | 34546 | 156-60-5 |
| 1,2-Dichloropropane | 34541 | 78-87-5 |
| cis-1,3-Dichloropropane | 34704 | 10061-01-5 |
| trans-1,3-Dichloropropane | 34699 | 10061-02-6 |
| Ethyl benzene | 34371 | 100-41-4 |
| Methylene chloride | 34423 | 75-09-2 |
| 1,1,2,2-Tetrachloroethane | 34516 | 79-34-5 |
| Tetrachloroethene | 34475 | 127-18-4 |
| Toluene | 34010 | 108-88-3 |
| 1,1,1-Trichloroethane | 34506 | 71-55-6 |
| 1,1,2-Trichloroethane | 34511 | 79-00-5 |
| Trichloroethane | 39180 | 79-01-6 |
| Trichlorofluoromethane | 34488 | 75-69-4 |
| Vinyl chloride | 39175 | 75-01-4 |

1.2 The method may be extended to screen samples for acrolein (STORET No. 34210, CAS No. 107-02-8) and acrylonitrile (STORET No. 34215, CAS No. 107-13-1), however, the preferred method for these two compounds in Method 603.

1.3 This is a purge and trap gas chromatographic/mass spectrometer (GC/MS) method applicable to the determination of the compounds listed above in municipal

and industrial discharges as provided under 40 CFR 136.1.

1.4 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.5 Any modification to this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5. Depending upon the nature of the modification and the extent of intended use, the applicant may be required to demonstrate that the modifications will produce equivalent results when applied to relevant wastewaters.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the purgeables are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.[2,3]

*3. Interferences*

3.1 Impurities in the purge gas, organic compounds outgassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high purgeable levels, it may be necessary to wash the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105 ° C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

*4. Safety*

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4-6] for the information of the analyst.

4.2. The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with a hole in the center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicane (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for 1 h before use.

5.2 Purge and trap system—The purge and trap system consists of three separate pieces of equipment: a purging device, trap, and desorber. Several complete systems are now commercially available.

5.2.1 The purging device must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass though the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging device illustrated in Figure 1 meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. The trap must be packed to contain the following minimum lengths of adsorbents: 1.0 cm of methyl silicone coated packing (Section 6.3.2), 15 cm of 2,6-dyphenylene oxide polymer (Section 6.3.1), and 8 cm of silica gel (Section 6.3.3). The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber should be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 200 °C. The desorber illustrated in Figure 2 meets these design criteria.

5.2.4 The purge and trap system may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

5.3 GC/MS system:

5.3.1 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, and gases.

5.3.2 Column—6 ft long x 0.1 in ID stainless steel or glass, packed with 1% SP-1000 on Carbopack B (60/80 mesh) or equivalent. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 11.1.

5.3.3 Mass spectrometer—Capable of scanning from 20 to 260 amu every 7 s or less, utilizing 70 V (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the criteria in Table 2 when 50 ng of 4-bromofluorobenzene (BFB) is injected through the GC inlet.

5.3.4 GC/MS interface—Any GC to MS interface that gives acceptable calibration points at 50 ng or less per injection for each of the parameters of interest and achieves all acceptable performance criteria (Section 10) may be used. GC to MS interfaces constructed of all glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

5.3.5 Data system—A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for specific m/z (masses) and plotting such m/z abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits.

5.4 Syringes—5-mL, glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25-μL, 0.006 in. ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Syringe—5-mL, gas-tight with shut-off valve.

5.8 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighing 0.0001 g.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrasorb–300, Calgon Corp., or equivalent).

6.1.2 A water purification system (Millipore Super–Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for 1 h. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Trap materials:

6.3.1 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.3.2 Methyl silicone packing—3% OV–1 on Chromosorb-W (60/80 mesh) or equivalent.

6.3.3 Silica gel—35/60 mesh, Davison, grade-15 or equivalent.

6.4 Methanol—Pesticide quality or equivalent.

6.5 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the compounds, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.5.1 Place about 9.8 mL of methanol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.5.2 Add the assayed reference material:

6.5.2.1 Liquids—Using a 100-μL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.5.2.2 Gases—To prepare standards for any of the four halocarbons that boil below 30 °C (bromomethane, chloroethane, chloromethane, and vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid (the heavy gas will rapidly dissolve in the methanol).

6.5.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in μg/μL from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

6.5.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at −10 to −20 °C and protect from light.

6.5.5 Prepare fresh standards weekly for the four gases and 2-chloroethylvinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.

6.6 Secondary dilution standards—Using stock solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Section 7.3 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7 Surrogate standard spiking solution—Select a minimum of three surrogate compounds from Table 3. Prepare stock standard solutions for each surrogate standard in methanol as described in Section 6.5. Prepare a surrogate standard spiking solution from these stock standards at a concentration of 15 μg/mL in water. Store the solutions at 4 °C in Teflon-sealed glass containers with a minimum of headspace. The solutions should be checked frequently for stability. The addition of 10 μL of this solution of 5 mL of sample or standard is equivalent to a concentration of 30 μg/L of each surrogate standard.

6.8 BFB Standard—Prepare a 25 μg/mL solution of BFB in methanol.

6.9 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Assemble a purge and trap system that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Condition the trap for 10 min once daily prior to use.

7.2 Connect the purge and trap system to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate conditions equivalent to those given in Table 1.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select three or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Some recommended internal standards are listed in Table 3.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 μL of one or more secondary dilution standards to 50, 250, or 500 mL of reagent water. A 25-μL syringe with a 0.006 in. ID needle should be used for this operation. One of the calibration standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC/MS system. These aqueous standards can be stored up to 24 h, if held in sealed vials with zero headspace as described in Section 9.2. If not so stored, they must be discarded after 1 h.

7.3.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.5 and 6.6. It is recommended that the secondary dilution standard be prepared at a concentration of 15 μg/mL of each internal standard compound. The addition of 10 μL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 μg/L.

7.3.3 Analyze each calibration standard according to Section 11, adding 10 μL of internal standard spiking solution directly to the syringe (Section 11.4). Tabulate the area response of the characteristic m/z against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Area of the characteristic m/z for the parameter to be measured.

$A_{is}$ = Area of the characteristic m/z for the inernal standard.

$C_{is}$ = Concentration of the internal standard.

$C_s$ = Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<35% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.4 The working calibration curve or RF must be verified on each working day by the measurement of a QC check sample.

7.4.1 Prepare the QC check sample as described in Section 8.2.2.

7.4.2 Analyze the QC check sample according to the method beginning in Section 10.

7.4.3 For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in Table 5. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual Q falls outside the range, proceed according to Section 7.4.4.

**Note.**—The large number of parameters in Table 5 present a substantial probability that one or more will not meet the calibration

acceptance criteria when all parameters are analyzed.

7.4.4 Repeat the test only for those parameters that failed to meet the calibration acceptance criteria. If the response for a parameter does not fall within the range in this second test, a new calibration curve or RF must be prepared for that parameter according to Section 7.3.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.1 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 11.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Each day, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system are under control.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 5% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 5% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must spike all samples with surrogate standards to monitor continuing laboratory performance. This procedure is described in Section 8.5.

8.1.7 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.6.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 10 μg/mL in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Prepare a QC check sample to contain 20 μg/L of each parameter by adding 200 μL of QC check sample concentrate to 100 mL of reagent water.

8.2.3 Analyze four 5-mL aliquots of the well-mixed QC check sample according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter of interest using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 5. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter.

**Note.**—The large number of parameters in Table 5 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.3.

8.2.6.2 Beginning with Section 8.2.3, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.3.

8.3 The laboratory must, on an ongoing basis, spike at least 5% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing 1 to 20 samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 20 μg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.2 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second 5-mL sample aliquot with 10 μL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 5. These acceptance criteria wer calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than 20 μg/L, the analyst must use either the QC acceptance criteria in Table 5, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recoveryof a parameter: (1) calculate accuracy (X') using the equation in Table 6, substituting the spike concentration (T) for C; (2) calculate overall precision (S') using the equation in Table 6, substituting X' for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X'/T) (±2.44(100 S'/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

**Note.**—The frequency for the required anlaysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of parameters in Table 5 must be measured in the sample in Section 8.3, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spiked sample.

8.4.1 Prepare the QC check standard by adding 10 μL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 5 mL of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 5.

Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As a quality control check, the laboratory must spike all samples with the surrogate standard spiking solutions as described in Section 11.4, and calculate the percent recovery of each surrogate compound.

8.6 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter a regular basis (e.g. after each five to ten new accuracy measurements).

8.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

*9. Sample Collection, Preservation, and Handling*

9.1 All samples must be iced or refrigerated from the time of collection until analysis. If the sample contains residual chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm $Cl_2$) to the empty sample bottle just prior to shipping to the sampling site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[8] Field test kits are available for this purpose.

9.2 Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for 1 min. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 Experimental evidence indicates that some aromatic compounds, notably benzene, toluene, and ethyl benzene are susceptible to rapid biological degradation under certain environmental conditions.[3] Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days. For this reason, a separate sample should be collected, acidified, and analyzed when these aromatics are to be determined. Collect about 500 mL of sample in a clean container. Adjust the pH of the sample to about 2 by adding 1+1 HCl while stirring vigorously. Check pH with narrow range (1.4 to 2.8) pH paper. Fill a sample container as described in Section 9.2.

9.4 All samples must be analyzed within 14 days of collection.[3]

*10. Daily GC/MS Performance Tests*

10.1 At the beginning of each day that analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria are achieved for BFB.[9] The performance test must be passed before any samples, blanks, or standards are analyzed, unless the instrument has met the DFTPP test described in Method 625 earlier in the day.[10]

10.2 These performance tests require the following instrumental parameters:

Electron Energy: 70 V (nominal)
Mass Range: 20 to 260 amu
Scan Time: To give at least 5 scans per peak but not to exceed 7 s per scan.

10.3 At the beginning of each day, inject 2 μL of BFB solution directly on the column. Alternatively, add 2 μL of BFB solution to 5.0 mL of reagent water or standard solution and analyze the solution according to section 11. Obtain a background-corrected mass spectrum of BFB and confirm that all the key m/z criteria in Table 2 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved.

*11. Sample Purging and Gas Chromatography*

11.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. An example of the separations achieved by this column is shown in Figure 5. Other packed columns or chromatographic conditions may be used if the requirements of Section 8.2 are met.

11.2 After achieving the key m/z abundance criteria in Section 10, calibrate the system daiy as described in Section 7.

11.3 Adjust the purge gas (helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the purge and trap system to purge (Figure 3). Open the syringe valve located on the purging device sample introduction needle.

11.4 Allow the sample to come to ambient temperature prior to introducing it into the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 μL of the surrogate spiking solution (Section 6.7) and 10.0 μL of the internal standard spiking solution (Section 7.3.2) through the valve bore, then close the valve. The surrogate and internal standards may be mixed and added as a single spiking solution.

11.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

11.6 Close both valves and purge the sample for 11.0±0.1 min at ambient temperature.

11.7 After the 11-min purge time, attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode (Figure 4), and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for 4 min. If rapid heating of the trap cannot be achieved, the GC cloumn must be used as a secondary trap by cooling it to 30 °C (subambient temperature, if problems persist) instead of the initial program temperature of 45 °C.

11.8 While the trap is being desorbed into the gas chromatograph, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

11.9 After desorbing the sample for 4 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C. After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

11.10 If the response for any m/z exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

*12. Qualitative Identification*

12.1 Obtain EICPs for the primary m/z (Table 4) and at least two secondary masses for each parameter of interest. The following criteria must be met to make a qualitative identification:

12.1.1 The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.

12.1.2 The retention time must fall within ±30 s of the retention time of the authentic compound.

12.1.3 The relative peak heights of the three characteristic masses in the EICPs must fall within ± 20% of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library.

12.2 Structural isomers that have very similar mass spectra and less than 30 s difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

*13. Calculations*

13.1 When a parameter has been identified, the quantitation of that parameter should be based on the integrated abundance

from the EICP of the primary characteristic m/z given in Table 4. If the sample produces an interference for the primary m/z, use a secondary characteristic m/z quantitate.

Calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.3 and Equation 2.

Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})}{(A_{is})(RF)}$$

where:

$A_s$ = Area of the characteristic m/z for the parameter or surrogate standard to be measured.

$A_{is}$ = Area of the characteristic m/z for the internal standard.

$C_{is}$ = Concentration of the internal standard.

13.2 Report results in μg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

*14. Method Performance*

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[11] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method was tested by 15 laboratories using reagent water, drinking water, surface water, and industrial wastewaters spiked at six concentrations over the range 5–600 μg/L.[12] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 5.

**References**


1. 40 CFR Part 136, Appendix B.

2. Bellar, T.A., and Lichtenberg, J.J. "Determining Volatile Organics at Microgram-per-Litre Levels by Gas Chromatography," *Journal American Water Works Association, 66,* 739 (1974).

3. Bellar, T.A., and Lichtenberg, J.J. "Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds, " Measurement of Organic Pollutants in Water and Wastewater, C.E. Van Hall, editor, American Society for Testing and Materials, Philadelphia, PA. Special Technical Publication 686, 1978.

4. "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory, 15,* 58–63 (1983). (The value 2.44 used in the equation in Section 8.2.3 is two times the value 1.22 derived in this report.)

8. "Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

9. Budde, W.L., and Eichelberger, J.W. "Performance Tests for the Evaluation of Computerized Gas Chromatography/Mass Spectrometry Equipment and Laboratories," EPA–600/4–80–025, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, April 1980.

10. Eichelberger, J.W., Harris, L.E., and Budde, W.L. "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography—Mass Spectrometry Systems," *Analytical Chemistry, 47,* 995–1000 (1975).

11. "Method Detection Limit for Methods 624 and 625," Olynyk, P., Budde, W.L., and Eichelberger, J.W. Unpublished report, October 1980.

12. "Interlaboratory Method Study for EPA Method 624—Purgeables," Final Report for EPA Contract 68–03–3102.

13. "Method Performance Data for Method 624," Memorandum from R. Slater and T. Pressley, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, January 17, 1984.


TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

| Parameter | Retention time (min) | Method detection limit (μg/L) |
|---|---|---|
| Chloromethane | 2.3 | nd |
| Bromomethane | 3.1 | nd |
| Vinyl chloride | 3.8 | nd |
| Chloroethane | 4.6 | nd |
| Methylene chloride | 6.4 | 2.8 |
| Trichlorofluoromethane | 8.3 | nd |
| 1,1-Dichloroethene | 9.0 | 2.8 |
| 1,1-Dichloroethane | 10.1 | 4.7 |
| trans-1,2-Dichloroethene | 10.8 | 1.6 |
| Chloroform | 11.4 | 1.6 |
| 1,2-Dichloroethane | 12.1 | 2.8 |
| 1,1,1-Trichloroethane | 13.4 | 3.8 |
| Carbon tetrachloride | 13.7 | 2.8 |
| Bromodichloromethane | 14.3 | 2.2 |
| 1,2-Dichloroproane | 15.7 | 6.0 |
| cis-1,3-Dichloropropene | 15.9 | 5.0 |
| Trichloroethene | 16.5 | 1.9 |
| Benzene | 17.0 | 4.4 |
| Dibromochloromethane | 17.1 | 3.1 |
| 1,1,2-Trichloroethane | 17.2 | 5.0 |
| trans-1,3-Dichloropropene | 17.2 | nd |
| 2-Chloroethylvinyl ether | 18.6 | nd |
| Bromoform | 19.8 | 4.7 |
| 1,1,2,2-Tetrachloroethane | 22.1 | 6.9 |
| Tetrachloroethene | 22.2 | 4.1 |

TABLE 1.—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS—Continued

| Parameter | Retention time (min) | Method detection limit (μg/L) |
|---|---|---|
| Toluene | 23.5 | 6.0 |
| Chlorobenzene | 24.6 | 6.0 |
| Ethyl benzene | 26.4 | 7.2 |
| 1,3-Dichlorobenzene | 33.9 | nd |
| 1,2-Dichlorobenzene | 35.0 | nd |
| 1,4-Dichlorobenzene | 35.4 | nd |

Column conditions: Carbopak B (60/80 mesh) coated with 1% SP–1000 packed in a 6 ft by 0.1 in. ID glass column with helium carrier gas at 30 mL/min flow rate. Column temperature held at 45°C for 3 min, then programmed at 8°C/min to 220°C and held for 15 min.

nd = not determined.

TABLE 2.—BFB KEY M/Z ABUNDANCE CRITERIA

| Mass | m/z Abundance criteria |
|---|---|
| 50 | 15 to 40% of mass 95. |
| 75 | 30 to 60% of mass 95. |
| 95 | Base Peak, 100% Relative Abundance. |
| 96 | 5 to 9% of mass 95. |
| 173 | <2% of mass 174. |
| 174 | >50% of mass 95. |
| 175 | 5 to 9% of mass 174. |
| 176 | >95% but < 101% of mass 174. |
| 177 | 5 to 9% of mass 176. |

TABLE 3.—SUGGESTED SURROGATE AND INTERNAL STANDARDS

| Compound | Retention time (min)* | Primary m/z | Secondary masses |
|---|---|---|---|
| Benzene d-6 | 17.0 | 84 | |
| 4-Bromofluorobenzene | 28.3 | 95 | 174, 176 |
| 1,2-Dichloroethane d-4 | 12.1 | 102 | |
| 1,4-Difluorobenzene | 19.6 | 114 | 63, 88 |
| Ethylbenzene d-5 | 26.4 | 111 | |
| Ethylbenzene d-10 | 26.4 | 98 | |
| Fluorobenzene | 18.4 | 96 | 70 |
| Pentafluorobenzene | 23.5 | 168 | |
| Bromochloromethane | 9.3 | 128 | 49, 130, 51 |
| 2-Bromo-1-chloropropane | 19.2 | 77 | 79, 156 |
| 1, 4-Dichlorobutane | 25.8 | 55 | 90, 92 |

*For chromatographic conditions, see Table 1.

TABLE 4.—CHARACTERISTIC MASSES FOR PURGEABLE ORGANICS

| Parameter | Primary | Secondary |
|---|---|---|
| Chloromethane | 50 | 52. |
| Bromomethane | 94 | 96. |
| Vinyl chloride | 62 | 64. |
| Chloroethane | 64 | 66. |
| Methylene chloride | 84 | 49, 51, and 86. |
| Trichlorofluoromethane | 101 | 103. |
| 1,1-Dichloroethene | 96 | 61 and 98. |
| 1,1-Dichloroethene | 63 | 65, 83, 85, 98, and 100. |
| trans-1,2-Dichloroethene | 96 | 61 and 98. |
| Chloroform | 83 | 85. |
| 1,2-Dichloroethane | 98 | 62, 64, and 100. |
| 1,1,1-Trichloroethane | 97 | 99, 117, and 119. |
| Carbon tetrachloride | 117 | 119 and 121. |
| Bromodichloromethane | 127 | 83, 85, and 129. |
| 1,2-Dichloropropane | 112 | 63, 65, and 114. |
| trans-1,3-Dichloropropene | 75 | 77. |
| Trichloroethene | 130 | 95, 97, and 132. |
| Benzene | 78 | |
| Dibromochloromethane | 127 | 129, 208, and 206. |
| 1,1,2-Trichloroethane | 97 | 83, 85, 99, 132, and 134. |
| cis-1,3-Dichloropropene | 75 | 77. |
| 2-Chloroethylvinyl ether | 106 | 63 and 65. |

TABLE 4.—CHARACTERISTIC MASSES FOR PURGEABLE ORGANICS—Continued

| Parameter | Primary | Secondary |
|---|---|---|
| Bromoform | 173 | 171, 175, 250, 252, 254, and 256. |
| 1,1,2,2-Tetrachloroethane | 168 | 83, 85, 131, 133, and 166. |
| Tetrachloroethene | 164 | 129, 131, and 166. |
| Toluene | 92 | 91. |
| Chlorobenzene | 112 | 114. |
| Ethyl benzene | 106 | 91. |
| 1,3-Dichlorobenzene | 146 | 148 and 113. |
| 1,2-Dichlorobenzene | 146 | 148 and 113. |
| 1,4-Dichlorobenzene | 146 | 148 and 113. |

TABLE 5.—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 624[a]

| Parameter | Range for Q (μg/L) | Limit for s (μg/L) | Range for $\bar{X}$ (μg/L) | Range for P, $P_s$ (%) |
|---|---|---|---|---|
| Benzene | 12.8–27.2 | 6.9 | 15.2–26.0 | 37–151 |
| Bromodichloromethane | 13.1–26.9 | 6.4 | 10.1–28.0 | 35–155 |
| Bromoform | 14.2–25.8 | 5.4 | 11.4–31.1 | 45–169 |
| Bromomethane | 2.8–37.2 | 17.9 | D–41.2 | D–242 |
| Carbon tetrachloride | 14.6–25.4 | 5.2 | 17.2–23.5 | 70–140 |
| Chlorobenzene | 13.2–26.8 | 6.3 | 16.4–27.4 | 37–160 |
| Chloroethane | 7.6–32.4 | 11.4 | 8.4–40.4 | 14–230 |
| 2-Chloroethylvinyl ether | D–44.8 | 25.9 | D–50.4 | D–305 |
| Chloroform | 13.5–26.5 | 6.1 | 13.7–24.2 | 51–138 |
| Chloromethane | D–40.8 | 19.8 | D–45.9 | D–273 |
| Dibromochloromethane | 13.5–26.5 | 6.1 | 13.8–26.6 | 53–149 |
| 1,2-Dichlorobenzene | 12.6–27.4 | 7.1 | 11.8–34.7 | 18–190 |
| 1,3-Dichlorobenzene | 14.6–25.4 | 5.5 | 17.0–28.8 | 59–156 |
| 1,4-Dichlorobenzene | 12.6–27.4 | 7.1 | 11.8–34.7 | 18–190 |
| 1,1-Dichloroethane | 14.5–25.5 | 5.1 | 14.2–28.5 | 59–155 |
| 1,2-Dichloroethane | 13.6–26.4 | 6.0 | 14.3–27.4 | 49–155 |
| 1,1-Dichlorothene | 10.1–29.9 | 9.1 | 3.7–42.3 | D–234 |
| trans-1,2-Dichloroethene | 13.9–26.1 | 5.7 | 13.6–28.5 | 54–156 |
| 1,2-Dichloropropane | 6.8–33.2 | 13.8 | 3.8–36.2 | D–210 |
| cis-1,3-Dichloropropene | 4.8–35.2 | 15.8 | 1.0–39.0 | D–227 |
| trans-1,3-Dichloropropene | 10.0–30.0 | 10.4 | 7.6–32.4 | 17–183 |
| Ethyl benzene | 11.8–28.2 | 7.5 | 17.4–26.7 | 37–162 |
| Methylene chloride | 12.1–27.9 | 7.4 | D–41.0 | D–221 |
| 1,1,2,2-Tetrachloroethane | 12.1–27.9 | 7.4 | 13.5–27.2 | 46–157 |
| Tetrachloroethene | 14.7–25.3 | 5.0 | 17.0–26.6 | 64–148 |
| Toluene | 14.9–25.1 | 4.8 | 16.6–26.7 | 47–162 |
| 1,1,1-Trichloroethane | 15.0–25.0 | 4.6 | 13.7–30.1 | 52–162 |
| 1,1,2-Trichloroethane | 14.2–25.8 | 5.5 | 14.3–27.1 | 52–150 |
| Trichloroethane | 13.3–26.7 | 6.6 | 18.6–27.6 | 71–157 |
| Trichlorofluoromethene | 9.6–30.4 | 10.0 | 8.9–31.5 | 17–181 |
| Vinyl chloride | 0.8–39.2 | 20.0 | D–43.5 | D–251 |

Q = Concentration measured in QC check sample, in μg/L (Section 7.5.3).
s = Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).
$\bar{X}$ = Average recovery of four recovery measurements, in μg/L (Section 8.2.4).
P, $P_s$ = Percent recovery measured, (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.
[a] Criteria were calculated assuming a QC check sample concentration of 20 μg/L.

NOTE.—These criteria are based directly upon the method performance data in Table 6. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 6.

TABLE 6.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 624

| Parameter | Accuracy, as recovery, $\bar{X}$ (μg/L) | Single analyst precision, $s_r$ (μg/L) | Overall precision, S (μg/L) |
|---|---|---|---|
| Benzene | 0.93C+2.00 | 0.26$\bar{X}$–1.74 | 0.25$\bar{X}$–1.33 |
| Bromodichloromethane | 1.03C–1.58 | 0.15$\bar{X}$+0.59 | 0.20$\bar{X}$+1.13 |
| Bromoform | 1.18C–2.35 | 0.12$\bar{X}$+0.34 | 0.17$\bar{X}$+1.38 |
| Bromomethane[a] | 1.00C | 0.43$\bar{X}$ | 0.58$\bar{X}$ |
| Carbon tetrachloride | 1.10C–1.68 | 0.12$\bar{X}$+0.25 | 0.11$\bar{X}$+0.37 |
| Chlorobenzene | 0.98C+2.28 | 0.16$\bar{X}$–0.09 | 0.26$\bar{X}$–1.92 |
| Chloroethane | 1.18C+0.81 | 0.14$\bar{X}$+2.78 | 0.29$\bar{X}$+1.75 |
| 2-Chloroethylvinyl ether[a] | 1.00C | 0.62$\bar{X}$ | 0.84$\bar{X}$ |
| Chloroform | 0.93C+0.33 | 0.16$\bar{X}$+0.22 | 0.18$\bar{X}$+0.16 |
| Chloromethane | 1.03C–1.81 | 0.37$\bar{X}$+2.14 | 0.58$\bar{X}$+0.43 |
| Dibromochloromethane | 1.01C–0.03 | 0.17$\bar{X}$–0.18 | 0.17$\bar{X}$+0.49 |
| 1,2-Dichlorobenzene[b] | 0.94C+4.47 | 0.22$\bar{X}$–1.45 | 0.30$\bar{X}$–1.20 |
| 1,3-Dichlorobenzene | 1.06C+1.68 | 0.14$\bar{X}$–0.48 | 0.18$\bar{X}$–0.82 |
| 1,4-Dichlorobenzene[b] | 0.94C+4.47 | 0.22$\bar{X}$–1.45 | 0.30$\bar{X}$–1.20 |
| 1,1-Dichloroethane | 1.05C+0.36 | 0.13$\bar{X}$–0.05 | 0.16$\bar{X}$+0.47 |
| 1,2-Dichloroethane | 1.02C+0.45 | 0.17$\bar{X}$–0.32 | 0.21$\bar{X}$–0.38 |
| 1,1-Dichloroethene | 1.12C+0.61 | 0.17$\bar{X}$+1.06 | 0.43$\bar{X}$–0.22 |
| trans-1,2,-Dichloroethene | 1.05C+0.03 | 0.14$\bar{X}$+0.09 | 0.19$\bar{X}$+0.17 |
| 1,2-Dichloropropane[a] | 1.00C | 0.33$\bar{X}$ | 0.45$\bar{X}$ |
| cis-1,3-Dichloropropene[a] | 1.00C | 0.38$\bar{X}$ | 0.52$\bar{X}$ |
| trans-1,3-Dichloropropene[a] | 1.00C | 0.25$\bar{X}$ | 0.34$\bar{X}$ |
| Ethyl benzene | 0.98C+2.48 | 0.14$\bar{X}$+1.00 | 0.26$\bar{X}$–1.72 |
| Methylene chloride | 0.87C+1.88 | 0.15$\bar{X}$+1.07 | 0.32$\bar{X}$+4.00 |
| 1,1,2,2-Tetrachloroethane | 0.93C+1.76 | 0.16$\bar{X}$+0.69 | 0.20$\bar{X}$+0.41 |

TABLE 6.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 624—Continued

| Parameter | Accuracy, as recovery, $\bar{X}'$ (μg/L) | Single analyst precision, $s_r$ (μg/L) | Overall precision, $S'$ (μg/L) |
|---|---|---|---|
| Tetrachloroethene | 1.06C+0.80 | 0.13$\bar{X}$−0.18 | 0.16$\bar{X}$−0.45 |
| Toluene | 0.98C+2.03 | 0.15$\bar{X}$−0.71 | 0.22$\bar{X}$−1.71 |
| 1,1,1-Trichloroethane | 1.06C+0.73 | 0.12$\bar{X}$−0.15 | 0.21$\bar{X}$−0.39 |
| 1,1,2-Trichloroethane | 0.95+1.71 | 0.14$\bar{X}$+0.02 | 0.18$\bar{X}$+0.00 |
| Trichloroethene | 1.04C+2.27 | 0.13$\bar{X}$+0.36 | 0.12$\bar{X}$+0.59 |
| Trichlorofluoromethane | 0.99C+0.39 | 0.33$\bar{X}$−1.48 | 0.34$\bar{X}$−0.39 |
| Vinyl chloride | 1.00C | 0.48$\bar{X}$ | 0.65$\bar{X}$ |

$\bar{X}'$ = Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
$S'$ = Expected single analyst standard deviation of measurements at an average concentration found of$\bar{X}$, in μg/L.
S = Expected interlaboratory standard deviation of measurements at an average concentration found of$\bar{X}$, in μg/L.
C = True value for the concentration, in μg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in μg/L.
[a] Estimates based upon the performance in a single laboratory.[13]
[b] Due to chromatographic resolution problems, performance statements for these isomers are based upon the sums of their concentrations.

BILLING CODE 6560-50-M

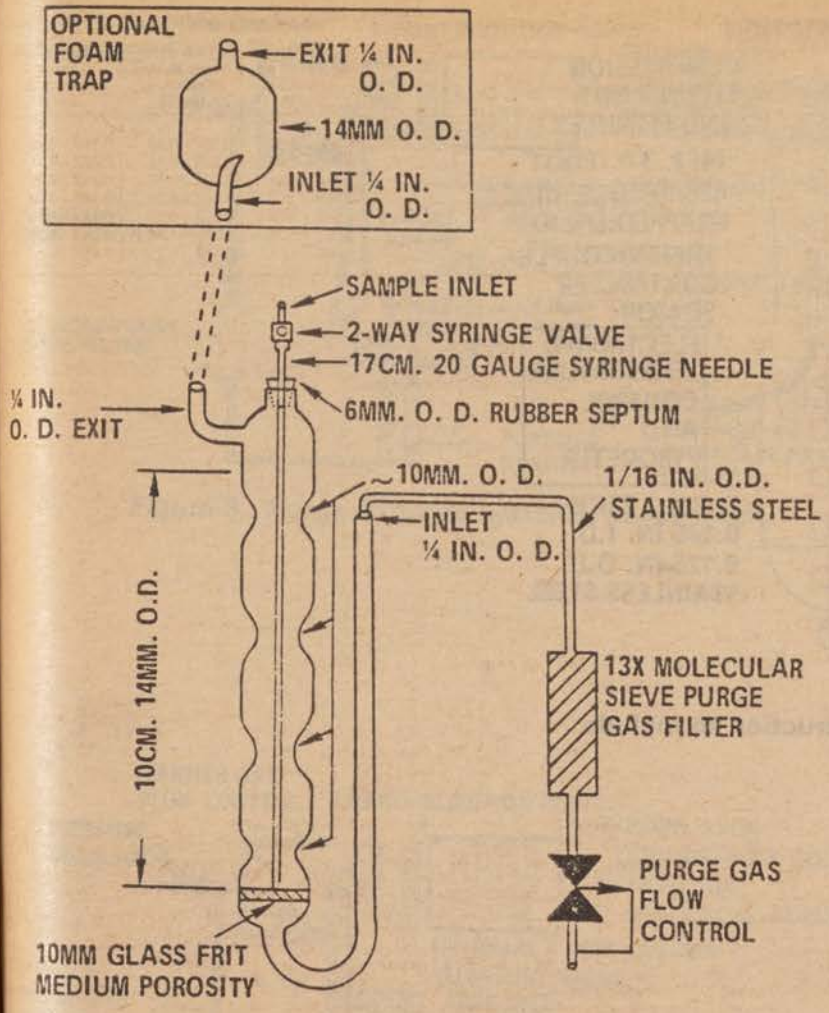



Figure 1. Purging device.

-704

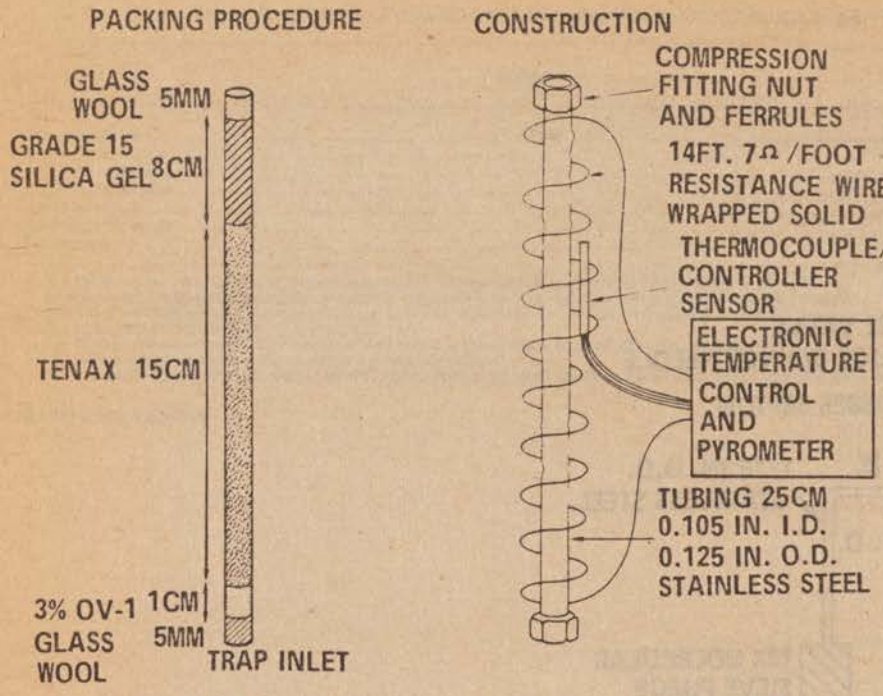



**Figure 2. Trap packings and construction to include desorb capability.**

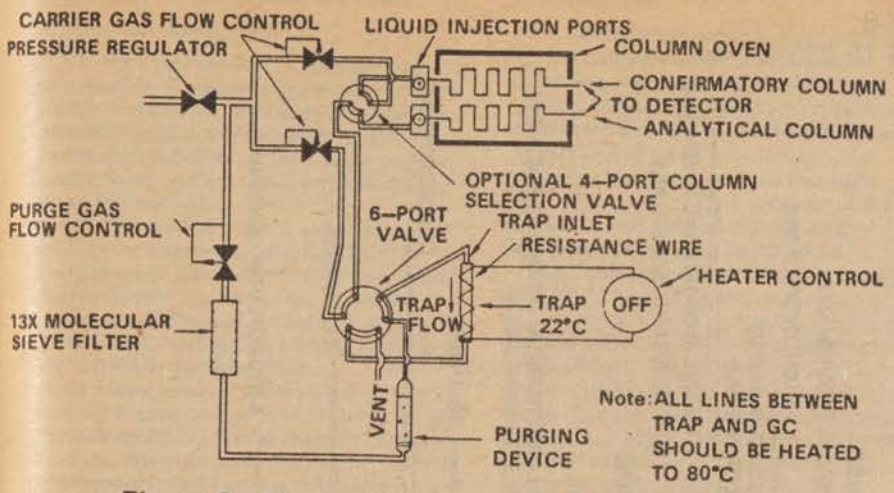



**Figure 3. Purge and trap system - purge mode.**

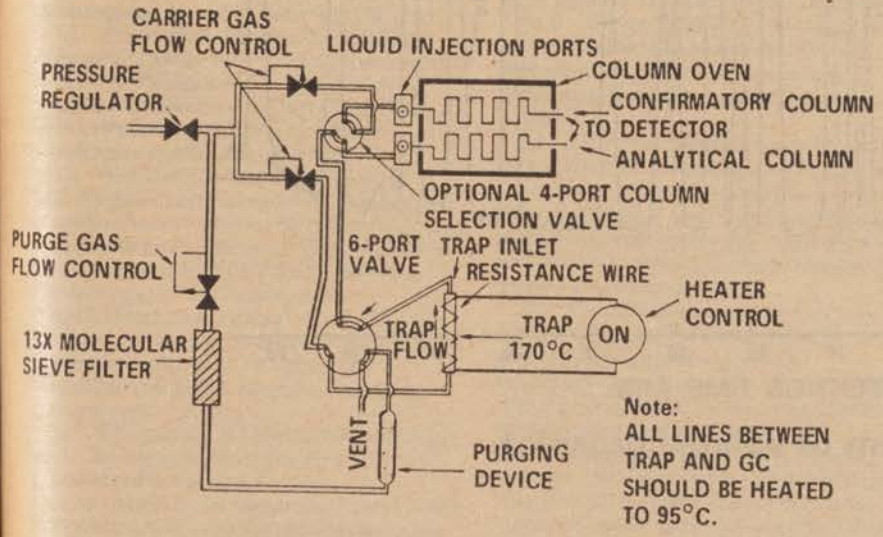



**Figure 4. Purge and trap system - desorb mode.**

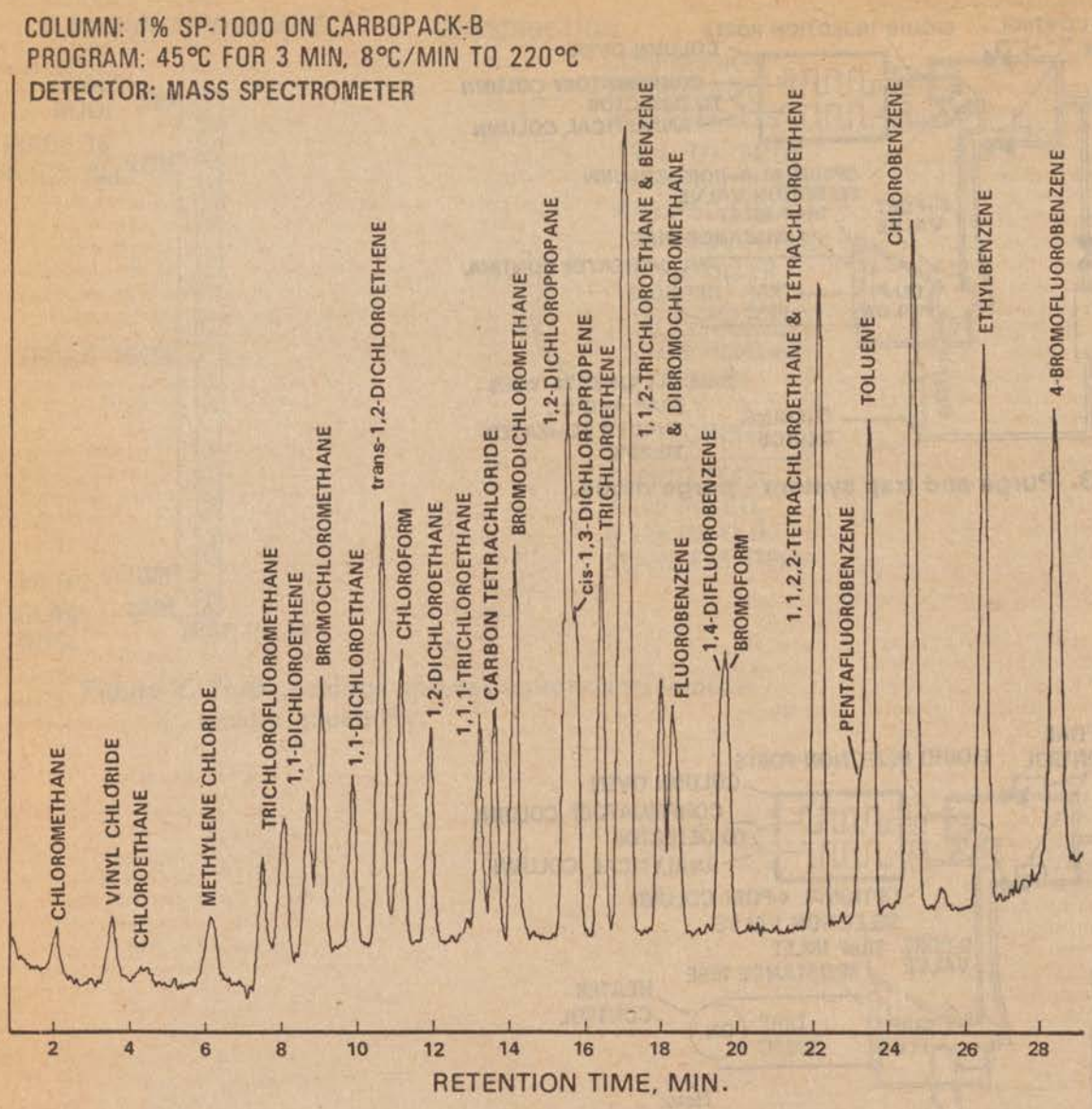



**Figure 5. Gas chromatogram of volatile organics.**

## Method 625—Base/Neutrals and Acids

*1. Scope and Application*

1.1 This method covers the determination of a number of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography. The parameters listed in Tables 1 and 2 may be qualitatively and quantitatively determined using this method.

1.2 The method may be extended to include the parameters listed in Table 3. Benzidine can be subject to oxidative losses during solvent concentration. Under the alkaline conditions of the extraction step, α-BHC, γ-BHC, endosulfan I and II, and endrin are subject to decomposition. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. The preferred method for each of these parameters is listed in Table 3.

1.3 This is a gas chromatographic/mass spectrometry (GC/MS) method applicable to the determination of the compounds listed in Tables 1, 2, and 3 in municipal and industrial discharges as provided under 40 CFR 136.1.

1.4 The method detection limit (MDL, defined in Section 16.1)[1] for each parameter is listed in Tables 4 and 5. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.5 Any modification to this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5. Depending upon the nature of the modification and the extent of intended use, the applicant may be required to demonstrate that the modifications will produce equivalent results when applied to relevant wastewaters.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

*2. Summary of Method*

2.1 A measured volume of sample, approximately 1-L, is serially extracted with methylene chloride at a pH greater than 11 and again at a pH less than 2 using a separatory funnel or a continuous extractor. The methylene chloride extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of three characteristic masses (m/z). Quantitative analysis is performed using either external or internal standard techniques with a single characteristic m/z.

*3. Interferences*

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled.

3.3 The base-neutral extraction may cause significantly reduced recovery of phenol, 2-methylphenol, and 2,4-dimethylphenol. The analyst must recognize that results obtained under these conditions are minimum concentrations.

3.4 The packed gas chromatographic columns recommended for the basic fraction may not exhibit sufficient resolution for certain isomeric pairs including the following: anthracene and phenanthrene; chrysene and benzo(a)anthracene; and benzo(b)fluoranthene and benzo(k)fluoranthene. The gas chromatographic retention time and mass spectra for these pairs of compounds are not sufficiently different to make an unambiguous identification. Alternative techniques should be used to identify and quantify these specific compounds, such as Method 610.

3.5 In samples that contain an inordinate number of interferences, the use of chemical ionization (CI) mass spectrometry may make identification easier. Tables 6 and 7 give characteristic CI ions for most of the compounds covered by this method. The use of CI mass spectrometry to support electron ionization (EI) mass spectrometry is encouraged but not required.

*4. Safety.*

4.1 The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [4-6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'-dichlorobenzidine, benzo(a)pyrene, α-BHC, β-BHC, δ-BHC, γ-BHC, dibenzo(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyls (PCBs). Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

*5. Apparatus and Materials*

5.1 Sampling equipment, for discrete or composit sampling.

5.1.1 Grab sample bottle—1-L or 1-gt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. before use, however, the compressible tubing should be throughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2-L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, 19 mm ID, with coarse frit filter disc.

5.2.3 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.4 Evaporative flask, Kuderna-Danish—500-mL (Kontes K–57001–0500 or equivalent). Attach to concentrator tube with springs.

5.2.5 Snyder column, Kuderna-Danish—Three all macro (Kontes K–503000–0121 or equivalent).

5.2.6 Snyder column, Kuderna-Danish—Two-ball macro (Kontes K–569001–0219 or equivalent).

5.2.7. Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.2.8 Continuous liquid—liquid extractor—Equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication. (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, N.J., P/N 6841–10 or equivalent.)

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min of Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2°C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 GC/MS system:

5.6.1 Gas Chromatograph—An analytical system complete with a temperature programmable gas chromatograph and all required accessores including syringes, analytical columns, and gases. The injection port must be designed for on-column injection when using packed columns and for splitless injection when using capillary columns.

5.6.2 Column for base/neutrals—1.8 m long x 2 mm ID glass, packed with 3% SP–2250 on Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statements in Section 16. Guidelines for the use of alternate column packings are provided in Section 13.1.

5.6.3 Column for acids—1.8 m long x 2 mm ID glass, packed with 1% SP–1240DA on Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statements in Section 16. Guidelines for the use of alternate column packings are given in Section 13.1.

5.6.4 Mass spectrometer—Capable of scanning from 35 to 450 amu every 7 s or less, utilizing a 70 V (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the criteria in Table 9 when 50 ng of decafluorotriphenyl phosphine (DFTPP; bis(perfluorophenyl) phenyl phosphine) is injected through the GC inlet.

5.6.5 GC/MS interface—Any GC to MS interface that gives acceptable calibration points at 50 ng per injection for each of the parameters of interest and achieves all acceptable performance criteria (Section 12) may be used. GC to MS interfaces constructed of all glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

5.6.6 Data system—A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for specific m/z and plotting such m/z abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits.

*6. Reagents*

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.3 Sodium thiosulfate—(ACS) Granular.

6.4 Sulfuric acid (1+1)—Slowly, add 50 mL of $H_2SO_4$ (ACS, sp. gr. 1.84) to 50 mL of reagent water.

6.5 Acetone, methanol, methlylene chloride—Pesticide quality or equivalent.

6.6 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray.

6.7 Stock standard solutions (1.00 µg/µL)—standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.7.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in pesticide quality acetone or other suitable solvent and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

6.7.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7.3 Stock standard solutions must be replaced after six months, or sooner if comparison with quality control check samples indicate a probelm.

6.8 Surrogate standard spiking solution—Select a minimum of three surrogate compounds from Table 8. Prepare a surrogate standard spiking solution containing each selected surrogate compound at a concentration of 100 µg/mL in acetone. Addition of 1.00 mL of this solution to 1000 mL of sample is equivalent to a concentration of 100 µg/L of each surrogate standard. Store the spiking solution at 4 °C in Teflon-sealed glass container. The solution should be checked frequently for stability. The solution must be replaced after six months, or sooner if comparison with quality control check standards indicates a problem.

6.9 DFTPP standard—Prepare a 25 µg/mL solution of DFTPP in acetone.

6.10 Quality control check sample concentrate—See Section 8.2.1.

*7. Calibration*

7.1 Establish gas chromatographic operating parameters equivalent to those indicated in Tables 4 or 5.

7.2 Internal standard calibration procedure—To use this approach, the analyst must select three or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standards is not affected by method or matrix interferences. Some recommended internal standards are listed in Table 8. Use the base peak m/z as the primary m/z for quantification of the standards. If interferences are noted, use one of the next two most intense masses for quantification.

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding appropriate volumes of one or more stock standards to a volumetric flask. To each calibration standard or standard mixture, add a known constant amount of one or more internal standards, and and dilute to volume with acetone. One of the calibration standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC/MS system.

7.2.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 13 and tabulate the area of the primary characteristic m/z (Tables 4 and 5) against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

$A_s$ = Area of the characteristic m/z for the parameter to be measured.

$A_{is}$ = Area of the characteristic m/z for the internal standard.

$C_{is}$ = Concentration of the internal standard (µg/L).

$C_s$ = Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (<35% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, $A_s/A_{is}$, vs. RF.

7.3 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±20%, the test must be repeated uning a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

*8. Quality Control*

8.1 Each laboratory that uses this method is required to operate a formal quality control

program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occuring in chromatography, the analyst is permitted certain options (detailed in Sections 10.6 and 13.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 5% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 5% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 100 μg/mL in acetone. Multiple solutions may be required. PCBs and multicomponent pesticides may be omitted from this test. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at a concentration of 100 μg/L by adding 1.00 mL of QC check sample concentrate to each of four 1-L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10 or 11.

8.2.4 Calculate the average recovery (X) in μg/L, and the standard deviation of the recovery (s) in μg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and X with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 6. If s and X for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual X falls outside the range for accuracy, the system performance is unacceptable for that parameter.

**Note.**—The large number of parameters in Table 6 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.2.6.2 Beginning with Section 8.2.2, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 5% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing 1 to 20 samples per month, at least one spiked sample per month is required.

8.3.1. The concentration of the spike in the sample should be determined as follows:

8.3.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 100 μg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or 100 μg/L.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 6. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than 100 μg/L, the analyst must use either the QC acceptance criteria in Table 6, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) calculate accuracy (X′) using the equation in Table 7, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 7, substituting X′ for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X′/T)±2.44(100 S′/T)%[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

**Note.**—The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of single-component parameters in Table 6 must be measured in the sample in Section 8.3, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spike sample.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery ($P_s$) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery ($P_s$) for each parameter with the corresponding QC acceptance criteria found in Table 6. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the

laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery ($s_p$). Express the accuracy assessment as a percent interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}=90\%$ and $s_p=10\%$, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 As a quality control check, the laboratory must spike all samples with the surrogate standard spiking solution as described in Section 10.2, and calculate the percent recovery of each surrogate compound.

8.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

*9. Sample Collection, Preservation, and Handling*

9.1 Grab samples must be collected in glass containers. Conventional sampling practices [8] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All sampling must be iced or refrigerated at 4 °C from the time of collection until extraction. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[9] Field test kits are available for this purpose.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.

*10. Separatory Funnel Extraction*

10.1 Samples are usually extracted using separatory funnel techniques. If emulsions will prevent achieving acceptable solvent recovery with separatory funnel extractions, continuous extraction (Section 11) may be used. The separatory funnel extraction scheme described below assumes a sample volume of 1 L. When sample volumes of 2 L are to be extracted, use 250, 100, and 100-mL volumes of methylene chloride for the serial extraction of the base/neutrals and 200, 100, and 100-mL volumes of methylene chloride for the acids.

10.2 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2-L separatory funnel. Pipet 1.00 mL of the surrogate standard spiking solution into the separatory funnel and mix well. Check the pH of the sample with wide-range pH paper and adjust to pH>11 with sodium hydroxide solution.

10.3 Add 60 mL of methylene chloride to the sample bottle, seal, and shake for 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask. If the emulsion cannot be broken (recovery of less than 80% of the methylene chloride, corrected for the water solubility of methylene chloride), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in Section 11.3.

10.4 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner. Label the combined extract as the base/neutral fraction.

10.5 Adjust the pH of the aqueous phase to less than 2 using sulfuric acid. Serially extract the acidified aqueous phase three times with 60-mL aliquots of methylene chloride. Collect and combine the extracts in a 250-mL Erlenmeyer flask and label the combined extracts as the acid fraction.

10.6 For each fraction, assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.7 For each fraction, pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.8 Add one or two clean boiling chips and attach a three-ball Snyder column to the evaporative flask for each fraction. Prewet each Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus from the water bath and allow it to drain and cool for at least 10 min. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of methylene chloride. A 5-mL syringe is recommended for this operation.

10.9 Add another one or two clean boiling chips to the concentrator tube for each fraction and attach a two-ball micro-Snyder column. Prewet the Snyder column by adding about 0.5 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches about 0.5 mL, remove the K–D apparatus from the water bath and allow it to drain and cool for at least 10 min. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with approximately 0.2 mL of acetone or methylene chloride. Adjust the final volume to 1.0 mL with the solvent. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extracts will be stored longer than two days, they should be transferred to Teflon-sealed screw-cap vials and labeled base/neutral or acid fraction as appropriate.

10.10 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

*11. Continuous Extraction*

11.1 When experience with a sample from a given source indicates that a serious emulsion problem will result or an emulsion is encountered using a separatory funnel in Section 10.3, a continuous extractor should be used.

11.2 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Check the pH of the sample with wide-range pH paper and adjust to pH >11 with sodium hydroxide solution. Transfer the sample to the continuous extractor and using a pipet, add 1.00 mL of surrogate standard spiking solution and mix well. Add 60 mL of methylene chloride to the sample bottle, seal, and shake for 30 s to rinse the inner surface. Transfer the solvent to the extractor.

11.3 Repeat the sample bottle rinse with an additional 50 to 100-mL portion of methylene chloride and add the rinse to the extractor.

11.4 Add 200 to 500 mL of methylene chloride to the distilling flask, add sufficient reagent water to ensure proper operation, and extract for 24 h. Allow to cool, then detach the distilling flask. Dry, concentrate,

and seal the extract as in Sections 10.6 through 10.9.

11.5 Charge a clean distilling flask with 500 mL of methylene chloride and attach it to the continuous extractor. Carefully, while stirring, adjust the pH of the aqueous phase to less than 2 using sulfuric acid. Extract for 24 h. Dry, concentrate, and seal the extract as in Sections 10.6 through 10.9.

*12. Daily GC/MS Performance Tests*

12.1 At the beginning of each day that analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria are achieved for DFTPP.[10] Each day that benzidine is to be determined, the tailing factor criterion described in Section 12.4 must be achieved. Each day that the acids are to be determined, the tailing factor criterion in Section 12.5 must be achieved.

12.2 These performance tests require the following instrumental parameters:

Electron Energy: 70 V (nominal)
Mass Range: 35 to 450 amu
Scan Time: To give at least 5 scans per peak but not to exceed 7 s per scan.

12.3 DFTPP performance test—At the beginning of each day, inject 2 μL (50 ng) of DFTPP standard solution. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 9 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed. The tailing factor tests in Sections 12.4 and 12.5 may be performed simultaneously with the DFTPP test.

12.4 Column performance test for base/neutrals—At the beginning of each day that the base/neutral fraction is to be analyzed for benzidine, the benzidine tailing factor must be calculated. Inject 100 ng of benzidine either separately or as a part of a standard mixture that may contain DFTPP and calculate the tailing factor. The benzidine tailing factor must be less than 3.0. Calculation of the tailing factor is illustrated in Figure 13.[11] Replace the column packing if the tailing factor criterion cannot be achieved.

12.5 Column performance test for acids—At the beginning of each day that the acids are to be determined, inject 50 ng of pentachlorophenol either separately or as a part of a standard mix that may contain DFTPP. The tailing factor for pentachlorophenol must be less than 5. Calculation of the tailing factor is illustrated in Figure 13.[11] Replace the column packing if the tailing factor criterion cannot be achieved.

*13. Gas Chromatography/Mass Spectrometry*

13.1 Table 4 summarizes the recommended gas chromatographic operating conditions for the base/neutral fraction. Table 5 summarizes the recommended gas chromatographic operating conditions for the acid fraction. Included in these tables are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by these columns are shown in Figures 1 through 12. Other packed or capillary (open-tubular) columns or chromatographic conditions may be used if the requirements of Section 8.2 are met.

13.2 After conducting the GC/MS performance tests in Section 12, calibrate the system daily as described in Section 7.

13.3 If the internal standard calibration procedure is being used, the internal standard must be added to sample extract and mixed thoroughly immediately before injection into the instrument. This procedure minimizes losses due to adsorption, chemical reaction or evaporation.

13.4 Inject 2 to 5 μL of the sample extract or standard into the GC/MS system using the solvent-flush technique.[12] Smaller (1.0 μL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 μL.

13.5 If the response for any m/z exceeds the working range of the GC/MS system, dilute the extract and reanalyze.

13.6 Perform all qualitative and quantitative measurements as described in Sections 14 and 15. When the extracts are not being used for analyses, store them refrigerated at 4°C, protected from light in screw-cap vials equipped with unpierced Teflon-lined septa.

*14. Qualitative Identification*

14.1 Obtain EICPs for the primary m/z and the two other masses listed in Tables 4 and 5. See Section 7.3 for masses to be used with internal and surrogate standards. The following criteria must be met to make a qualitative identification:

14.1.1 The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.

14.1.2 The retention time must fall within ±30 s of the retention time of the authentic compound.

14.1.3 The relative peak heights of the three characteristic masses in the EICPs must fall within ±20% of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library.

14.2 Structural isomers that have very similar mass spectra and less than 30 s difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

*15. Calculations*

15.1 When a parameter has been identified, the quantitation of that parameter will be based on the integrated abundance from the EICP of the primary characteristic m/z in Tables 4 and 5. Use the base peak m/z for internal and surrogate standards. If the sample produces an interference for the primary m/z, use a secondary characteristic m/z to quantitate.

Calculate the concentration in the sample using the response factor (RF) determined in Section 7.2.2 and Equation 3.

Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

$A_s$ = Area of the characteristic m/z for the parameter or surrogate standard to be measured.
$A_{is}$ = Area of the characteristic m/z for the internal standard.
$I_s$ = Amount of internal standard added to each extract (μg).
$V_o$ = Volume of water extracted (L).

15.2 Report results in μg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

*16. Method Performance*

16.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Tables 4 and 5 were obtained using reagent water.[13] The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

16.2 This method was tested by 15 laboratories using reagent water, drinking water, surface water, and industrial wastewaters spiked at six concentrations over the range 5 to 1300 μg/L.[14] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 7.

*17. Screening Procedure for 2,3,7,8-Tetrachlorodibenzo-p-dioxin (2,3,7,8,-TCDD)*

17.1 If the sample must be screened for the presence of 2,3,7,8-TCDD, it is recommended that the reference material not be handled in the laboratory unless extensive safety precautions are employed. It is sufficient to analyze the base/neutral extract by selected ion monitoring (SIM) GC/MS techniques, as follows:

17.1.1 Concentrate the base/neutral extract to a final volume of 0.2 ml.

17.1.2 Adjust the temperature of the base/neutral column (Section 5.6.2) to 220 °C.

17.1.3 Operate the mass spectrometer to acquire data in the SIM mode using the ions at m/z 257, 320 and 322 and a dwell time no greater than 333 milliseconds per mass.

17.1.4 Inject 5 to 7 μL of the base/neutral extract. Collect SIM data for a total of 10 min.

17.1.5 The possible presence of 2,3,7,8-TCDD is indicated if all three masses exhibit simultaneous peaks at any point in the selected ion current profiles.

17.1.6 For each occurrence where the possible presence of 2,3,7,8-TCDD is indicated, calculate and retain the relative abundances of each of the three masses.

17.2 False positives to this test may be caused by the presence of single or coeluting combinations of compounds whose mass spectra contain all of these masses.

17.3 Conclusive results of the presence and concentration level of 2,3,7,8-TCDD can

be obtained only from a properly equipped laboratory through the use of EPA Method 613 or other approved alternate test procedures.

**References**


1. 40 CFR Part 136, Appendix B.

2. "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants," U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1977, Revised April 1977. Available from Effluent Guidelines Division, Washington, DC 20460.

3. ASTM Annual Book of Standards, Part 31, D3694–78. "Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents," American Society for Testing and Materials, Philadelphia.

4. "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. "Interpretation of Percent Recovery Data," *American Laboratory*, 15, 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

8. ASTM Annual Book of Standards, Part 31, D3370–76. "Standard Practices for Sampling Water," American Society for Testing and Materials, Philadelphia.

9. "Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual," Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

10. Eichelberger, J.W., Harris, L.E., and Budde, W.L. "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectometry," *Analytical Chemistry*, 47, 995 (1975).

11. McNair, N.M. and Bonelli, E.J. "Basic Chromatography," Consolidated Printing, Berkeley, California, p. 52, 1969.

12. Burke, J.A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," *Journal of the Association of Official Analytical Chemists, 48*, 1037 (1965).

13. Olynyk, P., Budde, W.L., and Eichelberger, J.W. "Method Detection Limit for Methods 624 and 625," Unpublished report, October 1980.

14. "Interlaboratory Method Study for EPA Method 625—Base/Neutrals, Acids, and Pesticides," Final report for EPA Contract 68–03–3102 (In preparation).


TABLE 1.—BASE/NEUTRAL EXTRACTABLES

| Parameter | STORET No. | CAS No. |
|---|---|---|
| Acenaphthene | 34205 | 83-32-9 |
| Acenaphthylene | 34200 | 208-96-8 |
| Anthracene | 34220 | 120-12-7 |
| Aldrin | 39330 | 309-00-2 |
| Benzo(a)anthracene | 34526 | 56-55-3 |
| Benzo(b)fluoranthene | 34230 | 205-99-2 |
| Benzo(k)fluoranthene | 34242 | 207-08-9 |
| Benzo(a)pyrene | 34247 | 50-32-8 |
| Benzo(ghi)perylene | 34521 | 191-24-2 |
| Benzyl butyl phthalate | 34292 | 85-68-7 |
| β-BHC | 39338 | 319-85-7 |
| δ-BHC | 34259 | 319-86-8 |
| Bis(2-chloroethyl)ether | 34273 | 111-44-4 |
| Bis(2-chloroethoxy)methane | 34278 | 111-91-1 |
| Bis(2-ethylhexl)phthalate | 39100 | 117-81-7 |
| Bis(2-chloroisopropyl)ether | 34283 | 108-60-1 |
| 4-Bromophenyl phenyl ether | 34636 | 101-55-3 |
| Chlordane | 39350 | 57-74-9 |
| 2-Chloronaphthalene | 34581 | 91-58-7 |
| 4-Chlorophenyl phenyl ether | 34641 | 7005-72-3 |
| Chrysene | 34320 | 218-01-9 |
| 4,4'-DDD | 39310 | 72-54-8 |
| 4,4'-DDE | 39320 | 72-55-9 |
| 4,4'-DDT | 39300 | 50-29-3 |
| Dibenzo(a,h)anthracene | 34556 | 53-70-3 |
| Di-n-butylphthalate | 39110 | 84-74-2 |
| 1,3-Dichlorobenzene | 34566 | 541-73-1 |
| 1,2-Dichlorobenzene | 34536 | 95-50-1 |
| 1,4-Dichlorobenzene | 34571 | 106-46-7 |
| 3,3'-Dichlorobenzidine | 34631 | 91-94-1 |
| Dieldrin | 39380 | 60-57-1 |
| Diethyl phthalate | 34336 | 84-66-2 |
| Dimethyl phthalate | 34341 | 131-11-3 |
| 2,4-Dinitrotoluene | 34611 | 121-14-2 |
| 2,6-Dinitrotoluene | 34626 | 606-20-2 |
| Di-n-octylphthalate | 34596 | 117-84-0 |
| Endosulfan sulfate | 34351 | 1031-07-8 |
| Endrin aldehyde | 34366 | 7421-93-4 |
| Fluoranthene | 34376 | 206-44-0 |
| Fluorene | 34381 | 86-73-7 |
| Heptachlor | 39410 | 76-44-8 |
| Heptchlor epoxide | 39420 | 1024-57-3 |
| Hexachlorobenzene | 39700 | 118-74-1 |
| Hexachlorobutadiene | 34391 | 87-68-3 |
| Hexachloroethane | 34396 | 67-72-1 |
| Indeno(1,2,3-cd)pyrene | 34403 | 193-39-5 |
| Isophorone | 34408 | 78-59-1 |
| Naphthalene | 34696 | 91-20-3 |
| Nitrobenzene | 34447 | 98-95-3 |
| N-Nitrosodi-n-propylamine | 34428 | 621-64-7 |
| PCB-1016 | 34671 | 12674-11-2 |
| PCB-1221 | 39488 | 11104-28-2 |
| PCB-1232 | 39492 | 11141-16-5 |
| PCB-1242 | 39496 | 53469-21-9 |
| PCB-1248 | 39500 | 12672-29-6 |
| PCB-1254 | 39504 | 11097-69-1 |
| PCB-1260 | 39508 | 11096-82-5 |
| Phenanthrene | 34461 | 85-01-8 |
| Pyrene | 34469 | 129-00-0 |
| Toxaphene | 39400 | 8001-35-2 |
| 1,2,4-Trichlorobenzene | 34551 | 120-82-1 |

TABLE 2.—ACID EXTRACTABLES

| Parameter | STORET No. | CAS No. |
|---|---|---|
| 4-Chloro-3-methylphenol | 34452 | 59-50-7 |
| 2-Chlorophenol | 34586 | 95-57-8 |
| 2,4-Dichlorophenol | 34601 | 120-83-2 |
| 2,4-Dimethylphenol | 34606 | 105-67-9 |
| 2,4-Dinitrophenol | 34616 | 51-28-5 |
| 2-Methyl-4,6-dinitrophenol | 34657 | 534-52-1 |
| 2-Nitrophenol | 34591 | 88-75-5 |
| 4-Nitrophenol | 34646 | 100-02-7 |
| Pentachlorophenol | 39032 | 87-86-5 |
| Phenol | 34694 | 108-95-2 |
| 2,4,6-Trichlorophenol | 34621 | 88-06-2 |

TABLE 3.—ADDITIONAL EXTRACTABLE PARAMETERS*

| Parameter | STORET No. | CAS No. | Method |
|---|---|---|---|
| Benzidine | 39120 | 92-87-5 | 605 |
| β-BHC | 39337 | 319-84-6 | 608 |
| δ-BHC | 39340 | 58-89-8 | 608 |
| Endosulfan I | 34361 | 959-98-8 | 608 |
| Endosulfan II | 34356 | 33213-65-9 | 608 |
| Endrin | 39390 | 72-20-8 | 608 |
| Hexachlorocyclopentadiene | 34386 | 77-47-4 | 612 |
| N-Nitrosodimethylamine | 34438 | 62-75-9 | 607 |
| N-Nitrosodiphenylamine | 34433 | 86-30-6 | 607 |

* See Section 1.2.

TABLE 4.—CHROMATOGRAPHIC CONDITIONS, METHOD DETECTION LIMITS, AND CHARACTERISTIC MASSES FOR BASE/NEUTRAL EXTRACTABLES

| Parameter | Retention time (min) | Method detection limit (μg/L) | Characteristic masses | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | Electron impact | | | Chemical ionization | | |
| | | | Primary | Secondary | Secondary | Methane | Methane | Methane |
| 1,3-Dichlorobenzene | 7.4 | 1.9 | 146 | 148 | 113 | 146 | 148 | 150 |
| 1,4-Dichlorobenzene | 7.8 | 4.4 | 146 | 148 | 113 | 146 | 148 | 150 |
| Hexachloroethane | 8.4 | 1.6 | 117 | 201 | 199 | 199 | 201 | 203 |
| Bis(2-chloroethyl) ether | 8.4 | 5.7 | 93 | 63 | 95 | 63 | 107 | 109 |
| 1,2-Dichlorobenzene | 8.4 | 1.9 | 146 | 148 | 113 | 146 | 148 | 150 |
| Bis(2-chloroisopropyl) ether | 9.3 | 5.7 | 45 | 77 | 79 | 77 | 135 | 137 |
| N-Nitrosodi-n-propylamine | | | 130 | 42 | 101 | | | |
| Nitrobenzene | 11.1 | 1.9 | 77 | 123 | 65 | 124 | 152 | 164 |
| Hexachlorobutadiene | 11.4 | 0.9 | 225 | 223 | 227 | 223 | 225 | 227 |
| 1,2,4-Trichlorobenzene | 11.6 | 1.9 | 180 | 182 | 145 | 181 | 183 | 209 |
| Isophorone | 11.9 | 2.2 | 82 | 95 | 138 | 139 | 167 | 178 |
| Naphthalene | 12.1 | 1.6 | 128 | 129 | 127 | 129 | 157 | 169 |
| Bis(2-chloroethoxy) methane | 12.2 | 5.3 | 93 | 95 | 123 | 65 | 107 | 137 |
| Hexachlorocyclopentadiene* | 13.9 | | 237 | 235 | 272 | 235 | 237 | 239 |
| 2-Chloronaphthalene | 15.9 | 1.9 | 162 | 164 | 127 | 163 | 191 | 203 |
| Acenaphthylene | 17.4 | 3.5 | 152 | 151 | 153 | 152 | 153 | 181 |

TABLE 4.—CHROMATOGRAPHIC CONDITIONS, METHOD DETECTION LIMITS, AND CHARACTERISTIC MASSES FOR BASE/NEUTRAL EXTRACTABLES—Continued

| Parameter | Retention time (min) | Method detection limit (μg/L) | Characteristic masses | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | Electron impact | | | Chemical ionization | | |
| | | | Primary | Secondary | Secondary | Methane | Methane | Methane |
| Acenaphthene | 17.8 | 1.9 | 154 | 153 | 152 | 154 | 155 | 183 |
| Dimethyl phthalate | 18.3 | 1.6 | 163 | 194 | 164 | 151 | 163 | 164 |
| 2,6-Dinitrotoluene | 18.7 | 1.9 | 165 | 89 | 121 | 183 | 211 | 223 |
| Fluorene | 19.5 | 1.9 | 166 | 165 | 167 | 166 | 167 | 195 |
| 4-Chlorophenyl phenyl ether | 19.5 | 4.2 | 204 | 206 | 141 | | | |
| 2,4-Dinitrotoluene | 19.8 | 5.7 | 165 | 63 | 182 | 183 | 211 | 223 |
| Diethylphthalate | 20.1 | 1.9 | 149 | 177 | 150 | 177 | 223 | 251 |
| N-Nitrosodiphenylamine* | 20.5 | 1.9 | 169 | 168 | 167 | 169 | 170 | 198 |
| Hexachlorobenzene | 21.0 | 1.9 | 284 | 142 | 249 | 284 | 286 | 288 |
| β-BHC* | 21.1 | | 183 | 181 | 109 | | | |
| 4-Bromophenyl phenyl ether | 21.2 | 1.9 | 248 | 250 | 141 | 249 | 251 | 277 |
| δ-BHC* | 22.4 | | 183 | 181 | 109 | | | |
| Phenanthrene | 22.8 | 5.4 | 178 | 179 | 176 | 178 | 179 | 207 |
| Anthracene | 22.8 | 1.9 | 178 | 179 | 176 | 178 | 179 | 207 |
| β-BHC | 23.4 | 4.2 | 181 | 183 | 109 | | | |
| Heptachlor | 23.4 | 1.9 | 100 | 272 | 274 | | | |
| δ-BHC | 23.7 | 3.1 | 183 | 109 | 181 | | | |
| Aldrin | 24.0 | 1.9 | 66 | 263 | 220 | | | |
| Dibutyl phthalate | 24.7 | 2.5 | 149 | 150 | 104 | 149 | 205 | 279 |
| Heptachlor epoxide | 25.6 | 2.2 | 353 | 355 | 351 | | | |
| Endosulfan I* | 26.4 | | 237 | 338 | 341 | | | |
| Fluoranthene | 26.5 | 2.2 | 202 | 101 | 100 | 203 | 231 | 243 |
| Dieldrin | 27.2 | 2.5 | 79 | 263 | 279 | | | |
| 4,4'-DDE | 27.2 | 5.6 | 246 | 248 | 176 | | | |
| Pyrene | 27.3 | 1.9 | 202 | 101 | 100 | 203 | 231 | 243 |
| Endrin* | 27.9 | | 81 | 263 | 82 | | | |
| Endosulfan II* | 28.6 | | 237 | 339 | 341 | | | |
| 4,4'-DDD | 28.6 | 2.8 | 235 | 237 | 165 | | | |
| Benzidine* | 28.8 | 44 | 184 | 92 | 185 | 185 | 213 | 225 |
| 4,4'-DDT | 29.3 | 4.7 | 235 | 237 | 165 | | | |
| Endosulfan sulfate | 29.8 | 5.6 | 272 | 387 | 422 | | | |
| Endrin aldehyde | | | 67 | 345 | 250 | | | |
| Butyl benzyl phthalate | 29.9 | 2.5 | 149 | 91 | 206 | 149 | 299 | 327 |
| Bis(2-ethylhexyl) phthalate | 30.6 | 2.5 | 149 | 167 | 279 | 149 | | |
| Chrysene | 31.5 | 2.5 | 228 | 226 | 229 | 228 | 229 | 257 |
| Benzo(a)anthracene | 31.5 | 7.8 | 228 | 229 | 226 | 228 | 229 | 257 |
| 3,3'-Dichlorobenzidine | 32.2 | 16.5 | 252 | 254 | 126 | | | |
| Di-n-octyl phthalate | 32.5 | 2.5 | 149 | | | | | |
| Benzo(b)fluoranthene | 34.9 | 4.8 | 252 | 253 | 125 | 252 | 253 | 281 |
| Benzo(k)fluoranthene | 34.9 | 2.5 | 252 | 253 | 125 | 252 | 253 | 281 |
| Benzo(a)pyrene | 36.4 | 2.5 | 252 | 253 | 125 | 252 | 253 | 281 |
| Indeno(1,2,3-c,d)pyrene | 42.7 | 3.7 | 276 | 138 | 277 | 276 | 277 | 305 |
| Dibenzo(a,h)anthracene | 43.2 | 2.5 | 278 | 139 | 279 | 278 | 279 | 307 |
| Benzo(ghi)perylene | 45.1 | 4.1 | 276 | 138 | 277 | 276 | 277 | 305 |
| N-Nitrosodimethylamine* | | | 42 | 74 | 44 | | | |
| Chlordane[b] | 19–30 | | 373 | 375 | 377 | | | |
| Toxaphene[b] | 25–34 | | 159 | 231 | 233 | | | |
| PCB 1016[b] | 18–30 | | 224 | 260 | 294 | | | |
| PCB 1221[b] | 15–30 | 30 | 190 | 224 | 260 | | | |
| PCB 1232[b] | 15–32 | | 190 | 224 | 260 | | | |
| PCB 1242[b] | 15–32 | | 224 | 260 | 294 | | | |
| PCB 1248[b] | 12–34 | | 294 | 330 | 262 | | | |
| PCB 1254[b] | 22–34 | 36 | 294 | 330 | 362 | | | |
| PCB 1260[b] | 23–32 | | 330 | 362 | 394 | | | |

* See Section 1.2.
[b] These compounds are mixtures of various isomers. (See figures 2 thru 12.)

Column conditions: Supecoport (100/120 mesh) coated with 3% SP-2250 packed in a 1.8 m long × 2mm ID glass column with helium carrier gas at 30 mL/min flow rate. Column temperature held isothermal at 50 °C for 4 min, then programmed at 8 °C/min to 270 °C and held for 30 min.

TABLE 5.—CHROMATOGRAPHIC CONDITIONS, METHOD DETECTION LIMITS, AND CHARACTERISTIC MASSES FOR ACID EXTRACTABLES

| Parameter | Retention time (min) | Method detection limit (μg/L) | Characteristic masses | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | Electron impact | | | Chemical ionization | | |
| | | | Primary | Secondary | Secondary | Methane | Methane | Methane |
| 2-Chlorophenol | 5.9 | 3.3 | 128 | 64 | 130 | 129 | 131 | 157 |
| 2-Nitrophenol | 6.5 | 3.6 | 139 | 65 | 109 | 140 | 168 | 122 |
| Phenol | 8.0 | 1.5 | 94 | 65 | 66 | 95 | 123 | 135 |
| 2,4-Dimethylphenol | 9.4 | 2.7 | 122 | 107 | 121 | 123 | 151 | 163 |
| 2,4-Dichlorophenol | 9.8 | 2.7 | 162 | 164 | 98 | 163 | 165 | 167 |
| 2,4,6-Trichlorophenol | 11.8 | 2.7 | 196 | 198 | 200 | 197 | 199 | 201 |
| 4-Chloro-3-methylphenol | 13.2 | 3.0 | 142 | 107 | 144 | 143 | 171 | 183 |
| 2,4-Dinitrophenol | 15.9 | 42 | 184 | 63 | 154 | 185 | 213 | 225 |
| 2-Methyl-4,6-dinitrophenol | 16.2 | 24 | 198 | 182 | 77 | 199 | 227 | 239 |
| Pentachlorophenol | 17.5 | 3.6 | 266 | 264 | 268 | 267 | 265 | 269 |
| 4-Nitrophenol | 20.3 | 2.4 | 65 | 139 | 109 | 140 | 168 | 122 |

Column conditions: Supelcoport (100/120 mesh) coated with 1% SP-1240DA packed in a 1.8 m long x 2mm ID glass column with helium carrier gas at 30 mL/min flow rate. Column temperature held isothermal at 70 °C for 2 min then programmed at 8 °C/min to 200 °C.

TABLE 6.—QC ACCEPTANCE CRITERIA—METHOD 625

| Parameter | Test conclusion (µg/L) | Limits for s (µg/L) | Range for $\bar{X}$ (µg/L) | Range for P, $P_s$ (Percent) |
|---|---|---|---|---|
| Acenaphthene | 100 | 27.6 | 60.1–132.3 | 47–145 |
| Acenaphthylene | 100 | 40.2 | 53.5–126.0 | 33–145 |
| Aldrin | 100 | 39.0 | 7.2–152.2 | D–166 |
| Anthracene | 100 | 32.0 | 43.4–118.0 | 27–133 |
| Benzo(a)anthracene | 100 | 27.6 | 41.8–133.0 | 33–143 |
| Benzo(b)fluoranthene | 100 | 38.8 | 42.0–140.4 | 24–159 |
| Benzo(k)fluoranthene | 100 | 32.3 | 25.2–145.7 | 11–162 |
| Benzo(a)pyrene | 100 | 39.0 | 31.7–148.0 | 17–163 |
| Benzo(ghi)perylene | 100 | 58.9 | D–195.0 | D–219 |
| Benzyl butyl phthalate | 100 | 23.4 | D–139.9 | D–152 |
| β-BHC | 100 | 31.5 | 41.5–130.6 | 24–149 |
| δ-BHC | 100 | 21.6 | D–100.0 | D–110 |
| Bis(2-chloroethyl)ether | 100 | 55.0 | 42.9–126.0 | 12–158 |
| Bis(2-chloroethoxy)methane | 100 | 34.5 | 49.2–164.7 | 33–184 |
| Bis(2-chloroisopropyl)ether | 100 | 46.3 | 62.8–138.6 | 36–166 |
| Bis(2-ethylhexyl)phthalate | 100 | 41.1 | 28.9–136.8 | 8–158 |
| 4-Bromophenyl phenyl ether | 100 | 23.0 | 64.9–114.4 | 53–127 |
| 2-Chloronaphthalene | 100 | 13.0 | 64.5–113.5 | 60–118 |
| 4-Chlorophenyl phenyl ether | 100 | 33.4 | 38.4–144.7 | 25–158 |
| Chrysene | 100 | 48.3 | 44.1–139.9 | 17–168 |
| 4,4'-DDD | 100 | 31.0 | D–134.5 | D–145 |
| 4,4'-DDE | 100 | 32.0 | 19.2–119.7 | 4–136 |
| 4,4'-DDT | 100 | 61.8 | D–170.6 | D–203 |
| Dibenzo(a,h)anthracene | 100 | 70.0 | D–199.7 | D–227 |
| Di-n-butyl phthalate | 100 | 16.7 | 8.4–111.0 | 1–118 |
| 1,2-Dichlorobenzene | 100 | 30.9 | 48.6–112.0 | 32–129 |
| 1,3-Dichlorobenzene | 100 | 41.7 | 16.7–153.9 | D–172 |
| 1,4,-Dichlorobenzene | 100 | 32.1 | 37.3–105.7 | 20–124 |
| 3,3'-Dichlorobenzidine | 100 | 71.4 | 8.2–212.5 | D–262 |
| Dieldrin | 100 | 30.7 | 44.3–119.3 | 29–136 |
| Diethyl phthalate | 100 | 26.5 | D–100.0 | D–114 |
| Dimethyl phthalate | 100 | 23.2 | D–100.0 | D–112 |
| 2,4-Dinitrotoluene | 100 | 21.8 | 47.5–126.9 | 39–139 |
| 2,6-Dinitrotoluene | 100 | 29.6 | 68.1–136.7 | 50–158 |
| Di-n-octylphthalate | 100 | 31.4 | 18.6–131.8 | 4–146 |
| Endosulfan sulfate | 100 | 16.7 | D–103.5 | D–107 |
| Endrin aldehyde | 100 | 32.5 | D–186.8 | D–209 |
| Fluoranthene | 100 | 32.8 | 42.9–121.3 | 26–137 |
| Fluorene | 100 | 20.7 | 71.6–106.4 | 59–121 |
| Heptachlor | 100 | 37.2 | D–172.2 | D–192 |
| Heptachlor epoxide | 100 | 54.7 | 70.9–109.4 | 26–155 |
| Hexachlorobenzene | 100 | 24.9 | 7.8–141.5 | D–152 |
| Hexachlorobutadiene | 100 | 26.3 | 37.8–102.2 | 24–116 |
| Hexachloroethane | 100 | 24.5 | 55.2–100.0 | 40–113 |
| Indeno(1,2,3-cd)pyrene | 100 | 44.6 | D–150.9 | D–171 |
| Isophorone | 100 | 63.3 | 46.6–180.2 | 21–196 |
| Naphthalene | 100 | 30.1 | 35.6–119.6 | 21–133 |
| Nitrobenzene | 100 | 39.3 | 54.3–157.6 | 35–180 |
| N-Nitrosodi-n-propylamine | 100 | 55.4 | 13.6–197.9 | D–230 |
| PCB-1260 | 100 | 54.2 | 19.3–121.0 | D–164 |
| Phenanthrene | 100 | 20.6 | 65.2–108.7 | 54–120 |
| Pyrene | 100 | 25.2 | 69.6–100.0 | 52–115 |
| 1,2,4-Trichlorobenzene | 100 | 28.1 | 57.3–129.2 | 44–142 |
| 4-Chloro-3-methylphenol | 100 | 37.2 | 40.8–127.9 | 22–147 |
| 2-Chlorophenol | 100 | 28.7 | 36.2–120.4 | 23–134 |
| 2,4-Dichlorophenol | 100 | 26.4 | 52.5–121.7 | 39–135 |
| 2,4-Dimethylphenol | 100 | 26.1 | 41.8–109.0 | 32–119 |
| 2,4-Dinitrophenol | 100 | 49.8 | D–172.9 | D–191 |
| 2-Methyl-4,6-dinitrophenol | 100 | 93.2 | 53.0–100.0 | D–181 |
| 2-Nitrophenol | 100 | 35.2 | 45.0–166.7 | 29–182 |
| 4-Nitrophenol | 100 | 47.2 | 13.0–106.5 | D–132 |
| Pentachlorophenol | 100 | 48.9 | 38.1–151.8 | 14–176 |
| Phenol | 100 | 22.6 | 16.6–100.0 | 5–112 |
| 2,4,6-Trichlorophenol | 100 | 31.7 | 52.4–129.2 | 37–144 |

s = Standard deviation for four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$ = Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P, $P_s$ = Percent recovery measured (Section 8.3.2, Section 8.4.2).
D = Detected; result must be greater than zero.

**Note:** These criteria are based directly upon the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limts to concentrations below those used to develop Table 7

TABLE 7. METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 625

| Parameter | Accuracy, as recovery, $\bar{X}'$ (µg/L) | Single analyst precision, $S_r'$ (µg/L) | Overall precision, $S'$ (µg/L) |
|---|---|---|---|
| Acenaphthene | 0.96C+0.19 | $0.15\bar{X}-0.12$ | $0.21\bar{X}-0.67$ |
| Acenaphthylene | 0.89C+0.74 | $0.24\bar{X}-1.06$ | $0.26\bar{X}-0.54$ |
| Aldrin | 0.78C+1.66 | $0.27\bar{X}-1.28$ | $0.43\bar{X}+1.13$ |
| Anthracene | 0.80C+0.68 | $0.21\bar{X}-0.32$ | $0.27\bar{X}-0.64$ |
| Benzo(a)anthracene | 0.88C−0.60 | $0.15\bar{X}+0.93$ | $0.26\bar{X}-0.28$ |
| Benzo(b)fluoranthene | 0.93C−1.80 | $0.22\bar{X}+0.43$ | $0.29\bar{X}+0.96$ |
| Benzo(k)fluoranthene | 0.67C−1.56 | $0.19\bar{X}+1.03$ | $0.35\bar{X}+0.40$ |
| Benzo(a)pyrene | 0.90C−0.13 | $0.22\bar{X}+0.48$ | $0.32\bar{X}+1.35$ |
| Benzo(ghi)perylene | 0.98C−0.86 | $0.29\bar{X}+2.40$ | $0.51\bar{X}-0.44$ |
| Benzyl butyl phthalate | 0.66C−1.68 | $0.18\bar{X}+0.94$ | $0.53\bar{X}+0.92$ |
| β-BHC | 0.87C−0.94 | $0.20\bar{X}-0.58$ | $0.30\bar{X}-1.94$ |
| δ-BHC | 0.29C−1.09 | $0.34\bar{X}+0.86$ | $0.93\bar{X}-0.17$ |
| Bis(2-chloroethyl)ether | 0.86C−1.54 | $0.35\bar{X}-0.99$ | $0.35\bar{X}+0.10$ |

TABLE 7. METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 625—Continued

| Parameter | Accuracy, as recovery, $X'$ (µg/L) | Single analyst precision, $S_r'$ (µg/L) | Overall precision, $S'$ (µg/L) |
|---|---|---|---|
| Bis(2-chloroethoxy)methane | 1.12C−5.04 | 0.16$\bar{X}$+1.34 | 0.26$\bar{X}$+2.01 |
| Bis(2-chloroisopropyl)ether | 1.03C−2.31 | 0.24$\bar{X}$+0.28 | 0.25$\bar{X}$+1.04 |
| Bis(2-ethylhexyl)phthalate | 0.84C−1.18 | 0.26$\bar{X}$+0.73 | 0.36$\bar{X}$+0.67 |
| 4-Bromophenyl phenyl ether | 0.91C−1.34 | 0.13$\bar{X}$+0.66 | 0.16$\bar{X}$+0.66 |
| 2-Chloronaphthalene | 0.89C+0.01 | 0.07$\bar{X}$+0.52 | 0.13$\bar{X}$+0.34 |
| 4-Chlorophenyl phenyl ether | 0.91C+0.53 | 0.20$\bar{X}$−0.94 | 0.30$\bar{X}$−0.46 |
| Chrysene | 0.93C−1.00 | 0.28$\bar{X}$+0.13 | 0.33$\bar{X}$−0.09 |
| 4,4'-DDD | 0.56C−0.40 | 0.29$\bar{X}$−0.32 | 0.66$\bar{X}$−0.96 |
| 4,4'-DDE | 0.70C−0.54 | 0.26$\bar{X}$−1.17 | 0.39$\bar{X}$−1.04 |
| 4,4'-DDT | 0.79C−3.28 | 0.42$\bar{X}$+0.19 | 0.65$\bar{X}$−0.58 |
| Dibenzo(a,h)anthracene | 0.88C+4.72 | 0.30$\bar{X}$+8.51 | 0.59$\bar{X}$+0.25 |
| Di-n-butyl phthalate | 0.59C+0.71 | 0.13$\bar{X}$+1.16 | 0.39$\bar{X}$+0.60 |
| 1,2-Dichlorobenzene | 0.80C+0.28 | 0.20$\bar{X}$+0.47 | 0.24$\bar{X}$+0.39 |
| 1,3-Dichlorobenzene | 0.86C−0.70 | 0.25$\bar{X}$+0.68 | 0.41$\bar{X}$+0.11 |
| 1,4-Dichlorobenzene | 0.73C−1.47 | 0.24$\bar{X}$+0.23 | 0.29$\bar{X}$+0.36 |
| 3,3'-Dichlorobenzidine | 1.23C−12.65 | 0.28$\bar{X}$+7.33 | 0.47$\bar{X}$+3.45 |
| Dieldrin | 0.82C−0.16 | 0.20$\bar{X}$−0.16 | 0.26$\bar{X}$−0.07 |
| Diethyl phthalate | 0.43C+1.00 | 0.28$\bar{X}$+1.44 | 0.52$\bar{X}$+0.22 |
| Dimethyl phthalate | 0.20C+1.03 | 0.54$\bar{X}$+0.19 | 1.05$\bar{X}$−0.92 |
| 2,4-Dinitrotoluene | 0.92C−4.81 | 0.12$\bar{X}$+1.06 | 0.21$\bar{X}$+1.50 |
| 2,6-Dinitrotoluene | 1.06C−3.60 | 0.14$\bar{X}$+1.26 | 0.19$\bar{X}$+0.35 |
| Di-n-octylphthalate | 0.76C−0.79 | 0.21$\bar{X}$+1.19 | 0.37$\bar{X}$+1.19 |
| Endosulfan sulfate | 0.39C+0.41 | 0.12$\bar{X}$+2.47 | 0.63$\bar{X}$−1.03 |
| Endrin aldehyde | 0.76C−3.86 | 0.18$\bar{X}$+3.91 | 0.73$\bar{X}$−0.62 |
| Fluoranthene | 0.81C+1.10 | 0.22$\bar{X}$−0.73 | 0.28$\bar{X}$−0.60 |
| Fluorene | 0.90C−0.00 | 0.12$\bar{X}$+0.26 | 0.13$\bar{X}$+0.61 |
| Heptachlor | 0.87C−2.97 | 0.24$\bar{X}$−0.56 | 0.50$\bar{X}$−0.23 |
| Heptachlor epoxide | 0.92C−1.87 | 0.33$\bar{X}$−0.46 | 0.28$\bar{X}$+0.64 |
| Hexachlorobenzene | 0.74C+0.66 | 0.18$\bar{X}$−0.10 | 0.43$\bar{X}$−0.52 |
| Hexachlorobutadiene | 0.71C−1.01 | 0.19$\bar{X}$+0.92 | 0.26$\bar{X}$+0.49 |
| Hexachloroethane | 0.73C−0.83 | 0.17$\bar{X}$+0.67 | 0.17$\bar{X}$+0.80 |
| Indeno(1,2,3-cd)pyrene | 0.78C−3.10 | 0.29$\bar{X}$+1.46 | 0.50$\bar{X}$+0.44 |
| Isophorone | 1.12C+1.41 | 0.27$\bar{X}$+0.77 | 0.33$\bar{X}$+0.26 |
| Naphthalene | 0.76C+1.58 | 0.21$\bar{X}$−0.41 | 0.30$\bar{X}$−0.68 |
| Nitrobenzene | 1.09C−3.05 | 0.19$\bar{X}$+0.92 | 0.27$\bar{X}$+0.21 |
| N-Nitrosodi-n-propylamine | 1.12C−6.22 | 0.27$\bar{X}$+0.68 | 0.44$\bar{X}$+0.47 |
| PCB-1260 | 0.81C−10.86 | 0.35$\bar{X}$+3.61 | 0.43$\bar{X}$+1.82 |
| Phenanthrene | 0.87C−0.06 | 0.12$\bar{X}$+0.57 | 0.15$\bar{X}$+0.25 |
| Pyrene | 0.84C−0.16 | 0.16$\bar{X}$+0.06 | 0.15$\bar{X}$+0.31 |
| 1,2,4-Trichlorobenzene | 0.94C−0.79 | 0.15$\bar{X}$+0.85 | 0.21$\bar{X}$+0.39 |
| 4-Chloro-3-methylphenol | 0.84C+0.35 | 0.23$\bar{X}$+0.75 | 0.29$\bar{X}$+1.31 |
| 2-Chlorophenol | 0.78C+0.29 | 0.18$\bar{X}$+1.46 | 0.28$\bar{X}$+0.97 |
| 2,4-Dichlorophenol | 0.87C+0.13 | 0.15$\bar{X}$+1.25 | 0.21$\bar{X}$+1.28 |
| 2,4-Dimethylphenol | 0.71C+4.41 | 0.16$\bar{X}$+1.21 | 0.22$\bar{X}$+1.31 |
| 2,4-Dinitrophenol | 0.81C−18.04 | 0.38$\bar{X}$+2.36 | 0.42$\bar{X}$+26.29 |
| 2-Methyl-4,6-dinitrophenol | 1.04C−28.04 | 0.10$\bar{X}$+42.29 | 0.26$\bar{X}$+23.10 |
| 2-Nitrophenol | 1.07C−1.15 | 0.16$\bar{X}$+1.94 | 0.27$\bar{X}$+2.60 |
| 4-Nitrophenol | 0.61C−1.22 | 0.38$\bar{X}$+2.57 | 0.44$\bar{X}$+3.24 |
| Pentachlorophenol | 0.93C+1.99 | 0.24$\bar{X}$+3.03 | 0.30$\bar{X}$+4.33 |
| Phenol | 0.43C+1.26 | 0.26$\bar{X}$+0.73 | 0.35$\bar{X}$+0.58 |
| 2,4,6-Trichlorophenol | 0.91C−0.18 | 0.16$\bar{X}$+2.22 | 0.22$\bar{X}$+1.81 |

$X'$ = Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.
$s_r'$ = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
$S'$ = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
C = True value for the concentration, in µg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in µg/L.

TABLE 8.—SUGGESTED INTERNAL AND SURROGATE STANDARDS

| Base/neutral fraction | Acid fraction |
|---|---|
| Aniline-$d_5$ | 2-Fluorophenol. |
| Anthracene-$d_{10}$ | Pentafluorophenol. |
| Benzo(a)anthracene-$d_{12}$ | Phenol-$d_5$ |
| 4,4'-Dibromobiphenyl | 2-Perfluoromethyl phenol. |
| 4,4'-Dibromooctafluorobiphenyl. | |
| Decafluorobiphenyl | |
| 2,2'-Difluorobiphenyl | |
| 4-Fluoroaniline | |
| 1-Fluoronaphthylene | |
| 2-Fluoronaphthylene | |
| Naphthalene-$d_8$ | |
| Nitrobenzene-$d_5$ | |
| 2,3,4,5,6-Pentafluorobiphenyl | |
| Phenanthrene-$d_{10}$ | |
| Pyridine-$d_5$ | |

TABLE 9.—DFTPP KEY MASSES AND ABUNDANCE CRITERIA

| Mass | m/z Abundance criteria |
|---|---|
| 51 | 30–60 percent of mass 198. |
| 68 | Less than 2 percent of mass 69. |
| 70 | Less than 2 percent of mass 69. |
| 127 | 40–60 percent of mass 198. |
| 197 | Less than 1 percent of mass 198. |
| 198 | Base peak, 100 percent relative abundance. |
| 199 | 5–9 percent of mass 198. |
| 275 | 10–30 percent of mass 198. |
| 365 | Greater than 1 percent of mass 198. |
| 441 | Present but less than mass 443. |
| 442 | Greater than 40 percent of mass 198. |
| 443 | 17–23 percent of mass 442 |

BILLING CODE 6560-50-M

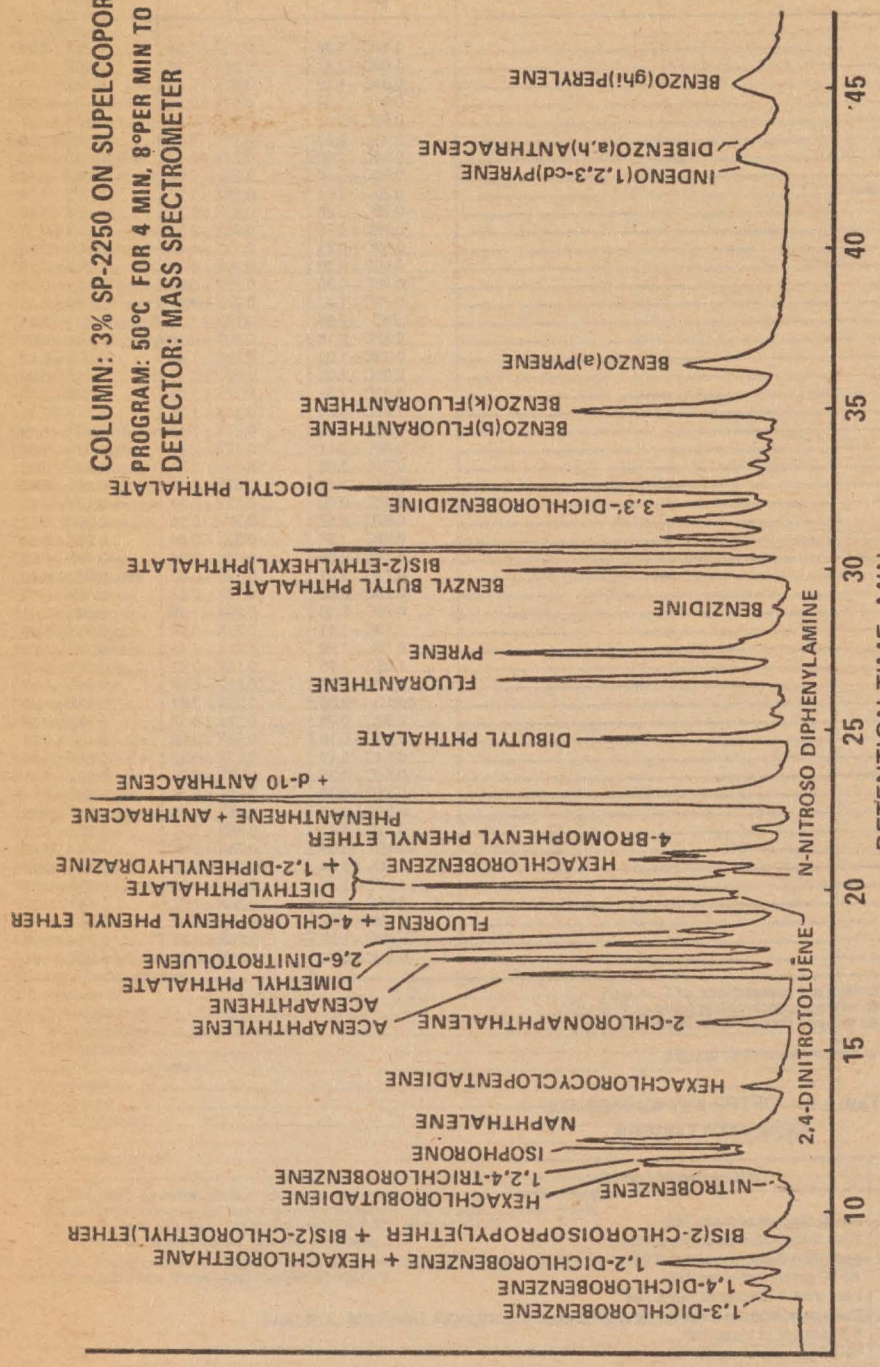



Figure 1. Gas chromatogram of base/neutral fraction.

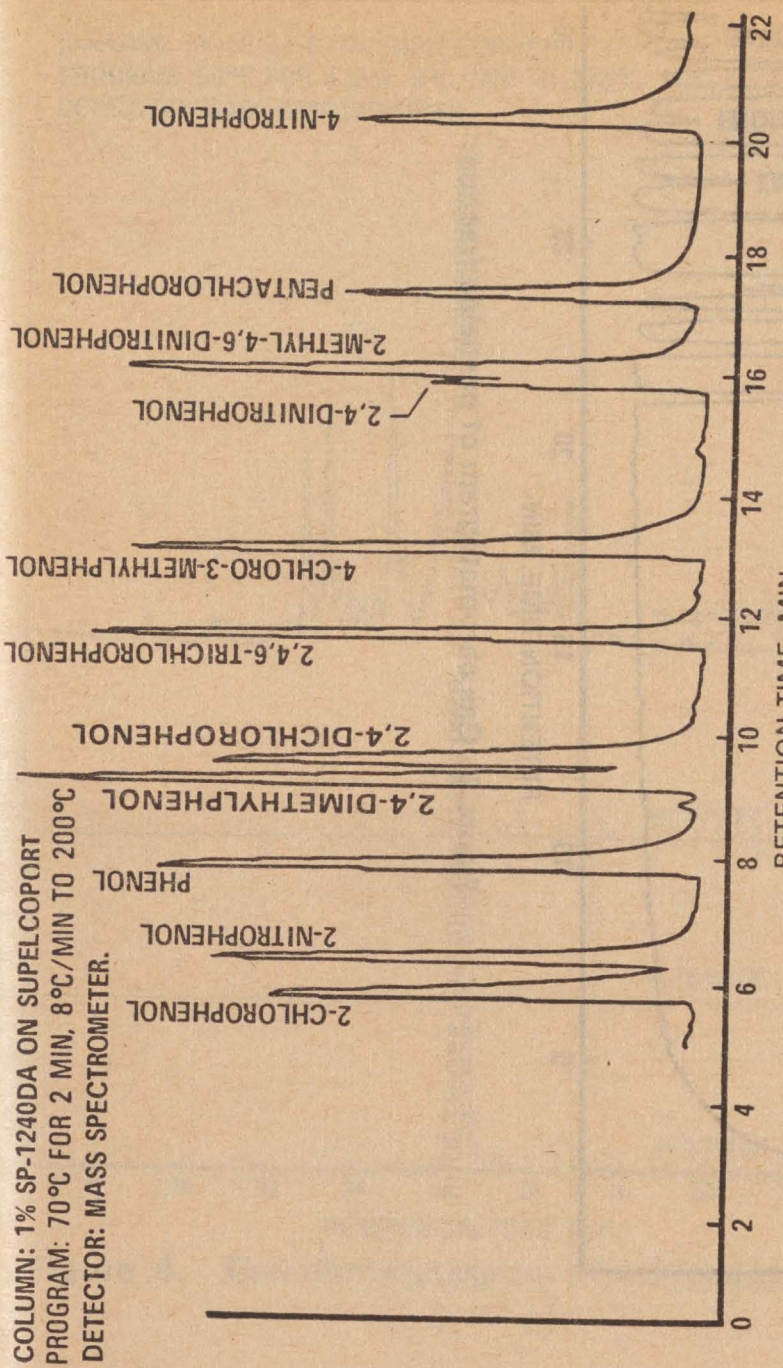



Figure 2. Gas chromatogram of acid fraction.

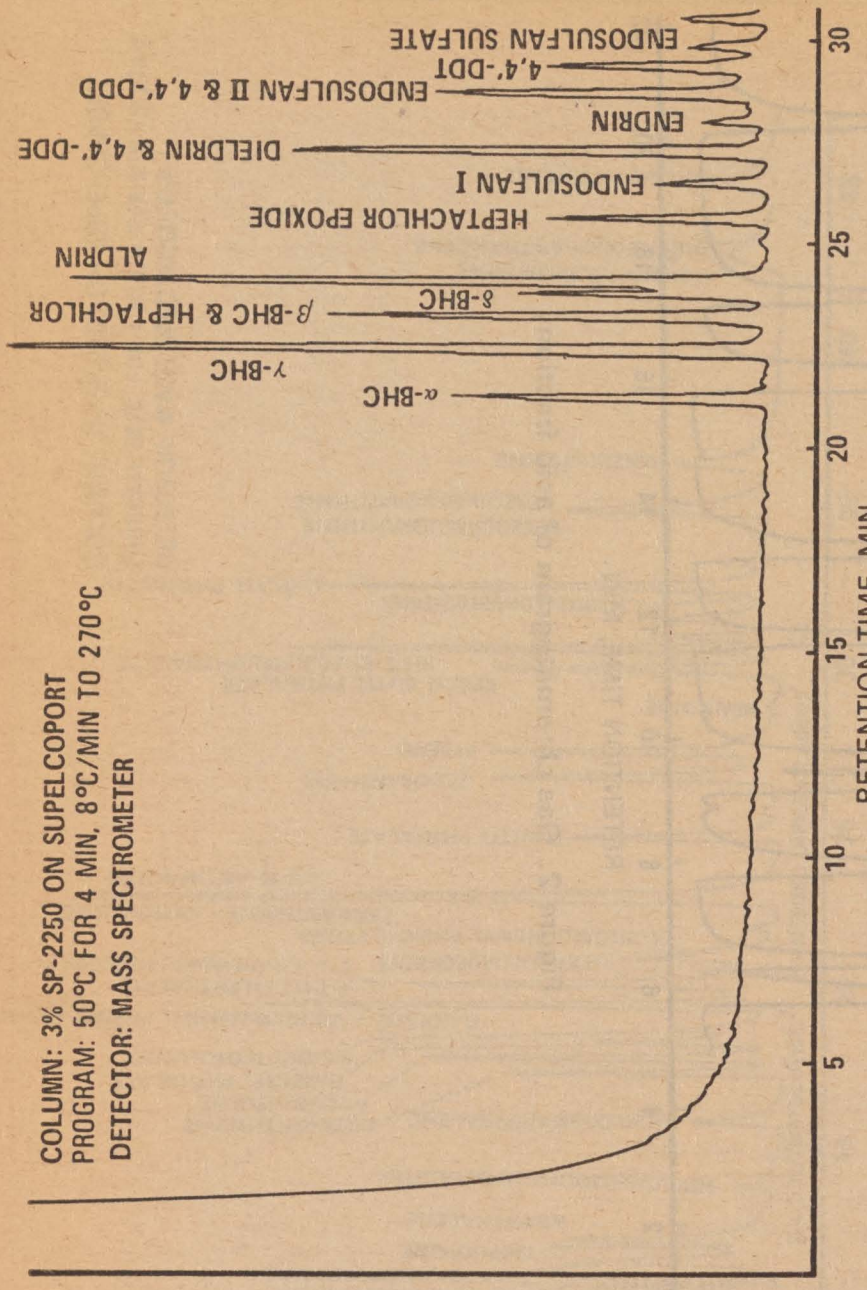



Figure 3. Gas chromatogram of pesticide fraction.

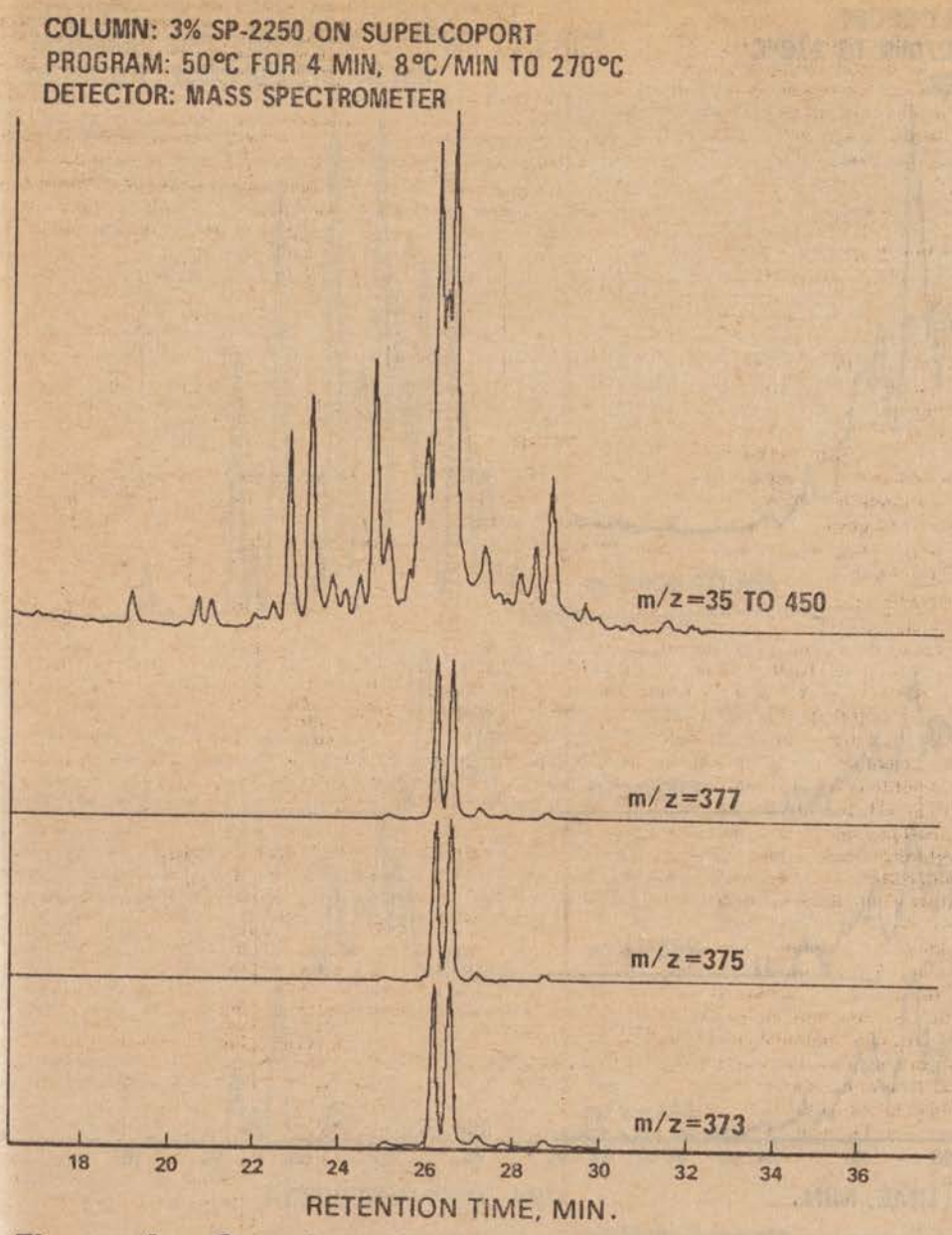



**Figure 4. Gas chromatogram of chlordane.**

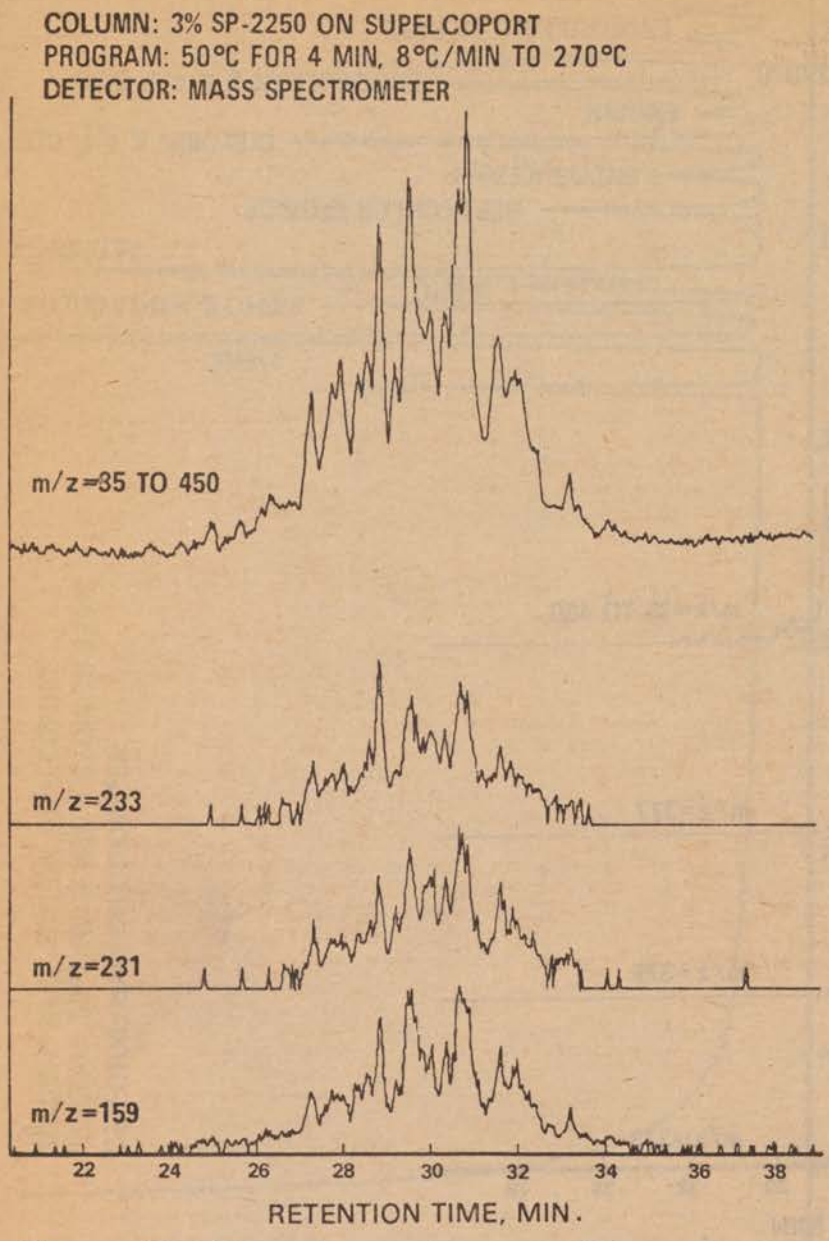



**Figure 5. Gas chromatogram of toxaphene.**

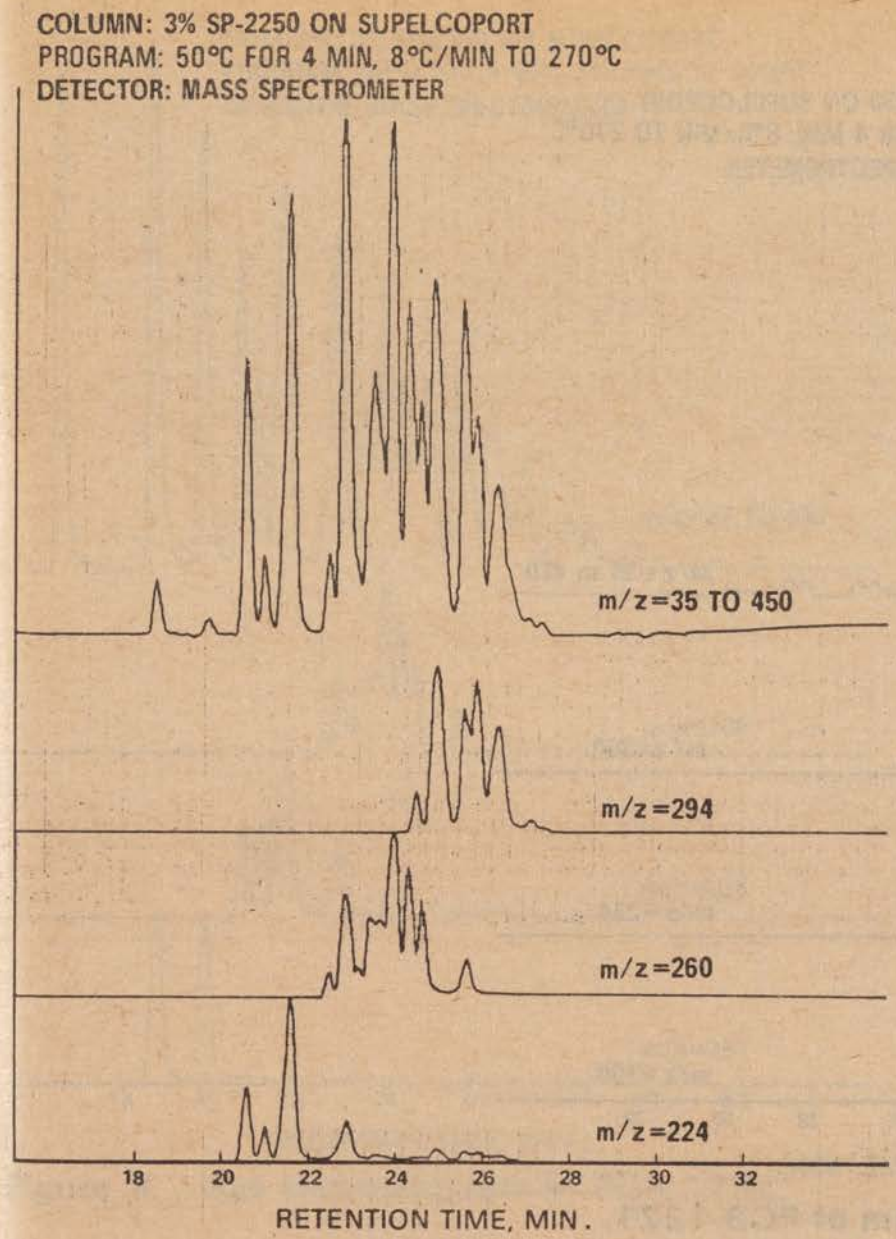



**Figure 6. Gas chromatogram of PCB-1016.**

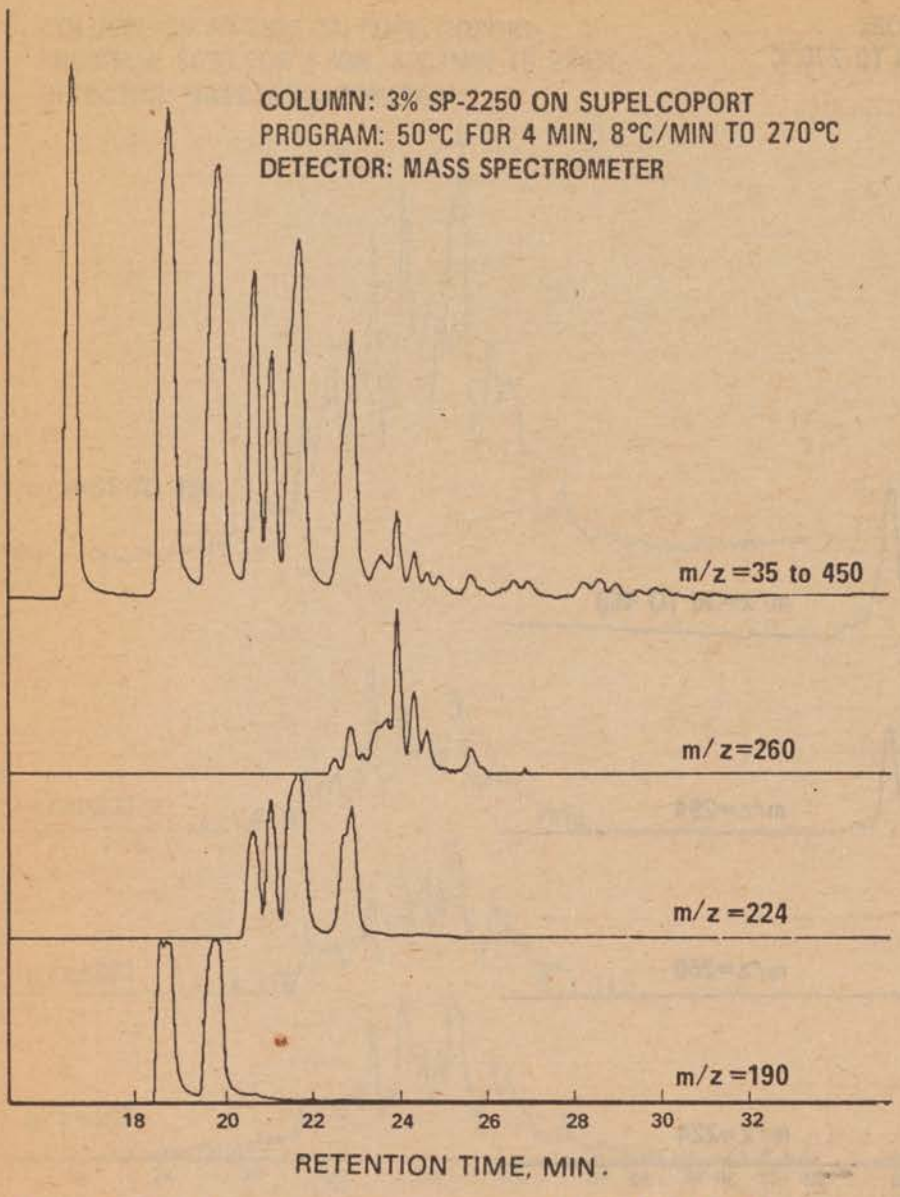



**Figure 7. Gas chromatogram of PCB-1221.**

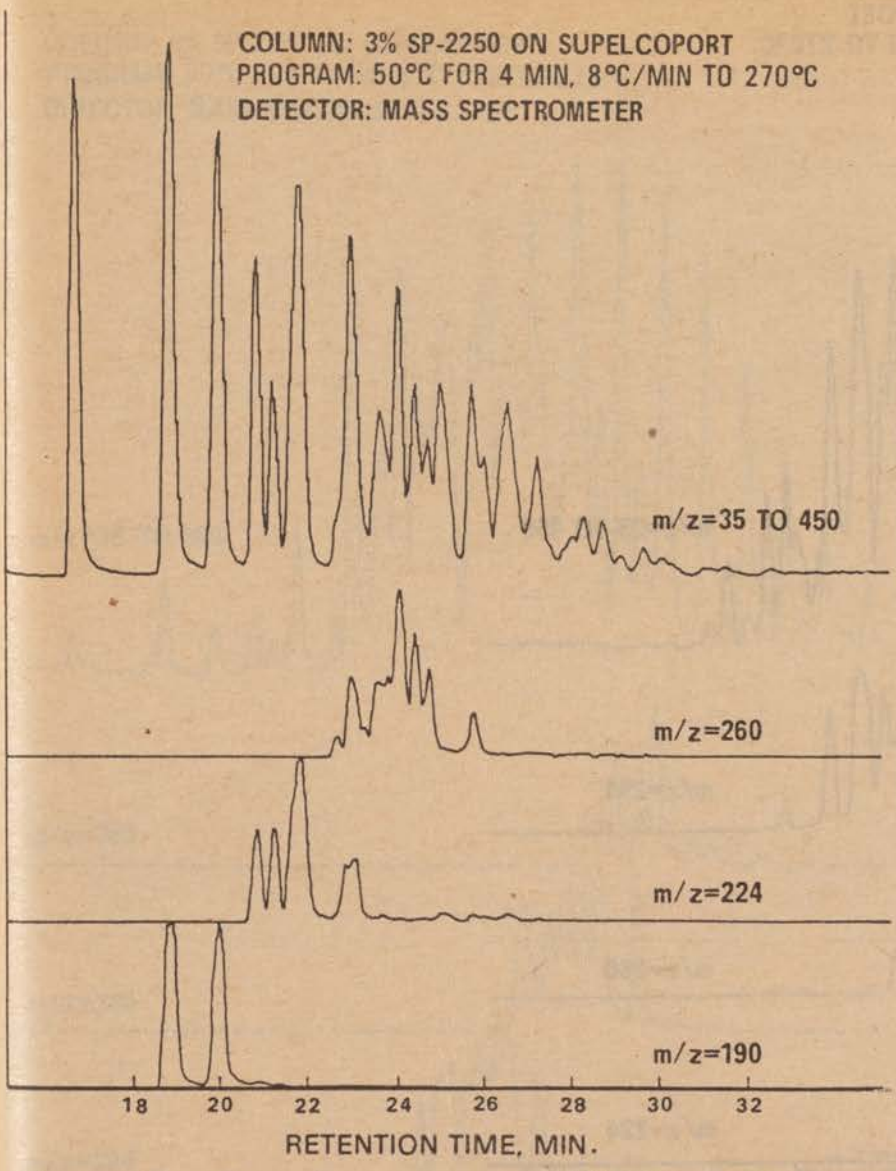



**Figure 8. Gas chromatogram of PCB-1232.**

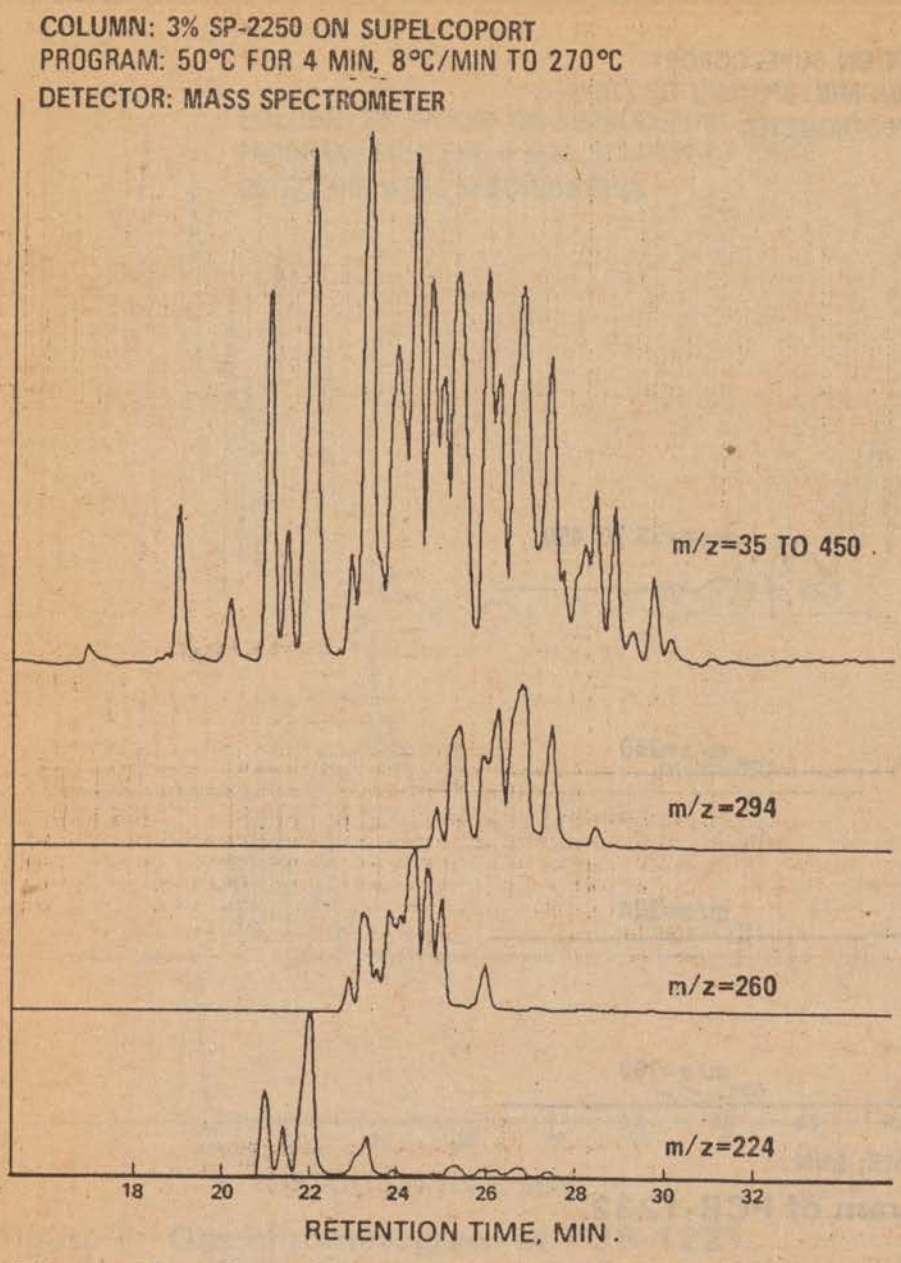



**Figure 9. Gas chromatogram of PCB-1242.**

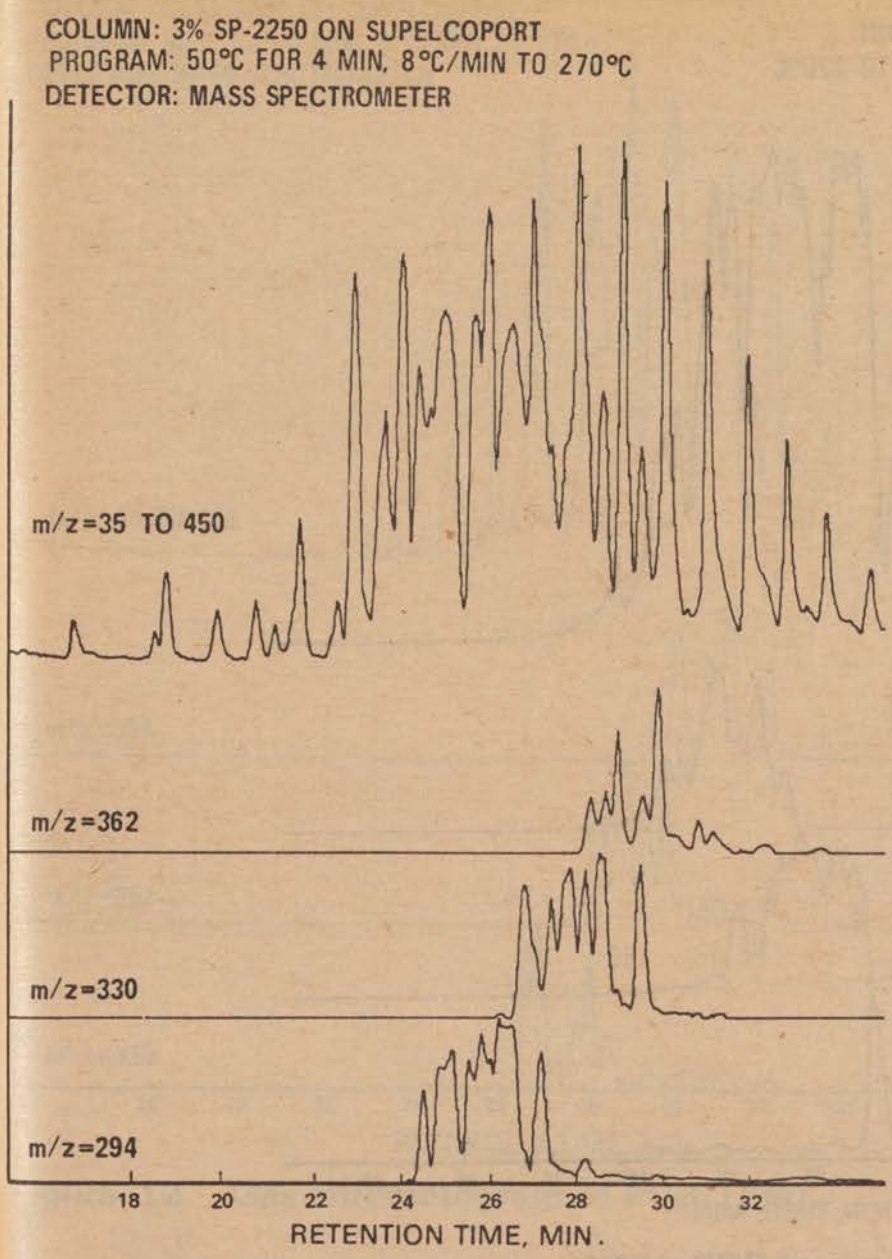



Figure 10. Gas chromatogram of PCB-1248.

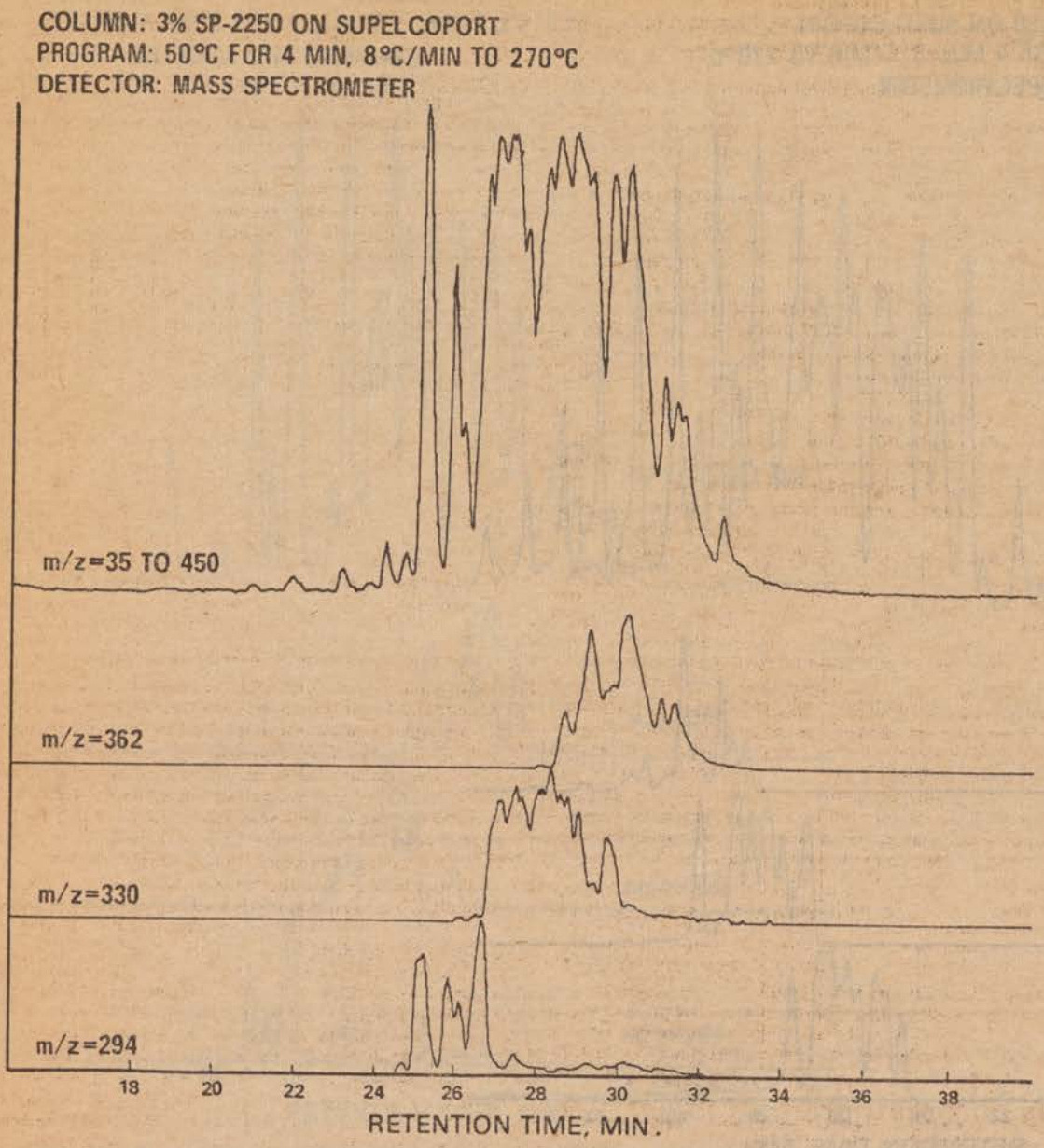



**Figure 11. Gas chromatogram of PCB-1254.**

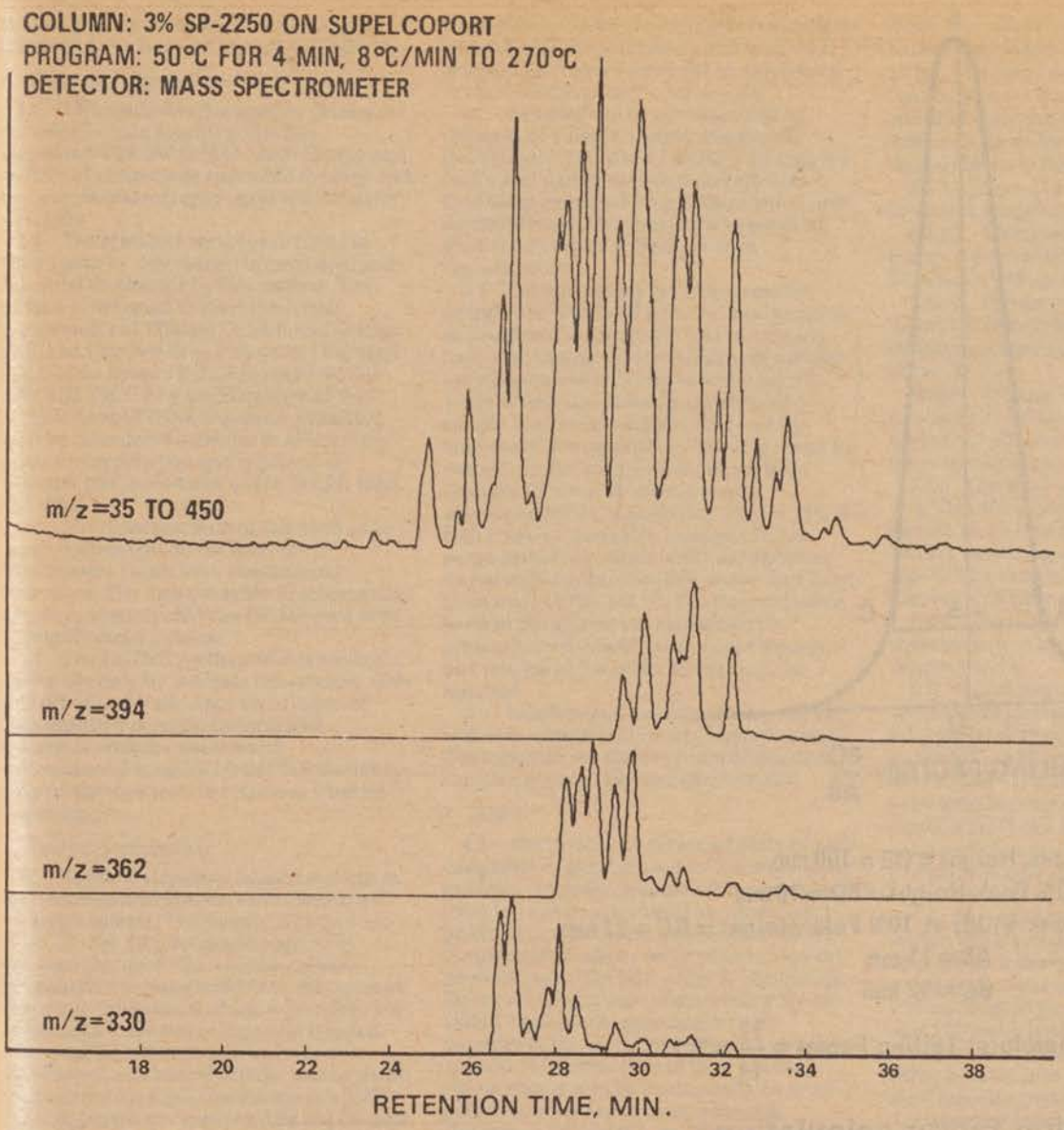
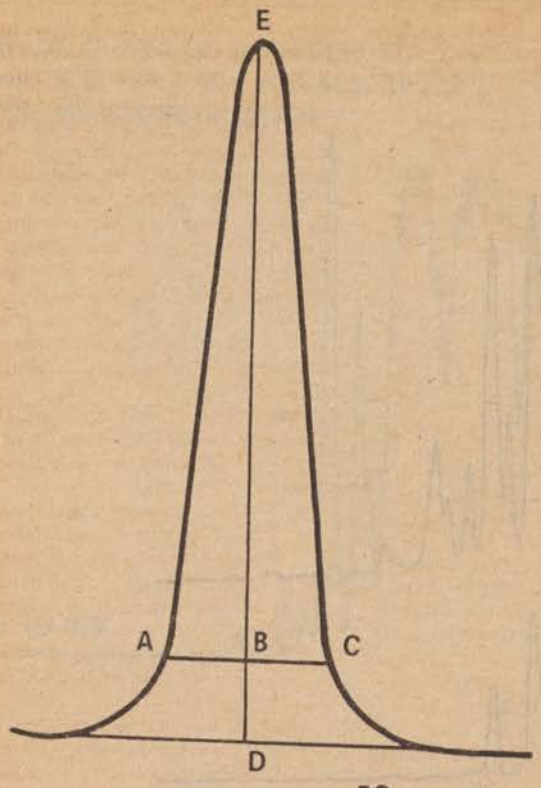



Figure 12. Gas chromatogram of PCB-1260.




$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor $= \frac{12}{11} = 1.1$

**Figure 13. Tailing factor calculation.**



BILLING CODE 6560-50-C

**Method 1624 Revision B—Volatile Organic Compounds by Isotope Dilution GC/MS**

*1 Scope and application*

1.1 This method is designed to determine the volatile toxic organic pollutants associated with the 1976 Consent Decree and additional compounds amenable to purge and trap gas chromatography-mass spectrometry (GC/MS).

1.2 The chemical compounds listed in table 1 may be determined in municipal and industrial discharges by this method. The method is designed to meet the survey requirements of Effluent Guidelines Division (EGD) and the National Pollutants Discharge Elimination System (NPDES) under 40 CFR 136.1 and 136.5. Any modifications of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.3 The detection limit of this method is usually dependent on the level of interferences rather than instrumental limitations. The limits in table 2 represent the minimum quantity that can be detected with no interferences present.

1.4 The GC/MS portions of this method are for use only by analysts experienced with GC/MS or under the close supervision of such qualified persons. Laboratories unfamiliar with the analyses of environmental samples by GC/MS should run the performance tests in reference 1 before beginning.

*2 Summary of method*

2.1 Stable isotopically labeled analogs of the compounds of interest are added to a 5 mL water sample. The sample is purged at 20–25 °C with an inert gas in a specially designed chamber. The volatile organic compounds are transferred from the aqueous phase into the gaseous phase where they are passed into a sorbent column and trapped. After purging is completed, the trap is backflushed and heated rapidly to desorb the compounds into a gas chromatograph (GC). The compounds are separated by the GC and detected by a mass spectrometer (MS) (references 2 and 3). The labeled compounds serve to correct the variability of the analytical technique.

2.2 Identification of a compound (qualitative analysis) is performed by comparing the GC retention time and the background corrected characteristic spectral masses with those of authentic standards.

2.3 Quantitative analysis is performed by GC/MS using extracted ion current profile (EICP) areas. Isotope dilution is used when labeled compounds are available; otherwise, an internal or external standard method is used.

2.4 Quality is assured through reproducible calibration and testing of the purge and trap and GC/MS systems.

*3 Contamination and interferences*

3.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing upstream of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system is demonstrated to be free from interferences under conditions of the analysis by analyzing blanks initially and with each sample lot (samples analyzed on the same 8 hr shift), as described in section 8.5.

3.2 Samples can be contaminated by diffusion of volatile organic compounds (particularly methylene chloride) through the bottle seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol serves as a check on such contamination.

3.3 Contamination by carry-over can occur when high level and low level samples are analyzed sequentially. To reduce carry-over, the purging device and sample syringe are rinsed between samples with reagent water. When an unusually concentrated sample is encountered, it is followed by analysis of a reagent water blank to check for carry-over. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds, or high levels or purgeable compounds, the purge device is washed with soap solution, rinsed with tap and distilled water, and dried in an oven at 100–125 °C. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

3.4 Interferences resulting from samples will vary considerably from source to source, depending on the diversity of the industrial complex or municipality being sampled.

*4 Safety*

4.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should also be made available to all personnel involved in these analyses. Additional information on laboratory safety can be found in references 4–6.

4.2 The following compounds covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator should be worn when high concentrations are handled.

5 Apparatus and materials

5.1 Sample bottles for discrete sampling

5.1.1 Bottle—25 to 40 mL with screw cap (Pierce 13075, or equivalent). Detergent wash, rinse with tap and distilled water, and dry at >105 °C for one hr minimum before use.

5.1.2 Septum—Teflon-faced silicone (Pierce 12722, or equivalent), cleaned as above and baked at 100–200 °C, for one hour minimum.

5.2 Purge and trap device—consists of purging device, trap, and desorber. Complete devices are commercially available.

5.2.1 Purging device—designed to accept 5 mL samples with water column at least 3 cm deep. The volume of the gaseous head space between the water and trap shall be less than 15 mL. The purge gas shall be introduced less than 5 mm from the base of the water column and shall pass through the water as bubbles with a diameter less than 3 mm. The purging device shown in figure 1 meets these criteria.

5.2.2 Trap—25 to 30 cm x 2.5 mm i.d. minimum, containing the following:

5.2.2.1 Methyl silicone packing—one ± 0.2 cm, 3 percent OV–1 on 60/80 mesh Chromosorb W, or equivalent.

5.2.2.2 Porous polymer—15 ± 1.0 cm, Tenax GC (2,6-diphenylene oxide polymer), 60/80 mesh, chromatographic grade, or equivalent.

5.2.2.3 Silica gel—8 ± 1.0 cm, Davison Chemical, 35/60 mesh, grade 15, or equivalent. The trap shown in figure 2 meets these specifications.

5.2.3 Desorber—shall heat the trap to 175 ± 5 °C in 45 seconds or less. The polymer section of the trap shall not exceed 180 °C, and the remaining sections shall not exceed 220 °C. The desorber shown in figure 2 meets these specifications.

5.2.4 The purge and trap device may be a separate unit or coupled to a GC as shown in figures 3 and 4.

5.3 Gas chromatograph—shall be linearly temperature programmable with initial and final holds, shall contain a glass jet separator as the MS interface, and shall produce results which meet the calibration (section 7), quality assurance (section 8), and performance tests (section 11) of this method.

5.3.1 Column—2.8 ± 0.4 m x 2 ± 0.5 mm i. d. glass, packekd with one percent SP–1000 on Carbopak B, 60/80 mesh, or equivalent.

5.4 Mass spectrometer—70 eV electron impact ionization; shall repetitively scan from 20 to 250 amu every 2–3 seconds, and produce a unit resolution (valleys between m/z 174–176 less than 10 percent of the height of the m/z 175 peak), background corrected mass spectrum from 50 ng 4-bromo-fluorobenzene (BFB) injected into the GC. The BFB spectrum shall meet the mass-intensity criteria in table 3. All portions of the GC column, transfer lines, and separator which connect the GC column to the ion source shall remain at or above the column temperature during analysis to preclude condensation of less volatile compounds.

5.5 Data system—shall collect and record MS data, store mass intensity data in spectral libraries, process GC/MS data and generate reports, and shall calculate and record response factors.

5.5.1 Data acquisition—mass spectra shall be collected continuously throughout the analysis and stored on a mass storage device.

5.5.2 Mass spectral libraries—user created libraries containing mass spectra obtained from analysis of authentic standards shall be employed to reverse search GC/MS runs for the compounds of interest (section 7.2).

5.5.3 Data processing—the data system shall be used to search, locate, identify, and quantify the compounds of interest in each GC/MS analysis. Software routines shall be employed to compute retention times and EICP areas. Displays of spectra, mass

chromatograms, and library comparisons are required to verify results.

5.5.4 Response factors and multipoint calibrations—the data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and generate multi-point calibration curves (section 7). Computations of relative standard deviation (coefficient of variation) are useful for testing calibration linearity. Statistics on initial and on-going performance shall be maintained (sections 8 and 11).

5.6 Syringes—5 mL glass hypodermic, with Luer-lok tips.

5.7 Micro syringes—10, 25, and 100 uL.

5.8 Syringe valves—2-way, with Luer ends (Telfon or Kel-F).

5.9 Syringe—5 mL, gas-tight, with shut-off valve.

5.10 Bottles—15 mL., screw-cap with Telfon liner.

5.11 Balance—analytical, capable of weighing 0.1 mg.

*6 Reagents and standards*

6.1 Reagent water—water in which the compounds of interest and interfering compounds are not detected by this method (section 11.7). It may be generated by any of the following methods:

6.1.1 Activated carbon—pass tap water through a carbon bed (Calgon Filtrasorb-300, or equivalent).

6.1.2 Water purifier—pass tap water through a purifier (Millipore Super Q, or equivalent).

6.1.3 Boil and purge—heat tap water to 90–100 °C and bubble contaminant free inert gas through it for approx one hour. While still hot, transfer the water to screw-cap bottles and seal with a Teflon-lined cap.

6.2 Sodium thiosulfate—ACS granular.

6.3 Methanol—pesticide quality or equivalent.

6.4 Standard solutions—purchased as solution or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If compound purity is 96 percent or greater, the weight may be used without correction to calculate the concentration of the standard.

6.5 Preparation of stock solutions—prepare in methanol using liquid or gaseous standards per the steps below. Observe the safety precautions given in section 4.

6.5.1 Place approx 9.8 mL of methanol in a 10 mL ground glass stoppered volumetric flask. Allow the flask to stand unstoppered for approximately 10 minutes or until all methanol wetted surfaces have dried. In each case, weigh the flask, immediately add the compound, then immediately reweigh to prevent evaporation losses from affecting the measurement.

6.5.1.1 Liquids—using a 100 μL syringe, permit 2 drops of liquid to fall into the methanol without contacting the neck of the flask. Alternatively, inject a known volume of the compound into the methanol in the flask using a micro-syringe.

6.5.1.2 Gases (chloromethane, bromomethane, chloroethane, vinyl chloride)—fill a valved 5 mL gas-tight syringe with the compound. Lower the needle to approx 5 mm above the methanol meniscus. Slowly introduce the compound above the surface of the meniscus. The gas will dissolve rapidly in the methanol.

6.5.2 Fill the flask to volume, stopper, then mix by inverting several times. Calculate the concentration in mg/mL (μg/μL) from the weight gain (or density if a known volume was injected).

6.5.3 Transfer the stock solution to a Teflon sealed screw-cap–bottle. Store, with minimal headspace, in the dark at −10 to −20 °C.

6.5.4 Prepare fresh standards weekly for the gases and 2-chloroethylvinyl ether. All other standards are replaced after one month, or sooner if comparison with check standards indicate a change in concentration. Quality control check standards that can be used to determine the accuracy of calibration standards are available from the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

6.6 Labeled compound spiking solution—from stock standard solutions prepared as above, or from mixtures, prepare the spiking solution to contain a concentration such that a 5–10 μL spike into each 5 mL sample, blank, or aqueous standard analyzed will result in a concentration of 20 μg/L of each labeled compound. For the gases and for the water soluble compounds (acrolein, acrylonitrile, acetone, diethyl ether, and MEK), a concentration of 100 μg/L may be used. Include the internal standards (section 7.5) in this solution so that a concentration of 20 μg/L in each sample, blank, or aqueous standard will be produced.

6.7 Secondary standards—using stock solutions, prepare a secondary standard in methanol to contain each pollutant at a concentration of 500 μg/mL For the gases and water soluble compounds (section 6.6), a concentration of 2.5 mg/mL may be used.

6.7.1 Aqueous calibration standards—using a 25 μL syringe, add 20 μL of the secondary standard (section 6.7) to 50, 100, 200, 500, and 1000 mL of reagent water to produce concentrations of 200, 100, 50, 20, and 10 μg/L, respectively. If the higher concentration standard for the gases and water soluble compounds was chosen (section 6.6), these compounds will be at cocentrations of 1000, 500, 250, 100, and 50 μg/L in the aqueous calibration standards.

6.7.2 Aqueous performance standard—an aqueous standard containing all pollutants, internal standards, labeled compounds, and BFB is prepared daily, and analyzed each shift to demonstrate performance (section 11). This standard shall contain either 20 or 100 μg/L of the labeled and pollutant gases and water soluble compounds, 10 μg/L BFB, and 20 μg/L of all other pollutants, labeled compounds, and internal standards. It may be the nominal 20 μg/L aqueous calibration standard (section 6.7.1).

6.7.3 A methanolic standard containing all pollutants and internal standards is prepared to demonstrate recovery of these compounds when syringe injection and purge and trap analyses are compared. This standard shall contain either 100 μg/mL or 500 μg/mL of the gases and water soluble compounds, and 100 μg/mL of the remaining pollutants and internal standards (consistent with the amounts in the aqueous performance standard in 6.7.2).

6.7.4 Othe standards which may be needed are those for test of BFB performance (section 7.1) and for collection of mass spectra for storage in spectral libraries (section 7.2).

*7 Calibration*

7.1 Assemble the gas chromatographic apparatus and establish operating conditions given in table 2. By injecting standards into the GC, demonstrate that the analytical system meets the detection limits in table 2 and the mass-intensity criteria in table 3 for 50 ng BFB.

7.2 Mass spectral libraries—detection and identification of the compound of interest are dependent upon the spectra stored in user created libraries.

7.2.1 Obtain a mass spectrumof each pollutant and labeled compound and each internal standard by analyzing an authentic standard either singly or as part of a mixture in which there is no interference between closely eluted components. That only a single compound is present is determined by examination of the spectrum. Fragments not attributable to the compound under study indicate the presence of an interfering compound. Adjust the analytical conditions and scan rate (for this test only) to produce an undistorted spectrum at the GC peak maximum. An undistorted spectrum will usually be obtained if five complete spectra are collected across the upper half of the GC peak. Software algorithms designed to "enhance" the spectrum may eliminate distortion, but may also eliminate authentic m/z's or introduce other distortion.

7.2.3 The authentic reference spectrum is obtained under BFB tuning conditions (section 7.1 and table 3) to normalize it to spectra from other instruments.

7.2.4 The spectrum is edited by saving the 5 most intense mass spectral peaks and all other mass spectral peaks greater than 10 percent of the base peak. This spectrum is stored for reverse search and for compound confirmation.

7.3 Assemble the purge and trap device. Pack the trap as shown in figure 2 and condition overnight at 170–180 °C by backflushing with an inert gas at a flow rate of 20–30 mL/min. Condition traps daily for a minimum of 10 minutes prior to use.

7.3.1 Analyze the aqueous performance standard (section 6.7.2) according to the purge and trap procedure in section 10. Compute the area at the primary m/z (table 4) for each compound. Compare these areas to those obtained by injecting one μL of the methanolic standard (section 6.7.3) to determine compound recovery. The recovery shall be greater than 20 percent for the water soluble compounds, and 60–110 percent for all other compounds. This recovery is demonstrated initially for each purge and trap GC/MS system. The test is repeated only if the purge and trap or GC/MS systems are modified in any way that might result in a change in recovery.

7.3.2 Demonstrate that 100 ng toluene (or toluene-d8) produces an area at m/z 91 (or 99) approx one-tenth that required to exceed

the linear range of the system. The exact value must be determined by experience for each instrument. It is used to match the calibration range of the instrument to the analytical range and detection limits required.

7.4 Calibration by isotope dilution—the isotope dilution approach is used for the purgeable organic compounds when appropriate labeled compounds are available and when interferences do not preclude the analysis. If labeled compounds are not available, or interferences are present, internal or external standard methods (section 7.5 or 7.6) are used. A calibration curve encompassing the concentration range of interest is prepared for each compound determined. The relative response (RR) vs concentration (μg/L) is plotted or computed using a linear regression. An example of a calibration curve for toluene using toluene-d8 is given in figure 5. Also shown are the ±10 percent error limits (dotted lines). Relative response is determined according to the procedures described below. A minimun of five data points are required for calibration (section 7.4.4).

7.4.1 The relative response (RR) of pollutant to labeled compound is determined from isotope ratio values calculated from acquired data. Three isotope ratios are used in this process:

$R_x$ = the isotope ratio measured in the pure pollutant (figure 6A).

$R_y$ = the isotope ratio of pure labeled compound (figure 6B).

$R_m$ = the isotope ratio measured in the analytical mixture of the pollutant and labeled compounds (figure 6C).

The correct way to calculate RR is: $RR=(R_y-R_m)(R_x+1)/(R_m-R_x)(R_y+1)$ If $R_m$ is not between $2R_y$ and $0.5R_x$, the method does not apply and the sample is analyzed by internal or external standard methods (section 7.5 or 7.6).

7.4.2 In most cases, the retention times of the pollutant and labeled compound are the same and isotope ratios (R's) can be calculated from the EICP areas, where: R=(area at $m_1/z$) (area at $m_2/z$) If either of the areas is zero, it is assigned a value of one in the calculations; that is, if: area of $m_1/z=50721$, and area of $m_2/z=0$, then $R=50721/1=50720$. The m/z's are always selected such that $R_x>R_y$. When there is a difference in retention times (RT) between the pollutant and labeled compounds, special precautions are required to determine the isotope ratios.

$R_x$, $R_y$, and $R_m$ are defined as follows:

$R_x$=[area $m_1/z$ (at $RT_1$)]/1

$R_y$=1/[area $m_2/z$ (at $RT_2$)]

$R_m$=[area $m_1/z$ (at $RT_1$)]/[area $m_2/z$ (at $RT_2$)]

7.4.3 An example of the above calculations can be taken from the data plotted in figure 5 for toluene and toluene-d8. For these data, $R_x=168920/1=168900$, $R_y=1/60960=0.00001640$, and $R_m=96868/82508=1.174$. The RR for the above data is then calculated using the equation given in section 7.4.1. For the example, RR=1.174.

*Note:* Not all labeled compounds elute before their pollutant analogs.

7.4.4 To calibrate the analytical system by isotope dilution, analyze a 5 mL aliquot of each of the aqueous calibration standards (section 6.7.1) spiked with an appropriate constant amount of the labeled compound spiking solution (section 6.6), using the purge and trap procedure in section 10. Compute the RR at each concentration.

7.4.5 Linearity—if the ratio of relative response to concentration for any compound is constant (less than 20 percent coefficient of variation) over the 5 point calibration range, an averaged relative response/concentration ratio may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point calibration range.

7.5 Calibration by internal standard—used when criteria for isotope dilution (section 7.4) cannot be met. The method is applied to pollutants having no labeled analog and to the labeled compounds. The internal standards used for volatiles analyses are bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane. Concentrations of the labeled compounds and pollutants without labeled analogs are computed relative to the nearest eluted internal standard, as shown in table 2.

7.5.1 Response factors—calibration requires the determination of response factors (RF) which are defined by the following equation: $RF=(A_s \times C_{is})/(A_{is} \times C_s)$, where $A_s$ is the EICP area at the characteristic m/z for the compound in the daily standard. $A_{is}$ is the EICP area at the characteristic m/z for the internal standard.

$C_{is}$ is the concentration (ug/L) of the internal standard

$C_s$ is the concentration of the pollutant in the daily standard.

7.5.2 The response factor is determined at 10, 20, 50, 100, and 200 ug/L for the pollutants (optionally at five times these concentrations for gases and water soluble pollutants—see section 6.7), in a way analogous to that for calibration by isotope dilution (section 7.4.4). The RF is plotted against concentration for each compound in the standard ($C_s$) to produce a calibration curve.

7.5.3 Linearity—if the response factor (RF) for any compound is constant (less than 35 percent coefficient of variation) over the 5 point calibration range, an averaged response factor may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point range.

7.6 Combined calibration—by adding the isotopically labeled compounds and internal standards (section 6.6) to the aqueous calibration standards (section 6.7.1), a single set of analyses can be used to produce calibration curves for the isotope dilution and internal standard methods. These curves are verified each shift (section 11.5) by purging the aqueous performance standard (section 6.7.2). Recalibration is required only if calibration and on-going performance (section 11.5) criteria cannot be met.

*8 Quality assurance/quality control*

8.1 Each laboratory that uses this method is required to operate a formal quality assurance program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

8.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in section 8.2.

8.1.2 The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance specifications are met. Each time a modification is made to the method, the analyst is required to repeat the procedure in section 8.2 to demonstrate method performance.

8.1.3 Analyses of blanks are required to demonstrate freedom from contamination and that the compounds of interest and interfering compounds have not been carried over from a previous analysis (section 3). The procedures and criteria for analysis of a blank are described in sections 8.5 and 11.7.

8.1.4 The laboratory shall spike all samples with labeled compounds to monitor method performance. This test is described in section 8.3. When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method performance within acceptable limits (section 14.2).

8.1.5 The laboratory shall, on an on-going basis, demonstrate through the analysis of the aqueous performance standard (section 6.7.2) that the analysis system is in control. This procedure is described in sections 11.1 and 11.5.

8.1.6 The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in sections 8.4 and 11.5.2.

8.2 Initial precision and accuracy—to establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

8.2.1 Analyze two sets of four 5-mL aliquots (8 aliquots total) of the aqueous performance standard (section 6.7.2) according to the method beginning in section 10.

8.2.2 Using results of the first set of four analyses in section 8.2.1, compute the average recovery ($\bar{X}$) in μg/L and the standard deviation of the recovery (s) in μg/L for each compound, by isotope dilution for polluitants with a labeled analog, and by internal standard for labeled compounds and pollutants with no labeled analog.

8.2.3 For each compound, compare s and $\bar{X}$ with the corresponding limits for initial precision and accuracy found in table 5. If s and $\bar{X}$ for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If individual $\bar{X}$ falls outside the range for accuracy, system performance is unacceptable for that compound. NOTE: The large number of compounds in table 5 present a substantial probability that one or more will fail one of the acceptance criteria when

all compounds are analyzed. To determine if the analytical system is out of control, or if the failure can be attributed to probability, proceed as follows:

8.2.4 Using the results of the second set of four analyses, compute s and $\bar{X}$ for only those compounds which failed the test of the first set of four analyses (section 8.2.3). If these compounds now pass, system performance is acceptable for all compounds and analysis of blanks and samples may begin. If, however, any of the same compounds fail again, the analysis system is not performing properly for the compound(s) in question. In this event, correct the problem and repeat the entire test (section 8.2.1).

8.3 The laboratory shall spike all samples with labeled compounds to assess method performance on the sample matrix.

8.3.1 Spike and analyze each sample according to the method beginning in section 10.

8.3.2 Compute the percent recovery (P) of the labeled compounds using the internal standard method (section 7.5).

8.3.3 Compare the percent recovery for each compound with the corresponding labeled compound recovery limit in table 5. If the recovery of any compound falls outside its warning limit, method performance is unacceptable for that compound in that sample. Therefore, the sample matrix is complex and the sample is to be diluted and reanalyzed, per section 14.2.

8.4 As part of the QA program for the laboratory, method accuracy for wastewater samples shall be assessed and records shall be maintained. After the analysis of five wastewater samples for which the labeled compounds pass the tests in section 8.3.3, compute the average percent recovery (P) and the standard deviation of the percent recovery ($s_p$) for the labeled compounds only. Express the accuracy assessment as a percent recovery interval from $P-2s_p$ to $P+2s_p$. For example, if $P=90\%$ and $s_p=10\%$, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each compound on a regular basis (e.g. after each 5–10 new accuracy measurements).

8.5 Blanks—reagent water blanks are analyzed to demonstrate freedom from carry-over (section 3) and contamination.

8.5.1 The level at which the purge and trap system will carry greater than 5 μg/L of a pollutant of interest (table 1) into a succeeding blank shall be determined by analyzing successively larger concentrations of these compounds. When a sample contains this concentration or more, a blank shall be analyzed immediately following this sample to demonstrate no carry-over at the 5 μg/L level.

8.5.2 With each sample lot (samples analyzed on the same 8 hr shift), a blank shall be analyzed immediately after analysis of the aqueous performance standard (section 11.1) to demonstrate freedom from contamination. If any of the compounds of interest (table 1) or any potentially interfering compound is found in a blank at greater than 10 μg/L (assuming a response factor of 1 relative to the nearest eluted internal standard for compounds not listed in table 1), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

8.6 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state.

The standards used for calibration (section 7), calibration verification (section 11.5) and for initial (section 8.2) and on-going (section 11.5) precision and accuracy should be identical, so that the most precise results will be obtained. The GC/MS instrument in particular will provide the most reproducible results if dedicated to the settings and conditions required for the analyses of volatiles by this method.

8.7 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when internal or external standard methods are used.

*9 Sample collection, preservation, and handling*

9.1 Grab samples are collected in glass containers having a total volume greater than 20 mL. Fill sample bottles so that no air bubbles pass through the sample as the bottle is filled. Seal each bottle so that no air bubbles are entrapped. Maintain the hermetic seal on the sample bottle until time of analysis.

9.2 Samples are maintained at 0–4 °C from the time of collection until analysis. If the sample contains residual chlorine, add sodium thiosulfate preservative (10 mg/40 mL) to the empty sample bottles just prior to shipment to the sample site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine (reference 8). If preservative has been added, shake bottle vigorously for one minute immediately after filling.

9.3 Experimental evidence indicates that some aromatic compounds, notably benzene, toluene, and ethyl benzene are susceptible to rapid biological degradation under certain environmental conditions. Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days. For this reason, a separate sample should be collected, acidified, and analyzed when these aromatics are to be determined. Collect about 500 mL of sample in a clean container.

Adjust the pH of the sample to about 2 by adding HCl (1+1) while stirring. Check pH with narrow range (1.4 to 2.8) pH paper. Fill a sample container as described in section 9.1. If residual chlorine is present, add sodium thiosulfate to a separate sample container and fill as in section 9.1.

9.4 All samples shall be analyzed within 14 days of collection.

*10 Purge, trap, and GC/MS analysis*

10.1 Remove standards and samples from cold storage and bring to 20–25 °.

10.2 Adjust the purge gas flow rate to 40 ±4 mL/min. Attach the trap inlet to the purging device and set the valve to the purge mode (figure 3). Open the syringe valve located on the purging device sample introduction needle (figure 1).

10.3 Remove the plunger from a 5–mL syringe and attach a closed syringe valve. Open the sample bottle and carefully pour the sample into the syringe barrel until it overflows. Replace the plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Because this process of taking an aliquot destroys the validity of the sample for future analysis, fill a second syringe at this time to protect against possible loss of data. Add an appropriate amount of the labeled compound spiking solution (section 6.6) through the valve bore, then close the valve.

10.4 Attach the syringe valve assembly to the syringe valve on the purging device. Open both syringe valves and inject the sample into the purging chamber.

10.5 Close both valves and purge the sample for 11.0 ± 0.1 minutes at 20–25 °C.

10.6 After the 11 minute purge time, attach the trap to the chromatograph and set the purge and trap apparatus to the desorb mode (figure 4). Desorb the trapped compounds into the GC column by heating the trap to 170–180 °C while backflushing with carrier gas at 20–60 mL/min for four minutes. Start MS data acquisition upon start of the desorb cycle, and start the GC column temperature program 3 minutes later. Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and detection limits that were achieved under these conditions. An example of the separations achieved by the column listed is shown in figure 5. Other columns may be used provided the requirements in section 8 can be met. If the priority pollutant gases produce GC peaks so broad that the precision and recovery specifications (section 8.2) cannot be met, the column may be cooled to ambient or sub-ambient temperatures to sharpen these peaks.

10.7 While analysis of the desorbed compounds proceeds, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL portions of reagent water. After the purging device has been emptied, allow the purge gas to vent through the chamber until the frit is dry, so that it is ready for the next sample.

10.8 After desorbing the sample for four minutes, recondition the trap by returning to the purge mode. Wait 15 seconds, then close the syringe valve on the purging device to begin gas flow through the trap. Maintain the trap temperature at 170–180 °C. After approximately seven minutes, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

*11 System performance*

11.1 At the beginning of each 8 hr shift during which analyses are performed, system calibration and performance shall be verified for all pollutants and labeled compounds. For these tests, analysis of the aqueous performance standard (section 6.7.2) shall be used to verify all performance criteria. Adjustment and/or recalibration (per section 7) shall be performed until all performance criteria are met. Only after all performance

criteria are met may blanks and samples be analyzed.

11.2 BFB spectrum validity—the criteria in table 3 shall be met.

11.3 Retention times—the absolute retention times of all compounds shall approximate those given in table 2.

11.4 GC resolution—the valley height between toluene and toluene-d8 (at m/z 91 and 99 plotted on the same graph) shall be less than 10 percent of the taller of the two peaks.

11.5 Calibration verification and on-going precision and accuracy—compute the concentration of each pollutant (table 1) by isotope dilution (section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant (table 1) which has no labeled analog by the internal standard method (section 7.5). Compute the concentration of the labeled compounds by the internal standard method. These concentrations are computed based on the calibration data determined in section 7.

11.5.1 For each pollutant and labeled compound, compare the concentration with the corresponding limit for on-going accuracy in table 5. If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may continue. If any individual value falls outside the range given, system performance is unacceptable for that compound. NOTE: The large number of compounds in table 5 present a substantial probability that one or more will fail the acceptance criteria when all compounds are analyzed. To determine if the analytical system is out of control, or if the failure may be attributed to probability, proceed as follows:

11.5.1.1 Analyze a second aliquot of the aqueous performance standard (section 6.7.2).

11.5.1.2 Compute the concentration for only those compounds which failed the first test (section 11.5.1). If these compounds now pass, system performance is acceptable for all compounds and analyses of blanks and samples may proceed. If, however, any of the compounds fail again, the measurement system is not performing properly for these compounds. In this event, locate and correct the problem or recalibrate the system (section 7), and repeat the entire test (section 11.1) for all compounds.

11.5.2 Add results which pass the specification in 11.5.1.2 to initial (section 8.2) and previous on-going data. Update QC charts to form a graphic representation of laboratory performance (Figure 7). Develop a statement of accuracy for each pollutant and labeled compound by calculating the average percentage recovery (R) and the standard deviation of percent recovery ($s_r$). Express the accuracy as a recovery interval from $R-2s_r$ to $R+2s_r$. For example, if R=95% and $s_r$=5%, the accuracy is 85–105 percent.

*12 Qualitative determination—accomplished by comparison of data from analysis of a sample or blank with data from analysis of the shift standard (section 11.1). Identification is confirmed when spectra and retention times agree per the criteria below.*

12.1 Labeled compounds and pollutants having no labeled analog:

12.1.1 The signals for all characteristic masses stored in the spectral library (section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

12.1.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two (0.5 to 2 times) for all masses stored in the library.

12.1.3 The retention time relative to the nearest eluted internal standard shall be within ±7 scans or ±20 seconds, whichever is greater.

12.2 Pollutants having a labeled analog:

12.2.1 The signals for all characteristic masses stored in the spectral library (section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

12.2.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the spectral library.

12.2.3 The retention time difference between the pollutant and its labeled analog shall agree within ±2 scans or ±6 seconds (whichever is greater) of this difference in the shift standard (section 11.1).

12.3 Masses present in the experimental mass spectrum that are not present in the reference mass spectrum shall be accounted for by contaminant or background ions. If the experimental mass spectrum is contaminated, an experienced spectrometrist (section 1.4) is to determine the presence or absence of the compound.

*13 Quantitative determination*

13.1 Isotope dilution—by adding a known amount of a labeled compound to every sample prior to purging, correction for recovery of the pollutant can be made because the pollutant and its labeled analog exhibit the same effects upon purging, desorption, and gas chromatography. Relative response (RR) values for sample mixtures are used in conjunction with calibration curves described in section 7.4 to determine concentrations directly, so long as labeled compound spiking levels are constant. For the toluene example given in figure 6 (section 7.4.3), RR would be equal to 1.174. For this RR value, the toluene calibration curve given in figure 5 indicates a concentration of 31.8 μg/L.

13.2 Internal standard—calculate the concentration using the response factor determined from calibration data (section 7.5) and the following equation:

Concentration $=(A_s \times C_{is})/(A_{is} \times RF)$

where the terms are as defined in section 7.5.1.

13.3 If the EICP area at the quantitation mass for any compound exceeds the calibration range of the system, the sample is diluted by successive factors of 10 and these dilutions are analyzed until the area is within the calibration range.

13.4 Report results for all pollutants and labeled compounds (table 1) found in all standards, blanks, and samples, in μg/L to three significant figures. Results for samples which have been diluted are reported at the least dilute level at which the area at the quantitation mass is within the calibration range (section 13.3) and the labeled compound recovery is within the normal range for the Method (section 14.2).

*14 Analysis of complex samples*

14.1 Untreated effluents and other samples frequently contain high levels (>1000 μg/L) of the compounds of interest and of interfering compounds. Some samples will foam excessively when purged; others will overload the trap/or GC column.

14.2 Dilute 0.5 mL of sample with 4.5 mL of reagent water and analyze this diluted sample when labeled compound recovery is outside the range given in table 5. If the recovery remains outside of the range for this diluted sample, the aqueous performance standard shall be analyzed (section 11) and calibration verified (section 11.5). If the recovery for the labeled compound in the aqueous performance standard is outside the range given in table 5, the analytical system is out of control. In this case, the instrument shall be repaired, the performance specifications in section 11 shall be met, and the analysis of the undiluted sample shall be repeated. If the recovery for the aqueous performance standard is within the range given in table 5, the method does not work on the sample being analyzed and the result may not be reported for regulatory compliance purposes.

14.3 Reverse search computer programs can misinterpret the spectrum of chromatographically unresolved pollutant and labeled compound pairs with overlapping spectra when a high level of the pollutant is present. Examine each chromatogram for peaks greater than the height of the internal standard peaks. These peaks can obscure the compounds of interest.

*15 Method performance*

15.1 The specifications for this method were taken from the inter-laboratory validation of EPA Method 624 (reference 9). Method 1624 has been shown to yield slightly better performance on treated effluents than Method 624. Additional method performance data can be found in Reference 10.

*References*

1. "Performance Tests for the Evaluation of Computerized Gas Chromatography/Mass Spectrometry Equipment and Laboratories," USEPA, EMSL/Cincinnati, OH 45268, EPA–600/4–80–025 (April 1980).

2. Bellar, T.A. and Lichtenberg, J.J., "Journal American Water Works Association," *66*, 739 (1974).

3. Bellar, T.A. and Lichtenberg, J.J., "Semi-automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds," in *Measurement of Organic Pollutants Water and Wastewater*, C.E. VanHall, ed., American Society for Testing Materials, Philadelphia, PA, Special Technical Publication 686, (1978).

4. "Working with Carcinogens," DHEW, PHS, NIOSH, Publication 77–206 (1977).

5. "OSHA Safety and Health Standards, General Industry," 29 CFR 1910, OSHA 2206, (1976).

6. "Safety in Academic Chemistry Laboratories," American Chemical Society Publication, Committee on Chemical Safety (1979).

7. "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL/Cincinnati, OH 45268, EPA–4–79–019 (March 1979).

8. "Methods 330.4 and 330.5 for Total Residual Chlorine," USEPA, EMSL/Cincinnati, OH 45268, EPA–4–79–020 (March 1979).

9. "Test Method: Purgeables—Method 624," USEPA, EMSL/Cincinnati, OH 45268.

10. "Colby, B.N., Beimer, R.G., Rushneck, D.R., and Telliard, W.A., "Isotope Dilution Gas Chromatography-Mass Spectrometry for the Determination of Priority Pollutants in Industrial Effluents," USEPA, Effluent Guidelines Division, Washington, DC 20460 (1980).

TABLE 1.—VOLATILE ORGANIC COMPOUNDS ANALYZED BY ISOTOPE DILUTION GC/MS

| Compound | Storet | CAS registry | EPA–EGD | NPDES |
|---|---|---|---|---|
| Acetone | 81552 | 67–64–1 | 516 V | |
| Acrolein | 34210 | 107–02–8 | 002 V | 001 V |
| Acrylonitrile | 34215 | 107–13–1 | 003 V | 002 V |
| Benzene | 34030 | 71–43–2 | 004 V | 003 V |
| Bromodichloromethane | 32101 | 75–27–4 | 048 V | 012 V |
| Bromoform | 32104 | 75–25–2 | 047 V | 005 V |
| Bromomethane | 34413 | 74–83–9 | 046 V | 020 V |
| Carbon tetrachloride | 32102 | 56–23–5 | 006 V | 006 V |
| Chlorobenzene | 34301 | 108–90–7 | 007 V | 007 V |
| Chloroethane | 34311 | 75–00–3 | 016 V | 009 V |
| 2-chloroethylvinyl ether | 34576 | 110–75–8 | 019 V | 010 V |
| Chloroform | 32106 | 67–66–1 | 023 V | 011 V |
| Chloromethane | 34418 | 74–87–3 | 045 V | 021 V |
| Dibromochloromethane | 32105 | 124–48–1 | 051 V | 008 V |
| 1,1-dichloroethane | 34496 | 75–34–3 | 013 V | 014 V |
| 1,2-dichloroethane | 32103 | 107–06–2 | 010 V | 015 V |
| 1,1-dichloroethane | 34501 | 75–35–4 | 029 V | 016 V |
| Trans-1,2-dichloroethane | 34546 | 156–60–5 | 030 V | 026 V |
| 1,2-dichloropropane | 34541 | 78–87–5 | 032 V | 017 V |
| Cis-1,3-dichloropropene | 34704 | 10061–01–5 | | |
| Trans-1,3-dichloropropene | 34699 | 10061–02–6 | 033 V | |
| Diethyl ether | 81576 | 60–29–7 | 515 V | |
| P-dioxane | 81582 | 123–91–1 | 527 V | |
| Ethylbenzene | 34371 | 100–41–4 | 038 V | 019 V |
| Methylene chloride | 34423 | 75–09–2 | 044 V | 022 V |
| Methyl ethyl ketone | 81595 | 78–93–3 | 514 V | |
| 1,1,2,2-tetrachloroethane | 34516 | 79–34–5 | 015 V | 023 V |
| Tetrachlorethene | 34475 | 127–18–4 | 085 V | 024 V |
| Toluene | 34010 | 108–88–3 | 086 V | 025 V |
| 1,1,1-trichloroethane | 34506 | 71–55–6 | 011 V | 027 V |
| 1,1,2-trichloroethane | 34511 | 79–00–5 | 014 V | 028 V |
| Trichloroethene | 39180 | 79–01–6 | 087 V | 029 V |
| Vinyl chloride | 39175 | 75–01–4 | 088 V | 031 V |

TABLE 2.—GAS CHROMATOGRAPHY OF PURGEABLE ORGANIC COMPOUNDS BY ISOTOPE DILUTION GC/MS

| EGD No. (1) | Compound | Ref EGD No. | Mean retention time (sec) | Minimum level (2) (µg/L) |
|---|---|---|---|---|
| 181 | Bromochloromethane (I.S.) | 181 | 730 | 10 |
| 245 | Chloromethane-d3 | 181 | 147 | 50 |
| 345 | Chloromethane | 245 | 148 | 50 |
| 246 | Bromomethane-d3 | 181 | 243 | 50 |
| 346 | Bromomethane | 246 | 246 | 50 |
| 288 | Vinyl chloride-d3 | 181 | 301 | 50 |
| 388 | Vinyl chloride | 288 | 304 | 10 |
| 216 | Chloroethene-d5 | 181 | 378 | 50 |
| 316 | Chloroethane | 216 | 386 | 50 |
| 244 | Methylene chloride-d2 | 181 | 512 | 10 |
| 344 | Methylene chloride | 244 | 517 | 10 |
| 616 | Acetone-d6 | 181 | 554 | 50 |
| 716 | Acetone | 616 | 565 | 50 |
| 002 | Acrolein | 181 | 586 | 50 |
| 203 | Acrylonitrile-d3 | 181 | 606 | 50 |
| 303 | Acrylonitrile | 203 | 612 | 50 |
| 229 | 1,1-dichloroethene-d2 | 181 | 696 | 10 |
| 329 | 1,1-dichloroethene | 229 | 696 | 10 |
| 213 | 1,1-dichloroethane-d3 | 181 | 778 | 10 |
| 313 | 1,1-dichloroethane | 213 | 786 | 10 |
| 615 | Diethyl ether-d10 | 181 | 804 | 50 |
| 715 | Diethyl ether | 615 | 820 | 50 |
| 230 | Trans-1,2-dichloroethene-d2 | 181 | 821 | 10 |
| 330 | Trans-1,2-dichloroethene | 230 | 821 | 10 |
| 614 | Methyl ethyl ketone-d3 | 181 | 840 | 50 |
| 714 | Methyl ethyl ketone | 614 | 848 | 50 |
| 223 | Chloroform-13C1 | 181 | 861 | 10 |
| 323 | Chloroform | 223 | 861 | 10 |
| 210 | 1,2-dichloroethane-d4 | 181 | 901 | 10 |
| 310 | 1,2-dichloroethane | 210 | 910 | 10 |
| 211 | 1,1,1-trichloroethane-13C2 | 181 | 989 | 10 |
| 311 | 1,1,1-trichloroethane | 211 | 999 | 10 |
| 527 | p-dioxane | 181 | 1001 | 10 |
| 206 | Carbon tetrachloride-13C1 | 182 | 1018 | 10 |
| 306 | Carbon tetrachloride | 206 | 1018 | 10 |
| 248 | Bromodichloromethane-13C1 | 182 | 1045 | 10 |
| 348 | Bromodichloromethane | 248 | 1045 | 10 |
| 232 | 1,2-dichloropropane-d6 | 182 | 1123 | 10 |
| 332 | 1,2-dichloropropane | 232 | 1134 | 10 |
| 233 | Trans-1,3-dichloropropene-d4 | 182 | 1138 | 10 |
| 333 | Trans-1,3-dichloropropene | 233 | 1138 | 10 |
| 287 | Trichloroethene-13C1 | 182 | 1172 | 10 |
| 387 | Trichloroethene | 287 | 1187 | 10 |
| 204 | Benzene-d6 | 182 | 1200 | 10 |
| 304 | Benzene | 204 | 1212 | 10 |
| 251 | Chlorodibromemethane-13C1 | 182 | 1222 | 10 |
| 351 | Chlorodibromomethane | 251 | 1222 | 10 |
| 214 | 1,1,2-trichloroethane-13C2 | 182 | 1224 | 10 |
| 314 | 1,1,2-trichloroethane | 214 | 1224 | 10 |
| 019 | 2-chloroethylvinyl ether | 182 | 1278 | 10 |
| 182 | 2-bromo-1-chloropropane (I.S.) | 182 | 1306 | 10 |
| 247 | Bromoform-13C1 | 182 | 1386 | 10 |
| 347 | Bromoform | 247 | 1386 | 10 |
| 215 | 1,1,2,2-tetrachloroethane-d2 | 183 | 1525 | 10 |
| 315 | 1,1,2,2-tetrachloroethane | 215 | 1525 | 10 |
| 285 | Tetrachloroethene-13C2 | 183 | 1528 | 10 |
| 385 | Tetrachloroethene | 285 | 1528 | 10 |
| 183 | 1,4-dichlorobutane (int std) | 183 | 1555 | 10 |
| 286 | Toluene-d8 | 183 | 1603 | 10 |
| 386 | Toluene | 286 | 1619 | 10 |
| 207 | Chlorobenzene-d5 | 183 | 1679 | 10 |
| 307 | Chlorobenzene | 207 | 1679 | 10 |
| 238 | Ethylbenzene-d10 | 183 | 1802 | 10 |
| 338 | Ethylbenzene | 238 | 1820 | 10 |
| 185 | Bromofluorobenzene | 183 | 1985 | 10 |

(1) Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a poliutant quantified by isotope dilution.

(2) This is a minimum level at which the analytical system shall give recognizable mass spectra (background corrected) and acceptable calibration points. Column: 2.4m (8 ft) x 2 mm i.d. glass, packed with one percent SP–1000 coated on 60/80 Carbopak B. Carrier gas: helium at 40 mL/min. Temperature program: 3 min at 45 °C, 8 °C per min to 240 °C, hold at 240 °C for 15 minutes.

**Note.**—The specifications in this table were developed from data collected from three wastewater laboratories.

TABLE 3.—BFB Mass-Intensity Specifications

| Mass | Intensity required |
|---|---|
| 50 | 15 to 40 percent of mass 95. |
| 75 | 30 to 60 percent of mass 95. |
| 95 | base peak, 100 percent. |
| 96 | 5 to 9 percent of mass 95. |
| 173 | <2 percent of mass 174. |
| 174 | >50 percent of mass 95. |
| 176 | 95 to 100 percent of mass 174. |
| 177 | 5 to 9 percent of mass 176. |

TABLE 4.—VOLATILE ORGANIC COMPOUND CHARACTERISTIC MASSES

| Labeled compound | Analog | Primary m/z's |
|---|---|---|
| Acetone | d6 | 58/64 |
| Acrolein | d2 | 56/58 |
| Acrylonitrile | d3 | 53/56 |
| Benzene | d6 | 78/84 |
| Bromodichloromethane | 13C | 83/86 |
| Bromoform | 13C | 173/176 |
| Bromomethane | d3 | 96/99 |
| Carbon tetrachloride | 13C | 47/48 |
| Chlorobenzene | d5 | 112/117 |
| Chloroethane | d5 | 64/71 |
| 2-chloroethylvinyl ether | d7 | 106/113 |
| Chloroform | 13C | 85/86 |
| Chloromethane | d3 | 50/53 |
| Dibromochloromethane | 13C | 129/130 |
| 1,1-dichloroethane | d3 | 63/66 |
| 1,2-dichloroethane | d4 | 62/67 |
| 1,1-dichloroethene | d2 | 61/65 |
| Trans-1,2-dichloroethene | d2 | 61/65 |
| 1,2-dichloropropane | d6 | 63/67 |
| Cis-1,3-dichloropropene | d4 | 75/79 |
| Trans-1,3-dichloropropene | d4 | 75/79 |
| Diethyl ether | d10 | 74/84 |
| p-dioxane | d8 | 88/96 |
| Ethylbenzene | d10 | 106/116 |
| Methylene chloride | d2 | 84/88 |
| Methyl ethyl ketone | d3 | 72/75 |
| 1,1,2,2-tetrachloroethane | d2 | 83/84 |
| Tetrachloroethene | 13C2 | 166/172 |
| Toluene | d8 | 92/99 |
| 1,1,1-trichloroethane | d3 | 97/102 |
| 1,1,2-trichloroethane | 13C2 | 83/84 |
| Trichloroethene | 13C | 95/133 |
| Vinyl chloride | d3 | 62/65 |

TABLE 5.—ACCEPTANCE CRITERIA FOR PERFORMANCE TESTS

| Compound | Acceptance criteria at 20 μg/L | | | |
|---|---|---|---|---|
| | Initial precision and accuracy section 8.2.3 | | Labeled compound recovery sec 8.3 and 14.2 | On-going accuracy sec 11.5 |
| | s (μg/L) | $\bar{X}$ (μg/L) | P (percent) | R (μg/L) |
| Acetone | | note 1 | | |
| Acrolein | | note 2 | | |
| Acrylonitrile | | note 2 | | |
| Benzene | 9.0 | 13.0–28.2 | ns–196 | 4–33 |
| Bromodichloromethane | 8.2 | 6.5–31.5 | ns–199 | 4–34 |
| Bromoform | 7.0 | 7.4–35.1 | ns–214 | 6–36 |
| Bromomethane | 25.0 | d–54.3 | ns–414 | d–61 |
| Carbon tetrachloride | 6.9 | 15.9–24.8 | 42–165 | 12–30 |
| Chlorobenzene | 8.2 | 14.2–29.6 | ns–205 | 4–35 |
| Chloroethane | 14.8 | 2.1–46.7 | ns–308 | d–51 |
| 2-chloroethylvinyl ether | 36.0 | d–69.8 | ns–554 | d–79 |
| Chloroform | 7.9 | 11.6–26.3 | 18–172 | 8–30 |
| Chloromethane | 26.0 | d–55.5 | ns–410 | d–64 |
| Dibromochloromethane | 7.9 | 11.2–29.1 | 16–185 | 8–32 |
| 1,1-dichloroethane | 6.7 | 11.4–31.4 | 23–191 | 9–33 |
| 1,2-dichloroethane | 7.7 | 11.6–30.1 | 12–192 | 8–33 |
| 1,1-dichloroethene | 11.7 | d–49.8 | ns–315 | d–52 |
| Trans-1,2-dichloroethene | 7.4 | 10.5–31.5 | 15–195 | 8–34 |
| 1,2-dichloropropane | 19.2 | d–46.8 | ns–343 | d–51 |
| Cis-1,3-dichloropropene | 22.1 | d–51.0 | ns–381 | d–56 |
| Trans-1,3-dichloropropene | 14.5 | d–40.2 | ns–284 | d–44 |
| Diethyl ether | | note 1 | | |
| P-dioxane | | note 1 | | |
| Ethyl benzene | 9.6 | 15.6–28.5 | ns–203 | 5–35 |
| Methylene chloride | 9.7 | d–49.8 | ns–316 | d–50 |
| Methyl ethyl ketone | | note 1 | | |
| 1,1,2,2-tetrachloroethane | 9.6 | 10.7–30.0 | 5–199 | 7–34 |
| Tetrachloroethene | 6.6 | 15.1–28.5 | 31–181 | 11–32 |
| Toluene | 6.3 | 14.5–28.7 | 4–193 | 6–33 |
| 1,1,1-trichloroethane | 5.9 | 10.5–33.4 | 12–200 | 8–35 |
| 1,1,2-trichloroethane | 7.1 | 11.8–29.7 | 21–184 | 9–32 |
| Trichloroethene | 8.9 | 16.6–29.5 | 35–196 | 12–34 |
| Vinyl chloride | 27.9 | d–58.5 | ns–452 | d–65 |

d=detected; result must be greater than zero.
ns=no specification; limit would be below detection limit.

**Note 1:** Specifications not available for these compounds at time of release of this method.
**Note 2:** Specifications not developed for these compounds; use method 603.

BILLING CODE 6560–50–M

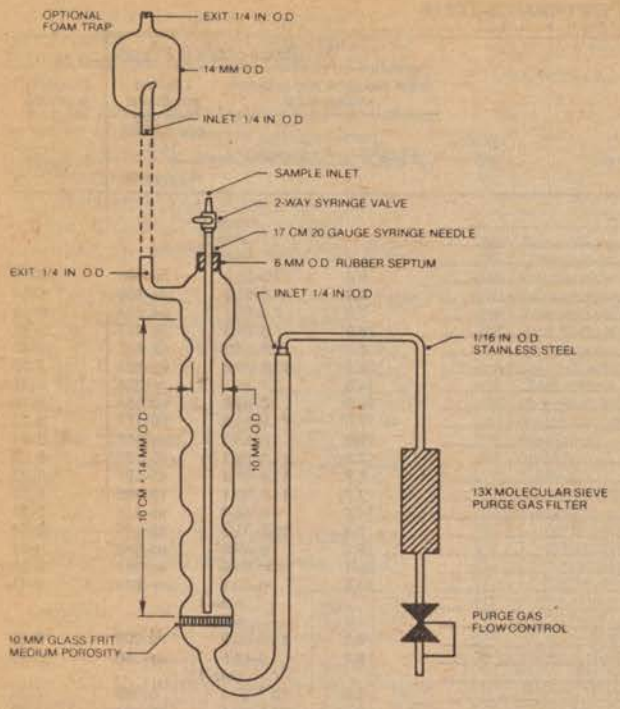



FIGURE 1 Purging Device.

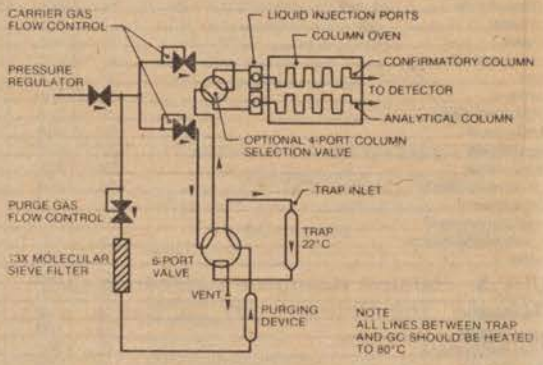



FIGURE 3 Schematic of Purge and Trap Device—Purge Mode.

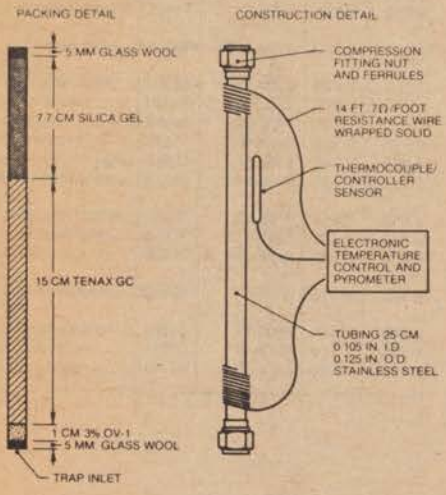



FIGURE 2 Trap Packings and Construction to Include Desorb Capability.

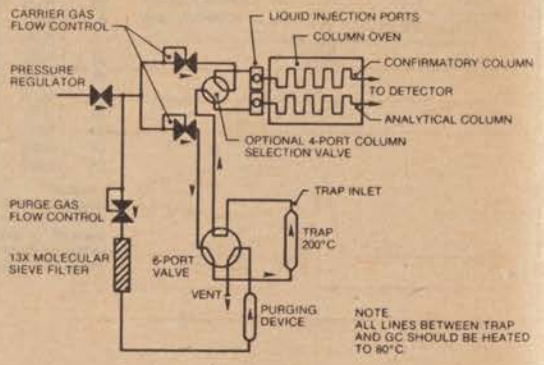



FIGURE 4 Schematic of Purge and Trap Device—Desorb Mode.

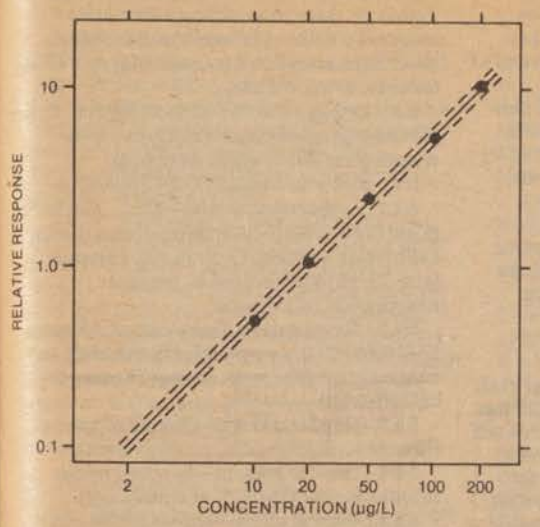



FIGURE 5 Relative Response Calibration Curve for Toluene. The Dotted Lines Enclose a ±10 Percent Error Window.

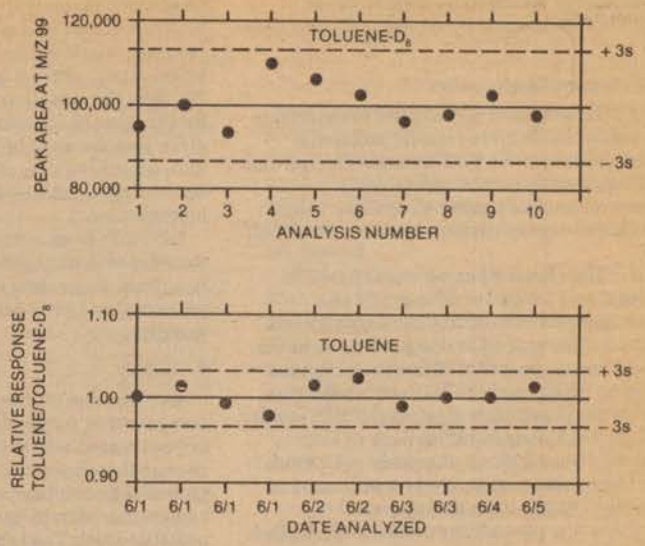



FIGURE 7 Quality Control Charts Showing Area (top graph) and Relative Response of Toluene to Toluene-$d_8$ (lower graph) Plotted as a Function of Time or Analysis Number.

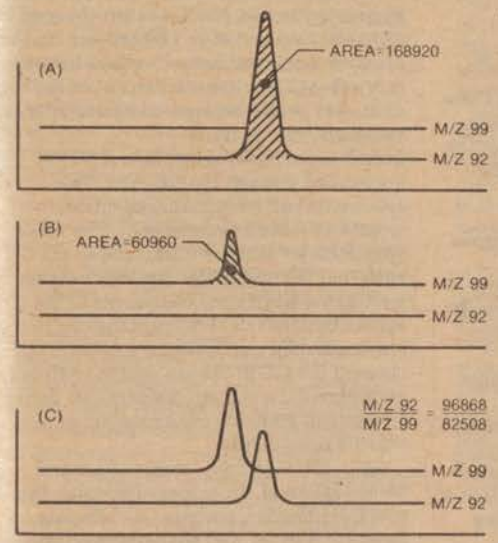



FIGURE 6 Extracted Ion Current Profiles for (A) Toluene, (B) Toluene-$d_8$, and a Mixture of Toluene and Toluene-$d_8$.

BILLING CODE 6560-50-C

**Method 1625 Revision B—Semivolatile Organic Compounds by Isotope Dilution GC/MS**

*1 Scope and application*

1.1 This method is designed to determine the semivolatile toxic organic pollutants associated with the 1976 Consent Decree and additional compounds amenable to extraction and analysis by capillary column gas chromatography-mass spectrometry (GC/MS).

1.2 The chemical compounds listed in tables 1 and 2 may be determined in municipal and industrial discharges by this method. The method is designed to meet the survey requirements of Effluent Guidelines Division (EGD) and the National Pollutants Discharge Elimination System (NPDES) under 40 CFR 136.1. Any modifications of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.3 The detection limit of this method is usually dependent on the level of interferences rather than instrumental limitations. The limits listed in tables 3 and 4 represent the minimum quantity that can be detected with no interferences present.

1.4 The GC/MS portions of this method are for use only by analysts experienced with GC/MS or under the close supervision of such qualified persons. Laboratories unfamiliar with analyses of environmental samples by GC/MS should run the performance tests in reference 1 before beginning.

*2 Summary of method*

2.1 Stable isotopically labeled analogs of the compounds of interest are added to a one liter wastewater sample. The sample is extracted at pH 12–13, then at pH <2 with methylene chloride using continuous extraction techniques. The extract is dried over sodium sulfate and concentrated to a volume of one mL. An internal standard is added to the extract, and the extract is injected into the gas chromatograph (GC). The compounds are separated by GC and detected by a mass spectrometer (MS). The labeled compounds serve to correct the variability of the analytical technique.

2.2 Identification of a compound (qualitative analysis) is performed by comparing the GC retention time and background corrected characteristic spectral masses with those of authentic standards.

2.3 Quantitative analysis is performed by GC/MS using extracted ion current profile (EICP) areas. Isotope dilution is used when labeled compounds are available; otherwise, an internal or external standard method is used.

2.4 Quality is assured through reproducible calibration and testing of the extraction and GC/MS systems.

*3 Contamination and interferences*

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms and spectra. All materials shall be demonstrated to be free from interferences under the conditions of analysis by running method blanks initially and with each sample lot (samples started through the extraction process on a given 8 hr shift, to a maximum of 20). Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Glassware and, where possible, reagents are cleaned by solvent rinse and baking at 450 °C for one hour minimum.

3.2 Interferences coextracted from samples will vary considerably from source to source, depending on the diversity of the industrial complex or municipality being samples.

*4 Safety*

4.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should also be made available to all personnel involved in these analyses. Additional information on laboratory safety can be found in references 2–4.

4.2 The following compounds covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: benzo(a)anthracene, 3,3'-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, N-nitrosodimethylamine, and B-naphthylamine. Primary standards of these compounds shall be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator should be worn when high concentrations are handled.

*5 Apparatus and materials*

5.1 Sampling equipment for discrete or composite sampling.

5.1.1 Sample bottle, amber glass, 1.1 liters minimum. If amber bottles are not available, samples shall be protected from light. Bottles are detergent water washed, then solvent rinsed or baked at 450 °C for one hour minimum before use.

5.1.2 Bottle caps—threaded to fit sample bottles. Caps are lined with Teflon. Aluminum foil may be substituted if the sample is not corrosive. Liners are detergent water washed, then reagent water (section 6.5) and solvent rinsed, and baked at approximately 200 °C for one hour minimum before use.

5.1.3 Compositing equipment—automatic or manual compositing system incorporating glass containers for collection of a minimum 1.1 liters. Sample containers are kept at 0 to 4 °C during sampling. Glass or Teflon tubing only shall be used. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used in the pump only. Before use, the tubing is thoroughly rinsed with methanol, followed by repeated rinsings with reagent water (section 6.5) to minimize sample contamination. An integrating flow meter is used to collect proportional composite samples.

5.2 Continuous liquid-liquid extractor—Teflon or glass conncecting joints and stopcocks without lubrication (Hershberg-Wolf Extractor) one liter capacity, Ace Glass 6841–10, or equivalent.

5.3 Drying column—15 to 20 mm i.d. Pyrex chromatographic column equipped with coarse glass frit or glass wool plug.

5.4 Kuderna-Danish (K–D) apparatus

5.4.1 Concentrator tube—10mL, graduated (Kontes K–570050–1025, or equivalent) with calibration verified. Ground glass stopper (size 19/22 joint) is used to prevent evaporation of extracts.

5.4.2 Evaporation flask—500 mL (Kontes K–570001–0500, or equivalent), attached to concentrator tube with springs (Kontes K–662750–0012).

5.4.3 Snyder column—three ball macro (Kontes K–503000–0232, or equivalent).

5.4.4 Snyder column—two ball micro (Kontes K–469002–0219, or equivalent).

5.4.5 Boiling chips—approx 10/40 mesh, extracted with methylene chloride and baked at 450 °C for one hr minimum.

5.5 Water bath—heated, with concentric ring cover, capable of temperature control (q 2 °C), installed in a fume hood.

5.6 Sample vials—amber glass, 2–5 mL with Teflon-lined screw cap.

5.7 Analytical balance—capable of weighing 0.1 mg.

5.8 Gas chromatograph—shall have splitless or on-column injection port for capillary column, temperature program with 30 °C hold, and shall meet all of the performance specifications in section 12.

5.8.1 Column—30±5 m×0.25±0.02 mm i.d. 5% phenyl, 94% methyl, 1% vinyl silicone bonded phase fused silica capillary column (J & W DB–5, or equivalent).

5.9 Mass spectrometer—70 eV electron impact ionization, shall repetitively scan from 35 to 450 amu in 0.95 to 1.00 second, and shall produce a unit resolution (valleys between m/z 441–442 less than 10 percent of the height of the 441 peak), backgound corrected mass spectrum from 50 ng decafluorotriphenylphosphine (DFTPP) introduced through the GC inlet. The spectrum shall meet the mass-intensity criteria in table 5 (reference 5). The mass spectrometer shall be interfaced to the GC such that the end of the capillary column terminates within one centimeter of the ion source but does not intercept the electron or ion beams. All portions of the column which connect the GC to the ion source shall remain at or above the column temperature during analysis to preclude condensation of less volatile compounds.

5.10 Data system—shall collect and record MS data, store mass-intensity data in spectral libraries, process GC/MS data, generate reports, and shall compute and record response factors.

5.10.1 Data acquisition—mass spectra shall be collected continuously throughout the analysis and stored on a mass storage device.

5.10.2 Mass spectral libraries—user created libraries containing mass spectra obtained from analysis of authentic standards shall be employed to reverse

search GC/MS runs for the compounds of interest (section 7.2).

5.10.3 Data processing—the data system shall be used to search, locate, identify, and quantify the compounds of interest in each GC/MS analysis. Software routines shall be employed to compute retention times and peak areas. Displays of spectra, mass chromatograms, and library comparisons are required to verify results.

5.10.4 Response factors and multipoint calibrations—the data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and multipoint calibration curves (section 7). Computations of relative standard deviation (coefficient of variation) are useful for testing calibration linearity. Statistics on initial (section 8.2) and on-going (section 12.7) performance shall be computed and maintained.

*6 Reagents and standards*

6.1 Sodium hydroxide—reagent grade, 6N in reagent water.

6.2 Sulfuric acid—reagent grade, 6N in reagent water.

6.3 Sodium sulfate—reagent grade, granular anhydrous, rinsed with methylene chloride (20 mL/g) and conditioned at 450 °C for one hour minimum.

6.4 Methylene chloride—distilled in glass (Burdick and Jackson, or equivalent).

6.5 Reagent water—water in which the compounds of interest and interfering compounds are not detected by this method.

6.6 Standard solutions—purchased as solutions or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If compound purity is 96 percent or greater, the weight may be used without correction to compute the concentration of the standard. When not being used, standards are stored in the dark at −20 to −10 °C in screw-capped vials with Teflon-lined lids. A mark is placed on the vial at the level of the solution so that solvent evaporation loss can be detected. The vials are brought to room temperature prior to use. Any precipitate is redissolved and solvent is added if solvent loss has occurred.

6.7 Preparation of stock solutions—prepare in methylene chloride, benzene, p-dioxane, or a mixture of these solvents per the steps below. Observe the safety precautions in section 4. The large number of labeled and unlabeled acid, base/neutral, and Appendix C compounds used for combined calibration (section 7) and calibration verification (12.5) require high concentrations (approx 40 mg/mL) when individual stock solutions are prepared, so that dilutions of mixtures will permit calibration with all compounds in a single set of solutions. The working range for most compounds is 10–200 ug/mL. Compounds with a reduced MS response may be prepared at higher concentrations.

6.7.1 Dissolve an appropriate amount of assayed reference material in a suitable solvent. For example, weigh 400 mg naphthalene in a 10 mL ground glass stoppered volumetric flask and fill to the mark with benzene. After the naphthalene is completely dissolved, transfer the solution to a 15 mL vial with Teflon-lined cap.

6.7.2 Stock standard solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards. Quality control check samples that can be used to determine the accuracy of calibration standards are available from the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

6.7.3 Stock standard solutions shall be replaced after six months, or sooner if comparison with quality control check samples indicates a change in concentration.

6.8 Labeled compound spiking solution—from stock standard solutions prepared as above, or from mixtures, prepare the spiking solution at a concentration of 200 μg/mL, or at a concentration appropriate to the MS response of each compound.

6.9 Secondary standard—using stock solutions (section 6.7), prepare a secondary standard containing all of the compounds in tables 1 and 2 at a concentration of 400 μg/mL, or higher concentration appropriate to the MS response of the compound.

6.10 Internal standard solution—prepare 2,2′-difluorobiphenyl (DFB) at a concentration of 10 mg/mL in benzene.

6.11 DFTPP solution—prepare at 50 μg/mL in acetone.

6.12 Solutions for obtaining authentic mass spectra (section 7.2)—prepare mixtures of compounds at concentrations which will assure authentic spectra are obtained for storage in libraries.

6.13 Calibration solutions—combine 0.5 mL of the solution in section 6.8 with 25, 50, 125, 250, and 500 μL of the solution in section 6.9 and bring to 1.00 μL total volume each. This will produce calibration solutions of nominal 10, 20, 50, 100, and 200 μg/mL of the pollutants and a constant nominal 100 μg/mL of the labeled compounds. Spike each solution with 10 uL of the internal standard solution (section 6.10). These solutions permit the relative response (labeled to unlabeled) to be measured as a function of concentration (section 7.4).

6.14 Precision and recovery standard—used for determination of initial (section 8.2) and on-going (section 12.7) precision and recovery. This solution shall contain the pollutants and labeled compounds at a nominal concentration of 100 μg/mL.

6.15 Stability of solutions—all standard solutions (sections 6.8–6.14) shall be analyzed within 48 hours of preparation and on a monthly basis thereafter for signs of degradation. Standards will remain acceptable if the peak area at the quantitation mass relative to the DFB internal standard remains within ±15 percent of the area obtained in the initial analysis of the standard.

*7 Calibration*

7.1 Assemble the GC/MS and establish the operating conditions in table 3. Analyze standards per the procedure in section 11 to demonstrate that the analytical system meets the detection limits in tables 3 and 4, and the mass-intensity criteria in table 5 for 50 ng DFTPP.

7.2 Mass spectral libraries—detection and identification of compounds of interest are dependent upon spectra stored in user created libraries.

7.2.1 Obtain a mass spectrum of each pollutant, labeled compound, and the internal standard by analyzing an authentic standard either singly or as part of a mixture in which there is no interference between closely eluted components. That only a single compound is present is determined by examination of the spectrum. Fragments not attributable to the compound under study indicate the presence of an interfering compound.

7.2.2 Adjust the analytical conditions and scan rate (for this test only) to produce an undistorted spectrum at the GC peak maximum. An undistorted spectrum will usually be obtained if five complete spectra are collected across the upper half of the GC peak. Software algorithms designed to "enhance" the spectrum may eliminate distortion, but may also eliminate authentic masses or introduce other distortion.

7.2.3 The authentic reference spectrum is obtained under DFTPP tuning conditions (section 7.1 and table 5) to normalize it to spectra from other instruments.

7.2.4 The spectrum is edited by saving the 5 most intense mass spectral peaks and all other mass spectral peaks greater than 10 percent of the base peak. This edited spectrum is stored for reverse search and for compound confirmation.

7.3 Analytical range—demonstrate that 20 ng anthracene or phenanthrene produces an area at m/z 178 approx one-tenth that required to exceed the linear range of the system. The exact value must be determined by experience for each instrument. It is used to match the calibration range of the instrument to the analytical range and detection limits required, and to diagnose instrument sensitivity problems (section 15.4). The 20 ug/mL calibration standard (section 6.13) can be used to demonstrate this performance.

7.3.1 Polar compound detection—demonstrate that unlabeled pentachlorophenol and benzidine are detectable at the 50 ug/mL level (per all criteria in section 13). The 50 ug/mL calibration standard (section 6.13) can be used to demonstrate this performance.

7.4 Calibration with isotope dilution—isotope dilution is used when 1) labeled compounds are available, 2) interferences do not preclude its use, and 3) the quantitation mass extracted ion current profile (EICP) area for the compound is in the calibration range. If any of these conditions preclude isotope dilution, internal or external standard methods (section 7.5 or 7.6) are used.

7.4.1 A calibration curve encompassing the concentration range is prepared for each compound to be determined. The relative response (pollutant to labeled) vs concentration in standard solutions is plotted or computed using a linear regression. The example in Figure 1 shows a calibration curve for phenol using phenol-d5 as the isotopic diluent. Also shown are the ± 10 percent error limits (dotted lines). Relative Reponse (RR) is determined according to the procedures described below. A minimum of five data points are employed for calibration.

7.4.2 The relative response of a pollutant to its labeled analog is determined from

isotope ratio values computed from acquired data. Three isotope ratios are used in this process:

$R_x$ = the isotope ratio measured for the pure pollutant.

$R_y$ = the isotope ratio measured for the labeled compound.

$R_m$ = the isotope ratio of an analytical mixture of pollutant and labeled compounds.

The m/z's are selected such that $R_x = R_y$. If $R_m$ is not between $2R_y$ and $0.5R_z$, the method does not apply and the sample is analyzed by internal or external standard methods.

7.4.3 Capillary columns usually separate the pollutant-labeled pair, with the labeled compound eluted first (figure 2). For this case, $R_x$ = [area $m_1/z$]/1, at the retention time of the pollutant ($RT_2$). $R_y$ = 1/[area $m_2/z$, at the retention time of the labeled compound $RT_1$). $R_m$ = [area at $m_1/z$ (at $RT_2$)]/[area at $RT_1$)], as measured in the mixture of the pollutant and labeled compounds (figure 2), and RR = $R_m$.

7.4.4 Special precautions are taken when the pollutant-labeled pair is not separated, or when another labeled compound with interfering spectral masses overlaps the pollutant (a case which can occur with isomeric compounds). In this case, it is necessary to determine the respective contributions of the pollutant and labeled compounds to the respective EICP areas. If the peaks are separated well enough to permit the data system or operator to remove the contributions of the compounds to each other, the equations in section 7.4.3 apply. This usually occurs when the height of the valley between the two GC peaks at the same m/z is less than 10 percent of the height of the shorter of the two peaks. If significant GC and spectral overlap occur, RR is computed using the following equation: RR = $(R_y - R_m)(R_x + 1)/(R_m - R_y + 1)$, where $R_x$ is measured as shown in figure 3A, $_y$ is measured as shown in figure 3B, and $R_m$ is measured as shown in figure 3C. For example, $R_x$ = 46100/4780 = 9.644, $R_y$ = 2650/43600 = 0.0608, $R_m$ = 49200/48300 = 1.019. amd RR = 1.114.

7.4.5 To calibrate the analytical system by isotope dilution, analyze a 1.0 µL aliquot of each of the calibration standards (section 6.13) using the procedure in section 11. Compute the RR at each concentration.

7.4.6 Linearity—if the ratio of relative response to concentration for any compound is constant (less than 20 percent coefficient of variation) over the 5 point calibration range, and averaged relative response/concentration ratio may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point calibration range.

7.5 Calibration by internal standard—used when criteria for istope dilution (section 7.4) cannot be met. The internal standard to be used for both acid and base/neutral analyses is 2,2'–difluorobiphenyl. The internal standard method is also applied to determination of compounds having no labeled analog, and to measurement of labeled compounds for intra-laboratory statistics (sections 8.4 and 12.7.4).

7.5.1 Response factors—calibration requires the determination of response factors (RF) which are defined by the following equation:

RF = $(A_s \times C_{is})/(A_{is} \times C_s$, where

$A_s$ is the area of the characteristic mass for the compound in the daily standard

$A_{is}$ is the area of the characteristic mass for the internal standard

$C_{is}$ is the concentration of the internal standard (µg/mL)

$C_s$ is the concentration of the compound in the daily standard (µg/mL)

7.5.1.1 The response factor is determined for at least five concentrations appropriate to the response of each compound (section 6.13); nominally, 10, 20, 50, 100, and 200 µg/mL. The amount of internal standard added to each extract is the same (100 µg/mL) so that $C_{is}$ remains constant. The RF is plotted vs concentration for each compound in the standard ($C_s$) to produce a calibration curve.

7.5.1.2 Linearity—if the response factor (RF) for any compound is constant (less than 35 percent coefficient of variation) over the 5 point calibration range, an averaged response factor may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point range.

7.6 Combined calibration—by using calibration solutions (section 6.13) containing the pollutants, labeled compounds, and the internal standard, a single set of analyses can be used to produce calibration curves for the isotope dilution and internal standard methods. These curves are verified each shift (section 12.5) by analyzing the 100 µg/mL calibration standard (section 6.13). Recalibration is required only if calibration verification (section 12.5) criteria cannot be met.

*8 Quality assurance/quality control*

8.1 Each laboratory that uses this method is required to operate a formal quality assurance program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

8.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in section 8.2.

8.1.2 The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance specifications are met. Each time a modification is made to the method, the analyst is required to repeat the procedure in section 8.2 to demonstrate method performance.

8.1.3 Analyses of blanks are required to demonstrate freedom from contamination. The procedures and criteria for analysis of a blank are described in section 8.5.

8.1.4 The laboratory shall spike all samples with labeled compounds to monitor method performance. This test is described in section 8.3. When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method performance within acceptable limits (section 15).

8.1.5 The laboratory shall, on an on-going basis, demonstrate through calibration verification and the analysis of the precision and recovery standard (section 6.14) that the analysis system is in control. These procedures are described in sections 12.1, 12.5, and 12.7.

8.1.6 The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in section 8.4.

8.2 Initial precision and accuracy—to establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

8.2.1 Extract, concentrate, and analyze two sets of four one-liter aliquots (8 aliquots total) of the precision and recovery standard (section 6.14) according to the procedure in section 10.

8.2.2 Using results of the first set of four analyses, compute the average recovery ($\bar{X}$) in µg/mL and the standard deviation of the recovery (s) in µg/mL for each compound, by isotope dilution for pollutants with a labeled analog, and by internal standard for labeled compounds and pollutants with no labeled analog.

8.2.3 For each compound, compare s and $\bar{X}$ with the corresponding limits for initial precision and accuracy in table 8. If s and $\bar{X}$ for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If, however, any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, system performance is unacceptable for that compound. **Note:** The large number of compounds in table 8 present a substantial probability that one or more will fail the acceptance criteria when all compounds are analyzed. To determine if the analytical system is out of control, or if the failure can be attributed to probability, proceed as follows:

8.2.4 Using the results of the second set of four analyses, compute s and $\bar{X}$ for only those compounds which failed the test of the first set of four analyses (section 8.2.3). If these compounds now pass, system performance is acceptable for all compounds and analysis of blanks and samples may begin. If, however, any of the same compounds fail again, the analysis system is not performing properly for these compounds. In this event, correct the problem and repeat the entire test (section 8.2.1).

8.3 The laboratory shall spike all samples with labeled compounds to assess method performance on the sample matrix.

8.3.1 Analyze each sample according to the method in section 10.

8.3.2 Compute the percent recovery (P) of the labeled compounds using the internal standard method (section 7.5).

8.3.3 Compare the labeled compound recovery for each compound with the corresponding limits in table 8. If the recovery of any compounds falls outside its warning limit, method performance is unacceptable for that compound in that sample. Therefore, the sample is complex and

is to be diluted and reanalyzed per section 15.4.

8.4 As part of the QA program for the laboratory, method accuracy for wastewater samples shall be assessed and records shall be maintained. After the analysis of five wastewater samples for which the labeled compounds pass the tests in section 8.3, compute the average percent recovery (P) and the standard deviation of the percent recovery ($s_p$) for the labeled compounds only. Express the accuracy assessment as a percent recovery interval from $P-2_{sp}$ to $P+2_{sp}$. For example, if P=90% and $s_p$=10%, the accuracy interval is expressed as 70–100%. Update the accuracy assessment for each compound on a regular basis (e.g. after each 5–10 new accuracy measurements).

8.5 Blanks—reagent water blanks are analyzed to demonstrate freedom from contamination.

8.5.1 Extract and concentrate a blank with each sample lot (samples started through the extraction process on the same 8 hr shift, to a maximum of 20 samples). Analyze the blank immediately after analysis of the precision and recovery standard (section 6.14) to demonstrate freedom from contamination.

8.5.2 If any of the compounds of interest (tables 1 and 2) or any potentially interfering compound is found in a blank at greater than 10 μg/L (assuming a response factor of 1 relative to the internal standard for compounds not listed in tables 1 and 2), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

8.6 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state. The standards used for calibration (section 7), calibration verification (section 12.5), and for initial (section 8.2) and on-going (section 12.7) precision and recovery should be identical, so that the most precise results will be obtained. The GC/MS instrument in particular will provide the most reproducible results if dedicated to the settings and conditions required for the analysis of semi-volatiles by this method.

8.7 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when internal or external standard methods are used.

*9 Sample collection, preservation, and handling*

9.1 Collect samples in glass containers following conventional sampling practices (reference 7). Composite samples are collected in refrigerated glass containers (section 5.1.3) in accordance with the requirements of the sampling program.

9.2 Maintain samples at 0–4 °C from the time collection until extraction. If residual chlorine is present, add 80 mg sodium thiosulfate per liter of water. EPA methods 330.4 and 330.5 may be used to measure residual chlorine (reference 8).

9.3 Begin sample extraction within seven days of collection, and analyze all extracts within 40 days of extraction.

*10 Sample extraction and concentration (See figure 4)*

10.1 Labeled compound spiking—measure 1.00 ± 0.01 liter of sample into a glass container. For untreated effluents, and samples which are expected to be difficult to extract and/or concentrate, measure an additional 10.0 ± 0.1 mL and dilute to a final volume of 1.00 ± 0.01 liter with reagent water in a glass container.

10.1.1 For each sample or sample lot (to a maximum of 20) to be extracted at the same time, place three 1.00 ± 0.10 liter aliquots of reagent water in glass containers.

10.1.2 Spike 0.5 mL of the labeled compound spiking solution (section 6.8) into all samples and one reagant water aliquot.

10.1.3 Spike 1.0 mL of the precision and recovery standard (section 6.14) into the two remaining reagent water aliquots.

10.1.4 Stir and equilibrate all solutions for 1–2 hr.

10.2 Base/neutral extraction—place 100–150 mL methylene chloride in each continuous extractor and 200–300 in each distilling flask.

10.2.1 Pour the sample(s), blank, and standard aliquots into the extractors. Rinse the glass containers with 50–100 mL methylene chloride and add to the respective extractor.

10.2.2 Adjust the pH of the waters in the extractors to 12–13 with 6N NaOH while monitoring with a pH meter. Begin the extraction by heating the flask until the methylene chloride is boiling. When properly adjusted, 1–2 drops of methylene chloride per second will fall from the condensor tip into the water. After 1–2 hours of extraction, test the pH and readjust to 12–13 if required. Extract for 18–24 hours.

10.2.3 Remove the distilling flask, estimate and record the volume of extract (to the nearest 100 mL), and pour the contents through a drying column containing 7 to 10 cm anhydrous sodium sulfate. Rinse the distilling flask with 30–50 mL of methylene chloride and pour through the drying column. Collect the solution in a 500 mL K–D evaporator flask equipped with a 10 mL concentrator tube. Seal, label as the base/neutral fraction, and concentrate per sections 10.4 to 10.5.

10.3 Acid extraction—adjust the pH of the waters in the extractors to 2 or less using 6N sulfuric acid. Charge clean distilling flasks with 300–400 mL of methylene chloride. Test and adjust the pH of the waters after the first 1–2 hr of extraction. Extract for 18–24 hours.

10.3.1 Repeat section 10.2.3, except label as the acid fraction.

10.4 Concentration—concentrate the extracts in separate 500 mL K–D flasks equipped with 10 mL concentrator tubes.

10.4.1 Add 1 to 2 clean boiling chips to the flask and attach a three-ball macro Snyder column. Prewet the column by adding approximately one mL of methylene chloride through the top. Place the K–D apparatus in a hot water bath so that the entire lower rounded surface of the flask is bathed with steam. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid has reached an apparent volume of 1 mL, remove the K–D apparatus from the bath and allow the solvent to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1–2 mL of methylene chloride. A 5-mL syringe is recommended for this operation.

10.4.2 For performance standards (sections 8.2 and 12.7) and for blanks (section 8.5), combine the acid and base/neutral extracts for each at this point. Do not combine the acid and base/neutral extracts for samples.

10.5 Add a clean boiling chip and attach a two ball micro Snyder column to the concentrator tube. Prewet the column by adding approx 0.5 mL methylene chloride through the top. Place the apparatus in the hot water bath. Adjust the vertical position and the water temperature as required to complete the concentration in 5–10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid reaches an apparent volume of approx 0.5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes. Remove the micro Snyder column and rinse its lower joint into the concentrator tube with approx 0.2 mL of methylene chloride. Adjust the final volume to 1.0 mL.

10.6 Transfer the concentrated extract to a clean screw-cap vial. Seal the vial with a Teflon-lined lid, and mark the level on the vial. Label with the sample number and fraction, and store in the dark at −20 to −10 °C until ready for analysis.

*11 GC/MS analysis*

11.1 Establish the operating conditions given in tables 3 or 4 for analysis of the base/neutral or acid extracts, respectively. For analysis of combined extracts (section 10.4.2), use the operating conditions in table 3.

11.2 Bring the concentrated extract (section 10.6) or standard (sections 6.13–6.14) to room temperature and verify that any precipitate has redissolved. Verify the level on the extract (sections 6.6 and 10.6) and bring to the mark with solvent if required.

11.3 Add the internal standard solution (section 6.10) to the extract (use 1.0 uL of solution per 0.1 mL of extract) immediately prior to injection to minimize the possibility of loss by evaporation, adsorption, or reaction. Mix thoroughly.

11.4 Inject a volume of the standard solution or extract such that 100 ng of the internal standard will be injected, using on-column or splitless injection. For 1 mL extracts, this volume will be 1.0 uL. Start the GC column initial isothermal hold upon injection. Start MS data collection after the solvent peak elutes. Stop data collection after the benzo (ghi) perylene or pentachlorophenol peak elutes for the base/neutral or acid fraction, respectively. Return

the column to the initial temperature for analysis of the next sample.

*12 System and laboratory performance*

12.1 At the beginning of each 8 hr shift during which analyses are performed, GC/MS system performance and calibration are verified for all pollutants and labeled compounds. For these test, analysis of the 100 μg/mL calibration standard (section 6.13) shall be used to verify all performance criteria. Adjustment and/or recalibration (per section 7) shall be performed until all performance criteria are met. Only after all performance criteria are met may samples, blanks, and precision and recovery standards be analyzed.

12.2 DFTPP spectrum validity—inject 1 μL of the DFTPP solution (section 6.11) either separately or within a few seconds of injection of the standard (section 12.1) analyzed at the beginning of each shift. The criteria in table 5 shall be met.

12.3 Retention times—the absolute retention time of 2,2'-difluorobiphenyl shall be within the range of 1078 to 1248 seconds and the relative retention times of all pollutants and labeled compounds shall fall within the limits given in tables 3 and 4.

12.4 GC resolution—the valley height between anthracene and phenanthrene at m/z 178 (or the analogs at m/z 188) shall not exceed 10 percent of the taller of the two peaks.

12.5 Calibration verification—compute the concentration of each pollutant (tables 1 and 2) by isotope dilution (section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant which has no labeled analog by the internal standard method (section 7.5). Compute the concentration of the labeled compounds by the internal standard method. These concentrations are computed based on the calibration data determined in section 7.

12.5.1 For each pollutant and labeled compound being tested, compare the concentration with the calibration verification limit in table 8. If all compounds meet the acceptance criteria, calibration has been verified and analysis of blanks, samples, and precision and recovery standards may proceed. If, however, any compound fails, the measurement system is not performing properly for that compound. In this event, prepare a fresh calibration standard or correct the problem causing the failure and repeat the test (section 12.1), or recalibrate (section 7).

12.6 Multiple peaks—each compound injected shall give a single, distinct GC peak.

12.7 On-going precision and accuracy.

12.7.1 Analyze the extract of one of the pair of precision and recovery standards (section 10.1.3) prior to analysis of samples from the same lot.

12.7.2 Compute the concentration of each pollutant (tables 1 and 2) by isotope dilution (section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant which has no labeled analog by the internal standard method (section 7.5). Compute the concentration of the labeled compounds by the internal standard method.

12.7.3 For each pollutant and labeled compound, compare the concentration with the limits for on-going accuracy in table 8. If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed. If, however, any individual concentration falls outside of the range given, system performance is unacceptable for that compound. NOTE: The large number of compounds in table 8 present a substantial probability that one or more will fail when all compounds are analyzed. To determine if the extraction/concentration system is out of control or if the failure is caused by probability, proceed as follows:

12.7.3.1 Analyze the second aliquot of the pair of precision and recovery standard (section 10.1.3).

12.7.3.2 Compute the concentration of only those pollutants or labeled compounds that failed the previous test (section 12.7.3). If these compounds now pass, the extraction/concentration processes are in control and analysis of blanks and samples may proceed. If, however, any of the same compounds fail again, the extraction/concentration processes are not being performed properly for these compounds. In this event, correct the problem, re-extract the sample lot (section 10) and repeat the on-going precision and recovery test (section 12.7).

12.7.4 Add results which pass the specifications in section 12.7.2 to initial and previous on-going data. Update QC charts to perform a graphic representation of continued laboratory performance (Figure 5). Develop a statement of laboratory accuracy for each pollutant and labeled compound by calculating the average percent recovery (R) and the standard deviation of percent recovery ($s_r$). Express the accuracy as a recovery interval from $R-2s_r$ to $R+2s_r$. For example, if R=95% and $s_r$=5%, the accuracy is 85–105%.

*13 Qualitative determination*

13.1 Qualititative determination is accomplished by comparison of data from analysis of a sample or blank with data from analysis of the shift standard (section 12.1) and with data stored in the spectral libraries (section 7.2.4). Identification is confirmed when spectra and retention times agree per the criteria below.

13.2 Labeled compounds and pollutants having no labeled analog:

13.2.1 The signals for all characteristic masses stored in the spectral library (section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

13.2.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two (0.5 to 2 times) for all masses stored in the library.

13.2.3 The retention time relative to the nearest eluted internal standard shall be within ±15 scans or ±15 seconds, whichever is greater of this difference in the shift standard (section 12.1).

13.3 Pollutants having a labled analog:

13.3.1 The signals for all characteristic masses stored in the spectral library (section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

13.3.2. Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the spectral library.

13.3.3. The retention time difference between the pollutant and its labeled analog shall agree within ± 6 scans or ± 6 seconds (whichever is greater) of this difference in the shift standard (section 12.1).

13.4 Massesd present in the experimental mass spectrum that are not present in the reference mass spectrum shall be accounted for by contaminant or background ions. If the experimental mass spectrum is contaminated, an experienced spectrometrist (section 1.4) is to determine the presence or absence of the compound.

*14 Quantitative determination*

14.1 Isotope dilution—by adding a known amount of a labeled compound to every sample prior to extraction, correction for recovery of the pollutant can be made because the pollutant and its labeled analog exhibit the same effects upon extraction, concentration, and gas chromatography. Relative response (RR) values for mixtures are used in conjunction with calibration curves described in section 7.4 to determine concentrations directly, so long as labeled compound spiking levels are constant. For the phenol example given in figure 1 (section 7.4.1), RR would be equal to 1.114. For this RR value, the phenol calibration curve given in figure 1 indicates a concentration of 27 μg/mL in the sample extract ($C_{ex}$).

14.2 Internal standard—compute the concentration in the extract using the response factor determined from calibration data (section 7.5) and the following equation: $C_{ex}(\mu g/mL)=(A_s \times C_{is})/(A_{is} \times RF)$ where $C_{ex}$ is the concentration of the compound in the extract, and the other terms are as defined in section 7.5.1.

14.3 The concentration of the pollutant in water is computed using the volumes of the original water sample (section 10.1) and the final extract volume (section 10.5), as follows: Concentration in water $(\mu g/L)=(C_{ex} \times V_{ex})/V_s$ where $V_{ex}$ is the extract volume in mL, and $V_s$ is the sample volume in liters.

14.4 If the EICP area at the quantitiation mass for any compound exceeds the calibration range of the system, the extract of the dilute aliquot (section 10.1) is analyzed by isotope dilution; otherwise, the extract is diluted by a factor of 10, 9 μL of internal standard solution (section 6.10) are added to a 1.0 mL aliquot, and this diluted extract is analyzed by the internal standard method (section 14.2). Quantify each compound at the highest concentration level within the calibration range.

14.5 Report results for all pollutants and labeled compounds (tables 1 and 2) found in all standards, blanks, and samples in μg/L, to three significant figures. Results for samples which have been diluted are reported at the least dilute level at which the area at the quantitation mass is within the calibration range (section 14.4) and the labeled compound recovery is within the normal range for the method (section 15.4).

*15 Analysis of complex samples*

15.1 Untreated effluents and other samples frequently contain high levels (>1000 μg/L) of the compounds of interest, interfering compounds, and/or polymeric materials. Some samples will not concentrate to one mL (section 10.5); others will overload the GC column and/or mass spectrometer.

15.2 Analyze the dilute aliquot (section 10.1) when the sample will not concentrate to 1.0 mL. If a dilute aliquot was not extracted, and the sample holding time (section 9.3) has not been exceeded, dilute an aliquot of the sample with reagent water and re-extract (section 10.1); otherwise, dilute the extract (section 14.4) and analyze by the internal standard method (section 14.2).

15.3 Recovery of internal standard— the EICP area of the internal standard should be within a factor of two of the area in the shift standard (section 12.1). If the absolute areas of the labeled compounds are within a factor of two of the respective areas in the shift standard, and the internal standard area is less than one-half of its respective area, then internal standard loss in the extract has occurred. In this case, use one of the labeled compounds (perferably a polynuclear aromatic hydrocarbon) to compute the concentration of a pollutant with no labeled analog.

15.4 Recovery of labeled compounds— in most samples, labeled compound recoveries will be similar to those from reagent water (section 12.7). If the labeled compound recovery is outside the limits given in table 8, the dilute extract (section 10.1) is analyzed as in section 14.4. If the recoveries of all labeled compounds and the internal standard are low (per the criteria above), then a loss in instrument sensitivity is the most likely cause. In this case, the 100 μg/mL calibration standard (section 12.1) shall be analyzed and calibration verified (section 12.5). If a loss in sensitivity has occurred, the instrument shall be repaired, the performance specifications in section 12 shall be met, and the extract reanalyzed. If a loss in instrument sensitivity has not occurred, the method does not work on the sample being analyzed and the result may not be reported for regulatory compliance purposes.

*16 Method performance*

16.1 Interlaboratory performance for this method is detailed in references 9 and 10.

16.2 A chromatogram of the 100 μg/Ml acid/base/neutral calibration standard (section 6.13) is shown in figure 6.

**References**

1. "Performance Tests for the Evaluation of Computerized Gas Chromatography/Mass Spectrometry Equipment and Laboratories" USEPA, EMSL/Cincinnati, OH 45268, EPA–600/4–80–025 (April 1980).

2. "Working with Carcinogens," DHEW, PHS, CDC, NIOSH, Publication 77–206, (Aug 1977).

3. "OSHA Safety and Health Standards, General Industry" OSHA 2206, 29 CFR 1910 (Jan 1976).

4. "Safety in Academic Chemistry Laboratories, " ACS Committee on Chemical Safety (1979).

5. "Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectrometry Systems," J.W. Eichelberger, L.E. Harris, and W.L. Budde, Anal. Chem., *47*, 955 (1975).

6. "Handbook of Analytical Quality Control in Water and Wastewater Laboratories," USEPA, EMSL/Cincinnati, OH 45268, EPA–600/4–79–019 (March 1979).

7. "Standard Practice for Sampling Water," ASTM Annual Book of Standards, ASTM, Philadelphia, PA, 76 (1980).

8. "Methods 330.4 and 330.5 for Total Residual Chlorine," USEPA, EMSL/Cincinnati, OH 45268, EPA 600/4–70–020 (March 1979).

9. Colby, B.N., Beimer, R.G., Rushneck, D.R., and Telliard, W.A., "Isotope Dilution Gas Chromatography-Mass Spectrometry for the determination of Priority Pollutants in Industrial Effluents." USEPA, Effluent Guidelines Division, Washington, DC 20460 (1980).

10. "Inter-laboratory Validation of US Environmental Protection Agency Method 1625," USEPA, Effluent Guidelines Division, Washington, DC 20460 (June 15, 1984).

TABLE 1.—Base/Neutral Extractable Compounds

| Compound | Storet | CAS registry | EPA-EGD | NPDES |
|---|---|---|---|---|
| Acenaphthene | 34205 | 83-32-9 | 001 B | 001 B |
| Acenaphthylene | 34200 | 208-96-8 | 077 B | 002 B |
| Anthracene | 34220 | 120-12-7 | 078 B | 003 B |
| Benzidine | 39120 | 92-87-5 | 005 B | 004 B |
| Benzo(a)anthracene | 34526 | 56-55-3 | 072 B | 005 B |
| Benzo(b)fluoranthene | 34230 | 205-99-2 | 074 B | 007 B |
| Benzo(k)fluoranthene | 34242 | 207-08-9 | 075 B | 009 B |
| Benzo(a)pyrene | 34247 | 50-32-8 | 073 B | 006 B |
| Benzo(ghi)perylene | 34521 | 191-24-2 | 079 B | 008 B |
| Biphenyl (Appendix C) | 81513 | 92-52-4 | 512 B | |
| Bis(2-chloroethyl) ether | 34273 | 111-44-4 | 018 B | 011 B |
| Bis(2-chloroethyoxy)methane | 34278 | 111-91-1 | 043 B | 010 B |
| Bis(2-chloroisopropyl) ether | 34283 | 108-60-1 | 042 B | 012 B |
| Bis(2-ethylhexyl) phthalate | 39100 | 117-81-7 | 066 B | 013 B |
| 4-bromophenyl phenyl ether | 34636 | 101-55-3 | 041 B | 014 B |
| Butyl benzyl phthalate | 34292 | 85-68-7 | 067 B | 015 B |
| n-C10 (Appendix C) | 77427 | 124-18-5 | 517 B | |
| n-C12 (Appendix C) | 77588 | 112-40-2 | 506 B | |
| n-C14 (Appendix C) | 77691 | 629-59-4 | 518 B | |
| n-C16 (Appendix C) | 77757 | 544-76-3 | 519 B | |
| n-C18 (Appendix C) | 77804 | 593-45-3 | 520 B | |
| n-C20 (Appendix C) | 77830 | 112-95-8 | 521 B | |
| n-C22 (Appendix C) | 77859 | 629-97-0 | 522 B | |
| n-C24 (Appendix C) | 77886 | 646-31-1 | 523 B | |
| n-C26 (Appendix C) | 77901 | 630-01-3 | 524 B | |
| n-C28 (Appendix C) | 78116 | 630-02-4 | 525 B | |
| n-C30 (Appendix C) | 78117 | 638-68-6 | 526 B | |
| Carbazole (4c) | 77571 | 86-74-8 | 528 B | |
| 2-chloronaphthalene | 34581 | 91-58-7 | 020 B | 016 B |
| 4-chlorophenyl phenyl ether | 34641 | 7005-72-3 | 040 B | 017 B |
| Chrysene | 34320 | 218-01-9 | 076 B | 018 B |
| P-cymene (Appendix C) | 77356 | 99-87-6 | 513 B | |
| Dibenzo(a,h)anthracene | 34556 | 53-70-3 | 082 B | 019 B |
| Dibenzofuran (Appendix C and 4c) | 81302 | 132-64-9 | 505 B | |
| Dibenzothiophene (Synfuel) | 77639 | 132-65-0 | 504 B | |
| Di-n-butyl phthalate | 39110 | 84-74-2 | 068 B | 026 B |
| 1,2-dichlorobenzene | 34536 | 95-50-1 | 025 B | 020 B |
| 1,3-dichlorobenzene | 34566 | 541-73-1 | 026 B | 021 B |
| 1,4-dichlorobenzene | 34571 | 106-46-7 | 027 B | 022 B |
| 3,3'-dichlorobenzidine | 34631 | 91-94-1 | 028 B | 023 B |
| Diethyl phthalate | 34336 | 84-66-2 | 070 B | 024 B |
| 2,4-dimethylphenol | 34606 | 105-67-9 | 034 A | 003 A |
| Dimethyl phthalate | 34341 | 131-11-3 | 071 B | 025 B |
| 2,4-dinitrotoluene | 34611 | 121-14-2 | 035 B | 027 B |
| 2,6-dinitrotoluene | 34626 | 606-20-2 | 036 B | 028 B |
| Di-n-octyl phthalate | 34596 | 117-84-0 | 069 B | 029 B |

TABLE 1.—Base/Neutral Extractable Compounds—Continued

| Compound | Storet | CAS registry | EPA-EGD | NPDES |
|---|---|---|---|---|
| Diphenylamine (Appendix C) | 77579 | 122-39-4 | 507 B | |
| Diphenyl ether (Appendix C) | 77587 | 101-84-8 | 508 B | |
| 1,2-diphenylhydrazine | 34346 | 122-66-7 | 037 B | 030 B |
| Fluoranthene | 34376 | 206-44-0 | 039 B | 031 B |
| Fluorene | 34381 | 86-73-7 | 080 B | 032 B |
| Hexachlorobenzene | 39700 | 118-74-1 | 009 B | 033 B |
| Hexachlorobutadiene | 34391 | 87-68-3 | 052 B | 034 B |
| Hexachloroethane | 34396 | 67-72-1 | 012 B | 036 B |
| Hexachlorocyclopentadiene | 34386 | 77-47-4 | 053 B | 035 B |
| Indeno(1,2,3-cd)pyrene | 34403 | 193-39-5 | 083 B | 037 B |
| Isophorone | 34408 | 78-59-1 | 054 B | 038 B |
| Naphthalene | 34696 | 91-20-3 | 055 B | 039 B |
| B-naphthylamine (Appendix C) | 82553 | 91-59-8 | 502 B | |
| Nitrobenzene | 34447 | 98-95-3 | 056 B | 040 B |
| N-nitrosodimethylamine | 34438 | 62-75-9 | 061 B | 041 B |
| N-nitrosodi-n-propylamine | 34428 | 621-64-7 | 063 B | 042 B |
| N-nitrosodiphenylamine | 34433 | 86-30-3 | 062 B | 043 B |
| Phenanthrene | 34461 | 85-01-8 | 081 B | 044 B |
| Phenol | 34694 | 108-95-2 | 065 A | 010 A |
| a-picoline (Synfuel) | 77088 | 109-06-89 | 503 B | |
| Pyrene | 34469 | 129-00-0 | 084 B | 045 B |
| styrene (Appendix C) | 77128 | 100-42-5 | 510 B | |
| a-terpineol (Appendix C) | 77493 | 98-55-5 | 509 B | |
| 1,2,3-trichlorobenzene (4c) | 77613 | 87-61-6 | 529 B | |
| 1,2,4-trichlorobenzene | 34551 | 120-82-1 | 008 B | 046 B |

TABLE 2.—Acid Extractable Compounds

| Compound | Storet | CAS registry | EPA-EGD | NPDES |
|---|---|---|---|---|
| 4-chloro-3-methylphenol | 34452 | 59-50-7 | 022 A | 008 A |
| 2-chlorophenol | 34586 | 95-57-8 | 024 A | 001 A |
| 2,4-dichlorophenol | 34601 | 120-83-2 | 031 A | 002 A |
| 2,4-dinitrophenol | 34616 | 51-28-5 | 059 A | 005 A |
| 2-methyl-4,6-dinitrophenol | 34657 | 534-52-1 | 060 A | 004 A |
| 2-nitrophenol | 34591 | 88-75-5 | 057 A | 006 A |
| 4-nitrophenol | 34646 | 100-02-7 | 058 A | 007 A |
| Pentachlorophenol | 39032 | 87-86-5 | 064 A | 009 A |
| 2,3,6-trichlorophenol (4c) | 77688 | 93-37-55 | 530 A | |
| 2,4,5-trichlorophenol (4c) | | 95-95-4 | 531 A | |
| 2,4,6-trichlorophenol | 34621 | 88-06-2 | 021 A | 011 A |

TABLE 3.—GAS CHROMATOGRAPHY OF BASE/NEUTRAL EXTRACTABLE COMPOUNDS

| EGD No.[1] | Compound | Retention time | | | Detection limit [2] (µg/L) |
|---|---|---|---|---|---|
| | | Mean (sec) | EGD Ref | Relative | |
| 164 | 2,2'-difluorobiphenyl (int std) | 1163 | 164 | 1.000–1.000 | 10 |
| 061 | N-nitrosodimethylamine | 385 | 164 | ns | 50 |
| 603 | alpha picoline-d7 | 417 | 164 | 0.326–0.393 | 50 |
| 703 | alpha picoline | 426 | 603 | 1.006–1.028 | 50 |
| 610 | styrene-d5 | 546 | 164 | 0.450–0.488 | 10 |
| 710 | styrene | 549 | 610 | 1.002–1.009 | 10 |
| 613 | p-cymene-d14 | 742 | 164 | 0.624–0.652 | 10 |
| 713 | p-cymene | 755 | 613 | 1.008–1.023 | 10 |
| 265 | phenol-d5 | 696 | 164 | 0.584–0.613 | 10 |
| 365 | phenol | 700 | 265 | 0.995–1.010 | 10 |
| 218 | bis(2-chloroethyl) ether-d8 | 696 | 164 | 0.584–0.607 | 10 |
| 318 | bis(2-chloroethyl) ether | 704 | 218 | 1.007–1.016 | 10 |
| 617 | n-decane-d22 | 698 | 164 | 0.585–0.615 | 10 |
| 717 | n-decane | 720 | 617 | 1.022–1.038 | 10 |
| 226 | 1,3-dichlorobenzene-d4 | 722 | 164 | 0.605–0.636 | 10 |
| 326 | 1,3-dichlorobenzene | 724 | 226 | 0.998–1.008 | 10 |
| 227 | 1,4-dichlorobenzene-d4 | 737 | 164 | 0.601–0.666 | 10 |
| 327 | 1,4-dichlorobenzene | 740 | 227 | 0.997–1.009 | 10 |
| 225 | 1,2-dichlorobenzene-d4 | 758 | 164 | 0.632–0.667 | 10 |
| 325 | 1,2-dichlorobenzene | 760 | 225 | 0.995–1.008 | 10 |
| 242 | bis(2-chloroisopropyl) ether-d12 | 788 | 164 | 0.664–0.691 | 10 |
| 342 | bis(2-chloroisopropyl) ether | 799 | 242 | 1.010–1.016 | 10 |
| 212 | hexachloroethane-13C | 819 | 164 | 0.690–0.717 | 10 |
| 312 | hexachloroethane | 823 | 212 | 0.999–1.001 | 10 |
| 063 | N-nitrosodi-n-propylamine | 830 | 164 | ns | 20 |
| 256 | nitrobenzene-d5 | 845 | 164 | 0.706–0.727 | 10 |
| 356 | nitrobenzene | 849 | 256 | 1.002–1.007 | 10 |
| 254 | isophorone-d8 | 881 | 164 | 0.747–0.767 | 10 |
| 354 | isophorone | 889 | 254 | 0.999–1.017 | 10 |
| 234 | 2,4-dimethylphenol | 921 | 164 | 0.781–0.803 | 10 |
| 334 | 2,4-dimethylphenol | 924 | 234 | 0.999–1.003 | 10 |
| 043 | bis(2-chloroethoxy) methane | 939 | 164 | ns | 10 |
| 208 | 1,2,4-trichlorobenzene-d3 | 955 | 164 | 0.813–0.830 | 10 |
| 308 | 1,2,4-trichlorobenzene | 958 | 208 | 1.000–1.005 | 10 |
| 255 | naphthalene-d8 | 963 | 164 | 0.819–0.836 | 10 |
| 355 | naphthalene | 967 | 255 | 1.001–1.006 | 10 |
| 609 | alpha-terpineol-d3 | 973 | 164 | 0.829–0.844 | 10 |
| 709 | alpha-terpineol | 975 | 609 | 0.998–1.008 | 10 |
| 606 | n-dodecane-d26 | 953 | 164 | 0.730–0.908 | 10 |
| 706 | n-dodecane | 981 | 606 | 0.986–1.051 | 10 |

TABLE 3.—GAS CHROMATOGRAPHY OF BASE/NEUTRAL EXTRACTABLE COMPOUNDS—Continued

| EGD No.[1] | Compound | Retention time | | | Detection limit [2] (μg/L) |
|---|---|---|---|---|---|
| | | Mean (sec) | EGD Ref | Relative | |
| 529 | 1,2,3-trichlorobenzene | 1003 | 164 | ns | 10 |
| 252 | hexachlorobutadiene-13C4 | 1005 | 164 | 0.856–0.871 | 10 |
| 352 | hexachlorobutadiene | 1006 | 252 | 0.999–1.002 | 10 |
| 253 | hexachlorocyclopentadiene-13C4 | 1147 | 164 | 0.976–0.986 | 10 |
| 353 | hexachlorocyclopentadiene | 1142 | 253 | 0.999–1.001 | 10 |
| 220 | 2-chloronaphthalene-d7 | 1185 | 164 | 1.014–1.024 | 10 |
| 320 | 2-chloronaphthalene | 1200 | 220 | 0.997–1.007 | 10 |
| 518 | n-tetradecane | 1203 | 164 | ns | 10 |
| 612 | Biphenyl-d10 | 1205 | 164 | 1.016–1.027 | 10 |
| 712 | Biphenyl | 1195 | 612 | 1.001–1.006 | 10 |
| 606 | Diphenyl ether-d10 | 1211 | 164 | 1.036–1.047 | 10 |
| 708 | Diphenyl ether | 1216 | 608 | 0.997–1.009 | 10 |
| 277 | Acenaphthylene-d8 | 1265 | 164 | 1.080–1.095 | 10 |
| 377 | Acenaphthylene | 1247 | 277 | 1.000–1.004 | 10 |
| 271 | Dimethyl phthalate-d4 | 1269 | 164 | 1.083–1.102 | 10 |
| 371 | Dimethyl phthalate | 1273 | 271 | 0.998–1.005 | 10 |
| 236 | 2,6-dinitrotoluene-d3 | 1283 | 164 | 1.090–1.112 | 10 |
| 336 | 2,6-dinitrotoluene | 1300 | 236 | 1.001–1.005 | 10 |
| 201 | Acenaphthene-d10 | 1298 | 164 | 1.107–1.125 | 10 |
| 301 | Acenaphthene | 1304 | 201 | 0.999–1.009 | 10 |
| 605 | Dibenzofuran-d8 | 1331 | 164 | 1.134–1.155 | 10 |
| 705 | Dibenzofuran | 1335 | 605 | 0.995–1.007 | 10 |
| 602 | Beta-naphthylamine-d7 | 1368 | 164 | 1.163–1.189 | 50 |
| 702 | Beta-naphthylamine | 1371 | 602 | 0.996–1.007 | 50 |
| 280 | Fluorene-d10 | 1395 | 164 | 1.185–1.214 | 10 |
| 380 | Fluorene | 1401 | 281 | 0.999–1.008 | 10 |
| 240 | 4-chlorophenyl phenyl ether-d5 | 1406 | 164 | 1.194–1.223 | 10 |
| 340 | 4-chlorophenyl phenyl ether | 1409 | 240 | 0.990–1.015 | 10 |
| 270 | Diethyl phthalate-d4 | 1409 | 164 | 1.197–1.229 | 10 |
| 370 | Diethyl phthalate | 1414 | 270 | 0.996–1.006 | 10 |
| 619 | n-hexadecane-d34 | 1447 | 164 | 1.010–1.478 | 10 |
| 719 | n-hexadecane | 1469 | 619 | 1.013–1.020 | 10 |
| 235 | 2,4-dinitrotoluene-d3 | 1359 | 164 | 1.152–1.181 | 10 |
| 335 | 2,4-dinitrotoluene | 1344 | 235 | 1.000–1.002 | 10 |
| 237 | 1,2-diphenylhydrazine-d8 | 1433 | 164 | 1.216–1.248 | 20 |
| 337 | 1,2-diphenylhydrazine [3] | 1439 | 237 | 0.999–1.009 | 20 |
| 607 | Diphenylamine-d10 | 1437 | 164 | 1.213–1.249 | 20 |
| 707 | Diphenylamine | 1439 | 607 | 1.000–1.007 | 20 |
| 262 | N-nitrosodiphenylamine-d6 | 1447 | 164 | 1.225–1.252 | 20 |
| 362 | N-nitrosodiphenylamine [4] | 1464 | 262 | 1.000–1.002 | 20 |
| 041 | 4-bromophenyl phenyl ether | 1498 | 164 | 1.271–1.307 | 10 |
| 209 | Hexachlorobenzene-13C6 | 1521 | 164 | 1.288–1.327 | 10 |
| 309 | Hexachlorobenzene | 1522 | 209 | 0.999–1.001 | 10 |
| 281 | Phenanthrene-d10 | 1578 | 164 | 1.334–1.380 | 10 |
| 520 | n-octadecane | 1580 | 164 | ns | 10 |
| 381 | Phenanthrene | 1583 | 281 | 1.000–1.005 | 10 |
| 278 | Anthracene-d10 | 1588 | 164 | 1.342–1.388 | 10 |
| 378 | Anthracene | 1592 | 278 | 0.998–1.006 | 10 |
| 604 | Dibenzothiophene-d8 | 1559 | 164 | 1.314–1.361 | 10 |
| 704 | Dibenzothiophene | 1564 | 604 | 1.000–1.006 | 10 |
| 528 | Carbazole | 1650 | 164 | ns | 20 |
| 621 | n-eicosane-d42 | 1655 | 164 | 1.184–1.662 | 10 |
| 721 | n-eicosane | 1677 | 621 | 1.010–1.021 | 10 |
| 268 | Di-n-butyl phthalate-d4 | 1719 | 164 | 1.446–1.510 | 10 |
| 368 | Di-n-butyl phthalate | 1723 | 268 | 1.000–1.003 | 10 |
| 239 | Fluoranthene-d10 | 1813 | 164 | 1.522–1.596 | 10 |
| 339 | Fluoranthene | 1817 | 239 | 1.000–1.004 | 10 |
| 284 | Pyrene-d10 | 1844 | 164 | 1.523–1.644 | 10 |
| 384 | Pyrene | 1852 | 284 | 1.001–1.003 | 10 |
| 205 | Benzidine-d8 | 1854 | 164 | 1.549–1.632 | 50 |
| 305 | Benzidine | 1853 | 205 | 1.000–1.002 | 50 |
| 522 | n-docosane | 1889 | 164 | ns | 10 |
| 623 | n-tetracosane-d50 | 1997 | 164 | 1.671–1.764 | 10 |
| 723 | n-tetracosane | 2025 | 612 | 1.012–1.015 | 10 |
| 067 | Butylbenzyl phthalate | 2060 | 164 | ns | 10 |
| 276 | Chrysene-d12 | 2081 | 164 | 1.743–1.837 | 10 |
| 376 | Chrysene | 2083 | 276 | 1.000–1.004 | 10 |
| 272 | Benzo(a)anthracene-d12 | 2082 | 164 | 1.735–1.846 | 10 |
| 372 | Benzo(a)anthracene | 2090 | 272 | 0.999–1.007 | 50 |
| 228 | 3,3'-dichlorobenzidine-d6 | 2088 | 164 | 1.744–1.848 | 50 |
| 328 | 3,3'-dichlorobenzidine | 2086 | 228 | 1.000–1.001 | 50 |
| 266 | Bis(2-ethylhexyl) phthalate-d4 | 2123 | 164 | 1.771–1.880 | 10 |
| 366 | Bis(2-ethylhexyl) phthalate | 2124 | 266 | 1.000–1.002 | 10 |
| 524 | n-hexacosane | 2147 | 164 | ns | 10 |
| 269 | di-n-octyl phthalate-d4 | 2239 | 164 | 1.867–1.982 | 10 |
| 369 | di-n-octyl phthalate | 2240 | 269 | 1.000–1.002 | 10 |
| 525 | n-octacosane | 2272 | 164 | ns | 10 |
| 274 | Benzo(b)fluoranthene-d12 | 2281 | 164 | 1.902–2.025 | 10 |
| 354 | Benzo(b)fluoranthene | 2293 | 274 | 1.000–1.005 | 10 |
| 275 | Benzo(k)fluoranthene-d12 | 2287 | 164 | 1.906–2.033 | 10 |
| 375 | Benzo(k)fluoranthene | 2293 | 275 | 1.000–1.005 | 10 |
| 273 | Benzo(a)pyrene-d12 | 2351 | 164 | 1.954–2.088 | 10 |
| 373 | Benzo(a)pyrene | 2350 | 273 | 1.000–1.004 | 10 |
| 626 | N-triacontane-d62 | 2384 | 164 | 1.972–2.127 | 10 |
| 726 | N-triacontane | 2429 | 626 | 1.011–1.028 | 10 |
| 083 | Indeno(1,2,3-cd)pyrene | 2650 | 164 | ns | 20 |
| 082 | Dibenzo(a,h)anthracene | 2660 | 164 | ns | 20 |
| 279 | Benzo(ghi)perylene-d12 | 2741 | 164 | 2.187–2.524 | 20 |
| 379 | Benzo(ghi)perylene | 2750 | 279 | 1.001–1.006 | 20 |

[1] Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.
[2] This is a minimum level at which the entire GC/MS system must give recognizable mass spectra (background corrected) and accepted calibration points.
[3] Detected as azobenzene.
[4] Detected as diphenylamine.

ns = specification not available at time of release of method.
Column: 30 ±2 m × 0.25 ±0.02 mm i.d. 94% methyl, 4% phenyl, 1% vinyl bonded phase fused silica capillary.
Temperature program: 5 min at 30 °C; 30 - 280 °C at 8 °C per min; isothermal at 280 °C until benzo(ghi)perylene elutes.
Gas velocity: 30 ±5 cm/sec.

### Table 4.—Gas Chromatography of Acid Extractable Compounds

| EGD No. [1] | Compound | Retention time | | | Detection limit [2] (µg/L) |
|---|---|---|---|---|---|
| | | Mean (sec) | EGD Ref | Relative | |
| 164 | 2,2'-difluorobiphenyl (ins std) | 1163 | 164 | 1.000–1.000 | 10 |
| 224 | 2-chlorophenol-d4 | 701 | 164 | 0.587–0.618 | 10 |
| 324 | 2-chlorophenol | 705 | 224 | 0.997–1.010 | 10 |
| 257 | 2-nitrophenol-d4 | 898 | 164 | 0.761–0.783 | 20 |
| 357 | 2-nitrophenol | 900 | 257 | 0.994–1.009 | 20 |
| 231 | 2,4-dichlorophenol-d3 | 944 | 164 | 0.802–0.822 | 10 |
| 331 | 2,4-dichlorophenol | 947 | 231 | 0.997–1.006 | 10 |
| 222 | 4-chloro-3-methylphenol-d2 | 1086 | 164 | 0.930–0.943 | 10 |
| 322 | 4-chloro-3-methylphenol | 1091 | 222 | 0.998–1.003 | 10 |
| 221 | 2,4,6-trichlorophenol-d2 | 1162 | 164 | 0.994–1.005 | 10 |
| 321 | 2,4,6-trichlorophenol | 1165 | 221 | 0.998–1.004 | 10 |
| 531 | 2,4,5-trichlorophenol | 1170 | 164 | ns | 10 |
| 530 | 2,3,6-trichlorophenol | 1195 | 184 | ns | 10 |
| 259 | 2,4-dinitrophenol-d3 | 1323 | 164 | 1.127–1.149 | 50 |
| 359 | 2,4-dinitrophenol | 1325 | 259 | 1.000–1.005 | 50 |
| 258 | 4-nitrophenol-d4 | 1349 | 164 | 1.147–1.175 | 50 |
| 358 | 4-nitrophenol | 1354 | 258 | 0.997–1.006 | 50 |
| 260 | 2-methyl-4,6-dinitrophenol-d2 | 1433 | 164 | 1.216–1.249 | 20 |
| 360 | 2-methyl-4,6-dinitrophenol | 1435 | 260 | 1.000–1.002 | 20 |
| 264 | Pentachlorophenol-13C6 | 1559 | 164 | 1.320–1.363 | 50 |
| 364 | Pentachlorophenol | 1561 | 264 | 0.998–1.002 | 50 |

[1] Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.
[2] This is a minimum level at which the entire GC/MS system must give recognizable mass spectra (background corrected) and acceptable calibration points.

ns = specification not available at time of release of method.
Column: 30±2m×0.25±0.02mm id.d. 94% methyl, 4% phenyl, 1% vinyl bonded phase fused silica capillary.
Temperature program: 5 min at 30 °C; 30—250 °C or until pentachlorophenol elutes.
Gas velocity: 30±5 cm/sec.

### Table 5.—DFTPP Mass Intensity Specifications

| Mass | Intensity required |
|---|---|
| 51 | 30–80 percent of mass 198. |
| 68 | Less than 2 percent of mass 69. |
| 70 | Less than 2 percent of mass 69. |
| 127 | 30–60 percent of mass 198. |
| 197 | Less than 1 percent of mass 198. |
| 199 | 5–9 percent of mass 198. |
| 275 | 10–30 percent of mass 198. |
| 441 | Less than mass 443. |
| 442 | 40–100 percent of mass 198. |
| 443 | 17–23 percent of mass 442. |

### Table 6.—Base/Neutral Extractable Compound Characteristic Masses

| Compound | Labeled analog | Primary m/z |
|---|---|---|
| Acenaphthene | d10 | 154/164 |
| Acenaphthylene | d8 | 152/160 |
| Anthracene | d10 | 178/188 |
| Benzidine | d8 | 184/192 |
| Benzo(a)anthracene | d12 | 228/240 |
| Benzo(b)fluoranthene | d12 | 252/264 |
| Benzo(k)fluoranthene | d12 | 252/264 |
| Benzo(a)pyrene | d12 | 252/264 |
| Benzo(ghi)perylene | d12 | 276/288 |
| Biphenyl | d10 | 154/164 |
| Bis(2-chloroethyl) ether | d8 | 93/101 |
| Bis(2-chloroethoxy)methane | | 93 |
| Bis(2-chloroisopropyl) ether | d12 | 121/131 |
| Bis(2-ethylhexyl) phthalate | d4 | 149/153 |
| 4-bromophenyl phenyl ether | | 248 |
| Butyl benzyl phthalate | | 149 |
| n-C10 | d22 | 55/66 |
| n-C12 | d26 | 55/66 |
| n-C14 | | 55 |
| n-C16 | d34 | 55/66 |

### Table 6.—Base/Neutral Extractable Compound Characteristic Masses—Continued

| Compound | Labeled analog | Primary m/z |
|---|---|---|
| n-C18 | | 55 |
| n-C20 | d42 | 55/66 |
| n-C22 | | 55 |
| n-C24 | d50 | 55/66 |
| n-C26 | | 55 |
| n-C28 | | 55 |
| n-C30 | d62 | 55/66 |
| Carbazole | d8 | 167/175 |
| 2-chloronaphthalene | d7 | 162/169 |
| 4-chlorophenyl phenyl ether | d5 | 204/209 |
| Chrysene | d12 | 228/240 |
| p-cymene | d14 | 114/130 |
| Dibenzo(a,h)anthracene | | 278 |
| Dibenzofuran | d8 | 168/176 |
| Dibenzothiophene | d8 | 184/192 |
| Di-n-butyl phthalate | d4 | 149/153 |
| 1,2-dichlorobenzene | d4 | 146/152 |
| 1,3-dichlorobenzene | d4 | 146/152 |
| 1,4-dichlorobenzene | d4 | 146/152 |
| 3,3'-dichlorobenzidine | d6 | 252/258 |
| Diethyl phthalate | d4 | 149/153 |
| 2,4-dimethylphenol | d3 | 122/125 |
| Dimethyl phthalate | d4 | 163/167 |
| 2,4-dinitrotoluene | d3 | 164/168 |
| 2,6-dinitrotoluene | d3 | 165/167 |
| Di-n-octyl phthalate | d4 | 149/153 |
| Diphenylamine | d10 | 169/179 |
| Diphenyl ether | d10 | 170/180 |
| 1,2-diphenylhydrazine* | d10 | 77/82 |
| Fluoranthene | d10 | 202/212 |
| Fluorene | d10 | 166/176 |
| Hexachlorobenzene | 13C6 | 284/292 |
| Hexachlorobutadiene | 13C4 | 225/231 |
| Hexachloroethane | 13C | 201/204 |
| Hexachlorocyclopentadiene | 13C4 | 237/241 |
| Ideno(1,2,3-cd)pyrene | | 276 |
| Isophorone | d8 | 82/88 |

### Table 6.—Base/Neutral Extractable Compound Characteristic Masses—Continued

| Compound | Labeled analog | Primary m/z |
|---|---|---|
| Naphthalene | d8 | 128/136 |
| B-naphthylamine | d7 | 143/150 |
| Nitrobenzene | d5 | 123/128 |
| N-nitrosodimethylamine | | 74 |
| N-nitrosodi-n-propylamine | | 70 |
| N-nitrosodiphenylamine** | d6 | 169/175 |
| Phenanthrene | d10 | 178/188 |
| Phenol | d5 | 94/71 |
| a-picoline | d7 | 93/100 |
| Pyrene | d10 | 202/212 |
| Styrene | d5 | 104/109 |
| a-terpineol | d3 | 59/62 |
| 1,2,3-trichlorobenzene | d3 | 180/183 |
| 1,2,4-trichlorobenzene | d3 | 180/183 |

*Detected as azobenzene.
**Detected as diphenylamine.

### Table 7.—Acid Extractable Compound Characteristic Masses

| Compound | Labeled analog | Primary m/z |
|---|---|---|
| 4-chloro-3-methylphenol | d2 | 107/109 |
| 2-chlorophenol | d4 | 128/132 |
| 2,4-dichlorophenol | d3 | 162/167 |
| 2,4-dinitrophenol | d3 | 184/187 |
| 2-methyl-4,6-dinitrophenol | d2 | 198/200 |
| 2-nitrophenol | d4 | 139/143 |
| 4-nitrophenol | d4 | 139/143 |
| Pentachlorophenol | 13C6 | 266/272 |
| 2,3,6-trichlorophenol | d2 | 196/200 |
| 2,4,5-trichlorophenol | d2 | 196/200 |
| 2,4,6-trichlorophenol | d2 | 196/200 |

TABLE 8.—ACCEPTANCE CRITERIA FOR PERFORMANCE TESTS

| EGD No.[1] | Compound | Acceptance criteria | | | | |
|---|---|---|---|---|---|---|
| | | Initial precision and accuracy section 8.2.3 (μg/L) | | Labeled compound recovery sec. 8.3 and 14.2 P (percent) | Calibration verification sec. 12.5 (μg/mL) | On-going accuracy sec. 11.6 R (μg/L) |
| | | s | X | | | |
| 301 | Acenaphthene | 21 | 79–134 | | 60–125 | 72–144 |
| 201 | Acenaphthene-d10 | 38 | 38–147 | 20–270 | 71–141 | 30–180 |
| 377 | Acenaphtylene | 38 | 69–186 | | 60–166 | 61–207 |
| 277 | Acenaphthylene-d8 | 31 | 38–146 | 23–239 | 66–152 | 33–168 |
| 378 | Anthracene | 41 | 58–174 | | 60–168 | 50–199 |
| 278 | Anthracene-d10 | 49 | 31–194 | 14–419 | 58–171 | 23–242 |
| 305 | Benzidine | 119 | 16–518 | | 34–296 | 11–672 |
| 205 | Benzidine-d8 | 269 | ns–ns | ns–ns | ns–ns | ns–ns |
| 372 | Benzo(a)anthracene | 20 | 65–168 | | 70–142 | 62–176 |
| 272 | Benzo(a)anthracene-d12 | 41 | 25–298 | 12–605 | 28–357 | 22–329 |
| 374 | Benzo(b)fluoranthene | 183 | 32–545 | | 61–164 | 20–ns |
| 274 | Benzo(b)fluoranthene-d12 | 168 | 11–577 | ns–ns | 14–ns | ns–ns |
| 375 | Benzo(k)fluoranthene | 26 | 59–143 | | 13–ns | 53–155 |
| 275 | Benzo(k)fluoranthene-d12 | 114 | 15–514 | ns–ns | 13–ns | ns–685 |
| 373 | Benzo(a)pyrene | 26 | 62–195 | | 78–129 | 59–206 |
| 273 | Benzo(a)pyrene-d12 | 24 | 35–181 | 21–290 | 12–ns | 32–194 |
| 379 | Benzo(ghi)perylene | 21 | 72–160 | | 69–145 | 58–168 |
| 279 | Benzo(ghi)perylene-d12 | 45 | 29–268 | 14–529 | 13–ns | 25–303 |
| 712 | Biphenyl (Appendix C) | 41 | 75–148 | | 58–171 | 62–176 |
| 612 | Biphenyl-d12 | 43 | 28–165 | ns–ns | 52–192 | 17–267 |
| 318 | Bis(2-chloroethyl) ether | 34 | 55–196 | | 61–164 | 50–213 |
| 218 | Bis(2-chloroethyl) ether-d8 | 33 | 29–196 | 15–372 | 52–194 | 25–222 |
| 043 | Bis(2-chloroethoxy)methane* | 27 | 43–153 | | 44–228 | 39–166 |
| 342 | Bis(2-chloroisopropyl) ether | 17 | 81–138 | | 67–148 | 77–145 |
| 242 | Bis(2-chloroisopropyl)ether-d12 | 27 | 35–149 | 20–260 | 44–229 | 30–169 |
| 366 | Bis(2-ethylhexyl) phthalate | 31 | 69–220 | | 76–131 | 64–232 |
| 266 | Bis(2-ethylhexyl) phthalate-d4 | 29 | 32–205 | 18–364 | 43–232 | 28–224 |
| 041 | 4-bromophenyl phenyl ether* | 44 | 44–140 | | 52–193 | 35–172 |
| 067 | Butyl benzyl phthalate* | 31 | 19–233 | | 22–450 | 35–170 |
| 717 | n-C10 (Appendix C) | 51 | 24–195 | | 42–235 | 19–237 |
| 617 | n-C10-d22 | 70 | ns–298 | ns–ns | 44–227 | ns–504 |
| 706 | n-C12 (Appendix C) | 74 | 35–369 | | 60–166 | 29–424 |
| 606 | n-C12-d26 | 53 | ns–331 | ns–ns | 41–242 | ns–408 |
| 518 | n-C14 (Appendix C)* | 109 | ns–985 | | 37–268 | ns–ns |
| 719 | n-C16 (Appendix C) | 33 | 80–162 | | 72–138 | 71–181 |
| 619 | n-C16-d34 | 46 | 37–162 | 18–308 | 54–186 | 28–202 |
| 520 | n-C18 (Appendix C)* | 39 | 42–131 | | 40–249 | 35–167 |
| 721 | n-C20 (Appendix C) | 59 | 53–263 | | 54–184 | 46–301 |
| 621 | n-C20-d42 | 34 | 34–172 | 19–306 | 62–162 | 29–198 |
| 522 | n-C22 (Appendix C)* | 31 | 45–152 | | 40–249 | 39–195 |
| 723 | n-C24 (Appendix C) | 11 | 80–139 | | 65–154 | 78–142 |
| 623 | n-C24-d50 | 28 | 27–211 | 15–376 | 50–199 | 25–229 |
| 524 | n-C26 (Appendix C)* | 35 | 35–193 | | 26–392 | 31–212 |
| 525 | n-C28 (Appendix C)* | 35 | 35–193 | | 26–392 | 31–212 |
| 726 | n-C30 (Appendix C) | 32 | 61–200 | | 66–152 | 56–215 |
| 626 | n-C30-d62 | 41 | 27–242 | 13–479 | 24–423 | 23–274 |
| 528 | Carbazole (4c)* | 38 | 36–165 | | 44–227 | 31–188 |
| 320 | 2-chloronaphthalene | 100 | 46–357 | | 58–171 | 35–442 |
| 220 | 2-chloronaphthalene-d7 | 41 | 30–168 | 15–324 | 72–139 | 24–204 |
| 322 | 4-chloro-3-methylphenol | 37 | 76–131 | | 65–115 | 62–159 |
| 222 | 4-chloro-3-methylphenol-d2 | 111 | 30–174 | ns–613 | 68–147 | 14–314 |
| 324 | 2-chlorophenol | 13 | 79–135 | | 78–129 | 76–138 |
| 224 | 2-chlorophenol-d4 | 24 | 36–162 | 23–255 | 55–180 | 33–176 |
| 340 | 4-chlorophenyl phenyl ether | 42 | 75–166 | | 71–142 | 63–194 |
| 240 | 4-chlorophenyl phenyl ether-d5 | 52 | 40–161 | 19–325 | 57–175 | 29–212 |
| 376 | Chrysene | 51 | 59–186 | | 70–142 | 48–221 |
| 276 | Chrysene-d12 | 69 | 33–219 | 13–512 | 24–411 | 23–290 |
| 713 | p-cymene (Appendix C) | 18 | 76–140 | | 79–127 | 72–147 |
| 613 | p-cymene-d14 | 67 | ns–359 | ns–ns | 66–152 | ns–468 |
| 082 | Dibenzo(a,h)anthracene* | 55 | 23–299 | | 13–761 | 19–340 |
| 705 | Dibenzofuran (Appendix C) | 20 | 85–136 | | 73–136 | 79–148 |
| 605 | Dibenzofuran-d8 | 31 | 47–136 | 28–220 | 66–150 | 39–160 |
| 704 | Diberizothiophene (Synfuel) | 31 | 79–150 | | 72–140 | 70–168 |
| 604 | Dibenzothiophene-d8 | 31 | 48–130 | 29–215 | 69–145 | 40–156 |
| 368 | Di-n-butyl phthalate | 15 | 76–165 | | 71–142 | 74–169 |
| 268 | Di-n-butyl phthalate-d4 | 23 | 23–195 | 13–346 | 52–192 | 22–209 |
| 325 | 1,2-dichlorobenzene | 17 | 73–146 | | 74–135 | 70–152 |
| 225 | 1,2-dichlorobenzene-d4 | 35 | 14–212 | ns–494 | 61–164 | 11–247 |
| 326 | 1,3-dichlorobenzene | 43 | 63–201 | | 65–154 | 55–225 |
| 226 | 1,3-dichlorobenzene-d4 | 48 | 13–203 | ns–550 | 52–192 | ns–260 |
| 327 | 1,4-dichlorobenzene | 42 | 61–194 | | 62–161 | 53–219 |
| 227 | 1,4-dichlorobenzene-d4 | 48 | 15–193 | ns–474 | 65–153 | 11–245 |
| 328 | 3,3'-dichlorobenzidine | 26 | 68–174 | | 77–130 | 64–185 |
| 228 | 3,3'-dichlorobenzidine-d6 | 80 | ns–562 | ns–ns | 18–558 | ns–ns |
| 331 | 2,4-dichlorophenol | 12 | 85–131 | | 67–149 | 83–135 |
| 231 | 2,4-dichlorophenol-d3 | 28 | 38–164 | 24–260 | 64–157 | 34–182 |
| 370 | Diethyl phthalate | 44 | 75–196 | | 74–135 | 65–222 |
| 270 | Djethyl phthalate-d4 | 78 | ns–260 | ns–ns | 47–211 | ns–ns |
| 334 | 2,4-dimethylphenol | 13 | 62–153 | | 67–150 | 60–156 |
| 234 | 2,4-dimethylphenol-d3 | 22 | 15–228 | ns–449 | 58–172 | 14–242 |
| 371 | Dimethyl phthalate | 36 | 74–188 | | 73–137 | 67–207 |
| 271 | Dimethyl phthalate-d4 | 108 | ns–640 | ns–ns | 50–201 | ns–ns |
| 359 | 2,4-dinitrophenol | 18 | 72–134 | | 75–133 | 68–141 |
| 259 | 2,4-dinitrophenol-d3 | 66 | 22–308 | ns–ns | 39–256 | 17–378 |
| 335 | 2,4-dinitrotoluene | 18 | 75–158 | | 79–127 | 72–164 |
| 235 | 2,4-dinitrotoluene-d3 | 37 | 22–245 | 10–514 | 53–187 | 19–275 |
| 336 | 2,6-dinitrotoluene | 30 | 80–141 | | 55–183 | 70–159 |
| 236 | 2,6-dinitrotoluene-d3 | 59 | 44–184 | 17–442 | 36–278 | 31–250 |

TABLE 8.—ACCEPTANCE CRITERIA FOR PERFORMANCE TESTS—Continued

| EGD No.[1] | Compound | Acceptance criteria | | | | |
|---|---|---|---|---|---|---|
| | | Initial precision and accuracy section 8.2.3 (μg/L) | | Labeled compound recovery sec. 8.3 and 14.2 P (percent) | Calibration verification sec. 12.5 (μg/mL) | On-going accuracy sec. 11.6 R (μg/L) |
| | | s | X | | | |
| 369 | Di-n-octyl phthalate | 16 | 77–161 | | 71–140 | 74–166 |
| 269 | Di-n-octyl phthalate-d4 | 46 | 12–383 | ns–ns | 21–467 | 10–433 |
| 707 | Diphenylamine (Appendix C) | 45 | 58–205 | | 57–176 | 51–231 |
| 607 | Diphenylamine-d10 | 42 | 27–206 | 11–488 | 59–169 | 21–249 |
| 708 | Diphenyl ether (Appendix C) | 19 | 82–136 | | 83–120 | 77–144 |
| 608 | Diphenyl ether-d10 | 37 | 36–155 | 19–281 | 77–129 | 29–186 |
| 337 | 1,2-diphenylhydrazine | 73 | 49–308 | | 75–134 | 40–360 |
| 237 | 1,2-diphenylhydrazine-d10 | 35 | 31–173 | 17–316 | 58–174 | 26–200 |
| 339 | Fluoranthene | 33 | 71–177 | | 67–149 | 64–194 |
| 239 | Fluoranthene-d10 | 35 | 36–161 | 20–278 | 47–215 | 30–187 |
| 380 | Fluorene | 29 | 81–132 | | 74–135 | 70–151 |
| 280 | Fluorene-d10 | 43 | 51–131 | 27–238 | 61–164 | 38–172 |
| 309 | Hexachlorobenzene | 16 | 90–124 | | 78–128 | 85–132 |
| 209 | Hexachlorobenzene-13C6 | 81 | 36–228 | 13–595 | 38–265 | 23–321 |
| 352 | hexachlorobutadiene | 56 | 51–251 | | 74–135 | 43–287 |
| 252 | hexachlorobutadiene-13C4 | 63 | ns–316 | ns–ns | 68–148 | ns–413 |
| 312 | hexachloroethane | 227 | 21–ns | | 71–141 | 13–ns |
| 212 | hexachloroethane-13C1 | 77 | ns–400 | ns–ns | 47–212 | ns–563 |
| 353 | hexachlorocyclopentadiene | 15 | 69–144 | | 77–129 | 67–148 |
| 253 | hexachlorocyclopentadiene-13C4 | 60 | ns–ns | ns–ns | 47–211 | ns–ns |
| 083 | ideno(1,2,3-cd)pyrene* | 55 | 23–299 | | 13–761 | 19–340 |
| 354 | isophorone | 25 | 76–156 | | 70–142 | 70–168 |
| 254 | isophorone-d8 | 23 | 49–133 | 33–193 | 52–194 | 44–147 |
| 360 | 2-methyl-4,6-dinitrophenol | 19 | 77–133 | | 69–145 | 72–142 |
| 260 | 2-methyl-4,6-dinitrophenol-d2 | 64 | 36–247 | 16–527 | 56–177 | 28–307 |
| 355 | naphthalene | 20 | 80–139 | | 73–137 | 75–149 |
| 255 | naphthalene-d8 | 39 | 28–157 | 14–305 | 71–141 | 22–192 |
| 702 | B-naphthylamine (Appendix C) | 49 | 10–ns | | 39–256 | ns–ns |
| 602 | B-naphthylamine-d7 | 33 | ns–ns | ns–ns | 44–230 | ns–ns |
| 356 | nitrobenzene | 25 | 69–161 | | 85–115 | 65–169 |
| 256 | nitrobenzene-d5 | 28 | 18–265 | ns–ns | 46–219 | 15–314 |
| 357 | 2-nitrophenol | 15 | 78–140 | | 77–129 | 75–145 |
| 257 | 2-nitrophenol-d4 | 23 | 41–145 | 27–217 | 61–163 | 37–158 |
| 358 | 4-nitrophenol | 42 | 62–146 | | 55–183 | 51–175 |
| 258 | 4-nitrophenol-d4 | 188 | 14–398 | ns–ns | 35–287 | ns–ns |
| 061 | N-nitrosodimethylamine* | 198 | 21–472 | | 40–249 | 12–807 |
| 063 | N-nitrosodi-n-proplyamine* | 198 | 21–472 | | 40–249 | 12–807 |
| 362 | N-nitrosodiphenylamine | 45 | 65–142 | | 68–148 | 53–173 |
| 262 | N-nitrosodiphenylamine-d6 | 37 | 54–126 | 26–256 | 59–170 | 40–166 |
| 364 | pentachlorophenol | 21 | 76–140 | | 77–130 | 71–150 |
| 264 | pentachlorophenol-13C6 | 49 | 37–212 | 18–412 | 42–237 | 29–254 |
| 381 | phenanthrene | 13 | 93–119 | | 75–133 | 87–126 |
| 281 | phenanthrene-d10 | 40 | 45–130 | 24–241 | 67–149 | 34–168 |
| 365 | phenol | 36 | 77–127 | | 65–155 | 62–154 |
| 265 | phenol-d5 | 161 | 21–210 | ns–ns | 48–208 | ns–ns |
| 703 | a-picoline (Synfuel) | 38 | 59–149 | | 60–165 | 50–174 |
| 603 | a-picoline-d7 | 138 | 11–380 | ns–ns | 31–324 | ns–608 |
| 384 | pyrene | 19 | 76–152 | | 76–132 | 72–159 |
| 284 | pyrene-d10 | 29 | 32–176 | 18–303 | 48–210 | 28–196 |
| 710 | styrene (Appendix C) | 42 | 53–221 | | 65–153 | 48–244 |
| 610 | styrene-d5 | 49 | ns–281 | ns–ns | 44–228 | ns–348 |
| 709 | a-terpineol (Appendix C) | 44 | 42–234 | | 54–186 | 38–258 |
| 609 | a-terpineol-d3 | 48 | 22–292 | ns–672 | 20–502 | 18–339 |
| 529 | 1,2,3-trichlorobenzene (4c)* | 69 | 15–229 | | 60–167 | 11–297 |
| 308 | 1,2,4-trichlorobenzene | 19 | 82–136 | | 78–128 | 77–144 |
| 208 | 1,2,4-trichlorobenzene-d3 | 57 | 15–212 | ns–592 | 61–163 | 10–282 |
| 530 | 2,3,6-trichlorophenol (4c)* | 30 | 58–137 | | 56–180 | 51–153 |
| 531 | 2,4,5-trichlorophenol (4c)* | 30 | 58–137 | | 56–180 | 51–153 |
| 321 | 2,4,6-trichlorophenol | 57 | 59–205 | | 81–123 | 48–244 |
| 221 | 2,4,6-trichlorophenol-d2 | 47 | 43–183 | 21–363 | 69–144 | 34–226 |

[1] Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.

* Measured by internal standard; specification derived from related compound.
ns = no specification; limit is outside the range that can be measured reliably.

BILLING CODE 6560-50-M

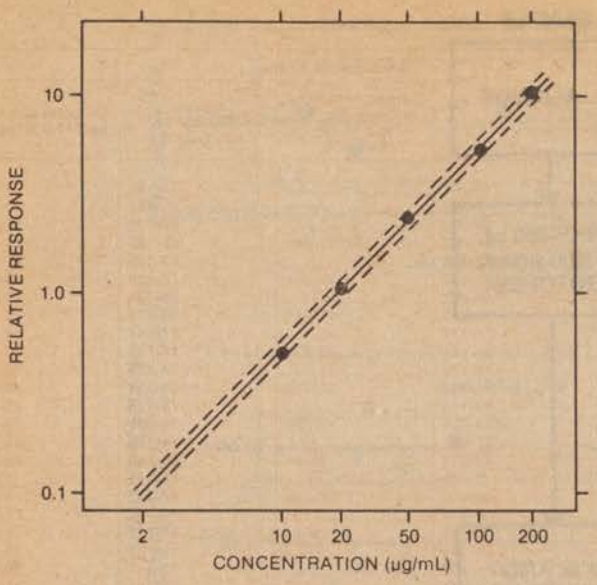



FIGURE 1 Relative Response Calibration Curve for Phenol. The Dotted Lines Enclose a ±10 Percent Error Window.

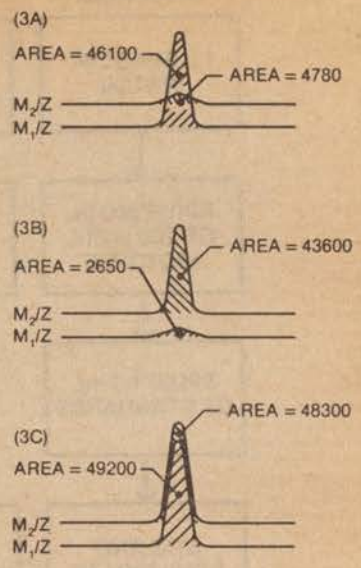



FIGURE 3 Extracted Ion Current Profiles for (3A) Unlabeled Compound, (3B) Labeled Compound, and (3C) Equal Mixture of Unlabeled and Labeled Compounds.

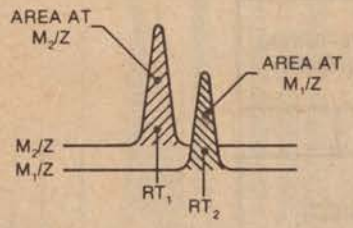



FIGURE 2 Extracted Ion Current Profiles for Chromatographically Resolved Labeled ($m_2/z$) and Unlabeled ($m_1/z$) Pairs.

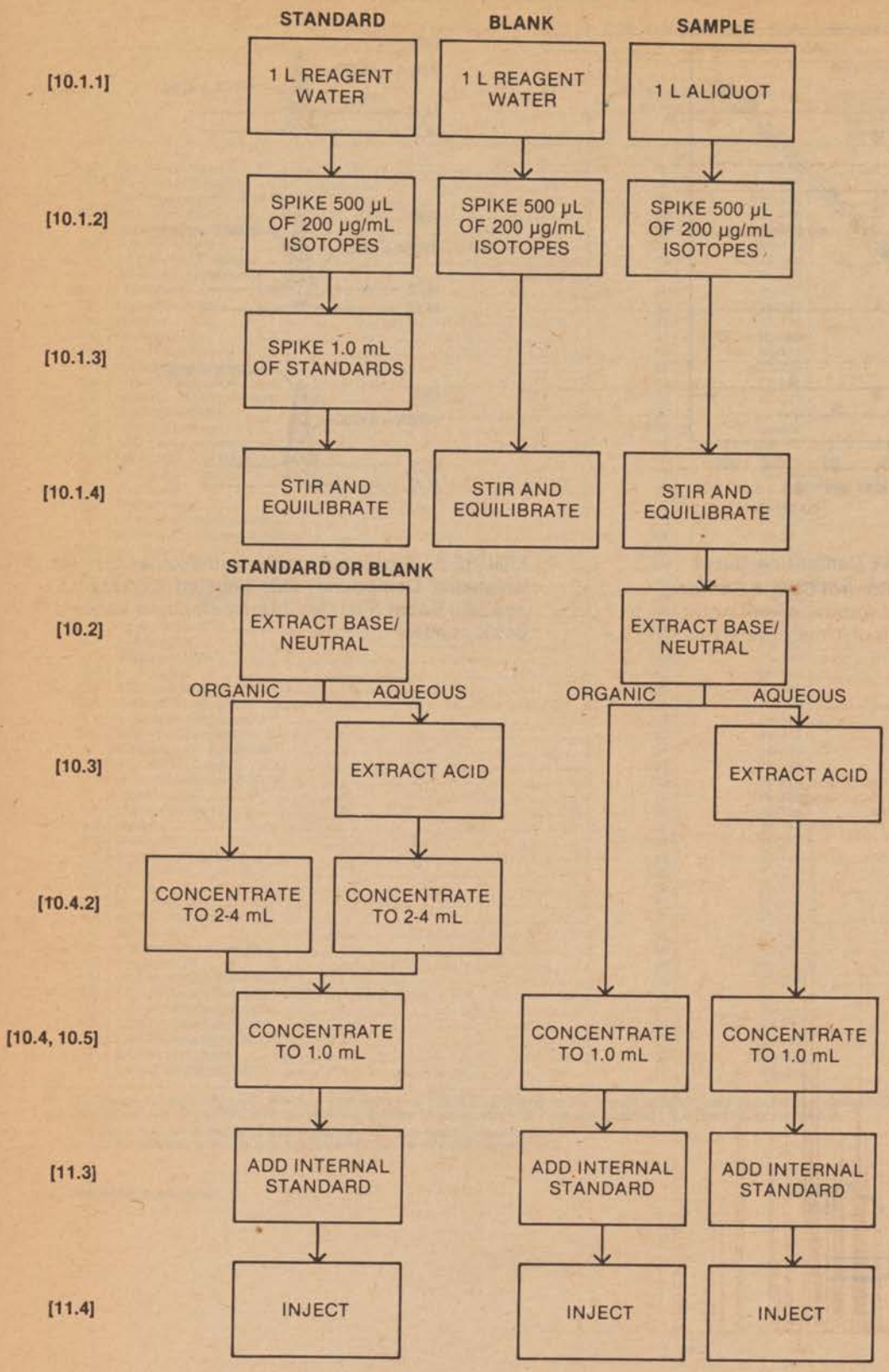



FIGURE 4 Flow Chart for Extraction/Concentration of Precision and Recovery Standard, Blank, and Sample by Method 1625. Numbers in Brackets [ ] Refer to Section Numbers in the Method.

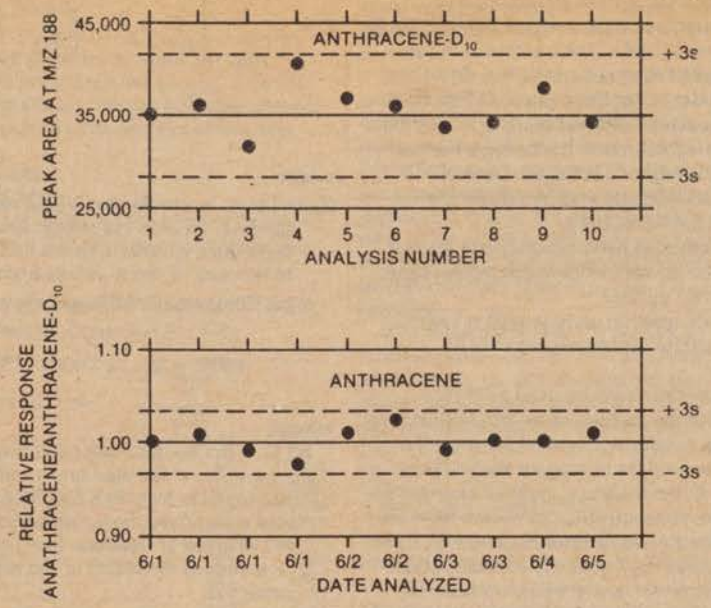



FIGURE 5 Quality Control Charts Showing Area (top graph) and Relative Response of Anthracene to Anthracene-$d_{10}$ (lower graph) Plotted as a Function of Time or Analysis Number.

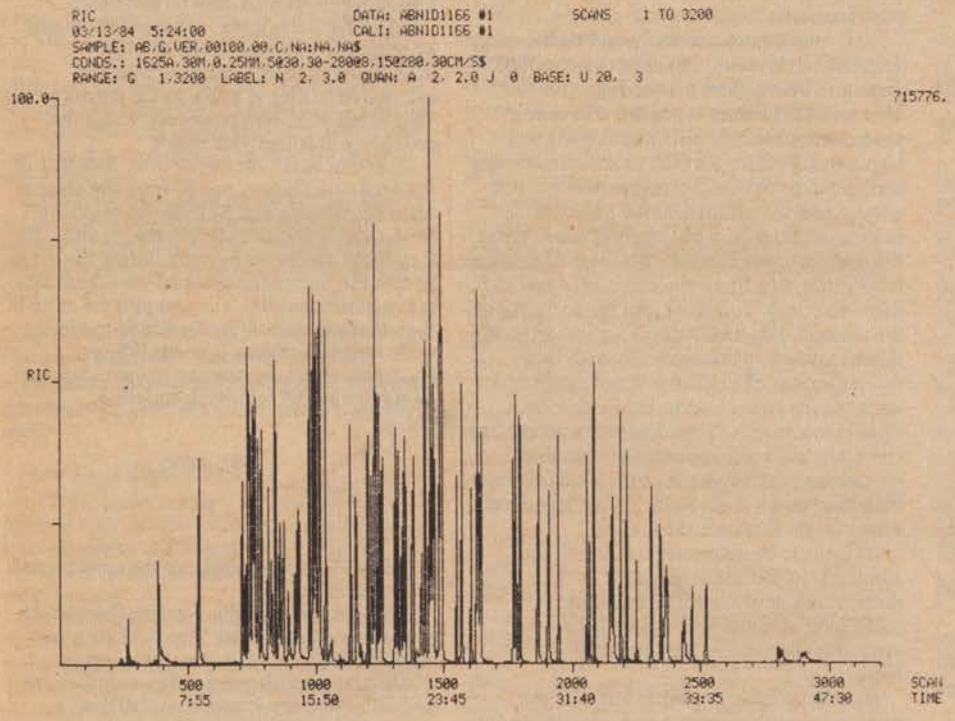



FIGURE 6 Chromatogram of Combined Acid/base/neutral Standard.

BILLING CODE 6560-50-C

## Appendix B to Part 136—Definition and Procedure for the Determination of the Method Detection Limit—Revision 1.11

*Definition*

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

*Scope and Application*

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instrument-independent.

*Procedure*

1. Make an estimate of the detection limit using one of the following:

(a) The concentration value that corresponds to an instrument signal/noise in the range of 2.5 to 5.

(b) The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.

(c) That region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.

(d) Instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the initial estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

3. (a) If the MDL is to be determined in reagent (blank) water, prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated method detection limit. (Recommend between 1 and 5 times the estimated method detection limit.) Proceed to Step 4.

(b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated detection limit, proceed to Step 4.

If the measured level of analyte is less than the estimated detection limit, add a known amount of analyte to bring the level of analyte between one and five times the estimated detection limit.

If the measured level of analyte is greater than five times the estimated detection limit, there are two options.

(1) Obtain another sample with a lower level of analyte in the same matrix if possible.

(2) The sample may be used as is for determining the method detection limit if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the method detection limit and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

(b) It may be economically and technically desirable to evaluate the estimated method detection limit before proceeding with 4a. This will: (1) Prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL even though the level of analyte is less than five times the calculated method detection limit. To insure that the estimate of the method detection limit is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower method detection limit. Take two aliquots of the sample to be used to calculate the method detection limit and process each through the entire method, including blank measurements as described above in 4a. Evaluate these data:

(1) If these measurements indicate the sample is in desirable range for determination of the MDL, take five additional aliquots and proceed. Use all seven measurements for calculation of the MDL.

(2) If these measurements indicate the sample is not in correct range, reestimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.

5. Calculate the variance ($S^2$) and standard deviation (S) of the replicate measurements, as follows:

$$S^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n} X_i^2 - \left(\sum_{i=1}^{n} X_i\right)^2 / n\right]$$

$$S = (S^2)^{1/2}$$

where:

$X_i$; i=1 to n, = are the analytical results in the final method reporting units obtained from the n sample aliquots and $\Sigma$ refers to the sum of the X values from i=1 to n.

6. (a) Compute the MDL as follows:

$$MDL = t_{(n-1,1-\alpha\,=\,0.99)}\ (S)$$

where:

MDL = the method detection limit

$t_{(n-1,1-\alpha\,=\,.99)}$ = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

(b) The 95% confidence interval estimates for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution ($\chi^2$/df).

LCL = 0.64 MDL

UCL = 2.20 MDL

where: LCL and UCL are the lower and upper 95% confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimate of the MDL and subsequent MDL determinations.

(a) If this is the initial attempt to compute MDL based on the estimate of MDL formulated in Step 1, take the MDL as calculated in Step 6, spike in the matrix at the calculated MDL and proceed through the procedure starting with Step 4.

(b) If this is the second or later iteration of the MDL calculation, use $S^2$ from the current MDL calculation and $S^2$ from the previous MDL calculation to compute the F-ratio. The F-ratio is calculated by substituting the larger $S^2$ into the numerator $S^2_A$ and the others into the denominator $S^2_B$. The computed F-ratio is then compared with the F-ratio found in the table which is 3.05 as follows: if $S^2_A/S^2_B < 3.05$, then compute the pooled standard deviation by the following equation:

$$S_{pooled} = \left[\frac{6S^2_A + 6S^2_B}{12}\right]^{1/2}$$

if $S^2_A/S^2_B > 3.05$, respike at the most recent calculated MDL and process the samples through the procedure starting with Step 4. If the most recent calculated MDL does not permit qualitative identification when samples are spiked at that level, report the MDL as a concentration between the current and previous MDL which permits qualitative identification.

(c) Use the $S_{pooled}$ as calculated in 7b to compute the final MDL according to the following equation:

$MDL = 2.681\ (S_{pooled})$

where 2.681 is equal to $t_{(12, 1-\alpha=.99)}$.

(d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from precentiles of the chi squared over degrees of freedom distribution.

LCL=0.72 MDL

UCL=1.65 MDL

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

TABLES OF STUDENTS' t VALUES AT THE 99 PERCENT CONFIDENCE LEVEL

| Number of replicates | Degrees of freedom (n-1) | $t_{(n, 1-.99)}$ |
|---|---|---|
| 7 | 6 | 3.143 |
| 8 | 7 | 2.998 |
| 9 | 8 | 2.896 |
| 10 | 9 | 2.821 |
| 11 | 10 | 2.764 |
| 16 | 10 | 2.602 |
| 21 | 20 | 2.528 |
| 26 | 25 | 2.485 |
| 31 | 30 | 2.457 |
| 61 | 60 | 2.390 |
| 00 | 00 | 2.326 |

*Reporting*

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options which affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with MDL value. Report the mean analyte level with the MDL and indicate if the MDL procedure was iterated. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, also report the mean recovery.

If the level of analyte in the sample was below the determined MDL or does not exceed 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.

## Appendix C to Part 136—Inductively Coupled Plasma—Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes Method 200.7

*1. Scope and Application*

1.1 This method may be used for the determination of dissolved, suspended, or total elements in drinking water, surface water, and domestic and industrial wastewaters.

1.2 Dissolved elements are determined in filtered and acidified samples. Appropriate steps must be taken in all analyses to ensure that potential interferences are taken into account. This is especially true when dissolved solids exceed 1500 mg/L. (See section 5.)

1.3 Total elements are determined after appropriate digestion procedures are performed. Since digestion techniques increase the dissolved solids content of the samples, appropriate steps must be taken to correct for potential interference effects. (See section 5.)

1.4 Table 1 lists elements for which this method applies along with recommended wavelengths and typical estimated instrumental detection limits using conventional pneumatic nebulization. Actual working detection limits are sample dependent and as the sample matrix varies, these concentrations may also vary. In time, other elements may be added as more information becomes available and as required.

1.5 Because of the differences between various makes and models of satisfactory instruments, no detailed instrumental operating instructions can be provided. Instead, the analyst is referred to the instruction provided by the manufacturer of the particular instrument.

*2. Summary of Method*

2.1 The method describes a technique for the simultaneous or sequential multielement determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in 5.1 (and tests for their presence as described in 5.2) should also be recognized and appropriate corrections made.

*3. Definitions*

3.1 *Dissolved*—Those elements which will pass through a 0.45 μm membrane filter.

3.2 *Suspended*—Those elements which are retained by a 0.45 μm membrane filter.

3.3 *Total*—The concentration determined on an unfiltered sample following vigorous digestion (Section 9.3), or the sum of the dissolved plus suspended concentrations. (Section 9.1 plus 9.2).

3.4 *Total recoverable*—The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid (Section 9.4).

3.5 *Instrumental detection limit*—The concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal at the same wavelength.

3.6 *Sensitivity*—The slope of the analytical curve, i.e. functional relationship between emission intensity and concentration.

3.7 *Instrument check standard*—A multielement standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis. (See 7.6.1)

3.8 *Interference check sample*—A solution containing both interfering and analyte elements of known concentration that can be used to verify background and interelement correction factors. (See 7.6.2.)

3.9 *Quality control sample*—A solution obtained from an outside source having known, concentration values to be used to verify the calibration standards. (See 7.6.3)

3.10 *Calibration standards*—A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). (See 7.4)

3.11 *Linear dynamic range*—The concentration range over which the analytical curve remains linear.

3.12 *Reagent blank*—A volume of deionized, distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme. (See 7.5.2)

3.13 *Calibration blank*—A volume of deionized, distilled water acidified with $HNO_3$ and HCl. (See 7.5.1)

3.14 *Method of standard addition*—The standard addition technique involves the use of the unknown and the unknown plus a known amount of standard. (See 10.6.1.)

*4. Safety*

4.1 The toxicity of carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is repsonsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [14.7, 14.8 and 14.9] for the information of the analyst.

*5. Interferences*

5.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

5.1.1 *Spectral interferences* can be categorized as (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; and (4) background contribution from stray light from

the line emission of high concentration elements. The first of these effects can be compensated by utilizing a computer correction of the raw data, requiring the monitoring and measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line. In addition, users of simultaneous multi-element instrumentation must assume the responsibility of verifying the absence of spectral interferenece from an element that could occur in a sample but for which there is no channel in the instrument array. Listed in Table 2 are some interference effects for the recommended wavelengths given in Table 1. The data in Table 2 are intended for use only as a rudimentary guide for the indication of potential spectral interferences. For this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed. The Interference information, which was collected at the Ames Laboratory,[1] is expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interferent element. The suggested use of this information is as follows: Assume that arsenic (at 193.696 nm) is to be determined in a sample containing approximately 10 mg/L of aluminum. According to Table 2, 100 mg/L of aluminum would yield a false signal for arsenic equivalent to approximately 1.3 mg/L. Therefore, 10 mg/L of aluminum would result in a false signal for arsenic equivalent to approximately 0.13 mg/L. The reader is cautioned that other analytical systems may exhibit somewhat different levels of interference than those shown in Table 2, and that the interference effects must be evaluated for each individual system.

Only those interferents listed were investigated and the blank spaces in Table 2 indicate that measurable interferences were not observed for the interferent concentrations listed in Table 3. Generally, interferences were discernible if they produced peaks or background shifts corresponding to 2–5% of the peaks generated by the analyte concentrations also listed in Table 3.

At present, information on the listed silver and potassium wavelengths are not available but it has been reported that second order energy from the magnesium 383.231 nm wavelength interferes with the listed potassium line at 766.491 nm.

5.1.2 *Physical interferences* are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem which can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aersol flow rate causing instrumental drift. Wetting the argon prior to nebulization, the use of a tip washer, or sample dilution have been used to control this problem. Also, it has been reported that better control of the argon flow rate improves instrument performance. This is accomplished with the use of mass flow controllers.

5.1.3 *Chemical Interferences* are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with the ICP technique, however, if observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

5.2 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in 5.2.1 through 5.2.4, will ensure the analyst that neither positive nor negative interference effects are operative on any of the analyte elements thereby distorting the accuracy of the reported values.

5.2.1 *Serial dilution*—If the analyte concentration is sufficiently high (minimally a factor of 10 above the instrumental detection limit after dilution), an analysis of a dilution should agree within 5 percent of the original determination (or within some acceptable control limit (14.3) that has been established for that matrix.). If not, a chemical or physical interference effect should be suspected.

5.2.2 *Spike addition*— The recovery of a spike addition added at a minimum level of 10X the instrumental detection limit (maximum 100X) to the original determination should be recovered to within 90 to 110 percent or within the established control limit for that matrix. If not, a matrix effect should be suspected. The use of a standard addition analysis procedure can usually compensate for this effect.

*Caution:* The standard addition technique does not detect coincident spectral overlap. If suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended (See 5.2.3).

5.2.3 *Comparison with alternate method of analysis*—When investigating a new sample matrix, comparison tests may be performed with other analytical techniques such as atomic absorption spectrometry, or other approved methodology.

5.2.4 *Wavelength scanning of analyte line region*—If the appropriate equipment is available, wavelength scanning can be performed to detect potential spectral interferences.

*6. Apparatus*

6.1 Inductively Coupled Plasma-Atomic Emission Spectrometer.

6.1.1 Computer controlled atomic emission spectrometer with background correction.

6.1.2 Radiofrequency generator.

6.1.3 Argon gas supply, welding grade or better.

6.2 Operating conditions—Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain quality control data confirming instrument performance and analytical results.

*7. Reagents and Standards*

7.1 Acids used in the preparation of standards and for sample processing must be ultra-high purity grade or equivalent. Redistilled acids are acceptable.

7.1.1 *Acetic acid,* conc. (sp gr 1.06).

7.1.2 *Hydrochloric acid,* conc. (sp gr 1.19).

7.1.3 *Hydrochloric acid,* (1+1): Add 500 mL conc. HCl (sp gr 1.19) to 400 mL deionized, distilled water and dilute to 1 liter.

7.1.4 *Nitric acid,* conc. (sp gr 1.41).

7.1.5 *Nitric acid,* (1+1): Add 500 mL conc. $HNO_3$ (sp gr 1.41) to 400 mL deionized, distilled water and dilute to 1 liter.

7.2 *Deionized, distilled water:* Prepare by passing distilled water through a mixed bed of cation and anion exchange resins. Use deionized, distilled water for the preparation of all reagents, calibration standards and as dilution water. The purity of this water must be equivalent to ASTM Type II reagent water of Specification D 1193 (14.6).

7.3 *Standard stock solutions* may be purchased or prepared from ultra high purity grade chemicals or metals. All salts must be dried for 1 h at 105 °C unless otherwise specified.

(CAUTION: Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling.)

Typical stock solution preparation procedures follow:

7.3.1 *Aluminum solution, stock,* 1 mL=μg Al: Dissolve 0.100 g of aluminum metal in an acid mixture of 4 mL of (1+1) HCl and 1 mL of conc. $HNO_3$ in a beaker. Warm gently to effect solution. When solution is complete, transfer quantitatively to a liter flask add an additional 10 mL of (1+1) HCl and dilute to 1,000 mL with deionized, distilled water.

7.3.2 *Antimony solution stock,* 1 mL=100 μg Sb: Dissolve 0.2669 g $K(SbO)C_4H_4O_6$ in deionized distilled water, add 10 mL (1+1) HCl and dilute to 1,000 mL with deionized, distilled water.

7.3.3 *Arsenic solution, stock,* 1 mL=100 μg As: Dissolve 0.1320 g of $As_2O_3$ in 100 mL of deionized, distilled water containing 0.4 g NaOH. Acidify the solution with 2 mL conc. $HNO_3$ and dilute to 1,000 mL with deionized, distilled water.

[1] Ames Laboratory, USDOE, Iowa State University, Ames Iowa 50011.

7.3.4 *Barium solution, stock,* 1 mL=100 μg Ba: Dissolve 0.1516 g $BaCl_2$ (dried at 250 °C for 2 hrs) in 10 mL deionized, distilled water with 1 mL (1+1) HCl. Add 10.0 mL (1+1) HCl and dilute to 1,000 with mL deionized, distilled water.

7.3.5 *Beryllium solution, stock,* 1 mL=100 μg Be: *Do not dry.* Dissolve 1.966 g $BeSO_4 \cdot 4H_2O$, in deionized, distilled water, add 10.0 mL conc. $HNO_3$ and dilute to 1,000 mL with deionized, distilled water.

7.3.6 *Boron solution, stock,* 1 mL=100 μg B: *Do not dry.* Dissolve 0.5716 g anhydrous $H_3BO_3$ in deionized, distilled water and dilute to 1,000 mL. Use a reagent meeting ACS specifications, keep the bottle tightly stoppered and store in a desiccator to prevent the entrance of atmospheric moisture.

7.3.7 *Cadmium solution, stock,* 1 mL=100 μg Cd: Dissolve 0.1142 g CdO in a minimum amount of (1+1) $HNO_3$. Heat to increase rate of dissolution. Add 10.0 mL conc. $HNO_3$ and dilute to 1,000 mL with deionized, distilled water.

7.3.8 *Calcium solution, stock,* 1 mL=100 μg Ca: Suspend 0.2498 g $CaCO_3$ dried at 180 °C for 1 h before weighing in deionized, distilled water and dissolve cautiously with a minimum amount of (1+1) $HNO_3$. Add 10.0 mL conc. $HNO_3$ and dilute to 1,000 mL with deionized, distilled water.

7.3.9 *Chromium solution, stock,* 1 mL=100 μg Cr: Dissolve 0.1923 g of $CrO_3$ in deionized, distilled water. When solution is complete, acidify with 10 mL conc. $HNO_3$ and dilute to 1,000 mL with deionized, distilled water.

7.3.10 *Cobalt solution, stock,* 1 mL=100 μg Co: Dissolve 0.1000 g of cobalt metal in a minimum amount of (1+1) $HNO_3$. Add 10.0 mL (1+1) HCl and dilute to 1,000 mL with deionized, distilled water.

7.3.11 *Copper solution, stock,* 1 mL=100 μg Cu: Dissolve 0.1252 g CuO in a minimum amount of (1+1) $HNO_3$. Add 10.0 mL conc. $HNO_3$ and dilute to 1,000 mL with deionized, distilled water.

7.3.12 *Iron solution, stock,* 1 mL=100 μg Fe: Dissolve 0.1430 g $Fe_2O_3$ in a warm mixture of 20 mL (1+1) HCl and 2 mL of conc. $HNO_3$. Cool, add an additional 5 mL of conc. $HNO_3$ and dilute to 1,000 mL with deionized, distilled water.

7.3.13 *Lead solution, stock,* 1 mL=100 μg Pb: Dissolve 0.1599 g $Pb(NO_3)_2$ in a minimum amount of (1+1) $HNO_3$. Add 10.0 mL conc. $HNO_3$ and dilute to 1,000 mL with deionized, distilled water.

7.3.14 *Magnesium solution, stock,* 1 mL=100 μg Mg: Dissolve 0.1658 g MgO in a minimum amount of (1+1) $HNO_3$. Add 10.0 mL conc. $HNO_3$ and dilute to 1,000 mL with deionized, distilled water.

7.3.15 *Manganese solution, stock,* 1 mL=100 μg Mn: Dissolve 0.1000 g of manganese metal in the acid mixture 10 mL conc. HCl and 1 mL conc. $HNO_3$, and dilute to 1,000 mL with deionized, distilled water.

7.3.16 *Molybdenum solution, stock,* 1 mL=100 μg Mo: Dissolve 0.2043 g $(NH_4)_2MoO_4$ in deionized, distilled water and dilute to 1,000 mL.

7.3.17 *Nickel solution, stock,* 1 mL=100 μg Ni: Dissolve 0.1000 g of nickel metal in 10 mL hot conc. $HNO_3$, cool and dilute to 1,000 mL with deionized, distilled water.

7.3.18 *Potassium solution, stock,* 1 mL=100 μg K: Dissolve 0.1907 g KCl, dried at 110 °C, in deionized, distilled water and dilute to 1,000 mL.

7.3.19 *Selenium solution, stock,* 1 mL=100 μg Se: *Do not dry.* Dissolve 0.1727 g $H_2SeO_3$ (actual assay 94.6%) in deionized, distilled water and dilute to 1,000 mL.

7.3.20 *Silica solution, stock,* 1 mL=100 μg $SiO_2$: *Do not dry.* Dissolve 0.4730 g $Na_2SiO_3 \cdot 9H_2O$ in deionized, distilled water. Add 10.0 mL conc. $HNO_3$ and dilute to 1,000 mL with deionized, distilled water.

7.3.21 *Silver solution, stock,* 1 mL=100 μg Ag: Dissolve 0.1575 g $AgNO_3$ in 100 mL of deionized, distilled water and 10 mL conc. $HNO_3$. Dilute to 1,000 mL with deionized, distilled water.

7.3.22 *Sodium solution, stock,* 1 mL=100 μg Na: Dissolve 0.2542 g NaCl in deionized, distilled water. Add 10.0 mL conc. $HNO_3$ and dilute to 1,000 mL with deionized, distilled water.

7.3.23 *Thallium solution, stock,* 1 mL=100 μg Tl: Dissolve 0.1303 g $TlNO_3$ in deionized, distilled water. Add 10.0 mL conc. $HNO_3$ and dilute to 1,000 mL with deionized, distilled water.

7.3.24 *Vanadium solution, stock,* 1 mL=100 μg V: Dissolve 0.2297 $NH_4VO_3$ in a minimum amount of conc. $HNO_3$. Heat to increase rate of dissolution. Add 10.0 mL conc. $HNO_3$ and dilute to 1,000 mL with deionized, distilled water.

7.3.25 *Zinc solution, stock,* 1 mL=100 μg Zn: Dissolve 0.1245 g ZnO in a minimum amount of dilute $HNO_3$. Add 10.0 mL conc. $HNO_3$ and dilute to 1,000 mL deionized, distilled water.

7.4 *Mixed calibration standard solutions*—Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. (See 7.4.1 thru 7.4.5) Add 2 mL of (1+1) $HNO_3$ and 10 mL of (1+1) HC1 and dilute to 100 mL with deionized, distilled water. (See Notes 1 and 6.) Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the mixed standards that the elements are compatible and stable. Transfer the mixed standard solutions to a FEP fluorocarbon or unused polyethylene bottle for storage. Fresh mixed standards should be prepared as needed with the realization that concentration can change on aging. Calibration standards must be initially verified using a quality control sample and monitored weekly for stability (See 7.6.3). Although not specifically required, some typical calibration standard combinations follow when using those specific wavelengths listed in Table 1.

7.4.1 *Mixed standard solution I*—Manganese, beryllium, cadmium, lead, and zinc.

7.4.2 *Mixed standard solution II*—Barium, copper, iron, vanadium, and cobalt.

7.4.3 *Mixed standard solution III*—Molybdenum, silica, arsenic, and selenium.

7.4.4 *Mixed standard solution IV*—Calcium, sodium, potassium, aluminum, chromium and nickel.

7.4.5 *Mixed standard solution V*—Antimony, boron, magnesium, silver, and thallium.

**Note 1.**—If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of deionized distilled water and warm the flask until the solution clears. Cool and dilute to 100 mL with deionized, distilled water. For this acid combination the silver concentration should be limited to 2 mg/L. Silver under these conditions is stable in a tap water matrix for 30 days. Higher concentrations of silver require additional HCl.

7.5 Two types of blanks are required for the analysis. The calibration blank (3.13) is used in establishing the analytical curve while the reagent blank (3.12) is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing.

7.5.1 *The calibration blank* is prepared by diluting 2 mL of (1+1) $HNO_3$ and 10 mL of (1+1) HCl to 100 mL with deionized, distilled water. (See Note 6.) Prepare a sufficient quantity to be used to flush the system between standards and samples.

7.5.2 *The reagent blank* must contain all the reagents and in the same volumes as used in the processing of the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

7.6 In addition to the calibration standards, an instrument check standard (3.7), an interference check sample (3.8) and a quality control sample (3.9) are also required for the analyses.

7.6.1 The *instrument check standard* is prepared by the analyst by combining compatible elements at a concentration equivalent to the midpoint of their respective calibration curves. (See 12.1.1.)

7.6.2 The *interference check sample* is prepared by the analyst in the following manner. Select a representative sample which contains minimal concentrations of the analytes of interest but known concentration of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest at the approximate concentration of either 100 μg/L or 5 times the estimated detection limits given in Table 1. (For effluent samples of expected high concentrations, spike at an appropriate level.) If the type of samples analyzed are varied, a synthetically prepared sample may be used if the above criteria and intent are met. A limited supply of a synthetic interference check sample will be available from the Quality Assurance Branch of EMSL-Cincinnati. (See 12.1.2).

7.6.3 The *quality control sample* should be prepared in the same acid matrix as the calibration standards at a concentration near 1 mg/L and in accordance with the instructions provided by the supplier. The Quality Assurance Branch of EMSL-Cincinnati will either supply a quality control sample or information where one of equal quality can be procured. (See 12.1.3.)

*8. Sample Handling and Preservation*

8.1 For the determination of trace elements, contamination and loss are of prime concern. Dust in the laboratory environment, impurities in reagents and impurities on laboratory apparatus which the sample contacts are all sources of potential contamination. Sample containers can introduce either positive or negative errors in the measurement of trace elements by (a) contributing contaminants through leaching or surface desorption and (b) by depleting concentrations through adsorption. Thus the collection and treatment of the sample prior to analysis requires particular attention. Laboratory glassware including the sample bottle (whether polyethylene, polyproplyene or FEP-fluorocarbon) should be thoroughly washed with detergent and tap water; rinsed with (1+1) nitric acid, tap water, (1+1) hydrochloric acid, tap and finally deionized, distilled water in that order (See Notes 2 and 3).

**Note 2.**—Chromic acid may be useful to remove organic deposits from glassware; however, the analyst should be cautioned that the glassware must be thoroughly rinsed with water to remove the last traces of chromium. This is especially important if chromium is to be included in the analytical scheme. A commercial product, NOCHROMIX, available from Godax Laboratories, 6 Varick St., New York, NY 10013, may be used in place of chromic acid. Chromic acid should not be used with plastic bottles.

**Note 3.**—If it can be documented through an active analytical quality control program using spiked samples and reagent blanks, that certain steps in the cleaning procedure are not required for routine samples, those steps may be eliminated from the procedure.

8.2 Before collection of the sample a decision must be made as to the type of data desired, that is dissolved, suspended or total, so that the appropriate preservation and pretreatment steps may be accomplished. Filtration, acid preservation, etc., are to be performed at the time the sample is collected or as soon as possible thereafter.

8.2.1 For the determination of dissolved elements the sample must be filtered through a 0.45-μm membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus are recommended to avoid possible contamination.) Use the first 50–100 mL to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1+1) $HNO_3$ to a pH of 2 or less. Normally, 3 mL of (1+1) acid per liter should be sufficient to preserve the sample.

8.2.2 For the determination of suspended elements a measured volume of unpreserved sample must be filtered through a 0.45-μm membrane filter as soon as practical after collection. The filter plus suspended material should be transferred to a suitable container for storage and/or shipment. No preservative is required.

8.2.3 For the determination of total or total recoverable elements, the sample is acidified with (1+1) $HNO_3$ to pH 2 or less as soon as possible, preferably at the time of collection. The sample is not filtered before processing.

*9. Sample Preparation*

9.1 For the determinations of dissolved elements, the filtered, preserved sample may often be analyzed as received. The acid matrix and concentration of the samples and calibration standards must be the same. (See Note 6.) If a precipitate formed upon acidification of the sample or during transit or storage, it must be redissolved before the analysis by adding additional acid and/or by heat as described in 9.3.

9.2 For the determination of suspended elements, transfer the membrane filter containing the insoluble material to a 150-mL Griffin beaker and add 4 mL conc. $HNO_3$. Cover the beaker with a watch glass and heat gently. The warm acid will soon dissolve the membrane. Increase the temperature of the hot plate and digest the material. When the acid has nearly evaporated, cool the beaker and watch glass and add another 3 mL of conc. $HNO_3$. Cover and continue heating until the digestion is complete, generally indicated by a light colored digestate. Evaporate to near dryness (2 mL), cool, and 10 mL HCl (1+1) and 15 mL deionized, distilled water per 100 mL dilution and warm the beaker gently for 15 min. to dissolve any precipitated or residue material. Allow to cool, wash down the watch glass and beaker walls with deionized distilled water and filter the sample to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the volume based on the expected concentrations of elements present. This volume will vary depending on the elements to be determined (See Note 6). The sample is now ready for analysis. Concentrations so determined shall be reported as "suspended."

**Note 4.**—In place of filtering, the sample after diluting and mixing may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

9.3 For the determination of total elements, choose a measured volume of the well mixed acid preserved sample appropriate for the expected level of elements and transfer to a Griffin beaker. (See Note 5.) Add 3 mL of conc. $HNO_3$. Place the beaker on a hot plate and evaporate to near dryness cautiously, making certain that the sample does not boil and that no area of the bottom of the beaker is allowed to go dry. Cool the beaker and add another 5 mL portion of conc. $HNO_3$. Cover the beaker with a watch glass and return to the hot plate. Increase the temperature of the hot plate so that a gently reflux action occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing.) Again, evaporate to near dryness and cool the beaker. Add 10 mL of 1+1 HCl and 15 mL of deionized, distilled water per 100 mL of final solution and warm the beaker gently for 15 min. to dissolve any precipitate or residue resulting from evaporation. Allow to cool, wash down the beaker walls and watch glass with deionized distilled water and filter the sample to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the sample to a predetermined volume based on the expected concentrations of elements present. The sample is now ready for analysis (See Note 6). Concentrations so determined shall be reported as "total."

**Note 5.**—If low determinations of boron are critical, quartz glassware should be used.

**Note 6.**—If the sample analysis solution has a different acid concentration from that given in 9.4, but does not introduce a physical interference or affect the analytical result, the same calibration standards may be used.

9.4 For the determination of total recoverable elements, choose a measured volume of a well mixed, acid preserved sample appropriate for the expected level of elements and transfer to a Griffin beaker. (See Note 5.) Add 2 mL of (1+1) $HNO_3$ and 10 mL of (1+1) HCl to the sample and heat on a steam bath or hot plate until the volume has been reduced to near 25 mL making certain the sample does not boil. After this treatment, cool the sample and filter to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the volume to 100 mL and mix. The sample is now ready for analysis. Concentrations so determined shall be reported as "total."

*10. Procedure*

10.1 Set up instrument with proper operating parameters established in Section 6.2. The instrument must be allowed to become thermally stable before beginning. This usually requires at least 30 min. of operation prior to calibration.

10.2 Initiate appropriate operating configuration of computer.

10.3 Profile and calibrate instrument according to instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Section 7.4. Flush the system with the calibration blank (7.5.1) between each standard. (See Note 7.) (The use of the average intensity of multiple exposures for both standardization and sample analysis has been found to reduce random error.)

**Note 7.**—For boron concentrations greater than 500 μg/L extended flush times of 1 to 2 minutes may be required.

10.4 Before beginning the sample run, reanalyze the highest mixed calibration standard as if it were a sample. Concentration values obtained should not deviate from the actual values by more than +5 percent (or the established control limits whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

10.5 Begin the sample run flushing the system with the calibration blank solution (7.5.1) between each sample. (See Note 7.) Analyze the instrument check standard (7.6.1) and the calibration blank (7.5.1) each 10 samples.

10.6 If it has been found that methods of standard addition are required, the following procedure is recommended.

10.6.1 The standard addition technique (14.2) involves preparing new standards in the sample matrix by adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constitutent that enhances or depresses the analyte signal thus

producing a different slope from that of the calibration standards. It will not correct for additive interference which causes a baseline shift. The simplest version of this technique is the single-addition method. The procedure is as follows. Two identical aliquots of the sample solution, each of volume $V_x$, are taken. To the first (labeled A) is added a small volume $V_s$ of a standard analyte solution of concentration $c_s$. To the second (labeled B) is added the same volume $V_s$ of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration $c_x$ is calculated:

$$c_x = \frac{S_B V_s c_s}{(S_A - S_B)\ V_x}$$

where $S_A$ and $S_B$ are the analytical signals (corrected for the blank) of solutions A and B, respectively. $V_s$ and $c_s$ should be chosen so that $S_A$ is roughly twice $S_B$ on the average. It is best if $V_s$ is made much less than $V_x$, and thus $c_s$ is much greater than $c_x$, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results from this technique to be valid, the following limitations must be taken into consideration:

1. The analytical curve must be linear.
2. The chemical form of the analyte added must respond the same as the analyte in the sample.
3. The interference effect must be constant over the working range of concern.
4. The signal must be corrected for any additive interference.

*11. Calculation*

11.1 Reagent blanks (7.5.2) should be subtracted from all samples. This is particularly important for digested samples requiring large quantities of acids to complete the digestion.

11.2 If dilutions were performed, the appropriate factor must be applied to sample values.

11.3 Data should be rounded to the thousandth place and all results should be reported in mg/L up to three significant figures.

*12. Quality Control (Instrumental)*

12.1 Check the instrument standardization by analyzing appropriate quality control check standards as follow:

12.1.1 Analyze and appropriate instrument check standard (7.6.1) containing the elements of interest at a frequency of 10%. This check standard is used to determine instrument drift. If agreement is not within ±5% of the expected values or within the established control limits, whichever is lower, the analysis is out of control. The analysis should be terminated, the problem corrected, and the instrument recalibrated.

Analyze the calibration blank (7.5.1) at a frequency of 10%. The result should be within the established control limits of 2 standard deviations of the mean value. If not, repeat the analysis two more times and average the three results. If the average is not within the control limit, terminate the analysis, correct the problem and recalibrate the instrument.

12.1.2 To verify interelement and background correction factors analyze the interference check sample (7.6.2) at the beginning, end, and at periodic intervals throughout the sample run. Results should fall within the established control limits of 1.5 times the standard deviation of the mean value. If not, terminate the analysis, correct the problem and recalibrate the instrument.

12.1.3 A quality control sample (7.6.3) obtained from an outside source must first be used for the initial verification of the calibration standards. A fresh dilution of this sample shall be analyzed every week thereafter to monitor their stability. If the results are not within ±5% of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

*13. Precision and Accuracy*

13.1 In an EPA round robin phase 1 study, even laboratories applied the ICP technique to acid-distilled water matrices that had been dosed with various metal concentrates. Table 4 lists the true value, the mean reported value and the mean % relative standard deviation.

*14. References*

14.1 Winge, R.K., V.J. Peterson, and V.A. Fassel, "Inductively Coupled Plasma-Atomic Emission Spectroscopy: Prominent Lines, EPA–600/4–79–017.

14.2 Winefordner, J.D., "Trace Analysis: Spectroscopic Methods for Elements," *Chemical Analysis*, Vol, *46*, pp. 41–42.

14.3 Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA–600/4–79–019.

14.4 Garbarino, J.R. and Taylor, H.E., "An Inductively-Coupled Plasma Atomic Emission Spectrometric Method for Routine Water Quality Testing," Applied Spectroscopy *33*, No. 3 (1979).

14.5 "Methods for Chemical Analysis of Water and Wastes," EPA–600/4–79–020.

14.6 Annual Book of ASTM Standards, Part 31.

14.7 "Carcinogens—Working With Carcinogens," Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, Aug. 1977.

14.8 "OSHA Safety and Health Standards, General Industry," (29 CFR 1910), Occupational Safety and Health Administration, OSHA 2206, (Revised, January 1976).

14.9 "Safety in Academic Chemistry Laboratories, American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

TABLE 1.—RECOMMENDED WAVELENGTHS [1] AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

| Element | Wavelength, nm | Estimated detection limit, µg/L [2] |
|---|---|---|
| Aluminum | 308.215 | 45 |
| Arsenic | 193.696 | 53 |
| Antimony | 206.833 | 32 |
| Barium | 455.403 | 2 |
| Beryllium | 313.042 | 0.3 |
| Boron | 249.773 | 5 |
| Cadmium | 226.502 | 4 |
| Calcium | 317.933 | 10 |
| Chromium | 267.716 | 7 |
| Cobalt | 228.616 | 7 |
| Copper | 324.754 | 6 |
| Iron | 259.940 | 7 |
| Lead | 220.353 | 42 |
| Magnesium | 279.079 | 30 |
| Manganese | 257.610 | 2 |
| Molybdenum | 202.030 | 8 |
| Nickel | 231.604 | 15 |
| Potassium | 766.491 | [3] |
| Selenium | 196.026 | 75 |
| Silica ($SiO_2$) | 288.158 | 58 |
| Silver | 328.068 | 7 |
| Sodium | 588.995 | 29 |
| Thallium | 190.864 | 40 |
| Vanadium | 292.402 | 8 |
| Zinc | 213.856 | 2 |

[1] The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. (See 5.1.1).

[2] The estimated instrumental detection limits as shown are taken from "Inductively Coupled Plasma-Atomic Emission Spectroscopy-Prominent Lines," EPA–600/4–79–017. They are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

[3] Highly dependent on operating conditions and plasma position.

TABLE 1.—ANALYTE CONCENTRATION EQUIVALENTS (MG/L) ARISING FROM INTERFERENTS AT THE 100 MG/L LEVEL

| Analyte | Wavelength, nm | Interferent— | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Al | Ca | Cr | Cu | Fe | Mg | Mn | Ni | Ti | V |
| Aluminum | 308.214 | | | | | | | 0.21 | | | 1.4 |
| Antimony | 206.833 | 0.47 | | 2.9 | | 0.08 | | | | 0.25 | 0.45 |
| Arsenic | 193.696 | 1.3 | | 0.44 | | | | | | | 1.1 |
| Barium | 455.403 | | | | | | | | | | |
| Beryllium | 313.042 | | | | | | | | | 0.04 | 0.05 |
| Boron | 249.773 | 0.04 | | | | 0.32 | | | | | |
| Cadmium | 226.502 | | | | | 0.03 | | | 0.02 | | |
| Calcium | 317.933 | | | 0.08 | | 0.01 | 0.01 | 0.04 | | 0.03 | 0.03 |
| Chromium | 267.716 | | | | | 0.003 | | 0.04 | | | 0.04 |
| Cobalt | 228.616 | | | 0.03 | | 0.005 | | | 0.03 | 0.15 | |
| Copper | 324.754 | | | | | 0.003 | | | | 0.05 | 0.02 |

TABLE 1.—ANALYTE CONCENTRATION EQUIVALENTS (MG/L) ARISING FROM INTERFERENTS AT THE 100 MG/L LEVEL—Continued

| Analyte | Wavelength, nm | Interferent— | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Al | Ca | Cr | Cu | Fe | Mg | Mn | Ni | Ti | V |
| Iron | 259.940 | | | | | | | 0.12 | | | |
| Lead | 220.353 | 0.17 | | | | | | | | | |
| Magnesium | 279.079 | | 0.02 | 0.11 | | 0.13 | | 0.25 | | 0.07 | 0.12 |
| Manganese | 257.610 | 0.005 | | 0.01 | | 0.002 | 0.002 | | | | |
| Molybdenum | 202.030 | 0.05 | | | | 0.03 | | | | | |
| Nickel | 231.604 | | | | | | | | | | |
| Selenium | 196.026 | 0.23 | | | | 0.09 | | | | | |
| Silicon | 288.158 | | | 0.07 | | | | | | | 0.01 |
| Sodium | 588.995 | | | | | | | | | 0.08 | |
| Thallium | 190.864 | 0.30 | | | | | | | | | |
| Vanadium | 292.402 | | | 0.05 | | 0.005 | | | | 0.02 | |
| Zinc | 213.856 | | | | 0.14 | | | | 0.29 | | |

TABLE 3. INTERFERENT AND ANALYTE ELEMENTAL CONCENTRATIONS USED FOR INTERFERENCE MEASUREMENTS IN TABLE 2

| Analytes | (mg/L) | Interferents | (mg/L) |
|---|---|---|---|
| Al | 10 | Al | 1,000 |
| AS | 10 | Ca | 1,000 |
| B | 10 | Cr | 200 |
| Ba | 1 | Cu | 200 |
| Be | 1 | Fe | 1,000 |
| Ca | 1 | Mg | 1,000 |
| Cd | 10 | Mn | 200 |
| Co | 1 | Ni | 200 |
| Cr | 1 | Ti | 200 |
| Cu | 1 | V | 200 |
| Fe | 1 | | |
| Mg | 1 | | |
| Mn | 1 | | |
| Mo | 10 | | |
| Na | 10 | | |
| Ni | 10 | | |
| Pb | 10 | | |
| Sb | 10 | | |
| Se | 10 | | |
| Si | 1 | | |
| Tl | 10 | | |
| V | 1 | | |
| Zn | 10 | | |

TABLE 4.—ICP PRECISION AND ACCURACY DATA

| Element | Sample No. 1 | | | Sample No. 2 | | | Sample No. 3 | | |
|---|---|---|---|---|---|---|---|---|---|
| | True value µg/L | Mean reported value µg/L | Mean percent RSD | True value µg/L | Mean reported value µg/L | Mean percent RSD | True value µg/L | Mean reported value µg/L | Mean percent RSD |
| Be | 750 | 733 | 6.2 | 20 | 20 | 9.8 | 180 | 176 | 5.2 |
| Mn | 350 | 345 | 2.7 | 15 | 15 | 6.7 | 100 | 99 | 3.3 |
| V | 750 | 749 | 1.8 | 70 | 69 | 2.9 | 170 | 169 | 1.1 |
| As | 200 | 208 | 7.5 | 22 | 19 | 23 | 60 | 63 | 17 |
| Cr | 150 | 149 | 3.8 | 10 | 10 | 18 | 50 | 50 | 3.3 |
| Cu | 250 | 235 | 5.1 | 11 | 11 | 40 | 70 | 67 | 7.9 |
| Fe | 600 | 594 | 3.0 | 20 | 19 | 15 | 180 | 178 | 6.0 |
| Al | 700 | 696 | 5.6 | 60 | 62 | 33 | 160 | 161 | 13 |
| Cd | 50 | 48 | 12 | 2.5 | 2.9 | 16 | 14 | 13 | 16 |
| Co | 500 | 512 | 10 | 20 | 20 | 4.1 | 120 | 108 | 21 |
| Ni | 250 | 245 | 5.8 | 30 | 28 | 11 | 60 | 55 | 14 |
| Pb | 250 | 236 | 16 | 24 | 30 | 32 | 80 | 80 | 14 |
| Zn | 200 | 201 | 5.6 | 18 | 19 | 45 | 80 | 82 | 9.4 |
| Se | 40 | 32 | 21.9 | 6 | 8.5 | 42 | 10 | 8.5 | 8.3 |

Not all elements were analyzed by all laboratories.

[Doc. 84-28189 Filed 10-23-84; 8:45 am]

BILLING CODE 6560-50-M

**ENVIRONMENTAL PROTECTION AGENCY**

**40 CFR Part 136**

**[FRL-2636-6]**

## Guidelines Establishing Test Procedures for the Analysis of Pollutants

**AGENCY:** Environmental Protection Agency (EPA).

**ACTION:** Proposed regulation.

**SUMMARY:** Elsewhere in this issue of the **Federal Register**, EPA has promulgated new test procedures under Clean Water Act (CWA) Section 304(h) for the analysis of many priority toxic organic and other pollutants, which are based upon gas chromatographic instrumental systems. This proposal would withdraw approval for outdated test procedures which had been approved for sixteen compounds, including chlorinated organic compounds, benzidine, and for fourteen pesticide compounds. EPA is also proposing to approve two methods (Methods 1624 and 1625) for new compounds. These methods were promulgated for other compounds elsewhere in today's **Federal Register**.

**DATE:** Comments on this proposal must be submitted on or before January 10, 1985.

**ADDRESS:** Send comments to Dr. Robert B. Medz, "Proposed 304(h) Guidelines," Water and Waste Management Monitoring Research Division, Office of Research and Development (RD-680), 401 M Street, SW., Washington, D.C. 20460.

**FOR FURTHER INFORMATION CONTACT:** Dr. Robert B. Medz at the address listed above, or call (202) 382-5788. The record for the rulemaking is available for review at Washington, D.C.

**SUPPLEMENTARY INFORMATION:**

### I. Authority and Background

This regulation is proposed under the authority of sections 301, 304(h) and 501(a) of the Clean Water Act of 1977 (CWA).

It would amend 40 CFR Part 136 in two ways. First, it would withdraw approval of outmoded methods for the analysis of 30 chemical compounds. Second, it would expand the scope of two recently approved methods to include thirty-two additional compounds for which no approved methods now exist.

Elsewhere in today's **Federal Register**, EPA has approved new test procedures for the analysis of 111 priority, toxic organic pollutants. The analytical test procedures are based on 12 gas chromatograph (GC) methods and 5 gas chromatograph/mass spectrometer (GC/MS) methods. These test procedures differ from those previously approved by the EPA at 40 CFR Part 136. Their most important improvement is that they include detailed quality control requirements and specify control limits indicating inadequate performance. If an analyst's performance falls outside those control limits, his analytical system is considered to be out-of-control and data generated with the system is not reportable for regulatory purposes.

EPA approved analytical methods for 3 classes of organic pollutants on December 1, 1976 (41 FR 25780). These classes included 30 compounds which were later included within the scope of the priority pollutants. The 1976 test procedures had neither detailed quality control requirements nor warning limits within their provisions. In complex industrial and municipal wastewater matricies, application of these 1976 test procedures results in data of poorly defined quality if the analyst fails to perform an adequate level of quality control. The procedures were updated in 1978 but the underlying problems with quality control were not addressed. Therefore, for the 30 organic compounds, there is an inconsistency between the test procedures approved in 1976 and the test procedures approved today. This proposal will withdraw approval for the test procedures as they apply to these 30 pollutants. In effect, the old GC procedures will be superseded by the 15 test procedures which have been approved today for the 111 priority toxic organic pollutants.

EPA has approved two GC/MS test procedures (Methods 1624 and 1625) today. These methods use stable, isotopically labeled analogs of the priority pollutants as internal standards. They have been extensively tested for their applicability to the analysis of the 32 pollutants included pursuant to paragraph 4(c) and Appendix C of the Consent Decree (NRDC v. Train, 8 ERC 2120 (D.D.C. 1976), as modified 12 ERC 1833 (D.D.C. 1979) and by the Court's Order of October 26, 1982, August 2, 1983, January 6, 1984, and July 5, 1984). The Consent Decree settled a suit between the Natural Resources Defense Council (NRDC) and EPA regarding regulation under the CWA. In paragraph 4(c), EPA agreed to study pollutants for possible regulation, including these specified in Appendix C. Today's proposal would extend the approved scope of the two methods to include the additional paragraph 4(c) and Appendix C pollutants.

### II. Summary and Rationale for Proposed Amendments

#### *A. Withdrawal of Former Method Approvals*

Thirty compounds which were approved as parameters in the 1976 amendments to 40 CFR Part 136 were also included in the 1976 Consent Decree as priority pollutants. In the 1976 regulation they were carried under the parameter designations Benzidine, Pentachlorophenol, Chlorinated organic compounds (except pesticides), and Pesticides. Approved test procedures for these parameters were available from EPA's Environmental Monitoring and Support Laboratory in Cincinnati, Ohio. The method for Benzidine was covered under "Method for Benzidine and Its Salts in Wastewater." The remaining parameters were covered under "Procedures for Pentachlorophenol, Chlorinated Organic Compounds, and Pesticides."

These analytical methods were updated in 1978 by the EPA publication, "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol, and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September 1978. However, the earlier methods and the 1978 update do not require a mandatory level of quality control and do not stipulate analytical control limits, outside of which an analysis would be considered to be out-of-control. Therefore, the analyst could generate data which would be unusable for regulatory purposes. The new methods being approved today for priority pollutant analysis do include these procedures. This leads to a significant inconsistency between these new methods and the updated methods which had not been anticipated at the time that the priority pollutant test procedures were proposed on December 3, 1979 (44 FR 69464).

The present proposed action will eliminate this inconsistency and ensure that these earlier methods will be superceded by the test procedures being approved today for the priority pollutants for the following specific parameters in Tables IC and ID of § 136.3: Table IC, Benzidine, Carbon Tetrachloride, Chlorobenzene, Chloroform, Methylene chloride, PCB-1016, PCB-1221, PCB-1232, PCB-1242, PCB-1248, PCB-1260, Pentachlorophenol, 1,1,2,2-Tetrachloroethane, Tetrachloroethene, 1,2,4-Trichlorobenzene, and 1,1,2-Trichloroethane; and Table ID, Aldrin, $\alpha$-BHC, $\gamma$-BHC, Chlordane, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Dieldrin, Endosulfan

I, Endosulfan II, Endrin, Heptachlor, Heptachlor expoxide, and Toxaphene.

The methods approved in 1976 and updated in 1978 may continue to be the basis for EPA enforcement action where proper quality control and quality assurance are used. Of course, EPA may also use the new methods where they are more appropriate or more economical.

*B. Extension of New Methods to Appendix C Parameters*

EPA is proposing to expand Table IC of § 136.3 by 32 paragraph 4(c) and Appendix C parameters (it will now include 129 parameters). The purge and trap test procedure, Method 1624, is approved for four of these purgeable compounds: Acetone, Diethyl ether, p-Dioxane, and Methyl ethyl ketone. Extraction test procedure, Method 1625, is approved for 28 compounds: Benzoic acid, Biphenyl, Carbazole, p-Cymene, n-Decane, Dibenzofuran, Dibenzothiophene, Diphenylamine, Diphenyl ether, 1,2-Diphenylhydrazine, n-Docosane, n-Dodecane, n-Eicosane, n-Hexacosane, n-Hexadecane, Hexanoic acid, β-Naphthylamine, n-Octacosane, n-Octadecane, α-Picoline, Styrene, α-Terpineol, n-Tetracosane, n-Tetradecane, n-Triacontane, 1,2,3-Trichlorobenzene, 2,3,6-Trichlorophenol, and 2,4,5-Trichlorophenol. These methods have been extensively applied to the analysis of these 32 compounds in industrial wastewaters and the methods have been validated for their applicability to analysis of these compounds by multi-laboratory testing.

*C. Other Part 136 Provisions*

EPA wishes to make clear that methods for the 32 new compounds that will be covered under Part 136 will be subject to all the existing definitions and provisions of Part 136. For example, EPA will be able to approve equivalent methods for these new compounds, as it can for any parameter subject to this Part.

**III. Regulatory Analysis**

(a) Under Executive Order 12291, EPA must judge whether a regulation is "major" and, therefore, subject to the requirement of a "Regulatory Impact Analysis." This regulation is not major for the following reasons:

(1) It proposes analytical methods and sample handling requirements that ensure a uniform measure of pollutants across all wastewater discharges within minimum acceptance criteria for 32 parameters. The purpose is to ensure that the quality of environmental monitoring data meets certain minimum standards. It would withdraw the use of outdated methods.

(2) The impact of this regulation will be far less than $100 million.

a. The regulation affects unit monitoring costs for other regulatory programs, e.g., effluent guidelines regulations and the implementation regulations of the National Pollutant Discharge Elimination System (NPDES), and the pretreatment program. However, it does not impose those costs. In fact, the monitoring costs for other programs are considered in those other rulemakings. This is appropriate because total (rather than unit) monitoring costs are determined by the monitoring provisions of those regulations.

b. Equivalency provisions will encourage the development of innovative analytical methods by the private sector and to encourage the competitive viability of the instrument manufacturing industry. The equivalency provision also allows individual dischargers to gain approval of analytical systems of their own design that may further reduce their total monitoring costs.

(3) The empact of compliance with these regulations will not be concentrated on any particular sectors of American industry.

This regulation was submitted to the Office of Management and Budget (OMB) for review as required by Executive Order 12291. Any comments from OMB to EPA and any EPA response to those comments will be available for public inspection at the Public Information Reference Unit, Room M2904 (EPA Library-Rear), TM-213, Environmental Protection Agency, 401 M Street, SW., Washington, D.C. 20460, Phone: (202) 382-5926, Office Hours 8:00 a.m. to 4:30 p.m.

(b) Under the Regulatory Flexibility Act, 5 U.S.C. 601, *et seq.*, EPA is required to determine whether a regulation will significantly affect a substantial number of small entities so as to require a regulatory analysis. The regulation requires no new reports beyond those already now required. The analytical techniques approved here either can be handled by small facilities, or are widely available by contract at a reasonable price. Therefore, in accordance with 5 U.S.C. 605(b), I hereby certify that this rule will not have a significant adverse economic impact on a substantial number of small facilities.

(c) Under the Paperwork Reduction Act of 1980, 44 U.S.C. 3501 *et seq.*, the information provisions in this rule associated with the analytical test procedures equivalency program, 40 CFR 136.3 (a), (c) and (d), 136.4 and 136.5, and the sample preservation and holding times variances, 40 CFR 136.3(e), have been submitted to the Office of Management and Budget (OMB) as part of the final and interim-final rule published elsewhere in today's **Federal Register**. All approvals made on the final rule will be applicable to this proposed rule.

(Secs. 301, 304(h), 307 and 501(a), Pub. L. 95-217, 91 Stat. 1566, et seq. (33 U.S.C. 1251, et seq.) (the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977))

Dated: September 26, 1984.

**William D. Ruckleshaus,**

*Administrator.*

**List of Subjects in 40 CFR Part 136**

Water pollution control.

In consideration of the preceding, EPA proposes to amend Chapter I, Subchapter D of Title 40, Code of Federal Regulations, as follows:

**Proposed Rule**

For the reasons set out in the Preamble, it is proposed to amend Part 136, Chapter 1, Subchapter D of Title 40 of the Code of Federal Regulations as follows:

1. In § 136.3, Table IC is amended by renumbering to have 129 parameters, and by alphabetically inserting the following thirty-two new parameters: Acetone, Benzoic acid, Biphenyl, Carbazole, p-Cymene, n-Decane, Dibenzofuran, Dibenzothiophene, Diethyl ether, p-Dioxane, Diphenylamine, Diphenyl ether, 1,2-Diphenylhydrazine, n-Docosane, n-Dodecane, n-Eicosane, n-Hexacosane, n-Hexadecane, Hexanoic acid, Methyl ethyl ketone, β-Naphthylamine, n-Octacosane, n-Octadecane, α-Picoline, Styrene, α-Terpineol, n-Tetracosane, n-Tetradecane, n-Triacontane, 1,2,3-Trichlorobenzene, 2,3,6-Trichlorophenol, and 2,4,5,-Trichlorophenol; by approving EPA Method 1624 for the analysis of Acetone, Diethyl ether, p-Dioxane, and Methyl ethyl ketone; by approving EPA Method 1625 for the analysis of Benzoic acid, Biphenyl, Carbazole, p-Cymene, n-Decane, Dibenzofuran, Dibenzothiophene, Diphenylamine, Diphenyl ether, 1,2-Diphenylhydrazine, n-Docosane, n-Dodecane, n-Eicosane, n-Hexacosane, n-Hexadecane, Hexanoic acid, β-Naphthylamine, n-Octacosane, n-Octadecane, α-Picoline, Styrene, α-Terpineol, n-Tetracosane, n-Tetradecane, n-Triacontane, 1,2,3-Trichlorobenzene, 2,3,6-Trichlorophenol, and 2,4,5-Trichlorophenol; by withdrawing approval of the following cited test procedures for the following

parameters: Benzidine, oxidation-colorimetric procedure, "Method for Benzidine and Its Salts in Wastewater", Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio (1976); and Carbon tetrachloride, Chlorobenzene, Chloroform, Methylene chloride, PCB-1016, PCB-1221, PCB-1232, PCB-1242, PCB-1248, PCB-1260, Pentachlorophenol, 1,1,2,2-Tetrachloroethane, Tetrachloroethene, 1,2,4-Trichlorobenzene and 1,1,2-Trichlolorethane, gas chromatography, "Procedures for Pentachlorphenol, Chlorinated Organic Compounds, and Pesticides", Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio (1976). As proposed to be revised, § 136.3, Table IC would read as follows:

**§ 136.3 Identification of Test Procedures.**

* * * * *

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR ORGANIC COMPOUNDS

| Parameter [1] | EPA method number [2,7] | | | |
|---|---|---|---|---|
| | GC | GC/MS | HPLC | Other |
| 1. Acenaphthene | 610 | 625, 1625 | 610 | |
| 2. Acenaphthylene | 610 | 625, 1625 | 610 | |
| 3. Acetone | | 1624 | | |
| 4. Acrolein | 603 | [4] 624, 1624 | | |
| 5. Acrylonitrile | 603 | [4] 624, 1624 | | |
| 6. Anthracene | 610 | 625, 1625 | 610 | |
| 7. Benzene | 602 | 624, 1624 | | |
| 8. Benzidine | | [5] 625, 1625 | 605 | |
| 9. Benzo(a)anthracene | 610 | 625, 1625 | 610 | |
| 10. Benzoic Acid | | 1625 | | |
| 11. Benzo(a)pyrene | 610 | 625, 1625 | 610 | |
| 12. Benzo(b)fluoranthene | 610 | 625, 1625 | 610 | |
| 13. Benzo(ghi)perylene | 610 | 625, 1625 | 610 | |
| 14. Benzo(k)fluoranthene | 610 | 625, 1625 | 610 | |
| 15. Benzyl Chloride | | | | Note 3, p. 130; Note 6, p. S102 |
| 16. Benzyl Butyl Phthalate | 606 | 625, 1625 | | |
| 17. Biphenyl | | 1625 | | |
| 18. Bis(2-chloroethoxy) methane | 611 | 625, 1625 | | |
| 19. Bis(2-chloroethyl) ether | 611 | 625, 1625 | | |
| 20. Bis(2-ethylhexyl) phthalate | 606 | 625, 1625 | | |
| 21. Bromodichloromethane | 601 | 624, 1624 | | |
| 22. Bromoethane | 601 | 624, 1624 | | |
| 23. Bromoform | 601 | 624, 1624 | | |
| 24. 4-Bromophenylphenyl ether | 611 | 625, 1625 | | |
| 25. Carbazole | | 1625 | | |
| 26. Carbon tetrachloride | 601 | 624, 1624 | | |
| 27. Chloroethane | 601 | 624, 1624 | | |
| 28. 4-Chloro-3-methylphenol | 604 | 625, 1625 | | |
| 29. Chlorobenzene | 601, 602 | 624, 1624 | | |
| 30. 2-Chloroethylvinyl ether | 601 | 624, 1624 | | |
| 31. Chloroform | 601 | 624, 1624 | | |
| 32. Chloromethane | 601 | 624, 1624 | | |
| 33. 2-Chloronaphthalene | 612 | 625, 1625 | | |
| 34. 2-Chlorophenol | 604 | 625, 1625 | | |
| 35. 4-Chlorophenylphenyl ether | 611 | 625, 1625 | | |
| 36. Chrysene | 610 | 625, 1625 | 610 | |
| 37. p-Cymene | | 1625 | | |
| 38. n-Decane | | 1625 | | |
| 39. Dibenzo(a,h)anthracene | 610 | 625, 1625 | 610 | |
| 40. Dibenzofuran | | 1625 | | |
| 41. Dibenzothiophene | | 1625 | | |
| 42. Dibromochlorobenzene | 601 | 624, 1624 | | |
| 43. 1,2-Dichlorobenzene | 601, 602 | 624, 625 | | |
| | 612 | 1625 | | |
| 44. 1,3-Dichlorobenzene | 601, 602 | 624, 625 | | |
| | 612 | 1625 | | |
| 45. 1,4-Dichlorobenzene | 601, 602 | 624, 625 | | |
| | 612 | 1625 | | |
| 46. 3,3'-Dichlorobenzidine | | 625, 1625 | 605 | |
| 47. Dichlorodifluoromethane | 601 | | | |
| 48. 1,1-Dichloroethane | 601 | 624, 1624 | | |
| 49. 1,2-Dichloroethane | 601 | 624, 1624 | | |
| 50. 1,1-Dichloroethene | 601 | 624, 1624 | | |
| 51. trans-1,2-Dichloroethene | 601 | 624, 1624 | | |
| 52. 2,4-Dichlorophenol | 604 | 625, 1625 | | |
| 53. 1,2-Dichloropropane | 601 | 624, 1624 | | |
| 54. cis-1,3-Dichloropropene | 601 | 624, 1624 | | |
| 55. trans-1,3-Dichloropropene | 601 | 624, 1624 | | |
| 56. Diethyl ether | | 1624 | | |
| 57. Diethyl phthalate | 606 | 625, 1625 | | |
| 58. 2,4-Dimethylphenol | 604 | 625, 1625 | | |
| 59. Dimethyl phthalate | 606 | 625, 1625 | | |
| 60. Di-n-butyl phthalate | 606 | 625, 1625 | | |
| 61. Di-n-octyl phthalate | 606 | 625, 1625 | | |
| 62. 2,4-Dinitrophenol | 604 | 625, 1625 | | |
| 63. 2,4-Dinitrotoluene | 609 | 625, 1625 | | |
| 64. 2,6-Dinitrotoluene | 609 | 625, 1625 | | |
| 65. p-Dioxane | | 1624 | | |
| 66. Diphenylamine | | 1625 | | |
| 67. Diphenyl ether | | 1625 | | |
| 68. 1,2-Diphenylhydrazine | | 1625 | | |
| 69. n-Docosane | | 1625 | | |
| 70. n-Dodecane | | 1625 | | |
| 71. n-Eicosane | | 1625 | | |
| 72. Epichlorohydrin | | | | Note 3, p. 130; Note 6, p. S102. |
| 73. Ethylbenzene | 602 | 624, 1624 | | |

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR ORGANIC COMPOUNDS—Continued

| Parameter [1] | EPA method number [2] [7] | | | |
|---|---|---|---|---|
| | GC | GC/MS | HPLC | Other |
| 74. Fluoranthene | 610 | 625, 1625 | 610 | |
| 75. Fluorene | 610 | 625, 1625 | 610 | |
| 76. Hexachlorobenzene | 612 | 625, 1625 | | |
| 77. Hexachlorobutadiene | 612 | 625, 1625 | | |
| 78. Hexachlorocyclopentadiene | 612 | [5] 625, 1625 | | |
| 79. Hexachloroethane | 612 | 625, 1625 | | |
| 80. n-Hexacosane | | 1625 | | |
| 81. n-Hexadecane | | 1625 | | |
| 82. Hexanoic acid | | 1625 | | |
| 83. Ideno(1,2,3-cd)pyrene | 610 | 625, 1625 | 610 | |
| 84. Isophorone | 609 | 625, 1625 | | |
| 85. Methylene Chloride | 601 | 624, 1624 | | |
| 86. 2-Methyl-4,6-Dinitrophenol | 604 | 625, 1625 | | |
| 87. Methyl ethyl ketone | | 1624 | | |
| 88. Naphthalene | 610 | 625, 1625 | | |
| 89. β-Naphthylamine | | 1625 | | |
| 90. Nitrobenzene | 609 | 625, 1625 | | |
| 91. 2-Nitrophenol | 604 | 625, 1625 | | |
| 92. 4-Nitrophenol | 604 | 625, 1625 | | |
| 93. N-Nitrosodimethylamine | 607 | [5] 625, 1625 | | |
| 94. N-Nitrosodi-n-propylamine | 607 | [5] 624, 1625 | | |
| 95. N-Nitrosodiphenylamine | 607 | [5] 625, 1625 | | |
| 96. n-Octacosane | | 1625 | | |
| 97. n-Octadecane | | 1625 | | |
| 98. 2,2-oxybis(1-chloropropane) | 611 | 625, 1625 | | |
| 99. PCB-1016 | 608 | 625 | | |
| 100. PCB-1221 | 608 | 625 | | |
| 101. PCB-1232 | 608 | 625 | | |
| 102. PCB-1242 | 608 | 625 | | |
| 103. PCB-1248 | 608 | 625 | | |
| 104. PCB-1254 | 608 | 625 | | |
| 105. PCB-1260 | 608 | 625 | | |
| 106. Pentachlorophenol | 604 | 625, 1625 | | |
| 107. Phenanthrene | 610 | 625, 1625 | 610 | |
| 108. Phenol | 604 | 625, 1625 | | |
| 109. α-Picoline | | 1625 | | |
| 110. Pyrene | 610 | 625, 1625 | 610 | |
| 111. Styrene | | 1625 | | |
| 112. α-Terpineol | | 1625 | | |
| 113. 2,3,7,8-Tetrachloro-dibenzo-p-dioxin (2,3,7,8-TCDD) | | 613 | | |
| 114. 1,1,2,2-Tetrachloroethane | 601 | 624, 1624 | | |
| 115. Tetrachloroethene | 601 | 624, 1624 | | |
| 116. n-tetracosane | | 1625 | | |
| 117. n-Tetradecane | | 1624 | | |
| 118. Toluene | 602 | 625, 1624 | | |
| 119. n-Triacontane | | 1625 | | |
| 120. 1,2,3-Trichlorobenzene | | 1625 | | |
| 121. 1,2,4-Trichlorobenzene | 612 | 625, 1625 | | |
| 122. 1,1,1-Trichloroethane | 601 | 624, 1624 | | |
| 123. 1,1,2-Trichloroethane | 601 | 624, 1624 | | |
| 124. Trichloroethene | 601 | 624, 1624 | | |
| 125. Trichlorofluoromethane | 601 | 624 | | |
| 126. 2,3,6-Trichlorophenol | | 1625 | | |
| 127. 2,4,5-Trichlorophenol | | 1625 | | |
| 128. 2,4,6-Trichlorophenol | 604 | 625, 1625 | | |
| 129. Vinyl Chloride | 601 | 624, 1624 | | |

[1] All parameters are expressed in micrograms per liter (μg/L).

[2] The full text of Methods 601–613, 624, 625, 1624, and 1625, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit," of this Part 136.

[3] "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978.

[4] Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds are Method 603 or Method 1624.

[5] Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625, are preferred methods for these compounds.

[6] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).

[7] Each analyst must make an initial, one-time, demonstration of his ability to generate acceptable precision and accuracy with Methods 601–613, 624, 625, 1624, and 1625 (See Appendix A of this Part 136) in accordanace with procedures given in section 8.2 of each of these Methods. Additionally, each laboratory, on an ongoing basis, must spike and analyze 10% (5% for Methods 624, 625, and 100% 1624, and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported for regulatory compliance purposes.

2. In § 136.3, Table ID is amended by withdrawing approval of the following cited test procedures for the following parameters: Aldrin, α–BHC γ–BHC (Lindane), Chlordane, 4,4'-DDD, 4,4'-DDE, 4,4'-DDT, Dieldrin, Endosulfan I, Endosulfan II, Endrin, Heptachlor, Heptachlor epoxide, and Toxaphene by the Gas Chromatography test procedures cited in, "Procedures for Pentachlorophenol, Chlorinated Organic Compounds, and Pesticides," Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio (1976). As proposed to be revised, § 136.3, Table ID would read as follows:

**§ 136.3 Identification of Test Procedures.**

* * * * *

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES [1]

| Parameter | Method | EPA [2, 3] | Standard methods 15th Ed | ASTM | Other |
|---|---|---|---|---|---|
| 1. Aldrin | GC | 608 | | | |
| | GC/MS | 625 | | | |
| 2. Ametryn | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 3. Aminocarb | TLC | | | | Note 3, p. 94; Note 6, p. S16. |
| 4. Atratpm | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 5. Atrazine | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 6. Azinphos methyl | GC | | | | Note 3, p. 25; Note 6, p. S51. |
| 7. Barban | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 8. α-BHC | GC | 608 | | | |
| | GC/MS | [5] 625 | | | |
| 9. β-BHC | GC | 608 | | | |
| | GC/MS | 625 | | | |
| 10. δ-BHC | GC | 608 | | | |
| | GC/MS | [5] 625 | | | |
| 11. γ-BHC (Lindane) | GC | 608 | | | |
| | GC/MS | 625 | | | |
| 12. Captan | GC | | 509A | | Note 3, p. 7. |
| 13. Carbaryl | TLC | | | | Note 3, p. 94; Note 6, p. S60. |
| 14. Carbophenothion | GC | | | | Note 4, p. 30; Note 6, p. S73. |
| 15. Chlordane | GC | 608 | | | |
| | GC/MS | 625 | | | |
| 16. Chloropropham | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 17. 2,4'-D | GC | | 509B | | Note 3, p. 115; Note 4, p. 35. |
| 18. 4,4'-DDD | GC | 608 | | | |
| 19. 4,4'-DDE | GC | 608 | | | |
| | GC/MS | 625 | | | |
| 20. 4,4'-DDT | GC | 608 | | | |
| | GC/MS | 625 | | | |
| 21. Demeton-O | GC | | | | Note 3, p. 25; Note 6, p. S51. |
| 22. Dementon-S | GC | | | | Note 3, p. 25; Note 6, p. S51. |
| 23. Diazinon | GC | | | | Note 3, p. 25; Note 4, p. 30; Note 6, p. S51. |
| 24. Dicamba | GC | | | | Note 3, p. 115. |
| 25. Dichlofenthion | GC | | | | Note 4, p. 30; Note 6, p. S73. |
| 26. Dichloran | GC | | 509A | | Note 3, p. 7. |
| 27. Dicofol | GC | | | D3086 | |
| 28. Dieldrin | GC | 608 | | | |
| 29. Dioxathion | GC | | | | Note 4, p. 30; Note 6, p. S73. |
| 30. Disulfoton | GC | | | | Note 3, p. 25; Note 6, p. S51. |
| 31. Diuron | TLC | | | | Note 3, p. 104. |
| 32. Endosulfan I | GC | 608 | | | |
| | GC/MS | [5] 625 | | | |
| 33. Endosulfan II | GC | 608 | | | |
| | GC/MS | [5] 625 | | | |
| 34. Endosulfan sulfate | GC | 608 | | | |
| | GC/MS | 625 | | | |
| 35. Endrin aldehyde | GC | 608 | | | |
| | GC/MS | 625 | | | |
| 37. Ethion | GC | | | | Note 4, p. 30. |
| 38. Fenuron | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 39. Fenuron-TCA | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 40. Heptachlor | GC | 608 | | | |
| | GC/MS | 625 | | | |
| 41. Heptachlor epoxide | GC | 608 | | | |
| | GC/MS | 625 | | | |
| 42. Isodrin | GC | | | | Note 4, p. 30; Note 6, p. S73. |
| 43. Linuron | TLC | | | | Note 3, p. 25; Note 6, p. S64. |
| 44. Malathion | GC | | 509A | | Note 3, p. 25; Note 4, p. 30; Note 6, p. S51. |
| 45. Methiocarb | TLC | | | | Note 3, p. 94; Note 6, p. S60. |
| 46. Methoxychlor | GC | | 509A | D3086 | Note 3, p. 7; Note 4, p. 30. |
| 47. Mexacarbate | TLC | | | | Note 3, p. 94; Note 6, p. S60. |
| 48. Mirex | GC | | 509A | | Note 3, p. 7. |
| 49. Monuron | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 50. Monuron-TCA | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 51. Neburon | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 52. Parathion methyl | GC | | 509A | | Note 3, p. 25; Note 4, p. 30. |
| 53. Parathion ethyl | GC | | 509A | | Note 3, p. 25. |
| 54. PCNB | GC | | 509A | | Note 3, p. 7. |
| 55. Perthane | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 56. Prometon | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 57. Prometryn | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 58. Propazine | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 59. Propham | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 60. Propoxur | TLC | | | | Note 3, p. 94; Note 6, p. S60. |
| 61. Secbumeton | TLC | | | | Note 3, p. 83; Note 6, p. S68. |
| 62. Siduron | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 63. Simazine | GC | | | | Note 3, p. 83; Note 6, p. S68. |
| 64. Strobane | GC | | 509A | | Note 3, p. 7. |
| 65. Swep | TLC | | | | Note 3, p. 104; Note 6, p. S64. |
| 66. 2,4,5-T | GC | | 509B | | Note 3, p. 115; Note 4, p. 35. |
| 67. 2,4,5-TP (Silvex) | GC | | 509B | | Note 3, p. 115. |
| 68. Terbuthylazine | GC | | | | Note 3, p. 83; Note 6, p. S68 69. |
| 69. Toxaphene | GC | 608 | | | |
| | GC/MS | 625 | | | |
| 70. Trifluralin | GC | | 509A | | Note 3, p. 7. |

[1] Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table IC, where entries are listed by chemical name.
[2] The full text of Methods 606 and 625, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B, "Definition and Procedure for the Determination of the Method Detection Limit", of this Part 136.
[3] "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods.

[4] "Methods for Analysis of Orgsnic Substances in Water," U.S. Geological Survey, Techniques of Water-Resources Investigations, Book 5, Chapter A3 (1972).
[5] The method may be extended to include α-BHC, δ-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.
[6] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency," Supplement to the Fifteenth Edition of *Standard Methods for the Examination of Water and Wastewater* (1981).
[7] Each analyst must make an initial, one-time, demonstration of his ability to generate acceptable precision and accuracy with Methods 608, and 625 (See Appendix A of this Part 136), in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an ongoing basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported for regulatory compliance purposes.

[FR Doc. 84-26349 Filed 10-23-84; 8:45 am]
BILLING CODE 6560-50-M

# Reader Aids

Federal Register

Vol. 49, No. 209

Friday, October 26, 1984

## INFORMATION AND ASSISTANCE

### SUBSCRIPTIONS AND ORDERS

| | |
|---|---|
| Subscriptions (public) | 202-783-3238 |
| Problems with subscriptions | 275-3054 |
| Subscriptions (Federal agencies) | 523-5240 |
| Single copies, back copies of FR | 783-3238 |
| Magnetic tapes of FR, CFR volumes | 275-2867 |
| Public laws (Slip laws) | 275-3030 |

### PUBLICATIONS AND SERVICES

**Daily Federal Register**

| | |
|---|---|
| General information, index, and finding aids | 523-5227 |
| Public inspection desk | 523-5215 |
| Corrections | 523-5237 |
| Document drafting information | 523-5237 |
| Legal staff | 523-4534 |
| Machine readable documents, specifications | 523-3408 |

**Code of Federal Regulations**

| | |
|---|---|
| General information, index, and finding aids | 523-5227 |
| Printing schedules and pricing information | 523-3419 |

**Laws**

| | |
|---|---|
| Indexes | 523-5282 |
| Law numbers and dates | 523-5282 |
| | 523-5266 |

**Presidential Documents**

| | |
|---|---|
| Executive orders and proclamations | 523-5230 |
| Public Papers of the President | 523-5230 |
| Weekly Compilation of Presidential Documents | 523-5230 |

**United States Government Manual** 523-5230

**Other Services**

| | |
|---|---|
| Library | 523-4986 |
| Privacy Act Compilation | 523-4534 |
| TDD for the deaf | 523-5229 |

## FEDERAL REGISTER PAGES AND DATES, OCTOBER


38525-38926 ..... 1
38927-39034 ..... 2
39035-39152 ..... 3
39153-39272 ..... 4
39273-39504 ..... 5
39505-39652 ..... 9
39653-39828 ..... 10
39829-39994 ..... 11
39995-40156 ..... 12
40157-40382 ..... 15
40383-40556 ..... 16
40557-40774 ..... 17
40775-41008 ..... 18
41009-41226 ..... 19
41227-42538 ..... 22
42539-42690 ..... 23
42691-42906 ..... 24
42907-43034 ..... 25
43035-43442 ..... 26


## PARTS AFFECTED DURING OCTOBER

At the end of each month, the Office of the Federal Register publishes separately a List of CFR Sections Affected (LSA), which lists parts and sections affected by documents published since the revision date of each title.


**1 CFR**

3 ..... 39511

**Proposed Rules:**

Ch. III ..... 40040

**3 CFR**

**Executive Orders:**

April 17, 1926
Revoked in part by PLO 6575 ..... 40407
5327 Revoked in part by PLO 6574 ..... 40406
6843 Revoked in part by PLO 6573 ..... 40406
11145 (See EO 12489) ..... 38927
11157 (Amended by EO 12488) ..... 38525
11183 (See EO 12489) ..... 38927
11287 (See EO 12489) ..... 38927
11776 (See EO 12489) ..... 38927
12131 (See EO 12489) ..... 38927
12190 (See EO 12489) ..... 38927
12196 (See EO 12489) ..... 38927
12216 (See EO 12489) ..... 38927
12296 (See EO 12489) ..... 38927
12345 (See EO 12489) ..... 38927
12367 (See EO 12489) ..... 38927
12395 (See EO 12489) ..... 38927
12488 ..... 38525
12489 ..... 38927
12490 ..... 40393

**Proclamations:**

5240 ..... 39273
5241 ..... 39275
5242 ..... 39277
5243 ..... 39279
5244 ..... 39281
5245 ..... 39283
5246 ..... 39285
5247 ..... 39505
5248 ..... 39507
5249 ..... 39509
5250 ..... 39653
5251 ..... 39655
5252 ..... 39829
5253 ..... 39831
5254 ..... 39833
5255 ..... 40383
5256 ..... 40385
5257 ..... 40387
5258 ..... 40389
5259 ..... 40391
5260 ..... 40557
5261 ..... 40775
5262 ..... 41227
5263 ..... 41229
5264 ..... 41231
5265 ..... 42539
5266 ..... 42541
5267 ..... 42543
5268 ..... 42545
5269 ..... 42547

**4 CFR**

81 ..... 38527

**5 CFR**

315 ..... 39287

**7 CFR**

2 ..... 38929
53 ..... 43045
272 ..... 39035
273 ..... 39035
301 ..... 38930, 40157, 42691
319 ..... 39038
355 ..... 42907
356 ..... 42907
371 ..... 38529, 41009
427 ..... 40777
431 ..... 41009
432 ..... 40781
444 ..... 39512
651 ..... 40395
910 ..... 39288, 39995, 41014, 43039
920 ..... 39657
928 ..... 40559
966 ..... 39153, 40397
981 ..... 38530, 40788
985 ..... 42549
1065 ..... 39995
1079 ..... 40397
1097 ..... 39835
1435 ..... 38532
1910 ..... 40789
1944 ..... 40789
1951 ..... 41220
1980 ..... 41223
3015 ..... 38533

**Proposed Rules:**

58 ..... 39166
443 ..... 41064
800 ..... 40582, 40583
910 ..... 40182
985 ..... 39166
1004 ..... 42737
1030 ..... 40183
1434 ..... 39167

**8 CFR**

103.....41014
214.....39663, 41014
292a.....41014

**Proposed Rules:**

248.....39685

**9 CFR**

78.....39836
81.....39517
92.....41015
201.....39516
203.....39516

**Proposed Rules:**

93.....41257
113.....39853
319.....39560
327.....39560, 42740

**10 CFR**

2.....38534
50.....38534, 42693
51.....42693
55.....42693
1035.....41233

**Proposed Rules:**

7.....43070
20.....38643
34.....39168
50.....39066

**11 CFR**

4.....41016
5.....41016

**12 CFR**

205.....40794
226.....40560
407.....41237
523.....43040
541.....43040
543.....41237
544.....41237
545.....43040
549.....43040
552.....41237
561.....43040
562.....41237
563.....38924, 43040
564.....38924
584.....43040
615.....39518
571.....41237
741.....40561, 42918
746.....40561

**Proposed Rules:**

5.....38954, 39066
563.....40584
564.....40584

**13 CFR**

Ch. IV.....42696
102.....40564
121.....39996, 40398
122.....39837
123.....39268
140.....38931

**Proposed Rules:**

121.....40877

**14 CFR**

21.....39650
25.....43182, 43188
29.....43188
39.....38534–38537, 38931, 39288–39291, 39997, 40799, 40800, 42556, 42696, 42918, 42919, 43046
71.....38538, 38539, 40158, 40400, 42557–42559, 42919
73.....42919
75.....39291, 40801
91.....38933
95.....39998
97.....40003, 42920
121.....43182, 43188
221.....40004
234.....40565
250.....40401
255.....40401
291.....40401
399.....40567

**Proposed Rules:**

Ch. I.....39336, 42943, 43020
21.....40154, 40184
23.....40186
25.....40041
39.....38643, 39336, 39565, 40042, 42742, 42944
45.....40154
71.....38644, 39686, 40042, 40187, 40878, 42575, 42740, 42945–42947
73.....42740
302.....39337
375.....42948
389.....39337
398.....40043
399.....39337, 40043, 40588

**15 CFR**

Ch. XIV.....42697
373.....40568
399.....40568

**Proposed Rules:**

371.....39154
373.....38955, 39154
376.....38955
390.....39154
399.....39154

**16 CFR**

13.....39040, 39159, 39839, 39840, 41017, 42699
305.....43047
1010.....39663
1030.....39292
1602.....39669
1632.....39790

**Proposed Rules:**

13.....39070

**17 CFR**

1.....39518, 40159, 43048
3.....39518
16.....40159, 43048
33.....40005
140.....39518
145.....39518
239.....40569
240.....40573
241.....40159
270.....40569
274.....40569

**Proposed Rules:**

210.....38956
229.....38956
240.....40188, 40416
270.....40879

**18 CFR**

2.....39293, 40802
35.....39293
154.....39042, 39293, 40802
201.....39293, 40802
270.....39293, 40802
271.....39293, 39535, 40802
294.....39536
301.....39293
341.....38540
342.....38540
343.....38540
344.....38540
345.....38540
346.....38540
347.....38540
385.....39538
389.....38934, 39293

**Proposed Rules:**

154.....38956
271.....39032

**19 CFR**

4.....41152, 43049
6.....41152
7.....41152
10.....39043, 41152
11.....41152
12.....41152
18.....39044, 41152
19.....41152
24.....41152
54.....41152
101.....41152
112.....41152
113.....41152
114.....41152
123.....39044, 41152
125.....41152
127.....41152
132.....41152
133.....41152
134.....40802, 41152
141.....41152
142.....41152
144.....39044, 41152
145.....41152
146.....41152
147.....41152
148.....41152
151.....41152
162.....41152
171.....39047
172.....39048, 41152
174.....41152
191.....41152

**Proposed Rules:**

Ch. I.....39854
4.....39072
6.....39075
19.....39077
24.....39075
151.....40882
158.....42576

**20 CFR**

404.....38935, 41244
618.....41017
621.....41018
632.....42559
655.....41018

**Proposed Rules:**

Ch. V.....39686

**21 CFR**

Ch. I.....41019
73.....42922
81.....38935, 38936
176.....40402
177.....43050
193.....40575
430.....39670
436.....39670, 40006
440.....39670
442.....40006
455.....39670
510.....39539
520.....43051
522.....38937, 38938
558.....39539, 39842
561.....42700
866.....40380
1308.....39307

**Proposed Rules:**

201.....40188
808.....38645, 38646

**22 CFR**

Ch. VIII.....40804
51.....40403
305.....38939

**23 CFR**

625.....38940
660.....40006

**Proposed Rules:**

Ch. III.....38648
658.....38956

**24 CFR**

17.....39675
115.....39676
251.....38943
571.....42701
905.....42701

**Proposed Rules:**

105.....40528
200.....39855, 41212
203.....39686, 40188, 41212
204.....41212
207.....39688, 39690, 41068
220.....39690, 41068, 41212
221.....39690, 41068, 41212
226.....39686
231.....41068
234.....39686
235.....41212
237.....41212
241.....40044
242.....40044, 40047
246.....39690
255.....39690
290.....40888
570.....39693
882.....41072
886.....40888
941.....39694

**25 CFR**

5.....39157
244.....39308

**26 CFR**

1.....39051, 39314, 39540, 39677, 40011, 40016, 41246, 42701–42715, 43052
5f.....39677
18.....38920

25.....38540, 39843
35a.....40403
41.....39544
51.....42922
150.....40804
601.....40808

**Proposed Rules:**
1.....39078, 39344, 39571, 41261, 42743, 42744
51.....40896
301.....39566, 42745

**27 CFR**

9.....42563, 42719, 43053
19.....42567
240.....42567
285.....41020

**Proposed Rules:**
4.....42577

**28 CFR**

0.....39843, 41246
2.....40403
545.....38914

**29 CFR**

1601.....39053
2619.....40161
2705.....38542

**30 CFR**

210.....40576
212.....40576
217.....40576
218.....40576
219.....40576
228.....40576
229.....40024, 40576
241.....40576
243.....40576
914.....41020
917.....39053
925.....43055
935.....41024
938.....40028
942.....38874

**Proposed Rules:**
250.....41077
779.....38958
780.....38958
783.....38958
784.....38958
816.....38958
817.....38958
906.....38653
917.....41262
926.....43071
935.....42745
944.....40421
950.....42579

**32 CFR**

199.....42569, 42570
242b.....42927
544.....39946

**Proposed Rules:**
505.....40589

**33 CFR**

100.....40403
110.....40809
117.....39157, 40404
165.....40405, 40810
181.....39327
183.....39327
320.....39478
323.....39478
325.....39478
330.....39478, 39843

**Proposed Rules:**
100.....38654, 38655
117.....38656, 40422, 40423 40900, 41264

**34 CFR**

760.....43226

**Proposed Rules:**
221.....40362
361.....38656
362.....38656
366.....38656
369.....38656
373.....38656
379.....38656
385.....38656
386.....38656
389.....38656

**35 CFR**

253.....41025

**Proposed Rules:**
121.....38660

**36 CFR**

50.....39677

**37 CFR**

**Proposed Rules:**
Ch. II.....39171
201.....39174

**38 CFR**

5.....39328
8.....39328
13.....39328
17.....39328
21.....39544, 40810, 42725
36.....42570, 43055

**Proposed Rules:**
21.....39572

**39 CFR**

10.....38543
447.....40768
601.....40858
956.....40768

**Proposed Rules:**
10.....39573, 41265
111.....38661

**40 CFR**

30.....36943
45.....41004
46.....41006
51.....43202
52.....39057-39062, 39545-39547, 39843, 40029, 40162, 40164, 41026-41029, 43072, 43202
60.....40031, 41030
61.....38946
62.....43057
81.....41029
87.....41000
123.....39063, 42572
133.....40405
136.....43234
147.....42727
158.....42856
180.....42728-42733
233.....38947
271.....39328, 39683, 41036, 41038
721.....42928, 43058-43061
790.....39774
799.....39810, 42932

**Proposed Rules:**
Ch. I.....39696
51.....43211
52.....38962, 39574-39582, 39696, 39866, 39869, 40051, 40052, 40607, 42670, 42746-42749, 42957, 42958, 43211
60.....40542
65.....38963, 39080, 39583-39587
81.....40053, 40424, 40425, 42750
86.....40258
122.....38812, 39697
136.....43437
180.....39698, 40608, 42752-42758
232.....39012
233.....39012
260.....38786
261.....38786, 42580
264.....38786
265.....38786
270.....38786
271.....38671, 38786, 39175, 40055, 40610, 42759-42761, 42959, 43072
300.....40320
406.....40611
407.....40611
408.....40611
409.....40611
411.....40611
422.....40611
424.....40611
426.....40611
430.....40611
431.....40611
432.....40611
439.....40611
721.....39703, 42762, 42960
761.....39966

**41 CFR**

Ch. 201.....39159
51-7.....43065
51-8.....43065
101-41.....42932
105-61.....42933
201-1.....38948
201-4.....38948
201-35.....38948
201-36.....38948

**Proposed Rules:**
101-11.....41265

**42 CFR**

57.....40406
405.....40167

**43 CFR**

3100.....39329
3200.....39329
3470.....39329
3500.....39329

**Public Land Orders:**
4690.....42934
6572.....40031
6573.....40406
6574.....40406
6575.....40407
6576.....42934

**Proposed Rules:**
2650.....41266
3160.....40354

**44 CFR**

64.....40552, 42572
67.....39684
150.....39844

**Proposed Rules:**
67.....39176, 40901, 43074

**45 CFR**

3.....39160

**Proposed Rules:**
90.....43074
205.....39488
305.....39488
1180.....39346

**46 CFR**

33.....40407
35.....40407
75.....40407
78.....40407
94.....40407
97.....40407
107.....39161
108.....39161
109.....39161
161.....40407
167.....40407
180.....40407
185.....40407
192.....40407
196.....40407
381.....39847
510.....38544
515.....38544
550.....38836, 42934
552.....38836, 42934
553.....38836, 42934
555.....38836, 42934
558.....38836, 42934
559.....38836, 42934
560.....38836, 42934
561.....38836, 42934
562.....38836, 42934
564.....38836, 42934
566.....38836, 42934
568.....38836, 42934
569.....38836, 42934

**Proposed Rules:**
Subchapter D (30-40).....38672
Subchapter H (70-89).....38672
Subchapter I (90-106).....38672
550.....40940
580.....40940

**47 CFR**

0.....42935
1.....40168, 40858
2.....39330, 40410, 41247
13.....39064, 40170
61.....40858
73.....38544-38548, 39064, 40032, 41249, 42936
78.....42938

81.....40170, 40410
83.....40170, 40172, 40410, 41249
87.....39330
90.....40175, 40177, 41247
97.....40414

**Proposed Rules:**

Ch. I.....39081
2.....39082, 40611
15.....42593, 42594
67.....40192
68.....39349
73.....38673-38679, 39352, 42964
81.....40193
90.....39082, 42594
97.....40194, 40611

**48 CFR**

Ch. 2.....38549, 38599, 38605
Ch. 5.....39335, 40576
Ch. 9.....38949, 42938
Ch. 18.....41038
501.....40032
504.....40577

**Proposed Rules:**

Ch. 5.....39872, 40615
5.....38680, 39151
6.....38680, 39151
14.....38680, 39151
15.....38680, 39151

**49 CFR**

1.....38609
173.....40033, 42733
177.....42733
178.....40033, 42733
179.....42733
533.....41250
571.....38610, 39335
574.....38610, 39335
575.....38610, 39335
700.....40036
1002.....39548, 43065
1011.....43065
1033.....39162
1103.....38613
1152.....43065
1177.....43065
1180.....43065
1182.....43065
1300.....38540, 38614
1303.....38614
1304.....38614
1305.....38614
1306.....38614
1307.....38614
1308.....38614
1309.....38614
1310.....38614
1312.....38614, 40415

**Proposed Rules:**

Ch. VI.....40426
107.....40056
173.....39177
571.....39872, 42965
701.....40057
1152.....39085

**50 CFR**

17.....40036, 43065
20.....40038
24.....42938
32.....38642
285.....38641, 38642, 39684, 39851, 40415, 40580
424.....38900
630.....40415
638.....40415
641.....39548, 41063
650.....39065
652.....39558, 40580
654.....39162
655.....41063
658.....39162

**Proposed Rules:**

13.....42594
17.....39179, 39353, 39873, 40058, 40196, 41266, 42594, 43076
611.....40615
630.....40621, 43076

## LIST OF PUBLIC LAWS

**Note:** No public bills which have become law were received by the Office of the Federal Register for inclusion in today's **List of Public Laws.**

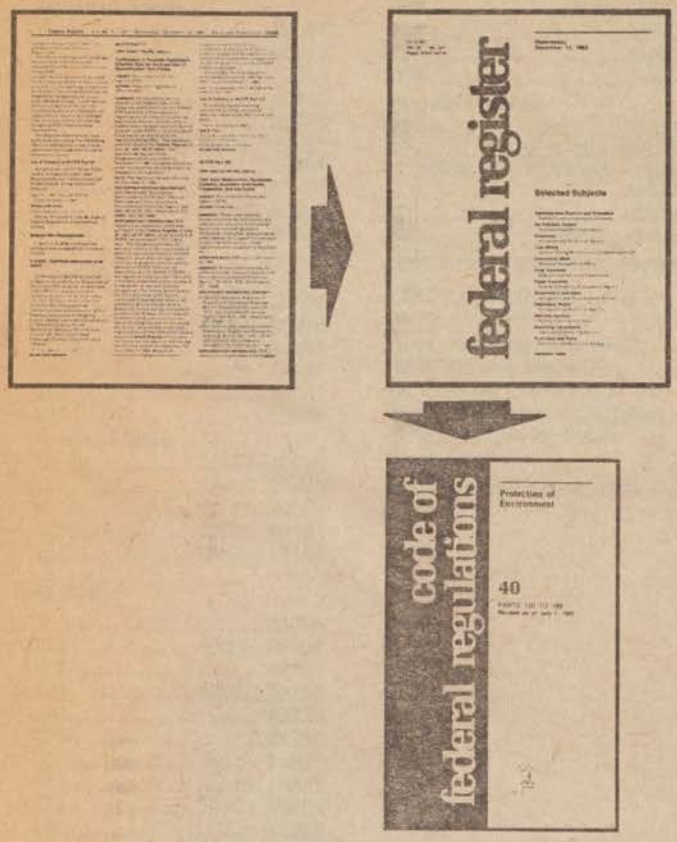
**Last List October 16, 1984**





# The Federal Register

**Regulations** appear as agency documents which are published daily in the **Federal Register** and codified annually in the **Code of Federal Regulations**




The **Federal Register**, published daily, is the official publication for notifying the public of proposed and final regulations. It is the tool for you to use to participate in the rulemaking process by commenting on the proposed regulations. And it keeps you up to date on the Federal regulations currently in effect.

Mailed monthly as part of a **Federal Register** subscription are: the LSA (List of CFR Sections Affected) which leads users of the **Code of Federal Regulations** to amendatory actions published in the daily **Federal Register**; and the cumulative **Federal Register Index.**

The **Code of Federal Regulations** (CFR) comprising approximately 180 volumes contains the annual codification of the final regulations printed in the **Federal Register.** Each of the 50 titles is updated annually.

**Subscription Prices:**

- **Federal Register**
  - **Paper:**
    - One year as issued: $300 domestic; $375 foreign
    - Six months: $150 domestic; $187.50 foreign
  - **24x Microfiche Format:**
    - One year as issued: $175 domestic; $218.75 foreign
    - Six months: $87.50 domestic; $109.40 foreign
- **Code of Federal Regulations**
  - **Paper:**
    - One year as issued: $550.00 domestic; $687.50 foreign
    - Single volumes: Individually priced.
  - **24x Microfiche Format:**
    - Current year (as issued): $200 domestic; $250 foreign
    - Previous year's full set (single shipment): $155 domestic; $193.75 foreign

## Order Form

Mail To: Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402

Enclosed is $_____ ☐ check, ☐ money order, or charge to my Deposit Account No.

☐☐☐☐☐☐☐–☐

Order No._____

**MasterCard and VISA accepted.**

MasterCard VISA

**Credit Card Orders Only**

Total charges $_____

Fill in the boxes below.

Credit Card No. ☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐☐

Expiration Date Month/Year ☐☐☐☐

Customer's Telephone No.'s

Area Code Home Area Code Office

Charge orders may be telephoned to the GPO order desk at (202)783-3238 from 8:00 a.m. to 4:00 p.m. eastern time, Monday-Friday (except holidays).

**Please send me**

**PAPER:**

_____ **Federal Register:** ___ $300 per year domestic; $375 foreign ___ $150 per six-month domestic; $187.50 foreign

_____ **Code of Federal Regulations:** ___ $550 per year domestic; $687.50 foreign

**24x MICROFICHE FORMAT:**

_____ **Federal Register:** ___ One year as issued: $175 domestic; $218.75 foreign ___ Six months: $87.50 domestic; $109.40 foreign

_____ **Code of Federal Regulations:** ___ Current year: $200 domestic; $250 foreign ___ Previous year's full set (single shipment): $155 domestic; $193.75 foreign

**PLEASE PRINT OR TYPE**

Company or Personal Name

Additional address/attention line

Street address

City State ZIP Code

(or Country)

**For Office Use Only**

Quantity Charges

Publications
Subscription
Special Shipping Charges
International Handling
Special Charges
OPNR
UPNS
Balance Due
Discount
Refund