

m³, the State's modeling shows a design 24-hour average concentration of 148.7 µg/m³, and USEPA's supplemental modeling shows a design concentration of 149.9 µg/m³. As compared to the annual average standard of 50 µg/m³, Ohio's modeling shows a highest concentration of 49.6 µg/m³, and USEPA's supplemental modeling also shows a highest concentration of 49.6 µg/m³. Thus, with or without consideration of the minor inventory issues, Ohio's plan may be judged to assure attainment of the air quality standards in the Steubenville area.

IV. Today's Action

With respect to Cuyahoga County, USEPA proposes to conclude that the revised rules now provide for RACT by December 1993, that the quench water test method issue and the associated attainment demonstration issue has been resolved, and that the further revisions to the limitation for Ford's Cleveland Casting Plant do not jeopardize attainment. With respect to the Steubenville area, USEPA proposes to conclude that the State has now submitted a fully approvable attainment demonstration for the area. USEPA also proposes in particular to approve the rule revisions for Cuyahoga County and the findings and order requiring control system enhancements at Wheeling-Pittsburgh Steel's basic oxygen furnace.

Based on the above proposed findings, USEPA proposes further to conclude that Ohio's particulate matter plans for the Cuyahoga County and Steubenville nonattainment areas now satisfy all applicable requirements under Part D of the Clean Air Act (except for new source review requirements, which are not addressed here or in the May 1994 rulemaking and are being addressed separately). More specifically, USEPA proposes to find that the deficiencies identified in the May 1994 rulemaking have been remedied. USEPA is publishing this finding as an interim final determination in a separate notice in the Rules section of this Federal Register issue. As a result, the sanctions which were to take effect December 27, 1995, are deferred and shall not be applied pending further rulemaking on these issues. If USEPA's final action finalizes the approval action proposed today, the sanctions clock shall be fully stopped. Only if USEPA publishes proposed or final disapproval action concluding that some deficiencies have not been remedied would sanctions be applied.

Under the Regulatory Flexibility Act, 5 U.S.C. 600 et seq., USEPA must prepare a regulatory flexibility analysis assessing the impact of any proposed or

final rule on small entities. (5 U.S.C. 603 and 604.) Alternatively, USEPA may certify that the rule will not have a significant impact on a substantial number of small entities. Small entities include small businesses, small not-for-profit enterprises, and government entities with jurisdiction over populations of less than 50,000.

SIP approvals under section 110 and subchapter I, part D of the Act do not create any new requirements, but simply approve requirements that the State is already imposing. Therefore, because the federal SIP approval does not impose any new requirements, I certify that it does not have a significant impact on any small entities affected. Moreover, due to the nature of the Federal-State relationship under the Act, preparation of a regulatory flexibility analysis would constitute federal inquiry into the economic reasonableness of state action. The Act forbids USEPA to base its actions concerning SIPs on such grounds. *Union Electric Co. v. USEPA*, 427 U.S. 246, 256-66 (1976); 42 U.S.C. 7410(a)(2).

Nothing in this action should be construed as permitting or allowing or establishing a precedent for any future request for revision to any SIP. Each request for revision to the SIP shall be considered separately in light of specific technical, economic, and environmental factors and in relation to relevant statutory and regulatory requirements.

This action has been classified as a Table 3 action by the Regional Administrator under the procedures published in the Federal Register on January 19, 1989 (54 FR 2214-2225), as revised by an October 4, 1993 memorandum from Michael H. Shapiro, Acting Assistant Administrator for Air and Radiation. The Office of Management and Budget exempted this regulatory action from Executive Order 12866 review.

Under Sections 202, 203 and 205 of the Unfunded Mandates Reform Act of 1995 ("Unfunded Mandates Act"), signed into law on March 22, 1995, EPA must undertake various actions in association with proposed or final rules that include a Federal mandate that may result in estimated costs of \$100 million or more to the private sector, or to State, local, or tribal governments in the aggregate.

EPA has determined that the approval action proposed today does not include a Federal mandate that may result in estimated costs of \$100 million or more to either State, local, or tribal governments in the aggregate, or to the private sector. This Federal action proposes to approve pre-existing

requirements under State or local law, and imposes no new Federal requirements. Accordingly, no additional costs to State, local, or tribal governments, or to the private sector, result from this action.

List of Subjects in 40 CFR Part 52

Air Pollution control, Environmental protection, Intergovernmental relations, Particulate matter, Reporting and recordkeeping requirements.

Dated: December 13, 1995.

Gail C. Ginsburg,

Acting Regional Administrator.

[FR Doc. 96-876 Filed 1-22-96; 8:45 am]

BILLING CODE 6560-50-P

40 CFR Part 136

[FRL-5401-7]

Guidelines Establishing Test Procedures for the Analysis of Oil and Grease and Total Petroleum Hydrocarbons

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed rule.

SUMMARY: This proposed regulation would amend the Guidelines Establishing Test Procedures for the Analysis of Pollutants under section 304(h) of the Clean Water Act to replace existing gravimetric test procedures for the conventional pollutant "oil and grease" (40 CFR 401.16) with EPA Method 1664 as part of EPA's effort to reduce dependency on the use of chlorofluorocarbons (CFCs). Method 1664 uses normal hexane (n-hexane) as the extraction solvent in place of 1,1,2-trichloro-1,2,2-trifluoroethane (CFC-113; Freon-113). CFC-113 is used in currently approved 40 CFR Part 136 methods for the determination of oil and grease. These methods are EPA Method 413.1 in Methods for Chemical Analysis of Water and Wastes (EPA-600/4-79-020) and Method 5520B in Standard Methods for the Examination of Water and Wastewater, 18th edition. This proposal would withdraw approval of Methods 413.1 and 5520B to preclude the unacceptable inconsistency between results produced by such methods and proposed Method 1664. In an effort to provide for the use and depletion of existing laboratory stocks of Freon-113, EPA plans to implement the required use of Method 1664 no sooner than six months after the final rule is published in the Federal Register. Method 1664 is also being proposed for the determination of total petroleum hydrocarbons.

DATES: Comments on this proposal must be submitted on or before March 25, 1996.

ADDRESSES: Send written comments on the proposed rule to "Method 1664" Comment Clerk; Water Docket MC-4101; Environmental Protection Agency; 401 M Street, SW.; Washington, DC 20460. Commenters are requested to submit any references cited in their comments. Commenters are also requested to submit an original and 3 copies of their written comments and enclosures. Commenters who want receipt of their comments acknowledged should include a self addressed, stamped envelope. All comments must be postmarked or delivered by hand by March 25, 1996. No facsimiles (faxes) will be accepted.

Data available: A copy of the comments and supporting documents cited in this proposal are available for review at EPA's Water Docket; 401 M Street, S.W., Washington, DC 20460. For access to the Docket materials, call (202) 260-3027 between 9 a.m. and 3:30 p.m. for an appointment.

Electronic versions of Method 1664 will be available via the Internet. EPA operates a "public access server," also known as "Earth 1," through which EPA will include all of the ways that copies of the test method manual are available. The Office of Water will put the directions about electronic retrieval of the test method manual on EPA's Internet "homepage." By doing so, persons interested in electronic copies of the method may obtain copies by either (1) retrieving the documents from EPA's file transfer protocol (FTP) site on the Internet at ftp.epa.gov or gopher.epa.gov (2) retrieving the documents by dial-in access at 919-558-0335.

FOR FURTHER INFORMATION CONTACT: Mr. Ben Honaker, Engineering and Analysis Division (4303), USEPA Office of Science and Technology, 401 M Street, S.W., Washington, DC, 20460, or call (202) 260-2272.

SUPPLEMENTARY INFORMATION:

I. Authority

A. *Clean Water Act*

Today's proposal is pursuant to the authority of sections 301, 304(h), and 501(a) of the Clean Water Act (CWA), 33 U.S.C. 1314(h), 1361(a) (the "Act"). Section 301 of the Act prohibits the discharge of any pollutant into navigable waters unless the discharge complies with an NPDES permit, issued under section 402 of the Act. Section 304(h) of the Act requires the Administrator of the EPA 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 this Act or permit applications pursuant to section 402 of this Act." Section 501(a) of the Act authorizes the Administrator to "prescribe such regulations as are necessary to carry out his function under this Act."

The Administrator also has made these test methods applicable to monitoring and reporting of NPDES permits (40 CFR part 122, §§ 122.21, 122.41, 122.44, and 123.25), and implementation of the pretreatment standards issued under section 307 of the Act (40 CFR part 403, §§ 403.10 and 402.12).

B. *Clean Air Act Amendments of 1990*

Today's proposal is also consistent with sections 604, 606 and 608 of the 1990 Clean Air Act Amendments (CAAA) to phase out production of Class I CFCs and reduce use and emissions of Class I CFCs to the lowest achievable level, and with section 613 of the CAAA to reduce the Federal procurement of products and services that employ Class I CFCs.

II. Background and History

A. *Oil and Grease and Petroleum Hydrocarbons Testing*

The background and history of the applicability of EPA's Stratospheric Ozone Protection Program to analytical methods requiring the use of CFCs in EPA regulatory programs was given in an earlier proposal of an alternate test procedure for the determination of oil and grease (56 FR 30519-30520, July 3, 1991).

As stated in the earlier proposal, preliminary efforts to find a suitable replacement solvent for Freon-113 in the determination of oil and grease were conducted by the Office of Research and Development's Environmental Monitoring Systems Laboratory in Cincinnati, Ohio (EMSL-Ci). Results of that study, presented in the document titled A Study to Select a Suitable Replacement Solvent for Freon-113 in the Gravimetric Determination of Oil and Grease by F.K. Kawahara, October 2, 1991, suggested the use of an 80/20 mixture of n-hexane and methyl tertiary butyl ether (MTBE) in place of Freon-113 for oil and grease determination. This led to the proposal (56 FR 30519-30524, July 3, 1991) to replace Freon-113 with the n-hexane:MTBE mixture in CWA and RCRA analytical methods for determination of oil and grease. Based on comments received concerning this

proposal, the EMSL-Ci study results, and the need to further investigate alternative solvents, the Office of Water and the Office of Solid Waste initiated a multi-phase Freon Replacement Study.

1. Phase I of the Freon Replacement Study

Phase I of the Freon Replacement Study evaluated alternative solvents and extraction systems for equivalency to the results produced by Freon-113 across a range of real world effluent and solid waste samples from a variety of industrial categories. More specifically, Phase I focused on (1) the use of five alternative solvents for gravimetric determination of oil and grease in aqueous samples by MCAWW Method 413.1 (with modifications) and in solid samples by SW-846 Method 9071A (with modifications) and (2) the use of alternative techniques for oil and grease analysis including sonication extraction, solid phase extraction (SPE) using cartridges and disks, and a solvent/non-dispersive infrared technique. The five alternative solvents studied in Phase I were n-hexane, methylene chloride (dichloromethane), perchloroethylene (tetrachloroethene), DuPont 123 (2,2-dichloro-1,1,1-trifluoroethane, a hydrofluorochlorocarbon), and the n-hexane:MTBE 80:20 mixture.

Results of the tests of gravimetric procedures in Phase I yielded the following conclusions: n-hexane should be retained as a possible extraction solvent for further study using gravimetric techniques; perchloroethylene should be retained for consideration in the use of infra-red techniques; and cyclohexane should be introduced for consideration with gravimetric techniques based on its similarity to n-hexane and because of its lower neurotoxicity when compared to n-hexane.

Results of tests of alternative techniques in Phase I indicated that only sonication extraction of soil and other solids-containing samples produced results equivalent to existing techniques that use Freon-113. Specifics of the study design, results, and conclusions can be found in the Preliminary Report of EPA Efforts to Replace Freon for the Determination of Oil and Grease, September 1993, that is included in the docket.

Prior to the commencement of Phase II of the study, two public workshops were held, one on May 4, 1993, in Norfolk, VA and the other on June 30, 1993, in Boston, MA. The objectives of these workshops were to inform interested parties of the results from Phase I and related studies, and to

provide a forum for the discussion of results, issues, and possible solutions to the problem of finding a non-ozone-depleting substance that would produce results identical to the results produced by CFC-113. Detailed records of the information presented, which included data from EPA, vendor, and laboratory representatives, as well as the proceedings of the question and answer sessions, can be found in the reports entitled Oil and Grease Workshop, Norfolk, Virginia, May 4, 1993 and Oil and Grease Workshop, Boston, Massachusetts, June 30, 1993 that are also included in the docket for today's proposed rule.

2. Phase II of the Freon Replacement Study

Based on the conclusions of Phase I of the Freon Replacement Study, Phase II was designed to further assess the use of n-hexane as a replacement solvent and to evaluate the use of cyclohexane as a replacement solvent for oil and grease determination in aqueous samples by MCAWW Method 413.1, with modifications to allow for solvent densities less than the density of water. In addition, gravimetric determination of "petroleum hydrocarbons" was evaluated by subjecting the extracted oil and grease samples to the silica gel adsorption procedure in Standard Methods for the Examination of Water and Wastewater (Standard Methods) 5520F (with modifications). This procedure removes non-aliphatic hydrocarbons such as detergents, surfactants, fatty acids, aromatic hydrocarbons, heterocyclics, and some chemical compounds containing nitrogen, phosphorus, and sulfur.

The final objectives of the Phase II study were to utilize these results and comments to choose a replacement solvent and to document the analytical protocol implemented to produce the first draft of Method 1664 (the "Method") for gravimetric determination of oil and grease and petroleum hydrocarbons. Following the formal documentation of the Method, a further objective was to initiate an interlaboratory study using the new method in order to characterize the method and generate method specifications in the form of quality control (QC) acceptance criteria.

A total of 34 samples from a combination of in-process and effluent waste streams were collected from 25 facilities encompassing 16 different industrial categories. Samples containing between 40–300 mg/L oil and grease, some from petroleum and some from non-petroleum sources, were collected. The study focused on this

concentration range to avoid the problems associated with the comparison and evaluation of "non-detect" results. In order to increase the types of matrices that could be analyzed using the Method and better assess the effect of different matrices on solvent extraction performance, the Agency collected samples from a variety of industrial facilities that were different from those collected during Phase I of the study.

Analysis of each sample was performed in triplicate for each of the three extraction solvents. Prior to the analysis of field samples, the evaluating laboratory was required to demonstrate its ability to generate acceptable accuracy and precision for each of the required procedures by performing a series of initial precision and recovery (IPR) analyses for determination of oil and grease and petroleum hydrocarbons using Freon-113, n-hexane, and cyclohexane as extraction solvents. IPR analyses involved extraction, concentration, and analysis of a set of four 1-L aliquots of reagent water that had been spiked with hexadecane and stearic acid. All IPR analyses included modifications necessary to allow for differences in solvent densities and other modifications necessary to apply Method 413.1 and Standard Method 5520F to the alternative solvents, in order to evaluate any effects that might result from the modified procedures.

An ongoing precision and recovery (OPR) analysis, the equivalent of a single IPR sample, was required with each analytical batch for each alternative solvent. An analytical batch consisted of a set of samples extracted at the same time, to a maximum of ten samples.

In addition, a reagent water method blank was analyzed with each IPR set and with each sample batch for each alternative solvent. These reagent water blanks were run through the same extraction and analysis procedure through which the samples were run. The analytical protocol required that the concentration of oil and grease in method blanks not exceed 5 mg/L and, if contamination above this level was detected in any method blank, the laboratory was required to isolate the source of contamination and reanalyze associated samples.

Multiple aliquots of each sample were collected in order to accommodate the numerous analyses required. The multiple aliquots were split from a homogenized sample and, to the extent practicable, contained identical concentrations of oil and grease. Within each of the three different solvent procedures and two modified methods

(413.1 and 5520F), it was expected that the relative standard deviation (RSD) of the triplicate measurements would be less than or equal to 10 percent for those results at or above 25 mg/L and less than or equal to 20 percent for those results less than 25 mg/L. The evaluating laboratory was required to notify EPA if the triplicate results exceeded these RSDs.

Addition of these QC requirements and data quality objectives to the usual method protocol, and careful monitoring of the analytical techniques ensured that reliable data were produced.

Alternative analytical techniques were also evaluated. These techniques were performed voluntarily by manufacturers of various devices on splits of samples collected as part of Phase II. EPA supplied additional volumes of each sample collected during the Phase II study to a number of vendors that tested alternative oil and grease extraction and measurement techniques similar to those tested in the Phase I study. These included solid phase extraction using cartridges and disks, non-dispersive infrared spectroscopy, and immunoassay.

Evaluation of Phase II data led to the conclusion that the results generated when using n-hexane and cyclohexane were statistically equivalent to one another, but that these results were significantly different from results generated when using Freon-113 as the extraction solvent. These findings were consistent with the Phase I study conclusion that, when all sample matrices are collectively considered, none of the solvents that were evaluated was statistically equivalent to Freon-113. Specifics of the study design, results, and conclusions are included in the Report of EPA Efforts to Replace Freon for the Determination of Oil and Grease and Total Petroleum Hydrocarbons: Phase II, April 1995, that is included in the docket.

The decision of which alternative solvent was best suited for the new method was therefore based on logistical analytical considerations, of which the primary factor was the difference between the boiling points of n-hexane (69 °C) and cyclohexane (81 °C). Based on laboratory comments regarding the extensive amount of time required to evaporate cyclohexane, n-hexane was determined to be a more suitable replacement for Freon-113.

Evaluation of vendor data was limited to the SPE disk and column extraction techniques with gravimetric determination, and demonstrated that results from both of the SPE techniques were not statistically equivalent to

results produced by either Freon-113 or n-hexane using separatory funnel extraction and gravimetric determination.

Results from the non-dispersive infrared and immunoassay analyses were not considered for this proposal because they represent completely different determinative techniques. EPA is, however, planning to further evaluate these techniques and consider other promising procedures in subsequent studies.

Though written as a separatory funnel extraction procedure, Method 1664 allows the use of alternative extraction and concentration techniques, such as SPE, under the performance-based option, provided that these techniques produce results that meet the specifications in Method 1664 when tested using reference standards and, when used for compliance monitoring, produce results equivalent to results produced by Method 1664 on the specific discharge to which they are to be applied.

The Agency solicits additional comparative data and information on SPE techniques and other alternative extraction and concentration techniques, and on the proposal to allow these techniques under the performance-based option in Method 1664. EPA is particularly interested in comparative data produced from alternative techniques and separatory funnel extraction with gravimetric determination when both techniques are concurrently applied to a given wastewater discharge.

The final product of Phase II was Method 1664, which uses n-hexane as the extraction solvent. Between April and September, 1994, the March 29, 1994 draft version of the Method was distributed extensively at several conferences and workshops and in response to requests to EPA.

EPA encouraged reviewers to submit any questions, clarifications, data, or issues for consideration in updating the Method for this proposal. As part of the effort to collect information from interested parties, a questionnaire pertaining to Phase II of the Freon Replacement Study and the content of Method 1664 was distributed on May 4, 1994 at EPA's 17th Annual Conference on Analysis of Pollutants in the Environment. EPA also collected information from studies performed by industry representatives, including a report produced by the American Petroleum Institute titled Method Development and Freon-113 Replacement in the Analysis of Oil and Grease in Petroleum Industry Samples, and a presentation delivered by Dave

Clampitt from the Uniform & Textile Service Association at EPA's 17th Annual Conference on Analysis of Pollutants in the Environment titled Impact of Detergents on the Determination of Oil and Grease by Gravimetric and Infra-red Analysis. Comments received as a result of these efforts were reviewed and considered when revising Method 1664 to produce the April 1995 version being proposed today. These comments are included in the docket.

The quality control acceptance criteria in Method 1664 were derived from the Phase II results and the results from an interlaboratory study conducted by 11 laboratories belonging to the Twin City Round Robin Group. In addition, Method Detection Limit (MDL) studies were performed to determine the MDL and minimum level (ML) specifications included in the version of the Method being proposed today. Details of these studies are included in the docket as part of the document titled Report of the Method 1664 Validation Studies, April 1995.

B. N-Hexane as the Extraction Solvent

In the process of deciding upon the most suitable extraction solvent for replacement of Freon-113, EPA considered the potential effects of the new solvent on compliance monitoring, logistics, and health and safety concerns. Of all the solvents evaluated in the Freon Replacement Study, n-hexane was the most appropriate choice for the following reasons: (1) It had been previously used as the extraction solvent for permit compliance analysis of oil and grease and TPH prior to the advent of Freon-113, (2) EPA Phase I and Phase II studies indicated that n-hexane produces results that are as or more comparable to Freon-113 results than other solvents (although no solvent produced results exactly equivalent to Freon-113), and (3) the Phase II study showed that there was no significant difference in results produced by n-hexane and Freon-113 for the analysis of reagent water samples spiked with reference standards. In addition, a comparison of the Phase I and Phase II data suggests that any change in oil and grease concentration that may result from using n-hexane instead of Freon-113 would be overshadowed by the variability that was observed in the currently approved Freon methods that did not impose these thorough QC requirements.

Once the solvent choices had been narrowed to cyclohexane and n-hexane, and the results of Phase II indicated that both solvents produced equivalent results, the decision was based on more

pragmatic issues. Of concern was the neurotoxicity of n-hexane compared to cyclohexane and the cost of the two solvents. EPA compared the Occupational Safety and Health Administration (OSHA) permissible exposure limits (PELs) for cyclohexane and n-hexane. This comparison showed that n-hexane is only 1.7 times more toxic than cyclohexane and that the time weighted average (TWA) for n-hexane is 300 ppm, compared to 500 ppm for cyclohexane. TWA is defined as the average airborne exposure that shall not be exceeded in any 8-hour work shift of a 40-hour work week. Based on these figures, the toxicity of n-hexane is not appreciably higher than that of cyclohexane and can be minimized by implementing effective safety controls and procedures in the occupational setting.

An examination of the relative costs of n-hexane and cyclohexane revealed that cyclohexane costs approximately 17 percent more than n-hexane from the four suppliers surveyed.

Based on the solvent evaporation time issue presented in the discussion on Phase II of the Freon Replacement Study and the cost considerations detailed above, n-hexane was selected over cyclohexane as the solvent to replace Freon-113.

III. Summary of Proposed Rule

A. Introduction

This proposed rule would allow the use of EPA Method 1664 for the determination of "oil and grease" and "total petroleum hydrocarbons", and would withdraw approval of EPA Method 413.1 and Standard Method 5520B. Though on May 10, 1995, a global exemption for laboratory and analytical essential uses of CFCs was granted for the 1996 and 1997 control periods (60 FR 24970), this proposed rule will nonetheless provide for the elimination of the use of Freon-113 because of the unacceptable inconsistencies that would be created by allowing analytical methods employing two different solvents for determination of oil and grease. The proposed replacement method, Method 1664: N-Hexane Extractable Material (HEM) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons), April 1995, is a gravimetric procedure applicable to aqueous matrices for the determination of n-hexane extractable material and silica gel treated n-hexane extractable material (oil and grease and total petroleum hydrocarbons, respectively). The proposed method contains more

thorough QA/QC procedures than the Freon methods proposed for withdrawal.

"Oil and Grease" is a conventional pollutant defined in the Clean Water Act and codified at 40 CFR 401.16. The term "n-hexane extractable material" (HEM) reflects the fact that this method can be applied to materials other than oils and greases. Similarly, the term "silica gel treated n-hexane extractable material" (SGT-HEM) reflects that this method can be applied to materials other than aliphatic petroleum hydrocarbons that are not adsorbed by silica gel.

Method 1664 is a performance-based method that allows alternative extraction and concentration techniques, provided that equivalent performance can be demonstrated using reference standards and by complying with all performance specifications given in the Method.

B. Summary of Proposed Method 1664

For determination of n-hexane extractable material (HEM), samples are acidified to pH <2 and serially extracted three times with 30-mL portions of n-hexane in a separatory funnel. The extract is filtered through sodium sulfate to remove residual water, and the solvent is evaporated by heating with a steam or water bath. The HEM that remains is weighed and the concentration calculated in mg/L.

For determination of silica gel treated n-hexane extractable material (SGT-HEM), the HEM is redissolved in n-hexane, and silica gel is added to remove adsorbable materials. The amount of silica gel added is proportional to the amount of HEM. The solution is filtered to remove the silica gel, the solvent is evaporated by heating, and the SGT-HEM is weighed to produce the concentration in mg/L.

C. Method Quality Control

The quality control criteria incorporated into the Method exceeds and improves upon that of the currently approved 40 CFR Part 136 oil and grease methods, and is consistent with the 40 CFR 136 Appendix A protocol for determination of organic analytes. Initial demonstrations of laboratory capability are required and consist of (1) a method detection limit (MDL) study to demonstrate that the laboratory is able to achieve the MDL and ML specified in the Method, and (2) an initial precision and recovery (IPR) test consisting of the analysis of four spiked reagent water samples to demonstrate the laboratory's ability to generate acceptable precision and accuracy.

An important component of these and other QC tests required in Method 1664 is the use of hexadecane and stearic acid as the reference standards for spiking. Hexadecane was chosen to simulate petroleum hydrocarbons; stearic acid was chosen to simulate animal fats and detergents, and serves to test the effects of the silica gel procedure. The use of standards of known composition and purity, which are not a requirement of the currently approved gravimetric methods for the determination of oil and grease, allows for more accurate determination of recovery and precision and minimizes variability that may be introduced from spiking with substances such as Wesson oil, #2 fuel oil, mineral oil, etc. that are comprised of unknown proportions of substances.

Routine quality control consists of an initial two point calibration of the analytical balance, and the following tests that must accompany each analytical batch (the set of samples extracted at the same time, to a maximum of 10 samples):

- Analysis of a matrix spike (MS) and matrix spike duplicate (MSD) to demonstrate method accuracy and precision, and to monitor matrix interferences. Hexadecane and stearic acid are the reference standards used for spiking.
- Analysis of a laboratory blank to demonstrate freedom from contamination.
- Verification of calibration of the analytical balance, to demonstrate that measurements are in control.
- Analysis of an ongoing precision and recovery (OPR) sample to demonstrate that the analysis system is in control and acceptable precision and accuracy is being maintained with each analytical batch. The OPR sample consists of reagent water spiked with hexadecane and stearic acid. It is the equivalent of one of the IPR samples.

The laboratory is required to meet the acceptance criteria listed in Method 1664 for these quality control tests and is encouraged to monitor performance over time to identify and anticipate problems or improvements to the procedure.

Aside from the use of a solvent other than Freon-113, the most significant difference between Method 1664 and approved and existing methods used for oil and grease and petroleum hydrocarbons determinations is that Method 1664 contains an extensive QA/QC program that allows the data user to evaluate the quality of the results. This promotes a consistent, careful evaluation of the data generated that increases the reliability of results produced by HEM and SGT-HEM

determinations, and provides a means for laboratories and data users to monitor analytical performance, thereby providing a basis for sound, defensible data.

D. Performance Based Approach

To allow for advances in technology and reductions in the cost of analyses, Method 1664 is performance-based. Alternate extraction and concentration procedures are permitted as long as the equivalency procedures in the Method are followed and all QC acceptance criteria are met. The equivalency procedures consist of performing the IPR test using reference standards to demonstrate that the results produced with the modified procedure meet the specifications in Method 1664. In addition, if the detection limit will be affected by the modification, performance of an MDL study is required to demonstrate that the modified procedure can achieve an MDL less than or equal to the MDL in the Method or, for those instances in which the regulatory compliance level is greater than the Minimum Level in the Method, one-third the regulatory compliance level, whichever is higher.

If the performance-based option is to be applied to analyses for compliance monitoring, the discharger must demonstrate that the modified method produces results equivalent to those produced by Method 1664 for each specific discharge. The reason that this additional demonstration over and above the demonstration of equivalency with reference standards is required is that the possibility exists that Method 1664 and the modified method may produce equivalent results with reference standards but not produce equivalent results when the discharge is analyzed. Both Phase I and Phase II of the Freon Replacement Study demonstrated that results produced by other solvents are not equivalent to the results produced by Freon-113 when comparative tests are performed on discharges. EPA is concerned that the amount of material extracted from a discharge by a modified method will not be equivalent to the amount of material extracted by Method 1664. If the amount of material extracted is less when the modified method is used, the amount discharged could be greater before a violation would occur. Similarly, if the amount of material extracted is greater when the modified method is used, the amount discharged would need to be less to prevent a violation. The requirement to verify the equivalence of the modified method to Method 1664 assures that the modified method and

Method 1664 exhibit equivalent performance on the specific discharge.

For those instances in which the results from the equivalence study of field samples are below the Minimum Level, and the test of the modified method is passed for spikes of reference standards into reagent water, the modified method is deemed to be equivalent to this method for determining HEM and/or SGT-HEM on that specific discharge. This allowance is based on the reasoning that the level of material in this discharge will be so low that it is unlikely a violation will ever occur with this discharge and, consequently, small differences in the amount measured with the modified method as compared to Method 1664 will be negligible.

The procedure required to demonstrate equivalency consists of the following: (1) Two sets of four one-liter aliquots of a specific discharge are collected for analysis—one set is analyzed according to Method 1664—the other set is analyzed according to the modified procedure, (2) the average percent recovery of HEM and/or SGT-HEM is calculated for each set of four analyses, and (3) the average percent recovery using the modified procedure must be 79–114 percent of the average percent recovery produced by Method 1664 for HEM and 66–114 percent of the average percent recovery produced by Method 1664 for SGT-HEM. Unless these criteria are met, the modified procedure cannot be used for compliance monitoring purposes.

Whether or not the modified procedure is applied to compliance monitoring, all modifications to the Method must be thoroughly documented and the documentation must be maintained in the format specified in Method 1664.

IV. Method Validation and Precision and Recovery of the Proposed Test Method

The version of Method 1664 being proposed today is the product of revisions to the March 29, 1994 draft, and the October 1994 and January 1995 versions, and reflects consideration of numerous peer review comments, survey results, data from industry studies, results from an interlaboratory method validation study, and results from several EPA single-laboratory method detection limit (MDL) studies.

A. Precision and Recovery Studies

The interlaboratory method validation study conducted by EPA consisted of tests of initial precision and recovery (IPR) and ongoing precision and recovery (OPR) in twelve different

analytical testing laboratories. Data produced in this study were used to derive the QC acceptance criteria for precision and recovery that are specified in Table 1 of Method 1664. Details of the analyses and results are described in a document titled Report of the Method 1664 Validation Studies, April 1995.

One of the twelve laboratories participating in the validation study performed the QC analyses, which included one set of IPR analyses and 30 OPR analyses, as part of the n-hexane, cyclohexane, and Freon-113 comparisons for Phase II of the Freon Replacement Study. Since many of the techniques incorporated into Method 1664 evolved as this evaluating laboratory performed analyses under Phase II of the Freon Replacement Study, some of the work was developmental in nature. For example, the decision to use hexadecane and stearic acid as spiking standards required determination of an appropriate concentration as well as an appropriate solvent for the stock solution.

Another issue was the applicability of the silica gel extraction procedure to HEM concentrations in excess of 100 mg/L. The adsorptive capacity of silica gel needed to be studied in order to determine the amount of silica gel required to adsorb increasing concentrations of HEM. In addition, it was necessary to determine an appropriate cutoff for the maximum amount of silica gel that realistically could be used. Through a series of tests, it was determined that if more than 30 g of silica gel is used, the potential for contamination from substances in the silica gel increases.

Analysis of IPRs prior to sample analysis and continuing evaluation of the analytical system through OPR analyses were necessary to evaluate the potential effects of all procedural changes implemented as a result of this developmental work.

Results from the evaluating laboratory's analysis of real world samples supports the Method 1664 criteria derived from the method validation data. Because each field sample was analyzed in triplicate, the standard deviation of the replicate values could be derived. The mean relative standard deviation (RSD) across all analyses was 11.5 percent, thereby demonstrating the precision of Method 1664 on real world sample matrices.

The other eleven laboratories involved in method validation, working cooperatively as part of the Twin City Round Robin (TCRR) Group, performed IPR and OPR analyses for the determination of HEM by Method 1664.

In addition to the QC analyses, this study consisted of the analysis of two sets of samples, one from a petroleum source and the other from a nonpetroleum source, in triplicate, for HEM. The mean RSD of the results across all laboratories and all samples was 9.5 percent, further demonstrating that Method 1664 is capable of producing precise results on real world samples. Results and evaluation of the TCRR study, including field sample analyses, are presented in the document titled Report of the Method 1664 Validation Studies, April 1995.

TCRR study participants submitted comments on the method, most of which focused on difficulties related to extracts containing excessive amounts of water and the longer time required for the evaporation of n-hexane. These issues have been addressed in the revision of Method 1664 being proposed today, the former by recommending more careful separation of the aqueous and solvent phases to avoid carryover of the water into the extract and that more sodium sulfate be used in the filtering process, and the latter by allowing the use of either a water bath or steam bath set at a temperature that results in evaporation of the solvent within 30 minutes.

Most laboratories in the interlaboratory study did not encounter difficulties with the analysis of IPR and OPR samples and were able to achieve acceptable recoveries of hexadecane and stearic acid. Statistical evaluation of the results from all twelve laboratories produced few outliers, indicating that Method 1664 is a reproducible procedure sufficiently reliable to be used by a variety of laboratories.

B. Development of Quality Control Acceptance Criteria

As stated above, data from the TCRR interlaboratory study were combined with data from EPA's data gathering in Phase II to produce performance specifications in the form of quality control (QC) acceptance criteria for Method 1664. The development of these criteria are described in the Report of the Method 1664 Validation Studies, April 1995, included in the docket. Criteria were developed for initial precision and recovery (IPR), ongoing precision and recovery (OPR), and recovery of hexadecane and stearic acid spiked into samples (matrix spikes) for both HEM and SGT-HEM. For HEM, the IPR and OPR acceptance criteria were constructed using analysis of variance (ANOVA) statistics. The criteria for recovery of a matrix spike (MS) and for the relative percent difference between an MS and a matrix spike duplicate

(MSD) were transferred from the IPR and OPR criteria, since neither the TCRR study or EPA's data gathering efforts required the spiking of field samples. EPA believes that this transfer is acceptable because the determinative technique in Method 1664 is gravimetry, which is not susceptible to interferences, and because the treated effluent to which Method 1664 is to be applied in monitoring is nearly identical to the reagent water used in the IPR and OPR tests. EPA used a similar transfer of data for development of specifications for acceptance criteria in the organic methods promulgated at 40 CFR Part 136, Appendix A.

For SGT-HEM, EPA received results from only two laboratories. EPA used these data to construct preliminary acceptance criteria for SGT-HEM and widened these preliminary criteria to those of HEM in those instances in which the calculated SGT-HEM criteria were more stringent than those for HEM. The acceptance criteria were widened based on the knowledge that the determination of SGT-HEM follows the determination of HEM in Method 1664, and therefore the results for SGT-HEM is likely to be at least as variable as results for HEM.

EPA solicits data on the variability of the determination of HEM and SGT-HEM, particularly data from interlaboratory studies using either the March 29, 1994 version of Method 1664 that was distributed at various conferences, the October 1994 or January 1995 versions of Method 1664, or the April 1995 version that is cited in today's proposed rule and which is included in the docket.

C. Method Detection Limit Studies

To date, five single-laboratory method detection limit (MDL) studies have been performed as part of the effort to determine MDLs and MLs for HEM and SGT-HEM. Results of these studies are detailed in the document titled Report of the Method 1664 Validation Studies, April 1995. The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. To determine the MDL, the laboratories were required to follow the procedure in Appendix B to 40 CFR Part 136. This procedure consists of the analysis of seven aliquots of reagent water that are spiked with the analyte(s) of interest. For EPA's MDL studies, the hexadecane and stearic acid specified in the quality control tests in Method 1664 were used. Spike levels were in the range of one to five times the estimated detection limit. The MDL is calculated by multiplying

the standard deviation of the seven replicate analyses by the Student's *t* value for ($n - 1$) degrees of freedom, where n equals the number of replicates. The Student's *t* value for seven replicates is 3.143.

The Minimum Level is defined as the level at which the entire analytical system produces a recognizable signal and an acceptable calibration point, and is determined by multiplying the MDL by 3.18 and rounding the resulting value to the number nearest to $(1, 2, \text{ or } 5) \times 10^n$, where n is an integer. The value "3.18" represents the ratio between the Student's *t* multiplier used to determine the MDL (3.143) and the 10 times multiplier used in the American Chemical Society (ACS) Limit of Quantitation (LOQ) (i.e., $10 \div 3.143 = 3.18$). For example, if the calculated MDL is 1.7, the ML will be equal to 1.7 times 3.18, which equals 5.1. Rounding to the number nearest to $(1, 2, \text{ or } 5) \times 10^n$ establishes the ML at 5.0.

The first MDL study was performed in a commercial laboratory by an analyst at the Ph.D. level who has more than 20 years of experience in the determination of oil and grease and TPH. This study yielded an MDL of 0.91 mg/L and a resultant ML of 2 mg/L for HEM and an MDL of 1.6 mg/L and a resultant ML of 5 mg/L for SGT-HEM.

Based on the disparity between the results obtained by this laboratory and the lower limit of the range in Method 413.1, it was decided that a second MDL study should be conducted in another commercial laboratory to verify the values obtained in the first study.

The second MDL study was also performed by a laboratory experienced in the determination of oil and grease and TPH, though the analysts performing the study were not at the Ph.D. level. In order to move expeditiously, the laboratory was required to perform the second MDL study within 24 hours. An MDL of 5.4 mg/L and an ML of 20 mg/L for HEM, and an MDL of 2.6 mg/L and an ML of 10 mg/L for SGT-HEM was determined in the second MDL study.

The second laboratory was contacted to determine if they encountered difficulties in performing the study. They stated that the results were the best that could be obtained under the imposed 24 hour turn-around time constraint, and that they believed they could achieve lower MDLs given more time. Based on these circumstances, the Agency decided that the MDLs to be included in the October version of Method 1664 should be those representing the better performing laboratory. Therefore, the MDL and associated ML values from the original

Method 1664 MDL study were incorporated into the October 1994 revision of the Method.

The high results produced in the second MDL study brought into question the reasonableness and effect of requiring a 24-hour turnaround. As a result, the second laboratory performed another MDL study (MDL study #3), this time without the turnaround constraint, and with the analytical objective to confirm the MDLs/MLs that had been obtained in the first MDL study. An MDL of 2.4 mg/L and an associated ML of 10 mg/L for HEM, and an MDL of 1.7 mg/L and an associated ML of 5 mg/L for SGT-HEM were obtained from this third MDL study. Although closer to the MDL and ML for HEM obtained in the first MDL study, the ML of 10 mg/L for HEM is still above the equivalent level in Method 413.1, and the result for SGT-HEM, the more complex procedure, is still less than the result for HEM.

From these results, the Agency concluded that the MDLs/MLs for HEM and SGT-HEM produced in the first MDL study are self-consistent, whereas the results produced in the second and third MDL studies are not. Therefore, the MDL and ML limits specified in the January 1995 version of the Method were those from the first MDL study.

The Agency still needed to address the issue that the HEM MDL values in both the October 1994 and the January 1995 versions of Method 1664 had not been verified with follow-up MDL studies. In contrast, a comparison of SGT-HEM results shows that the MDL/ML for SGT-HEM from the third MDL study supports the first MDL study results for SGT-HEM. (Both the first and third MDL studies produced an ML of 5 mg/L for SGT-HEM.)

To verify the HEM MDL and ML values specified in the October 1994 and January 1995 versions of Method 1664, which were the results obtained in MDL study #1, the laboratory that performed this MDL study conducted another study (MDL study #4). As with MDL study #1, the same Ph.D. level chemist with extensive analytical experience performed the analyses. Because the spike level in MDL study #1 was greater than five times the resulting MDL, the spike level was lowered to 5 mg/L. An MDL of 0.88 mg/L, with a resulting ML of 2 mg/L was obtained, thereby supporting the original MDL results.

In response to comments received from laboratories and other interested parties regarding the difficulties encountered when attempting to achieve the HEM MDL of 0.91 mg/L specified in the October 1994 and January 1995 versions of Method 1664,

and because most technicians performing HEM analysis for commercial laboratories will not have the experience or qualifications of the Ph.D. level chemist who performed MDL studies 1 and 4, an analyst with a bachelor's degree and one month's laboratory experience performed another HEM MDL study at this laboratory. The results of MDL study #5 were an HEM MDL of 1.4 mg/L and a resulting ML of 5 mg/L.

EPA has concluded that the MDL appropriate for Method 1664 should be representative of a better performing laboratory. However, to realistically address the qualifications of the laboratory personnel most likely to perform this procedure, the MDL should reflect the results obtained when using qualified, but not Ph.D. level, personnel. Therefore, the HEM MDL specified in the April 1995 version of Method 1664 (the version being proposed) is 1.4 mg/L and the HEM ML is 5 mg/L. Unchanged from the January 1995 version of Method 1664, the SGT-HEM MDL is 1.6 mg/L and the SGT-HEM ML is 5 mg/L.

EPA solicits comment on the appropriateness of these MDLs and data from other MDL studies conducted with the goal of achieving an MDL of 1 mg/L or less for HEM and SGT-HEM.

V. Withdrawal of Currently Approved Methods

The Clean Air Act Amendments of 1990 (CAAA) established schedules for phasing out the production and importation of CFCs in the U.S. Pursuant to section 606, production of most class I substances, including Freon, is phased-out as of January 1, 1996, except for a few exemptions for essential uses. Existing supplies may be used after that date, but the substances will become increasingly scarce and costly over time. On May 10, 1995, a global exemption for laboratory and essential analytical uses of CFCs was granted for the 1996 and 1997 control periods (60 FR 24970). This exemption explicitly allows for the production of CFCs for laboratory use through December 31, 1997. Therefore, it would be possible to allow continued use of the currently approved analytical methods that employ CFCs along with the use of Method 1664.

EPA has considered allowing continued use of the currently approved methods, but believes that unacceptable conflicts would be created by allowing the simultaneous use of oil and grease methods that employ different extraction solvents. As is detailed above, EPA's Freon Replacement Studies indicated that no solvent

produces results sufficiently equivalent to the results produced by Freon-113. By allowing two or more methods that employ different solvents, the possibility exists that a regulatory authority and a discharger could produce different results for the same analyte in the same sample. Indeed, the same analyst testing the same sample could produce unacceptably different results using the different methods. If one of these results showed a permit violation and the other did not, an unfair conflict would result.

As is also detailed above, Method 1664 contains extensive quality control procedures to assure that precise and accurate results are produced. If use of the currently approved methods is continued, the possibility also exists that analytical results could indicate a permit violation due to the greater variability of results produced by these methods when compared to the proposed Method 1664. For example, if the permit limit is 20 mg/L and the true concentration of oil and grease in the discharge is slightly less than this limit, Method 1664 is more likely to produce a result closer to the true value than the currently approved methods because of the improved precision of the Method.

The conflict between results obtained using the existing approved methods and results obtained using the proposed Method 1664 arises because oil and grease is a "method-defined" parameter. Much like biochemical oxygen demand measured over five days ("BOD₅"), and total suspended solids ("TSS"), which measures the amount of non-filterable material suspended in water, the quantification of oil and grease depends on the procedures used to measure the parameter in the first place. The analytical result is dependent on how the measurement is conducted. In the case of BOD₅, the sample pH, the seed quality, the incubation time and temperature, and other factors define how much BOD occurs. In the case of TSS, the sample homogeneity, the filter type and pore size, the drying time and temperature, and other factors determine the amount of solids that will be measured. In the specific case of oil and grease, one portion of the test sample preparation procedure, the addition of a specific solvent, *defines* how much "oil and grease" will be extracted. The oil and grease parameter is that material which is extracted by the solvent and not lost during solvent drying or evaporation.

Given these concerns, and to avoid other potential conflicts, EPA is proposing to withdraw approval of the use of methods for oil and grease

determination that are currently promulgated at 40 CFR Part 136.

In an effort to provide for the use and depletion of existing laboratory stocks of Freon-113, EPA plans to implement the withdrawal of the existing Freon methods no sooner than six months after the final rule is published in the Federal Register. In this scenario, Freon-113 and the currently approved methods would continue to be used until the implementation date. N-hexane and Method 1664 would be required on that date and thereafter. EPA seeks comment on the desirability of this scenario, alternate scenarios, and whether the 6-month period is sufficient or, if insufficient, the length of the desired period. EPA also seeks comment as to whether the 6-month period is too long, in that persons and organizations affected by this rule may desire to switch to n-hexane sooner to reduce the costs associated with the purchase of Freon-113. When submitting comments on this issue, please indicate the amount of Freon-113 being used by your organization for oil and grease determinations using the currently approved 40 CFR Part 136 methods so that EPA can assess the number of parties affected and the extent of the effect.

VI. Regulatory Requirements

A. Regulatory Impact Analysis

Executive Order 12866 requires that regulatory agencies prepare an analysis of the regulatory impact of major rules. Major rules are defined as those likely to result in: (1) An annual cost to the economy of \$100 million or more; or (2) a major increase in costs or prices for consumers or individual industries; or (3) significant adverse effects on competition, investment, innovation, or international trade. This regulation is not a major regulation for the reasons discussed below.

The impact of this proposed regulation will be far less than \$100 million annually. Laboratories are switching to CFC substitutes (or substitute methods) as CFCs become more costly due to restriction in supply and due to the excise tax that, as a result of the 1989 and 1990 Budget Reconciliation Acts, is imposed on all ozone-depleting chemicals listed in the Montreal Protocol and the 1990 CAAA. Thus, the true cost of this regulation is the difference in expense of switching to CFC substitutes now as opposed to later. The Agency believes that these increased transitional costs will be minimal for the following reasons:

First, laboratory testing is a very small part of Freon-113 consumption (less

than 1 percent) and the testing required by EPA is only a fraction of this total. EPA estimates that the total market for Freon-113 for laboratory use is less than \$2 million annually.

Second, this rule is not likely to cause a major increase in costs or prices for individuals or consumers. Laboratories may experience some increase in costs due to longer testing procedures because of the increased number of sample manipulations and the additional quality control in the method. However, the price for n-hexane is actually cheaper per pound than the CFCs and this difference may increase as CFC production is reduced and supply becomes more limited.

Third, this regulation is unlikely to cause significant adverse effects on competition, investment, innovation, or international trade. As noted above, laboratory use of these products is estimated to be much less than 1 percent of the total market for these products. Further, in some cases this proposed rule and notice would result in a switch back to procedures commonly used in the 1970s, which did not have a significant impact on competition, investment, or trade at that time.

On March 9, 1995, this proposal was granted a waiver from review by the Office of Management and Budget.

B. Unfunded Mandates

Under Section 202 of the Unfunded Mandates Reform Act of 1995, signed into law on March 22, 1995, EPA must prepare a statement to accompany any rule where the estimated costs to State, local, or tribal governments, or to the private sector, will be \$100 million or more in any one year. Under Section 205, EPA must select the most cost-effective and least burdensome alternative that achieves the objective of the rule and is consistent with statutory requirements. Section 203 requires EPA to establish a plan for informing and advising any small governments that may be significantly impacted by the rule.

EPA estimates that the costs to State, local, or tribal governments, or to the private sector, from this rule will be less than \$100 million. This rulemaking should have minimal impact on the current regulatory burden imposed on permittees because the rulemaking will simply replace an existing test procedure with a new procedure. EPA has determined that an unfunded mandates statement is therefore unnecessary. Similarly, the method in today's rule does not establish any regulatory requirements that might significantly or uniquely affect small governments.

C. Regulatory Flexibility Act

Pursuant to the Regulatory Flexibility Act, 5 U.S.C. 601, whenever an agency is required to publish a notice of rulemaking for any proposed or final rule, it must prepare and make available for public comment a Regulatory Flexibility Analysis that describes the effect of the rule on small entities (i.e., small businesses, small organizations, and small governmental jurisdictions). This analysis is unnecessary if the Agency's Administrator certifies that the rule will not have a significant economic effect on a substantial number of small entities.

This proposed rule will not have a significant economic impact on a substantial number of small facilities. This regulation simply approves an analytical technique to be available for use by all laboratories.

D. Paperwork Reduction Act

This rule contains no requests for information and is, therefore, exempt from the requirements of the Paperwork Reduction Act, 44 U.S.C. 3501 *et seq.*

VII. Materials Proposed for Incorporation by Reference Into 40 CFR Part 136

1. Method 1664: N-Hexane Extractable Material (HEM) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons), April 1995, Document

No. EPA-821-B-94-004b, available from the EPA Water Resource Center, Mail Code RC-4100, 401 M Street, S.W., Washington, D.C. 20460, phone: 202/260-7786 or 202/260-2814.

VIII. Request for Comments

EPA requests public analysis, comments, and information on the replacement of Freon-113 with n-hexane, the utility of Method 1664 for monitoring, the QC acceptance criteria in Method 1664, the MDL and ML levels, the performance-based option criteria, and the 6-month implementation scenario.

List of Subjects in 40 CFR Part 136

Environmental protection, Reporting and recordkeeping requirements, Water pollution control, Incorporation by reference.

Dated: December 12, 1995.
Carol M. Browner,
Administrator.

In consideration of the preceding, USEPA proposes to amend 40 CFR Part 136 as follows:

PART 136—[AMENDED]

1. The authority citation of 40 CFR Part 136 continues to read as follows:

Authority: Secs. 301, 304(h), 307, and 501(a) Pub. L. 95-217, 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 and the Water Quality Act of 1987), 33 U.S.C. 1314 and 1361; 86 Stat. 816, Pub. L. 92-500; 91 Stat. 1567, Pub. L. 92-217; Stat. 7, Pub. L. 100-4 (The "Act").

2. In § 136.3(a), Table 1B.-List of Approved Inorganic Test Procedures, is proposed to be amended by revising entry 41. Oil and grease-Total recoverable; by adding an entry for petroleum hydrocarbons, total recoverable; and by adding a note to Table 1B to reference Method 1664 to read as follows:

§ 136.3 Identification of test procedures.
* * * * *

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, units and methods	Reference (Method No. or page)				
	EPA 1. 35	Std. meth- ods 18th Ed.	ASTM	USGS2	Other
41. Oil and grease—Total recoverable, mg/L; Gravimetric (extraction)	xx1664				
— Petroleum hydrocarbons—Total recoverable; mg/L; Gravimetric (extraction)	xx1664				

TABLE 1B.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and methods	Reference (Method No. or page)				
	EPA 1. 35	Std. meth- ods 18th Ed.	ASTM	USGS2	Other

^{xx} Method 1664: N-Hexane Extractable Material (HEM) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM) by Extraction and Gravimetry (Oil and Grease and Total Petroleum Hydrocarbons), April 1995, Document No. EPA-821-B-94-004b, can be obtained from the EPA Water Resource Center, Mail Code RC-4100, 401 M Street, S.W., Washington, D.C. 20460.

* * * * *
 3. In § 136.3(e), Table II—Required Containers, Preservation Techniques, and Holding Times, is proposed to be amended by adding an entry for petroleum hydrocarbons to read as follows:
§ 136.3 Identification of test procedures.
 * * * * *

TABLE II.—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter	Con- tainer ¹	Preservation ^{2,3}	Maximum holding time ⁴
(Add the following entry.) — —. Petroleum hydrocarbons	G	Cool to 4° C, H ₂ SO ₄ or HCL to pH<2	28 days.

[FR Doc. 96-877 Filed 1-22-96; 8:45 am]
 BILLING CODE 6560-50-P

DEPARTMENT OF COMMERCE

National Oceanic and Atmospheric Administration

50 CFR Part 663

[Docket No. 960111002-6002-01; I.D. 112495B]

RIN 0648-AG31

Pacific Coast Groundfish Fishery; Designation of Routine Management Measures

AGENCY: National Marine Fisheries Service (NMFS), National Oceanic and Atmospheric Administration (NOAA), Commerce.

ACTION: Proposed rule; request for comments.

SUMMARY: NMFS issues a proposed rule that would designate certain management measures as "routine" in the Pacific coast groundfish fishery off Washington, Oregon, and California. Once management measures have been designated as routine, they may be modified after a single meeting and recommendation of the Pacific Fishery Management Council (Council). Such action is authorized under the Pacific

Coast Groundfish Fishery Management Plan (FMP) and is intended to provide for responsive inseason management of the groundfish resource.

DATES: Comments must be received by March 8, 1996.

ADDRESSES: Comments may be mailed to William Stelle, Jr., Director, Northwest Region, National Marine Fisheries Service, 7600 Sand Point Way NE., BIN C15700, Seattle, WA 98115-0070; or Hilda Diaz-Soltero, Director, Southwest Region, National Marine Fisheries Service, 501 W. Ocean Blvd., Suite 4200, Long Beach, CA 90802-4213. Information relevant to this proposed rule has been compiled in aggregate form and is available for public review during business hours at the Office of the Director, Northwest Region, NMFS. Copies of the Environmental Assessment/Regulatory Impact Review (EA/RIR) can be obtained from the Council, 2000 SW First Avenue, Suite 420, Portland, OR 97201.

FOR FURTHER INFORMATION CONTACT: William L. Robinson at 206-526-6140, or Rodney R. McInnis at 310-980-4030.

SUPPLEMENTARY INFORMATION: The FMP authorizes the designation of certain management measures as "routine." Routine management measures are specific for species, gear types and purposes. Implementation and adjustment of those routine measures

may occur after consideration at a single Council meeting, subsequent approval by NMFS and announcement in the Federal Register. Adjustments must be within the scope of the analysis performed when the management measure originally is designated routine. A list of routine management measures is found at 50 CFR 663.23, specifying the species and gear types to which they apply.

At its August 1994 meeting, the Council announced its preliminary recommendation to designate the management measures contained in this proposed rule as routine. A draft EA/RIR was distributed to the public. At its October 1994 meeting, after hearing public testimony, the Council confirmed its preliminary recommendations to establish additional routine designations as follows: (1) Trip limits for all groundfish species, separately or in any combination, taken with open access gear; and (2) trip and size limits for lingcod, and trip limits for canary rockfish, shortspine thornyheads and longspine thornyheads taken with any legal gear in the limited entry (or open access) fisheries.

The most common type of routine management measure is "trip landing and frequency limits," which applies to the harvest of most major groundfish species. Trip landing and frequency limits (trip limits) include limits on the