

ENVIRONMENTAL PROTECTION AGENCY

40 CFR Part 136

[FRL-5308-4]

Guidelines Establishing Test Procedures for the Analysis of Pollutants: New Methods

AGENCY: Environmental Protection Agency (EPA).

ACTION: Proposed rule and request for comment.

SUMMARY: Under the Clean Water Act, Section 304(h), EPA proposes to amend its list of approved analytical techniques by adding new or revised test procedures for certain metal and inorganic chemical pollutants, by adding method citations to Table IB and by amending the incorporation by reference section of the regulation accordingly.

EPA is also proposing to substitute reagents that are more environmentally friendly for certain hazardous and toxic chemical reagents currently used in certain approved methods.

In addition, EPA is proposing to withdraw approval of certain outdated or little used analytical methods, as well as, certain methods that require the use of hazardous or toxic reagents. For each method that is proposed for withdrawal, one or more commonly used methods have been previously approved.

Comments are requested on this proposed rulemaking. After considering comments received in response to this proposal, EPA will promulgate a final rule.

This action amends guidelines establishing test procedures for the analysis of pollutants by adding clarifying notes to lists of approved test procedures, by adding and updating certain method citations in Tables IB and IC, and by amending the incorporation by reference section of the regulation accordingly.

DATES: Comments on the proposed amendments will be accepted until December 18, 1995.

ADDRESSES: Send written comments to the 304(h) Docket Clerk (Ben Honaker), Water Docket (MC-4101), U.S. Environmental Protection Agency, 401 M Street, SW., Washington, DC 20460. Please submit any references cited in your comments. EPA would appreciate an original and three copies of your comments and enclosures (including references). Commenters who want EPA to acknowledge receipt of their comments should enclose a self-addressed, stamped envelope. No

facsimiles (faxes) will be accepted because EPA cannot assure that they will be submitted to the Water Docket.

The supporting materials are available for review at the Water Docket at the address above. For access to Docket Materials, call (202) 260-3027 between 9 am and 3:30 pm for an appointment.

FOR FURTHER INFORMATION CONTACT: Mr. James E. Longbottom, Aquatic Research Division, National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268. Telephone number: (513) 569-7308.

SUPPLEMENTARY INFORMATION:

Materials in the public docket include:

- "Methods for the Determination of Metals in Environmental Samples" Supplement I, EPA-600/R-94/111, May 1994.
- "Methods for the Determination of Inorganic Substances in Environmental Samples", EPA-600/R-93/100, August 1993.
- "Determination of Trace Elements in Water by Inductively Coupled Plasma-Mass Spectrometry: Collaborative Study", Journal of AOAC-International, 77, pp. 1004-1023, 1994.
- "Determination of Inorganic Anions in Water by Ion Chromatography: Collaborative Study", Journal of AOAC-International, 77, pp. 1253-1263, 1994.
- "Ion Chromatographic Method for Dissolved Hexavalent Chromium in Drinking Water, Ground Water, and Industrial Wastewater Effluents: Collaborative Study", Journal of AOAC-International, 77, pp. 994-1004, 1994.
- "Low-level Chlorine Analysis by Amperometric Titration", Journal of the Water Pollution Control Federation, 51, pp. 2636-2640, 1979.

A copy of the first two items can also be obtained from EPA through a facsimile request to the Chemistry Research Branch, Aquatic Research Division, Cincinnati, Ohio, on (513) 569-7757.

I. Authority

This regulation is proposed under authority of sections 301, 304(h) and 501(a) of the Clean Water Act, 33 U.S.C. 1251 *et seq.* (the Federal Water Pollution Control Act Amendments of 1972 as amended) (the "Act"). Section 301 prohibits the discharge of any pollutant into navigable waters unless the discharge complies with a 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 application 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 functions under this Act".

The use of approved test procedures or approved alternate procedures is required whenever the waste constituent specified is required to be measured for: an NPDES permit application; discharge monitoring reports; state certification; and other requests from the permitting authority for quantitative or qualitative effluent data. Use of approved test procedures is also required for the expression of pollutant amounts, characteristics, or properties in effluent limitations guidelines and standards of performance and pretreatment standards, unless otherwise specifically noted or defined.

II. Regulatory Background

The CWA establishes two principal bases for effluent limitations. First, existing discharges are required to meet technology-based effluent limitations. New source discharges must meet new source performance standards based on the best demonstrated technology-based controls. Second, where necessary, additional requirements are imposed to assure attainment and maintenance of water quality standards established by the States under Section 303 of the CWA. In establishing or reviewing NPDES permit limits, EPA must ensure that permitted discharges will not cause or contribute to a violation of water quality standards, including designated water uses.

For use in permit applications, discharge monitoring reports, and state certification and to ensure compliance with effluent limitations, standards of performance, and pretreatment standards, EPA has promulgated regulations providing nationally-approved testing procedures at 40 CFR Part 136. Test procedures have previously been approved for 262 different parameters. Those procedures apply to the analysis of inorganic (metal, non-metal, mineral) and organic chemical (including pesticides), radiological, biological, nutrient, demand, residue, and physical parameters.

Additionally, some particular industries may discharge pollutants for which test procedures have not been proposed and approved under 40 CFR Part 136. Under 40 CFR 122.41 permit writers may impose monitoring

requirements and establish test methods for pollutants for which no approved Part 136 method exists. 40 CFR 122.41(j) (4). EPA may also approve additional test procedures when establishing industry-wide technology-based effluent limitations guidelines and standards as described at 40 CFR 401.13.

The procedures for approval of alternate test procedures (ATPs) are described at 40 CFR 136.4 and 136.5. Under these procedures the Administrator may approve alternate test procedures for nationwide use which are developed and proposed by any person. 40 CFR 136.4 (a). Persons wishing to use such alternate procedures, must apply to the State or Regional EPA permitting office [for limited approval under 136.4 (b)], and to the Director of the Environmental Monitoring Systems Laboratory in Cincinnati [for nationwide approval under 136.4 (d)]. As specified below, today's proposed rule would approve additional nationwide test procedures for certain metals, anions, Cr(VI) and VOCs in wastewater and related samples.

III. Summary of Proposed Methods

The proposed methods have been reviewed by appropriate members of and written in the EPA Environmental Monitoring Management Council Format.

The methods proposed for addition include new methods for: preparation of samples for metals analysis, inductively coupled plasma/mass spectrometry (ICP/MS), a stabilized temperature graphite furnace atomic absorption (STGFAA) method for metals, and ion chromatography (IC) methods for anions and for hexavalent chromium [Cr(VI)]. A revised EPA inductively coupled plasma atomic emission spectrometry (ICP-AES) method for metals to replace the currently approved method, and a low-level extension of the approved method for the determination of low level total residual chlorine are also being proposed.

Methods 200.7, 200.8, and 200.9 were approved for use in EPA's drinking water programs at 40 CFR Part 141.23(k)(1) on December 5, 1994 (59 FR 62466). When the methods proposed today are promulgated at 40 CFR Part 136 for use in EPA's wastewater programs, these three methods will be conformed for the two programs.

Although not part of today's proposal, the Agency's Environmental Monitoring Management Council (EMMC) will soon propose integrated methods for citation in several regulations, including those at Part 136. The methods include a graphite furnace atomic absorption

spectrometry method for certain metals and an inductively coupled plasma atomic emissions spectrometry method for metals. These methods may be included in the final rule for today's proposal.

In the interest of pollution prevention, EPA is proposing to replace mercuric sulfate with copper sulfate in the total Kjeldahl nitrogen methods and to permit the substitution of the AMCO-AEPA-1 Standard for the formazin standard in the turbidity method. Replacement of these two reagents would remove hazardous or potentially carcinogenic chemicals from use in EPA approved methods.

A. Sample Preparation for Total Recoverable Elements

EPA is proposing a new broad-purpose digestion procedure for total recoverable elements. It has the advantage of being compatible with several of the approved measurement techniques, which will allow laboratories to achieve some cost savings by reducing preparations and increasing flexibility in choosing approved analytical techniques after digestion. This preparation procedure is a variation of the procedures given in the previously approved EPA 200 Series Methods for "total recoverable" metals published in "Methods for Chemical Analysis of Water and Wastes". It utilizes the same acid combination (nitric + hydrochloric) as the previous "total recoverable" procedures, but the acid concentration has been modified by lowering the amount of hydrochloric acid.

This sample preparation procedure has been incorporated into three EPA Methods proposed today, including, a May 1994 revision of the approved ICP/AES Method 200.7, a new measurement technology ICP/MS Method 200.8, and a stabilized temperature graphite furnace automatic absorption (GFAA) method (Method 200.9). In addition, EPA has prepared a stand-alone version of the digestion procedure: Method 200.2 "Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements". EPA proposes to permit the substitution of Method 200.2 for the digestion procedure in certain non-EPA approved direct aspiration flame atomic absorption procedures. The digestion procedure has been tested on various matrices using determinative methods 200.7, 200.8 and 200.9 and has been found comparable to previously approved NPDES preparation procedures. Method 200.2 was also validated through interlaboratory testing in the joint EPA/AOAC International study of Method

200.8. Each of the new EPA procedures includes: a list of elements to which it applies, sample preservation practices, and preparation conditions.

EPA proposes to withdraw the EPA AA direct aspiration and AA furnace methods contained in "Methods for Chemical Analysis of Water and Wastes". The methods are out of date and lack many of the necessary instructions included in more recently produced EPA methods. The remaining approved non-EPA methods for AA direct aspiration are sufficiently identical to the withdrawn methods as to result in no impact on the regulated community. The EPA furnace methods are being replaced by EPA Method 200.9. Appendix D to this regulation, which contains the interlaboratory measures of precision and accuracy for the EPA methods that are being withdrawn, would also be removed by this proposal.

B. Elemental Analysis by ICP/AES

The EPA proposes to approve the May 1994 revision of Method 200.7, "Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry" and to cite the revised method by reference. Accordingly, Appendix C to 40 CFR Part 136 would be deleted. The May 1994 edition of the method has been demonstrated to produce precision and recovery for the applicable elements that is equal to or better than that achieved by the currently approved edition and published in Table 4 of Appendix C. The procedure includes: A list of elements to which it applies, sample collection practices, preparation conditions, and quality control practices. The EMMC inductively coupled plasma-atomic emission spectrometry method being proposed elsewhere is proposed to be interchangeable with Method 200.7 for the purposes of this regulation.

C. Elemental Analysis by ICP/MS

The Agency is proposing a new multielement test procedure, ICP/MS, Method 200.8 "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry" for the detection and quantification of 20 metals in aqueous samples. Sample material in solution is introduced by pneumatic nebulization into a radio-frequency plasma where energy transfer processes cause desolvation, atomization and ionization. The ions are extracted from the plasma through a differentially pumped vacuum interface and separated on the basis of their mass-to-charge ratio by a

quadrupole mass spectrometer having a minimum resolution capability of 1 amu peak width at 5% peak height. The ions transmitted through the quadrupole are registered by an electron multiplier or Faraday detector and the ion information processed by a data handling system. Interferences relating to the technique must be identified and results must be corrected accordingly. Such corrections must include compensation for isobaric elemental interferences and interferences from polyatomic ions derived from the plasma gas, reagents or sample matrix. Instrumental drift, as well as suppressions or enhancements of instrument response caused by the sample matrix, must be corrected by the use of internal standardization.

The Agency developed ICP/MS Method 200.8 under a contract and in cooperation with AOAC International jointly conducted an interlaboratory validation study of the method. This method (May 1994) represents the current state-of-the-art for the determination of metals in wastewater and water related media. The method description includes a list of the elements to which the method applies, sample collection practices, recommended analytical conditions, quality control practices, instrumental and method detection limits, and performance criteria based on the interlaboratory study data.

AOAC-International has approved and published this ICP/MS method along with the study results as AOAC-International Method 993.14. The same method is in the final stages of the ASTM consensus process. EPA proposes to approve the AOAC-International method, as well as, consider approval of the ASTM ICP/MS Method if full ASTM Society acceptance is achieved prior to final EPA rulemaking.

D. Elemental Analysis by Stabilized Temperature Platform GFAA

The May 1994 Revision of Method 200.9 "Determination of Trace Metals by Stabilized Temperature Graphite Furnace Atomic Absorption" is being proposed as a replacement for the currently approved EPA furnace methods. Method 200.9 determines elements by stabilized temperature graphite furnace atomic absorption (STGFAA). In STGFAA, the sample and required matrix modifier are first pipetted onto the platform or a device which provides delayed atomization. The sample is then dried at a relatively low temperature ($\approx 120^\circ\text{C}$) to avoid spattering. Once dried, the sample is normally pretreated in a char or ashing step which is designed to minimize the

interference effects caused by the concomitant sample matrix. After the char step, the furnace is allowed to cool prior to atomization. The atomization cycle is characterized by rapid heating of the furnace to a temperature where the metal (analyte) is atomized from the pyrolytic graphite surface. The resulting atomic cloud absorbs the element specific atomic emission produced by a hollow cathode lamp or a electrodeless discharge lamp.

Because the resulting absorbance usually has a nonspecific component associated with the actual analyte absorbance, an instrumental background correction device is necessary to subtract from the total signal the component which is nonspecific to the analyte. In the absence of interferences, the background corrected absorbance is directly related to the concentration of the analyte. Interferences relating to suppression or enhancement of instrument response caused by the sample matrix, must be corrected by the method of standard addition.

The method description includes a list of elements to which the method applies, sample collection practices, recommended analytical conditions, quality control practices, method detection limits and performance criteria based on single laboratory study data. The EMMC furnace atomic absorption method being proposed elsewhere is proposed to be interchangeable with Method 200.9 for the purposes of this regulation.

E. Anions by Ion Chromatography

EPA developed IC Method 300.0 for the following anions: Bromide, chloride, fluoride, nitrate-N, nitrite-N, orthophosphate, and sulfate. Using IC, a water sample is injected into a stream of carbonate-bicarbonate eluent and passed through a series of ion exchangers. The anions of interest are separated on the basis of their relative affinities for a low capacity, strongly basic anion exchanger (guard and separator columns). The separated anions are directed through a hollow fiber cation exchanger membrane (fiber suppressor) or micromembrane suppressor bathed in continuously flowing strongly acid solution (regenerant solution). In the suppressor the separated anions are converted to their highly conductive acid forms and the carbonate-bicarbonate eluent is converted to weakly conductive carbonic acid. The separated anions in their acid forms are measured by conductivity. They are identified on the basis of retention time as compared to reference standards. Quantitation is by measurement of peak area or peak height.

EPA, in cooperation with ASTM Committee D-19 on Water, has conducted an interlaboratory validation study of EPA method 300.0 "The Determination of Inorganic Anions in Water by Ion Chromatography", August 1993. The method represents current state-of-the-art for determination of the anions listed above. The method description includes: Sample collection practices, recommended analytical conditions, quality control practices and estimated detection limits for the applicable analytes and performance criteria based on the interlaboratory study data. ASTM, Standard Methods, and AOAC-International have approved the method under their consensus systems and have published the method in their Books of Standards. EPA also proposes to approve the ASTM Method D-4327, as well as, Method 4110 B published in the 18th edition of "Standard Methods for the Examination of Water and Wastewater" (SMEWW) and AOAC-International Method 993.30 all of which were derived from EPA Method 300.0.

F. Cr (VI) by Ion Chromatography

Using the IC Cr(VI) Method (Method 218.6), an aqueous sample is filtered through a 0.45 μm filter and the filtrate is adjusted to a pH of 9 to 9.5 with a buffer solution. A measured volume of sample (50–250 μl) is introduced into the ion chromatograph. A guard column is employed to remove organics from the sample prior to separation of Cr(VI) as CrO_4^{-2} on an anion exchange separator column. Cr(VI) is determined by post column derivatization with diphenylcarbazide and passing through a low-volume flow-through cell and detection of the colored complex with a visible lamp detector at 530 nm.

EPA, in cooperation with ASTM Committee D-19 on Water, has conducted an interlaboratory validation study of EPA Method 218.6 "Determination of Dissolved Hexavalent Chromium in Drinking Water, Ground Water, and Industrial Wastewater Effluents by Ion Chromatography". The method description includes: Sample collection practices, recommended analytical conditions, quality control practices and method detection limits for Cr(VI), as well as performance criteria based on the interlaboratory study data.

ASTM, Standard Methods, and AOAC-International have approved this method as a standard test method under their consensus systems and have published it in their manuals of methods. EPA also proposes to approve the ASTM Method D-5257, Standard Methods Method 3500-Cr E, and

Method 993.23 from the AOAC-International Official Methods of Analysis, 16th Edition. All three of these methods were derived from EPA Method 218.6.

G. Method for Determination of Low Level Residual Chlorine

EPA is proposing to additionally approve SMEWW, Method 4500-Cl E for the detection and quantification of low levels of chlorine in water. This method is a minor modification of the approved amperometric Method 4500-Cl D and is capable of measuring down to 10 µg/L chlorine. Federal and State permitting authorities have requested such a method in order to assess compliance with effluent limits based on EPA and State water quality criteria for chlorine. Supporting performance data for the method can be found at Journal of the Water Pollution Control Federation, Vol. 51, pages 2636-2640 (1979), a copy of which is included in the docket for this proposal.

H. Replacement of Mercury Catalyst in Total Kjeldahl Nitrogen Methods

Due to demonstrated toxic hazards of mercuric sulfate, and difficulty of disposal of laboratory wastes, EPA is proposing to replace this chemical with copper sulfate in the total Kjeldahl Nitrogen Methods. Copper sulfate exhibits significantly less toxicity than mercuric sulfate. The European community has already eliminated mercuric chloride from their total nitrogen methods in favor of less toxic catalysts.

I. EPA is proposing to amend EPA Method 180.1—Turbidity by accepting the use of styrene divinylbenzene beads (AMCO-AEPA-1 Standard) as a substitute for the presently used formazine standard, which is prepared from hydrazine sulfate. The AEPA-1 Standard is an alternate to formazine in the previously approved Standard Methods, Method 2130 B. This substitution will eliminate the need to use hydrazine sulfate, a known carcinogen.

J. EPA is proposing to delete liquid-liquid extraction (LLE) methods, including EPA Methods 611 and 625 and Standard Methods Method 6410 B, as approved procedures for 1,2-dichlorobenzene, 1,3-dichlorobenzene, and 1,4-dichlorobenzene. While these compounds can be determined by these LLE methods, due to their volatility, significant losses can occur when using the prescribed sample collection procedures in the LLE methods resulting in low recovery of these compounds. These compounds are more properly analyzed by EPA Method 624

or Method 1625 (an isotope-dilution method that compensates for any evaporation losses).

K. EPA proposes to delete extraneous method citations, including those EPA methods that reference another source for method details and certain older EPA methods that are readily available from another source. Some measurement technologies are proposed for removal because they are no longer supported by the group that authored them or they use toxic reagents. EPA plans to provide an 18-month transition period after final rule to permit continued use of these methods following the practice of the drinking water program (see 59 FR 62456). These deletions include: colorimetric methods for cadmium, lead and zinc (dithizone), copper (neocuprione), nickel (heptoxime), nitrate-nitrite (automated hydrazine), potassium (cobaltinitrite), vanadium (gallic acid); AA furnace methods for gold, iridium, platinum, rhodium, ruthenium, titanium, and zinc; flame photometric methods for potassium and sodium; voltametry for cadmium and lead; titration (mercuric nitrate) for chloride; and the AA chelation-extraction for total chromium. Table IB of Part 136(a) would be further revised to reorder and clarify footnotes and method citations.

IV. Request for Comments

The Agency requests general comments on its policy to remove or reject methods from this regulation on the basis of pollution prevention when comparable approved methods are available. Comments on the specific methods removed today are also solicited, as well as, comments on other pollution prevention changes that should be considered by the Agency. EPA considered the elimination of the Nessler method for Total Kjeldahl because the Nessler reagent contains 10% mercuric iodide. However, an assessment of the data submitted by participants in recent Water Pollution (WP) performance evaluation studies indicated that the Nessler method is broadly used and produces above-average data compared to the other approved methods. Further, the Nessler method may be necessary to this regulation because it is more sensitive than the other methods. The Agency requests comments on whether the Nessler methods should be removed from Part 136.

V. Regulatory Requirements

A. Executive Order 12866

Under Executive Order 12866, EPA must determine whether a regulation is

“significant” and, therefore subject to OMB review and the requirements of the Executive Order. EPA has determined that this regulation is not a “significant regulatory action” under the terms of Executive Order 12866 and is therefore not subject to OMB review. This rule will not result in an effect on the economy of \$100 million or more, a significant increase in cost or prices, or any of the effects described in the Executive Order. This proposed rule simply specifies analytical techniques which may be used by laboratories in measuring concentrations of certain metals and, therefore, has no adverse economic impacts.

B. Regulatory Flexibility Act

This proposed amendment is consistent with the objectives of the Regulatory Flexibility Act (5 U.S.C. 601 *et seq.*) because it will not have a significant economic impact on a substantial number of small entities. The procedures proposed in this rule give all laboratories the flexibility to use the new proposed procedures or already approved alternate procedures.

C. Paperwork Reduction Act

This proposed rule contains no requests for information activities and, therefore, no information collection request (ICR) was submitted to the Office of Management and Budget (OMB) for review in compliance with the Paperwork Reduction Act, 44 U.S.C. 3501 *et seq.*

D. Unfunded Mandates

Under Section 202 of the Unfunded Mandates Reform Act of 1995, signed into law on March 22, 1995, EPA must prepare a written statement to accompany rules 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 such a rule and that is consistent with statutory requirements. Section 203 requires EPA to establish a plan for informing and advising any small governments that may be significantly and uniquely affected by the rule.

EPA estimates that the costs to State, local or tribal governments, or the private sector, from this proposed rule will be far less than \$100 million. This proposed rule should have minimal impact, if any, on the existing regulatory burden imposed on NPDES permittees required to monitor for regulated pollutants because the proposed rule would merely make additional options

available to the laboratory analyst conducting an existing approved test method. EPA has determined that an unfunded mandates statement therefore is unnecessary. Similarly, the methods proposed today do not establish any regulatory requirements that might significantly or uniquely affect small governments.

List of Subjects in 40 CFR Part 136

Environmental protection,
Incorporation by reference, Water
pollution control.

Dated: October 10, 1995.
Carol M. Browner,
Administrator.

In consideration of the preceding, EPA proposes to amend part 136 of title 40 of the Code of Federal Regulations as follows:

PART 136—[AMENDED]

1. The authority citation for part 136 continues to read as follows:

Authority: Secs. 301, 304(h), 307, and 501(a) Public Law 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).

2. Section 136.3(a) is proposed to be amended by revising Table IB to read as follows:

§ 136.3 Identification of test procedures.

* * * * *

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, Units and Method	EPA ¹	Reference (Method No. or Page)			AOAC-inter-national ³ and others
		Std. Methods 18th Ed.	ASTM	USGS ²	
10. Boron ⁸ —Total, mg/L: Colorimetric (curcumin) ICP/AES, or DCP.	200.7	4500-B B 3120 B	D4190-82(88)	I-3112-85	Note 16
11. Bromide, mg/L; Titrimetric, or Ion Chromatography	300.0	4110 B	D1246-82(88)(C) D4327-91	I-1125-85	p. S44 ¹⁹ 993.30
12. Cadmium—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration AA graphite furnace ICP/AES ICP/MS, or DCP	200.9 200.7 200.8	3111 B or C 3113 B 3120 B	D3557-90(A or B) D3557-90(D) D4190-82(88)	I-3135-85 or I-3136-85	974.2 p. 37 ¹⁸ 993.14 Note 16
13. Calcium—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration ICP/AES DCP, or Titrimetric (EDTA)	200.7	3111 B 3120 B	D511-93(B) D511-93(A)	I-3152-85	Note 16
14. Carbonaceous biochemical oxygen demand (CBOD ⁵), mg/L ⁹ Dissolved Oxygen Depletion with nitrification inhibitor	410.3 ¹⁵ 410.4	5220 B 5220 D	D1252-88(A) D1252-88(B)	I-3560-85, I-3562-85 I-3561-85	973.46, p. 17 ¹⁸ Notes 20, 21
15. Chemical oxygen demand (COD), mg/L: Titrimetric, or Spectrophotometric, manual or automated		4500-CI B	D512-89(B)	I-1183-85 I-1187-85 I-2187-85	993.30
16. Chloride, mg/L: Titrimetric (silver nitrate) Colorimetric, Manual, or Automated (Ferricyanide), or Ion Chromatography	300.0	4500-CI-E 4110 B	D4327-91		
17. Chlorine—Total residual, mg/L: Titrimetric: Amperometric direct Iodometric direct Back titration either end-point ¹⁰ DPD-FAS Spectrophotometric (DPD), or Electrode		4500-CI D or E 4500-CI B 4500-CI C 4500-CI F 4500-CI G	D1253-86(92)		Note 22
18. Chromium VI dissolved, mg/L: 0.45 micron filtration followed by: AA chelation-extraction Ion Chromatography, or Colorimetric (Diphenylcarbazide)	218.6	3111 C 3500-Cr E ²³ 3500-Cr D	D5257-93 D1687-92(A)	I-1232-85 I-1230-85	993.23
19. Chromium—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration AA graphite furnace ICP/AES ICP/MS, or DCP Colorimetric (Diphenylcarbazide)	200.9 200.7 200.8	3113 B 3120 B	D1687-92(B) D1687-92(C) D4190-82(88)	I-3236-85	974.27 993.14 Note 16
20. Cobalt—Total ⁴ , mg/L: Colorimetric (Diphenylcarbazide)		3500-Cr D			

Digestion ⁵ followed by: AA direct aspiration AA graphite furnace ICP/AES ICP/MS, or DCP	200.9 200.7 200.8	3111 B or C 3113 B 3120 B	D3558-90(A or B) D3558-90(C) D4190-82(88)	I-3239-85	p.37 ¹⁸ 993.14 Note 16
21. Color, platinum cobalt units or dominant wavelength, hue, luminance purity: Colorimetric (ADMI) Colorimetric (Platinum cobalt), or Spectrophotometric Copper—Total ⁴ , mg/L: Digestion ^{5,6} followed by: AA direct aspiration AA graphite furnace ICP/AES ICP/MS DCP, or Colorimetric (Bicinchoninate)	2120 E 2120B 2120 C	2120 E 2120B 2120 C	D1688-90(A or B) D1688-90(C) D4190-82(88)	I-3270-85 or I-3271-85	974.27p.37 ¹⁸
22. Copper—Total ⁴ , mg/L: Digestion ^{5,6} followed by: AA direct aspiration AA graphite furnace ICP/AES ICP/MS DCP, or Colorimetric (Bicinchoninate)	200.9 200.7 200.8	3111 B or C 3113 B 3120 B	D2036-91(A) D2036-91(A) D2036-91(B)	I-3300-85	993.14 Note 16 Note 25
23. Cyanide—Total, mg/L: Manual distillation ¹¹ with MgCl ₂ followed by: Titrimetric Spectrophotometric, manual or Spectrophotometric, automated	335.4 335.4	4500-CN C 4500-CN D 4500-CN E	D2036-91(A) D2036-91(A) D2036-91(B)	I-3300-85	p.22 ¹⁹
24. Cyanide amenable to chlorination, mg/L: Manual distillation with MgCl ₂ , followed by titrimetric or spectrophotometric.	335.4	4500-CN G	D2036-91(B)	I-3300-85	p.22 ¹⁹
25. Fluoride—Total, mg/L: Manual distillation ⁷ , followed by: Electrode, Manual Electrode, Automated Colorimetric (SPADNS) Colorimetric (Auto complexone), or Ion Chromatography	300.0	4500-F B 4500-F C 4500-F D 4500-F E 4110 B	D1179-93(B) D1179-93(A) D4327-91	I-4327-85	993.30
26. Gold—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration, or DCP	300.0	3111 B	D4327-91	I-4327-85	993.30
27. Hardness—Total, as CaCO ₃ , mg/L: Automated colorimetric Titrimetric (EDTA), or Ca plus Mg as their carbonates, by ICP/AES, AA direct aspiration, or DCP. (Parameters 13 and 33).	130.1 ¹⁵	2340 B or C	D1126-86(92)	I-1338-85	973.52B
28. Hydrogen ion (pH), pH units: Electrometric measurement, or Automated electrode	150.2 ¹⁵	4500-H+ B	D1293-84(90) (A or B)	I-1586-85	973.41 Note 26
29. Iridium—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration	150.2 ¹⁵	3111 B	D1293-84(90) (A or B)	I-1586-85	973.41 Note 26
30. Iron—Total ⁴ , mg/L: Digestion ^{5,6} followed by: AA direct aspiration AA graphite furnace ICP/AES DCP, or Colorimetric (Phenanthroline)	200.9 200.7	3111 B D 3113 B 3120 B	D1068-90(A or B) D1068-90(C) D4190-82(88)	I-3381-85	974.27 Note 16 Note 27
31. Kjeldahl nitrogen—Total, (as N), mg/L: Digestion ¹² and distillation.	200.9 200.7	3111 B D 3113 B 3120 B	D1068-90(A or B) D1068-90(C) D4190-82(88)	I-3381-85	974.27 Note 16 Note 27

Ion Chromatography, or Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40)	300.0	4110 B	D4327-91	993.30
39. Nitrate-nitrite (sum as N), mg/L: Cadmium reduction, Manual Cadmium reduction, Automated, or Ion Chromatography	353.2 300.0	4500-NO ₃ E 4500-NO ₃ F 4110 B	D3867-90(B) D3867-90(A) D4327-91	993.30
40. Nitrite (as N), mg/L: Spectrophotometric, Manual, or Automated (Diazotization) Cadmium reduction, Manual Cadmium reduction, Automated, or Ion Chromatography	300.0	4500-NO ₂ B 4500-NO ₃ E 4500-NO ₃ F 4110 B I-4540-85 I-4545-85	Note 32 993.30
41. Oil and grease—Total recoverable, mg/L: Gravimetric (extraction)	413.1	5520-B ³³	973.47, p.14 ³⁵
42. Organic carbon—Total (TOC), mg/L: Combustion or oxidation	5310-B,C or D	D2579-93(A or B)
43. Organic nitrogen (as N), mg/L: Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4)
44. Orthophosphate (as P), mg/L: Ascorbic acid method: Automated, or Manual single reagent Manual two reagent, or Ion Chromatography	365.1 365.3 ¹⁵ 300.0	4500-P F 4500-P E 4110 B D515-88(A) D4327-91	973.56 973.55 993.30
45. Osmium—Total, ⁴ mg/L: Digestion ⁵ followed by: AA direct aspiration, or AA graphite furnace	3111 D
46. Oxygen dissolved, mg/L: Winkler (Azide modification), or Electrode	4500-O C 4500-O G	D888-92(A) D888-92(B)	973.45B
47. Palladium—Total, ⁴ mg/L: Digestion ⁵ followed by: AA direct aspiration, AA graphite furnace, or DCP	3111 B	p.S27 ¹⁹ Note 16
48. (Reserved)
49. Phenols, mg/L: Manual distillation, ¹³ followed by Colorimetric (4AAP) manual, or Colorimetric (4AAP) Automated	¹⁵ 420.1 420.4	Note 35
50. Phosphorus (elemental), mg/L Gas-liquid chromatography	Note 36
51. Phosphorus—Total, mg/L: Persulfate digestion followed by: Ascorbic acid reduction, Manual, or Automated 365.3 ¹⁵ 365.1	4500-P B,5 4500-P E 4500-P F	D515-88(A) D515-88(B)	973.55 973.56
52. Semi-automated block digester Platinum—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration, or DCP	3111 B	Note 16
53. Potassium—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration, or ICP/AES	200.7	3111 B 3120 B I-3630-85	973.53
54. Residue—Total, mg/L: Gravimetric, 103-105°	2540 B
55. Residue—filterable, mg/L: Gravimetric, 180°	2540 C

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, Units and Method	EPA ¹	Reference (Method No. or Page)			AOAC-inter-national ³ and others
		Std. Methods 18th Ed.	ASTM	USGS ²	
56. Residue—nonfilterable (TSS), mg/L: Gravimetric, 103–105° post washing of residue		2540 D		I-3765–85	
57. Residue—settleable, mg/L: Volumetric (Imhoff cone), or Gravimetric		2540 F			
58. Residue—Volatile, mg/L: Gravimetric, 550° C	160.4 ¹⁵			I-3753–85	
59. Rhodium—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration		3111 B			
60. Ruthenium—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration		3111 B			
61. Selenium—Total ⁴ , mg/L: Digestion ^{5,6} followed by: AA graphite furnace	200.9	3113 B	D3859–93(B)		
AA gaseous hydride	200.7	3114 B	3859–93(A)	I-3667–85	
ICP/AES, or	200.8	3120 B			993.14
ICP/MS					
62. Silica ⁸ Dissolved, mg/L: 0.45 micron filtration followed by: Colorimetric, Manual, or Automated (Molybdate), or ICP	200.7	4500–Si D	D859–88	I-1700–85 I-2700–85	
63. Silver—Total ⁴ , mg/L: Digestion ^{5,14} followed by: AA direct aspiration		3111 B or C			974.27, p.37 ¹⁸
AA graphite furnace	200.9	3113 B			
ICP/AES	200.7	3120 B			993.14
ICP/MS, or	200.8				Note 16
DCP					
64. Sodium—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration		3111 B		I-3735–85	973.54
ICP/AES, or	200.7	3120 B			Note 16
DCP					
65. Specific conductance, micromhos/cm at 25°C: Wheatstone bridge		2510 B	D1125–91(A)	I-1780–85	973.40
66. Sulfate (as SO ₄), mg/L: Colorimetric, Automated (Barium chloranilate)	375.1 ¹⁵	4500–SO ₄ ⁻² C or D			925.54
Gravimetric		4500–SO ₄ ⁻² E	D516–90		993.30
Turbidimetric, or Ion Chromatography	300.0	4110 B	D4327–91		
67. Sulfide (as S), mg/L: Titrimetric (iodine), or Colorimetric (methylene blue)		4500–S ⁻² E		I-3840–85	
Colorimetric (methylene blue)		4500–S ⁻² D			
68. Sulfite (as SO ₃), mg/L: Titrimetric (iodine-iodate)		4500–SO ₃ ⁻² B			
69. Surfactants, mg/L: Colorimetric (methylene blue)		5540 C	D2330–88		
70. Temperature, °C:					

Thermometric	2550 B	Note 37
71. Thallium—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration	3111 B
AA graphite furnace	200.9
ICP/AES, or	200.7	3120 B	993.14
ICP/MS	200.8
72. Tin—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration, AA graphite furnace, or	3111 B
ICP/AES	200.9	3113 B
ICP/AES	200.7
73. Titanium—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration	3111 D	Note 16
ICP/AES, or	200.7
DCP
74. Turbidity, NTU: Nephelometric	2130 B	D1889-88(A)	I-3860-85
75. Vanadium—Total ⁴ , mg/L: Digestion ⁵ followed by: AA direct aspiration	180.1
AA graphite furnace	3111 D	D3373-93
ICP/AES
ICP/MS, or	200.7	3120 B	993.14
DCP	200.8	D4190-82(88)	Note 16
76. Zinc—Total ⁴ , mg/L: Digestion ^{5,6} followed by: AA direct aspiration	3111 (B or C)	D1691-90 (A or B)	974.27, p. 37 ¹⁸
ICP/AES	200.7	3120 B	993.14
ICP/MS	200.8	D4190-82(88)	Note 16
DCP, or	Note 38
Colorimetric (zinccon)	3500-Zn F

Table IB—Notes

- ¹"Methods for the Determination of Metals in Environmental Samples", Supplement 1, EMSL-Cl, EPA/600/R-94/111, May 1994; and "Methods for the Determination of Inorganic Substances in Environmental Samples", EMSL-Cl, EPA/600/R-93/100, August, 1993.
- ²Fishman, M. J., et al., "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments", U.S. Department of the Interior, Techniques of Water—Resource Investigations of the U.S. Geological Survey (USGS), Denver, CO, Revised 1989.
- ³"Official Methods of Analysis of the Association of Official Analytical Chemists", methods manual, 16th ed (1995).
- ⁴For the determination of total metals the sample is not filtered before processing and a digestion procedure is required a) to solubilize analytes in the suspended material and b) to render the analyte available for analysis when colorimetric methods are used.
- ⁵For non-platform graphite furnace atomic absorption determinations a digestion using nitric acid is required prior to analysis. The procedure used should subject the sample to gentle, acid refluxing and at no time should the sample be taken to dryness. For direct aspiration flame atomic absorption determinations (FLAA) a combination acid (nitric and hydrochloric acids) digestion is preferred prior to analysis. Such a digestion is described as Method 200.2 in Supplement I of "Methods for the Determination of Metals in Environmental Samples" EPA-600/R-94/111 May, 1994. However, when using the gaseous hydride technique or for the determination of certain elements such as antimony, arsenic, selenium, silver, and tin by non-EPA graphite furnace atomic absorption methods, mercury by cold vapor atomic absorption, the noble metals and titanium by FLAA, a specific or modified sample digestion procedure may be required and in all cases the referenced method write-up should be consulted for specific instruction and/or cautions. For analyses using inductively coupled plasma-atomic emission spectrometry (ICP-AES), the direct current plasma (DCP) technique or the EPA spectrochemical techniques (platform furnace AA, ICP-AES, and ICP-MS) the appropriate sample digestion procedure to be used is included in the referenced Standard Methods, ASTM, Fisons, or EPA methods, respectively.
- ⁶The digestion procedure "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", CEM Corporation, P.O. Box 200, Matthews, NC 28106-0200, April 16, 1992, is acceptable for use with any AA direct aspiration, ICP/AES, or DCP measurement system listed for this parameter. Available from the CEM Corporation.
- ⁷Manual 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.
- ⁸When determining boron and silica, only plastic, PFTE, or quartz labware may be used from start until completion of the analysis.
- ⁹Carbonaceous biochemical oxygen demand (CBOD₅) must not be confused with the traditional BOD₅ test which measures "total BOD". The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD₅ parameter. A discharger whose permit reporting the traditional BOD₅ may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD₅ is required can the permittee report data using the nitrification inhibitor.
- ¹⁰The back titration method will be used to resolve controversy.
- ¹¹When using EPA digestion procedure, must use EPA determinative procedure. When using Standard Methods digestion procedure, must use Standard Methods determinative procedure.
- ¹²Copper sulfate or other mercury-free catalyst shown to be effective must be used in place of mercuric sulfate in the digestion step.

- ¹³ Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.
- ¹⁴ The digestion procedures in the approved Standard Methods direct aspiration flame atomic absorption, and inductively coupled plasma-atomic emission spectrometry methods are useful for the analysis of silver up to concentrations of 1 mg/l. However, where silver concentrations > 1 mg/L exist as an inorganic halide the referenced methods sample preparation procedures are inadequate. For sample preparation in these situations a well-mixed representative 20 ml sample aliquot should be diluted to 100 mL by adding 40 mL each of 2 M Na₂S₂O₃ and NaOH prior to analysis by direct aspiration flame atomic absorption. Calibration standards should be prepared in the same manner. The approved EPA methods are useful for the analysis of silver up to concentrations of 0.1 mg/L.
- ¹⁵ "Methods for Chemical Analysis of Water and Wastes", U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory-Cincinnati (EMSL-CI), Cincinnati, OH, EPA-600/4-79-020, Revised March 1983 and 1979 where applicable.
- ¹⁶ "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AE50029", 1986—Revised 1991, Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923.
- ¹⁷ Ammonia, Automated Electrode Method, Industrial Method Number 379-75 WE, dated February 19, 1976, Bran & Luebbe (Technicon) Auto Analyzer II, Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.
- ¹⁸ American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.
- ¹⁹ "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).
- ²⁰ OIC Chemical Oxygen Demand Method Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, TX 77840.
- ²¹ Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- ²² Orion Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138.
- ²³ "Standard Method for the Examination of Water and Wastewater—Supplement to the 18th Edition", 1994.
- ²⁴ National Council of the Paper Industry for Air and Stream Improvement (Inc.), Technical Bulletin 253, December 1971.
- ²⁵ Copper, Biocinchoninate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- ²⁶ Hydrogen ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA, October 1976, Technicon AutoAnalyzer II, Technicon Industrial Systems, Tarrytown, NY 10591.
- ²⁷ Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- ²⁸ Nitrogen, Total Kjeldahl, Method PAI-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection), revised 12/22/94, Perstop Analytical.
- ²⁹ Nitrogen, Total Kjeldahl, Method PAI-DK02 (Block Digestion, Steam Distillation, Colorimetric Detection), revised 12/22/94, Perstop Analytical.
- ³⁰ Nitrogen, Total Kjeldahl, Method PAI-DK03 (Block Digestion, Automated FIA Gas Diffusion), revised 12/22/94, Perstop Analytical.
- ³¹ Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2-113 and 2-117, Hach Chemical Company, Loveland, CO 80537.
- ³² Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.
- ³³ Only the trichlorofluoroethane extraction solvent option is approved.
- ³⁴ Wershaw, R.L., et al, "Methods for Analysis of Organic Substances in Water", Techniques of Water-Resources Investigation of the U.S. Geological Survey, Book 5, Chapter A3 (1972 Revised 1987), p.14.
- ³⁵ The approved method is 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.
- ³⁶ 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.
- ³⁷ 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.
- ³⁸ Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2-231 and 2-333, Hach Chemical Company, Loveland, CO 80537.

3. Section 136.3(a) is proposed to be amended by revising entries 35–37 of Table IC to read:

136.3 Identification of Test Procedures.

* * * * *

TABLE 1C.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter ¹	EPA Method Number ^{2,7}			Standard methods 18th Ed.	ASTM	Other
	GC	GC/MS	HPLC			
* * * * *						
35. 1,2-Dichlorobenzene	601,602	624, 1625	—	6220 B, 6230 B.		
36. 1,3-Dichlorobenzene	601,602	624, 1625	—	6220 B, 6230 B.		
37. 1,4-Dichlorobenzene	601,602	624, 1625	—	6220 B, 6230 B.		
* * * * *						

Table 1C 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.

* * * * *

⁷ Each Analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601–603, 624, 625, 1624, and 1625 (See Appendix A of this Part 136) in accordance with procedures in section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis 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.”

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4. Section 136.3(b) is proposed to be amended by revising references 1 through 38 and adding references 39 and 40 to read as follows:

§ 136.3 Identification of test procedures.

* * * * *
(b) * * *

References, Sources, Costs, and Table Citations

(1) The full text 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. Table IC, Note 7; and Table ID, Note 2.

(2) “Microbiological Methods for Monitoring the Environment, Water and Wastes,” U.S. Environmental Protection Agency, EPA–600/8–78–017, 1978. Available from: ORD Publications, CERL, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268. Table IA, Note 2.

(3) “Methods for Chemical Analysis of Water and Wastes,” U.S. Environmental Protection Agency, EPA 600/4–79–020, March 1979, or “Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, EPA–600/4–79–020, Revised March 1983. Available from: ORD Publications, CERL, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268. Table IB, Note 15.

(4) “Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater,” U.S. Environmental Protection Agency, 1978. Available from: ORD Publications, CERL, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Table IC, Note 3; Table D, Note 3.

(5) “Prescribed Procedures for Measurement of Radioactivity in Drinking Water,” U.S. Environmental Protection Agency, EPA–600/4–80–032, 1980. Available from: ORD Publications, CERL, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Table IE, Note 1.

(6) “Standard Methods for the Examination of Water and Wastewater”, Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Environment Federation, 18th Edition, 1992. Available from: American Public Health Association, 1015 Fifteenth Street, NW., Washington, DC 20005, Cost \$160.00. Table IA, IB, IC, ID and IE.

(7) (Reserved)

(8) *Ibid*, 14th Edition, 1975. Table IB, Note 35.

(9) “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 NW.,

Washington, DC 20036. Cost available from publisher. Table IB, Note 19; Table IC, Note 6; Table ID, Note 6.

(10) “Annual Book of Standards—Water and Environmental Technology”, Section 11, Parts 11.01 Water (I) and 11.02 Water (II), American Society for Testing and Materials, 1994. 1916 Race Street, Philadelphia, PA 19103. Cost available from publisher. Tables IB, IC, ID, and IE.

(11) “Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples,” edited by Britton, L.J. and P.E. Greason, Techniques of Water Resources Investigations, of the U.S. Geological Survey, Book 5, Chapter A4 (1989). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: \$9.25 (subject to change). Table IA.

(12) “Methods for Determination of Inorganic Substances in Water and Fluvial Sediments,” by M.J. Fishman and Linda C. Friedman, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5 Chapter A1 (1989). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost \$108.75 (subject to change). Table IB, Note 2.

(13) (Reserved)

(14) “Methods for the Determination of Organic Substances in Water and Fluvial Sediments, Wershaw, R.L., et al., Techniques of Water-Resources

Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425. Cost: \$0.90 (subject to change). Table IB, Note 34, Table ID, Note 4.

(15) "Water Temperature-Influential Factors, Field Measurement and Data Presentation," by H.H. Stevens, Jr., J. Ficke, and G.F. Smoot, Techniques of Water Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1, 1975. Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: \$1.60 (subject to change). Table IB, Note 37.

(16) "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). Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Cost: \$13.50 (subject to change). Table IE, Note 2.

(17) "Official Methods of Analysis of AOAC-International", 16th Edition (1995). Price: \$359.00. Available from: AOAC-International, 1970 Chain Bridge Rd., Dept. 0742, McLean, VA 22109-0742. Table IB, Note 3.

(18) "American National Standard on Photographic Processing Effluents," April 2, 1975. Available from: American National Standards Institute, 1430 Broadway, New York, New York 10018. Table IB, Note 18.

(19) "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, New York, NY 10016. Cost available from publisher. Table IB, Note 24.

(20) Ammonia, Automated Electrode Method, Industrial Method Number 379-75WE, dated February 19, 1976. Technicon Auto Analyzer II. Method and price available from Technicon Industrial Systems, Tarrytown, New York 10591. Table IB, Note 17.

(21) Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Method price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 21.

(22) OIC Chemical Oxygen Demand Method, 1978. Method and price available from Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840. Table IB, Note 20.

(23) ORION Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977. Method and price available from ORION Research Incorporation, 840 Memorial Drive, Cambridge, Massachusetts 02138. Table IB, Note 22.

(24) Bicinchoninate Method for Copper. Method 8506, Hach Handbook of Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 300, Loveland, Colorado 80537. Table IB, Note 25.

(25) Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378-75WA. October 1976. Bran & Luebbe (Technicon) Auto Analyzer II. Method and price available from Bran & Luebbe Analyzing Technologies, Inc. Elmsford, N.Y. 10523. Table IB, Note 26.

(26) 1,10-Phenanthroline Method using FerroVer Iron Reagent for Water, Hach Method 8008, 1980. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 27.

(27) 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. Table IB, Note 31.

(28) Nitrogen, Nitrite-Low Range, Diazotization Method for Water and Wastewater, Hach Method 8507, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 32.

(29) 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. Table IB, Note 38.

(30) "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 Center, 52 Vanderbilt Avenue, New York, NY 10164. Cost available from publisher, Table IB, Note 36.

(31) "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes", Method AES 0029, 1986-Revised 1991. Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923. Table IB, Note 16.

(32) "Closed Vessel Microwave Digestion of Wastewater Samples for

Determination of Metals, CEM Corporation, P.O. Box 200, Matthews, North Carolina 28106-0200, April 16, 1992. Available from the CEM Corporation. Table IB, Note 6.

(33) Reserved.

(34) "Organochlorine Pesticides and PCB's in Wastewater Using Empore™ Disk", Test Method 3M 0222, Revised 10/28/94. 3M Corporation, 3M Center Building 220-9E-10, St. Paul, MN 55144-1000. Method available from 3M Corporation. Tables IC and ID, Note 9.

(35) "Nitrogen, Total Kjeldahl, Method PAI-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection)", revised 12/22/94, Perstorp Analytical. Method available from Perstorp Analytical Corporation, P.O. Box 648, Wilsonville, OR 97070. Table IB, Note 28.

(36) "Nitrogen, Total Kjeldahl, Method PAI-DK02 (Block Digestion, Steam Distillation, Colorimetric Detection)", revised 12/22/94, Perstorp Analytical. Method available from Perstorp Analytical Corporation, P.O. Box 648, Wilsonville, OR 97070. Table IB, Note 29.

(37) "Nitrogen, Total Kjeldahl, Method PAI-DK03 (Block Digestion, Automated FIA Gas Diffusion)", revised 12/22/94, Perstorp Analytical. Method available from Perstorp Analytical Corporation, P.O. Box 648, Wilsonville, OR 97070. Table IB, Note 30.

(38) Methods for the Determination of Metals in Environmental Samples, Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268, EPA 600 R-94/111, May 1994. Table IB, Notes 1 and 5.

(39) "Methods for the Determination of Inorganic Substances in Environmental Samples", Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268, Revised EPA 600 R-93/100 August 1993. Table IB, Note 1.

(40) "Standard Methods for the Examination of Water and Wastewater", American Public Health Association, American Water Works Association, Water Environment Federation, 18th Edition Supplement, 1994. Table IB, Note 23.

5. Section 136.3 (e) is proposed to be amended by revising Table II to read as follows:

136.3 Identification of test procedures.

* * * * *

(e) * * *

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

Test procedure table parameter number/name	Container ¹	Preservation procedure ²⁻⁴	Maximum holding time ⁵
Table IA—Biological Tests			
1. Coliform (fecal)	P,G	a,b ⁶	6 hours.
2. Coliform (fecal), Pres. of chlorine	P,G	a,b ⁶	6 hours.
3. Coliform (total)	P,G	a,b ⁶	6 hours.
4. Coliform (total), Pres. of chlorine	P,G	a,b ⁶	6 hours.
5. Fecal streptococci	P,G	a,b ⁶	6 hours.
Table IB—Inorganic Tests			
1. Acidity	P,G	a	14 days.
2. Alkalinity	P,G	a	14 days.
3. Aluminum ⁷	P,G	c	6 months.
4. Ammonia	P,G	a,d	28 days.
5. Antimony ⁷	P,G	c	6 months.
6. Arsenic ⁷	P,G	c	6 months.
7. Barium ⁷	P,G	c	6 months.
8. Beryllium ⁷	P,G	c	6 months.
9. Biochemical oxygen demand (BOD)	P,G	a	48 hours.
10. Boron ⁷	P,G	c	6 months.
11. Bromide	P,G	e	28 days.
12. Cadmium ⁷	P,G	c	6 months.
13. Calcium ⁷	P,G	c	6 months.
14. Carbonaceous biochemical oxygen demand (CBOD)	P,G	a	48 hours.
15. Chemical oxygen demand (COD)	P,G	a,d	28 days.
16. Chloride	P,G	e	28 days.
17. Chlorine	P,G	e	Immediate.
18. Chromium VI (dissolved)	P,G	a	24 hours.
19. Chromium ⁷	P,G	c	6 months.
20. Cobalt ⁷	P,G	c	6 months.
21. Color	P,G	a	48 hours.
22. Copper ⁷	P,G	c	6 months.
23. Cyanide	P,G	a,f ⁶ ,g	14 days. ⁸
24. Cyanide amenable	P,G	a,f ⁶ ,g	14 days. ⁸
25. Fluoride	P	e	28 days.
26. Gold ⁷	P,G	c	6 months.
27. Hardness	P,G	c or d	6 months.
28. Hydrogen ion (pH)	P,G	e	Immediate.
29. Iridium ⁷	P,G	c	6 months.
30. Iron ⁷	P,G	c	6 months.
31. Kjeldahl nitrogen	P,G	a,d	28 days.
32. Lead ⁷	P,G	c	6 months.
33. Magnesium ⁷	P,G	c	6 months.
34. Manganese ⁷	P,G	c	6 months.
35. Mercury	P,G	c	28 days.
36. Molybdenum ⁷	P,G	c	6 months.
37. Nickel ⁷	P,G	c	6 months.
38. Nitrate	P,G	a	48 hours.
39. Nitrate-nitrite	P,G	a,d	28 days.
40. Nitrite	P,G	a	48 hours.
41. Oil and grease	G	a; d or h	28 days.
42. Organic carbon	P,G	a; d or h or i	28 days.
43. Organic nitrogen	P,G	a,d	28 days.
44. Orthophosphate	P,G	a,j	48 hours.
45. Osmium ⁷	P,G	c	6 months.
46. Oxygen, dissolved:			
By probe	G	e	Immediate.
By winkler	G bottle and top	k	8 hours. 4
47. Palladium ⁷	P,G	c	6 months.
48. (Reserved)			
49. Phenols	G	a,d	28 days.
50. Phosphorus (elemental)	G	a	48 hours.
51. Phosphorus	P,G	a,d	28 days.
52. Platinum ⁷	P,G	c	6 months.
53. Potassium ⁷	P,G	c	6 months.
54. Residue, total	P,G	a	7 days.
55. Residue, filterable	P,G	a	7 days.
56. Residue, nonfilterable (TSS)	P,G	a	7 days.
57. Residue, settleable	P,G	a	48 hours.
58. Residue, Volatile	P,G	a	7 days.

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

Test procedure table parameter number/name	Container ¹	Preservation procedure ²⁻⁴	Maximum holding time ⁵
59. Rhodium ⁷	P,G	c	6 months.
60. Ruthenium ⁷	P,G	c	6 months.
61. Selenium ⁷	P,G	c	6 months.
62. Silica ⁷	P	a	28 days.
63. Silver ⁷	P,G	c	6 months.
64. Sodium ⁷	P,G	c	6 months.
65. Specific conductance	P,G	a	28 days.
66. Sulfate	P,G	a	28 days.
67. Sulfide	P,G	a,l,m	7 days.
68. Sulfite	P,G	e	Immediate.
69. Surfactants	P,G	a	48 hours.
70. Temperature	P,G	e	Immediate.
71. Thallium ⁷	P,G	c	6 months.
72. Tin ⁷	P,G	c	6 months.
73. Titanium ⁷	P,G	c	6 months.
74. Turbidity	P,G	a	48 hours.
75. Vanadium ⁷	P,G	c	6 months.
76. Zinc ⁷	P,G	c	6 months.

Table IC—Non-Pesticide Organic Compounds^{9, 10}

1. Acenaphthene	G,T	a,b ⁶ ,n	7 days. ¹¹
2. Acenaphthylene	G,T	a,b ⁶ ,n	7 days. ¹¹
3. Acrolein	G,T	a,o ⁶ ,p	14 days. ¹²
4. Acrylonitrile	G,T	a,o ⁶	14 days.
5. Anthracene	G,T	a,b ⁶ ,n	7 days. ¹¹
6. Benzene	G,T	a,h,o ⁶	14 days.
7. Benzidine	G,T	a,b ⁶ , ¹³	7 days. ¹⁴
8. Benzo(a)anthracene	G,T	a,b ⁶ ,n	7 days. ¹¹
9. Benzo(a)pyrene	G,T	a,b ⁶ ,n	7 days. ¹¹
10. Benzo(b)fluoranthene	G,T	a,b ⁶ ,n	7 days. ¹¹
11. Benzo(g,h,i)perylene	G,T	a,b ⁶ ,n	7 days. ¹¹
12. Benzo(k)fluoranthene	G,T	a,b ⁶ ,n	7 days. ¹¹
13. Benzyl chloride	G,T	a,o ⁶	14 days.
14. Benzyl butyl phthalate	G,T	a	7 days. ¹¹
15. Bis(2-chloroethoxy) methane	G,T	a,b ⁶	7 days. ¹¹
16. Bis(2-chloroethyl) ether	G,T	a,b ⁶	7 days. ¹¹
17. Bis(2-ethylhexyl) phthalate	G,T	a	7 days. ¹¹
18. Bromodichloromethane	G,T	a,o ⁶	14 days.
19. Bromoform	G,T	a,o ⁶	14 days.
20. Bromomethane	G,T	a,o ⁶	14 days.
21. 4-Bromophenylphenyl ether	G,T	a,b ⁶	7 days. ¹¹
22. Carbon tetrachloride	G,T	a,o ⁶	14 days.
23. 4-Chloro-3-methylphenol	G,T	a,b ⁶	7 days. ¹¹
24. Chlorobenzene	G,T	a,o ⁶	14 days.
25. Chloroethane	G,T	a,o ⁶	14 days.
26. 2-Chloroethylvinyl ether	G,T	a,o ⁶	14 days.
27. Chloroform	G,T	a,o ⁶	14 days.
28. Chloromethane	G,T	a,o ⁶	14 days.
29. 2-Chloronaphthalene	G,T	a	7 days. ¹¹
30. 2-Chlorophenol	G,T	a,b ⁶	7 days. ¹¹
31. 4-Chlorophenylphenyl ether	G,T	a,b ⁶	7 days. ¹¹
32. Chrysene	G,T	a,b ⁶ ,n	7 days. ¹¹
33. Dibenzo(a,h)anthracene	G,T	a,b ⁶ ,n	7 days. ¹¹
34. Dibromochloromethane	G,T	a,o ⁶	14 days.
35. 1,2-Dichlorobenzene	G,T	a,o ⁶	14 days.
36. 1,3-Dichlorobenzene	G,T	a,o ⁶	14 days.
37. 1,4-Dichlorobenzene	G,T	a,h,o ⁶	14 days.
38. 3,3-Dichlorobenzidine	G,T	a,b ⁶	7 days. ¹³
39. Dichlorodifluoromethane	G,T	a,o ⁶	14 days.
40. 1,1-Dichloroethane	G,T	a,o ⁶	14 days.
41. 1,2-Dichloroethane	G,T	a,o ⁶	14 days.
42. 1,1-Dichloroethene	G,T	a,o ⁶	14 days.
43. trans-1,2-Dichloroethene	G,T	a,o ⁶	14 days.
44. 2,4-Dichlorophenol	G,T	a,b ⁶	7 days. ¹¹
45. 1,2-Dichloropropane	G,T	a,o ⁶	14 days.
46. cis-1,3-Dichloropropene	G,T	a,o ⁶	14 days.
47. trans-1,3-Dichloropropene	G,T	a,o ⁶	14 days.
48. Diethyl phthalate	G,T	a	7 days. ¹¹
49. 2,4-Dimethylphenol	G,T	a,b ⁶	7 days. ¹¹
50. Dimethyl phthalate	G,T	a	7 days. ¹¹

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

Test procedure table parameter number/name	Container ¹	Preservation procedure ²⁻⁴	Maximum holding time ⁵
51. Di-n-butyl phthalate	G,T	a	7 days. ¹¹
52. Di-n-octyl phthalate	G,T	a	7 days. ¹¹
53. 2,4-Dinitrophenol	G,T	a,b ⁶	7 days. ¹¹
54. 2,4-Dinitrotoluene	G,T	a,b ⁶ ,n	7 days. ¹¹
55. 2,6-Dinitrotoluene	G,T	a,b ⁶ ,n	7 days. ¹¹
56. Epichlorohydrin	G,T	a,o ⁶	14 days.
57. Ethylbenzene	G,T	a,h,o ⁶	14 days.
58. Fluoranthene	G,T	a,b ⁶ ,n	7 days. ¹¹
59. Fluorene	G,T	a,b ⁶ ,n	7 days. ¹¹
60. Hexachlorobenzene	G,T	a	7 days. ¹¹
61. Hexachlorobutadiene	G,T	a	7 days. ¹¹
62. Hexachlorocyclopentadiene	G,T	a,b ⁶	7 days. ¹¹
63. Hexachloroethane	G,T	a,b ⁶	7 days. ¹¹
64. Ideno(1,2,3-cd)pyrene	G,T	a,b ⁶ ,n	7 days. ¹¹
65. Isophorone	G,T	a,b ⁶ ,n	7 days. ¹¹
66. Methylene chloride	G,T	a,o ⁶	14 days.
67. 2-Methyl-4,6-dinitrophenol	G,T	a,b ⁶	7 days. ¹¹
68. Naphthalene	G,T	a,b ⁶ ,n	7 days. ¹¹
69. Nitrobenzene	G,T	a,b ⁶ ,n	7 days. ¹¹
70. 2-Nitrophenol	G,T	a,b ⁶	7 days. ¹¹
71. 4-Nitrophenol	G,T	a,b ⁶	7 days. ¹¹
72. N-Nitrosodimethylamine	G,T	a,b ⁶ ,n	7 days. ¹¹
73. N-Nitrosodi-n-propylamine	G,T	a,b ⁶ ,n	7 days. ¹¹
74. N-Nitrosodiphenylamine	G,T	a,b ⁶ ,n ¹⁵	7 days. ¹¹
75. 2,2-Oxybis(1-chloropropane)	G,T	a,b ⁶	7 days. ¹¹
76. PCB-1016	G,T	a,b ⁶	7 days. ¹¹
77. PCB-1221	G,T	a,b ⁶	7 days. ¹¹
78. PCB-1232	G,T	a,b ⁶	7 days. ¹¹
79. PCB-1242	G,T	a,b ⁶	7 days. ¹¹
80. PCB-1248	G,T	a,b ⁶	7 days. ¹¹
81. PCB-1254	G,T	a,b ⁶	7 days. ¹¹
82. PCB-1260	G,T	a,b ⁶	7 days. ¹¹
83. Pentachlorophenol	G,T	a,b ⁶	7 days. ¹¹
84. Phenanthrene	G,T	a,b ⁶ ,n	7 days. ¹¹
85. Phenol	G,T	a,b ⁶	7 days. ¹¹
86. Pyrene	G,T	a,b ⁶ ,n	7 days. ¹¹
87. 2,3,7,8-Tetrachlorodibenzo-p-dioxin	G,T	a,b ⁶	7 days. ¹¹
88. 1,1,2,2-Tetrachloroethane	G,T	a,o ⁶	14 days.
89. Tetrachloroethene	G,T	a,o ⁶	14 days.
90. Toluene	G,T	a,h,o ⁶	14 days.
91. 1,2,4-Trichlorobenzene	G,T	a,o ⁶	14 days.
92. 1,1,1-Trichloroethane	G,T	a,o ⁶	14 days.
93. 1,1,2-Trichloroethane	G,T	a,o ⁶	14 days.
94. Trichloroethene	G,T	a,o ⁶	14 days.
95. Trichlorofluoromethane	G,T	a,o ⁶	14 days.
96. 2,4,6-Trichlorophenol	G,T	a,b ⁶	7 days. ¹¹
97. Vinyl chloride	G,T	a,o ⁶	14 days.

Table ID—Pesticides^{9, 10}

1-70. Pesticides	G,T	a,q ¹⁶	7 days. ¹¹
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Table IE—Radiologic Tests

1-5. Alpha, beta and radium	P,G	c	6 months.
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Table II Notes

¹ Container abbreviations: P = Polyethylene, G = Glass and T = PTFE-lined cap or septum.

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 Procedure abbreviations:

- a: Cool 4°C
- b: 0.008% Na₂S₂O₃⁶
- c: HNO₃ to pH<2
- d: H₂SO₄ to pH<2
- e: None required
- f: 0.6 g ascorbic acid⁶
- g: NaOH to pH>12
- h: HCl to pH<2
- i: H₃PO₄ to pH <2
- j: Filter immediately

- k: Fix on site and store in dark
- l: Add zinc acetate
- m: NaOH to pH>9
- n: Store in dark
- o: 0.025 g ascorbic acid⁶
- p: Adjust pH to 4–5¹⁰
- q: Adjust pH to 5–9

⁴When 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 (HNO₃) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H₂SO₄) 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).

⁵Samples 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 for the specific types of samples under study, the analytes 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. The term "analyze immediately" usually means within 15 minutes or less of sample collection.

⁶Should only be used in the presence of residual chlorine.

⁷Samples should be filtered immediately on-site before adding preservative for dissolved metals.

⁸Maximum 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.

⁹Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

¹⁰When samples are to be extracted and analyzed for multiple analytes, the most stringent preservation procedures and shortest maximum holding times should be observed for optimum safeguard of sample integrity. Samples extracted for a wide range of analytes 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 footnotes 6, 13, and 14.

¹¹Samples must be extracted within seven days of collection. The extract must be analyzed within 40 days of extraction.

¹²Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

¹³If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0± 0.2 to prevent rearrangement to benzidine.

¹⁴Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

¹⁵Adjust pH to 7–10 with NaOH within 24 hours of sampling.

¹⁶The 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% N₂S₂O₃.

Appendices C and D—[Removed]

6. In Part 136, Appendices C and D are proposed to be removed.

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