

**NATURAL RESOURCES BOARD AGENDA ITEM**

**SUBJECT:** Adoption of Board Order SS-16-08, revisions to NR219 relating to analytical test methods and procedures.

**FOR:** DECEMBER 2008 BOARD MEETING

**TO BE PRESENTED BY:** David Webb, Chief, Environmental Science Services  
Bureau of Science Services

**SUMMARY:**

Chapter NR 219 establishes analytical methods required for the analysis of wastewater samples for WPDES permit compliance. The purpose of amending chapter NR 219, Wis. Adm. Code, is to incorporate revisions to the federal regulations (March 12 and 26, 2007 Federal Registers), and add methods developed by EPA and approved by their Alternate Test Procedure program. The proposed rulemaking also incorporates updated methods published by ASTM, in "Standard Methods for the Examination of Water and Wastewater" 21st edition and on-line versions and those developed by instrument manufacturers.

**RECOMMENDATION:** Adopt Board Order SS-16-08, revisions to NR219 relating to analytical test methods and procedures.

**LIST OF ATTACHED MATERIALS:**

- |    |                                     |   |     |                                     |          |
|----|-------------------------------------|---|-----|-------------------------------------|----------|
| No | <input type="checkbox"/>            | Fiscal Estimate Required                              | Yes | <input checked="" type="checkbox"/> | Attached |
| No | <input checked="" type="checkbox"/> | Environmental Assessment or Impact Statement Required | Yes | <input type="checkbox"/>            | Attached |
| No | <input type="checkbox"/>            | Background Memo                                       | Yes | <input checked="" type="checkbox"/> | Attached |

**APPROVED:**

/s/ /s/

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Bureau Director, Jack Sullivan, Russell Rassmussen

/s/ /s/

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Administrator, Susan Crawford, Todd Ambs

/s/

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Secretary, Matt Frank

10/20/08 10/21/08

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Date

10/28/08 10/28/08

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Date

11/07/08

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Date

cc: Laurie J. Ross - AD/8  
J. Renville, LS/8

D. Drinkman, SS/7  
D. Webb, SS/7  
T. Mungan, WT/3

R. Rassmussen, WT/3

DATE: October 30, 2008

TO: Natural Resources Board Members

FROM: Matthew J. Frank, Secretary

SUBJECT: Background Memo on Proposed Amendments to Chapter 219, Wis. Adm. Code, Analytical Test Methods and Procedures

1. Why is rule being proposed?

- a. What event or action triggered the proposal?
- b. What are issues addressed by the rule?

On March 12, 2007, EPA published "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations; Analysis and Sampling Procedures; Final Rule". In addition, on March 26<sup>th</sup>, 2007, "Guidelines Establishing Test Procedures for the Analysis of Pollutants; Analytical Methods for Biological Pollutants in Wastewater and Sewage Sludge; Final Rule" was published. In these final rules, EPA updated the approved analytical test methods to be used in the National Pollutant Discharge Elimination System (NPDES) program, which regulates discharges from industrial and municipal wastewater treatment facilities. Wisconsin must incorporate these changes its administrative rules to maintain delegated authority for the Clean Water Act.

The proposed rule revisions to ch. NR 219, Wis. Adm. Code, reflect the changes made at the federal level, add additional methods approved by U.S. EPA Office of Water Alternate Test Procedure, and incorporate equivalent methods from newer versions of methods published by the American Society for Testing and Materials, (ASTM), contained in "Standard Methods for the Analysis of Water and Wastewater", and developed by instrument manufacturers. Chapter NR 219, Wis. Adm. Code, contains the analytical test methods and procedures for the Wisconsin Pollutant Discharge Elimination System (WPDES).

The proposed rule provides flexibility to laboratories that analyze samples in support of a WPDES permit. Although the rule revision deleted many analytical methods, the proposal retains at least one method for any regulated analyte. The proposed rule package also reduces the use of reagents containing mercury with the elimination of 14 additional methods.

The rule clarifies the maximum holding time for total residual chlorine, pH, dissolved oxygen, sulfite, and temperature by changing language to read "analyze within fifteen minutes" from "analyze immediately". It also addresses known interferences for cyanide determinations, extends holding time for hexavalent chromium, and specifies hold times for chlorinated dioxin and furan analyses by sample matrix.

2. Summary of the rule.

Chapter NR 219, Wis. Adm. Code, lists the approved analytical methods for analyses for the WPDES permit program. The methods, found in nine tables, contain approved analytical

methods for the analyses of Biological and Inorganic analytes, Metals Digestions, Non-Pesticide Organics, Pesticides, as well as Specific Methods for Sludge and Pharmaceutical Pollutants, as required for compliance with a WPDES permit. Sample Preservation requirements are contained in the final table. Language in sections NR 210.04 (2) and (3) has also been modified to be consistent with the thermal preservation requirements in the final table.

The Rule Making Order, included in Attachment A provides additional details in its rule summary.

### 3. How does this proposal effect existing policy?

This proposal does not affect existing policy, it merely provides lists of updated analytical methods approved for analyses in support of a WPDES permit.

### 4. Hearing synopsis.

#### a. When was the hearing held?

Public hearings were held on Wednesday, August 27, 2008.

#### b. Where was the hearing held?

Public hearings were concurrently held in Madison and Stevens Point utilizing videoconferencing technology.

#### c. Number of appearances.

One member of the regulated community appeared.

#### d. Summary of hearing comments.

Hearing comments were received from two municipal sewerage districts, one industrial permittee and three commercial laboratories that perform analyses in support of the WPDES program. Their comments were limited to four areas of concern: clarification and correction of approved analytical methods identified in this chapter, sample preservation and hold times, requesting approval of an additional analytical method and clarification of hierarchy of sample preservation and holding time content included in rule and analytical methods.

#### Clarification and Correction of Approved Analytical Methods

- One permittee sought clarification of the use of the luminescence technique for Biochemical Oxygen Demand (BOD<sub>5</sub>) and Carbonaceous Biochemical Oxygen Demand (CBOD<sub>5</sub>) determinations.
- One laboratory questioned the differences in approved methods for parameter 21- color in table B from *Standard Methods for the Analysis of Water and Wastewater*, 18-21<sup>st</sup> editions and the on-line version.
- One permittee sought clarification of acceptable methods for metals digestions.
- Four typographical errors were indentified by two laboratories in analytical methods tables.

#### Sample Preservation and Holding Times

- One permittee sought clarification of documentation of sample temperature when the facility's effluent stream temperature dramatically exceeds 6°C, even when collected in composite autosamplers maintained at  $\leq 4^\circ\text{C}$ .
- One permittee sought clarification of and development of guidance by the department, to address apparent discrepancies in sample temperature requirements contained in this chapter and ch. NR 149, Wis. Adm. Code, Laboratory Certification and Registration.
- Two commenters sought clarification and development of guidance to address cyanide preservation and interference mitigation procedures contained in Notes 5 and 6 to Table F.
- One laboratory suggested standardized data qualification procedures to address hold time exceedances for tests generally considered "field tests".
- One laboratory sought clarification of maximum holding time and filtration language for parameter 45, orthophosphate, in table F.

Requesting Approval of Additional Analytical Method

- One laboratory sought inclusion of cold vapor atomic fluorescence spectroscopy for the determination of mercury in wastewater sludges, as identified in table EM.

Clarification of Hierarchy of Sample Preservation and Holding Time

- One permittee sought inclusion of language within this chapter to clarify the hierarchy of sample preservation and holding times when this information is included in both rule and analytical methods.

Response to Legislative Council Rules Clearinghouse Report

The Legislative Council Rules Clearinghouse Report did not contain any comments or suggestions to modify the content of this chapter.

Please refer to the "Summary and Response to Public Hearing Comments", attached.

e. Public contacts after hearing.

There were no additional public contacts after the public hearing.

5. Information on environmental analysis, if needed.

This is a Type III action and does not require an environmental analysis or environmental impact statement.

6. Final Regulatory Flexibility Analysis.

Does the proposed rule have a significant economic impact on a substantial number of small businesses?

No, the proposed rule does not have a significant economic impact on a substantial number of small businesses.

If no, justify this response.

The small businesses impacted by the proposed rule are commercial laboratories that perform compliance monitoring for WPDES permittees. The vast majority of these laboratories have implemented use of newer analytical methods that were retained in Tables A-EM of this chapter. For most parameters where small businesses may have to update their method references, the Department has maintained at least one method from the current language. There were only four specific techniques eliminated— three of these included mercury-containing reagents for which other alternatives using similar techniques were retained. Hexane extractable materials, an alternative to the freon extraction for oil and grease, has been in use by laboratories for over a decade. All of the small businesses that perform oil and grease determinations currently maintain certification for the hexane method. The Department no longer offers certification for the freon extraction method.

Sample preservation requirements for tests that are typically considered field parameters and are not required to be performed by certified laboratories, were clarified so there is no question as to whether data from these time-sensitive tests is valid. The hold times for hexavalent chromium, increased from 48 hours to 14 days, and polychlorinated biphenyls (PCBs), increased from 7 days to one year before extraction, allow small businesses more flexibility in scheduling and performing these analyses. Clarification of procedures for potential interferences in cyanide analyses will allow small business laboratories to continue to perform these analyses in 14 days without requiring investment in new instrumentation required to conduct these analyses.

I. Identify and discuss why the rule includes or fails to include any of the following methods for reducing the impact on small business.

The following items require response only if there is significant economic impact on a substantial number small businesses. The proposed rule as explained above, does not have a substantial impact on this segment of the regulated community.

- A. Less stringent compliance or reporting requirements.
- B. Less stringent schedules or deadlines for compliance or reporting requirements.
- C. Consolidation or simplification of compliance or reporting requirements.
- D. The establishment of performance standards in lieu of design or operational standards.
- E. The exemption from any or all requirements of the rule.

II. Summarize the issues raised by small business during the rule hearings, any changes made in the proposed rule as a result of alternatives suggested by small business and the reasons for rejecting any alternatives suggested by small business.

The issues raised by small business included clarification of sample holding times and preservation requirements, inclusion of an additional analytical method, and correction of typographical errors in the draft rule. The Department agreed to issue guidance to further clarify sample holding times and preservation requirements and added the analytical method, as requested.

The Department did not reject any alternatives suggested by small business.

III. Identify and describe any reports required by the rule that must be submitted by small business and estimate the cost of their preparation. Manual Code 1022.4 Secretary's Directive - 5 - 11-23-05

The rule does not include any reporting requirements.

IV. Identify and describe any measures or investments that small business must take to comply with the rule and provide an estimate of the associated cost.

This rule maintained at least one approved analytical method for each parameter and three previously-approved editions of *Standard Methods for the Determination of Water and Wastewater*, in an effort to minimize additional costs to permittees and laboratories. Many of updated and newer methods use the same equipment and instruments as methods that were deleted in the federal rule. Updated methods published by U.S. EPA are readily available for download from various websites. Methods from other consensus organizations, such as ASTM, are available on inter-library loan.

Small business would only encounter additional cost or investment if they voluntarily modify their procedures to utilize newer techniques and those incorporated into this chapter for the first time.

V. Identify the additional cost, if any, to the state in administering or enforcing a rule which includes any of the methods listed in 1. A through E.

Compliance with this chapter is determined by the Bureau of Watershed Management and the Department's Laboratory Certification and Registration Program. The state will not incur additional cost to administer or enforce this rule.

VI. Describe the impact on public health, safety and welfare, if any, caused by including in the rule any of the methods listed in 1. A through E.

There are no impacts on public health, safety and welfare as a result of the methods contained in 1. A through E.

## Summary of and Response to Public Hearing Comments

Public comments were received from the following:

Facility (comment type)	Commenter
Davy Laboratories (written)	Paul Harris
Georgia-Pacific Corporation (written)	Jacqueline K. Powell
Milwaukee Metropolitan Sewerage District (verbal)	Sharon Mertens
Northern Lake Service (written)	Tom Priebe
S-F Analytical Laboratories, Inc. (written)	Gary G. Geipel
Waukesha Wastewater Treatment Plant (written)	Randall Thater

### A. Clarification and Correction of Approved Analytical Methods

#### Waukesha Wastewater Treatment Plant

*Biochemical Oxygen Demand – Table B, parameter 9. It is my understanding that this update was meant to include acceptance of luminescence technology for BOD testing. The analytical technology cited is 'Dissolved oxygen depletion' without specifying how DO is determined. The cited methods for BOD; Standard Methods in various editions, USGS, and AOAC do not specifically include luminescence determination. The NR 219 Table B, parameter 47, Oxygen, dissolved specifies Winkler, Electrode, and Luminescence as acceptable technologies for DO. The cited luminescence method is ASTM D888-05(C) and ASTM does not have an accepted method for BOD. If in fact it is intended to include BOD by luminescence, I believe the language should be clarified to make this apparent. I support the inclusion of the luminescence technology.*

#### WDNR Response

BOD methods direct laboratories to use methods for dissolved oxygen (4500-O C or G) to determine initial DO (e.g., ¶14.f 2). Standard Methods 5210B, 20<sup>th</sup> edition). The approved methods for dissolved oxygen, listed as Parameter 47, includes three analytical technologies- Winkler (azide modification), electrode, or luminescence (LDO). Laboratories may use any of these methods, including the LDO methods ASTM D888-05(C) or Hach 10360 for the determination of dissolved oxygen during BOD and Carbonaceous BOD analyses.

#### Davy Laboratories

*On Page 13 – Parameter 21 – Color. Why is 2120 E the only cited method versus 2120B or 2120D?*

#### WDNR Response

The inclusion of Standard Methods On-Line was based on an Alternate Test Procedure (ATP) approval granted to the American Water Works Association in April 2007. In this approval, the Office of Ground Water and Drinking Water's Technical Support Center (OGWDW/TSC) and the Engineering and Analytical Support Branch (EASB) declared equivalency between the 21<sup>st</sup> Edition and On-Line versions of Standard Methods for over 100 analytical methods.

For color analyses, EPA only identified Standard Methods 2120 B-01 as equivalent to approved methods in the ATP. The On-Line version of Standard Methods 2120 C, Spectrophotometric-Single Wavelength Method, identified as "PROPOSED" differs dramatically from the approved method contained in the 20<sup>th</sup> edition. Another approved method from Standard Methods 20<sup>th</sup>

ed., 2120 E, is also significantly different from the on-line version, 2120 E-01. Although EPA only approved a single on-line version, Table B includes Standard Methods 2120 B [18<sup>th</sup>, 19<sup>th</sup>, 20<sup>th</sup> and 21<sup>st</sup> editions], 2120 C [18<sup>th</sup>, 19<sup>th</sup>, 20<sup>th</sup> and 21<sup>st</sup> editions] and 2120 E [18<sup>th</sup>, 19<sup>th</sup>, 20<sup>th</sup> and 21<sup>st</sup> editions] as well as USGS I-1250-85.

U.S. EPA has not declared the remaining on-line versions of Standard Methods 2120 B and 2120 C as equivalent through the Alternate Test Procedure program. The Department does not see a need to include additional methods that have not been deemed as equivalent at this time.

### **Waukesha Wastewater Treatment Plant**

*Silver - Total – Table B, parameter 63. The prior version of NR 219 included a separate table for metals digestions. The proposed revision drops that table and uses footnotes to address digestion. Footnotes 9, 38, 40 follow the parameter listing and footnotes 9, 11, 45 follow the digestion listing. Footnote 9 addresses the general digestion for metals by acid reflux citing various references. Footnotes 38 and 40 are silver specific digestion modifications. Footnote 38 is stated to apply to industrial wastewaters above 1 mg/L. Footnote 40 is stated to apply to samples containing in excess of 4 mg/L. The practice in this laboratory, which is analyzing samples for silver to support the plant industrial pretreatment program, is to use the method as detailed in footnote 40, but for all samples regardless of expected silver concentration. We find it confusing to determine whether our current practice is allowable under the draft NR 219. We believe our protocol is a good laboratory practice, and should be allowed. We ask that the language be changed to clarify and allow such practice.*

*We understand that the language of this rule basically reiterates that in 40 CFR Part 136 from the US EPA. If this fact makes including these suggested clarifications problematic, then we suggest the department at least endeavor to make such clarifications by guidance.*

### **WDNR Response**

Metals digestion procedures are contained in Table EM of ch. NR 219. The department did not delete or modify the content of this table in this update effort. Table EM will be included, in the updated version of this chapter, unchanged, when rulemaking is completed.

Footnotes associated with silver determinations in table B, numbers 38 and 40, require specific digestion to ensure dissolution of silver in the presence of halide ion (e.g., chloride). If the notes were enforced literally, the digestion in note 38 would only be allowed for sample concentrations between 1-4 mg/L; this is not the intent, rather that labs must use either alternative if anticipated silver concentrations exceed 1 mg/L.

### **Davy Laboratories**

*On Page 14 – Parameter 25 – Fluoride. The ISE method is “C” not “B”. B is the distillation step.*

### **WDNR Response**

This is correct; the content of table B, parameter 25- Fluoride, Electrode, manual has been corrected to read "4500-F C [18<sup>th</sup>, 19<sup>th</sup>, 20<sup>th</sup>, 21<sup>st</sup>]" under the heading "Standard Methods".

### **Davy Laboratories**

*On Page 15 – Parameter 28 – pH. Standard method Online is missing a zero. Should read 4500-H+ B-00.*

### **WDNR Response**

This is correct; the content of table B, parameter 28- Hydrogen ion (pH), Electrometric measurement has been corrected to read "4500- H<sup>+</sup> B-00 under the heading "Standard Methods Online".

### **Davy Laboratories**

*On Page 19 – Parameter 40 - Standard method Online is missing a zero. Should read 4500-NO<sub>2</sub> B-00.*

### **WDNR Response**

This is correct; the content of table B, parameter 40- Nitrite, Spectrophotometric, manual, has been corrected to read "4500- NO<sub>2</sub> B-00 under the heading "Standard Methods Online".

### **Northern Lake Service**

*First off, a little housekeeping matter on Table EM regarding the Selenium method citations. It appears that ICP method citations are cited under both EPA and Standard Methods columns for Gaseous Hydride.*

### **WDNR Response**

This is correct; the content of table EM, Selenium, Gaseous Hydride, has been corrected. The only approved method for sample preparation and analysis of selenium by gaseous hydride is from "Test Methods for Evaluating Solid Water, Physical/Chemical Methods," SW-846, Method 7471A, September 1994.

## **B. Sample Preservation and Holding Time**

### **Georgia Pacific**

*In NR 219.04, Table F, Note 2, has the following wording "...For a composite sample to be split into separate aliquots for preservation and analysis, maintain the sample at <6°C, unless specified elsewhere in this table or in the method(s) until collection, splitting and preservation is completed. ...The temperature of the samples shall be documented upon receipt at the laboratory..." Although the intent of these statements is to ensure that the integrity of the sample is maintained during transport to the laboratory, Georgia-pacific believes that this requirement is unnecessary when samples are submitted to the laboratory within 15 minutes of collection and requests that the note be removed or modified.*

*Georgia-Pacific's final effluent stream has a temperature that is typically between 30°C and 35°C especially during the warmer months of the year. There are two 24-hour composite sampler refrigerators sampling this effluent stream both of which are maintained at <4°C. There will be instances (given that flow proportional samples are collected) where the composite sample exceeds the 6C temperature requirement due to the warm nature of the effluent. For example, an aliquot of effluent collected prior to the sampling period would exceed the 6°C temperature requirement since there may not be sufficient time to cool the sample below the required temperature. As a result, the temperature of the sample upon receipt by the laboratory, which is within 15 minutes of collection, could result in a temperature above 6°C. To meet the 6°C temperature requirement, the samples would need to be maintained at a lower temperature thereby risking the integrity of the sample as a result of the potential to freeze the sample, particularly during the early hours of the sampling period. We believe that documenting that the composite samplers and/or refrigerators are maintained at*

*temperatures <4°C should be sufficient.*

*The alternative is to place the sample jars on ice. Georgia-Pacific believes that this alternative, although satisfying the requirement for samples to be received on ice, is ineffective at cooling a sample to <6°C when delivered to the laboratory within 15 minutes of collection. It would also be quite cumbersome, in a practical sense, to transport two ten-liter glass jars on ice to the laboratory. We do not believe that this is the intent of the rule and suggests that the wording of Note 2 in Table F to be changed to the following "...The temperature of the samples shall be documented upon receipt at the laboratory is not received within 15 minutes of collection..."*

### **WDNR Response**

Footnote 2 specifies that "For a composite sample collected with an automated sampler, refrigerate the samples at  $\leq 6^{\circ}\text{C}$  during collection, unless specified elsewhere in this table or in the method(s) until collection, splitting and preservation is completed. For a composite sample to be split into separate aliquots for preservation and analysis, maintain the sample at  $\leq 6^{\circ}\text{C}$ , unless specified elsewhere in this table or in the method(s) until collection, splitting and preservation is completed.

Your facility's sample is properly preserved, provided that it is collected with a refrigerated autosampler. Regulations cannot preclude general laws of thermodynamics; it would likely take several hours for your facility's composite sample to drop  $25^{\circ}\text{C}$  ( $\sim 50^{\circ}\text{F}$ ) or more to literally meet these temperature requirements. Provided that your composite samples are either analyzed immediately or stored at  $\leq 6^{\circ}\text{C}$  upon arrival at the laboratory, they would be considered "properly preserved" under these requirements.

Samples shipped to your laboratory must also follow the preservation and holding time requirements for laboratories specified in s. NR 149.46(4) Wis. Adm. Code. In this section, samples requiring preservation at  $6^{\circ}\text{C}$  shall be considered preserved if they are received at a temperature from above their freezing point to  $6^{\circ}\text{C}$  or if they are received surrounded by ice. If the samples are not received on ice, the laboratory is required to determine actual sample temperature— either that of the actual sample, a temperature blank or melt water in the shipping container. A  $30\text{-}35^{\circ}\text{C}$  sample collected at  $\leq 6^{\circ}\text{C}$ , transported to the laboratory immediately after the completion of the compositing period "on ice", has been properly preserved, regardless of the actual sample temperature.

### **Milwaukee Metropolitan Sewerage District**

*The Milwaukee Metropolitan Sewerage District requests that the Department provide guidance on priority when there are discrepancies between NR 149 and NR 219 and to consider this when revising NR 149 again. The determination of sample temperature upon receipt, for example, appear to be in conflict.*

### **WDNR Response**

Sample preservation procedures, specified at ss. NR 219.04(2) Wis. Adm. Code, requires sludge samples be preserved by cooling to  $\leq 6^{\circ}\text{C}$  where required. All samples requiring thermal preservation at  $\leq 6^{\circ}\text{C}$  must be cooled immediately after collection (e.g., refrigerated autosampler for composite samples), and that temperature be maintained during shipping. s. NR 219.04(3) Wis. Adm. Code, requires that samples not cooled during collection shall be chilled to  $\leq 6^{\circ}\text{C}$  prior to shipping with a temperature blank. Samples shipped "on ice" can be recorded as "Received On Ice" if solid ice is present in the cooler upon receipt by the lab. If samples are shipped with ice packs, or no ice is present, labs must record the actual temperature of a sample, temperature blank or melt water temperature.

The sample preservation and holding time requirements listed in s. NR 149.46(4)a, Wis. Adm. Code, establishes that sample preservation and hold time requirements identified in state or federal regulations take precedence over that contained in analytical methods or authoritative sources (see General Comment, above, for further details). The following subsection, b., specifies that samples are properly preserved if, they are either surrounded by ice or actual temperature of a sample, temperature blank or melt water in the shipping container is between 0-6°C. This language mirrors that contained in ch. NR 219, Wisc. Adm. Code. In addition, permittees may petition the EPA Region V Administrator for a variance from prescribed sample preservation procedures applicable to samples from a specific discharge.

The WDNR Laboratory Certification Program may consider clarification of this issue in future revisions of ch. NR 149, Wisconsin Administrative Code.

### **Milwaukee Metropolitan Sewerage District**

*The language contained in Footnote 6, Table F, which addresses required preservation for cyanide determinations is complex and confusing, especially when dealing with unknown samples. The Milwaukee Metropolitan Sewerage District requests that the Department provide guidance on the proper procedure for preserving samples for cyanide.*

### **Northern Lake Service**

*The second issue is the cyanide preservation procedure, which involves a lengthy list of requirements to be performed at the time of collection. This may prove to be a source of potential problems for the laboratories, and possible data validation issues when sample collection is in the hands of our clients. If there are issues, they tend to be blamed on the laboratory conducting the analyses. I believe that WDNR will have to work with their existing database to help industries and municipalities identify potential interference's or processes which may require special handling in terms of sample collection. This responsibility cannot be placed on the laboratory community, since it is more of a consulting activity, and the alternative of having all clients sample based upon a worse case scenario is not economically justifiable. I believe the time required for client services staff to attempt to administer this collection procedure to our clients would be additional economic burden. If this section is not addressed further, the field collection is going to be an absolute nightmare. In addition, I haven't found anything in the method, or in 40CFR, part 136 that is quite like what is being proposed in the new NR 219.*

### **WDNR Response**

In the proposed Methods Update Rule (April 4, 2004), the preservation requirements for cyanide and cyanide amenable to chlorination were listed as "Cool, <6°C, NaOH to pH>12, reducing agent" and hold time as "14 days". Footnote 5 called for addition of dechlorinating agent, either ascorbic acid or sodium borohydride, if residual chlorine was present, and warned about use of excess. Footnote 6 called for samples to be adjusted to pH ≥12 by addition of sodium hydroxide and refrigeration, after checking for sulfide, particulate, aldehyde and chlorine, hypochlorite or other oxidizer interferences. Only *untreated* samples were required to be analyzed within 24 hours when sulfide was present. There are no provisions that require a specific analytical method in the event that sulfide or other interfering substances are determined to be present in the sample.

In the preamble to the final rule, EPA declares "The most significant of the changes are those made to Footnote 6, which addresses the preservation of samples to be analyzed for cyanide. Based on information gathered during the development of new cyanide methods approved in this rulemaking, and information collated from various commenters and experts in cyanide

analyses, EPA revised footnote 6 to Table II by adding text that describes procedures that are recommended for removal or suppression of cyanide interferences, including interferences from sulfur, sulfide, sulfite, thiocyanate, and aldehydes. The recommended procedures may differ from those described in the older approved methods."

The language contained in notes 5 and 6, were expanded by EPA in the final federal rule, which was based on information gathered during the development of new cyanide methods approved in this rulemaking, and information collected from various commenters and experts in cyanide analyses.

Historically, in Wisconsin cyanide monitoring has been required upon initial permit application as part of a pretreatment suite that includes metals and priority pollutants. The number of WPDES permittees that require routine cyanide monitoring is relatively small (approximately 30), when compared to the overall number of WPDES permits; of these the highest frequency is monthly. The department acknowledges that many interferences identified in table F are not present in well-treated municipal wastewater sampled at the proper locations. (Oxic wastewater usually does not contain sulfide and, by sampling prior to disinfection, the possibility of chlorine, sulfite, bisulfate and thiocyanate may be bypassed). Industrial permittees, on the other hand, can simply look to their processes to determine whether these compounds are likely to be present based on their facility's operation.

All samples for WPDES compliance must be screened for the presence of sulfide at the time of sample collection. If sulfide is not present, the sample must either be preserved to pH>12, refrigerated and analyzed within 48 hours or be screened for other interferences (sulfur, chlorine, carbonate, aldehyde, sulfite, thiosulfate and thiocyanate), preserved to pH>12, refrigerated and analyzed within 14 days. If sulfide is present, the sample must undergo one of three mitigation procedures to eliminate the interference.

Laboratories that perform WPDES-required cyanide analyses must either analyze samples that have been preserved to pH>12 and  $\leq 6^{\circ}\text{C}$  within 48 hours or document that the presence and removal of all interferences have been carried out for those samples preserved to pH>12 and  $\leq 6^{\circ}\text{C}$  and analyzed within 14 days.

Approved methods typically describe procedures for the determination and removal of some of these interferences. Laboratories can eliminate known interferences prior to sample distillation, as specified by method in lieu of interference mitigation after sample collection. When reporting cyanide results, laboratories should identify any interferences that were removed prior to distillation.

The department agrees issuance of guidance that explains interference screening and mitigation processes will assist the regulated community in this matter.

### **Northern Lake Service**

*Documentation of holding time, sample collection requirements, and preservation issues are going to come up with the new language of "within 15 minutes" being used. It is acknowledged and accepted by the regulatory community that many of the analyses performed for wastewater treatment facilities are performed beyond traditionally accepted holding times. My concern involves the data flagging procedure that would be required of all laboratories if we are to flag these items. I believe this will be necessary in order to put all labs on an even playing field, as qualified data from one laboratory is perceived as lower in quality than unqualified data from another laboratory for the same analytical procedure. This does*

*have a potential economic impact on laboratories. I would suggest that all laboratories have a specific data qualifier for these items, or at a minimum have a disclaimer regarding holding times for specific laboratory tests or sample preparation steps.*

### **WDNR Response**

Tests such as dissolved oxygen, pH, temperature and total residual chlorine, are exempted from laboratory certification requirements as they are considered "field testing". These tests are usually carried out by wastewater treatment plant operators as part of the facility's daily process control. If these results indicate adjustment is needed to the plant's operation, these changes can be made immediately, instead of possibly waiting several hours, or even days, for a report from a commercial laboratory. It is highly unlikely that samples can be transported to and analyzed by an external laboratory within hold time.

When field testing is performed after the hold time has been exceeded, these measurements cannot be used to demonstrate compliance with a permit. The only exception would be if a facility's permit specifies "Lab pH", which assumes that the data was not generated within 15 minutes of collection.

Samples that are not immediately analyzed must also be properly preserved. This includes addition of chemical preservatives or storage at  $\leq 6^{\circ}\text{C}$ . Many commercial laboratories provide pre-preserved sample containers to ensure proper preservation after sample is added. Samples that are shipped to external laboratories must also contain sufficient volume of ice to ensure sample temperatures do not exceed  $6^{\circ}\text{C}$  on arrival. When reporting results of samples that have been improperly preserved, data must be qualified.

Inclusion of certification for these field tests merely *allows* laboratories that perform sample collection for their clients to perform these analyses as a service to their clients. These samples still must be properly preserved and analyzed within the hold times established by this or other chapters of the Wisconsin Administrative Code. Any laboratory or facility that performs these analyses beyond the hold times *must* properly qualify the data when submitting results to the department. Qualified data is not necessarily perceived by the program as being of a lesser quality, merely that there were circumstances that resulted in the data not meeting all analytical or regulatory requirements.

The WDNR Laboratory Certification and Registration Program, in conjunction with the Watershed Management Permits Section, is developing guidance to more specifically address sample preservation and hold time requirements contained in chs. NR 219 and NR 149, Wis. Adm. Code.

### **Davy Laboratories**

*On Page 45 – Table F – 345. There should be some clarification under the information given in the maximum holding time column. If you are doing total orthophosphate why are you filtering? If you are filtering what type of filter do you use? (i.e. 0.45 um?). Should not the data be reported as dissolved orthophosphate?*

### **WDNR Response**

There are several forms of orthophosphorus included in EPA Method 365.1 (1993); these include total phosphorus, total orthophosphate (P-ortho), total hydrolysable phosphorus (P-hydro), total organic phosphorus (P-org), dissolve phosphorus (P-D), dissolve orthophosphate (P-D Ortho), dissolved hydrolysable phosphorus (P-D, hydro), dissolved organic phosphorus (P-D, org), and calculation of insoluble phosphorus. The requirement for filtration applies only to determination

of dissolved phosphorus species. The Department agrees that this content is confusing and has clarified the intent with the addition of footnote 22, which reads:

"Filtration is only required when reporting dissolved orthophosphate, dissolved hydrolyzable phosphorus or dissolved organic phosphorus as described in EPA Method 365.1 (1993). Filtration must be completed within 15 minutes of collection using a 0.45µm filter; sample shall be maintained at ≤6°C and analyzed within 48 hours."

## C. Requesting Approval of Additional Analytical Method

### S-F Analytical Laboratories, Inc.

#### SECTION I

*Our primary concern is that there is only one methodology which would be approved for the analysis of Mercury in sludge samples and that this methodology is Cold Vapor Atomic Absorption Spectroscopy (CVAAS). It is our assumption that the EPA has not approved any other method, such as Cold Vapor Atomic Fluorescence Spectroscopy (CVAFS), due to concerns for instrument cross contamination and/or the problems associated with the digestion and dilution procedures.*

*It is our opinion that the lack of alternative methodologies for this analysis places a financial burden on smaller analytical facilities which would, technically, need to have a CVAAS instrument to perform analysis of Mercury in sludges but would also need to have a CVAFS instrument in order to analyze aqueous samples to the low concentrations required by some permits.*

#### SECTION II – Considerations

*EPA method 245.7 allows for the extension of the working range by dilution prior to oxidation and addition of extra Bromide/Bromate solution in the presence of large amounts of organic matter.*

*The Appendix to EPA method 1631B specifically addressed the analysis of Total Mercury in Tissue, Sludge, Sediment and Soil by Acid Digestion and BrCl oxidation followed by determination by CVAFS. This method also gave performance criteria for calibration, calibration verification, MDL, ML, IPR, OPR and Blanks.*

*EPA SW-846 method 7474 which is specifically approved for the analysis of sediment and tissue samples uses a combination of microwave assisted digestion, Bromide/Bromate oxidation and CVAFS for the determination of Total Mercury. The initial digestion is based on method 3051A which is approved as a preparatory step for Mercury analysis. The digestate from the modified 3051A digestion is then subjected to the same oxidation and reduction steps used in method 245.7 for aqueous samples.*

*ISO/CEN 13506 is a CVAFS method using a BrCl oxidation and CVAFS.*

*Instrument Manufacturers have based CVAFS applications on the EPA and ISO methodologies. Teledyne-Leeman Labs application note 1034 is one such procedure and PS Analytical APP013 is another. Applications chemists from the manufacturers have commented that only the USA is still requiring the use of CVAAS techniques.*

*Revisions to NR 149 which take effect on September 1, 2008 do not make a distinction between*

*Sediment, Sludge and Tissue which are all placed in the Solid Matrix.*

*An Attachment to this letter is included to show method performance when using SW-846 method 7474 for the analysis of NIST SRM 2781 Domestic Sludge. The attachment shows sample recoveries obtained over a six (6) month period. It should be noted that during this period only one recovery fell outside of the performance criteria for method 1631B Appendix. For these determinations the old traps were not used and there was no measurable carryover from the sludge samples to blanks which followed in the analytical sequence.*

*Instrumentation has progressed to the point where the user can select whether or not to use the Gold traps. When not in use the traps are isolated from the gas flow and are not subject to contamination. When in use the traps are protected by software/hardware which will isolate the traps and purge the instrument and sampling system if a sample with high Mercury concentration is sensed.*

#### **SUMMARY**

*CVAFS is a methodology which has greater sensitivity and fewer interferences than CVAAS. In fact, the increased sensitivity of the method makes sample contamination the most likely form of interference.*

*There is precedent within the EPA for the use of CVAFS for the determination of Mercury in sludges. ISO has also approved a method for this determination. EPA methods do allow for extending the range of the analysis by not using one or both Gold traps in the system and also by allowing samples to be diluted prior to the oxidation procedure.*

*It is our hope that consideration of this method will be given for the analysis of sludge and solid samples, other than sediment and tissue, as it should be possible to meet the performance criteria in method 1631B Appendix when analyzing most sample matrices. Our experience working with the technique has shown that the control of cross contamination between matrices is not an insurmountable problem if the initial acid digestion procedure for solid samples is carried out in a separate location from the area designated for the preparation of water samples.*

*The laboratory also provided data to substantiate their ability to recover mercury from NIST 2781, a dried and pulverized domestic wastewater sludge. This lot has been certified by NIST's Standard Reference Material Program to contain  $3.64 \pm 0.25$  mg/kg mercury. Recoveries ranged from 85-128%, with an average recovery of 96.8%.*

#### **WDNR Response**

Analysis of mercury using the fluorescence technique for WPDES-compliance was first included in ch. NR 219, Wis. Adm. Code in 2002. Rulemaking by the department added two analytical methods developed by EPA, 1631D, which utilizes a purge and trap system, and 245.7, which uses a liquid-gas separation and drying tube to isolate mercury. Revision B of Method 1631 incorporated a digestion for mercury determination in tissue sludge, sediment, soil, industrial samples and certified reference materials as an appendix. This digestion suggested mercury determination at concentrations ranging from 1-5000 nanograms/gram in solid and semi-solid matrices. The language contained in the appendix is no longer included in the current revision, E, of EPA method 1631.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Update IV, February 2007, includes Method 7474, Mercury in Sediment and Tissue Samples by Atomic Fluorescence Spectrometry. In this method, samples are microwave digested, and an aliquot

of the digestate is diluted and subjected to a cold digestion with the same reagents and under the same conditions as in EPA Methods 245.7 and 1631E.

The following table compares the quality control requirements of Methods 1631E, 245.7 and 7474.

QC Element	Method 1631E	Method 245.7	Method 7474
Initial Precision & Recovery	79-121%, <21% RSD	78-108%, <16% RSD	No criteria
Ongoing Precision and Recovery or Laboratory Control Sample	77-123%	76-113%	80-120%
Matrix Spike/Matrix Spike Duplicate	71-125%, <24% RPD	63-111%, <18% RPD	80-120%, <20% RPD
Initial Calibration Verification (ICV)			90-110%
Continuing Calibration Verification (CCV)			90-110%

Each of these methods also incorporates a variety of blanks to assess potential contamination from sampling, reagents and the analytical system. Method 7474 includes additional calibration verification standards that are not required by either of the currently approved methods.

The department agrees that the fluorescence technique may be appropriate for sludges regulated under this chapter. The rigorous microwave digestion technique described in SW-846 Method 7474, when combined with fluorescence detection will result in data of similar quality to other currently approved analytical methods. Table EM has been modified to incorporate this addition.

## D. Clarification of Sample Preservation and Holding Time Hierarchy

### GEORGIA-PACIFIC CORPORATION:

*We understand that the Department has adopted Table II.—Required Containers, Preservation Techniques, and Holding Times referenced in 40 CFR Part 136. The analytical methods identified in Section 4: NR 219.04, Table B, lists Standard Methods, 18<sup>th</sup>, 19<sup>th</sup>, 20<sup>th</sup> and 21<sup>st</sup> Editions. These methodologies still refer to sampling and handling preservation temperatures of 4°C. To avoid confusion between the analytical methodology and the requirements outline in NR 219.04, table F, Georgia-Pacific proposes that the following language, similar to the language uses to indicate which items take precedence, be incorporated or referenced in NR 219.04.*

*"Sample preservation procedures, container materials, and maximum allowable holding times for parameters care cited in Table F. Information in the table takes precedence over information in specific methods or elsewhere. Any person may apply for a variance from the prescribes preservation techniques, container materials and maximum holding times applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the..."*

### WDNR RESPONSE

The hierarchy for sample preservation for laboratories performing analyses for covered programs, including permitted wastewater discharges under, ch. NR 219, Wis. Adm. Code, is contained in the Laboratory Certification and Registration code chapter, specifically s. NR 149.46(4)(a), Wis. Adm. Code, which states:

"Laboratories shall follow the sample preservation procedures and holding times required by state and federal regulations. If the sample preservation procedures are not required by state or federal regulations, laboratories shall follow the sample preservation procedures and holding times established in the analytical

method. If the analytical method does not establish sample preservation procedures and holding times, laboratories shall follow the procedures in authoritative sources specified in Appendix III of this chapter."

This language means that laboratories must follow all sample preservation and holding times included in state regulations, including ch. NR 219, Table F, Wis. Adm. Code, or 40 CFR Part 136 Table II. Sample preservation for other covered programs are typically contained in the corresponding program's code, e.g., s. NR 809.725 Tables F and G, Wis. Adm. Code for regulated drinking waters. The sample preservation and holding time language contained in approved methods in ch. NR 219, Wis. Adm. Code, for example, is applicable only when there are no preservation or hold time requirements established in state or federal regulations. The sample preservation and holding time requirements contained in authoritative sources, such as *Standard Methods for the Analysis of Water and Wastewater*, 20<sup>th</sup> ed., are applicable only if there are no requirements in state or federal regulations or in approved methods for analyses.

The language contained in the Laboratory Certification and Registration Program code clearly dictates the hierarchy for sample preservation and holding time requirements. This program verifies that samples have been collected, properly preserved and analyzed within holding times for WPDES compliance as part of the on-site evaluation process.

The department does not believe that inclusion of the suggested language is necessary as the hierarchy is addressed elsewhere in Wisconsin Administrative Code.

### Fiscal Estimate — 2007 Session

<input checked="" type="checkbox"/> Original <input type="checkbox"/> Updated  <input type="checkbox"/> Corrected <input type="checkbox"/> Supplemental	LRB Number  Bill Number	Amendment Number if Applicable  Administrative Rule Number NR219
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Subject  
 Analytical Test Procedures and Methods

**Fiscal Effect**

State:  No State Fiscal Effect  
 Indeterminate

Check columns below only if bill makes a direct appropriation or affects a sum sufficient appropriation.

- |  |   |
|--|---|
| <input type="checkbox"/> Increase Existing Appropriation | <input type="checkbox"/> Increase Existing Revenues |
| <input type="checkbox"/> Decrease Existing Appropriation | <input type="checkbox"/> Decrease Existing Revenues |
| <input type="checkbox"/> Create New Appropriation        |   |

Increase Costs — May be possible to absorb within agency's budget.

Yes     No

Decrease Costs

Local:  No Local Government Costs

Indeterminate

1.  Increase Costs  
 Permissive     Mandatory
2.  Decrease Costs  
 Permissive     Mandatory

3.  Increase Revenues  
 Permissive     Mandatory
4.  Decrease Revenues  
 Permissive     Mandatory

5. Types of Local Governmental Units Affected:

Towns     Villages     Cities

Counties     Others    Sanitary Districts

School Districts       WTCS Districts

**Fund Sources Affected**

GPR     FED     PRO     PRS     SEG     SEG-S

**Affected Chapter 20 Appropriations**

**Assumptions Used in Arriving at Fiscal Estimate**

Local governmental laboratories that reference analytical methods that have been deleted may incur small costs to update their procedures to one approved in this chapter. The bulk of the changes that will be required by this sector will be updating analytical method references. If the change is to an updated version of a similar method, where there are no changes to the analytical process, costs will be minimal. Some facilities may opt to use newer techniques that take less time, or utilize new instrumentation. The proposed rule does not require laboratories to make expenditures of this type to maintain compliance.

Five sanitary district laboratories currently maintain certification to perform oil and grease determinations utilizing freon as an extraction solvent; one district also performs the hexane extractable materials method. The four remaining laboratories have indicated that they will no longer perform this testing in-house due to time constraints, complexity of the method or the cost to implement the new method, when compared to subcontracting the analyses. The cost to subcontract these analyses will likely be much lower than the purchase of freon; one laboratory indicated their last purchase of freon, 60 pounds, cost in excess of \$1400. Manufacture of this solvent has been eliminated in the U.S. as a result of the Montreal Protocol legislation of the 1990's, which restricted the manufacture and sale of ozone-depleting substances.

It is highly possible that the vast majority of governmental laboratories will not be affected by this rule, since 500 methods are being maintained, 62 have been proposed for deletion, and over 300 new methods will be added to the various tables in this chapter.

**Long-Range Fiscal Implications**

None.

Prepared By:	Telephone No.	Agency
Joe Polasek	266-2794	Department of Natural Resources
Authorized Signature	Telephone No.	Date (mm/dd/ccyy)
	266-2794	

## Fiscal Estimate — 2007 Session

### Page 2 Assumptions Narrative Continued

LRB Number	Amendment Number if Applicable
Bill Number	Administrative Rule Number

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Assumptions Used in Arriving at Fiscal Estimate – Continued

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**Fiscal Estimate Worksheet — 2007 Session**  
 Detailed Estimate of Annual Fiscal Effect

Original       Updated  
 Corrected       Supplemental

LRB Number	Amendment Number if Applicable
Bill Number	Administrative Rule Number NR 219

Subject  
 Analytical Test Procedures and Methods

One-time Costs or Revenue Impacts for State and/or Local Government (do not include in annualized fiscal effect):

Annualized Costs:		Annualized Fiscal Impact on State Funds from:	
		Increased Costs	Decreased Costs
<b>A. State Costs by Category</b>			
State Operations — Salaries and Fringes		\$ 0	\$ - 0
(FTE Position Changes)		( 0.00 FTE )	(- 0.00 FTE )
State Operations — Other Costs		0	- 0
Local Assistance		0	- 0
Aids to Individuals or Organizations		0	- 0
<b>Total State Costs by Category</b>		\$ 0	\$ - 0
<b>B. State Costs by Source of Funds</b>		<b>Increased Costs</b>	<b>Decreased Costs</b>
GPR		\$ 0	\$ - 0
FED		0	- 0
PRO/PRS		0	- 0
SEG/SEG-S		0	- 0
State Revenues	Complete this only when proposal will increase or decrease state revenues (e.g., tax increase, decrease in license fee, etc.)	<b>Increased Revenue</b>	<b>Decreased Revenue</b>
GPR Taxes		\$	\$ -
GPR Earned			-
FED			-
PRO/PRS			-
SEG/SEG-S			-
<b>Total State Revenues</b>		\$	\$ -

**Net Annualized Fiscal Impact**

	<u>State</u>	<u>Local</u>
Net Change in Costs	\$ 0	\$
Net Change in Revenues	\$ 0	\$

Prepared By: Joe Polasek	Telephone No. 266-2794	Agency Department of Natural Resources
Authorized Signature	Telephone No. 266-2794	Date (mm/dd/ccyy)

ORDER OF THE STATE OF WISCONSIN NATURAL RESOURCES BOARD  
REPEALING AND RECREATING, AND CREATING RULES

The Wisconsin Natural Resources Board proposes an order to amend s. NR 219.04 (2) and (3) (intro), to repeal and recreate NR 210.04 Tables A, B, C, D, E, EM and F; and to create s. NR 219.04 (4) relating to analytical methods used for Wisconsin Pollutant Discharge Monitoring System (WPDES) compliance monitoring.

SS-16-08

Summary Prepared by the Department of Natural Resources

1. Statutes Interpreted

Sections 283.31, 283.55 (1), 299.11 and 299.15(2), Stats.

2. Statutory Authority

Sections 227.11 (2), 281.19(1), 283.31, 283.55(1), 299.11, and 299.15(2), Stats.

3. Explanation of Agency Authority

Section 281.19(1), Stats. authorizes the department to issue general orders, and adopt rules applicable throughout the state for the construction, installation, use and operation of systems, methods and means to prevent and abate pollution of the waters of the state. Section 283.31, Stats. prohibits the discharge of any pollutant into any waters of the state or the disposal of sludge from a treatment work by any person unless such discharge or disposal is done under a permit issued by the department. Section 283.55(1), Stats. requires permittees to use monitoring methods, including where appropriate, biological monitoring methods, to identify and determine the amount of each pollutant discharged from each point source under the owner's or operator's ownership or control. Section 299.15(2)(a), Stats. authorizes the department by rule to prescribe methods of analysis for pollutants.

4. Related Statute or Rule

Chs. NR 101, 102, 106, 149, 157, 200, 204, 205, 206, 210, 211, 212, 214, 216, 217, 218, 230, 233, 243, 252, 254, 256, 260, 261, 262, 263, 270, 273, 274, 279, 290, and 347, Wis. Adm. Code and Chapter 283, Stats.

5. Plain Language Rule Analysis

The analytical methods contained in 40 CFR Part 136, which are used by laboratories in support of WPDES compliance monitoring, have not been substantively updated in several years. Many of the methods that were deleted by EPA were originally published twenty or more years ago. Use of newer laboratory instruments and methods have been hindered by the lack of inclusion in the Federal rule. In March of 2007, EPA published updated analytical test methods. The proposed rule revisions to chapter NR 219 incorporate these updated methods for the WPDES permit program.

The department is also incorporating methods that have been approved through the Office of Water's Alternative Test Procedure program, including the luminescence technique for dissolved oxygen, and selected methods from the 21<sup>st</sup> Edition of "Standard Methods for the Examination of Water and Wastewater". The tables also include updated references from "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Updates III and IV", the "2007 Annual Book of Standards" from ASTM and developed by instrument manufacturers.

The maximum holding time requirement for some tests, as listed in Table F, has been clarified. The current language, "analyze immediately", has been interpreted to mean within 15 minutes or less of sample collection. This has been clarified in the federal rule so that the holding time for these tests is now "analyze within 15 minutes". Other changes to this table include identification of holding times for the various matrices that require dioxins and furan analysis, and allowing metals samples to be transported to a laboratory without pH adjustment. Preservation requirements for available and total cyanide have been expanded to describe procedures that are recommended for removal or suppression of known cyanide interferences.

This proposal also changes language in ss. NR 219.04 (2) and (3) to make this language consistent with the thermal preservation requirements contained in Table F.

6. Summary of, and Comparison with, Existing or Proposed Federal Regulations

The federal counterpart to this rule is 40 CFR 136. On March 12, 2007, EPA published "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; National Primary Drinking Water Regulations; and National Secondary Drinking Water Regulations; Analysis and Sampling Procedures; Final Rule" In addition, on March 26, 2007, EPA published the "Guidelines Establishing Test Procedures for the Analysis of Pollutants; Analytical Methods for Biological Pollutants in Wastewater and Sewage Sludge; Final Rule." The proposed revision to ch. NR 219 incorporates changes and are consistent with the federal regulations.

7. Comparison with Rules in Adjacent States

The States of Illinois, Iowa, Michigan, and Minnesota have wastewater programs delegated to them from the U.S. Environmental Protection Agency. The Illinois laboratory certification program required laboratories to update their scope of accreditation to incorporate the revisions starting June 1, 2007. The IL Environmental Protection Agency did not require rulemaking to address these updates. The Iowa Department of Natural Resources incorporated the changes at 40 CFR 136 in rulemaking initiated in November 2007; the changes were effective January 9, 2008. The State of Michigan does not certify laboratories that perform analyses of wastewater. The Minnesota laboratory certification program allowed laboratories to use the analytical methods listed in the federal rules on the effective dates, April 11 and 25, 2007, respectively. Minnesota's program limits analytical methods to those from the 20<sup>th</sup> and 21<sup>st</sup> Editions and on-line version of "Standard Methods for the Examination of Water and Wastewater", and limits ASTM methods to the most-recently published version. All other sources of analytical methods were incorporated as described in the federal rule. MN Department of Public Health did not require rulemaking to address these updates.

8. Summary of Factual Data and Analytical Methodologies

This proposed rule revision brings Wisconsin up to date with the current federal rules that establish analytical test methods.

9. Analysis and Supporting Documents Used to Determine Effect on Small Business or in Preparation of Economic Impact Report

Many of the analytical methods that are being deleted have been replaced with methods that are nearly identical. The quality control requirements for analyses are established in ch. NR 149; these will not change with updating methods tables. For several analyses, newer methods and techniques have been added. It is possible for a laboratory to change the analytical method it uses and not incur additional costs or, in some cases, actually reduce costs.

The requirements imposed upon small business include following approved analytical methods listed in the rule. There are no reporting requirements in NR 219. The small businesses that will likely be impacted by this rule are commercial laboratories certified under ch. NR 149. The vast majority of these laboratories

likely follow approved analytical methods that are being maintained in this chapter. The certification status for each small business that was certified for oil and grease was examined to determine whether they performed the deleted and retained methods. Only one laboratory would have to change their certification status. The impacted laboratory has since obtained certification for the retained method for oil and grease determinations.

This proposal does not change the frequency of analytical testing, nor does it address any change in reporting, schedule or deadline requirements. Consequently, the impacts to small businesses will be minimal.

#### 10. Effect on Small Business

The proposed changes to ch. NR 219 potentially affect all certified and registered laboratories and wastewater facilities that collect samples for compliance with their Wisconsin Pollutant Discharge Elimination System (WPDES) permit. There are 420 laboratories certified or registered to perform analyses by the department. Of these, 264 municipal wastewater laboratories, 60 industrial environmental, and 78 commercial environmental will be impacted by this rule. The remaining laboratories are public health laboratories that analyze drinking water and those that perform testing at hazardous waste facilities. This update will allow laboratories to utilize new techniques that currently require approval by the U. S. Environmental Protection Agency and deletes outdated methods that lack adequate quality control procedures.

Of the approximately 400 certified or registered laboratories that will be impacted by this rule, there are less than 12, 3% of the total, that fit the definition of "small businesses". This proposed rule would affect commercial, industrial, and municipal laboratories that analyze effluent discharges to determine compliance with WPDES permit limitations. All laboratories performing analyses to determine WPDES compliance are expected to keep detailed records of all laboratory information, including: analytical methods and results, corrective action logs, equipment records, quality control records, and sample receipt logbooks.

This proposal does not change analytical testing requirements, nor does it address any change in reporting, schedule or deadline requirements. Sections 299.11 and 283.55, Stats., do not allow for less stringent schedules, deadlines or reporting requirements. Laboratories may seek approval of an alternate test procedure, as specified in s. NR 219.05, Wisc. Adm. Code, from U.S. EPA. WPDES permit terms and conditions and other related regulations require that permitted facilities conduct analytical tests in compliance with the procedures of ch. NR 219. The Laboratory Certification and Registration Program audits laboratories performing these analyses to ensure that testing is performed in compliance with the proposed rule. If a laboratory does not follow an approved method, it is cited during the audit process; a laboratory must correct all identified deficiencies to maintain their certification under ch. NR 149.

#### 11. Anticipated Costs Incurred by Private Sector

The vast majority of laboratories will not incur additional costs as a result of this rulemaking. The replacement of deleted methods with similar, and in some cases, identical language, does not require laboratories to incur additional costs. The laboratory community was advised of EPA's intent to delete analytical methods when the draft rule was published in 2003. In 2004 revisions to chapter NR 219, Wis. Adm. Code, the department incorporated many updated methods from "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846".

Many of the laboratories that perform oil and grease determinations have maintained certifications for both the freon and hexane extractable materials techniques. The use of freon as an extraction solvent for oil and grease determinations, was eliminated by EPA in 2001. Since that time, the department has worked to modify permit analytical requirements to utilize the hexane extractable material method and eliminate the use of freon. Those laboratories that perform oil and grease determinations will see a cost-savings by the elimination of the freon technique. One laboratory noted a 60-pound container of freon cost approximately

\$1,400; the same volume of hexane costs approximately \$140. Utilization of hexane instead of freon as an extraction solvent for oil and grease results in significant cost-savings for laboratories.

12. Agency Contact Person

Diane Drinkman, Audit Chemist  
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Section 1: NR 219.04 (2) and (3)(intro) are amended to read:

(2) SAMPLE PRESERVATION PROCEDURES. Sample preservation techniques, container materials, and maximum allowable holding times for parameters identified in tables A to E are prescribed in table F. Sludge samples shall be preserved at the time of collection by cooling to 4 less than or equal to 6° C where required. All samples requiring thermal preservation at 4 less than or equal to 6° C shall be cooled immediately after collection, and the required temperature maintained during shipping. Any person may apply for a variance from the prescribed preservation procedures applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the regional administrator and shall provide sufficient data to assure that the variance does not adversely affect the integrity of the sample. The regional administrator will make a decision on whether to approve or deny a variance within 90 days of receipt of the application.

(3) TEMPERATURE REPORTING PROCEDURES. (intro) Samples cooled with ice packs or not in direct contact with ice during shipping shall be cooled to -4 less than or equal to 6° C prior to shipping, and a temperature blank shall be submitted with the samples. Samples cooled during shipping with ice packs may not be recorded as received on ice. Samples may be recorded as received on ice only if solid ice is present in the cooler at the time the samples are received. If the samples are not received on ice, the laboratory shall record one of the following at the time of receipt:

Section 2: NR 219.04 (4) is created to read:

(4). INCORPORATION BY REFERENCE. The materials in this section are incorporated by reference for the purposes of the permit program under chapter 283, Stats.

**Note:** Copies of the publications referenced in Tables A-F are available for inspection at the offices of the department of natural resources, ~~the secretary of state and the revisor of statutes~~ and the legislative reference bureau. Many of these materials are also available through inter-library loan.

Section 3: NR 219.04 Table A is repealed and recreated to read:

Table A  
List of Approved Biological Analytical Methods

Parameter and Units	Analytical Technology <sup>1</sup>	EPA	Standard Methods <sup>4</sup>	Standard Methods Online <sup>12</sup>	AOAC, ASTM, USGS	Other
<b>Bacteria:</b>						
1. Coliform (fecal), number per 100 mL or number per gram dry weight	Most Probable Number (MPN), 5 tube 3 dilution, or	p. 132 <sup>3</sup> , 1680 <sup>12</sup> , 14, 1681 <sup>12, 19</sup>	9221 C or E [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9221 C-99 or 9221 E-99		
	Membrane filter (MF) <sup>2</sup> , single step	p. 124 <sup>3</sup>	9222 D [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9222 D-97	B-0050-85 <sup>5</sup>	
	MPN, multiple tube/multiple well		9223 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9223 B-97	991.15 <sup>11</sup>	Colilert <sup>®13, 17</sup> , Colilert-18 <sup>® 13, 16, 17</sup>
2. Coliform (fecal) in presence of chlorine, number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 132 <sup>3</sup>	9221 C or E [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9221 C-99 or 9221 E-99		
	MPN, multiple tube/multiple well, or		9223 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9223 B-97	991.15 <sup>11</sup>	Colilert <sup>®13, 17</sup> , Colilert-18 <sup>® 13, 16, 17</sup>
	MF <sup>2</sup> , single step	p. 124 <sup>3</sup>	9222 D [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9222 D-97		
3. Coliform (total), in number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 114 <sup>3</sup>	9221 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9221 B-99		
	MF <sup>2</sup> , single step or two step	p. 108 <sup>3</sup>	9222 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9222 B-97	B-0025-85 <sup>5</sup>	
4. Coliform (total), in presence of chlorine, number per 100 mL	MPN, 5 tube, 3 dilution, or	p. 114 <sup>3</sup>	9221 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9221 B-99		
	MF <sup>2</sup> with enrichment	p. 111 <sup>3</sup>	9222 (B+B, 5c) [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9222 (B+B, 5c)-97		
5. <i>E. coli</i> , number per 100 mL <sup>20</sup>	MPN <sup>7, 9, 15</sup> , multiple tube		9221 B.1 or 9221F [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ] <sup>22, 25</sup>	9221 B.1 or F-99 <sup>22, 25</sup>		
	MPN, multiple tube/multiple well		9223 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ] <sup>13</sup>	9223 B-97 <sup>13</sup>	991.15 <sup>11</sup>	Colilert <sup>®13, 17</sup> , Colilert-18 <sup>® 13, 16, 17</sup>
	MF <sup>2, 6, 7, 8, 9</sup> two step, or	1103.1 <sup>27</sup>	9222 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ] <sup>26</sup> , 9222 G [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ] <sup>26</sup> , 9213 D [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]	9222 B-97 <sup>26</sup> or G-97 <sup>26</sup>	D5392-93 <sup>10</sup>	
	MF <sup>2, 6, 7, 8, 9</sup> single step	1603 <sup>21</sup> , 1604 <sup>28</sup>				mColiBlue-24 <sup>® 18</sup>
6. Fecal streptococci, number per 100 mL	MPN, 5 tube, 3 dilution,	p. 139 <sup>3</sup>	9230 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9230 B-93		
	MF <sup>2</sup> , or	P. 136 <sup>3</sup>	9230 C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9230 C-93	B-0055-85 <sup>5</sup>	
	Plate count	p. 143 <sup>3</sup>				
7. Enterococci, number per 100 mL <sup>20</sup>	MPN <sup>7, 9</sup> , multiple tube,		9230 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9230 B-93		
	MPN, multiple tube/multiple well,				D6503-99 <sup>10</sup>	Enterolert <sup>® 13, 23</sup>
	MF <sup>2</sup> , two step,	1106.1 <sup>29</sup>	9230 C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	9230 C-93	D5259-92 <sup>10</sup>	
	MF <sup>2, 6, 7, 8, 9</sup> single step, or	1600 <sup>24</sup>				
	Plate count	p. 143 <sup>3</sup>				
<b>Protozoa:</b>						
8. Cryptosporidium	Filtration/IMS/FA	1622 <sup>30</sup> , 1623 <sup>31</sup>				
9. Giardia	Filtration/IMS/FA	1623 <sup>31</sup>				
<b>Aquatic Toxicity:</b>						
10. Toxicity, acute, fresh water organisms, percent effluent	Ceriodaphnia, 48-h static-renewal mortality					Note 32

Parameter and Units	Analytical Technology <sup>1</sup>	EPA	Standard Methods <sup>4</sup>	Standard Methods Online <sup>12</sup>	AOAC, ASTM, USGS	Other
	Fathead minnow, 96-h static renewal mortality, or 96-h flow-through mortality					Note 32
11. Toxicity, chronic, fresh water organisms, percent effluent	Ceriodaphnia survival and reproduction					Note 32
	Fathead minnow larval survival and growth					Note 32

<sup>1</sup> The method must be specified when results are reported.

<sup>2</sup> A 0.45 µm membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.

<sup>3</sup> U.S. EPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH, EPA/600/8-78/017.

<sup>4</sup> "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 21<sup>st</sup> Edition (2005), 20th Edition (1998), 19th Edition (1995), and 18th Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

<sup>5</sup> USGS. 1989. U.S. Geological Survey Techniques of Water-Resource Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, U.S. Geological Survey, U.S. Department of the Interior, Reston, VA.

<sup>6</sup> Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.

<sup>7</sup> Tests must be conducted to provide organism enumeration (density). Select the appropriate configuration of tubes/filtrations and dilutions/volumes to account for the quality, character, consistency, and anticipated organism density of the water sample.

<sup>8</sup> When the MF method has been used previously to test waters with high turbidity, large numbers of noncoliform bacteria, or samples that may contain organisms stressed by chlorine, a parallel test should be conducted with a multiple-tube technique to demonstrate applicability and comparability of results.

<sup>9</sup> To assess the comparability of results obtained with individual methods, it is suggested that side-by-side tests be conducted across seasons of the year with the water samples routinely tested in accordance with the most current Standard Methods for the Examination of Water and Wastewater or EPA alternate test procedure (ATP) guidelines.

<sup>10</sup> ASTM. 2000, 1999, 1996. Annual Book of ASTM Standards—Water and Environmental Technology, Section 11.02. ASTM International. 100 Barr Harbor Drive, West Conshohocken, PA 19428.

<sup>11</sup> AOAC. 1995. Official Methods of Analysis of AOAC International, 16th Edition, Volume I, Chapter 17. Association of Official Analytical Chemists International. 481 North Frederick Avenue, Suite 500, Gaithersburg, MD 20877-2417.

<sup>12</sup> "Standard Methods for the Examination of Water and Wastewater On-Line", Joint Editorial Board, American Public Health Association, American Water Works Association, Water Environment Federation, 2006. Subscription service available at: <http://www.standardmethods.org>.

<sup>13</sup> These tests are collectively known as defined enzyme substrate tests, where, for example, a substrate is used to detect the enzyme b-glucuronidase produced by *E. coli*.

<sup>14</sup> U.S. EPA. July 2006. Method 1680: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation Using Lauryl-Tryptose Broth (LTB) and EC Medium. US Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-012.

<sup>15</sup> Samples shall be enumerated by the multiple-tube or multiple-well procedure. Using multiple-tube procedures, employ an appropriate tube and dilution configuration of the sample as needed and report the Most Probable Number (MPN). Samples tested with Colilert<sup>®</sup> may be enumerated with the multiple-well procedures, Quanti-Tray<sup>®</sup> Quanti-Tray<sup>®</sup> 2000, and the MPN calculated from the table provided by the manufacturer.

<sup>16</sup> Colilert-18<sup>®</sup> is an optimized formulation of the Colilert<sup>®</sup> for the determination of total coliforms and *E. coli* that provides results within 18 h of incubation at 35 °C rather than the 24 h required for the Colilert<sup>®</sup> test and is recommended for marine water samples.

<sup>17</sup> Descriptions of the Colilert<sup>®</sup>, Colilert-18<sup>®</sup>, Quanti-Tray<sup>®</sup>, and Quanti-Tray<sup>®</sup>/2000 may be obtained from IDEXX Laboratories, Inc., 1 IDEXX Drive, Westbrook, ME 04092.

<sup>18</sup> A description of the mColiBlue24<sup>®</sup> test, Total Coliforms and *E. coli*, is available from Hach Company, 100 Dayton Ave., Ames, IA 50010.

<sup>19</sup> U.S. EPA. July 2006. Method 1681: Fecal Coliforms in Sewage Sludge (Biosolids) by Multiple-Tube Fermentation using A-1 Medium. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-013.

<sup>20</sup> Recommended for enumeration of target organism in wastewater effluent.

<sup>21</sup> U.S. EPA. July 2006. Method 1603: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using Modified membrane-Thermotolerant *Escherichia coli* Agar (modified mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-011.

<sup>22</sup> The multiple-tube fermentation test is used in 9221B.1. Lactose broth may be used in lieu of lauryl tryptose broth (LTB), if at least 25 parallel tests are conducted between this broth and LTB using the water samples normally tested, and this comparison demonstrates that the false-positive rate and false-negative rate for total coliform using lactose broth is less than 10 percent. No requirement exists to run the completed phase on 10 percent of all total coliform-positive tubes on a seasonal basis.

<sup>23</sup> A description of the Enterolert<sup>®</sup> test may be obtained from IDEXX Laboratories, Inc., 1 IDEXX Drive, Westbrook, ME 04092.

<sup>24</sup> U.S. EPA. July 2006. Method 1600: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus Indoxyl-b-D-Glucoside Agar (mEI). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-009.

<sup>25</sup> After prior enrichment in a presumptive medium for total coliform using 9221B.1, all presumptive tubes or bottles showing any amount of gas, growth or acidity within 48 h ± 3 h of incubation shall be submitted to 9221F. Commercially available EC-MUG media or EC media supplemented in the laboratory with 50 µg/mL of MUG may be used.

<sup>26</sup> Subject total coliform positive samples determined by 9222B or other membrane filter procedure to 9222G using NA-MUG media.

<sup>27</sup> U.S. EPA. July 2006. Method 1103.1: *Escherichia coli* (*E. coli*) in Water by Membrane Filtration Using membrane-Thermotolerant *Escherichia coli* Agar (mTEC). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-010.

<sup>28</sup> U. S. EPA, September 2002, Method 1604: Total Coliforms and *Escherichia coli* (*E. coli*) in Water by Membrane Filtration by Using a Simultaneous Detection Technique (MI Medium). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA 821-R-02-024.

<sup>29</sup> U.S. EPA. July 2006. Method 1106.1: Enterococci in Water by Membrane Filtration Using membrane-Enterococcus-Esculin Iron Agar (mE- EIA). U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-06-008.

<sup>30</sup> Method 1622 uses filtration, concentration, immunomagnetic separation of oocysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the detection of *Cryptosporidium*. U.S. EPA. 2001. Method 1622: *Cryptosporidium* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-01-026.

<sup>31</sup> Method 1623 uses filtration, concentration, immunomagnetic separation of oocysts and cysts from captured material, immunofluorescence assay to determine concentrations, and confirmation through vital dye staining and differential interference contrast microscopy for the simultaneous detection of *Cryptosporidium* and *Giardia* oocysts and cysts. U.S. EPA. 2001. Method 1623. *Cryptosporidium* and *Giardia* in Water by Filtration/IMS/FA. U.S. Environmental Protection Agency, Office of Water, Washington, DC EPA-821-R-01-025.

<sup>32</sup> Compliance monitoring must be performed in accordance with the specifications in the "State of Wisconsin Aquatic Life Toxicity Testing Methods Manual, 2<sup>nd</sup> Edition," Wisconsin Department of Natural Resources, 2004. This publication is available for inspection at the offices of the Department of Natural Resources and the Legislative Reference Bureau. Copies are available from the Department of Natural Resources, Bureau of Science Services, P.O. Box 7921, Madison, WI 53707.

Section 4: NR 219.04 Table B is repealed and recreated to read:

Table B  
List of Approved Inorganic Analytical Methods in Wastewater

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
1. Acidity, as CaCO <sub>3</sub> , mg/L:	Electrometric or phenolphthalein endpoint			2310 B(4a) [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	2310 B(4a)-97	D1067-92, 02, 06	I-1020-85 <sup>2</sup>	
2. Alkalinity, as CaCO <sub>3</sub> , mg/L:	Electrometric or colorimetric titration to pH 4.5, manual, or automatic	310.2, (Rev. 1974) <sup>1</sup>		2320 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	2320 B-97	D1067-92, 02, 06	I-1030-85 <sup>2</sup>	973.43 <sup>3</sup>
3. Aluminum- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:						I-2030-85 <sup>2</sup>	
	AA direct aspiration (FLAA)		7000B	3111 D [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 D-99		I-3051-85 <sup>2</sup>	
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99			

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120B [21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>2</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP), or					D4190-94, 99, 03		Note 43
	Colorimetric (Eriochrome cyanine R)			3500-Al D [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500 Al B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-Al B-01			
4. Ammonia (as N), mg/L:	Manual distillation (at pH 9.5) followed by:	350.1, Rev. 2.0 (1993)		4500- NH <sub>3</sub> B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500- NH <sub>3</sub> B-97			973.49 <sup>3</sup>
	Titration			4500- NH <sub>3</sub> E [18 <sup>th</sup> ], 4500- NH <sub>3</sub> C [19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500- NH <sub>3</sub> -C-97			
	Electrode			4500- NH <sub>3</sub> F or G [18 <sup>th</sup> ], 4500- NH <sub>3</sub> D or E [19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500- NH <sub>3</sub> D or E-97	D1426-98, 03 (B)		
	Automated phenate	350.1, Rev. 2.0, (1993) <sup>70</sup>		4500- NH <sub>3</sub> H [18 <sup>th</sup> ], 4500- NH <sub>3</sub> G [19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500- NH <sub>3</sub> G-97		I-4523--85 <sup>2</sup>	
	Automated electrode, or							Note 15
	Ion chromatography					D6919-03		
5. Antimony- Total <sup>9</sup> , ug/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99			
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99			
	Inductively coupled plasma-atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
6. Arsenic- -Total <sup>9</sup> , ug/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA gaseous hydride		7061A	3114 B <sup>10</sup> [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3114 B 4.d.-97	D2972-97, 03 (B)	I-3062-85 <sup>2</sup>	

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99	D2972-97, 03 (C)	I-4063-98 <sup>59</sup>	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Colorimetric (SDCC)			3500-As C [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-As B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-As B-97	D2972-97, 03(A)	I-3060-85 <sup>2</sup>	
7. Barium- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 D-99		I-3084-85 <sup>2</sup>	
	AA graphite furnace (GFAA)		7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99	D4382-95, 02		
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP)							Note 43
8. Beryllium- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 D-99	D3645-93 (88), 03 (A)	I-3095-85 <sup>2</sup>	
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99	D3645-93 (88), 03 (B)		
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP), or					D4190-94, 99, 03		Note 43
	Colorimetric (Aluminon)			3500-Be D [18 <sup>th</sup> , 19 <sup>th</sup> ]				

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
9. Biochemical Oxygen Demand (BOD <sub>5</sub> ), mg/L:	Dissolved oxygen depletion			5210 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	5210 B-01		I-1578-78 <sup>16</sup>	973.443 <sup>3</sup> , p17 <sup>16</sup>
10. Boron <sup>46</sup> , mg/L:	Colorimetric (Curcumin)			4500-B B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-B B-00		I-3112-85 <sup>2</sup>	
	Inductively coupled plasma-atomic emission spectrometry (ICP) <sup>11, 45</sup>	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
	Direct current plasma (DCP) <sup>11, 45</sup>					D4190-94, 99, 03		Note 43
11. Bromide, mg/L:	Titrimetric					D1246-95, 99 (C)	I-1125-85 <sup>2</sup>	p. S44 <sup>18</sup>
	Ion chromatography, or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	4110 B-00	D4327-97, 03		993.30 <sup>3</sup>
	CIE/UV					D6508-00 (05)		D6508, Rev 2 <sup>64</sup>
12. Cadmium- Total <sup>9</sup> mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B or C-99	D3557-95, 02 (A or B)	I-3135-85 <sup>2</sup> or I-3136-85 <sup>2</sup>	974.27 <sup>3</sup> , p. 37 <sup>17</sup>
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99	D3557-95, 02 (D)	I-4138-89 <sup>61</sup>	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-1472-85 <sup>2</sup> or I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP)					D4190-94, 99, 03		Note 43
	Voltammetry <sup>19</sup> , or					D3557-95, 02 (C)		
	Colorimetric (Dithizone)			3500-Cd D [18 <sup>th</sup> , 19 <sup>th</sup> ]				

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
13. Calcium- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99	D511-93, 03 (B)	I-3152-85 <sup>2</sup>	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
	Direct current plasma (DCP)							Note 43
	Titrimetric (EDTA) , or			3500-Ca D [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-Ca B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-Ca B-97	D511-93, 03 (A)		
	Ion chromatography					D6919-03		
14. Carbonaceous Biochemical Oxygen Demand (CBOD <sub>5</sub> ) <sup>20</sup> , mg/L:	Dissolved oxygen depletion with nitrification inhibitor			5210 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	5210 B-01			
15. Chemical Oxygen Demand (COD), mg/L:	Titrimetric, or	410.3, (Rev. 1978) <sup>1</sup>		5220 C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	5220 C-97	D-1252-95, 00, 06 (A)	I-3560-85 <sup>2</sup>	973.46 <sup>3</sup> and p.17 <sup>17</sup>
	Spectrophotometric, manual or automatic	410.4, Rev 2.0 (1993)		5520 D [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	5220 D-97	D1252-95, 00, 06 (B)	I-3561-85 <sup>2</sup>	Notes 21, 22
16. Chloride, mg/L:	Titrimetric (Silver nitrate) or		9253	4500-Cl B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-Cl B-97	D512-89 (99), 04 (B)	I-1183-85 <sup>2</sup>	
	Colorimetric; manual or,						I-1187-85 <sup>2</sup>	
	automated (Ferricyanide)		9250	4500-Cl E [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-Cl E-97		I-2187-85 <sup>2</sup>	
	Potentiometric titration			4500-Cl D [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-Cl D-97			
	Ion selective electrode					D512-89 (99), 04 (C)		
	Ion chromatography, or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	4110 B-00	D4327-97, 03		993.30 <sup>3</sup>
	CIE/UV					D6508-00 (05)		D6508, Rev. 2 <sup>64</sup>

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
17. Chlorine, Total Residual, mg/L:	Amperometric direct			4500-Cl D [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-Cl D-00	D1253-86 (96), 03		
	Amperometric direct (low level)			4500-Cl E [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-Cl E-00			
	Iodometric direct			4500-Cl B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-Cl B-00			
	Back titration either end-point <sup>23</sup>			4500-Cl C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-Cl C-00			
	DPD-FAS			4500-Cl F [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-Cl F-00			
	Spectrophotometric, DPD, or			4500-Cl G [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-Cl G-00			
	Ion selective electrode							Note 24
18. Chromium VI, dissolved, ug/L:	0.45 micron filtration followed by:							
	AA chelation-extraction		7197	3111 C [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 C-99		I-1232-85 <sup>2</sup>	
	Ion chromatography, or	218.6, Rev. 3.3 (1994)	7199	3500-Cr E [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-Cr C [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-Cr C-01	D5257-97, 03		993.23 <sup>3</sup>
	Colorimetric (Diphenylcarbazide)			3500-Cr D [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-Cr B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-Cr B-01	D1687-92, 02 (A)	I-1230-85 <sup>2</sup>	
19. Chromium- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99	D1687-92, 02 (B)	I-3236-85 <sup>2</sup>	974.27 <sup>3</sup>
	AA chelation-extraction			3111 C [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 C-99			
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99	D1687-92, 02 (C)	I-3233-93 <sup>56</sup>	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP), or					D4190-94, 99, 03		Note 43
	Colorimetric (Diphenylcarbazide)			3500-Cr D [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-Cr B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-Cr B-01			

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
20. Cobalt- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B or C-99	D3558-94, 03 (A or B)	I-3239-85 <sup>2</sup>	p 37 <sup>17</sup>
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99	D3558-94, 03 (C)	I-4243-89 <sup>61</sup>	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP)					D4190-94, 99, 03		Note 43
21. Color, platinum cobalt units or dominant wavelength, hue, luminance, purity:	Colorimetric (ADMI)			2120 E [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]				Note 26
	(Platinum cobalt), or			2120 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	2120 B-01		I-1250-85 <sup>2</sup>	
	Spectrophotometric			2120 C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]				
22. Copper- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B or C-99	D1688-95, 02 (A or B)	I-3270-85 <sup>2</sup> or I3271-85 <sup>2</sup>	974.37 <sup>3</sup> , p 37 <sup>17</sup>
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99	D1688-95, 02 (C)	I-4274-89 <sup>61</sup>	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP)					D4190-94, 99, 03		Note 43
	Colorimetric (Neocuproine), or			3500-Cu D [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-Cu B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-Cu B-99			
	(Bicinchoninate)			3500-Cu E [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-Cu C [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-Cu C-99			Note 27

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
23. Cyanide, Total, ug/L:	Automated Distillation and Colorimetry							Kelada-01 <sup>65</sup>
	Manual distillation with MgCl <sub>2</sub> followed by:	335.4, Rev. 1.0 (1993) <sup>67</sup>	9010B, 9010C	4500-CN C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]		D2036-98, 06(A)		10-204-00-1-X <sup>66</sup>
	Titrimetric		9014	4500-CN D [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-CN D-99			p.22 <sup>17</sup>
	Spectrophotometric, manual		9014	4500-CN E [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-CN E-99	D2036-98, 06 (A)	I-3300-85 <sup>2</sup>	
	Automated <sup>28</sup> , or	335.4, Rev. 1.0 (1993) <sup>67</sup>	9012A, 9012B				I-4302-85 <sup>2</sup>	10-204-00-1-X <sup>66</sup>
	Ion selective electrode			4500-CN F [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-CN F-99	D2036-98, 06 (A)		
24. Cyanide, Available, ug/L:	Cyanide Amenable to Chlorination (CATC); Manual distillation with MgCl <sub>2</sub> followed by Titrimetric or Spectrophotometric		9010B, 9010C, 9014	4500-CN G [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-CN G-99	D2036-98, 06 (B)		
	Flow injection and ligand exchange, followed by amperometry <sup>71</sup> , or					D6888-04		OIA-1677 <sup>54</sup>
	Automated distillation and colorimetry		9012A					Kelada-01 <sup>65</sup>
25. Fluoride- Total, mg/L:	Manual distillation <sup>14</sup> followed by:			4500-F B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-F B-97			
	Electrode, manual			4500-F C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-F C-97	D1179-93, 99, 04 (B)		
	Automated						I-4327-85 <sup>2</sup>	
	Colorimetric (SPADNS)			4500-F D [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-F D-97	D1179-93, 99, 04 (A)		
	Automated complexone			4500-F E [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-F E-97			
	Ion chromatography, or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	4110 B-00	D4327-97, 03		993.30 <sup>3</sup>

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
	CIE/UV					D6508-00 (05)		D6508, Rev. 2 <sup>64</sup>
26. Gold- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99			
	AA graphite furnace (GFAA)	231.2, Rev. 1978 <sup>1</sup>						
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
	Direct current plasma (DCP)							Note 43
27. Hardness, Total as CaCO <sub>3</sub> , mg/L:	Automated colorimetric	130.1, (Issued 1971) <sup>1</sup>						
	Titrimetric (EDTA), or			2340 B or C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	2340 B or C-97	D1126-86 (92), 02	I-1338-85 <sup>2</sup>	973.52B <sup>3</sup>
	Ca plus Mg as their carbonates by inductively coupled plasma-atomic emission spectrometry (ICP) or AA direct aspiration (See Parameters 13 and 33)							
28. Hydrogen ion (pH), pH units:	Electrometric measurement or		9040C	4500-H <sup>+</sup> B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-H <sup>+</sup> B-00	D1293-84 (90), 99 (05) (A or B)	I-1586-85 <sup>2</sup>	973.41 <sup>3</sup>
	Automated electrode	150.2, (Dec. 1982) <sup>1</sup>					I-2587-85 <sup>2</sup>	Note 29
29. Iridium- Total <sup>9</sup> , ug/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)			3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99			
	AA graphite furnace (GFAA)	235.2, (Issued 1978) <sup>1</sup>	7010					
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
30. Iron- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B or C-99	D1068-96, 03, 05 (A or B)	I-3381-85 <sup>2</sup>	974.27 <sup>3</sup>
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99	D1068-96, 03, 05 (C)		
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
	Direct current plasma (DCP), or					D4190-94, 99, 03		Note 43
	Colorimetric (Phenanthroline)			3500-Fe D [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-Fe B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-Fe B-97	D1068-96, 03, 05 (D)		Note 30
31. Kjeldahl Nitrogen- Total <sup>12</sup> , (as N), mg/L:	Digestion and distillation <sup>28</sup> followed by:			4500-N <sub>org</sub> B or C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ] and 4500-NH <sub>3</sub> B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-N <sub>org</sub> B or C-97 and 4500-NH <sub>3</sub> B-97	D3590-89, 02 (06) (A)		
	Titration			4500- NH <sub>3</sub> E [18 <sup>th</sup> ], 4500- NH <sub>3</sub> C [19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500- NH <sub>3</sub> C-97	D3590-89, 02 (06) (A)		973.48 <sup>3</sup>
	Electrode			4500- NH <sub>3</sub> F or G [18 <sup>th</sup> ], 4500- NH <sub>3</sub> D or E [19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500- NH <sub>3</sub> D or E-97			
	Automated phenate colorimetric	351.1, (Rev. 1978) <sup>1</sup>					I-4551-78 <sup>16</sup>	
	Semi-automated block digester colorimetric	351.2, Rev. 2.0 (1993)				D3590-89, 02 (06) (B)	I-4515-91 <sup>55</sup>	
	Manual or block digester potentiometric					D3590-89, 02 (06) (A)		
	Block digester, followed by auto distillation and titration, or							Note 48

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
	Flow injection gas diffusion							Note 49
32. Lead- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B or C-99	D3559-96, 03 (A or B)	I-3399-90 <sup>2</sup>	974.27 <sup>3</sup>
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99	D3559-96, 03 (D)	I-4403-89 <sup>61</sup>	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP)					D4190-94, 99, 03		Note 43
	Voltammetry, or					D3559-96, 03 (C)		
	Colorimetric (Dithizone)			3500-Pb D [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-Pb B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-Pb B-97			
33. Magnesium- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99	D511-93, 03 (B)	I-3447-85 <sup>2</sup>	974.27 <sup>3</sup>
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
	Direct current plasma (DCP)							Note 43
	Gravimetric, or			3500-Mg D [18 <sup>th</sup> , 19 <sup>th</sup> ]				
	Ion chromatography					D6919-03		
34. Manganese- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99	D858-95, 02 (A or B)	I-3454-85 <sup>2</sup>	974.27 <sup>3</sup>
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99	D858-95, 02 (C)		

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP)					D4190-94, 99, 03		Note 43
	Colorimetric (Persulfate), or			3500-Mn D [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-Mn B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-Mn B-99			920.203 <sup>3</sup>
	(Periodate)							Note 31
35. Mercury- Total <sup>9</sup> , ug/L:	Cold vapor, manual or	245.1	7470A	3112 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3112 B-99	D3223-97, 02	I-3462-85 <sup>2</sup>	977.22 <sup>3</sup>
	Automated	245.2						
	Purge and trap cold vapor atomic fluorescence spectrometry (CVAFS), or	1631E <sup>52</sup>						
	Cold vapor atomic fluorescence spectrometry (CVAFS) <sup>53</sup>	245.7 <sup>69</sup>						
36. Molybdenum- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 D-99		I-3490-85 <sup>2</sup>	
	AA graphite furnace (GFAA)		7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99		I-3492-96 <sup>57</sup>	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP)							Note 43
37. Nickel- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B or C-99	D1886-90, 94 (98), 03 (A or B)	I-3499-85 <sup>2</sup>	

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99	D1886-90, 94 (98), 03 (C)	I-4503-89 <sup>61</sup>	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP)					D4190-94, 99, 03		Note 43
38. Nitrate (as N), mg/L:	Nitrate-nitrite N minus Nitrite N							
	Ion chromatography	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)		4110 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	4110 B-00	D4327-97, 03		993.30 <sup>3</sup>
	Ion selective electrode, or			4500-NO <sub>3</sub> D [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-NO <sub>3</sub> D-00			
	CIE/UV					D6508-00 (05)		D6508, Rev 2 <sup>64</sup>
39. Nitrate + Nitrite (as N), mg/L:	Cadmium reduction, manual			4500-NO <sub>3</sub> E [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-NO <sub>3</sub> E-00	D3867-99, 04(B)		
	Automated	353.2, Rev. 2.0 (1993)		4500-NO <sub>3</sub> F [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-NO <sub>3</sub> F-00	D3867-99, 04(A)	I-4545-85 <sup>2</sup>	
	Automated hydrazine			4500-NO <sub>3</sub> H [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-NO <sub>3</sub> H-00			
	Ion chromatography <sup>34</sup> , or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	4110 B-00	D4327-03		993.30 <sup>3</sup>
	CIE/UV					D6508-00 (05)		D6508, Rev 2 <sup>64</sup>
40. Nitrite (as N), mg/L:	Spectrophotometric, Manual			4500-NO <sub>2</sub> B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-NO <sub>2</sub> B-00			Note 33
	Automated (Diazotization)						I-4540-85 <sup>2</sup>	

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
	Automated (*bypass cadmium reduction)	353.2, Rev. 2.0 (1993)		4500-NO <sub>3</sub> F [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-NO <sub>3</sub> F-00	D3867-99, 04(A)	I-4545-85 <sup>2</sup>	
	Manual (*bypass cadmium reduction)			4500-NO <sub>3</sub> E [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-NO <sub>3</sub> E-00	D3867-99, 04(B)		
	Ion chromatography <sup>34</sup> , or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	4110 B-00	D4327-97, 03		993.30 <sup>3</sup>
	CIE/UV					D6508-00 (05)		D6508, Rev 2 <sup>64</sup>
41. Oil and Grease- Total recoverable, mg/L:	Hexane extractable material (HEM), or	1664A <sup>50</sup>			5520 B-01			
	Silica-gel treated HEM (SGT-HEM); Silica gel treatment and gravimetry	1664A <sup>50</sup>						
42. Organic Carbon, Total (TOC), mg/L:	Combustion or oxidation			5310 B, C or D [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	5310 B, C or D-00			973.47 <sup>3</sup> , p.14 <sup>32</sup>
43. Organic Halides, Adsorbable (AOX), ug/L:	Adsorption and coulometric titration	1650 <sup>51</sup>						
44. Organic Nitrogen (as N), mg/L:	Kjeldahl nitrogen, total (Parameter 31) minus Ammonia nitrogen (Parameter 4)							
45. Orthophosphate (as P), mg/L:	Ascorbic acid method:							
	Automated	365.1, Rev. 2.0 (1993) <sup>1</sup>		4500-P F [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]			I-4601-85 <sup>2</sup>	973.56 <sup>3</sup>
	Manual single reagent			4500-P E [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]		D515-88(A)		973.55 <sup>3</sup>
	Manual two reagent	365.3, (Issued 1978)						
	Ion chromatography, or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	4110 B-00	D4327-97, 03		993.30 <sup>3</sup>

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
	CIE/UV					D6508-00 (05)		D6508, Rev. 2 <sup>64</sup>
46. Osmium- Total <sup>9</sup> , ug/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 D-99			
	AA furnace	252.2, (Issued 1978) <sup>1</sup>	7010					
	Inductively coupled plasma-atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994) <sup>13</sup>		3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
47. Oxygen, Dissolved, mg/L:	Winkler (azide modification)			4500-O C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-O C-01	D888-92, 03, 05 (A)	I-1575-78 <sup>16</sup>	973.45B <sup>3</sup>
	Electrode, or			4500-O G [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-O G-01	D888-92, 03, 05 (B)	I1576-78 <sup>16</sup>	
	Luminescence					D888-05(C)		Note 72
48. Palladium- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)			3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99			p. S27 <sup>18</sup>
	AA graphite furnace (GFAA)	253.2, (Issued 1978) <sup>1</sup>	7010					p. S28 <sup>18</sup>
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>		3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
	Direct current plasma (DCP)							Note 43
49. Phenols, ug/L:	Manual distillation <sup>35</sup> followed by:	420.1, (Rev. 1978) <sup>1</sup>						Note 36
	Colorimetric (4AAP) manual, or	420.1, (Rev. 1978) <sup>1</sup>	9065					Note 36

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
	Automated	420.4, Rev. 1.0 (1993)	9066					
50. Phosphorus (elemental) mg/L:	Gas-liquid chromatography							Note 37
51. Phosphorus- Total, mg/L:	Persulfate digestion <sup>28</sup> followed by:			4500-P B.5 [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]				973.55 <sup>3</sup>
	Manual	365.3, (Issued 1978) <sup>1</sup>		4500-P F [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]		D515-88(A)	I-4600-85 <sup>2</sup>	973.56 <sup>3</sup>
	Automated ascorbic acid reduction, or	365.1, Rev. 2.0, (1993)		4500-P E [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]				
	Semi-automated block digester	365.4, (Issued 1974) <sup>1</sup>				D515-88(B)	I-4610-91 <sup>58</sup>	
52. Platinum- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)			3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99			
	AA graphite furnace (GFAA)	255.2 <sup>1</sup>	7010					
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>		3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
	Direct current plasma (DCP)							Note 43
53. Potassium- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99		I-3630-85 <sup>2</sup>	973.53 <sup>3</sup>
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
	Flame photometric			3500-K D [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-K B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-K B-99			

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
	Colorimetric, or							317B <sup>25</sup>
	Ion chromatography					D6919-03		
54. Residue, total, mg/L:	Gravimetric, 103-105°C			2540 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	2540 B-97		I-3750-85 <sup>2</sup>	
55. Residue-- filterable, mg/L:	Gravimetric, 180°C			2540 C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	2540 C-97		I-1750-85 <sup>2</sup>	
56. Residue, non-filterable, mg/L:	Gravimetric, 103-105°C post-washing of residue			2540 D [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	2540 D-97		I-3765-85 <sup>2</sup>	
57. Residue,-settleable, mg/L:	Volumetric (Imhoff cone), or gravimetric			2540 F [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	2540 F-97			
58. Residue, volatile mg/L:	Gravimetric, 550° C	160.4 <sup>1</sup>					I-3753-85 <sup>2</sup>	
59. Rhodium- total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)			3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99			
	AA graphite furnace (GFAA)	265.2 <sup>1</sup>	7010					
	Inductively coupled plasma-atomic emission spectrometry (ICP) , or	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
60. Ruthenium- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)			3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99			
	AA graphite furnace (GFAA)	267.2 <sup>1</sup>	7010					
	Inductively coupled plasma-atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
61. Selenium- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA gaseous hydride		7741A	3114 B <sup>10</sup> [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3114 B-97 <sup>10</sup>	D3859-98, 03 (A)	I-3667-85 <sup>2</sup>	

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>		3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99	D3859-98, 03 (B)	I-4668-98 <sup>59</sup>	
	Inductively coupled plasma-atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
62. Silica- Dissolved <sup>46</sup> , mg/L:	0.45 micron filtration followed by:							
	Colorimetric, Manual			4500-Si D [18 <sup>th</sup> , 19 <sup>th</sup> ], 4500-SiO <sub>2</sub> C [20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-SiO <sub>2</sub> C-97	D859-94, 00, 05	I-1700-85 <sup>2</sup>	
	Automated (Molybdosilicate)						I-2700-85 <sup>2</sup>	
	Inductively coupled plasma-atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
63. Silver- Total <sup>9, 38, 40</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B or C-99		I-3720-85 <sup>2</sup>	974.27 <sup>3</sup>
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99		I-4724-89 <sup>61</sup>	
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>2</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP)							Note 43
64. Sodium- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99		I-3735-85 <sup>2</sup>	973.54 <sup>3</sup>

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
	Direct current plasma (DCP)							Note 43
	Flame photometric, or			3500-Na D [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-Na B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-Na B-97			
	Ion chromatography					D6919-03		
65. Specific conductance, micromohs/cm at 25° C:	Wheatstone bridge	120.1 <sup>1</sup>	9050A	2510 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	2510 B-97	D1125-95 (99), (05) (A)	I-2781-85 <sup>2</sup>	973.40 <sup>3</sup>
66. Sulfate (as SO <sub>4</sub> <sup>2-</sup> ), mg/L:	Automated colorimetric	375.2, Rev. 2.0 (1993)	9035					
	Gravimetric			4500-SO <sub>4</sub> <sup>2-</sup> C or D [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]				925.54 <sup>3</sup>
	Turbidimetric					D516-90, 02	426C <sup>39</sup>	
	Ion chromatography, or	300.0, Rev. 2.1 (1993) and 300.1, Rev. 1.0 (1997)	9056	4110 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	4110 B-00	D4327-97, 03		993.30 <sup>3</sup>
	CIE/UV					D6508-00 (05)		D6508, Rev 2 <sup>64</sup>
67. Sulfide (as S), mg/L:	Titrimetric (iodine)			4500-S <sup>2-</sup> E [18 <sup>th</sup> ], 4500-S <sup>2-</sup> F [19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-S <sup>2-</sup> F-00		I-3840-85 <sup>2</sup>	
	Colorimetric (methylene blue), or			4500-S <sup>2-</sup> D [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-S <sup>2-</sup> D-00			
	Ion selective electrode			4500-S <sup>2-</sup> G [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-S <sup>2-</sup> G-00	D4658-03		
68. Sulfite (as SO <sub>3</sub> ), mg/L:	Titrimetric (iodine-iodate)			4500-SO <sub>3</sub> <sup>-</sup> B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	4500-SO <sub>3</sub> <sup>-</sup> B-00			
69. Surfactants, mg/L:	Colorimetric (methylene blue)			5540 C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	5540 C-00	D2330-88, 02		
70. Temperature, °C:	Thermometric			2550 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	2550 B-00			Note 41

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
71. Thallium- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99			
	AA graphite furnace (GFAA)	279.2 (Issued 1978) <sup>1</sup> , 200.9, Rev. 2.2 (1994) <sup>13</sup>	7010					
	Inductively coupled plasma-atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
72. Tin- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B-99		I-3850-78 <sup>16</sup>	
	AA graphite furnace (GFAA)	200.9, Rev. 2.2 (1994) <sup>13</sup>	7010	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3113 B-99			
	Inductively coupled plasma-atomic emission spectrometry (ICP), or	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C					
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
73. Titanium- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)			3111 D [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 D-99			
	AA graphite furnace (GFAA)	283.2	7010					
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99			
	Inductively coupled plasma-mass spectrometry (ICP-MS), or	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A					
	Direct current plasma (DCP)							Note 43

Parameter, Units	Analytical Technology <sup>68</sup>	EPA <sup>44, 62</sup>	SW-846 <sup>4, 5</sup>	Standard Methods [Editions] <sup>6</sup>	Standard Methods-Online <sup>7</sup>	ASTM <sup>8</sup>	USGS	Other
74. Turbidity <sup>63</sup> , NTU:	Nephelometric	180.1		2130 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	2130 B-01	D1889-94, 00	I-3860-85 <sup>2</sup>	
75. Vanadium- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 D [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 D-99			
	AA graphite furnace (GFAA)		7010			D3373-93, 03		
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP), or					D4190-94, 99, 03		Note 43
	Colorimetric (Gallic acid)			3500-V D [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-V B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-V B-97			
76. Zinc- Total <sup>9</sup> , mg/L:	Digestion <sup>9, 11, 45</sup> followed by:							
	AA direct aspiration (FLAA)		7000B	3111 B or C [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ]	3111 B or C-99	D1691-95, 02 (A or B)	I-3900-85 <sup>2</sup>	974.27 <sup>3</sup> , p 37 <sup>17</sup>
	AA graphite furnace (GFAA)	289.2 (Issued 1978) <sup>1</sup>	7010					
	Inductively coupled plasma-atomic emission spectrometry (ICP)	200.7, Rev. 4.4 (1994) <sup>13</sup>	6010B, 6010C	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3120 B-99		I-4471-97 <sup>60</sup>	
	Inductively coupled plasma-mass spectrometry (ICP-MS)	200.8, Rev. 5.4 (1994) <sup>13</sup>	6020A			D5673-03, 05		993.14 <sup>3</sup>
	Direct current plasma (DCP)					D4190-94, 99, 03		Note 43
	Colorimetric (Dithizone), or			3500-Zn E [18 <sup>th</sup> , 19 <sup>th</sup> ]				
	Colorimetric (Zincon)			3500-Zn F [18 <sup>th</sup> , 19 <sup>th</sup> ], 3500-Zn B [20 <sup>th</sup> , 21 <sup>st</sup> ]	3500-Zn B-97			Note 42

<sup>1</sup> "Methods for Chemical Analysis of Water and Wastes," Environmental Protection Agency, Environmental Monitoring Systems Laboratory-Cincinnati (EMSL-CI), EPA-600/4-79-020 (NTIS PB 84-128677), Revised March 1983 and 1979 where applicable. Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

<sup>2</sup> Fishman, 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, Denver, CO, Revised 1989, unless otherwise stated.

<sup>3</sup> "Official Methods of Analysis of the Association of Official Analytical Chemists," Methods Manual, Sixteenth Edition, 4th Revision, 1998.

- <sup>4</sup> "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, EPA, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, September 1986 (Third edition), including July 1992 (Update I), September 1994 (Update II), August 1993 (Update IIA), January 1995 (Update IIB), December 1996 (Update III), April 1998 (Update IIIA), November 2004 (Update IIIB), February 2007 (Update IV) updates. Available from: U.S. Government Printing Office (GPO), Superintendent of Documents, Washington, DC 20402, (202) 512-1800 (Publication Number: 955-001-00000-1). Also, available on-line at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.
- <sup>5</sup> "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, Update IV includes methods 7000B, Flame Atomic Absorption Spectrophotometry and 7010, Graphite Furnace Atomic Absorption Spectrophotometry, general method descriptions.
- <sup>6</sup> "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 21<sup>st</sup> Edition (2005), 20th Edition (1998), 19th Edition (1995), and 18th Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.
- <sup>7</sup> "Standard Methods for the Examination of Water and Wastewater On-Line", Joint Editorial Board, American Public Health Association, American Water Works Association, Water Environment Federation, 2006. Subscription service available at: <http://www.standardmethods.org>.
- <sup>8</sup> "Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1994, 1996, 1999, 2007. Available from: the American Society for Testing and Materials, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959. Also available online at [www.astm.org](http://www.astm.org).
- <sup>9</sup> For the determination of total metals (which are the equivalent to total recoverable metals) the sample is not filtered before processing. A digestion procedure is required to solubilize analytes in suspended material and to break down organic-metal complexes (to convert the analyte to a detectable form for colorimetric analysis). For non-platform graphite furnace atomic absorption determinations, a digestion using nitric acid (as specified in Section 4.1.3 of Methods for the Chemical Analysis of Water and Wastes) 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. The approved total recoverable digestion is described as Method 200.2 in Supplement I of "Methods for the Determination of Metals in Environmental Samples" EPA/600R-94/111, May, 1994, and is reproduced in EPA Methods 200.7, 200.8, and 200.9 from the same Supplement. 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, STGFAA, ICP-AES, and ICP-MS) use EPA Method 200.2 or an approved alternate procedure (e.g., CEM microwave digestion, which may be used with certain analytes as indicated in Table B); the total recoverable digestion procedures in EPA Methods 200.7, 200.8, and 200.9 may be used for those respective methods. Regardless of the digestion procedure, the results of the analysis after digestion procedure are reported as "total" metals.
- <sup>10</sup> Use the digestion given in the method.
- <sup>11</sup> "Test Methods for Evaluating Solid Waste", SW-846 method 3015A. United States EPA SW-846, 3rd Edition and updates. Footnote 4 lists the complete reference.
- <sup>12</sup> Copper sulfate may be used in place of mercuric sulfate.
- <sup>13</sup> "Methods for the Determination of Metals in Environmental Samples", EPA-600/4-91-010, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, June 1991. Available from: the National Technical Information Service (NTIS), 5258 Port Royal Road, Springfield, Virginia 22161.
- <sup>14</sup> Manual distillation is not required if comparability data on representative effluent samples are on file to show that this preliminary distillation step is not necessary: however, manual distillation will be required to resolve any controversies.
- <sup>15</sup> Ammonia, Automated Electrode Method, Industrial Method Number 379-75 WE, February 19, 1976, Bran & Luebbe (Technicon) Auto Analyzer II, Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.
- <sup>16</sup> The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", USGS TWRI, Book 5, Chapter A1 (1979).
- <sup>17</sup> American National Standard on Photographic Processing Effluents, April 2, 1975. Available from ANSI, 25 West 43rd St., New York, NY 10036.
- <sup>18</sup> "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).
- <sup>19</sup> The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.
- <sup>20</sup> Carbonaceous biochemical oxygen demand (CBOD<sub>5</sub>) must not be confused with the traditional BOD<sub>5</sub> test method which measures "total BOD." The addition of the nitrification inhibitor is not a procedural option, but must be included to report the CBOD<sub>5</sub> parameter. A discharger whose permit requires reporting the traditional BOD<sub>5</sub> may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states CBOD<sub>5</sub> is required can the permittee report data using a nitrification inhibitor.
- <sup>21</sup> OIC Chemical Oxygen Demand Method, Oceanography International Corporation, 1978, 512 West Loop, P.O. Box 2980, College Station, TX 77840.
- <sup>22</sup> Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Company, P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- <sup>23</sup> The back titration method will be used to resolve controversy.
- <sup>24</sup> Orion Research Instruction Manual, Residual Chlorine Electrode Model 97-70, 1977, Thermo Scientific, 81 Wyman Street, Waltham, MA 02454. The calibration graph for the Orion residual chlorine method must be derived using a reagent blank and three standard solutions, containing 0.2, 1.0, and 5.0 mL 0.00281 N potassium iodate/100 mL solution, respectively.
- <sup>25</sup> The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1976. Available on interlibrary loan.
- <sup>26</sup> "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.
- <sup>27</sup> Copper, Bicinchoninate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Company, P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- <sup>28</sup> When using a method with block digestion, this treatment is not required.

- <sup>29</sup> Hydrogen ion (pH) Automated Electrode Method, Industrial Method Number 378–75WA, October 1976, Bran & Luebbe (Technicon) Autoanalyzer II. Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.
- <sup>30</sup> Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Company, P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- <sup>31</sup> Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, Hach Chemical Company, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- <sup>32</sup> 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. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.
- <sup>33</sup> Nitrogen, Nitrite, Method 8507, Hach Company, P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- <sup>34</sup> Nitrate + nitrite determinations by ion chromatography must be analyzed within 48 hours.
- <sup>35</sup> Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.
- <sup>36</sup> 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 spectrometric procedure. Available on interlibrary loan.
- <sup>37</sup> 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. Available in most public libraries. Back volumes of the Journal of Chromatography are available from: Elsevier/North–Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164.
- <sup>38</sup> Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.
- <sup>39</sup> The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 15th Edition. Available on interlibrary loan.
- <sup>40</sup> For samples known or suspected to contain high levels of silver (e.g., in excess of 4 mg/L), cyanogen iodide should be used to keep the silver in solution for analysis. Prepare a cyanogen iodide solution by adding 4.0 mL of concentrated NH<sub>4</sub>OH, 6.5 g of KCN, and 5.0 mL of a 1.0 N solution of I<sub>2</sub> to 50 mL of reagent water in a volumetric flask and dilute to 100.0 mL. After digestion of the sample, adjust the pH of the digestate to >7 to prevent the formation of HCN under acidic conditions. Add 1 mL of the cyanogen iodide solution to the sample digestate and adjust the volume to 100 mL with reagent water (NOT acid). If cyanogen iodide is added to sample digestates, then silver standards must be prepared that contain cyanogen iodide as well. Prepare working standards by diluting a small volume of a silver stock solution with water and adjusting the pH>7 with NH<sub>4</sub>OH. Add 1 mL of the cyanogen iodide solution and let stand 1 hour. Transfer to a 100-mL volumetric flask and dilute to volume with water.
- <sup>41</sup> Stevens, H.H., Ficke, J. F., and Smoot, G. F., "Water Temperature—Influential Factors, Field Measurement and Data Presentation," Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1, 1975. Available from: U.S. Geological Survey, 604 S. Pickett Street, Alexandria, VA 22304.
- <sup>42</sup> Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, Hach Company, Loveland, CO 80537. Available on-line at <http://www.hach.com>.
- <sup>43</sup> "Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029," 1986—Revised 1991. Available from: Thermo Scientific, 81 Wyman Street, Waltham, MA 02454.
- <sup>44</sup> Precision and recovery statements for the atomic absorption direct aspiration and graphite furnace methods, and for the spectrophotometric SDDC method for arsenic are provided in Appendix D of this part titled, "Precision and Recovery Statements for Methods for Measuring Metals."
- <sup>45</sup> Microwave-assisted digestion may be employed for this metal, when analyzed by this methodology. "Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals", April 16, 1992, CEM Corporation, P.O. Box 200, Matthews, NC 28106–0200.
- <sup>46</sup> When determining boron and silica, only plastic, PTFE, or quartz laboratory ware may be used from start until completion of analysis.
- <sup>47</sup> Only use n-hexane extraction solvent when determining Oil and Grease parameters—Hexane Extractable Material (HEM), or Silica Gel Treated HEM (analogous to EPA Method 1664A). Use of other extraction solvents (e.g., those in the 18th and 19th editions) is prohibited.
- <sup>48</sup> Nitrogen, Total Kjeldahl, Method PAI-DK01 (Block Digestion, Steam Distillation, Titrimetric Detection), revised 12/22/94, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.
- <sup>49</sup> Nitrogen, Total Kjeldahl, Method PAI–DK03 (Block Digestion, Automated FIA Gas Diffusion), revised 12/22/94, OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.
- <sup>50</sup> Method 1664, Revision A "n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry" EPA–821–R–98–002, February 1999. Available at NTIS, PB–121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, VA 22161.
- <sup>51</sup> The full text of Method 1650 is given in Appendix A, "Methods 1650 and 1653", of 40 CFR Part 430. Available from: The Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.
- <sup>52</sup> US EPA. 2001. Method 1631, Revision E, "Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry" September 2002, Office of Water, U.S. Environmental Protection Agency (EPA–821–R–02–024). The application of clean techniques described in EPA's draft Method 1669: *Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (EPA–821–R–96–011) are recommended to preclude contamination at low-level, trace metal determinations. Available at NTIS, PB–121949, U.S. Department of Commerce, 5285 Port Royal, Springfield, Virginia 22161.
- <sup>53</sup> Quality control requirements for low level mercury are found in s. NR 106.145 (9) and (10), Wis. Adm. Code. Low-level mercury methods are performance based so some method modifications are allowable, provided quality control requirements are met. If an atomic absorption detector is substituted for atomic fluorescence detector, the appropriate method citation is 245.1 (manual) or 245.2 (automated). If method 1631E is modified to eliminate the purge and trap step, the appropriate method citation is 245.7.
- <sup>54</sup> Available Cyanide, Method OIA–1677, "Available Cyanide by Flow Injection, Ligand Exchange, and Amperometry," OI Analytical/ALPKEM, P.O. Box 9010, College Station, TX 77842.
- <sup>55</sup> "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Ammonia Plus Organic Nitrogen by a Kjeldahl Digestion Method," Open File Report (OFR) 00–170.
- <sup>56</sup> "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Chromium in Water by Graphite Furnace Atomic Absorption Spectrophotometry," Open File Report (OFR) 93–449.

- <sup>57</sup> "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Molybdenum by Graphite Furnace Atomic Absorption Spectrophotometry," Open File Report (OFR) 97–198.
- <sup>58</sup> "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Total Phosphorus by Kjeldahl Digestion Method and an Automated Colorimetric Finish That Includes Dialysis," Open File Report (OFR) 92–146.
- <sup>59</sup> "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Arsenic and Selenium in Water and Sediment by Graphite Furnace-Atomic Absorption Spectrometry," Open File Report (OFR) 98–639.
- <sup>60</sup> "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Elements in Whole-water Digests Using Inductively Coupled Plasma-Optical Emission Spectrometry and Inductively Coupled Plasma-Mass Spectrometry," Open File Report (OFR) 98-165.
- <sup>61</sup> "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediment," Open File Report (OFR) 93–125.
- <sup>62</sup> All EPA methods, excluding EPA Method 300.1, are published in "Methods for the Determination of Metals in Environmental Samples," Supplement I, National Exposure Risk Laboratory-Cincinnati (NERL–CI), EPA/600/R–94/111, May 1994; and "Methods for the Determination of Inorganic Substances in Environmental Samples," NERL–CI, EPA/600/R–93/100, August, 1993. EPA Method 300.1 is available from <http://www.epa.gov/safewater/methods/pdfs/met300.pdf>.
- <sup>63</sup> Styrene divinyl benzene beads (e.g. AMCO–AEPA–1 or equivalent) and stabilized formazin (e.g., Hach StablCal™ or equivalent) are acceptable substitutes for formazin.
- <sup>64</sup> Method D6508, Rev. 2, "Test Method for Determination of Dissolved Inorganic Anions in Aqueous Matrices Using Capillary Ion Electrophoresis and Chromate Electrolyte," available from Waters Corp, 34 Maple St., Milford, MA, 01757, Telephone: 508/482–2131, Fax: 508/482–3625.
- <sup>65</sup> Kelada-01, "Kelada Automated Test Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate," EPA 821–B–01–009, Revision 1.2, August 2001, National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 [Order Number PB 2001–108275]. The toll free telephone number is: 800–553–6847. Note: A 450–W UV lamp may be used in this method instead of the 550–W lamp specified if it provides performance within the quality control (QC) acceptance criteria of the method in a given instrument. Similarly, modified flow cell configurations and flow conditions may be used in the method, provided that the QC acceptance criteria are met.
- <sup>66</sup> QuikChem Method 10–204–00–1–X, "Digestion and Distillation of Total Cyanide in Drinking and Wastewaters using MICRO DIST and Determination of Cyanide by Flow Injection Analysis" is available from Hach Company, P.O. Box 389, Loveland, CO 80537.
- <sup>67</sup> When using sulfide removal test procedures described in Method 335.4, reconstitute particulate that is filtered with the sample prior to distillation.
- <sup>68</sup> Unless otherwise stated, if the language of this table specifies a sample digestion and/or distillation "followed by" analysis with a method, approved digestion and/or distillation are required prior to analysis.
- <sup>69</sup> Method 245.7, Rev. 2.0, "Mercury in Water by Cold Vapor Atomic Fluorescence Spectrometry," February 2005, EPA–821–R–05–001, available from the U.S. EPA Sample Control Center (operated by CSC), 6101 Stevenson Avenue, Alexandria, VA 22304.
- <sup>70</sup> The use of EDTA may decrease method sensitivity in some samples. Analysts may omit EDTA provided that all method specified quality control acceptance criteria are met.
- <sup>71</sup> Samples analyzed for available cyanide using Methods OIA–1677 or D6888–04 that contain particulate matter may be filtered only after the ligand exchange reagents have been added to the samples, because the ligand exchange process converts complexes containing available cyanide to free cyanide, which is not removed by filtration. Analysts are further cautioned to limit the time between the addition of the ligand exchange reagents and sample analysis to no more than 30 minutes to preclude settling of materials in samples.
- <sup>72</sup> Oxygen, Dissolved, Luminescence, Hach Method 10360- Luminescence Measurement of Dissolved Oxygen (LDO®) in Water and Wastewater, Revision 1.1, January 2006, Hach Chemical Company, Loveland, CO 80537. Available from: Hach Company, P.O. Box 389, Loveland, CO 80537. Available on-line at <http://www.hach.com>.

Section 5: NR 219.04 Table C is repealed and recreated to read:

Table C  
List of Approved Analytical Methods for Non-Pesticide Organic Compounds

Parameter <sup>1</sup>	Analytical Technology	EPA <sup>2, 11</sup>	SW-846 <sup>3</sup>	Standard Methods [Edition(s)] <sup>4</sup>	Standard Methods Online <sup>5</sup>	Other
1. Acenaphthene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B–00	
	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]		
2. Acenaphthylene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B–00	
	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
3. Acrolein	GC	603				
	GC/MS	624 <sup>7</sup> , 1624B				

Parameter <sup>1</sup>	Analytical Technology	EPA <sup>2, 11</sup>	SW-846 <sup>3</sup>	Standard Methods [Edition(s)] <sup>4</sup>	Standard Methods Online <sup>5</sup>	Other
	HPLC		8316			
4. Acrylonitrile	GC	603	8031			
	GC/MS	624 <sup>7</sup> , 1624B				
	HPLC		8315A, 8316			
5. Anthracene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
6. Benzene	GC	602	8021B	6220 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
7. Benzidine	GC		8121			Note 6, p.1
	GC/MS	625 <sup>8</sup> , 1625B	8270C, 8270D			
	HPLC	605				
8. Benzo(a)anthracene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
9. Benzo(a)pyrene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
10. Benzo(b)fluoranthene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
11. Benzo(g,h,i) perylene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
12. Benzo(k) fluoranthene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
13. Benzyl chloride	GC		8121			Note 6, p. 130, Note 10, p. S102
	GC/MS		8260B			
14. Benzyl butyl phthalate	GC	606	8061A			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
15. Bis(2-chloroethoxy) methane	GC	611	8111			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
16. Bis(2-chloroethyl) ether	GC	611	8111			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
17. Bis(2-ethylhexyl) phthalate	GC	606				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
18. Bromodichloromethane	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
19. Bromoform	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
20. Bromomethane	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
21. 4-Bromophenyl phenyl ether	GC	611	8111			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
22. Carbon tetrachloride	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	

Parameter <sup>1</sup>	Analytical Technology	EPA <sup>2, 11</sup>	SW-846 <sup>3</sup>	Standard Methods [Edition(s)] <sup>4</sup>	Standard Methods Online <sup>5</sup>	Other
23. 4-Chloro-3-methylphenol	GC	604	8041A	6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
24. Chlorobenzene	GC	601, 602	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	Note 6, p.130
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
25. Chloroethane	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
26. 2-Chloroethylvinyl ether	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
27. Chloroform	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	Note 6, p.130
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
28. Chloromethane	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
29. 2-Chloronaphthalene	GC	612	8121			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
30. 2-Chlorophenol	GC	604	8041A	6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
31. 4-Chlorophenyl phenyl ether	GC	611	8111			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
32. Chrysene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
33. Dibenzo(a,h)anthracene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
34. Dibromochloromethane	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
35. 1,2-Dichlorobenzene	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
	GC	601, 602		6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	Note 13, p.27
36. 1,3-Dichlorobenzene	GC/MS	624, 1625B	8260B, 8270C, 8270D	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
	GC	601, 602		6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	Note 13, p.27
37. 1,4-Dichlorobenzene	GC/MS	624, 1625B	8260B, 8270C, 8270D	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
	GC	601, 602		6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	Note 13, p.27
38. 3,3-Dichlorobenzidine	GC/MS	624, 1625B	8260B, 8270C, 8270D	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
	GC		8121			
	HPLC	605				
39. Dichlorodifluoromethane	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
40. 1,1-Dichloroethane	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
41. 1,2-Dichloroethane	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
42. 1,1-Dichloroethene	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
43. <i>trans</i> -1,2-Dichloroethene	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
44. 2,4-Dichlorophenol	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
	GC	604	8041A	6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	

Parameter <sup>1</sup>	Analytical Technology	EPA <sup>2,11</sup>	SW-846 <sup>3</sup>	Standard Methods [Edition(s)] <sup>4</sup>	Standard Methods Online <sup>5</sup>	Other
45. 1,2-Dichloropropane	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
46. <i>cis</i> -1,3-Dichloropropene	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
47. <i>trans</i> -1,3-Dichloropropene	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
48. Diethyl phthalate	GC	606	8061A			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
49. 2,4-Dimethylphenol	GC	604	8041A	6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
50. Dimethyl phthalate	GC	606	8061A			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
51. Di- <i>n</i> -butyl phthalate	GC	606	8061A			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
52. Di- <i>n</i> -octyl phthalate	GC	606	8061A			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
53. 2,3-Dinitrophenol	GC	604	8041A	6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
54. 2,4-Dinitrophenol	GC	604	8041A	6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
55. 2,6-Dinitrophenol	GC	604	8041A	6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
56. 2,3-Dinitrotoluene	GC	609	8091			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
57. 2,4-Dinitrotoluene	GC	609	8091			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
58. 2,6-Dinitrotoluene	GC	609	8091			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
59. Epichlorohydrin	GC		8121			Note 6, p.130, Note 10, p. S102
	GC/MS		8260B			
60. Ethylbenzene	GC	602	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
61. Fluoranthene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
62. Fluorene	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
63. 1,2,3,4,6,7,8-Heptachlorodibenzofuran	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
	HRGC/LRMS		8280B			
64. 1,2,3,4,7,8,9-Heptachlorodibenzofuran	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
	HRGC/LRMS		8280B			
65. 1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
	HRGC/LRMS		8280B			

Parameter <sup>1</sup>	Analytical Technology	EPA <sup>2, 11</sup>	SW-846 <sup>3</sup>	Standard Methods [Edition(s)] <sup>4</sup>	Standard Methods Online <sup>5</sup>	Other
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
66. Hexachlorobenzene	GC	612	8081A, 8081B, 8121			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
67. Hexachlorobutadiene	GC	612	8121			Note 13, p.27
	GC/MS	625, 1625B	8260B, 8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
68. Hexachlorocyclopentadiene	GC	612	8081A, 8081B, 8121			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
69. 1,2,3,4,7,8-Hexachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
70. 1,2,3,6,7,8-Hexachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
71. 1,2,3,7,8,9-Hexachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
72. 2,3,4,6,7,8-Hexachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
73. 1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
74. 1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
75. 1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
76. Hexachloroethane	GC	612	8121			Note 13, p.27
	GC/MS	625, 1625B	8260B, 8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
77. Indeno(1,2,3-cd) pyrene	GC	610				
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	Note 13, p.27
	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
78. Isophorone	GC	609	8091			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
79. Methylene chloride	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	Note 6, p.130
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
80. 2-Methyl-4,6- dinitrophenol	GC	604	8041A	6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
81. Naphthalene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
82. Nitrobenzene	GC	609	8091			Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
83. 2-Nitrophenol	GC	604	8041A	6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
84. 4-Nitrophenol	GC	604	8041A	6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	Note 13, p.27

Parameter <sup>1</sup>	Analytical Technology	EPA <sup>2, 11</sup>	SW-846 <sup>3</sup>	Standard Methods [Edition(s)] <sup>4</sup>	Standard Methods Online <sup>5</sup>	Other
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
85. N-Nitrosodimethylamine	GC	607				Note 13, p.27
	GC/MS	6255, 1625B		6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
86. N-Nitrosodi-n-propylamine	GC	607				Note 13, p.27
	GC/MS	6255, 1625B		6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
87. N-Nitrosodiphenylamine	GC	607				Note 13, p.27
	GC/MS	6255, 1625B		6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
88. Octachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
89. Octachlorodibenzo-p-dioxin	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
90. 2,2'-Oxybis(2-chloropropane)	GC	611	8111			
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
91. PCB-1016 (Aroclor or congeners) <sup>16, 17</sup>	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HRGC/HRMS	1668A <sup>18</sup>				
92. PCB-1221 (Aroclor or congeners) <sup>16, 17</sup>	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HRGC/HRMS	1668A <sup>18</sup>				
93. PCB-1232 (Aroclor or congeners) <sup>16, 17</sup>	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HRGC/HRMS	1668A <sup>18</sup>				
94. PCB-1242 (Aroclor or congeners) <sup>16, 17</sup>	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HRGC/HRMS	1668A <sup>18</sup>				
95. PCB-1248 (Aroclor or congeners) <sup>16, 17</sup>	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HRGC/HRMS	1668A <sup>18</sup>				
96. PCB-1254 (Aroclor or congeners) <sup>16, 17</sup>	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HRGC/HRMS	1668A <sup>18</sup>				
97. PCB-1260 (Aroclor or congeners) <sup>16, 17</sup>	GC	608	8082			Note 6, p.43, Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HRGC/HRMS	1668A <sup>18</sup>				
98. 1,2,3,7,8-Pentachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
99. 2,3,4,7,8-Pentachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
100. 1,2,3,7,8-Pentachlorodibenzo-p-dioxin	HRGC/LRMS		8280B			

Parameter <sup>1</sup>	Analytical Technology	EPA <sup>2, 11</sup>	SW-846 <sup>3</sup>	Standard Methods [Edition(s)] <sup>4</sup>	Standard Methods Online <sup>5</sup>	Other
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
101. Pentachlorophenol	GC	604	8041A	6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
102. Phenanthrene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]		
103. Phenol	GC	604	8041A	6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
104. Pyrene	GC	610				Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
	HPLC	610	8310	6440 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		
105. 2,3,7,8-Tetrachlorodibenzofuran	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
106. 2,3,7,8-Tetrachlorodibenzo-p-dioxin	GC/MS	625 <sup>9</sup>				
	HRGC/LRMS		8280B			
	HRGC/HRMS	1613B <sup>14</sup>	8290A <sup>14</sup>			
107. 1,1,2,2-Tetrachloroethane	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	Note 6, p.130
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
108. Tetrachlorocatechol	GC			6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	
	GC/MS	1653 <sup>15</sup>		6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
109. Tetrachloroethene	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	Note 6, p.130
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
110. Tetrachloroguaicol	GC			6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	
	GC/MS	1653 <sup>15</sup>		6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
111. 2,3,4,6-Tetrachlorophenol	GC			6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	
	GC/MS	1653 <sup>15</sup>		6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
112. Toluene	GC	602	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
113. 1,2,4-Trichlorobenzene	GC	612	8021B			Note 6, p.130, Note 13, p.27
	GC/MS	625, 1624B	8260B, 8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
114. 3,4,5-Trichlorocatechol	GC			6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	
	GC/MS	1653 <sup>15</sup>		6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
115. 3,4,6-Trichlorocatechol	GC			6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	
	GC/MS	1653 <sup>15</sup>		6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
116. 1,1,1-Trichloroethane	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
117. 1,1,2-Trichloroethane	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
118. Trichloroethene	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
119. Trichlorofluoromethane	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	
120. 3,4,5-Trichloroguaicol	GC			6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	
	GC/MS	1653 <sup>15</sup>		6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
121. 3,4,6-Trichloroguaicol	GC			6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	
	GC/MS	1653 <sup>15</sup>		6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	

Parameter <sup>1</sup>	Analytical Technology	EPA <sup>2, 11</sup>	SW-846 <sup>3</sup>	Standard Methods [Edition(s)] <sup>4</sup>	Standard Methods Online <sup>5</sup>	Other
122. 4,5,6-Trichloroguaiacol	GC			6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	
	GC/MS	1653 <sup>15</sup>		6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
123. 2,4,5-Trichlorophenol	GC			6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	
	GC/MS	1653 <sup>15</sup>		6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
124. 2,4,6-Trichlorophenol	GC	604	8041A	6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	Note 13, p.27
	GC/MS	625, 1625B	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
125. Trichlorosyringol	GC			6420 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6420 B-00	
	GC/MS	1653 <sup>15</sup>		6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00	
126. Vinyl chloride	GC	601	8021B	6230 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 C [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 C-97	
	GC/MS	624, 1624B	8260B	6210 B [18 <sup>th</sup> , 19 <sup>th</sup> ], 6200 B [20 <sup>th</sup> , 21 <sup>st</sup> ]	6200 B-97	

<sup>1</sup>. All parameters are expressed in micrograms per liter (µg/L) except for Method 1613B in which the parameters are expressed in picograms per liter (pg/L).

<sup>2</sup>. The full text of Methods 601–613, 624, 625, 1624B, and 1625B, are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," of this Part 136. The full text of Method 1613B is incorporated by reference into this Part 136 and is available from the National Technical Information Services as stock number PB95–104774. 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.

<sup>3</sup>. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, EPA, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, September 1986 (third edition), including July 1992 (update I), September 1994 (update II), August 1993 (update IIA), January 1995 (update IIB), December 1996 (update III), April 1998 (update IIIA), November 2004 (update IIIB), February 2007 (update IV) updates. Available from: U.S. Government Printing Office (GPO), Superintendent of Documents, Washington, DC 20402, (202) 512–1800 (Publication Number: 955–001–00000–1). Also, available on-line at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.

<sup>4</sup>. "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 20<sup>th</sup> Edition (1998), 19<sup>th</sup> Edition (1995), and 18<sup>th</sup> Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

<sup>5</sup>. "Standard Methods for the Examination of Water and Wastewater Online", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 2006. Subscription service available at <http://www.standardmethods.org>.

<sup>6</sup>. "Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September, 1978.

<sup>7</sup>. Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 603 or Method 1624B.

<sup>8</sup>. Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625B, are preferred methods for these compounds.

<sup>9</sup>. 5a 625, screening only.

<sup>10</sup>. "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).

<sup>11</sup>. Each analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601–603, 624, 625, 1624B, and 1625B (See Appendix A of this Part 136) in accordance with procedures each in Section 8.2 of each of these methods. Additionally, each laboratory, on an ongoing basis must spike and analyze 10% (5% for methods 624 and 625 and 100% for methods 1624B and 1625B) 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. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

<sup>12</sup>. "Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk" 3M Corporation Revised 10/28/94.

<sup>13</sup>. USGS Method 0–3116–87 from "Methods of Analysis by U.S. Geological Survey National Water Quality Laboratory—Determination of Inorganic and Organic Constituents in Water and Fluvial Sediments," U.S. Geological Survey, Open File Report 93–125.

<sup>14</sup>. Analysts may use Fluid Management Systems, Inc. PowerPrep system in place of manual cleanup provided that the analysis meet the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities.

<sup>15</sup>. The full text of Method 1653 is given in Appendix A, "Methods 1650 and 1653", of 40 CFR Part 430. Available from: Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402. Also available on-line at <http://www.gpoaccess.gov/>.

<sup>16</sup>. EPA Method 1668A may be used to test for all PCB congeners. If this method is employed, all PCB congeners shall be delineated. Non-detects shall be treated as zero. The values that are between the limit of detection and the limit of quantitation shall be used when calculating the total value of all congeners. All results shall be added together and the total PCB concentration reported. It is recognized a number of congeners will co-elute with others, so there will not be 209 results to sum.

<sup>17</sup>. EPA Method 8082A shall be used for PCB-Aroclor analysis and may be used for congener specific analysis as well. If congener specific analysis is performed using Method 8082A, the list of congeners tested shall include at least congener numbers 5, 18, 31, 44, 52, 66, 87, 101, 110, 138, 141, 151, 153, 170, 180, 183, 187, and 206 plus any other additional congeners which might be reasonably expected to occur in the particular

sample. If Aroclor analysis is performed using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference. If congener specific analysis is done using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference. If the aforementioned limits of detection cannot be achieved after using the appropriate clean up techniques, a reporting limit that is achievable for the Aroclors or each congener for sample shall be determined. This report limit should be reported and qualified indicating the presence of an interference. The laboratory conducting the analysis shall perform as many the following methods as necessary to remove interference:

- 3620C – Florisil
- 3640A – Gel Permeation
- 3630C – Silica Gel
- 3611B – Alumina
- 3660B – Sulfur Clean Up
- 3665A – Sulfuric Acid Clean Up.

<sup>18</sup> “Method 1668A, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS”, EPA-821-R-00-002, Environmental Protection Agency, Office of Water, Washington, D.C., December 1999. Available from: the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

Section 6: NR 219.04 Table D is repealed and recreated to read:

Table D  
List of Approved Analytical Methods for Pesticides<sup>1</sup> in Wastewater

Parameter	Analytical Technology	EPA <sup>2, 11</sup>	SW-846 <sup>3</sup>	Standard Methods <sup>4</sup>	Standard Methods Online <sup>5</sup>	ASTM <sup>6</sup>	Other
1. Aldrin	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p.7; Note 8, p.27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
2. Ametryn	GC						Note 7, p. 83; Note 10, p. S68
3. Aminocarb	HPLC						Note 7, p. 94; Note 10, p. S16; Note 14
	LC/MS		8321A				
4. Atraton	GC						Note 7, p. 83; Note 10, p. S68
5. Atrazine	GC						Note 7, p. 83; Note 10, p. S68; Note 13
6. Azinphos methyl	GC						Note 7, p. 25; Note 10, p. S51
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
7. Barban	HPLC						Note 7, p.104; Note 10, p. S64; Note 14
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
	LC/MS		8321A				
8. α-BHC	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7; Note 12
	GC/MS	625 <sup>5</sup>	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
9. β-BHC	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 12
	GC/MS	625 <sup>5</sup>	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
10. δ-BHC	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 12
	GC/MS	625 <sup>5</sup>	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
11. γ-BHC (Lindane)	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7; Note 8, p 27; Note 12
	GC/MS	625 <sup>5</sup>	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
12. Captan	GC			6630 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7
13. Carbaryl	HPLC		8318				Note 7, p. 94; Note 10, p. S60; Note 14
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
	LC/MS		8321A, 8325				
14. Carbophenthion	GC		8141B				Note 8, p. 27; Note 10, p. S73
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
15. Chlordane	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12

Parameter	Analytical Technology	EPA <sup>2, 11</sup>	SW-846 <sup>3</sup>	Standard Methods <sup>4</sup>	Standard Methods Online <sup>5</sup>	ASTM <sup>6</sup>	Other
	GC/MS	625 <sup>5</sup>	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
16. Chloropropham	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14
	LC/MS		8321A				
17. 2,4-D	GC			6640 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 7, p. 115; Note 8, p. 40
	LC/MS		8321A				
18. 4,4'-DDD	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
19. 4,4'-DDE	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
20. 4,4'-DDT	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
21. Demeton-O	GC		8141A, 8141B				Note 7, p. 25; Note 10, p. S51
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
22. Demeton-S	GC		8141A, 8141B				Note 7, p. 25; Note 10, p. S51
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
23. Diazinon	GC		8141A, 8141B				Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
24. Dicamba	GC						Note 7, p. 115
	LC/MS		8321A				
25. Dichlofenthion	GC		8141A, 8141B				Note 8, p. 27; Note 10, p. S73
26. Dieldrin	GC			6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 7, p. 7
27. Dicofol	GC					D5812- 96 (02)	
28. Dieldrin	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
29. Dioxathion	GC		8141A, 8141B				Note 8, p. 27; Note 10, p. S73
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
30. Disulfoton	GC		8141A, 8141B				Note 7, p. 25; Note 10, p. S51
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
31. Diuron	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14
	LC/MS		8321A, 8325				
32. Endosulfan I	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625 <sup>9</sup>	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
33. Endosulfan II	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
34. Endosulfan sulfate	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
35. Endrin	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625 <sup>9</sup>	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
36. Endrin Aldehyde	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
37. Ethion	GC						Note 8, p. 27; Note 10, p. S73
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
38. Fenuron	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14
	LC/MS		8321A				
39. Fenuron-TCA	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14
40. Heptachlor	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625 <sup>9</sup>	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
41. Heptachlor epoxide	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 10, p. S73;

Parameter	Analytical Technology	EPA <sup>2, 11</sup>	SW-846 <sup>3</sup>	Standard Methods <sup>4</sup>	Standard Methods Online <sup>5</sup>	ASTM <sup>6</sup>	Other
							Note 12
	GC/MS	625 <sup>9</sup>	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
42. Isodrin	GC		8081A, 8081B				Note 8, p. 27; Note 10, p. S73
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
43. Linuron	HPLC						Note 7, p.104; Note 10 p. S64; Note 14
	LC/MS		8321A, 8325				
44. Malathion	GC			6630 C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 7, p. 25; Note 8, p. 27; Note 10, p. S51
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
45. Methiocarb	HPLC		8318				Note 7, p. 94; Note 10, p. S60; Note 14
	LC/MS		8321A, 8325				
46. Methoxychlor			8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
47. Mexacarbate	HPLC						Note 7, p.94; Note 10, p. S60; Note 14
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
48. Mirex	GC		8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 7, p. 7; Note 8, p. 27
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
49. Monuron	HPLC						Note 7, p.104; Note 10, p. S64; Note 14
	LC/MS		8321A, 8325				
50. Monuron-TCA	HPLC						Note 7, p.104; Note 10, p. S64; Note 14
51. Neburon	HPLC						Note 7, p.104; Note 10, p. S64; Note 14
	LC/MS		8321A				
52. Parathion methyl	GC		8141A, 8141B	6630 C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 7, p. 25; Note 8, p. 27
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
	LC/MS		8321A				
53. Parathion ethyl	GC		8141A, 8141B	6630 C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 7, p. 25; Note 8, p. 27
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
54. PCNB	GC		8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 7, p. 7
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
55. Perthane	GC		8081A, 8081B			D5812- 96 (02)	Note 8, p. 27
56. Prometon	GC						Note 7, p. 83; Note 10, p. S68; Note 13
57. Prometryn	GC						Note 7, p. 83; Note 10, p. S68; Note 13
58. Propazine	GC						Note 7, p. 83; Note 10, p. S68; Note 13
59. Propham	HPLC						Note 7, p.104; Note 10, p. S64; Note 14
	LC/MS		8321A				
60. Propoxur	HPLC		8318				Note 7, p. 94; Note 10, p. S60; Note 14
	LC/MS		8321A				
61. Secbumeton	HPLC						Note 7, p. 83; Note 10, p. S68; Note 14
62. Siduron	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14
	LC/MS		8321A, 8325				
63. Simazine	GC		8141A, 8141B				Note 7, p. 83; Note 10, p. S68; Note 13
64. Strobane	GC		8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 7, p. 7
65. Sweb	HPLC						Note 7, p. 104; Note 10, p. S64; Note 14
66. 2,4,5-T	GC		8151A	6640 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 7, p. 115; Note 8, p. 40
	LC/MS		8321A				
67. 2,4,5-TP (Silvex)	GC		8151A	6640 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 7, p. 115; Note 8, p. 40
	LC/MS		8321A				
68. Terbutylazine	GC						Note 7, p. 83; Note 10, p. S68

Parameter	Analytical Technology	EPA <sup>2, 11</sup>	SW-846 <sup>3</sup>	Standard Methods <sup>4</sup>	Standard Methods Online <sup>5</sup>	ASTM <sup>6</sup>	Other
69. Toxaphene	GC	608	8081A, 8081B	6630 B and C [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]		D5812- 96 (02)	Note 7, p. 7; Note 8, p. 27; Note 12
	GC/MS	625	8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		
70. Trifluralin	GC		8081A, 8081B	6630 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> ]			Note 7, p. 7; Note 13
	GC/MS		8270C, 8270D	6410 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ]	6410 B-00		

<sup>1</sup>. Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table IC, where entries are listed by chemical name.

<sup>2</sup>. The full text of Methods 608 and 625 are given at Appendix A, "Test Procedures for Analysis of Organic Pollutants," 40 CFR, 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," 40 CFR, Part 136.

<sup>3</sup>. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, EPA, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, November, 1986 (third edition), including July 1992 (update I), September 1994 (update II), August 1993 (update IIA), January 1995 (update IIB), December 1996 (update III), April 1998 (update IIIA), November 2004 (update IIIB), February 2007 (update IV) updates. Available from: U.S. Government Printing Office (GPO), Superintendent of Documents, Washington, DC 20402, (202) 512-1800 (Publication Number: 955-001-00000-1). Also, available on-line at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.

<sup>4</sup>. "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 21<sup>st</sup> Edition (2001), 20<sup>th</sup> Edition (1998), 19<sup>th</sup> Edition (1995), and 18<sup>th</sup> Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

<sup>5</sup>. "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 2006. On-line subscription service available at <http://www.standardmethods.org>.

<sup>6</sup>. "Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 2007. Available from: the American Society for Testing and Materials, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959. Also available online at [www.astm.org](http://www.astm.org).

<sup>7</sup>. "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater," U.S. Environmental Protection Agency, September 1978. This EPA publication includes thin-layer chromatography (HPLC) methods.

<sup>8</sup>. "Methods for Analysis of Organic Substances in Water and Fluvial Sediments," Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987).

<sup>9</sup>. The method may be extended to include a-BHC, g-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

<sup>10</sup>. "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).

<sup>11</sup>. Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A, 40 CFR, Part 136) in accordance with procedures given in Section 8.2 of each of these methods. Additionally, each laboratory, on an on-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect. The results should be reported, but cannot be used to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other methods cited.

<sup>12</sup>. "Organochlorine Pesticides and PCBs in Wastewater Using Empore<sup>TM</sup> Disk", 3M Corporation, Revised 10/28/94.

<sup>13</sup>. USGS Method 0-3106-93 from "Methods of Analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of Triazine and Other Nitrogen-containing Compounds by Gas Chromatography with Nitrogen Phosphorus Detectors" U.S. Geological Survey Open File Report 94-37.

<sup>14</sup>. HPLC method 623 from "Methods for Nonconventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater", EPA 440/1-83/079-C, United States Environmental Protection Agency. Available from National Technical Information Service, 5258 Port Royal Road, Springfield, Virginia, 22161 (703) 487-4650.

Section 7: NR 219.04 Table E is repealed and recreated to read:

Table E  
List of Approved Radiological Analytical Methods for Wastewater

Parameter and Units	Analytical Technology	EPA <sup>1</sup>	Standard Methods <sup>2</sup>	Standard Methods Online <sup>3</sup>	ASTM <sup>4</sup>	USGS <sup>5</sup>
1. Alpha-Total, pCi per liter	Proportional or scintillation counter	900.0	7110 B	7110 B-00	D1943-05	pp. 75 and 78 <sup>6</sup>
2. Alpha-Counting error, pCi per liter	Proportional or scintillation counter	Appendix B	7110 B	7110 B-00	D1943-05	p. 79
3. Beta-Total, pCi per liter.	Proportional counter	900.0	7110 B	7110 B-00	D1890-05	pp. 75 and 78 <sup>6</sup>
4. Beta-Counting error, pCi	Proportional counter	Appendix B	7110 B	7110 B-00	D1890-05	p. 79
5. (a) Radium Total pCi per liter	Proportional counter	903.0	7500-Ra B	7500-Ra B-01	D2460-05	
(b) Ra, pCi per liter	Scintillation counter	903.1	7500-Ra C	7500-Ra C-01	D3454-05	p. 81

<sup>1</sup> "Prescribed Procedures for Measurement of Radioactivity in Drinking Water", EPA-600/-4-80-032, U.S. Environmental Protection Agency.

<sup>2</sup> "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 21<sup>st</sup> Edition (2005), 20<sup>th</sup> Edition (1998), 19<sup>th</sup> Edition (1995), and 18<sup>th</sup> Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

<sup>3</sup> "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 2006. On-line subscription service available at <http://www.standardmethods.org>.

<sup>4</sup> "Annual Book of Standards, Section 11.01 and 11.02, Water and Environmental Technology", American Society for Testing and Materials, 2007. Available from: the American Society for Testing and Materials, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959. Also available online at [www.astm.org](http://www.astm.org).

<sup>5</sup> Fishman, M. J. and Brown, Eugene, "Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters," U.S. Geological Survey, Open-File Report 76-177 (1976).

<sup>6</sup> The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the "total."

Section 8: NR 219.04 Table EM is repealed and recreated to read:

Table EM  
List of Approved Analytical Methods for Sludge

Parameter	Analytical Technology	Sample Preparation		Method			
		SW-846 <sup>1</sup>	EPA <sup>4</sup>	SW-846 <sup>1</sup>	EPA <sup>2,3</sup>	Standard Methods <sup>8,9</sup>	Other
Metals							
Arsenic	Gaseous Hydride <sup>5</sup>	7061A		7061A			
	Graphite Furnace	3050B, 3051A	200.2	7010	200.9	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ], 3113 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Beryllium	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 D [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ], 3111 D-99	
	Graphite Furnace	3050B, 3051A	200.2	7010	200.9	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ], 3113 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Cadmium	Flame Atomic Absorption	3050B, 3051A	200.2	7000B			
	Graphite Furnace	3050B, 3051A	200.2	7010	200.9	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ], 3113 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ], 3120 B-99	

Parameter	Analytical Technology	Sample Preparation		Method			
		SW-846 <sup>1</sup>	EPA <sup>4</sup>	SW-846 <sup>1</sup>	EPA <sup>2,3</sup>	Standard Methods <sup>8,9</sup>	Other
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Chromium	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ], 3111 B-99	
	Graphite Furnace	3050B, 3051A	200.2	7010	200.9	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ], 3113 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Copper	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 B or C [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ], 3111 B-99 or C-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Lead	Flame Atomic Absorption	3050B, 3051A	200.2	7000B			
	Graphite Furnace <sup>6</sup>	3050B, 3051A	200.2	7010	200.9	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ], 3113 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Mercury	Cold Vapor Atomic Absorption	7471A, 7471B		7471A, 7471B			
	Cold vapor atomic fluorescence spectrometry	7474					
Molybdenum	Graphite Furnace <sup>6</sup>	3050B, 3051A	200.2	7010	200.9	3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ], 3113 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Nickel	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 B or C [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ], 3111 B-99 or C-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Selenium	Gaseous Hydride <sup>5</sup>	7741A		7741A			
	Graphite Furnace	3050B, 3051A	200.2	7010		3113 B [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ], 3113 B-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Zinc	Flame Atomic Absorption	3050B, 3051A	200.2	7000B		3111 B or C [18 <sup>th</sup> , 19 <sup>th</sup> , 21 <sup>st</sup> ], 3111 B-99 or C-99	
	Inductively Coupled Plasma Emission	3050B, 3051A	200.2	6010B, 6010C	200.7	3120 B [20 <sup>th</sup> , 21 <sup>st</sup> ], 3120 B-99	
	Inductively Coupled Plasma/Mass Spectrometry	3050B, 3051A		6020A	200.8		
Organics							

Parameter	Analytical Technology	Sample Preparation		Method			
		SW-846 <sup>1</sup>	EPA <sup>4</sup>	SW-846 <sup>1</sup>	EPA <sup>2,3</sup>	Standard Methods <sup>8,9</sup>	Other
Dioxins and Furans	Gas Chromatography/Mass Spectrometry	8290A <sup>11</sup>	1613B <sup>11</sup>	8290A	1613B		
PCB (Aroclor or Congeners)	Gas Chromatography	3540B, 3540C, 3545A		8082, 8082A <sup>12</sup>			
PCB (Congeners)	Gas Chromatography/Mass Spectrometry	1668A <sup>13, 14, 15</sup>			1668A <sup>13, 14, 15</sup>		
Biological							
Enteric Viruses	Centrifuge Concentration						D 4994–89 (02) <sup>7</sup> , or Appendix H <sup>10</sup>
Fecal Coliform	Most Probable Number Membrane Filter					9221 E [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ], 9221 E-99, 9222 D, 9222 D-97	Appendix F <sup>10</sup>
Helminth ova	Density Gradient Flotation						Note 9 or Appendix I <sup>9</sup>
Specific Oxygen Uptake Rate	Respirometer					2710 B [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ], 2710 B-04	Appendix D.2. <sup>10</sup>
Salmonella	Most Probable Number Selective Media Culture						9260 D.1 <sup>8</sup> or Appendix G <sup>10</sup>
Physical							
Solids	Gravimetric					2540 G [18 <sup>th</sup> , 19 <sup>th</sup> , 20 <sup>th</sup> , 21 <sup>st</sup> ], 2540 G-97	
Percent Volatiles Solids Reduction	Calculation						Appendix D.1. and 3 <sup>10</sup>

<sup>1</sup>. "Test Methods for Evaluating Solid Waste", Physical/Chemical Methods," SW-846, Environmental Protection Agency, Office of Solid Waste and Emergency Response, 401 M Street, S.W., Washington D.C. 20460, September 1986 (Third edition), including July 1992 (Update I), September 1994 (Update II), August 1993 (Update IIA), January 1995 (Update IIB), December 1996 (Update III), April 1998 (Update IIIA), November 2004 (Update IIIB), February 2007 (Update IV) updates. Available from: The Superintendent of Documents, U.S. Government Printing Office, Room 190, Federal Building, P.O. Box 371954, Pittsburgh, PA 15250-7954. Available online at <http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm>.

<sup>2</sup>. If an alternative digestion procedure is specified in the analytical method, the digestion in this table shall be used. In all cases, consult the analytical method for special requirements and cautions. SW-846 method 3051A is an acceptable alternate digestion procedure to SW-846 method 3050B.

<sup>3</sup>. "Methods for the Determination of Metals in Environmental Samples", EPA-600/4-91-010, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, June 1991. Available from: the National Technical Information Service (NTIS), 5258 Port Royal Road, Springfield, Virginia 22161.

<sup>4</sup>. "Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements", Method 200.2, Revision 2.8, Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, OH 45268, 1994. Available from: the National Technical Information Service (NTIS), 5258 Port Royal Road, Springfield, Virginia 22161.

<sup>5</sup>. High levels of chromium, copper, mercury, silver, cobalt, or molybdenum may interfere with the analysis. Consult Method 3114, of "Standard Methods for the Examination of Water and Wastewater", 18<sup>th</sup>, 19<sup>th</sup>, 20<sup>th</sup>, or 21<sup>st</sup> edition, for more information.

<sup>6</sup>. Concentrations of lead in municipal sludge may exceed the working range of graphite furnace.

<sup>7</sup>. 1993 Annual Book of ASTM Standards, Section 11.02, Water and Environmental Technology", American Society for Testing and Materials, 1993, 1916 Race Street, Philadelphia, PA 19103. Available from: the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

<sup>8</sup>. "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, , 21<sup>st</sup> Edition (2005), 20<sup>th</sup> Edition (1998), 19<sup>th</sup> Edition (1995), and 18<sup>th</sup> Edition, (1992). Available from: American Public Health Association, 1015 Fifteenth Street, N.W., Washington, D.C. 20005.

<sup>9</sup>. "Standard Methods for the Examination of Water and Wastewater", Joint Editorial Board, American Public Health Association, American Water Works Association, and Water Pollution Control Federation, 2006. On-line subscription service available at <http://www.standardmethods.org>.

<sup>10</sup>. "Occurrence of Pathogens in Distribution and Marketing Municipal Sludges", EPA 600/1-87-014, Environmental Protection Agency, 1987. Available from: the National Technical Information Service, order # PB 88-154273/AS, 5285 Port Royal Road, Springfield, Virginia 22161.

<sup>11</sup>. "Environmental Regulations and Technology – Control of Pathogens and Vectors Attraction in Sewage Sludge", EPA-625/R-92/013, Revised October 1999, Environmental Protection Agency, Cincinnati, OH, 1999. Available from: the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

<sup>12</sup>. Analysts may use Fluid Management Systems, Inc. PowerPrep system in place of manual cleanup provided that the analysis meet the requirements of Method 1613B (as specified in Section 9 of the method) and permitting authorities.

<sup>13</sup>. EPA Method 1668A may be used to test for all PCB congeners. If this method is employed, all PCB congeners shall be delineated. Non-detects shall be treated as zero. The values that are between the limit of detection and the limit of quantitation shall be used when calculating the total value of all congeners. All results shall be added together and the total PCB concentration by dry weight reported. It is recognized that a number of the congeners will co-elute with others, so there will not be 209 results to sum.

<sup>14</sup>. EPA Method 8082A shall be used for PCB-Aroclor analysis and may be used for congener specific analysis as well. If congener specific analysis is performed using Method 8082A, the list of congeners tested shall include at least congener numbers 5, 18, 31, 44, 52, 66, 87, 101, 110, 138, 141, 151, 153, 170, 180, 183, 187, and 206 plus any other additional congeners which might be reasonably expected to occur in the particular sample. For either type of analysis, the sample shall be extracted using Soxhlet extraction Method 3540C or Pressurized Fluid Extraction Method 3545A. If Aroclor analysis is performed using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference and achieve as close to a limit of detection of 0.11 mg/kg as possible. If congener specific analysis is done using Method 8082A, clean up steps of the extract shall be performed as necessary to remove interference and to achieve as close to a limit of detection of 0.003 mg/kg as possible for each congener. If the aforementioned limits of detection cannot be achieved after using the appropriate clean up techniques, a reporting limit that is achievable for the Aroclors or each congener for sample shall be determined. This report limit should be reported and qualified indicating the presence of an interference. The laboratory conducting the analysis shall perform as many of the following methods as necessary to remove interference:

- 3620C – Florisil
- 3640A – Gel Permeation
- 3630C – Silica Gel
- 3611B – Alumina
- 3660B – Sulfur Clean Up
- 3665A – Sulfuric Acid Clean Up.

<sup>15</sup>. “Method 1668A, Revision A: Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS”, EPA-821-R-00-002, Environmental Protection Agency, Office of Water, Washington, D.C., December 1999. Available from: the National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161.

Section 9: Table F is repealed and recreated to read:

Table F  
Required Containers, Preservation Techniques, and Holding Time for Wastewater

Parameter number/name	Container <sup>1</sup>	Preservation <sup>2, 3</sup>	Maximum Holding Time <sup>4</sup>
Table A— Bacterial Tests:			
1-5. Coliform, total, fecal and <i>E. coli</i>	PA, G	Cool, <10°C, 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>5</sup>	6 hours
6. Fecal streptococci	PA, G	Cool, <10°C, 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>5</sup>	6 hours
7. Enterococci	PA, G	Cool, <10°C, 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>5</sup>	6 hours
Table A: Protozoan Tests:			
8. Cryptosporidium	LDPE, field filtration	0-8°C	96 hours <sup>21</sup>
9. Giardia	LDPE, field filtration	0-8°C	96 hours <sup>21</sup>
Table A: Aquatic Toxicity Tests			
10-11. Toxicity, acute and chronic	P, FP, G	Cool, ≤6°C <sup>16</sup>	36 hours
Table B— Inorganic Tests			
1. Acidity	P, FP, G	Cool, ≤6°C <sup>18</sup>	14 days
2. Alkalinity	P, FP, G	Cool, ≤6°C <sup>18</sup>	14 days
4. Ammonia	P, FP, G	Cool, ≤6°C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
9. Biochemical oxygen demand	P, FP, G	Cool, ≤6°C <sup>18</sup>	48 hours
11. Bromide	P, FP, G	None required	28 days
14. Carbonaceous biochemical oxygen demand	P, FP, G	Cool, ≤6°C <sup>18</sup>	48 hours
15. Chemical oxygen demand	P, FP, G	Cool, ≤6°C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
16. Chloride	P, FP, G	None required	28 days
17. Chlorine, total residual	P, G	None required	Analyze within 15 minutes
21. Color	P, FP, G	Cool, ≤6°C <sup>18</sup>	48 hours
23-24. Cyanide, total or available (CATC)	P, FP, G	Cool, ≤6°C <sup>18</sup> , NaOH to pH>12 <sup>6</sup> , reducing agent <sup>5</sup>	14 days
25. Fluoride	P	None required	28 days
27. Hardness	P, FP, G	HNO <sub>3</sub> or H <sub>2</sub> SO <sub>4</sub> to pH<2	6 months
28. Hydrogen ion (pH)	P, FP, G	None required	Analyze within 15 minutes
31, 44. Kjeldahl and organic N	P, FP, G	Cool, ≤6°C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
38. Nitrate	P, FP, G	Cool, ≤6°C <sup>18</sup>	48 hours
39. Nitrate + nitrite	P, FP, G	Cool, ≤6°C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
40. Nitrite	P, FP, G	Cool, ≤6°C <sup>18</sup>	48 hours
41. Oil and grease	G	Cool, ≤6°C <sup>18</sup> , HCl or H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
42. Organic carbon	P, FP, G	Cool, ≤6°C <sup>18</sup> , HCl, H <sub>2</sub> SO <sub>4</sub> or H <sub>3</sub> PO <sub>4</sub> to pH<2	28 days

Parameter number/name	Container <sup>1</sup>	Preservation <sup>2,3</sup>	Maximum Holding Time <sup>4</sup>
45. Orthophosphate	P, FP, G	Cool, ≤6°C <sup>18</sup>	Analyze within 48 hours; filter, if needed, within 15 minutes <sup>22</sup>
47. Oxygen, dissolved (Probe or Luminescence)	G, Bottle and top	None required	Analyze within 15 minutes
47. Oxygen, dissolved (Winkler)	G, Bottle and top	Fix on site and store in dark	8 hours
49. Phenols	G	Cool, ≤6°C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
50. Phosphorus (elemental)	G	Cool, ≤6°C <sup>18</sup>	48 hours
51. Phosphorus, total	P, FP, G	Cool, ≤6°C <sup>18</sup> , H <sub>2</sub> SO <sub>4</sub> to pH<2	28 days
54. Residue, total	P, FP, G	Cool, ≤6°C <sup>18</sup>	7 days
55. Residue, filterable	P, FP, G	Cool, ≤6°C <sup>18</sup>	7 days
56. Residue, nonfilterable (TSS)	P, FP, G	Cool, ≤6°C <sup>18</sup>	7 days
57. Residue, settleable	P, FP, G	Cool, ≤6°C <sup>18</sup>	48 hours
58. Residue, volatile	P, FP, G	Cool, ≤6°C <sup>18</sup>	7 days
62. Silica	P or Quartz	Cool, ≤6°C <sup>18</sup>	28 days
65. Specific conductance	P, FP, G	Cool, ≤6°C <sup>18</sup>	28 days
66. Sulfate	P, FP, G	Cool, ≤6°C <sup>18</sup>	28 days
67. Sulfide	P, FP, G	Cool, ≤6°C <sup>18</sup> , add zinc acetate plus sodium hydroxide to pH>9	7 days
68. Sulfite	P, FP, G	None required	Analyze within 15 minutes
69. Surfactants	P, FP, G	Cool, ≤6°C <sup>18</sup>	48 hours
70. Temperature	P, FP, G	None required	Analyze within 15 minutes
74. Turbidity	P, FP, G	Cool, ≤6°C <sup>18</sup>	48 hours
Table B— Metals: <sup>7</sup>			
3, 5-8, 12, 13, 19, 20, 22, 26, 29, 30, 32-34, 36, 37, 46, 48, 52, 53, 59-61, 63, 64, 71-73, 75, 76. Metals, except boron, chromium VI, and mercury.	P, FP, G	HNO <sub>3</sub> to pH<2, or at least 24 hours prior to analysis <sup>19</sup>	6 months
10. Boron	P, FP, or Quartz	HNO <sub>3</sub> to pH<2	6 months
18. Chromium IV	P, FP, G	Cool, ≤6°C <sup>18</sup> , pH = 9.3 - 9.7 <sup>20</sup>	28 days
35. Mercury (CVAA)	P, FP, G	HNO <sub>3</sub> to pH<2	28 days
35. Mercury (CVAFS)	FP, G; and FP-lined cap <sup>17</sup>	5 mL/L 12N HCl or 5 mL/L BrCl <sup>17</sup>	90 days <sup>17</sup>
Table C— Organic Tests <sup>8</sup>			
3, 4. Acrolein and acrylonitrile	G, FP-lined septum	Cool, ≤6°C <sup>18</sup> , 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>5</sup>	14 days <sup>10</sup>
7, 38. Benzidines <sup>11,12</sup>	G, FP-lined cap	Cool, ≤6°C <sup>18</sup> , 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>5</sup>	7 days until extraction <sup>13</sup>
29, 35-37, 66-68, 76, 113. Chlorinated hydrocarbons <sup>11</sup>	G, FP-lined cap	Cool, ≤6°C <sup>18</sup>	7 days until extraction, 40 days after extraction
15, 16, 21, 31, 90. Haloethers	G, FP-lined cap	Cool, ≤6°C <sup>18</sup> , 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>5</sup>	7 days until extraction, 40 days after extraction
56-58, 78, 82. Nitroaromatics and Isophorone <sup>11</sup>	G, FP-lined cap	Cool, ≤6°C <sup>18</sup> , store in dark, 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>5</sup>	7 days until extraction, 40 days after extraction
85-87. Nitrosamines <sup>11,14</sup>	G, FP-lined cap	Cool, ≤6°C <sup>18</sup> , store in dark, 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>5</sup>	7 days until extraction, 40 days after extraction
91-97. PCBs <sup>11</sup>	G, FP-lined cap	Cool, ≤6°C <sup>18</sup>	1 year until extraction, 1 year after extraction
63-65, 69-75, 88, 89, 98-100, 105, 106. PCDDs/PCDFs <sup>11</sup>			
Aqueous Samples: Field and Laboratory Preservation	G	Cool, ≤6°C <sup>18</sup> , 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>5</sup> , pH<9	1 year
Solids and Mixed Phase Samples: Field Preservation	G	Cool, ≤6°C <sup>18</sup>	7 days
Tissue Samples: Field Preservation	G	Cool, ≤6°C <sup>18</sup>	24 hours
Solids, Mixed-Phase, and Tissue Samples: Laboratory Preservation	G	Freeze, ≤-10°C	1 year
23, 30, 44, 49, 53-55, 80, 83, 84, 101, 103, 108, 110, 111, 114, 115, 120-125. Phenols <sup>11</sup>	G, FP-lined cap	Cool, ≤6°C <sup>18</sup> , 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>5</sup>	7 days until extraction, 40 days after extraction
14, 17, 48, 50-52. Phthalate esters <sup>11</sup>	G, FP-lined cap	Cool, ≤6°C <sup>18</sup> , 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>18</sup>	7 days until extraction, 40 days after extraction <sup>13</sup>
1, 2, 5, 8-12, 32, 33, 61, 62, 77, 81, 102, 104. Polynuclear aromatic hydrocarbons <sup>11</sup>	G, FP-lined cap	Cool, ≤6°C <sup>18</sup> , store in dark, 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>5</sup>	7 days until extraction, 40 days after extraction
13, 18-20, 22, 24-28, 34-37, 39-43, 45-47, 59, 79, 107, 109, 113, 116-119, 126. Purgeable halocarbons.	G, FP-lined septum	Cool, ≤6°C <sup>18</sup> , 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>5</sup>	14 days
6, 60, 112. Purgeable aromatic hydrocarbons	G, FP-lined septum	Cool, ≤6°C <sup>18</sup> , HCl to pH<2, 0.0008% Na <sub>2</sub> SO <sub>3</sub> <sup>5</sup>	14 days <sup>9</sup>
Table D. Pesticides Tests			
1-70. Pesticides <sup>11</sup>	G, FP-lined cap	Cool, ≤6°C <sup>18</sup> , pH 5-9 <sup>15</sup>	7 days until extraction, 40 days after extraction
Table E— Radiological Tests:			
1-5. Alpha, beta and radium	P, FP, G	HNO <sub>3</sub> to pH<2	6 months

<sup>1</sup>. "P" is polyethylene; "FP" is fluoropolymer (polytetrafluoroethylene (PTFE; Teflon<sup>®</sup>), or other fluoropolymer, unless stated otherwise in this Table F; "G" is glass; "PA" is any plastic that is made of a sterilizable material (polypropylene or other autoclavable plastic); "LDPE" is low density polyethylene.

<sup>2</sup>. Except where noted in this Table F each grab sample shall be preserved within 15 minutes of collection. For a composite sample collected with an automated sampler, refrigerate the sample at  $\leq 6^{\circ}\text{C}$  during collection unless specified elsewhere in this table or in the method(s). For a composite sample to be split into separate aliquots for preservation and analysis, maintain the sample at  $\leq 6^{\circ}\text{C}$ , unless specified elsewhere in this table or in the method(s) until collection, splitting and preservation is completed. Add the preservative to the sample container prior to sample collection when the preservative will not compromise the integrity of the sample. Preserve (e.g., addition of acid, base or other chemical) the grab sample, composite sample, or aliquot split from the composite sample within 15 minutes of collection. The temperature of the samples shall be documented upon receipt at the laboratory. If the samples are shipped in crushed or cube ice (not "blue ice" packs) and solid ice is still present in the cooler, the lab may simply report the samples as "received on ice". If the ice has melted, the lab must report the either the temperature of the meltwater or of a temperature blank. A temperature blank is defined as an aliquot of deionized water, in an appropriate sample container, which is transported along with the samples. Since shipping simply with "blue ice" packs does not insure that samples are maintained at the appropriate temperatures, the sample collector must submit a temperature blank when using these ice packs for shipping.

<sup>3</sup>. When any sample is to be shipped by common carrier or sent via the U.S. Postal Service, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO<sub>3</sub>) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) 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).

<sup>4</sup>. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before the start of analysis and still be considered valid (e.g., samples analyzed for fecal coliforms may be held up to 6 hours prior to commencing analysis). 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 (s. NR 219.05). A permittee or monitoring laboratory is obligated to hold the sample for a shorter time if it knows that a shorter time is necessary to maintain sample stability.

<sup>5</sup>. Add a reducing agent only in the presence of residual chlorine.

<sup>6</sup>. Sample collection and preservation: Collect a volume of sample appropriate to the analytical method in a bottle of the material specified. If the sample can be analyzed within 48 hours and sulfide is not present, adjust the pH to  $>12$  with sodium hydroxide solution (e.g., 5 % w/v), refrigerate as specified, and analyze within 48 hours. Otherwise, to extend the holding time to 14 days and mitigate interferences, treat the sample immediately using any or all of the following techniques, as necessary, followed by adjustment of the sample pH to  $>12$  and refrigeration as specified. There may be interferences that are not mitigated by approved procedures. Any procedure for removal or suppression of an interference may be employed, provided the laboratory demonstrates that it more accurately measures cyanide. Particulate cyanide (e.g., ferric ferrocyanide) or a strong cyanide complex (e.g., cobalt cyanide) are more accurately measured if the laboratory holds the sample at room temperature and pH  $>12$  for a minimum of 4 hours prior to analysis, and performs UV digestion or dissolution under alkaline (pH=12) conditions, if necessary. **Sulfur:** To remove elemental sulfur (S<sub>8</sub>), filter the sample immediately. If the filtration time will exceed 15 minutes, use a larger filter or a method that requires a smaller sample volume (e.g., EPA Method 335.4 or Lachat Method 01). Adjust the pH of the filtrate to  $>12$  with NaOH, refrigerate the filter and filtrate, and ship or transport to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in  $\mu\text{g}$  or mg), and divide by the original sample volume to obtain the cyanide concentration.

(1) Sulfide: If the sample contains sulfide as determined by lead acetate paper, or if sulfide is known or suspected to be present, immediately conduct one of the volatilization treatments or the precipitation treatment as follows: Volatilization—Headspace expelling. In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a 4.4-L collapsible container (e.g., Cubitainer<sup>™</sup>). Acidify with concentrated hydrochloric acid to pH  $<2$ . Cap the container and shake vigorously for 30 seconds. Remove the cap and expel the headspace into the fume hood or open area by collapsing the container without expelling the sample. Refill the headspace by expanding the container. Repeat expelling a total of five headspace volumes. Adjust the pH to  $>12$ , refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide ( $>10\%$ ). Dynamic stripping: In a fume hood or well-ventilated area, transfer 0.75 liter of sample to a container of the material specified and acidify with concentrated hydrochloric acid to pH  $<2$ . Using a calibrated air sampling pump or flowmeter, purge the acidified sample into the fume hood or open area through a fritted glass aerator at a flow rate of 2.25 L/min for 4 minutes. Adjust the pH to  $>12$ , refrigerate, and ship or transport to the laboratory. Scaling to a smaller or larger sample volume must maintain the air to sample volume ratio. A larger volume of air will result in too great a loss of cyanide ( $>10\%$ ). Precipitation: If the sample contains particulate matter that would be removed by filtration, filter the sample prior to treatment to assure that cyanide associated with the particulate matter is included in the measurement. Ship or transport the filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in  $\mu\text{g}$  or mg), and divide by the original sample volume to obtain the cyanide concentration. For removal of sulfide by precipitation, raise the pH of the sample to  $>12$  with NaOH solution, then add approximately 1 mg of powdered cadmium chloride for each mL of sample. For example, add approximately 500 mg to a 500-mL sample. Cap and shake the container to mix. Allow the precipitate to settle and test the sample with lead acetate paper. If necessary, add cadmium chloride but avoid adding an excess. Finally, filter through 0.45 micron filter. Cool the sample as specified and ship or transport the filtrate and filter to the laboratory. In the laboratory, extract the filter with 100 mL of 5% NaOH solution for a minimum of 2 hours. Filter the extract and discard the solids. Combine the 5% NaOH-extracted filtrate with the initial filtrate, lower the pH to approximately 12 with

concentrated hydrochloric or sulfuric acid, and analyze the combined filtrate. Because the detection limit for cyanide will be increased by dilution by the filtrate from the solids, test the sample with and without the solids procedure if a low detection limit for cyanide is necessary. Do not use the solids procedure if a higher cyanide concentration is obtained without it. Alternatively, analyze the filtrates from the sample and the solids separately, add the amounts determined (in g or mg), and divide by the original sample volume to obtain the cyanide concentration. If a ligand-exchange method is used (e.g., ASTM D6888), it may be necessary to increase the ligand exchange reagent to offset any excess of cadmium chloride.

(2) Sulfite, thiosulfate, or thiocyanate: If sulfite, thiosulfate, or thiocyanate is known or suspected to be present, use UV digestion with a glass coil (Method Kelada-01) or ligand exchange (Method OIA-1677) to preclude cyanide loss or positive interference.

(3) Aldehyde: If formaldehyde, acetaldehyde, or another water-soluble aldehyde is known or suspected to be present, treat the sample with 20 mL of 3.5% ethylenediamine solution per liter of sample.

(4) Carbonate: Carbonate interference is evidenced by noticeable effervescence upon acidification in the distillation flask, a reduction in the pH of the absorber solution, and incomplete cyanide spike recovery. When significant carbonate is present, adjust the pH to  $\geq 12$  using calcium hydroxide instead of sodium hydroxide. Allow the precipitate to settle and decant or filter the sample prior to analysis (also see Standard Method 4500-CN.B.3.d).

(5) Chlorine, hypochlorite, or other oxidant: Treat a sample known or suspected to contain chlorine, hypochlorite, or other oxidant as directed in footnote 5.

<sup>7</sup> For dissolved metals, filter grab samples within 15 minutes of collection and before adding preservatives. For a composite sample collected with an automated sampler, filter the sample within 15 minutes after completion of collection and before adding preservatives.

<sup>8</sup> Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

<sup>9</sup> If the sample is not adjusted to  $\text{pH} < 2$ , then the samples must be analyzed within seven days of sampling.

<sup>10</sup> The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within three days of sampling.

<sup>11</sup> When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity (i.e., use all necessary preservatives and hold for the shortest time listed). When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to  $\leq 6^\circ\text{C}$ , reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (regarding the requirement for thiosulfate reduction), and footnotes 12, 13 (regarding the analysis of benzidine).

<sup>12</sup> If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to  $4.0 \pm 0.2$  to prevent rearrangement to benzidine.

<sup>13</sup> Extracts may be stored up to 30 days at  $< 0^\circ\text{C}$ .

<sup>14</sup> For the analysis of diphenylnitrosamine, add 0.008%  $\text{Na}_2\text{S}_2\text{O}_3$  and adjust pH to 7-10 with NaOH within 24 hours of sampling.

<sup>15</sup> The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008%  $\text{Na}_2\text{S}_2\text{O}_3$ .

<sup>16</sup> Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that  $\leq 6^\circ\text{C}$  has not been exceeded. In the isolated cases where it can be documented that this holding temperature cannot be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

<sup>17</sup> Samples collected for the determination of trace level mercury ( $< 100\text{ ng/L}$ ) using EPA Method 1631 must be collected in tightly-capped fluoropolymer or glass bottles and preserved with BrCl or HCl solution within 48 hours of sample collection. The time to preservation may be extended to 28 days if a sample is oxidized in the sample bottle. A sample collected for dissolved trace level mercury should be filtered in the laboratory within 24 hours of the time of collection. However, if circumstances preclude overnight shipment, the sample should be filtered in a designated clean area in the field in accordance with procedures given in Method 1669. If sample integrity will not be maintained by shipment to and filtration in the laboratory, the sample must be filtered in a designated clean area in the field within the time period necessary to maintain sample integrity. A sample that has been collected for determination of total or dissolved trace level mercury must be analyzed within 90 days of sample collection.

<sup>18</sup> Aqueous samples must be preserved at  $\leq 6^\circ\text{C}$ , and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulatory authority. The specification of " $\leq 6^\circ\text{C}$ " is used in place of the " $4^\circ\text{C}$ " and " $< 4^\circ\text{C}$ " sample temperature requirements listed in some methods. It is not necessary to measure the sample temperature to three significant figures (1/100th of 1 degree); rather, three significant figures are specified so that rounding down to  $6^\circ\text{C}$  may not be used to meet the  $\leq 6^\circ\text{C}$  requirement. The preservation temperature does not apply to samples that must be analyzed within 15 minutes.

<sup>19</sup> An aqueous sample may be collected and shipped without acid preservation. However, acid must be added at least 24 hours before analysis to dissolve any metals that adsorb to the container walls. If the sample must be analyzed within 24 hours of collection, add the acid immediately (see footnote 2). Soil and sediment samples do not need to be preserved with acid. The allowances in this footnote supersede the preservation and holding time requirements in the approved metals methods.

<sup>20</sup> To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6. The allowance in this footnote supersedes preservation and holding time requirements in the approved hexavalent chromium methods, unless this supersession would compromise the measurement, in which case requirements in the method must be followed.

<sup>21</sup> Holding time is calculated from time of sample collection to elution for samples shipped to the laboratory in bulk and calculated from the time of sample filtration to elution for samples filtered in the field.

<sup>22</sup> Filtration is only required when reporting dissolved orthophosphate, dissolved hydrolyzable phosphorus or dissolved organic phosphorus as described in EPA Method 365.1 (1993). Filtration must be completed within 15 minutes of collection using a  $0.45\mu\text{m}$  filter, sample shall be maintained at  $6^\circ\text{C}$  and analyzed within 48 hours.

**SECTION 10. Effective date.** This rule shall take effect the first day of the month following publication in the Wisconsin administrative register as provided in s. 227.22(2)(intro.), Stats.

SECTION 11. Board adoption. This rule was approved and adopted by the State of Wisconsin Natural Resources Board on \_\_\_\_\_.

Dated at Madison, Wisconsin \_\_\_\_\_.

STATE OF WISCONSIN  
DEPARTMENT OF NATURAL RESOURCES

By \_\_\_\_\_  
Matthew Frank, Secretary

(SEAL)