Summary of the 2021 Methods Update Rule

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On May 19, 2021, EPA finalized changes to analytical test procedures that are used to analyze the chemical, physical, and biological components of wastewater and other environmental samples that are required by regulations under the Clean Water Act. (https://www.govinfo.gov/content/pkg/FR-2021-05-19/pdf/2021-09596.pdf) The changes include:

- One revised EPA method (1623.1);
- 47 revised methods and 1 new method from ASTM International
- 16 revised and 5 new methods from *Standard Methods*;
- 7 new/updated methods developed by the U.S. Geological Survey;
- 3 methods approved under the Alternate Test Procedures (ATP) program; and
- Other minor changes.

The rule will become effective July 19, 2021, but many states may take longer to implement.

New EPA Method

EPA added the revised version of EPA Method 1623 (1623.1) for Giardia and Cryptosporidium to Table IH and added footnote 31which states:

> Method 1623.1 includes updated acceptance criteria for IPR, OPR, and MS/MSD and clarifications and revisions based on the use of Method 1623 for years and technical support questions

A copy of this method can be found here: https://www.regulations.gov/document/EPA-HQ-OW-2018-0826-0061

Revised Standard Methods Tables 1A and 1H

Method	Analyte(s)	Technology
9221 (B, D, E, F)-14	Coliform (total), Coliform (fecal), *E. coli*	most probable number (MPN), 5 tube 3 dilution.
9222 (B, D, I)-15	Coliform (total), Coliform (fecal), *E. coli*	membrane filtration (MF).
9223 B-16	*E. coli*	multiple tube/multiple well.
9230 (B, C)-13	Fecal Streptococci, Enterococci	MPN, 5 tube 3 dilution or MF.
9221 F.2-14*	Coliform (fecal)	MPN, 5 tube 3 dilution

* Note: In the proposed rule EPA indicated they planned to approve Method 9221 F.2- 2014 as an acceptable method for detecting thermotolerant coliforms and *E. coli* simultaneously and to add footnote 33 with this language: “This method analyzes Coliform (fecal) and *E. coli* using EC broth with 4- methylumbelliferyl-β-D-glucuronide (EC-MUG) with inverted vials.” This change was not discussed in the preamble to the 2021 final rule, but the method and footnote are in Tables 1A and 1H.

Revised Standard Methods Table 1B

Method	Analyte(s)	Technology
2540 B, C, D, E, and F-15	total, filterable, non-filterable, volatile, and settleable residue	gravimetric
4500-CN^- (B-F)-16	cyanide	manual distillation with magnesium chloride ($MgCl_2$) followed by: titrimetric, manual or semiautomated spectrophotometric, ion selective electrode
4500-CN^- G-16	cyanide available (cyanide amenable to chlorination (CATC))	manual distillation with $MgCl_2$, followed by titrimetric or spectrophotometric
4500-NO_3^- D-16	nitrate (as nitrogen)	ion-selective electrode (ISE).
4500-NO_3^- (E, F, and H)-16	nitrate-nitrite (as nitrogen),	colorimetric: cadmium reduction—manual and automated; and colorimetric: automated hydrazine
4500-NO_3^- (E and F)-16	nitrite (as nitrogen)	colorimetric: cadmium reduction—manual and automated.
4500-O (B-F, and G)-16	oxygen (dissolved)	Winkler (azide modification), and electrode
5210 B-16	biochemical oxygen demand (BOD_5) and CBOD	dissolved oxygen depletion
5310 (B, C)-14	total organic carbon (TOC)	combustion, heated persulfate or UV persulfate oxidation

New Standard Methods Table 1B

Method	Analyte(s)	Technology
4500-CN^- N-16	Cyanide, total	semi-automated spectrophotometry.
4500-NO_3^- I-16	combined nitrate-nitrite, nitrite (bypass the reduction column) and nitrate by subtraction	automated cadmium reduction and spectrophotometry
4500-NO_3^- J-18	combined nitrate-nitrite, nitrite when bypassing the enzymatic reduction step, and nitrate by subtraction	enzymatic reduction followed by colorimetric, manual
4500-O H-16	dissolved oxygen	luminescent-based sensor

New United States Geological Survey (USGS) Methods

Method	Analytes	Technology
I-2057-85	bromide, chloride, fluoride, nitrate, nitrite, orthophosphate, and sulfate	ion-exchange chromatographic, automat
I-2522-90	Nitrogen, ammonia	colorimetry, salicylate-hypochlorite, automated-segmented flow
I-2540-90	Nitrogen, nitrite	colorimetry, diazotization, automated-segmented flow
I-2601-90	Phosphorus, orthophosphate	colorimetry, phosphomolybdate, automated-segmented flow
I-4472-97	Metals	inductively coupled plasma-mass spectrometry
O-4127-96	Volatile Organic Compounds	Gas Chromatography/Mass Spectrometry
O-4436-16	Heat Purgeable and Ambient Purgeable Volatile Organic Compounds	Gas Chromatography/Mass Spectrometry

New Alternate Test Procedures (ATPs)

Method	Analytes	Technology
FIAlab 100	Ammonia	automated flow injection analysis with gas diffusion and fluorescence
MACHEREY-NAGEL GmbH 036/038	Chemical Oxygen Demand	spectrophotometry, manual
Micrology Laboratories KwikCount™	*Escherichia coli* (Table 1 H only)	enzyme substrate test

Revised ASTM Methods Table 1A

[Editor's Note: The information in the preamble was significantly revised by converting the information into a table, adding a line for each individual analyte (as in anions), and then sorting by analyte name for easier comparison to Table 1B. This table does not list any metal other than gold for Method D5673-16 as that was the only change to the method, but EPA has updated this version for all other metals previously approved.]

Analyte	Method	Technology	Comment
Acidity	D1067-16	electrometric endpoint or phenolphthalein endpoint; electrometric or colorimetric titration to pH 4.5, manual.	No procedural changes.
Alkalinity	D1067-16	electrometric endpoint or phenolphthalein endpoint; electrometric or colorimetric titration to pH 4.5, manual.	No procedural changes.
Aluminum	D4190-15	direct current plasma.	Added a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.
Ammonia	D1426-15 (A, B)	Nesslerization, electrode.	A lengthy section of QC requirements was added to the D1426A) that parallels the QC discussion that was already in the B procedure. Both procedures added information on use of commercially prepared standards and filter paper
	D6919-17	ion chromatography.	No procedural changes.
Arsenic	D2972-15 (A–C)	colorimetric, AA gaseous hydride, AA furnace	QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now tied to a laboratory-defined batch of up to 20 samples.
Barium	D4382-18	AA furnace	Changed the description of the hot block digester equipment. The new version specifies the capability to heat samples between 65 and 95 degrees C, instead of "approximately 95 degrees C." That change recognizes the operational characteristics of hot block digesters that will experience a temperature drop below 95 degrees when samples are added. This should not adversely affect use of this method.
Beryllium	D4190-15	direct current plasma.	Added a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.

Analyte	Method	Technology	Comment
Beryllium	D3645-15 (A, B)	AA direct aspiration AA furnace	Added specifications for filter paper. Clarified the requirements for a three-point calibration by discussing it in the calibration section as well as the QC section of both procedures. Added a new section with the QC requirements to the direct aspiration AA procedure that was already present in the AA furnace portion of this procedure (D3645-15 [B]).
Boron	D4190-15	direct current plasma.	Added a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.
Bromide	D1246-16	electrode.	No procedural changes.
	D6508-15	capillary ion electrophoresis with indirect UV detection.	No procedural changes.
	D4327-17	ion chromatography	Changes include updating the equipment and reagent descriptions to reflect more modern instrumentation, such as use of hydroxide eluents and eluent regeneration systems.
Cadmium	D3557-17 (A–D)	AA direct aspiration, voltammetry, AA furnace	Clarified requirements for a multi-point calibration by discussing it in the calibration section as well as the QC section of all three procedures. The QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now tied to a laboratory-defined batch of up to 20 samples, as opposed to 10 samples previously.
	D4190-15	direct current plasma.	Added a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.
Calcium	D511-14 (A, B)	titrimetric, (EDTA), AA direct aspiration.	Added specifications for filter paper.
	D6919-17	ion chromatography.	No procedural changes.
Chemical Oxygen Demand	D1252-06 (A, B) (12)	titrimetric, spectrophotometric.	No procedural changes.
Chloride	D512 (A, B)-12	titrimetric (mercuric nitrate), titration (silver nitrate).	Corrected one term in the calculation of the chloride calculation.
	D6508-15	capillary ion electrophoresis with indirect UV detection.	No procedural changes.

Analyte	Method	Technology	Comment
Chloride	D4327-17	ion chromatography	Changes include updating the equipment and reagent descriptions to reflect more modern instrumentation, such as use of hydroxide eluents and eluent regeneration systems.
Chlorine, residual	D1253-14	amperometric direct.	No procedural changes.
Chromium (total)	D1687-17 (B, C)	AA direct aspiration; AA furnace	The QC frequencies for method blank, continuing calibration verification (CCV), continuing calibration blank (CCB), matrix spike, and duplicate analyses are now tied to a laboratory-defined batch of up to 20 samples.
	D4190-15	direct current plasma.	Added a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.
Chromium, hexavalent	D1687-17 (B, C)	colorimetric (diphenyl–carbazide); AA direct aspiration; AA furnace	The QC frequencies for method blank, continuing calibration verification (CCV), continuing calibration blank (CCB), matrix spike, and duplicate analyses are now tied to a laboratory-defined batch of up to 20 samples.
	D5257-17	ion chromatography	Added a few additional warnings or recommendations.
Cobalt	D3558-15 (A–C)	AA direct aspiration, chelation extraction AA, and AA furnace	Clarified requirements for a multi-point calibration by discussing it in the calibration section as well as the QC section of all three procedures. The QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now tied to a laboratory-defined batch of up to 20 samples, as opposed to 10 samples previously.
	D4190-15	direct current plasma.	Added a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.
Copper	D1688-17 (A–C)	AA direct aspiration, AA furnace	Clarified the requirements for a multi-point calibration by discussing it in the calibration section as well as the QC section of all three procedures. The QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now tied to a laboratory-defined batch of up to 20 samples.
	D4190-15	direct current plasma.	Added a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.

Analyte	Method	Technology	Comment
Cyanide, available	D6888-16	flow injection and ligand exchange, followed by gas diffusion amperometry	Added a new mixed ligand exchange reagent, but also retains the original two ligand reagents that had to be mixed together during the testing.
	D2036-09 (A, B) (15)	manual distillation followed by titrimetric or spectrophotometric.	No procedural changes.
Cyanide, free	D4282-15	manual micro-diffusion and colorimetry.	No procedural changes.
	D7237-15 (A)	flow injection, followed by gas diffusion amperometry	Applicable range of the method has been changed from 2 to 500 µg/L to 5 to 500 µg/L. New information about interferences from floatation reagents has been added to Section 6.3. New materials in Section 8 discuss alternative reagents or concentrations.
Cyanide, total	D2036-09 (A, B) (15)	manual distillation followed by gas diffusion amperometry, titrimetric, spectrophotometric, ion chromatography, ion selective electrode	No procedural changes.
	D7284-13 (17)	manual distillation with $MgCl_2$ followed by flow injection, gas diffusion amperometry.	No procedural changes.
	D7511-12 (17)	segmented flow injection, in-line ultraviolet digestion, followed by gas diffusion amperometry.	No procedural changes.
Fluoride	D1179-16 (A, B)	colorimetric, (SPADNS), electrode, manual.	No procedural changes.
	D6508-15	capillary ion electrophoresis with indirect UV detection.	No procedural changes.
	D4327-17	ion chromatography	Changes include updating the equipment and reagent descriptions to reflect more modern instrumentation, such as use of hydroxide eluents and eluent regeneration systems.
Gold	D5673-16	ICP/MS	Gold was added to the list of target analytes. Some of the changes address the analysis of gold.
Hardness	D1126-17	titrimetric (EDTA).	No procedural changes.
Iron	D4190-15	direct current plasma.	Added a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.
	D1068-15 (A-C)	AA direct aspiration; AA furnace; colorimetric (Phenanthroline).	Added specifications for filter paper.

Analyte	Method	Technology	Comment
Lead	D4190-15	direct current plasma.	Added a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.
	D3559-15 (A–D)	AA direct aspiration, voltammetry, AA furnace	Clarified the requirements for a multi-point calibration by discussing it in the calibration section as well as the QC section of all three procedures. Also added a new section with the QC requirements to the direct AA procedure that was already present in the AA furnace portion of this procedure (D3559-15 [D]).
Magnesium	D511-14 (A, B)	titrimetric, (EDTA), AA direct aspiration.	Added specifications for filter paper.
	D6919-17	ion chromatography.	No procedural changes.
Manganese	D858-17 (A–C)	AA direct aspiration, AA furnace.	No procedural changes.
	D4190-15	direct current plasma.	Added a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.
Mercury	D3223-17	cold vapor, manual	Changed the acceptance limit for the CCV from 10% to 15% and added a requirement for a CCB. Given that the most comparable EPA procedure, Method 245.1, does not include a CCV requirement or an acceptance limit, the change of the acceptance limit from 10% to 15% in the revised method represents a requirement that is more stringent than that in EPA's procedure and therefore, the change is not an impediment.
Nickel	D4190-15	direct current plasma.	Added a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.
	D1886-14 (A–C)	AA direct aspiration, chelation extraction AA and AA furnace	QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now tied to a laboratory-defined batch of up to 20 samples.
Nitrate	D6508-15	capillary ion electrophoresis with indirect UV detection.	No procedural changes.
	D4327-17	ion chromatography	Changes include updating the equipment and reagent descriptions to reflect more modern instrumentation, such as use of hydroxide eluents and eluent regeneration systems.

Analyte	Method	Technology	Comment
	D3867-16 (A, B)	automated cadmium reduction, manual cadmium reduction, bypass cadmium reduction and subtraction	Added more detailed QC requirements, including specifically calling out the laboratory control sample (LCS), method blank, and matrix spike analyses. The 2016 version adds specifications for filter paper. It also changes the LCS frequency from 10% of samples to once per batch (up to 20) and sets the CCB and CCV frequencies at 10%.
Nitrate-nitrite	D6508-15	capillary ion electrophoresis with indirect UV detection.	No procedural changes.
	D3867-16 (A, B)	automated cadmium reduction, manual cadmium reduction, bypass cadmium reduction and subtraction	Added more detailed QC requirements, including specifically the laboratory control sample (LCS), method blank, and matrix spike. Added specifications for filter paper. Changed the LCS frequency from 10% of samples to once per batch (up to 20) and sets the CCB and CCV frequencies at 10%.
Nitrite	D6508-15	capillary ion electrophoresis with indirect UV detection.	No procedural changes.
	D4327-17	ion chromatography	Changes include updating the equipment and reagent descriptions to reflect more modern instrumentation, such as use of hydroxide eluents and eluent regeneration systems.
	D3867-16 (A, B	automated cadmium reduction, manual cadmium reduction, bypass cadmium reduction and subtraction	Added more detailed QC requirements, including laboratory control sample (LCS), method blank, and matrix spike analyses. Added specifications for filter paper. Changed the LCS frequency from 10% of samples to once per batch (up to 20) and sets the CCB and CCV frequencies at 10%.
Nitrogen, ammonia	D1426-15 (A, B)	Nesslerization, electrode.	QC requirements were added to D1426A that parallels the QC discussion that was already in the B procedure. Both procedures added information on use of commercially prepared standards and filter paper.
Nitrogen, Kjeldahl	D3590-17 (A, B)	manual digestion and distillation or gas diffusion; semi-automated block digester colorimetric (distillation not required)	Changed the acceptance limit for the CCV from 10% to 15% and adds a requirement for a CCB. Given that neither the approved Standard Methods method for measuring ammonia after the TKN digestion, nor the comparable EPA Method 350.1, include a CCV requirement or an acceptance limit, the change of the acceptance limit from 10% to 15% in the revised ASTM method represents a requirement that is more stringent than that required in other approved procedures and therefore is not an impediment to its approval.

Analyte	Method	Technology	Comment
Orthophosphate	D6508-15	capillary ion electrophoresis with indirect UV detection.	No procedural changes.
	D4327-17	ion chromatography	Changes include updating the equipment and reagent descriptions to reflect more modern instrumentation, such as use of hydroxide eluents and eluent regeneration systems.
Oxygen, dissolved	D888-12 (A–C)	Winkler, electrode, luminescent-based sensor.	Added information on a two-point calibration and updated performance information from an interlaboratory study to D888-12 (C).
Phenols	D1783-01 (A, B) (12)	manual distillation followed by manual colorimetric (4AAP).	No procedural changes.
Potassium	D6919-17	ion chromatography.	No procedural changes.
Selenium	D3859-15 (A, B)	AA gaseous hydride, AA furnace	The changes to the gaseous hydride portion of the method clarified the requirement for a 6-point calibration by discussing it in the calibration section as well as the QC section. Added an updated discussion of block digesters. The QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now tied to a laboratory-defined batch. The GFAA portion contains similar editorial and technical changes. Technical changes also included specifications for filter paper. The calibration requirement for three standards has been clarified by discussing it in the calibration section as well as the QC section.
Silica	D859-16	colorimetric, manual.	No procedural changes.
Sodium	D6919-17	ion chromatography.	No procedural changes.
Sulfate	D6508-15	capillary ion electrophoresis with indirect UV detection.	No procedural changes.
	D4327-17	ion chromatography	Changes include updating the equipment and reagent descriptions to reflect more modern instrumentation, such as use of hydroxide eluents and eluent regeneration systems.
	D516-16	turbidimetric.	Added specifications for filter paper.
Sulfide	D4658-15	ion selective electrode.	No procedural changes.
Total Organic carbon	D4839-03 (17)	heated persulfate or UV persulfate oxidation.	No procedural changes.
	D7573-09 (17)	combustion	No procedural changes.

Analyte	Method	Technology	Comment
Vanadium	D4190-15	direct current plasma.	Added a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.
	D3373-17	AA furnace	Clarified the requirements for a multi-point calibration by discussing it in the calibration section as well as the QC section of all three procedures. The QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now tied to a laboratory-defined batch of up to 20 samples.
Zinc	D4190-15	direct current plasma.	Added a requirement to run at least four calibration standards for all metals, as opposed to running four standards for only lithium to demonstrate linearity.
	D1691-17 (A, B)	AA direct aspiration	QC frequencies for method blank, CCV, CCB, matrix spike, and duplicate analyses are now tied to a laboratory-defined batch of up to 20 samples.

Revised ASTM Methods Table 1C

Method	Analytes	Technology	Comment
D7065-17	nonylphenol, bisphenol A, p-tert-octylphenol, nonylphenol monoethoxylate, nonylphenol diethoxylate	gas chromatography/mass spectrometry (GC/MS)	A large number of editorial and structural changes were made. A new QC section was added.

New ASTM Method Table 1B

Method	Analytes	Technology
D7781-14	nitrate-nitrite, nitrite (bypass the enzymatic reduction step) and nitrate by subtraction	enzymatic reduction followed by automated colorimetry

Other Changes

1. Changes to Tables IA and IH

Table IA

- Moved Colilert-18 from Parameter #1 Coliform (fecal) to Parameter #2 Coliform (fecal), to eliminate confusion as to whether it is approved for sewage sludge in addition to wastewater.
- Added *E. coli*, number per 100 mL - MF, two-step, Standard Methods Method 9222 B/9222 I, to the table along with footnote 31 "Subject coliform positive samples determined by 9222 B-2015 or other membrane filter procedure to 9222 I-2015 using NA-MUG media." The method was inadvertently omitted from Table IA when Table IA was split into two tables (IA and IH) in an earlier rulemaking.
- Revised Parameter #2 Coliform (fecal), deleting "in presence of chlorine." The phrase caused confusion because the methods cited were the same for the analyte/matrix combination that did not state "in the presence of chlorine."
- Deleted Parameter #4 Coliform (total) in presence of chlorine. Except for "MF with enrichment," all the methods were duplicative (e.g., Parameters #3 and #4). No approved methods for coliform (total) were removed from Table IA.
- Added the EPA Errata Sheet to footnotes 25, 26, and 27 for Whole Effluent Toxicity Testing. Available at:

Table IH

- Deleted Parameters #2 Coliform (fecal) and #4 Coliform (total). Except for "MF with enrichment" for coliform (total), all the methods were duplicative (e.g., Parameters #1 and #2). In addition to the methods being duplicative, Table IH is for ambient water which would not be expected to contain chlorine. No approved methods for coliform (fecal) or coliform (total) were removed from Table IH. The remaining parameters were renumbered.
- Deleted "or number per gram dry weight" from Parameter #1. Table IH is specifically for ambient waters, which does not require reporting results on a per gram dry weight basis.
- Adding the Alternate Test Procedure KwikCount™ EC for *E. coli*, number per 100 mL under "Other."
- Added EPA Method 1623.1 for Parameters 6 and 7. EPA Method 1623.1.
- Deleted footnote 5, "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." Table IH is specifically for ambient waters, so the footnote is not applicable. The remaining footnotes are renumbered accordingly.
- Revised footnote 20, to reference only EPA Method 1604. The literature reference was deleted from the footnote because it resulted in confusion as to whether EPA Method 1604 provided all the necessary information required by stakeholders to conduct analyses of ambient waters under the CWA.

Tables IA and IH

- Revised footnote 13 to Table IA and footnote [11] to Table IH as follows "These tests are collectively known as defined enzyme substrate tests." The remaining text, "where, for example, a substrate is used to detect the enzyme β-glucuronidase produced by *E. coli*" has been deleted because the example has caused some confusion to stakeholders.
- Quanti-Tray 2000 was added as an option to footnotes 13 (IH), 15 (IH), 16 (IA) and 18 (IA).

The addition of Quanti-Tray 2000 is to address matrices with high bacterial concentrations and to ensure Tables IA and IH are accurate and consistent.

- Revised footnote 30 to Table IA and footnote 27 to Table IH to specify a verification procedure. The footnotes contain the following language: "On a monthly basis, at least ten sheen colonies from positive samples must be verified using Lauryl Tryptose Broth and brilliant green lactose bile broth, followed by count adjustment based on these results; and representative non-sheen colonies should be verified using Lauryl Tryptose Broth. Where possible, verifications should be done from randomized sample sources." Adding the footnotes addresses the change in Standard Methods Method 9222 B-15 that stated that five typical and five atypical colonies should be verified per membrane, which could be burdensome to laboratories analyzing samples other than drinking water. In most cases, analysis of ambient waters and wastewaters could result in multiple plates per sample with typical and atypical colonies, whereas drinking water analyses would seldom result in any typical or atypical colonies. In addition, the language in footnotes 29 (IA) and 26 (IH), was revised as follows "the medium" was replaced with "positive samples" for clarity and consistency.
- Added footnote 32 to Table IA and footnote 30 to Table IH. The footnotes contain the following language "Verification of colonies by incubation of BHI agar at 10 ± 0.5 °C for 48 ± 3 h is optional." As per the Errata to the 23rd Edition of *Standard Methods*, "Growth on a BHI agar plate incubated at 10 ± 0.5°C for 48 ± 3 h is further verification that the colony belongs to the genus Enterococcus."
- Updated the Aquatic Toxicity Table by adding the common names of the genus and species.

2. Changes to Table IB

In the proposal, EPA listed ASTM Method D1179-16(A) in the wrong row in the Table IB entry for Fluoride. The method was erroneously listed in the row for colorimetric methods.

EPA also corrected the publication date of the Macherey-Nagel COD method in Footnote 83.

EPA added Footnote 84 to the heading for Standard Methods which states:

> Please refer to the following applicable Quality Control Sections: Part 2000 Methods, Physical and Aggregate Properties 2020 (2017); Part 3000 Methods, Metals, 3020 (2017); Part 4000 Methods, Inorganic Nonmetallic Constituents, 4020 (2014); Part 5000 Methods, and Aggregate Organic Constituents, 5020 (2017). These Quality Control Standards are available for download at www.standardmethods.org at no charge.

This footnote was not discussed in the preamble, but is consistent with the 2012 Rule that added Section 136.7 to Part 136 and with Standard Methods Part 1000 which states:

> Parts 2000 through 9000 contain sections describing QC practices specific to the methods in the respective parts; these practices are considered to be integral to the methods.

EPA added Footnote 85 to the heading for Standard Methods which states:

> Each laboratory may establish its own control limits by performing at least 25 glucose-glutamic acid (GGA) checks over several weeks or months and calculating the mean and standard deviation. The laboratory may then use the mean ± 3 standard deviations as the control limit for future GGA checks. However, GGA acceptance criteria can be no wider than 198 ± 30.5 mg/L for BOD_5. GGA

acceptance criteria for CBOD must be either 198 ± 30.5 mg/L, or the lab may develop control charts under the following conditions:

- Dissolved oxygen uptake from the seed contribution is between 0.6 – 1.0 mg/L.
- Control charts are performed on at least 25 GGA checks with three standard deviations from the derived mean.
- The RSD must not exceed 7.5%.
- Any single GGA value cannot be less than 150 mg/L or higher than 250 mg/L.

This footnote was not discussed in the preamble.

3. Changes to Table II

EPA updated footnote 6 to the preservation and holding time requirements for cyanide to cite the latest version of ASTM method D7365-09a that was reapproved in 2015. The recommended sampling and preservation procedures in the ASTM method have not changed since 2009, but the change to footnote 6 will simplify identification of the current method that is available from ASTM International.

EPA revised footnote 9 for the preservation and holding time requirements for purgeable halocarbons. This change allows the flexibility to collect a single sample with no acidification to be used for analysis of both purgeable halocarbons and purgeable aromatic hydrocarbons within seven days of collection, or to collect a single sample with acidification to be used for analysis of both purgeable halocarbons (except 2-CEVE) and purgeable aromatic hydrocarbons within the 14-day maximum holding time.

In response to a comment that pointed out that EPA did not update Table II to capture the microbiological method changes in Tables IA and IH, EPA modified Table II to make of these changes.

A commenter pointed out that EPA did not include organic parameter #73, hexachloroethane in Table II. EPA has corrected this error that dates to the Methods Update Rule proposed in 2004. The parameter #73 has been added to the list of chlorinated hydrocarbons in Table II.

A typographical error in Table II of the proposed rulemaking resulted in the specifications for four matrices listed under the dioxin and furan (CDDs/CDFs) entry to not be indented. This caused some confusion for commenters. EPA has ensured that Table II in the final rule appears as intended.

4. Microbial Methods for Biosolids

EPA clarified that Method 9221E-14 is approved for testing sewage sludge. In Table IA, EPA changed Footnote 11 from ‘approved’ to ‘recommended’ in the proposed rulemaking because the 2017 Methods Update Rule erroneously changed the footnote from ‘recommended’ to ‘approved.’ EPA corrected this error and changed the footnote back to “recommended”.
EPA has approved all biosolid methods listed in Table 1A for parameter #1, including those listed in Footnote 11. More method validation data is available for EPA Methods 1680 and 1681 than Standard Method 9221. EPA methods are recommended over 9221 and 9222, although all four methods are approved for biosolids.

5. Changes to 136.6 Method modifications and analytical requirements

EPA added a new paragraph to 136.6 that explicitly allows the use of closed- vessel microwave digestion as a modification to the approved metals digestion procedure that does not require prior approval. Microwave digestion has the same fundamental chemistry as a hot plate digestion.