

EPA Finalizes Methods Update Rule

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The EPA has finalized extensive changes to the analysis and sampling procedures in the wastewater and drinking water regulations. These changes, included in a regulation known as the Methods Update Rule (MUR), approve new methods and update versions of currently approved methods, revise method modification and analytical requirements, withdraw certain outdated methods and modify the sample collection, preservation, and holding time requirements. The rule was originally proposed in 2004, and its effective date is 30 days from the date of publication in the Federal Register, which is expected to be sometime in February.

Highlights from the final rule are summarized below:

New Chemical Test Methods

This rule approved 22 new procedures for monitoring chemical pollutants:

- ASTM D6508, Dissolved Inorganic Anions by Capillary Ion Electrophoresis.
- QuikChem Method 10-204-00-1-X, Cyanide using MICRO DIST and flow injection analysis.
- Kelada-01, Automated Methods for Total Cyanide, Acid Dissociable Cyanide, and Thiocyanate.
- Method CP-86.07, Chlorinated Phenolics by In situ Acetylation and GC/MS.
- EPA Method 245.7, Mercury by Cold Vapor Atomic Fluorescence Spectrometry.
- Standard Methods 4500-Cl, Chlorine by Low Level Amperometry.
- ASTM D6888-04 Available Cyanide by Ligand Exchange-FIA.
- ASTM D 6919-03, Cations and Ammonium in by Ion Chromatography.
- Standard Method 4500-Cl-D. Chloride by Potentiometry.
- ASTM D512-89 Chloride by Ion Selective Electrode.
- Standard Method 4500-CN-F, Cyanide by Ion Selective Electrode.
- ASTM D2036-98 A, Cyanide by Ion Selective Electrode.
- Standard Method 4500-S2-G, Sulfide by Ion Selective Electrode.
- ASTM D4658-92, Sulfide by Ion Selective Electrode.
- Standard Method 4500-NO3-D, Nitrate by Ion Selective Electrode.
- Method D99-003, Free Chlorine by Color Comparison Test Strip.
- Method OIA-1677, DW Available Cyanide by Ligand Exchange_FIA.
- Radium-226 and 228 by Gamma Spectrometry.
- EPA Method 327.0, Chlorine Dioxide by Colorimetry.
- EPA Method 300.1 for Anions.
- EPA Method 552.3 for Dalapon.
- Determination of Radium-226 and Radium-228 in Drinking Water by Gamma-ray Spectrometry Using HPGE or Ge(Li) Detectors.

Note that some of these methods were approved for wastewater only, some for drinking water only, and some for both wastewater and drinking water.

Updated Chemical Test Methods

The rule also approves methods that were first proposed in 1995, including:

- Method 200.2, Total Recoverable Elements Digestion.
- Method 200.8, Metals by Inductively Coupled Plasma-Mass Spectrometry.
- Method 200.9, Metals by Stabilized Temperature Graphite Furnace Atomic Absorption.
- Method 218.6, Hexavalent Chromium by Ion Chromatography.

- Method 300.0, Inorganic Anions by Ion Chromatography.
- Method 353.2, Nitrate and Nitrite by Colorimetry.
- Revisions to Methods 180.1, 200.7, 245.1, 335.3, 350.1, 351.2, 353.2, 365.1, 375.2, 410.4, and 420.4.

The ASTM and Standard Methods versions of many of these methods were also approved.

Updated Versions of Currently Approved Methods

This rule approved about 200 updated methods, including:

- An errata sheet for the whole effluent toxicity manuals.
- 74 newer versions of ASTM methods.
- 88 newer versions of Standard Methods from the 18th, 19th and 20th editions, but not the 21st.
- 19 methods published in the 16th edition of Official Methods of Analysis of AOAC International, 1995.

Method Modifications, Analytical Requirements, and Reporting Requirements

The final rule includes a new section to introduce greater flexibility in the use of approved methods. The section describes the circumstances in which approved methods may be modified and the requirements that analysts must meet to use these modified methods in required measurements without prior EPA approval. The rule also includes language to clarify that analysts need only meet method performance requirements for target analytes when using multi-analyte methods for compliance monitoring purposes. The 2004 proposal included language to clarify that a QC failure does not grant relief of timely reporting of results to a regulated entity, and that results be reported to the level specified in the method or required in the permit, whichever is lower. In the final rule, the EPA has clarified this language to allow greater flexibility.

As well, the rule approves the replacement of the mercuric sulfate catalyst with copper sulfate in methods approved for the determination of total Kjeldahl nitrogen, and approves the use of styrene divinylbenzene beads and Hach StablCal as alternatives to the formazin standard for turbidity. The rule also approves the use of capillary GC columns with Methods 601-613, 624, 625, and 1624B.

Sample Collection, Preservation, and Holding Time Requirements

The rule includes many detailed changes to Table II, including:

- The general sample preservation temperature from has changed 4 C to < 6.00 C.
- For metals other than boron, hexavalent chromium, and mercury, the EPA will allow sample preservation with nitric acid 24 hours prior to analysis. In other words, acid preservation in the field for metals is not required.
- Clarification that the start of a holding time for a grab sample would start at the time of sample collection. The holding time for a composite sample would start at the time the last grab sample component is collected.

Withdrawal of Methods

The rule deletes Methods 612 and 625 as approved procedures for 1,2-dichlorobenzene, 1,3-dichlorobenzene, and 1,4-dichlorobenzene, and withdraws approval for all oil and grease methods that use Freon-113 as an extraction solvent. In addition, the rule withdraws 105 methods contained in the EPA's *Methods for the Chemical Analysis of Water and Wastes* for which approved alternatives published by voluntary consensus standards bodies (i.e., ASTM and Standard Methods) are available. The methods that are deleted are listed below:

110.1	208.2	236.1	272.1	330.3	360.2
110.2	210.1	236.2	272.2	330.4	365.2
110.3	210.2	239.1	273.1	330.5	370.1
130.2	212.3	239.2	279.1	335.1	375.1
150.1	213.1	242.1	282.1	335.2	375.3
160.1	213.2	243.1	282.2	335.3	375.4
160.2	215.1	243.2	283.1	340.1	376.1
160.3	215.2	246.1	286.1	340.2	376.2
160.5	218.1	246.2	286.2	340.3	377.1
170.1	218.2	249.1	289.1	350.2	405.1
202.1	218.3	249.2	305.1	350.2	410.1
202.2	218.4	252.1	310.1	350.3	410.2
204.1	219.1	253.1	320.1	351.3	413.1
204.2	219.2	255.1	325.1	351.4	415.1
206.2	220.1	258.1	325.2	353.1	425.1
206.3	220.2	265.1	325.3	353.3	
206.4	231.1	267.1	330.1	354.1	
208.1	235.1	270.2	330.2	360.1	

Because the 20th edition of Standard Methods is out of print, the Standards Methods Board is considering a special publication of their methods which are “equivalent” to the EPA methods listed above.

To view a pre-publication copy of the final rule, go to
<http://www.epa.gov/waterscience/methods/update2003/mur-pre-pubfinal.pdf>