



# Department of Environmental Protection

## Low-Level Mercury Method

### Mercury Testing

In accordance with Rule 62-620.610, Florida Administrative Code (F.A.C.), all sampling and monitoring data, required to be reported to the Department, shall be collected and analyzed in accordance with Rule 62-4.246, Chapters 62-160 and 62-600, F.A.C., and 40 CFR 136, as appropriate. Effective August 25, 2003, Chapter 62-620, F.A.C., was revised to adopt, and incorporate by reference, various sections of Title 40 of the Code of Federal Regulations revised as of July 1, 2003, including the revised 40 CFR 136. The revised 40 CFR 136 includes a method for low-level mercury analysis, EPA Method 1631(Revision E), Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry (Method 1631E).

### Who is Required to Use Method 1631E?

Applicants for a wastewater facility permit and wastewater facility permittees are required to use the low-level mercury Method 1631E when reporting results associated with water quality standards (WQSs) below 0.2 micrograms per liter (ug/L). The following facilities are required to use Method 1631E for all **effluent samples**:

- Facilities discharging to Class I and Class II surface waters, including wetlands.
- Facilities discharging to Class III Marine or Fresh surface waters, including wetlands.
- Facilities with Water Quality Based Effluent Limits (WQBELs), or any other limit for mercury specified in a permit, below 0.2 ug/L.

This includes effluent samples collected for any of the following requirements:

- Monitoring specified in Section I, *Reclaimed Water and Effluent Limitations and Monitoring*, section of permits.
- Monitoring performed under Section 3.A. of *Wastewater Permit Application Form 2A For Domestic Wastewater Facilities*; Part VII.C. of *Application to Discharge Process Wastewater from New or Existing Industrial Wastewater Facilities to Surface Water - Form 2CS*; or Part V.C. of *Application to Discharge Process Wastewater from New or Existing Industrial Wastewater Facilities to Ground Water - Form 2CG*.
- Priority pollutant scans performed in accordance with pretreatment program annual report requirements.
- Monitoring performed for the development or re-evaluation of local discharge limitations.
- Monitoring required in Table 4 of the Generic Permit for Discharges from Petroleum Contaminated Sites.

The low-level mercury method provides the ability to assess compliance with mercury water quality standards (WQSs) below 0.2 ug/L. Your permit requires that surface water discharges shall be analyzed using a sufficiently sensitive method in accordance with 40 CFR 136. *Wastewater Permit Application Forms 2A, 2CS, and 2CG* require effluent testing be conducted using methods that are able to detect pollutants at levels adequate to meet WQSs and to provide reasonable assurance that the WQSs will not be violated in the future.

Additionally, in order to develop technically and legally defensible local discharge limitations for domestic wastewater facilities that have pretreatment programs, Method 1631E must be used to provide data that clearly establishes the basis for any calculated mercury limitations. Note, regarding local discharge limitations, the requirement to use Method 1631E may be expanded to other locations in the collection and treatment system on a case-by-case basis depending on the initial results from effluent analysis using Method 1631E.

## **Mercury Laboratory Analysis**

Method 1631E has a minimum level of quantitation of 0.0005 ug/L, or 0.5 nanograms per liter (ng/L), which is 400-times more sensitive than Method 245.1 (“Manual Cold Vapor Technique”). Due to the sensitivity of Method 1631E, the results are typically measured in parts per trillion (ng/L) rather than in parts per billion (µg/L). The Department established a target MDL of 0.2 ng/L and PQL of 0.8 ng/L for EPA method 1631E in the document entitled “FAC 62-4 MDL/PQL Table (April 26, 2006)” which is available at [www.dep.state.fl.us/labs/library/index.htm](http://www.dep.state.fl.us/labs/library/index.htm). All laboratory analysis must be done by a NELAP accredited laboratory with current certification by Florida Department of Health for Method 1631E.

## **Mercury Clean Sampling Techniques**

Clean sample handling techniques should be used when collecting samples for low-level mercury analysis to preclude false positives arising from sample collection, handling, or analysis. Sample collection methods should be consistent with *DEP-SOP-001/01: FS 8200 Clean Sampling For Ultratrace Metals in Surface Waters* and *EPA Method 1669: Sampling Ambient Water for Trace Metals at EPA Water Quality Criteria Levels* (EPA-821-R-96-011). Because FS 8200 and Method 1669 are performance-based procedures, sample collection personnel may modify these procedures or eliminate steps if the modification does not lead to unacceptable contamination of samples or blanks. Any modifications should be thoroughly evaluated and demonstrated to be effective before field samples are collected. This may be accomplished through documentation of uncontaminated samples, equipment blanks and/or other quality control samples.

Note, discrete and composite samplers have been found to contaminate samples with mercury at the ng/L level. Therefore, grab samples are permissible when using Method 1631E. However, grab samples must be representative of the wastewater discharge and a field blank should be collected along with the sample.

In order for a permittee to justify a claim that any reported mercury is due to outside contamination, a blank must have been collected. For this reason, permittees should consider collecting at least one blank at each site for each day a sample is collected. If more than one sample is collected in a day, at least one blank for each 10 samples collected on that day should also be collected. The blank may either be an equipment blank or a field blank. Once a permittee demonstrates the ability to collect samples from a given site using an established procedure that prevents contamination, the permittee may choose to decrease the number of blanks being taken. Specific definitions and procedures for collecting blanks are found in DEP SOP FQ 1000.

Field blanks should be collected only if no equipment other than the sample container is used to collect samples. If the sampling procedure involves the use of additional equipment, such as a peristaltic pump and pump tubing, equipment blanks should be collected. All blanks are subject to the same preservation, digestion, and analysis protocols as regular samples and should have a concentration at least five times lower than the sample concentration. The permittee may not subtract field blank concentrations when reporting sample results.

Sample collection, preservation, and shipping requirements should be discussed with contract laboratories to ensure the requirements of Method 1631E are met.

## **Additional Assistance and Information**

For additional information on Method 1631:  
<https://www.epa.gov/cwa-methods/cwa-methods-regulatory-history>

Please refer questions concerning sample collection to:  
Michael Blizzard: 850-245-8073

[Michael.Blizzard@dep.state.fl.us](mailto:Michael.Blizzard@dep.state.fl.us)

Additional information concerning NELAP certified laboratories can be obtained from:

Department of Health Bureau of Laboratories  
P.O. Box 210 Jacksonville, FL 32231

(904) 791-1599

<https://fldeplac.dep.state.fl.us/aams/>