Quality Control Guidance Information for the sampling and analysis of Low Level Mercury in Water following EPA Method 1631, Revision E August 2002

The following tables summarize **some** of the minimum quality control requirements for the routine sampling and analysis of Low Level Mercury in water following *US EPA Method 1631, Revision E: Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry.* Additional quality control requirements can be found in US EPA Method 1631, Revision E. See EPA 821-R-01-023 Guidance for Implementation and Use of EPA Method 1631 for the Determination of Low-Level Mercury (40 CFR part 136), March 2001, Section 5-18 for further details.

- All data are the responsibility of the discharger/permittee if the data are to be used for permitting or regulatory compliance purposes under a permit.
- It is the responsibility of the discharger/permittee to ensure that all minimum quality control sampling requirements for EPA Method 1631, Revision E are met. (see Table 1 below and EPA Method 1631, Revision E)
- It is the laboratory's responsibility to make sure that all analytical QC acceptance criteria for EPA Method 1631, Revision E are met (see Table 2 below and EPA Method 1631, Revision E).
- Data not meeting the QC requirements of Method 1631, Revision E may not be reported by a discharger/permittee for compliance purposes.
- Additionally, a laboratory cannot be responsible for activities over which it has no control.
- **Blank correction is not allowed except as specified in tables 1 and 2 below and Method 1631, Revision E.**

Table 1: Minimum Quality Control Sampling Requirements per Sampling Event

Test	Definition per Method 1631E	Spike Amount	Minimum Frequency	Acceptance Criteria	Blank Correction Criteria
Bottle Blanks	To determine that the bottle is free from contamination prior to use. Reagent water is added to a bottle, acidified to pH<2 with BrCl, and allowed to stand for a minimum of 24 hours. After standing, the water is analyzed.	NA	At least 5% from a given lot should be tested prior to collection of samples.	<0.5 ng/L	Blank correction of Bottle Blanks is not allowed as the bottles must be demonstrated to be free from contamination prior to use.
Equipment Blanks	Reagent water that has been processed through the sampling device at a laboratory or other equipment cleaning facility prior to shipment of the sampling equipment to the sampling site. The equipment blank is used to demonstrate that the sampling equipment is free from contamination prior to use.	NA	1 following each cleaning	<0.5 ng/L	Blank correction of Equipment Blanks is not allowed as the equipment must be demonstrated to be free of Hg and interferences before equipment may be used in the field
Field Blanks	Reagent water that has been transported to the sampling site and exposed to the same equipment and operations as a sample at the sampling site. The field blank is used to demonstrate that the sample has not been contaminated by the sampling and sample transport systems.	NA	10% from same site at same time	<0.5 ng/L or no greater than one-fifth Hg in associated sample(s), whichever is greater	Must meet acceptance criteria prior to blank correction of laboratory data. Must be reported separately with associated sample(s). Only Field Blanks *or* Method Blanks may be used for blank correction but not both.

Table 2: Minimum Quality Control Requirements per Laboratory Analytical Batch

Test	Definition per Method 1631E	Spike Amount	Minimum Frequency	Acceptance Criteria	Blank Correction Criteria
Bubbler Blanks	The bubbler blank is specific to the bubbler system and is used to determine that the analytical system is free from contamination. A minimum of three bubbler blanks is required for system calibration	NA	Three during calibration if using the Bubbler System	Each bubbler blank must be <0.5 ng/L; Mean of 3 bubbler blanks must be <0.25ng/L with a standard deviation of <0.1ng/L	If the mean of 3 Bubbler Blanks is <0.25ng/L, the average peak height or area is subtracted from all raw data before results are calculated
System Blanks	The system blank is specific to the flow-injection system and is used to determine contamination in the analytical system and in the reagents used to prepare the calibration standards. A minimum of three system blanks is required during system calibration.	NA	Three during calibration if using the Flow-Injection System	Each system blank must be <0.5 ng/L; Mean of 3 system blanks must be <0.5 ng/L with a standard deviation of <0.1ng/L	If the mean of 3 System Blanks is <0.5ng/L, the average peak height or area is subtracted from all raw data before results are calculated
Reagent Blanks	Reagent blanks are used to determine the concentration of mercury in the reagents that are used to prepare and analyze the samples. Reagent blanks are required when each new batch of reagents is prepared	NA	Each new batch of reagents prepared for laboratory analysis	<0.2 ng/L	Blank correction of reagent blanks is not allowed
Method Blanks	Method blanks are used to determine the concentration of mercury in the analytical system during sample preparation and analysis, and consist of a volume of reagent water that is carried through the entire sample preparation and analysis. Method blanks are prepared by placing reagent water in a sample bottle and analyzing the water using reagents and procedures identical to those used to prepare and analyze the corresponding samples	NA	3 per analytical batch	<0.5 ng/L	Must meet acceptance criteria prior to blank correction. Must be reported separately with associated sample(s). Only Method Blanks *or* Field Blanks may be used for blank correction but not both.
Quality Control Sample (QCS)	A sample containing mercury at known concentrations. The QCS is obtained from a source external to the laboratory, or is prepared from a source of standards different from the source of calibration standards. It is used as an independent check of instrument calibration	Within calibration range	1 per analytical batch	% Recovery - follow the specifications provided by the supplier of the standard	NA
Ongoing Precision and Recover (OPR)	To demonstrate that the analytical system is within the performance criteria of this Method and that acceptable precision and recovery is being maintained with each analytical batch	5 ng/L	Prior to and after analysis of each analytical batch	% Recovery: 77-123%	NA
Matrix Spike/Matrix Spike Duplicate (MS/MSD)	Aliquots of an environmental sample to which known quantities of the analyte(s) of interest is added in the laboratory. The MS and MSD are analyzed exactly like a sample. Their purpose is to quantify the bias and precision caused by the sample matrix	Compliance limit or 1-5x background level of the sample used for the MS/MSD, whichever is greater	10% from a given sampling site or discharge	% Recovery: 71-125% Relative % Difference Maximum: 24%	NA

NA= Not Applicable