

Data Acceptability Criteria for Methyl Mercury in Tissue and Sediment

Sample Type	Objective	Frequency of Analysis	Recommended Control Limits	Recommended Corrective Action
<p>External Calibration Calibration Standards (3-5 standards over the expected range of sample target analyte conc., with the lowest conc. Std at or near the MDL).</p>	Full calibration: Establish relationship between instrument response and target analyte conc.	Follow manufacturer's or procedures in specific analytical protocols. A min., 3 point calib. Each set up, major disruption, and when routine calib check exceeds specific control limits.	Linear regression, $r > 0.995$	Determine cause and take appropriate corrective action. Recalibrate and reanalyze all suspect samples or flag all suspect data.
<p>Calibration Verification Calibration Check Standards (minimum of one mid-range standard prepared independently from initial calibration standards: an instrument internal standard must be added to each calib. check std. when internal std. calib. is being used).</p>	Verify calibration.	After initial calibration or recalibration. Every 10 samples.	%R = 80-120%	Determine cause and take appropriate corrective action. Recalibrate and reanalyze all suspect samples or flag all suspect data.
<p>Method Detection Limit Determination Spiked matrix samples (analyte-free tissue or sediment samples to which known amounts of target analytes have been added; one spike for each target analyte at 3-10 times the estimated MDL).</p>	Establish or confirm MDL for analyte of interest.	Seven replicate analyses prior to use of method. Reevaluation of MDL annually.	Determined by program manager	Redetermine MDL.
<p>Accuracy and Precision Assessment Reference materials (SRMs or CRMs, prepared from actual contaminated fish or shellfish tissue and sediments if possible, covering the range of expected target analyte conc.). The actual reference material used for MMHg must be approved by SWAMP QA Program, as some have been proven unstable for MMHg (SRM 1944 for example).</p>	Assess method performance (initial method validation and routine accuracy assessment).	Method validation: As many as required to assess accuracy and precision of method before routine analysis of samples. Routine accuracy assessment: one (preferably blind) per 20 samples or one batch.	Method validation and Routine accuracy assessment: %R = 70-130%	If matrix spikes are in control then proceed. If not, determine cause and take appropriate corrective action. Recalibrate and reanalyze all suspect samples or flag all suspect data.
<p>Matrix spikes (tissue and sediment homogenates of field samples to which known amounts of target analytes have been added: 5 times the concentration of the analyte of interest or 10 times the MDL).</p>	Assess matrix effects and accuracy (%Recovery) routinely.	One per 20 samples or one per batch, whichever is more frequent.	%R = 70-130%	If SRMs are in control then proceed. If not, determine cause and take appropriate corrective action. Recalibrate and reanalyze all suspect samples or flag all suspect data. Zero percent recovery requires rejection of all suspect data.
<p>Matrix spike replicates (replicate aliquots of matrix spike samples; 5 times the concentration of the analyte of interest or 10 times the MDL).</p>	Assess method precision routinely.	One duplicate per 20 samples or one per batch, whichever is more frequent.	RPD <25%	Determine cause and take appropriate corrective action. Recalibrate and reanalyze all suspect samples or flag all suspect data.
<p>Field Replicate (replicate aliquots of tissue and sediment field samples).</p>	Assess method precision routinely. Assess total variability (i.e., population variability, field or sampling variability, and analytical method variability).	One field duplicate sample per 20 samples or one per batch, whichever is more frequent.	RPD <25% for duplicates.	Determine cause and take appropriate corrective action. Recalibrate and reanalyze all suspect samples or flag all suspect data.

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Contamination Assessment Laboratory Blanks (method, processing, bottle, reagent).	Assess contamination from equipment, reagents, etc.	Three method blanks per 20 samples or one per batch, whichever is more frequent. At least one bottle blank per batch. One reagent blank prior to use of a new batch of reagent and whenever method blank exceeds control limits.	Blanks < MDL	Determine cause of problem (e.g., contaminated reagents, equipment), remove sources of contamination, and reanalyze all suspect samples or flag all suspect data.
Field Blanks, Travel Blanks, Equipment Blanks.	Assess contamination from equipment, from air, from surrounding environment, etc.	Random performance evaluation conducted during periodic field audits, in which field blanks demonstrate contamination < MDL. If acceptable performance, no further field blanks required until next field audit. If non-acceptable, 5% field blanks must be conducted until next field audit.	Blanks < MDL	Determine cause of problem (e.g., contaminated preservatives, equipment contamination, improper cleaning, exposure to airborne contaminants, etc.), remove sources of contamination, and reanalyze all suspect samples or flag all suspect data.
External QA Assessment Accuracy-based performance evaluation samples submitted to new laboratories by SWAMP QA Program.	Initial demonstration of laboratory capability.	Once prior to routine analysis of field samples.	Determined by study manager.	Determine cause of problem and reanalyze sample. Do not begin analysis of field samples until laboratory initial capability is clearly demonstrated.
Mandatory interlaboratory exercises overseen by 3rd party external ("referee") SWAMP QA Program officials for all SWAMP participant laboratories.	Ongoing demonstration of laboratory capability.	One exercise per year.	Determined by study manager.	Determine cause of problem and reanalyze sample. Further corrective action to be determined by QA manager.
Voluntary, but encouraged, participation in NOAA-NIST intercalibration studies and CA-ELAP annual performance evaluations, as appropriate.	Ongoing demonstration of laboratory capability.	One exercise per year.	Determined by study manager.	Determine cause of problem and reanalyze sample. Further corrective action to be determined by QA manager.
General Provisions For a Data Set to be considered acceptable the CCV Recoveries must be within control limits and either the SRM or Spiked Matrix recoveries must also be within control limits				