WSC – CAM – III B

Quality Control Requirements and Performance Standards for the ***Analysis of Mercury by Cold Vapor Atomic Absorption (CVAA) Spectrometry*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

III. Metals Methods

B. Quality Control Requirements and Performance Standards for WSC-CAM-III B (Mercury by CVAA)

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ACRONYM LIST

CAM	Compendium of Analytical Methods
CASN	Chemical Abstracts Service Number
CCB	Continuing calibration blank
CCV	Continuing calibration verification
COC	Chain-of-custody
CVAA	Cold vapor atomic absorption spectrometry
DF	Dilution factor
HNO_3	Nitric acid
ICB	Initial calibration blank
ICV	Initial calibration verification
IRAs	Immediate Response Actions
LCS/LCSD	Laboratory control sample / Laboratory control sample duplicate
LLCV	Low-level calibration verification
LLOQ	Lower limit of quantitation
MassDEP	Massachusetts Department of Environmental Protection
MB	Method blank
MCP	Massachusetts Contingency Plan
MD	Matrix duplicate
MOHML	Massachusetts Oil and Hazardous Materials List
MS	Matrix spike
QA	Quality assurance
QC	Quality control
r	Correlation coefficient
RCs	Reportable Concentrations
RL	Reporting limit
RPD	Relative percent difference
RQs	Reportable Quantities
SRM	Standard reference material
USEPA	United States Environmental Protection Agency

UNITS:

mg/kg	Milligram per kilogram
mL	Milliliter
µg/L	Microgram per liter
µm	Micron

1.0 Quality Control Requirements and Performance Standards for WSC-CAM-III B

1.1 Overview of WSC-CAM-III B

WSC-CAM-III B, *Quality Control Requirements and Performance Standards for the Analysis of Mercury by Cold Vapor Atomic Absorption (CVAA) Spectrometry in Support of Response Actions under the Massachusetts Contingency Plan (MCP)*, is a component of MassDEP's Compendium of Analytical Methods (CAM). Effective February 15, 2024, this revised CAM protocol, WSC-CAM-III B, replaces the previous version of the Mercury CAM document, WSC-CAM-III B (effective date, July 1, 2010). Refer to WSC- CAM-I A for an overview of the CAM process. Please note that while this protocol must be followed on and after the effective date of February 15, 2024 for the purpose of "Presumptive Certainty," the revised protocol may be used optionally prior to its effective date upon its publication on November 15, 2023.

This document provides Quality Control (QC) requirements and performance standards to be used in conjunction with the required analytical methods SW-846 7470A & 7471B (or the most current versions), analysis for mercury in aqueous and solid samples using CVAA spectrometry. The QC requirements and performance standards specified in this document in Table III B-1 together with the analytical procedures described in EPA SW-846 Methods 7470A *Mercury in Liquid Waste (Manual Cold-Vapor Technique)* & 7471B, *Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)* constitute the WSC-CAM-III B protocol. All protocols included in the CAM are considered "methods" published by the MassDEP pursuant to the provisions of 310 CMR 40.0017(2). Use of EPA SW-846 7470A & 7471B is a "Presumptive Certainty" requirement of WSC-CAM-III B. It should be noted that if the laboratory utilizes the analytical procedures in SW-846 Method 7471A instead of 7471B, it is NOT acceptable to answer "YES" to Question B on the MassDEP Analytical Protocol Certification Form since there are analytical procedural differences between 7471A and 7471B (i.e., 7471B requires the use of a well-homogenized 0.6 gram aliquot instead of three 0.2 gram aliquots).

Sample preservation, container and analytical holding time specifications for aqueous, soil, and sediment matrices for mercury analyzed in support of MCP decision-making are presented in Appendix III B-1 of this document and Appendix VII-A of WSC-CAM-VII A *Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)*. Data reporting requirements are also provided in WSC-CAM-VII A.

Overall usability of data produced using this CAM protocol should be evaluated for compliance with project-specific data quality objectives, regardless of "Presumptive Certainty" status. For more guidance on data usability, refer to MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

1.1.1 Reporting Limits or Lower Limits of Quantitation for Mercury by WSC-CAM-III B

The reporting limit (RL) or lower limit of quantitation (LLOQ) for mercury using WSC-CAM-III B is dependent on the concentration of the lowest non-zero standard in the initial calibration or low-level calibration verification (LLCV), analyzed under identical conditions as the sample, with adjustments made for the sample size, preparation factors, percent solids, dilution factors, etc., as required. The CAM RLs/LLOQs for mercury using the WSC-CAM-III B protocol are:

- 0.2 µg/L for aqueous samples (surface water, groundwater and drinking water); and
- 0.1 mg/kg for soil/sediment samples (assuming 100% solids).

For "Presumptive Certainty" purposes, if the typical CAM RLs/LLOQs are not achieved, respond "NO" to Question G of the "MassDEP MCP Analytical Protocol Certification Form" and address the CAM RL/LLOQ exceedance in the laboratory narrative.

RLs/LLOQs lower than the above referenced CAM RLs/LLOQs for mercury may be required to satisfy project requirements. The RL/LLOQ (based on the concentration of the lowest calibration standard or the LLCV) for mercury must be less than or equal to the MCP standards or criteria that the contaminant concentrations are being compared to (e.g., Method 1 Standards, benchmark values, background, etc.). Meeting MCP standards or criteria may require analytical modifications to improve sensitivity or the use of a different analytical method. All such modifications must be described in the laboratory narrative. RLs/LLOQs for mercury will be proportionately higher for samples that require dilution, when a reduced sample size is used, or when the sample has a relatively high percent moisture (low percent solids).

1.1.2 Initial Demonstration of Proficiency for WSC-CAM-III B

Each laboratory that uses the WSC-CAM-III B protocol is required to operate a formal quality assurance (QA) program. The minimum requirements of this program consist of an initial demonstration of laboratory proficiency, ongoing analysis of standards and blanks to confirm acceptable continuing performance, the preparation/analysis of laboratory control samples (LCS) and LCS duplicates (LCSDs) to assess analytical accuracy and precision. Matrix spikes (MS) or matrix duplicates (MD) may also be used to evaluate accuracy and/or precision when such samples are analyzed either at the discretion of the laboratory or at the request of the data user.

Laboratories must document and have on file an Initial Demonstration of Proficiency for each combination of sample preparation and determinative method being used. These data must meet or exceed the performance standards as presented in Table III B-1 of this protocol. The data associated with the Initial Demonstration of Proficiency must be kept on file at the laboratory and made available to potential data users on request. The data associated with the Initial Demonstration of Proficiency for WSC-CAM-III B must include the following:

QC Element	Performance Criteria
Initial Calibration	See WSC-CAM-III B, Table III B-1
Continuing Calibration Verification	
Method Blanks	
Percent Recovery for LCS & MS	
Relative Percent Difference (RPD) for LCSD or MD	

Laboratories are encouraged to actively monitor pertinent QC performance standards described in Table III B-1 to assess analytical trends (i.e., systematic bias, etc.) and improve overall method performance by preempting potential non-conformances.

For the WSC-CAM-III B protocol, laboratory-specific control limits must meet or exceed (demonstrate less variability than) the performance standards for each QC element listed in Table III B-1. It should be noted that the performance standards listed in Table III B-1 are based on multiple-laboratory data, which are in most cases expected to demonstrate more variability than performance standards developed by a single laboratory.

This protocol is restricted to use by, or under the supervision of, analysts who are experienced in the use of CVAA spectrometry as a quantitative tool for environmental analyses.

1.2 Summary of SW-846 Methods 7470A and 7471B

SW-846 Method 7470A is a CVAA procedure for determining the concentration of total mercury (organic and inorganic forms) in mobility-procedure extracts, aqueous wastes, surface water and groundwater. SW-846 Method 7471B is a CVAA procedure for determining total mercury (organic and inorganic forms) in soils, sediments, bottom deposits, and sludge-type materials. Prior to analysis, the liquid samples must be pretreated according to the procedure described in Section 7.0 of SW-846 Method 7470A and solid samples are prepared using an appropriate dissolution step, as described in Section 11.0 of SW-846 Method 7471B.

Quantitation is based on the absorption of radiation at the 253.7-nm wavelength by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

1.3 Method Interferences

Samples submitted to a laboratory for trace metal analysis may become contaminated by numerous routes during both sampling and analysis. Potential sources of contamination may include:

- Metallic or metal-containing containers and sampling equipment,
- Laboratory acids or reagents,
- Improperly cleaned or stored equipment, and
- Atmospheric inputs such as dirt and dust.

Refer to SW-846 Methods 7470A and 7471B for further information on method interferences and contamination. A summary of common interferences and corrective measures is provided below.

- Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/kg of sulfide, as sodium sulfide, do not interfere with the recovery of inorganic mercury added to reagent water.
- Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm, thereby giving a potential high bias for mercury. An excess of hydroxylamine sulfate reagent (25 mL) can be added to ensure that free chlorine is absent before the mercury is reduced and swept into the cell.

1.4 Quality Control Requirements and Performance Standards for WSC-CAM-III B

Specific QC requirements and performance standards for mercury using the WSC-CAM-III B protocol are presented in Table III B-1. Refer to WSC-CAM-VII A for field QC requirements. ***Note that a project-specific matrix spike (MS) must be performed for mercury to evaluate accuracy in a solid matrix (soil/sediment) at a frequency of one per 20 samples per matrix***. Strict compliance with the QC requirements and performance standards, as well as satisfying the CAM's other analytical and reporting requirements will provide a data user with "Presumptive Certainty" in support of Response Actions under the MCP. The concept of "Presumptive Certainty" is explained in detail in Section 2.0 of WSC-CAM-VII A.

While optional, parties electing to utilize these protocols will be assured of "Presumptive Certainty" of data acceptance by agency reviewers. In order to achieve "Presumptive Certainty" for analytical data, parties must:

(a) Use the analytical method specified for the selected CAM protocol;
(b) Incorporate **all** required analytical QC elements specified for the selected CAM protocol;
(c) Implement, as necessary, required corrective actions and analytical response actions for **all** non-conforming analytical performance standards;
(d) Evaluate and narrate, as necessary, **all** identified CAM protocol non-compliances; and
(e) Comply with **all** the reporting requirements specified in WSC-CAM-VII A, including retention of reported and unreported analytical data and information for a period of ten (10) years.

In achieving "Presumptive Certainty" status, parties will be assured that analytical data sets:

- ✓ Satisfy the broad QA/QC requirements of 310 CMR 40.0017 and 40.0191 regarding the scientific defensibility, precision and accuracy, and reporting of analytical data; and

- ✓ May be used in a data usability and representativeness assessment, as required in 310 CMR 40.1056(2)(k) and 40.1057(2)(k) for Permanent and Temporary Solution submittals, respectively, consistent with the guidance described in MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

1.5 Special Analytical Considerations for WSC-CAM-III B

The following bullets highlight potential issues that may be encountered with the analysis of mercury using this protocol.

- Matrix Spike (MS) Recovery – ***A MS is required for WSC-CAM-III B for solid matrices (soil/sediment) at a frequency of one per 20 samples per matrix***. Consistent with the United States Environmental Protection Agency (USEPA) Region I data validation guidance, MassDEP requires rejection of non-detected metals results with <30% recovery in the MS if the concentration of the metal in the unspiked sample is <4x the amount spiked.. If the MS recovery is <30% and non-detected results were reported for mercury, the laboratory must follow the required corrective actions listed on Table III B-1.
    - Laboratories are not required to monitor whether or not MSs are performed on soil/sediment samples at a frequency of one per 20 samples per matrix. This is the responsibility of the data user.

  - For "Presumptive Certainty" purposes, if the data user does not submit a soil/sediment sample for MS analysis, Question H of the "MassDEP MCP Analytical Protocol Certification Form" must be answered NO and this must be noted in the laboratory narrative.
- Soils/Sediments – Because of the small sample size generally used for solid sample analyses (0.6-grams), "as-received" field samples of solid or semisolid materials must be thoroughly homogenized in the laboratory prior to mercury analysis to obtain a representative aliquot for analysis. If mercury is a contaminant of concern at an MCP site or if poor precision and/or accuracy (that would adversely affect MCP decision making) associated with sample matrix heterogeneity is anticipated, then the following corrective measures should be considered by the data user:
  - Direct the laboratory to use a larger sample aliquot (up to 10 grams) to enhance sensitivity and/or precision. The laboratory must adjust the concentration/volume of all reagents for the larger sample aliquot.
  - Direct the laboratory to prepare and analyze replicate aliquots (two or more) for each field sample to better assess variability, associated with sample matrix heterogeneity.
  - Use more effective field sample homogenization procedures (prior to submitting sample to laboratory for mercury analysis).
  - Increase frequency of field duplicate collection and analysis to improve understanding of heterogeneity and representativeness.
  - If overall site heterogeneity for mercury is a specific concern (i.e., distribution of mercury contamination is inconsistent with the conceptual site model), it is recommended that the number of field samples be increased to improve the representativeness of mercury results for site assessment and characterization.
- Appendix III B-3 provides a typical analysis sequence for mercury analyzed using this CAM protocol.

1.6 Analyte List for WSC-CAM-III B

The MCP analyte list for WSC-CAM-III B consists of Mercury, total (organic and inorganic), Chemical Abstracts Service Number (CASN) 7439-97-9.

It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analyte. Sources of various MassDEP standards and criteria are as follows:

- Reportable Quantities (RQs) and Reportable Concentrations (RCs) as described in 310 CMR 40.1600, The Massachusetts Oil and Hazardous Materials List (MOHML), in Subpart P of the MCP may be found at the following URL: http://www.mass.gov/dep/cleanup/laws/regulati.htm#mcp
- An online searchable Oil & Hazardous Materials List of RQs and RCs values may be found at the following URL: http://eeaonline.eea.state.ma.us/DEP/MOMHL/hazmat.aspx
- An updated list of MCP Method 1 Standards may be found at the following URL: https://www.mass.gov/regulations/310-CMR-4000-massachusetts-contingency-plan

Mercury has promulgated MCP Method 1 groundwater/soil standards.

Table III B-1: Specific QC Requirements and Performance Standards for Mercury (SW-846 7470A & 7471B) Using WSC-CAM-III B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must follow procedures in Section 9.2 of SW-846 7471B and Section 9.4 of SW-846 7000B.	No	NA	Refer to Section 9.2 of SW-846 7471B, Section 9.4 of SW-846 7000B, and Section 1.1.2 of this protocol.	NA
Preparation of Samples	Accuracy and Representativeness	(1) All aqueous and solid samples must be prepared (digested) prior to analysis. See SW-846 7470A and 7471B for details.	No	NA	NA	NA
Initial Calibration	Laboratory Analytical Accuracy	(1) Daily prior to sample analysis. (2) Minimum calibration blank plus 5 calibration standards (multi-point); high level standard in calibration defines the upper end of the linear calibration range. (3) Linear regression with correlation coefficient r $\geq$0.995.	No	NA	Perform instrument maintenance as necessary; re-optimize instrument; re-calibrate as required by SW-846 7470A and 7471B.	Suspend all analyses until initial calibration meets criteria.
Initial Calibration Verification (ICV)	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Prepared using standard source different than used for initial calibration. (3) Concentration level near midpoint of curve. (4) Percent recovery must be between 90-110%.	No	NA	(1) Reanalyze ICV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV.	Suspend all analyses until ICV meets criteria.
Initial Calibration Blank (ICB)	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Immediately after ICV. (2) Prepared using same concentration of acids as calibration standards. (3) Mercury must be <RL/LLOQ.	No	NA	(1) Reanalyze ICB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV & ICB.	Suspend all analyses until ICB meets criteria.
Low-Level Calibration Verification (LLCV) Laboratory Analytical Sensitivity (verify low-end of calibration range / verify RL/LLOQ)		(1) Daily prior to sample analysis if initial calibration did not contain a low- level standard at the RL/LLOQ. If initial calibration includes the RL/LLOQ as the low-level standard in the initial calibration curve, then LLCV is not	No	NA	(1) Reanalyze LLCV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and concentrations of mercury are $\leq$10x RL/LLOQ in associated field samples,	Suspend all analyses until LLCV meets criteria unless the concentrations of mercury are >10x RL/LLOQ in the associated field samples.

Table III B-1: Specific QC Requirements and Performance Standards for Mercury (SW-846 7470A & 7471B) Using WSC-CAM-III B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		required. (2) Prepared using same source as initial calibration standards. (3) Concentration level must be at the level of the RL/LLOQ for mercury. (4) Percent recovery must be 70-130%.			recalibrate and reanalyze LLCV and associated samples. (3) If concentrations of mercury are >10x RL/LLOQ in associated field samples, include explanation in laboratory narrative; no further action required.	
Continuing Calibration Verification (CCV)	Laboratory Analytical Accuracy	(1) Every 10 samples and at the end of the analytical run. (2) Prepared using same source as initial calibration standards. (3) Concentration level near midpoint of curve. (4) Percent recovery must be 80-120%.	No	NA	(1) Reanalyze CCV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCV – unless (3) applies. (3) If recovery is high (>120%) and all associated sample results are not-detected, no corrective action required.	If (3) applies, include explanation in laboratory narrative.
Continuing Calibration Blank (CCB)	Laboratory Analytical Sensitivity (instrument drift & contamination)	(1) Every 10 samples following CCV and at the end of the analytical run. (2) Prepared using same concentration of acids as calibration standards. (3) Mercury must be <RL/LLOQ.	No	NA	(1) Reanalyze CCB; if acceptable, no further action required. (2) If reanalysis still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCB – unless (3) applies. (3) If concentration of mercury in CCB is >RL/LLOQ but all associated sample results are either not detected or >10x concentration of mercury in CCB, no corrective action required.	If (3) applies, include explanation in laboratory narrative.
Method Blank (MB)	Laboratory Method Sensitivity (contamination evaluation)	(1) One per digestion batch of ≤20 field samples. (2) Must be digested with the samples using the same preparation method as the samples.	Yes	NA	(1) Reanalyze MB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, redigest and reanalyze MB and all associated field samples in batch – unless (3)	If (3) applies, include explanation in laboratory narrative.

Table III B-1: Specific QC Requirements and Performance Standards for Mercury (SW-846 7470A & 7471B) Using WSC-CAM-III B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(3) Mercury must be <RL/LLOQ.			applies. (3) If concentration of mercury in MB is >RL/LLOQ but all associated sample results are either not detected or >10x concentration of mercury in MB, no corrective action required.	
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) One per digestion batch of ≤20 field samples. (2) Must be matrix-matched by digesting with the samples using the same preparation method. CAM requires a solid Standard Reference Material (SRM) be prepared and analyzed with solid field samples as the "solid LCS." An SRM is a soil or sediment matrix that contains mercury at a known concentration and with 95% confidence limits. (3) Concentration level for aqueous LCS near midpoint of curve. (4) Percent recovery for mercury must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS.	Yes	Aqueous LCS: Recovery <50%: mercury results in associated samples may be rejected.	(1) Reanalyze LCS; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and LCSD is in-control for mercury, no corrective action required. (3) If LCS and LCSD are both outside of criteria, redigest and reanalyze LCS/LCSD and all associated field samples in batch.	Report recovery exceedances in laboratory narrative.
LCS Duplicate (LCSD)	Laboratory Analytical Accuracy & Precision	(1) One per digestion batch of ≤20 field samples ONLY if not performing project-specific MD. (2) Must be matrix-matched by digesting with the samples using the same preparation method. CAM requires a solid SRM be prepared and analyzed with solid field samples as the "solid LCSD." An SRM is a soil or sediment matrix that contains mercury at a known concentration and with 95% confidence limits. (3) Concentration level must be same as LCS. Analyze immediately following LCS. (4) Percent recovery for mercury must be	Yes ONLY if no MD	Same as above for LCS for recovery evaluation	(1) Reanalyze LCSD; if acceptable, no further action required. (2) If reanalysis is still outside of recovery criteria and LCS is in-control for mercury, no corrective action required. (3) If LCSD and LCS are both outside of recovery criteria, redigest and reanalyze LCS/LCSD and all associated field samples in batch.	Report recovery and RPD exceedances in laboratory narrative.

Table III B-1: Specific QC Requirements and Performance Standards for Mercury (SW-846 7470A & 7471B) Using WSC-CAM-III B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS. (5) RPDs must be ≤20 for aqueous LCS/LCSD and ≤30 for solid LCS/LCSD.				
Matrix Spike (MS) Project-Specific	Method Accuracy in Sample Matrix	(1) Solid Samples (Soil/Sediment): One per 20 field samples per matrix; designated by data user on chain-of-custody (COC) or at project set-up. Aqueous Samples :One per digestion batch of ≤20 field samples per matrix strongly recommended (designated by data user on COC or at project set-up). (2) Concentration levels near midpoint of curve. (3) Percent recovery for mercury must be 75-125%.	Yes ONLY when requested by the data user	Recovery <30%: affects non-detects for mercury in all associated samples.	(1) Reanalyze MS; if acceptable, no further action required. (2) After reanalysis, if MS recovery is 30-74% or >125% and LCS was in- control, no corrective action is required. (3) If MS recovery is <30% and associated with non-detected results, redigest (homogenize sample well) and reanalyze sample/MS pair. Report results and narrate.	Report MS exceedances in laboratory narrative. If redigested due to recoveries <30%, report both sets of sample/MS data.
Matrix Duplicate (MD) Project-Specific	Method Precision in Sample Matrix	(1) One per digestion batch of ≤20 field samples per matrix is strongly recommended (designated by data user on COC or at project set-up). (2) Prepare by digesting and analyzing an additional aliquot of the same field sample used for MS. (3) RPD for mercury must be <20 for aqueous and <35 for solids.	Yes ONLY when requested by the data user	NA	Narrate.	Report exceedances in laboratory narrative.
General Reporting Issues	NA	(1) Non-detected values must be reported with the sample-specific RL/LLOQ for mercury using all preparation/dilution factors. (2) The laboratory must only report values ≥ the sample-specific RL/LLOQ; optionally, values below the sample-specific RL/LLOQ can be reported as estimated, if requested. The laboratory must report results for samples and blanks in a consistent manner.	NA	NA	NA	(1) Qualification of the data is required if reporting values below the sample-specific RL/LLOQ. (2) Complete analytical documentation for diluted and undiluted analyses must be made available for review during an audit. (3) The performance of dilutions must be

Table III B-1: Specific QC Requirements and Performance Standards for Mercury (SW-846 7470A & 7471B) Using WSC-CAM-III B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[1]	Required Corrective Action	Required Analytical Response Action
		(3) Sample concentrations that exceed the highest calibration standard must be diluted and reanalyzed to fall within the linear calibration range. (4) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards. (5) For aqueous samples, the laboratory must note whether the results are "total" or "dissolved" in the laboratory narrative or on the report form. In addition, if "dissolved", the laboratory must note whether the samples were filtered in the field or at the laboratory. (6) Results must be reported with 2 or more "significant figures" if ≥RL/LLOQ. If reporting values below the RL/LLOQ, report with 1 or more "significant figures".[2] (7) Refer to Appendix III B-1 for COC requirements regarding preservation, cooler temperature, and holding times.				documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of mercury, reasons for dilutions must be explained in the laboratory narrative. (4) If samples are not preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory narrative. (5) If samples are prepared and/or analyzed outside of the holding time, note the non-conformances in the laboratory narrative. (6) Narrate any additional method non-compliance or sample-specific anomaly.

[1]As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, if these results are observed, data users should consider nondetect results as unusable and detected results as estimated with a significant low bias.

[2]Reporting protocol for "significant figures" is a policy decision included for standardization and consistency for reporting of results and is not a definition of "significant" in the scientific or mathematical sense.

2.0 Data Usability Assessment

Specific guidance applicable to all Permanent and Temporary Solutions, including Permanent and Temporary Solutions on a portion of a disposal site, for preparation of Representativeness Evaluations and Data Usability Assessments pursuant to 310 CMR 40.1056(2)(k) and 40.1057(2)(k), respectively, of the MCP is provided in *MCP Representativeness Evaluations and Data Usability Assessments* (Policy #WSC-07-350). This document provides general information regarding the purpose and content of these required evaluations as a component of and in support of a Permanent or Temporary Solution submittal. The most current version of this document may be found at the following URL: http://www.mass.gov/dep/cleanup/laws/policies.htm#finpol.

Overall usability of data produced using this CAM protocol should be evaluated for compliance with project-specific data objectives using MassDEP Policy #WSC-07-350, regardless of "Presumptive Certainty" status.

3.0 Reporting Requirements for WSC-CAM-III B

3.1 General Reporting Requirements for WSC-CAM-III B

General environmental laboratory reporting requirements for analytical data used in support of assessment and evaluation decisions at MCP disposal sites are presented in WSC-CAM-VII A, Section 2.4. This guidance document provides limited recommendations for field QC, as well as the required content of the laboratory report, which includes:

- Laboratory identification information,
- Analytical results and supporting information,
- Sample- and batch-specific QC information,
- Laboratory Report Certification Statement,
- Copy of the Analytical Protocol Certification Form,
- Laboratory narrative contents, and
- Chain-of-custody form requirements.

3.2 Specific Reporting Requirements for WSC-CAM-III B

Specific QC requirements and performance standards for WSC-CAM-III B are presented in Table III B-1. Specific reporting requirements for WSC-CAM-III B are summarized below in Table III B-2 as "Required Analytical Deliverables (**YES**)". Requirements listed as "YES" must always be included as part of the laboratory deliverable for this method. It should be noted that data for those items listed as "NO" under "Required Analytical Deliverables" must be available for review during an audit and may also be requested for inclusion in the analytical deliverable on a client-specific basis.

Soil and sediment results must be reported on a dry-weight basis. Refer to ASTM Method D2216, Determination of Moisture Content of Soils and Sediments, for more detailed analytical and equipment specifications.

Table II B-2 Routine Reporting Requirements for WSC-CAM-III B (SW-846 7470A/7471B)

Parameter	Required Analytical Deliverable
Initial Calibration	***NO***
Initial Calibration Verification (ICV)	***NO***
Initial Calibration Blank (ICB)	***NO***
Low-Level Calibration Verification (LLCV)	***NO***
Continuing Calibration Verification (CCV)	***NO***
Continuing Calibration Blank (CCB)	***NO***
Method Blank (MB)	***YES***
Laboratory Control Sample (LCS)	***YES***
LCS Duplicate	***YES (if no MD)***
Matrix Spike (MS)	***YES*** *(if requested by data user)*
Matrix Duplicate (MD)	***YES*** *(if requested by data user)*
Identification and Quantitation	***NO***
General Reporting Issues	***YES***

3.2.1 Sample Dilution

Under circumstances that sample dilution is required because the concentration of mercury exceeds the concentration of the highest calibration standard or due to matrix interference, the RL/LLOQ for mercury must be adjusted (increased) in direct proportion to the Dilution Factor (DF).

The revised RL/LLOQ for the diluted sample, $RL/LLOQ_d$:

$$RL/LLOQ_d = DF \times \text{Lowest Calibration Standard for Mercury}$$

It should be understood that samples with elevated RLs/LLOQs as a result of a dilution may not be able to satisfy MCP standards/criteria in some cases if the $RL/LLOQ_d$ is greater than the applicable MCP standard or criterion to which the concentration is being compared. Such increases in RLs/LLOQs are the unavoidable but acceptable consequence of sample dilution that enable quantification of target analytes which exceed the linear range. All dilutions must be fully documented in the laboratory narrative.

NOTE: **Over dilution is an unacceptable laboratory practice.** The post-dilution concentration of the target analyte must be detected within the calibration range.

Appendix III B-1

Sample Collection, Preservation, and Handling Procedures for Mercury Analyses

Sample preservation, container, and analytical holding time specifications for aqueous, soil, and sediment matrices for mercury analyzed in support of MCP decision-making are summarized below and presented in Appendix VII A-1 of WSC-CAM-VII A, *Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data Conducted in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)*. Additional guidance may be found in SW-846, Chapter Three.

Matrix	Container[1]	Preservation[5]	Holding Time[2]
Aqueous Total Mercury	500 mL glass or Polyethylene Bottle	HNO_3 to pH <2	28 days
Aqueous Dissolved Mercury (Filtered)	500 mL glass or Polyethylene Bottle	Filter (0.45 µm) on site or at the laboratory (***prior to acid preservation)*** within 24 hours of collection; then preserve with HNO_3 to pH <2[3]	28 days
Soil and Sediment	4-ounce glass jar with teflon-lined cap	Cool to ≤ 6ºC	28 days[4]
Concentrated Waste	125 mL wide-mouth glass or polyethylene bottle	Cool to ≤ 6ºC	28 days

[1]The collection of multiple sample containers per sample location may be required to collect enough sample for matrix QC. It is also acceptable to use smaller containers to reduce waste and as consistent with laboratory procedures.

[2]Holding Time begins from time of sample collection or date thawed (see note #4 below). As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, if the holding time is exceeded by >2x, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias.

[3]If samples are filtered and preserved at the laboratory, the laboratory must wait 24 hours prior to analysis to allow enough time for metals to become solubilized.

[4]Alternatively, soil and sediment samples for mercury analyses may be held for up to one (1) year if frozen within 24 hours of collection at <-10°C. Sampling container should only be filled to 2/3 of capacity to avoid breakage caused by expansion during freezing. Temperature must never be allowed to go below -20°C to avoid damage to seals, etc. Preparation must be commenced within 28 days of thawing. Once the thawing process begins, samples must be kept at 0-6◦C until preparation.

[5]If samples were received by the laboratory on the same day of collection and were stored and transported to the laboratory on ice, cooler temperatures above 6ºC are acceptable.

Appendix III B-2

Data Deliverable Requirements for Data Audits

If requested by MassDEP, submission of the information listed below may be required to perform a data audit to verify compliance with the analytical methods and to evaluate accuracy and reliability of the reported results. These deliverables represent a "full data package" including all sample documentation from receipt through preparation, analysis, and data reporting. The laboratory must ensure that these deliverables are available, in the event a data audit is performed. The laboratory is required to retain these deliverables for a period of 10 years from the date generated.

DELIVERABLE REQUIREMENTS FOR DATA AUDITS WSC-CAM-III B (Mercury by CVAA: SW-846 7470A/7471B)	
Laboratory Narrative	Must comply with the required laboratory narrative contents as described in WSC-CAM-VII A
Sample Handling Information	Chain-of-custody (external and internal), sample receipt logs (cooler temperatures and sample pH), correspondences
Miscellaneous Logs	Dry weight logs; Analytical logs; Freezer logs; Sample preparation logs (initial and final weights/volumes; preparation method reference); Filtration logs (if applicable)
Initial Calibration Data	Raw instrument data for initial calibration, including calculation of linear regression and correlation coefficients; Concentrations of calibration standards used must be clearly presented.
Initial Calibration Verification and Initial Calibration Blank Data	Percent recoveries for all ICVs; ICV source & true value; Results and raw instrument data for ICV and ICB
Low-Level Calibration Verification, Continuing Calibration Verification, and Continuing Calibration Blank Data	Percent recoveries for all LLCV and CCV; LLCV and CCV source & true value; Results and raw instrument data for LLCV, CCV, and CCB
Sample Results	Sample result forms with dilution factors, units, RLs/LLOQs, method reference, date of preparation, date of analysis; raw instrument data; percent solids results
Method Blank Results	Method Blank results, units, RLs/LLOQs; raw instrument data
LCS/LCS Duplicate Results and/or SRM results	Summary of results, including concentrations detected, concentrations spiked or known (vendor limits) if SRM, percent recoveries and RPDs; raw instrument data
MS Results – if analyzed **MD Results – if analyzed**	Summary of results, project-specific sample ID, unspiked sample concentration, concentration detected, concentration spiked, percent recoveries and RPDs; raw instrument data

Appendix III B-3

Analysis Sequence for Mercury by WSC-CAM-III B

Typical analytical sequence for mercury by CVAA using WSC-CAM-III B:

- Initial Calibration
- ICV
- ICB
- LLCV – only required if initial calibration curve does not have a low-level standard at the level of the RL/LLOQ
- CCV – initial
- CCB – initial
- MB
- LCS
- LCSD – only required if not performing a project-specific MD
- 8 samples – include the project-specific MS and/or MD, if applicable
- CCV
- CCB
- 10 samples
- CCV
- CCB
- Etc. (continue 10 samples and CCV/CCB pairs)
- CCV – ending
- CCB – ending