WSC – CAM – III A

Quality Control Requirements and Performance Standards for the ***Analysis of Trace Metals by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

III. Metals Methods

A. Quality Control Requirements and Performance Standards for WSC-CAM-III A (Metals by ICP-OES)

Table of Contents

List of Tables and Appendices

ACRONYM LIST

CAM	Compendium of Analytical Methods
CASN	Chemical Abstracts Service Number
CCB	Continuing calibration blank
CCV	Continuing calibration verification
COC	Chain-of-custody
DF	Dilution factor
FLAA	Flame atomic absorption
GFAA	Graphite furnace atomic absorption
HNO_3	Nitric acid
ICB	Initial calibration blank
ICP-AES	Inductively Coupled Plasma-Atomic Emission Spectrometry
ICP-OES	Inductively Coupled Plasma-Optical Emission Spectrometry
ICP-MS	ICP-Mass Spectrometry
ICSA/AB	Interelement interference check samples
ICV	Initial calibration verification
IEC	Interelement correction factors
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
MSD	Matrix spike duplicate
%D	Percent difference
PDS	Post-digestion spike
QA	Quality assurance
QC	Quality control
r	Correlation coefficient
r^2	Coefficient of determination
RCs	Reportable Concentrations
RL	Reporting limit
RPD	Relative percent difference
RQs	Reportable Quantities
SIC	Spectral interference check
SRM	Standard reference material
USEPA	United States Environmental Protection Agency

<u>UNITS</u>:

mg/kg	Milligrams per kilogram
mL	Milliliter
µg/L	Micrograms per liter
µm	Micron

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

1.1 Overview of WSC-CAM-III A

WSC-CAM-III A, *Quality Control Requirements and Performance Standards for the Analysis of Trace Metals by Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) in Support of Response Actions under the Massachusetts Contingency Plan (MCP)*, is a component of MassDEP's Compendium of Analytical Methods (CAM). Note: EPA interchangeably uses "Optical" or "Atomic" in reference to the ICP instrumentation for this method; it should be noted that ICP-OES is equivalent to ICP-Atomic Emission Spectrometry [AES]). Effective January 15, 2024, this revised CAM protocol, WSC-CAM-III A, replaces the previous version of the Trace Metals CAM document, WSC-CAM-III A (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 January 15, 2024 for the purpose of "Presumptive Certainty," the revised protocol may be used optionally prior to its effective date upon its publication on October 16, 2023.

This document provides Quality Control (QC) requirements and performance standards to be used in conjunction with the required analytical method SW-846 6010D (or the most current version), analysis for Trace Metals in aqueous and solid samples using ICP-OES preceded by conventional sample preparation methods via SW-846 Methods, as described in Section 1.3 of this protocol. The QC requirements and performance standards specified in this document in Table III A-1 together with the analytical procedures described in EPA SW-846 Method 6010D, *Inductively Coupled Plasma-Optical Emission Spectrometry*, constitute the WSC-CAM-III A protocol. All protocols included in the CAM are considered "methods" published by the MassDEP pursuant to the provisions of 310 CMR 40.0017(2). Since the analytical techniques for EPA SW-846 6010D and EPA SW-846 6010C are substantially the same, use of either of these analytical methods (or a subsequent/more current version) meets the "Presumptive Certainty" requirement of WSC-CAM-III A. Analysts should note that though EPA SW-846 6010D and EPA SW-846 6010C have consistent instrument set-up and initial calibration criteria, they do have differing method QC requirements; where QC criteria differ in the EPA SW-846 methods, QC criteria in the current CAM protocol take precedence.

Sample preservation, container and analytical holding time specifications for aqueous, soil, and sediment matrices for Trace Metals analyzed in support of MCP decision-making are presented in Appendix III A-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 Trace Metals by WSC-CAM-III A

The reporting limit (RL) or lower limit of quantitation (LLOQ) for an individual analyte using WSC-CAM-III A is dependent on the concentration of the lowest non-zero standard in the initial calibration or the 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 WSC-CAM-III A target analytes are:

- Aqueous samples (surface water, groundwater, and drinking water)
  - 5-10 µg/L for arsenic, barium, beryllium, cadmium, chromium, lead, selenium, silver, and vanadium
  - 25-50 µg/L for antimony, nickel, thallium, and zinc

- Soil and Sediment samples (assuming 100% solids)
  - 0.2-1 mg/kg (wet weight) for arsenic, barium, beryllium, cadmium, chromium, nickel, silver, and vanadium
  - 2-5 mg/kg (wet weight) for antimony, lead, selenium, thallium, and zinc

For "Presumptive Certainty" purposes, if the 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 WSC-CAM-III A target analytes may be required to satisfy project requirements. The RL/LLOQ (based on the concentration of the lowest calibration standard or the LLCV) for each target metal 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 (e.g., EPA SW-846 6020B). All such modifications must be described in the laboratory narrative. RLs/LLOQs for the WSC-CAM-III A target metals 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 A

Each laboratory that uses the WSC-CAM-III A 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 digestion/analysis of laboratory control samples (LCS) and LCS duplicates (LCSD) to assess analytical accuracy and precision. Matrix spikes (MS), matrix spike duplicates (MSD), 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 A-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 A must include the following information:

QC Element	Performance Criteria
Initial Calibration	WSC-CAM-III A, Table III A-1

QC Element	Performance Criteria
Continuing Calibration Verification	
Method Blanks	
Percent Recovery for LCS & MS	
Relative Percent Difference (RPD) for LCSD, MSD, or MD	
Other Instrument QC Samples including: Spectral Interference Checks (SICs)	

Laboratories are encouraged to actively monitor pertinent QC performance standards described in Table III A-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 A protocol, laboratory-specific control limits must meet or exceed (demonstrate less variability than) the performance standards for each QC element listed in Table III A-1. It should be noted that the performance standards listed in Table III A-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 experienced in the use of ICP-OES as a quantitative tool for environmental analyses and knowledgeable in the correction of spectral, chemical, and physical interferences described in this method.

1.2 Summary of SW-846 Method 6010D

ICP-OES is used to determine Trace Metals in solution. The method is applicable for all of the analytes listed in Table III A-2 as well as numerous other metals (refer to Section 1.1 of SW-846 Method 6010D). All aqueous matrices (except dissolved/filtered samples) and solid matrices require digestion prior to analysis.

The method describes multi-elemental determinations by ICP-OES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency ICP. The spectra are dispersed by a grating spectrometer and the intensities of the emission lines are monitored by photosensitive devices.

Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line.

1.3 Sample Digestion/Preparation Methods for WSC-CAM-III A

Samples for analysis by SW-846 Method 6010D must be prepared (digested) to solubilize the sample prior to analysis, except for dissolved/filtered aqueous samples. Dissolved/filtered aqueous samples may be analyzed directly, without digestion, as long as an internal standard is used to monitor for interferences and samples are acidified to match the calibration standards. If matrix interferences are observed, see Section 1.6 of this CAM protocol for further information. Note: For the purposes of the WSC-CAM-III A protocol, “dissolved” samples are operationally defined as those samples which have been filtered through a 0.45 µm filter.

Preparation methods for Trace Metals are described in Chapter Three of SW-846 and are summarized below.

SW-846 Digestion/Preparation Method	Matrix	Title/Description
3005A	Aqueous: Surface Water/ Groundwater	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by Flame Atomic Absorption (FLAA) or ICP Spectroscopy
3010A	Aqueous: Surface Water/ Groundwater/ Mobility-procedure extracts/ Aqueous waste	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy
3015A	Aqueous: Drinking Water/ Surface Water/ Groundwater/ Mobility-procedure extracts/ Aqueous waste	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts
3031	Solid: Oily Waste/Tar/ Wax/Paint/ Petroleum Product	Acid Digestion of Oils for Metals Analysis by Atomic Absorption or ICP Spectrometry
3040A	Solid: Oil/Grease/Wax	Dissolution Procedure for Oils, Greases, or Waxes
3050B	Solid: Soil/Sediment/ Sludge	Acid Digestion of Sediments, Sludges, and Soils
3051A	Solid: Soil/Sediment/ Sludge/Oil	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils
3052	Solid: Biological Tissue/Oil/Ash Soil/Sediment/ Sludge	Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices

1.4 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 Method 6010D for further information on method interferences and contamination. A summary of several common interferences and corrective measures is provided below.

- Spectral interferences (described in Section 4.1 of SW-846 Method 6010D) – caused by background emission, stray light from high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra. Common spectral interferents, which cause suppression or enhancement of other analytes present in a sample, include aluminum, calcium, iron, and magnesium (though other analytes can also contribute to spectral interference and should be monitored – see Table 1 of SW-846 Method 6010D for a list of 21 potential interelement interferents and the analytes that they affect). Spectral interferences are minimized by using background corrections and interelement corrections, which can be applied either automatically by the ICP data system or manually by the spectroscopist. It is recommended that automatic (computerized) corrections for both background and interelement interferences be utilized during analysis of all samples under this protocol. If not, the laboratory must narrate how spectral interferences were minimized and what hand-calculations, if any, were performed to correct sample results. The acceptable analysis of SICs (see Table III A-1 for acceptance criteria) provides evidence of acceptable background and interelement corrections.
- Physical interferences (described in Section 4.2 of SW-846 Method 6010D) – caused by sample viscosity and surface tension effects on the sample nebulization. Samples with high dissolved solids or high acid content can exhibit physical interference. Physical interferences can be minimized by diluting the sample, using an internal standard, or using a high solids nebulizer to introduce the sample to the ICP. The common use of mass flow controllers also minimizes the effects of physical interferences and improves ICP performance.
- Memory interferences (described in Section 4.4 of SW-846 Method 6010D) – caused by a high concentration sample contributing to signals measured in a subsequent sample. Optimizing rinse times between sample analyses (including both field and QC samples) will minimize the potential for memory interferences.
- High salt concentrations (described in Section 4.5 of SW-846 Method 6010D) – cause analyte signal suppression (e.g., seawater samples). Samples with high salt content can cause both physical interference, by salting-over the torch, and significant suppression of analyte response in the sample. See Section 1.6 of this CAM protocol for further information.

1.5 Quality Control Requirements and Performance Standards for WSC-CAM-III A

Specific QC requirements and performance standards for the WSC-CAM-III A protocol are presented in Table III A-1. Refer to WSC-CAM-VII A for field QC requirements. ***Note that a project-specific matrix spike (MS) must be performed for target Trace Metals 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*.

1.6 Special Analytical Considerations for WSC-CAM-III A

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

- Matrix Spike (MS) Recovery – ***A MS is required for WSC-CAM-III A 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 the affected metal, the laboratory must follow the required corrective actions listed on Table III A-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.

- RLs/LLOQs, sensitivity, and the optimum and linear concentration ranges of the analytes can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1 of SW-846 Method 6010D lists the recommended analytical wavelengths for numerous metals, including all analytes listed in Table III A-2 of this protocol. Trace Metals other than those listed in Table III A-2 may be analyzed by this method if the Initial Demonstration of Proficiency, as described in Section 1.1.2 of this protocol, is demonstrated.

- Appendix III A-3 provides a typical analysis sequence for Trace Metals analyzed using this CAM protocol.

- If unusual interelement or matrix interferences are encountered, a description of any corrective measures utilized by the laboratory must be included in the laboratory narrative. Such measures may include use of an alternative analytical wavelength, non-standard computerized compensation, sample dilution to overcome physical or chemical interferences, use of standard additions, or other method-specific corrective actions.

- Iron interference on thallium may not be adequately corrected in all matrices by ICP interelement correction factors (IECs). Data users should consider that detected thallium results above the applicable regulatory standard may be false positives and should be confirmed by an alternate analysis, such as ICP-Mass Spectrometry (ICP-MS) (WSC-CAM-III D).

- Samples with high salt content can cause both physical interference, by salting-over the torch, and significant suppression of analyte response in the sample. Samples should be diluted to bring the sodium (and other analytes) within the linear range of the instrument. Note, however, that this approach (dilution) may raise the sample-specific RL/LLOQ for analytes of interest above the MCP or data user requirements. Therefore, it is recommended that alternate preparation/extraction methods such as chelation/extraction be used to remove significant salt interference prior to ICP-OES analysis. Any non-routine modifications to the method must be described in the laboratory narrative.

- If dissolved/filtered aqueous samples are analyzed directly (not digested) and matrix interferences are present, as measured by low (outside of control limits) internal standard responses, the laboratory should digest the samples to try and reduce the interferences and repeat the analyses to obtain more accurate results.

- Metals not listed in Table III A-2 and identified and quantified in the course of SW-846 Method 6010D analysis of field samples to evaluate interelement spectral interferences, etc., need not be reported as contaminants, unless they were designated by the data user as project-specific target analytes.

- Data users should note that RLs/LLOQs for antimony, beryllium, and thallium using WSC-CAM-III A will not meet MCP Method 1 GW-1 Standards. If GW-1 standards need to be achieved for these metals, alternate methods should be used (such as ICP-MS method WSC-CAM-III D or Graphite Furnace Atomic Absorption [GFAA] method WSC-CAM-III C).

- For many applications, SW-846 Method 6020B (ICP-MS CAM WSC-CAM-III D) may be the preferred analytical approach for the determination of Trace Metals in various environmental media to support ecological risk assessment decisions under the MCP. This method has the requisite sensitivity and flexibility to cost-effectively identify and quantify a wide range of ecologically-significant metals. The data user should consult with the ecological risk assessor to develop data quality objectives for the sampling program to include contaminants of concern and program-specific RLs/LLOQs.

- Mercury is not included on the standard analyte list for WSC-CAM-III A because of its elevated instrument detection limit by ICP-OES.

  Although mercury is not required to be reported to obtain “Presumptive Certainty” status for data using this WSC-CAM-III A protocol, it must be given consideration as a contaminant of concern when sites with unknown, uncertain or complex history are assessed for potential contamination associated with “total metals” pursuant to 310 CMR 40.0191. Under these circumstances, the preferred analytical method for mercury is the WSC-CAM-III B protocol, based on SW-846 Methods 7470A and 7471B (cold vapor atomic absorption).

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010D) Using WSC-CAM-III A

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 contain all target analytes. (4) Must follow procedures in Section 9.4 of SW-846 6010D and the applicable preparation method (SW-846 3000 series).	No	NA	Refer to Section 9.4 of SW-846 6010D, the applicable preparation method requirements in SW-846 3000 series methods, and Section 1.1.2 of this protocol.	NA
LLOQ Verification	Sensitivity	(1) Initial Verification: Must follow procedure in Section 9.8.1 of SW-846 6010D. (2) Quarterly Verification: Must follow procedure in Section 9.8.2 of SW-846 6010D.	No	NA	Recalibrate if needed or raise the LLOQ and repeat the verification.	NA
Preparation of Samples	Accuracy and Representativeness	(1) All aqueous (except dissolved/filtered samples) and solid samples must be prepared (digested) prior to analysis. See Section 1.3 for preparation method references. See Sections 1.3 and 1.6 for information on digestion of dissolved/filtered samples.	No	NA	NA	NA
Linear Range	Laboratory Analytical Accuracy	(1) Following calibration (Section 9.6 of SW-846 6010D). (2) Utilize the high standard in the calibration curve or analyze a standard at higher concentration than high standard in calibration curve. (3) Percent recoveries must be 90-110% for all target analytes.	No	NA	NA	NA
Initial Calibration	Laboratory Analytical Accuracy	(1) Following profiling and optimization of ICP; daily prior to sample analysis. (2) Minimum calibration blank plus one calibration standard for each target analyte or a multi-point curve (minimum of three standards). (3) Linear regression with correlation coefficient $r \geq 0.995$; non-linear regression with coefficient of determination (r^2) ≥ 0.990.	No	NA	Perform instrument maintenance as necessary; re-optimize instrument; re-calibrate as required by SW-846 6010D.	Suspend all analyses until initial calibration meets criteria.

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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
		(4) For multi-point curve, recalculate the concentrations in the low-level standard using the new curve; percent recoveries must be within 80-120% for each metal. (5) For multi-point curve, recalculate the concentrations in the mid-level standard using the new curve; percent recoveries must be within 90-110% for each metal.				
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) Must contain all target analytes. (5) Percent recoveries must be between 90-110% for each target analyte.	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) Target analytes must be <RL/LLOQ.	No	NA	(1) Reanalyze ICB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and all associated sample concentrations are either not detected or > 10x blank level, no further action required. Otherwise, recalibrate and reanalyze ICV & ICB. (3) If ICBs consistently have target metals > RL/LLOQ, re-evaluate the RL/LLOQ.	Suspend all analyses until ICB meets criteria.

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010D) Using WSC-CAM-III A

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
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 for each target analyte. If initial calibration includes the RL/LLOQ as the low-level standard in the initial calibration curve, then LLCV is not required. (2) Prepared using same source as initial calibration standards. (3) Concentration level must be at the level of the RL/LLOQ for all target analytes. (4) Percent recoveries must be 70-130% for all target analytes.	No	NA	(1) Reanalyze LLCV; if acceptable, no further action required. (2) If reanalysis is still outside of criteria and associated analytes are ≤10x RL/LLOQ in associated field samples, recalibrate and reanalyze LLCV and associated samples. (3) If associated analytes are >10x RL/LLOQ in associated field samples, include explanation in laboratory narrative; no further action required.	Suspend all analyses until LLCV meets criteria unless the concentrations of the affected target analytes are >10x RL/LLOQ in the associated field samples.
Spectral Interference Checks (SIC) Individual Element SIC and Mixed Element SIC (formerly ICSA and ICSAB)	Laboratory Analytical Accuracy (Individual element SIC used to evaluate possible spectral interferences and to set interelement corrections if necessary) (Mixed element SIC used daily to check instrument free from interference from metals typically observed in high concentration and to check interference corrections applied are still valid)	(1) Mixed Element SIC: Daily prior to sample analysis; Individual Element SIC: Every 6 months. (2) Individual Element SIC and Mixed Element SIC must contain known amounts of interfering analytes (see Section 9.9 of SW-846 6010D). (3) Individual Element SIC: Absolute value of non-spiked analytes must be <2x the RL/LLOQ for that analyte. (4) Mixed Element SIC: must contain the known interferents Aluminum, Calcium, Iron, & Magnesium (see Section 7.12.2 of SW-846 6010D). Measured concentrations for any target analyte other than the 4 interferents must be < ± the RL/LLOQ with the exception of metals that have been demonstrated to be contaminants in the Individual Element SIC; these can be present up to the documented concentration plus the RL/LLOQ.	No	NA	(1) The criteria for the Individual Element SIC must be met prior to analysis. (2) Reanalyze Mixed Element SIC; if acceptable, no further action required. (3) If Mixed Element SIC is still outside of criteria, correct the problem or raise the RL/LLOQ to twice the concentration observed in the Mixed Element SIC. (4) Recalibrate and reanalyze all samples since last compliant Mixed Element SIC.	Suspend all analyses until Individual Element SIC meets criteria. Suspend all analyses until Mixed Element SIC meets criteria or elevate RL/LLOQ, as described in corrective action (3).
Continuing Calibration	Laboratory Analytical	(1) Every 10 samples and at the end of the	No	NA	(1) Reanalyze CCV; if	If (3) applies, include

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010D) Using WSC-CAM-III A

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
Verification (CCV)	Accuracy	analytical run. (2) Prepared using same source as initial calibration standards. (3) Concentration level near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries must be 90-110% for each target analyte.			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 (>110%) and all associated sample results are not detected, no corrective action required.	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) Target analytes must be <RL/LLOQ.	No	NA	(1) Reanalyze CCB; if acceptable, no further action required. (2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCB – unless (3) applies. (3) If concentration of contaminant in CCB is >RL/LLOQ but all associated sample results are either not detected or >10x concentration 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. (3) Matrix-specific (e.g., water, soil): reagent water for water samples and clean empty container, glass beads, or target-analyte free sand for soil samples (4) Target analytes must be < RL/LLOQ.	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) applies. (3) If concentration of contaminant in MB is >RL/LLOQ but all associated	If (3) applies, include explanation in laboratory narrative.

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010D) Using WSC-CAM-III A

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
					sample results are either not detected or >10x concentration 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 the analytes of interest at known concentrations and with 95% confidence limits. (3) Concentration levels for aqueous LCS near midpoint of curve. (4) Must contain all target analytes. (5) Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS.	Yes	Aqueous LCS: Recovery <50%: affected analytes 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 same analyte, 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 the analytes of interest at known concentrations and with 95% confidence limits. (3) Concentration levels must be same as LCS. (4) Must contain all target analytes; analyze	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 same analyte, 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 A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010D) Using WSC-CAM-III A

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
		immediately following LCS. (5) Percent recoveries for all target analytes must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS. (6) 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) Must contain all target analytes. (4) Percent recoveries for all target analytes must be 75-125%.	Yes ONLY when requested by the data user	Recovery <30%: affects non-detects for affected metal 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. (4) Perform dilution test and/or post-digestion spike. (see QC requirements below).	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 each target analyte must be ≤20 for aqueous and ≤35 for solids.	Yes ONLY when requested by the data user	NA	Narrate.	Report exceedances in laboratory narrative.

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010D) Using WSC-CAM-III A

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
Dilution Test	Accuracy in Sample Matrix	(1) One per ≤20 field samples per matrix; only if project-specific MS outside acceptance limits and analyte concentration is >25x RL/LLOQ. (2) Perform 5x serial dilution on same sample used for MS/MD. (3) %D of the sample & dilution results for target analytes at levels >25x RL/LLOQ must be ±20% for all matrices.	Yes ONLY if project-specific MS outside acceptance limits and analyte >25x RL/LLOQ	NA	Narrate.	Report exceedances in laboratory narrative.
Post-Digestion Spike (PDS)	Accuracy in Sample Matrix	(1) One per ≤20 field samples per matrix; only if project-specific MS outside of acceptance limits and high concentration sample (>25x RL/LLOQ) not available for dilution test. (2) Only analyzed for metals that fail the MS and only if spike concentration added in MS was > concentration in unspiked sample. (3) Percent recoveries must be 75-125%.	Yes ONLY if project-specific MS outside acceptance limits and analyte >25x RL/LLOQ not available for dilution test	Recovery <30%: affects non-detects for affected metal in all associated samples. Only use PDS in evaluation of analyte in sample data when the same analyte in the MS/MSD fails.	Narrate.	Report exceedances in laboratory narrative.
General Reporting Issues	NA	(1) Non-detected values must be reported with the sample-specific RL/LLOQ for each target analyte 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. (3) Sample concentrations that exceed the linear range must be diluted and reanalyzed to fall within the linear range. (4) Samples with concentrations of metals	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 documented in the laboratory narrative or

Table III A-1: Specific QC Requirements and Performance Standards for Metals (SW-846 6010D) Using WSC-CAM-III A

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
		higher than Individual Element SIC must be diluted until concentration < the SIC, even if the high concentration element is not required to be reported for the specific sample. (5) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards. (6) 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. (7) 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] (8) Refer to Appendix III A-1 for COC requirements regarding preservation, cooler temperature, and holding times.				on the report form. Unless due to elevated concentrations of target analytes, 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 digested 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*, September 2007, 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.

1.7 Analyte List for WSC-CAM-III A

The MCP analyte list for WSC-CAM-III A includes 13 target metals as presented in Table III A-2. These include: antimony, arsenic, barium, beryllium, cadmium, chromium, lead, nickel, selenium, silver, thallium, vanadium, and zinc.

It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analytes. 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

The target metals listed on Table III A-2 have promulgated MCP Method 1 groundwater/soil standards.

1.7.1 Analyte List Reporting Requirements for WSC-CAM-III A

While it is not necessary to request and report all the WSC-CAM-III A analytes listed in Table III A-2 to obtain “Presumptive Certainty” status, it is necessary to document use and reporting of a reduced analyte list, for site characterization and data representativeness considerations. MassDEP strongly recommends use of the full analyte list during the initial stages of site investigations, and/or at sites with an unknown or complicated history of uses of oil or hazardous materials. These assessment activities may include but are not limited to:

- ✓ Immediate Response Actions (IRAs) performed in accordance with 310 CMR 40.0410;
- ✓ Initial Site Investigation Activities performed in accordance with 310 CMR 40.0405(1);
- ✓ Phase I Initial Site Investigation Activities performed in accordance with 310 CMR 40.0480 through 40.0483; and
- ✓ Phase II Comprehensive Site Investigation Activities performed in accordance with 310 CMR 40.0830.

In some cases, the use of the full analyte list for a chosen analytical method may not be necessary, with respect to data representativeness concerns, including:

- ✓ Sites where substantial site/use history information is available to rule-out all but a limited number of contaminants of concern, and where use of the full analyte list would significantly increase investigative costs; or

- ✓ Well-characterized sites where initial full-analyte list testing efforts have sufficiently narrowed the list of contaminants of concern.

Note: a data user who avoids detection and quantitation of a contaminant that is present or likely present at a site above background levels by limiting an analyte list could be found in criminal violation of MGL c. 21E or any regulations or orders adopted or issued thereunder.

In cases where a reduced list of analytes is requested, laboratories must still employ the specified QC requirements and performance standards in WSC-CAM-III A to obtain "Presumptive Certainty" status.

Table III A-2: Analyte List for WSC-CAM-III A (SW-846 6010D)

Analyte	CASN
Antimony[1]	7440360
Arsenic	7440382
Barium	7440393
Beryllium[1]	7440417
Cadmium	7440439
Chromium (Total)	7440473
Lead	7439921
Nickel	7440020
Selenium	7782492
Silver	7440224
Thallium[1]	7440280
Vanadium	7440622
Zinc	7440666

[1] Standard laboratory RL/LLOQ for this metal may not be able to achieve regulatory compliance limit. A more sensitive analytical procedure (e.g., ICP-MS by WSC-CAM-III D or GFAA by WSC-CAM-III C) may be required.

CASN – Chemical Abstracts Service Number

NOTE: Other Trace Metals may also be analyzed using the WSC-CAM-III A Protocol but are not considered part of the CAM target analyte list.

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 A

3.1 General Reporting Requirements for WSC-CAM-III A

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 A

Specific QC requirements and performance standards for WSC-CAM-III A are presented in Table III A-1. Specific reporting requirements for WSC-CAM-III A are summarized below in Table III A-3 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 III A-3 Routine Reporting Requirements for WSC-CAM-III A (SW-846 6010D)

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***
Spectral Interference Checks (SICs; formerly ICS A and AB)	***NO***
Method Blank	***YES***
Laboratory Control Sample (LCS)	***YES***
LCS Duplicate (LCSD)	***YES (if no MSD or MD)***
Matrix Spike (MS)	***YES*** *(if requested by data user)*
Matrix Duplicate (MD)	***YES*** *(if requested by data user)*
Dilution Test	***YES*** *(if MS requested by data user & MS fails criteria)*
Post-digestion Spike	***YES*** *(if MS requested by data user & MS fails criteria)*
Identification and Quantitation	***NO***
General Reporting Issues	***YES***

3.2.1 Sample Dilution

Under circumstances that sample dilution is required because the concentration of one or more of the target or non-target metals exceeds the concentration of the linear range, the RL/LLOQ for the affected metal 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 X Lowest Calibration Standard (or the concentration of the LLCV) for target metal

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 A-1

Sample Collection, Preservation, and Handling Procedures for Trace Metals Analyses

Sample preservation, container and analytical holding time specifications for aqueous, soil, and sediment matrices for Trace Metals 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)*.

Matrix	Container[1]	Preservation[6]	Holding Time[2]
Aqueous Total Metals	500 mL Polyethylene Bottle	HNO_3 to pH <2	180 days
Aqueous Dissolved Metals (Filtered)	500 mL 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]	180 days
Soil and Sediment	4-ounce glass jar with teflon-lined cap	Cool to ≤ 6°C[4]	180 days[5]
Concentrated Waste	125 mL wide mouth glass or plastic	Cool to ≤ 6°C[4]	180 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 #5 below). As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if the holding time is exceeded by >2x the allowable holding time, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias. Note: The holding time is for the target Trace Metals CAM list of 13 metals, not including mercury (mercury holding time is 28 days; analyze by alternate method).

[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] SW-846 does not require preservation for Trace Metals (other than mercury) in solid samples; however, as a practical consideration since one sample container is generally collected for solid samples for all total metals analyses, preservation (cooling ≤6°C) for this CAM protocol has been defined.

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

[6] 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 A-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 A (Trace Metals by ICP-OES: SW-846 6010D)	
Laboratory Narrative	Must comply with the required laboratory narrative contents as described in WSC-CAM-VII A
Sample Handling Information	Chains-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)
Linear Range	Results and raw instrument data for determination of linear range of ICP-OES
Initial Calibration Data	Raw instrument data for initial calibration, including calculation of linear or non-linear regression, correlation coefficients, or coefficients of determination; 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
Spectral Interference Checks (formerly ICSA/AB)	Results and raw instrument data for Mixed Element SIC; summary of results from most recent Individual Element SIC
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** **PDS Results – if analyzed** **Dilution Test Results – if analyzed**	Summary of results, project-specific sample ID, unspiked sample concentration, concentration detected, concentration spiked, percent recoveries, RPDs, and %Ds, as applicable; raw instrument data

Appendix III A-3

Analysis Sequence for Trace Metals by WSC-CAM-III A

Typical analytical sequence for Trace Metals by ICP-OES using WSC-CAM-III A:

- 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
- Mixed Element Spectral Interference Check
- MB
- LCS
- LCSD – only required if not performing a project-specific MD
- 7 samples – include the project-specific MS and/or MD if applicable, plus Dilution test and/or PDS sample, if applicable
- CCV
- CCB
- 10 samples
- CCV
- CCB
- Etc. (continue 10 samples and CCV/CCB pairs)
- CCV – ending
- CCB – ending