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The requirement to use a separate-source standard to prepare the project-specific matrix spike (MS) and MS duplicate (MSD) has been eliminated.

BWSC


**Implementation Statement for MCP
Analytical Methods**

For “Presumptive Certainty” purposes, the implementation date for all MCP Analytical Methods will be thirty (30) calendar days after their respective publication date to allow for necessary changes and modifications in laboratory processes and procedures. At the discretion of the laboratory, a revised MCP Analytical Method may

be used anytime from the date of publication to the implementation date to satisfy “Presumptive Certainty” status the requirements.



Title: **Table III A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 6010B**

Required QA/QC	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
LCS Duplicate	Laboratory Method Precision	(1) Frequency - One per digestion batch of ≤ 20 field samples. If samples are undigested (dissolved metals) an ICV duplicate may be substituted for an LCS duplicate. (2) Prepared using same standard source and concentration as LCS. (3) Recommended to be run immediately after LCS in analytical sequence. (4) LCS duplicate must be matrix-matched to samples (aqueous/solid) and digested with the samples (5) Laboratory-determined Relative Percent Difference (RPD) must be ≤ 20 (aqueous) and ≤ 30 (solids), and (6) A project-specific MD or MSD may be substituted to evaluate precision in lieu of an LCS duplicate.	Yes	Recalculate RPD; Locate source of problem; Narrate non-conformances	(1) Locate and rectify source of non-conformance before proceeding with the analyses of subsequent sample batches. (2) Narrate non-conformances
 Project-specific Matrix Spike Sample (MS)	Method Accuracy in Sample Matrix	(1) Frequency - One per digestion batch of < 20 field samples <u>if requested by data-user</u> (see Appendix III A-2 for required matrix frequency) . <u>If samples are undigested (dissolved metals) perform an analytical spike.</u> (2) MS % recoveries for all analytes 75-125% for all media (recommended).	Yes Only when requested by the data-user	If MS % recovery is $> 30\%$, and LCS was in-control, no corrective action is required. If MS %recovery is $< 30\%$, and non-detected results were found, one or more of the following may be performed to confirm matrix effect: 1) homogenize sample well, re-digest, and re-analyze sample/MS; 2) perform method of standard additions for quantitation; 3) perform serial dilution; 4) perform post-digestion spike.	Narrate non-compliance. Note to data users: USEPA Region I data validation guidance requires rejection of non-detected results with MS $< 30\%$ recovery. Therefore, the 1st corrective action listed may be required for critical sample data to obtain usable results.



Title: **Table III A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 6010B**

Required QA/QC	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
Project-specific Matrix Duplicate Sample (MD) or Matrix Spike Duplicate (MSD)	Method Precision in Sample Matrix	(1) Frequency - One per digestion batch of < 20 field samples, at discretion of laboratory or at request of data-user <u>If samples are undigested (dissolved metals) perform an analytical duplicate.</u> (2) MD: Prepared by digesting and analyzing an additional aliquot of a field sample. (3) MSD: Prepared by fortifying an additional aliquot of the field sample used for MS. (4) MD relative percent difference (RPD): aqueous results > 5x RL: $\frac{20}{RL}$; aqueous results < 5x RL: $\frac{20}{RL}$; solid results > 5x RL: $\frac{20}{RL}$; solid results < 5x RL: $\frac{20}{RL}$	Yes Only when requested by the data-user	Check LCS; if recoveries acceptable in LCS, narrate nonconformance.	Note exceedances in Environmental Laboratory case narrative.
Linear Range Analysis	Laboratory Analytical Accuracy	(1) Frequency - Annually for most metals, every 6 months for those metals that periodically approach the upper limit. (2) Determine the upper limit of the linear dynamic range for each wavelength utilized by determining the signal responses from a minimum of 3 (preferably 5) different concentration standards across the range, see method for details.	No	Not applicable.	See method for requirements. Data must be kept on file to document initial demonstration of performance (IDP) of linear range analysis for each ICP instrument.
Inter-element Spectral Interference Correction Analysis	Laboratory Analytical Accuracy	(1) Frequency - Inter-element spectral corrections must be verified every 6 months. (2) Routine analysis of ICSA and ICSAB verifies inter-element spectral interference corrections. (3) See method for details.	No	Not applicable.	See method for requirements. Data must be kept on file to document initial demonstration of performance (IDP) of interelement correction factors/equations and background correction points for each ICP instrument.



Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup

Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 6010B, Trace Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

WSC-CAM

Table: III A-1

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Title: **Table III A-1 Specific QA/QC Requirements and Performance Standards for SW-846 Method 6010B**

Required QA/QC	Data Quality Objective	Performance Standard	Required Deliverable	Recommended Corrective Action	Analytical Response Action
General Reporting	NA	(1) Non detected values must be reported with the sample-specific reporting limit for each ICP analyte. (2) The RL must be \leq the applicable regulatory compliance standard for each metal reported (3) The RL must be verified at least daily with a low-level calibration check standard following the calibration curve or supported by the low-level standard in the calibration curve. (4) Results for soils/sediments must be reported on a dry weight basis for comparison to MCP regulatory standards (5) Sample concentrations that exceed the highest calibration standard must be diluted (in the same acid matrix) to fall within the calibration range when reanalyzed	Yes	Not applicable.	



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 6010B, Trace Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

1.5 List of Analytes for SW-846 Method 6010B

The MCP analyte list for SW-846 Method 6010B presented in Table III A-2, is intended to be protective of human health and the environment and the Commonwealth's groundwater resources. The list is comprised of potential toxic metal contaminants that are readily-analyzable by SW-846 Method 6010B and have a Method 1 Groundwater/Soil Standards as described in 310 CMR 40.0974

The MCP Method 1 Groundwater/Soil Standards used to characterize the risk of harm posed by oil or hazardous materials at a disposal site are described in 310 CMR 40.0974(2), Table 1. This list of groundwater/soil standards, developed by the Department, takes into account a defined set of conservative potential exposure pathways likely to be encountered at most disposal sites. Method 1 Standards have been developed by the Department for over one hundred organic and inorganic contaminants that are commonly encountered at MCP disposal sites. The MCP Method 1 Groundwater/Soil Standards list is periodically reviewed and updated by the Department. When compounds are added to the MCP Method 1 Groundwater/Soil Standards list that are suitable for analysis by SW-846 Method 6010B, the analyte list for this method will be updated accordingly.

1.6 Additional Analyte Reporting Requirements for SW-846 Method 6010B

1.6.1 Analysis and Reporting recommendations for SW-846 Method 6010B

While it is not necessary to request and report all the SW-846 Method 6010B analytes listed in Table III A-2 to obtain Presumptive Certainty, it is necessary to document such a limitation, for site characterization and data representativeness considerations. DEP 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 a limited number of cases, the use of the full analyte list for a chosen analytical method may not be necessary, with respect to data representativeness concerns, including:

- ✓ Uncharacterized 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



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 6010B, Trace Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

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

Note that a desire to avoid detection and quantitation of a contaminant that is present or likely present at a site above background levels is not a valid reason to limit an analyte list, and that such an action could constitute a criminal violation of MGL c. 21E.

In cases where a truncated list of method analytes is selected, laboratories must still employ the method-specific quality control requirements and performance standards associated with the requested analytes list to obtain Presumptive Certainty status.

1.6.2 Elements Used to Evaluate Inter-Element Spectral Interferences

Elements not listed in Table III A-2 and identified and quantified in the course of SW-846 Method 6010B analysis of field samples to evaluate inter-element spectral interferences, etc., need not be reported as contaminants.

1.6.3 Special Analytical and Reporting Considerations for Mercury

Because of its elevated Estimated Instrumental Detection Limit (Table 1, SW-846 Method 6010B) Mercury, a common environmental contaminant, is not included on Table III A-2, Analyte List for SW-846 Method 6010B.

Although Mercury is not required to be reported to obtain "Presumptive Certainty" status for SW-846 Method 6010B, 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" to satisfy due diligence requirements pursuant to 310 CMR 40.019. Under these circumstances, the preferred analytical method for Mercury is SW-846 Method 7470/7471 (Cold Vapor AAS).



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 6010B, Trace Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

Table III A-2 Analyte List for SW-846 Method 6010B

ANALYTE	CASRN	MCP METHOD 1	
		GW-1	S-1/GW-1
		µG/L (PPB)	mg/Kg (PPM)
Antimony	7440360	6 ¹	10
Arsenic	7440382	50	30
Barium	7440393	2000	1000
Beryllium	7440417	4	0.7
Cadmium	7440439	5	30
Chromium (Total)	7440473	100	200 ²
Lead	7439921	15 ¹	300
Nickel	7440020	100	300
Selenium	7782492	50 ¹	400
Silver	7440224	7 (GW-3) 40 (GW-1)	100
Thallium	7440280	2 ¹	8
Vanadium	7440622	50	400
Zinc	7440666	900 (GW-3) 2000 (GW-1)	2500

1. Laboratory Reporting Limits for this element may not be able to achieve MCP regulatory compliance limit using SW-846 Method 6010B.

2. Dermal contact updated Aug 99



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 6010B, Trace Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

2.0 Data Usability Assessment for Method 6010B

Overall data usability is influenced by uncertainties associated with both sampling and analytical activities. This document provides detailed quality control requirements and performance standards for SW-846 Method 6010B which may be used to assess the analytical component of data usability. The sampling component of data usability, an independent assessment of the effectiveness of sampling activities to meet data quality objectives, is not substantively addressed in this document.

3.0 Reporting Requirements for SW-846 Method 6010B

3.1 General Reporting Requirements for SW-846 Method 6010B

General reporting requirements for analytical data used in support of assessment and evaluation decisions at MCP disposal sites are presented in WSC-CAM-VIIA. This guidance document provides recommendations for field QC, as well as the required content of the Environmental Laboratory Report, including

- Laboratory identification information presented in WSC-CAM-VII A, Section 2.4.1,
- Analytical results and supporting information in WSC-CAM-VII A, Section 2.4.2,
- Sample- and batch-specific QC information in WSC-CAM-VII A, Section 2.4.3,
- Laboratory Report Certification Statement in WSC-CAM-VII A, Section 2.4.4,
- Copy of the Analytical Report Certification Form in WSC-CAM-VII A, Exhibit VII A-1,
- Environmental Laboratory case narrative contents in WSC-CAM-VII A, Section 2.4.5,
- Chain of Custody Form requirements in WSC-CAM-VII A, Section 2.4.6

3.2 Specific Reporting Requirements for SW-846 Method 6010B

Specific QA/QC Requirements and Performance Standards for SW-846 Method 6010B are presented in Table III A-1. Specific reporting requirements for SW-846 Method 6010B are summarized below in Table III A-3 as "Required Analytical Deliverables (**YES**)". These routine reporting requirements should always be included as part of the laboratory deliverable for this method. It should be noted that although certain items are not specified as "Required Analytical Deliverables (**NO**)", these data are to be available for review during an audit and may also be requested on a client-specific basis.

3.2.1 Reporting Data for Spectral Interference Corrections

If unusual inter-element or matrix interferences are encountered, a description of any corrective measures utilized by the laboratory must be included in the Environmental Laboratory case 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.



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Table III A-3 Routine QA/QC Reporting Requirements for SW-846 Method 6010B

Parameter	Required Analytical Deliverable
Initial Calibration	NO
Initial Calibration Verification (ICV)	NO
Initial Calibration Blank (ICB)	NO
Low Level Calibration Check standard	NO
Continuing Calibration Verification (CCV)	NO
Continuing Calibration Blank (CCB)	NO
Interference Check Standards (ICS A and B)	NO
Method (Preparation) Blank	YES
Laboratory Control Samples (LCS)	YES
LCS Duplicate (or project-specific MD or MSD)	YES
Project-specific Matrix Spike Sample (MS)	YES, <u>only</u> if requested by the LSP
Project-specific Matrix Duplicate (MD)	YES, <u>only</u> if requested by the LSP
Project-specific Matrix Spike Duplicate (MSD)	YES, <u>only</u> if requested by the LSP
Linear Range Analysis	NO
Inter-element Spectral Interference Correction Analysis	NO
General Reporting – sample specific reporting limits	YES



Title: Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 6010B, Trace Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

4.0 Regulatory Limits for Metals under 310 CMR 40.000

The most stringent (lowest) MCP Reportable Concentrations (RCs) and Method 1 Standards for metals analyzable by SW-846 Method 6010B are as follows:

Metal	RQ Pounds	RC GW-1 mg/L - (ppm)	RC S-1 mg/kg - (ppm)	Method 1 Groundwater ug/L - ppb	Method 1 Soils ug/g - ppm
Antimony	50	0.006	10	6	10
Arsenic	1	0.05	30	50	30
Barium	100	2	1000	2000	1000
Beryllium	5	0.004	0.7	4	0.7
Cadmium	5	0.005	30	5	30
Chromium (III)	100	0.1	1000	100	1000
Chromium (VI)	100	0.1	1000	50	200
Cobalt	50	5	500	NS	NS
Copper	100	10	1000	NS	NS
Lead	5	0.02	300	15	300
Lithium	10	1	100	NS	NS
Mercury ¹	1	0.001	20	2	20
Nickel	10	0.08	300	80 (GW-3)	300
Phosphorous	1	NA	NA	NS	NS
Potassium	10	NA	NA	NS	NS
Selenium	10	0.05	400	50	400
Silver	50	0.007	100	7 (GW-3)	100
Sodium	5	NA	NA	NS	NS
Thallium	50	0.002	8	2	8
Vanadium	50	0.05	400	50	400
Zinc	50	0.9	2500	900 (GW-3)	2500

NA – Not Applicable

RQ – Reportable Quantity

RC – Reportable Concentration for Groundwater (GW-1) and Soils (S-1)

Method 1 Groundwater – GW-1 Category unless otherwise noted

Method 1 Soils – Category S-1/GW-1 in all cases

NS – No MCP Method 1 Standard has been promulgated by the Department.

¹ Mercury values presented for completeness only. Analyze mercury by SW-846 Methods 7470A and 7471



Title: Sample Collection, Preservation, And Handling Procedures for SW-846 Method 6010B, Trace Metals by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil, sediment and wastes matrices for metal analyses conducted in support of MCP decision-making are summarized below and presented in Appendix VII-A of WSC-CAM-VIIA, Quality Assurance and Quality Control Guidelines for Sampling, Data Evaluation, and Reporting Activities for the Massachusetts Contingency Plan (MCP). Additional guidance may be found in SW-846, Chapter Three

Matrix	Sample Container(s) ¹	Preservative	Holding Time ²
Total Metals Groundwater and Surface Water	(1) 1-L Polyethylene Bottle for Total Metals	HNO ₃ to pH < 2,	180 days: all metals except mercury 28 days: mercury
Dissolved Metals Groundwater and Surface Water	(1) 1-L Polyethylene Bottle for field-filtered sample for Dissolved Metals	Filter (0.45 µm) unpreserved sample on site; or at the laboratory (prior to acid preservation) within 24 hours of collection, then HNO ₃ to pH <2,	180 days: all metals except mercury 28 days: mercury
Suspended Metals Groundwater and Surface Water	Submit Suspended Solids on Filter to Laboratory	Filter on site Filter 100 – 500 ml of unpreserved sample	180 days: all metals except mercury 28 days: mercury
Soils and Sediments	(1) 4-ounce glass jar	Cool, 4°C	180 days: all metals except mercury 28 days: mercury
Concentrated Waste Samples	125 mL wide mouth glass or plastic	Cool to 4°C	180 days: all metals except mercury 28 days: mercury

1 The number of sampling containers specified is not a requirement. For specific analyses, the collection of multiple sample containers is encouraged to avoid resampling if sample is consumed or compromised.

2 From date of sample collection.



Title: Methods for Sample Digestion or Preparation by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES)

SW-846 Method	Method Description
3005	Method prepares ground water and surface water samples for total recoverable and dissolved metal determinations by FLAA, ICP-AES, or ICP-MS. The unfiltered or filtered sample is heated with dilute HCl and HNO prior to metal determination.
3010	Method prepares waste samples for total recoverable metal determinations by FLAA, ICP-AES, or ICP-MS. The samples are vigorously digested with nitric acid followed by dilution with hydrochloric acid. The method is applicable to aqueous samples, EP and mobility-procedure extracts.
3015	Method prepares aqueous samples, mobility-procedure extracts, and wastes that contain suspended solids for total recoverable metal determinations by FLAA, GFAA, ICP-AES, or ICP-MS. Nitric acid is added to the sample in a Teflon digestion vessel and heated in a microwave unit prior to metals determination.
3031	Method prepares waste oils, oil sludges, tars, waxes, paints, paint sludges and other viscous petroleum products for analysis by FLAA, GFAA, and ICP-AES. The samples are vigorously digested with nitric acid, sulfuric acid, hydrochloric acid, and potassium permanganate prior to analysis.
3040	Method prepares oily waste samples for determination of soluble metals by FLAA, GFAA, and ICP-AES methods. The samples are dissolved and diluted in organic solvent prior to analysis. The method is applicable to the organic extract in the oily waste EP procedure and other samples high in oil, grease, or wax content
3050	Method prepares waste samples for total recoverable metals determinations by FLAA and ICP-AES, or GFAA and ICP-MS depending on the options chosen. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either nitric or hydrochloric acid. The method is applicable to soils, sludges, and solid waste samples.
3051	Method prepares sludges, sediments, soils and oils for total recoverable metal determinations by FLAA, GFAA, ICP-AES or ICP-MS. Nitric acid is added to the representative sample in a fluorocarbon digestion vessel and heated in a microwave unit prior to metals determination.
3052	Method prepares siliceous and organically based matrices including ash, biological tissue, oil, oil contaminated soil, sediment, sludge, and soil for total analysis by FLAA, CVAA, GFAA, ICP-AES, and ICP-MS. Nitric acid and hydrofluoric acid are added to a representative sample in a fluorocarbon digestion vessel and heated in a microwave unit prior to analysis