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Page 1 of 2

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)

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)



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)

VII. Sampling, Data Evaluation and Reporting Procedures for MCP Activities

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)

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ACRONYM LIST

APH	Air-phase petroleum hydrocarbon
CAM	Compendium of Analytical Methods
COC	Chain-of-custody
CVAA	Cold vapor atomic absorption
ESI/MS	Electrospray ionization/mass spectrometry
EPA	Environmental Protection Agency
EPH	Extractable petroleum hydrocarbons
FID	Flame ionization detector
GC/MS	Gas chromatography/mass spectrometry
ICP/OES	Inductively coupled plasma/optical emission spectroscopy
ICP/MS	Inductively coupled plasma/mass spectrometry
IRA	Immediate Response Action
LC/MS/MS	Liquid chromatography/dual mass spectrometry
LCS	Laboratory control sample
LLOQ	Lower limit of quantitation
MassDEP	Massachusetts Department of Environmental Protection
MCP	Massachusetts Contingency Plan
MS	Matrix spike
MSD	Matrix spike duplicate
OPR	Ongoing Precision and Recovery
PAC	Physiologically available cyanide
PAH	Polynuclear aromatic hydrocarbons
PCB	Polychlorinated biphenyl
PFAS	Per- and polyfluoroalkyl substances
PID	Photoionization detector
QA	Quality assurance
QC	Quality control
REDUA	Representativeness Evaluation and Data Usability Assessment
RL	Reporting limit
RPD	Relative percent difference
RTN	Release tracking number
SVOC	Semi-volatile organic compounds
TIC	Tentatively identified compound
TSS	Total suspended solids
VOC	Volatile organic compounds
VPH	Volatile petroleum hydrocarbons



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)

1.0 Introduction

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)*, is a component of MassDEP's Compendium of Analytical Methods (CAM). Effective September 30, 2025, this revised CAM protocol, WSC-CAM-VII A, replaces Revision No. 2 of the Acquisition and Reporting document, WSC-CAM-VII A (effective date, January 19, 2017). Please note that while this protocol must be followed on and after the effective date of September 30, 2025 for the purpose of "Presumptive Certainty," the revised protocol may be used optionally prior to its effective date upon its publication on June 30, 2025.

The purpose of this document (WSC-CAM-VII A) is to provide the regulated community with quality assurance (QA) and quality control (QC) guidance regarding the acquisition and reporting of analytical data submitted in support of response actions conducted at disposal sites regulated under M.G.L. c. 21E and 310 CMR 40.0000, the MCP.

Analytical data conforming to the specifications detailed in Section 2.0, including compliance with the QC requirements and performance standards detailed in the analyte/method-specific CAM protocols, assures data users of overall "Presumptive Certainty" and are considered by the MassDEP to meet the broad data quality requirements of 310 CMR 40.0017 and 40.0191 regarding the acquisition, analysis, and reporting of the analytical and environmental monitoring data used to support MCP response actions.

Analytical data that achieve "Presumptive Certainty" status are data for which the MassDEP stipulates the precision, accuracy, and sensitivity have been adequately determined. Depending on the nature and use of the analytical data, a separate evaluation may be necessary to confirm that the quality and representativeness of data are sufficient for its intended use in support of a response action decision. Data that achieve "Presumptive Certainty" status must still be assessed for usability and representativeness in comparison to project objectives as specified in 310 CMR 40.1056(2)(k) and further described in the MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

MassDEP's issuance of these CAM requirements and performance standards is in no way intended to preempt the exercise of professional judgment by the data user in regards to the selection of analytical methods and associated QC requirements. However, it must be clearly understood that if an alternative analytical method (non-CAM method) is chosen to support MCP decisions, the data user is responsible for independently demonstrating the accuracy, precision, sensitivity, representativeness and overall usability for all analytical data pursuant to the requirements of 310 CMR 40.0017, 40.0191(2), 40.1056(2)(k), and 40.1057(2)(k).

Section 3.0 provides a general overview of data usability guidance under the MCP. This information is provided as background for parties who elect not to use the "Presumptive Certainty" option described in Section 2.0. Detailed guidance regarding data usability and representativeness requirements specified in 310 CMR 40.1056(2)(k) and 40.1057(2)(k) is provided in Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.



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It should be noted that this document does not provide any specific guidance regarding proper sampling procedures, approaches to achieve representative sampling, or the type and frequency of field QC samples required to evaluate overall data representativeness or usability.

2.0 “Presumptive Certainty” for Analytical Data

2.1 Overview of the “Presumptive Certainty” Process

310 CMR 40.0017, 40.0191(2)(c) and 40.1056(2)(k) require that analytical and environmental monitoring data be scientifically valid and defensible, and of a level of precision and accuracy commensurate with its stated or intended use, taking into consideration relevant policies and guidelines issued by the MassDEP and the Environmental Protection Agency (EPA). 310 CMR 40.0017(3)(i) and 310 CMR 40.1056(2)(k) further provide that response action submittals to the MassDEP shall include details regarding any known conditions or findings which may affect the validity of analytical data, including unsatisfactory results obtained for blank, duplicate, surrogate or spiked samples and the requirements for a comprehensive data representativeness and usability evaluation.

To facilitate compliance with these broad performance standards, MassDEP has published the CAM, which provides a series of required protocols for the acquisition, analysis, and reporting of analytical data in support of MCP decisions. Analytical data that comply with the QC and performance standards detailed in the individual CAM protocols will be assured of “Presumptive Certainty” status.

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*.

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.

“Presumptive Certainty” requirements are to be considered minimum requirements. Efforts that go beyond these minimum requirements (e.g., including additional calibration points, etc.) are considered



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to be compliant with the requirements for “Presumptive Certainty” and need not be identified and discussed as a non-conformance in the laboratory narrative.

A logic diagram detailing the concept of “Presumptive Certainty” and its relationship to MassDEP Policy #WSC-07-350 *MCP Representativeness Evaluations and Data Usability Assessments* is presented in Figure VII A-1. Additional details on the concept and status of “Presumptive Certainty” may be obtained in WSC-CAM-I, *Overview of the Analytical Data Enhancement Process for the Massachusetts Contingency Plan (MCP)* at the following URL:

<https://www.mass.gov/info-details/compendium-of-analytical-methods-cam>

2.2 Summary of Sample Acquisition and Submittal Requirements for “Presumptive Certainty”

2.2.1 Sample Acquisition and Submittal Requirements

Parties seeking “Presumptive Certainty” are required to provide the laboratory with a sufficient weight or volume of sample, in the specified container, properly preserved and within a time period that will not compromise the extraction/analytical holding time for the analysis specified. Sample collection, preservation and holding time information for individual CAM protocols and matrices are described in Appendix VII A-1. In addition, sample collection information and analytical instructions should be clearly documented on a Chain-of-Custody (COC) form that must accompany all samples submitted to the laboratory for analysis in support of MCP decision-making. The COC form must include the following information, if applicable to the samples submitted for analysis:

- Sample identification,
- Sample type,
- Date and time of collection,
- Sample collector’s name,
- Sample preservative,
- Field filtration or other field preparation procedures used, and
- Relinquished and receipt signatures, dates, and times.

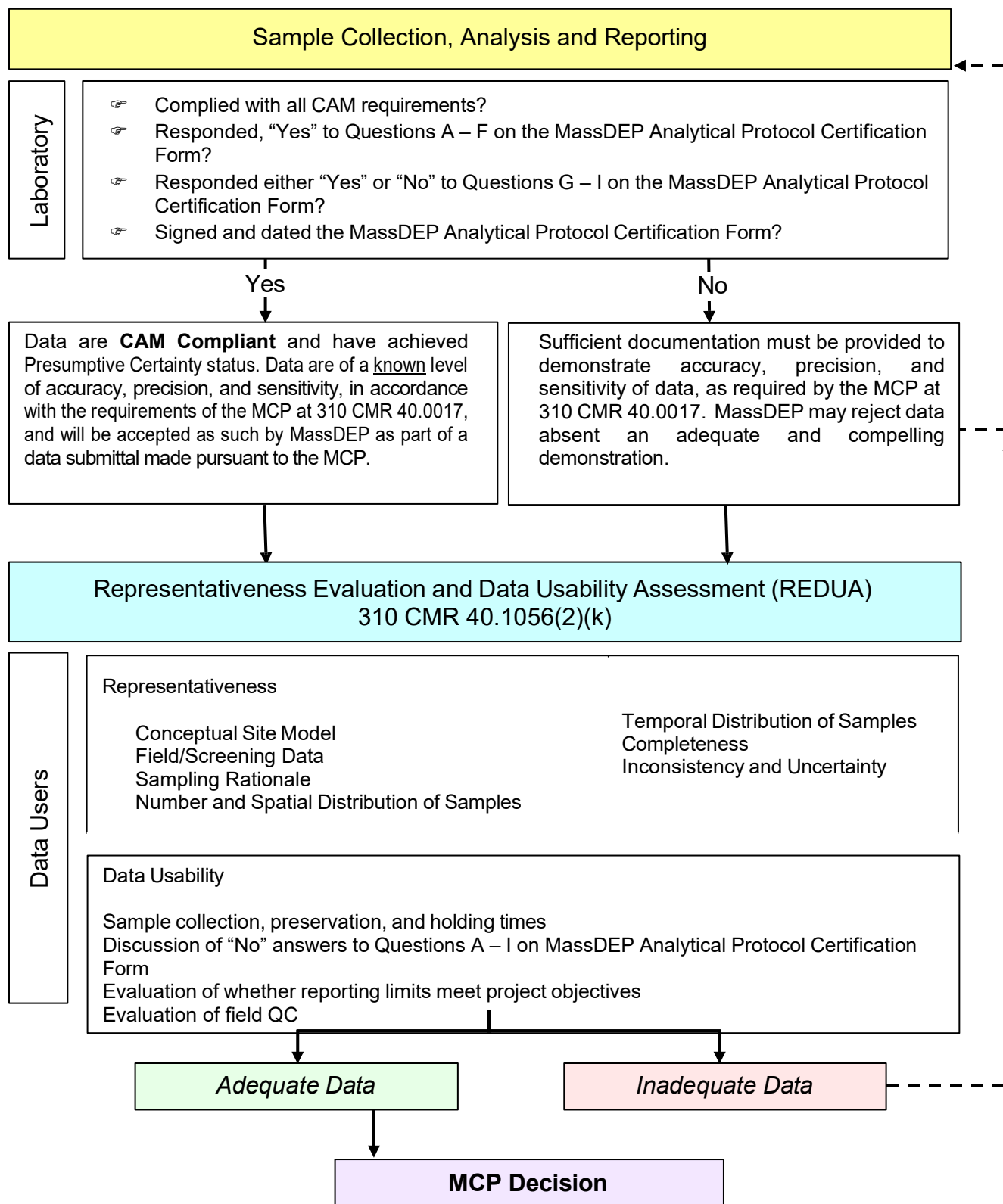
2.2.2 Optional MCP Analytical Services Request Form

In many instances, the information provided with the COC form does not provide adequate instruction to the laboratory for MCP analytical requests, including whether or not “Presumptive Certainty” status is requested. At a minimum, the data user should provide the laboratory with additional information that (1) clearly articulates whether CAM “Presumptive Certainty” status is being requested; (2) affirms that samples were collected in appropriate containers, and properly preserved or require additional laboratory preservation; (3) specifies required analyte lists and reporting limits (RLs)/lower limits of quantitation (LLOQs) or MCP criteria; and (4) identifies any field QC support to be provided by the laboratory. In addition, drinking water samples, as described in Section 2.5, should be identified and specific instruction regarding tentatively identified compound (TIC) reporting and the analysis of contingency field QC samples as described in Table VII A-3 must be provided, as appropriate.



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Figure VII A-1: MassDEP “Presumptive Certainty” and REDUA Concept





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Exhibit VII A-1, Optional MCP Analytical Services Request Form, provides a convenient means for providing this pertinent information to the laboratory. This form, or an equivalent listing of supplemental information, should be provided to the laboratory and may be attached to or included with the COC form for each data set for which MCP analytical services are being requested.

2.3 Use of CAM Protocols

The CAM is a compilation of protocols based on common analytical methods (e.g., EPA's SW-846 Methods, MassDEP's volatile petroleum hydrocarbons [VPH] Method, etc.) routinely used in support of response actions conducted under the MCP. In addition to providing a succinct summary of the required analytical method, the CAM further articulates detailed QC procedures and performance standards, analyte lists, reporting formats, and other methodological elements and details that may not have been specified and/or are cited as discretionary in the original publications (e.g., EPA's SW-846 Methods). Incorporation of all such provisions into a specified analytical method constitutes a "CAM Protocol." All protocols included in the CAM are considered "methods" published by the MassDEP pursuant to the provisions of 310 CMR 40.0017(2). All "CAM Protocols" are available in electronic format at the following URL:

<https://www.mass.gov/info-details/compendium-of-analytical-methods-cam>

2.3.1 Performance Standards for CAM Protocols

Individual CAM Protocols describe detailed method-specific QC requirements and associated performance standards. Compliance with these performance standards is evaluated by the analysis of various batch QC samples (data quality indicators such as laboratory control samples [LCSs], etc.) and the comparison of these analytical results to pre-established ranges of acceptable analytical variability.

While it is not expected that every performance standard will be met for every analytical batch for every method and analyte, it is required that all non-compliances must be identified and discussed in the laboratory narrative. This information must be given due consideration when evaluating overall analytical data usability in support of MCP decision-making.

2.3.2 Analyte Lists for CAM Protocols

It is not necessary to request and report all listed analytes in a CAM Protocol to obtain "Presumptive Certainty." However, 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



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- ✓ 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:

- ✓ 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;
- ✓ Most sites that are known to be contaminated **only** by a release of diesel fuel and/or No. 2 fuel oil (see MassDEP extractable petroleum hydrocarbons [EPH] Method, Section 3.9); 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 method analytes is selected, laboratories must still employ the method-specified QC requirements and performance standards associated with the requested analyte lists.

2.3.3 Reporting Limits or Lower Limits of Quantitation for CAM Protocols

The RL/LLOQ for CAM compliant data is empirically derived directly from the concentration of the lowest non-zero standard in the initial calibration, analyzed under identical conditions as the sample with adjustments for the sample size, extraction concentration factor, percent solids, dilution factors, etc., as required.

Non-detect results may be reported as “< RL/LLOQ.” For example, for an aqueous sample, if the lowest calibration standard for an 8260B analysis for benzene is 2.0 µg/L, then all non-detect results should be reported as < 2.0 µg/L or 2.0 U.

The laboratory may report extrapolated sample concentrations below the RL/LLOQ at the request of the data user. Such data must be qualified as “estimated” or “J.” Reporting of concentrations below the RL/LLOQ is only allowed for the following CAM Protocols:

- Volatile organic compounds (VOCs) and semivolatile organic compounds (SVOCs) by gas chromatography/mass spectrometry (GC/MS) (WSC-CAM-II A and II B)
- Metals by inductively coupled plasma/optical emission spectrometry (ICP/OES) and inductively coupled plasma/mass spectrometry (ICP/MS) (WSC-CAM-III A and III D)
- Mercury by cold vapor atomic absorption (CVAA) (WSC-CAM-III B)
- VPH by GC/MS (WSC-CAM-IV C)
- Perchlorate by electrospray ionization/mass spectrometry (ESI/MS) or liquid chromatography/dual mass spectrometry (LC/MS/MS) (WSC-CAM-VIII B)
- VOCs and air-phase petroleum hydrocarbons (APH) in Air by GC/MS (WSC-CAM-IX A and IX B)
- Per- and polyfluoroalkyl substances (PFAS) by LC/MS/MS (WSC-CAM-X A)



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Data that achieve “Presumptive Certainty” status may not necessarily meet the data quality objectives for sensitivity for some MCP response actions. It is the affirmative obligation of the data user to notify the laboratory of the RL/LLOQ requirements for the project.

2.4 Laboratory Report

When laboratories are directed to follow the CAM Protocols and achieve “Presumptive Certainty” the information specified in Table VII A-1 must be provided in the laboratory report. Individual CAM protocols should be consulted for required QC requirements, associated performance standards and any other protocol-specific reporting elements. It should be noted that WSC-CAM-IX A (MassDEP APH) and WSC-CAM-IX B (TO-15 VOC) have additional sampler-provided reporting requirements. It is required that both reported and non-reported analytical data and information be retained by the laboratory for ten (10) years to facilitate further in-depth review or for audit support.

2.4.1 Laboratory Report Certification Statement

To obtain “Presumptive Certainty” status, every laboratory report must include a certification attesting to compliance with the protocol-specific QC requirements and associated performance standards for all analytical data included in the laboratory report.

The required certification is provided in Exhibit VII A-2. While laboratories are not required to adopt the specific reporting format provided in this exhibit for CAM compliance, all the information and data specified must be succinctly and clearly presented in the alternative laboratory-reporting format. Moreover, the alternative certification format must clearly indicate each and every sample for which the attestations are being made, to be included at the beginning of such submittals.

The analytical report certification includes a series of “Yes” or “No” questions, followed by a statement attesting to the accuracy and completeness of those responses and of the attached laboratory report(s), which is signed by an authorized laboratory representative. In order to achieve a status of “Presumptive Certainty,” it is necessary to answer, “**YES**” to the first six (6) questions (**A-F**). A “**NO**” designation must be fully discussed in an attached laboratory narrative. Although the associated submittal will not have “Presumptive Certainty” due to a “**NO**” designation, the use of the CAM protocols and certification form is still recommended to facilitate review by the data user and MassDEP staff.

Three (3) additional questions (**G-I**) are asked that have relevance to data usability and representativeness considerations. In order to achieve a status of “Presumptive Certainty,” the three (3) additional questions **must be answered**, although it is **not** necessary to respond in the affirmative to preserve the “Presumptive Certainty” option. Once again, a “**NO**” designation must be fully discussed in an attached laboratory narrative.

2.4.1.1 Clarification of MassDEP Analytical Protocol Certification Form Questions A through I

Table VII A-2 provides additional clarification regarding the appropriate laboratory responses to Questions A through I described on the MassDEP Analytical Protocol Certification Form (See Exhibit VII A-2).



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Exhibit VII A-1: Optional MCP Analytical Services Request Form

MCP Analytical Services Request Form <i>Attach to Chain-of-Custody Form for Data Set</i>	
Client Name: _____	Project Name: _____
Project Location: _____	MCP RTN ¹ : _____
Applicable Samples: _____	
General Questions:	
Is MCP Presumptive Certainty status being requested for the referenced data set*? <i>* Laboratory must use approved CAM Protocols</i>	• Yes ² • No
Were all samples that comprise this data set collected in appropriate containers as specified in WSC-CAM-VII A, Appendix VII A-1 for requested analytes?	• Yes • No
Were all samples preserved as specified in WSC-CAM-VII A, Appendix VII A-1 for requested analytes?	• Yes • No
Were all samples placed in a cooler with ice, if required?	• Yes • No
Are any of the soil/sediment samples (or aqueous samples for PFAS) in the data set preserved by freezing, do any of the VOC samples require freezing (< -7°C) by the laboratory (within 48 hours of the time of collection), or do any of the non-VOC samples, where allowed, require freezing (<-10°C) by the laboratory within 24 hours to extend the holding time?	• Yes • No
Should the laboratory report the standard CAM analyte list for the requested analytical protocols?	• Yes • No ³
Should protocol-specific CAM RLs/LLOQs be used for all requested aqueous samples? <i>If lower reporting limits are required, please specify.</i>	• Yes • No
Should protocol-specific CAM RLs/LLOQs be used for all requested soil/sediment/tissue samples? <i>If lower reporting limits are required, please specify.</i>	• Yes • No
Are Matrix Spikes (MS) or MS Duplicates (MSD) required for this data set?	• Yes ⁴ • No
Has adequate sample volume been provided for the MS/MSD?	• Yes • No
Have the samples which require MS or MS Duplicate analysis been identified?	• Yes • No
Are any of the samples in the data set characterized as "drinking water" as described in WSC-CAM-VII A, Section 2.5?	• Yes • No
If YES , samples identified as "drinking water" must be analyzed using CAM Protocols and require the reporting of Tentatively Identified Compounds (TICs), if GC/MS analyses requested.	
Are Field Duplicate Samples provided and identified for all "drinking water" samples*? <i>* Analysis required only if a target analyte is detected above the RL/LLOQ in the original sample.</i>	• Yes • No
Are Trip Blanks provided and identified for all "drinking water" samples submitted for VOCs and VPH*? <i>* Analysis required only if a target analyte is detected above the RL/LLOQ in any of the associated samples.</i>	• Yes • No
Is any alternative, supplemental or non-routine QC required for this data set? <i>(Please specify)</i>	• Yes ⁵ • No
1. MCP Release Tracking Number, as applicable. 2. Laboratory must use approved CAM Protocols. 3. Attach modified analyte list (may include non-routine analytes). 4. Samples that require MS and/or MSD analysis should be designated on the COC. Data user responsible for providing the laboratory with adequate sample volume to prepare MS/MSD samples. 5. Attached description of alternative, supplemental or non-routine QC that is required.	
Signature _____	Date _____



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Table VII A-1 Required Laboratory Report Information

Laboratory Information

Laboratory Name, Address, Phone Number

Client Name, Client Contact, Address, Phone Number

Project Identification

Sample Results Section

Copy of Chain-of-Custody Form

Sample Identification: Field and Laboratory

Sample Matrix

CAM Protocol Reference

Preparation Method Reference

Analytical Method Reference

Analyst Initials

Target Analytes, Reported Concentrations and RL/LLOQ – based upon the lowest calibration standard and adjusted for sample size, % moisture, dilution factors, etc.

Units for soil/sediment/tissue and aqueous samples: (mass/mass [i.e., mg/kg] or mass/volume [i.e., µg/L] – not “ppm” or “ppb” – **soil/sediments must be reported on a dry weight basis; tissues must be reported on a wet weight basis**).

Units for air samples: ppbV or mass/volume (i.e., µg/m³).

Data Qualifiers, if applicable

Date of Collection

Date and Time of Preservation or Filtration, if performed by the laboratory

Date and Time Low-level VOC soil/sediment samples placed in the freezer, if applicable

Date of Preparation, if applicable

Date of Methylation, for herbicides

Date and Time of Analysis

Dilution/Concentration Factors

% Moisture or % Solids for solid samples

Air Samples: Vacuum of canister upon receipt at the laboratory

Air Samples: Flow controller calibration relative percent difference (RPD)

Air Samples: Canister and flow controller serial numbers, pre-sampling canister vacuums.

Required Sample- and Batch-Specific Quality Control Information

Method Blank Results

Surrogate Spike Recoveries (organics [non-PFAS] only)

Extracted Internal Standard Recoveries (PFAS only)

Laboratory Control Sample (LCS) and Ongoing Precision and Recovery (OPR) Recoveries

LCS Duplicate Recoveries and RPDs

Matrix Spike Recoveries, if applicable

Matrix Spike Duplicate Recoveries; if applicable

MS/MSD RPDs, if applicable

Laboratory Matrix Duplicate RPDs, if applicable

Air Samples: Media Certification results

Analytical holding time and preservation information, including temperature on receipt, as applicable

VPH only: Summary of column used (manufacturer, column name, length, ID, film thickness)

VPH only: Summary of trap used (manufacturer, trap contents)



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Exhibit VII A-2: MassDEP Analytical Protocol Certification Form

MassDEP Analytical Protocol Certification Form					
Laboratory Name:			Project #:		
Project Location:			RTN:		
This Form provides certifications for the following data set: list Laboratory Sample ID Number(s):					
Matrices: ☐ Groundwater/Surface Water/Wastewater/Landfill Leachate ☐ Soil/Sediment ☐ Biosolids ☐ Tissue ☐ Drinking Water ☐ Air ☐ Other:					
CAM Protocol (check all that apply below):					
8260 VOC CAM II A ☐	7470/7471 Hg CAM III B ☐	MassDEP VPH CAM IV A ☐ PID/FID CAM IV C ☐ GC/MS	8081 Pesticides CAM V B ☐	7196 Hex Cr CAM VI B ☐	MassDEP APH CAM IX A ☐
8270 SVOC CAM II B ☐	7010 Metals CAM III C ☐	MassDEP EPH CAM IV B ☐	8151 Herbicides CAM V C ☐	8330 Explosives CAM VIII A ☐	TO-15 VOC CAM IX B ☐
6010 Metals CAM III A ☐	6020 Metals CAM III D ☐	8082 PCB CAM V A ☐	9014 Total Cyanide/PAC CAM VI A ☐	6860 Perchlorate CAM VIII B ☐	1633 PFAS CAM X A ☐
Affirmative Responses to Questions A through F are required for "Presumptive Certainty" status					
A	Were all samples received in a condition consistent with those described on the Chain-of-Custody, properly preserved (including temperature) in the field or laboratory, and prepared/analyzed within method holding times?				☐ Yes ☐ No ¹
B	Were the analytical method(s) and all associated QC requirements specified in the selected CAM protocol(s) followed?				☐ Yes ☐ No ¹
C	Were all required corrective actions and analytical response actions specified in the selected CAM protocol(s) implemented for all identified performance standard non-conformances?				☐ Yes ☐ No ¹
D	Does the laboratory report comply with all the reporting requirements specified in CAM VII A, "Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data"?				☐ Yes ☐ No ¹
E1	VPH, EPH, and APH Methods only: Was each method conducted without significant modification(s)? (Refer to the individual method(s) for a list of significant modifications).				☐ Yes ☐ No ¹
E2	PFAS only: Were aqueous samples extracted without filtering?				☐ Yes ☐ No ¹
F	Were all applicable CAM protocol QC and performance standard non-conformances identified and evaluated in a laboratory narrative (including all "No" responses to Questions A through E)?				☐ Yes ☐ No ¹
Responses to Questions G, H and I below are required for "Presumptive Certainty" status					
G	Were the reporting limits/lower limits of quantitation at or below all CAM reporting limits/lower limits of quantitation specified in the selected CAM protocol(s)?				☐ Yes ☐ No ¹
Data User Note: Data that achieve "Presumptive Certainty" status may not necessarily meet the data usability and representativeness requirements described in 310 CMR 40.1056 (2)(k) and WSC-07-350.					
H	Were all QC performance standards specified in the CAM protocol(s) achieved?				☐ Yes ☐ No ¹
I	Were results reported for the complete analyte list specified in the selected CAM protocol(s)?				☐ Yes ☐ No ¹
¹ All negative responses must be addressed in an attached laboratory narrative.					
I, the undersigned, attest under the pains and penalties of perjury that, based upon my personal inquiry of those responsible for obtaining the information, the material contained in this analytical report is, to the best of my knowledge and belief, is accurate and complete.					
Signature: _____			Position: _____		
Printed Name: _____			Date: _____		



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Table VII A-2: Questions A through I Clarifications

Question	Clarifications
A	Respond "YES" only if (1) the physical characteristics and descriptions documented on the COC form accompanying the referenced data set were confirmed on initial inspection of all samples (e.g., did the sample IDs on the labels match the IDs on the COC form?); (2) all samples were properly preserved (in the field or in the lab) for the analyses requested; (3) the temperature on receipt and all holding times are consistent with requirements specified in Appendix VII A-1 for the requested CAM protocol(s); and (4) all preservation/preparation/analysis holding times specified in Appendix VII A-1 for the requested CAM protocol(s) were achieved for all samples. Otherwise respond "NO" and narrate. Notes: (a) If samples are delivered to the laboratory on the same day of collection and temperatures are outside of the acceptance criteria, respond "YES" if samples are received on ice, but narrate. (b) If the proper field QC samples are not delivered with drinking water samples, respond "NO." (c) If soil/sediment samples that are preserved in methanol for VOC or VPH analysis are not completely covered by the methanol preservative, respond "NO." (d) If soil/sediment samples collected for VPH are outside of the 1:1 $\pm 25\%$ range for sample/methanol ratio, respond "YES," but narrate this deviation. (e) If soil/sediment samples for hexavalent chromium are collected in the same jar as other analytical parameters and the digestion of hexavalent chromium is not conducted prior to other parameters, respond "NO."
B	Respond "YES" only if (1) the analytical method specified in the CAM protocol(s) was followed and (2) all required CAM protocol-specific QC parameters (e.g., calibration curves, blanks, LCSs, LCS Duplicates, etc.) were utilized without exception. Otherwise respond "NO" and narrate. In addition, for PFAS only: Respond "NO" if calibration standards did not include required linear and branched isomers for 11 PFAS identified in the EPA method (PFOA, PFOS, PFHxS, PFOSA, PFNA, NMeFOSAA, NEtFOSAA, NMeFOSA, NEtFOSA, NMeFOSE, NEtFOSE). Respond "NO" if the extracted internal standard used for quantitation of a specific PFAS does not agree with the requirements from Table 10 of EPA Method 1633. Respond "NO" and narrate if additional extracted internal standard was added to a sample extract which was diluted.
C	Respond "YES" only if all required CAM protocol-specific corrective actions and analytical response actions were implemented for all identified performance standard non-conformances. Otherwise respond "NO" and narrate.
D	Respond "YES" only if the laboratory report complies with all requirements specified in Section 2.4 of CAM VII A, <i>Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted Under the MCP</i>. For example, does the report contain required (1) laboratory information, (2) sample results, (3) sample- and batch-specific QC data, and (4) a signed MassDEP Analytical Protocol Certification Form? Otherwise respond "NO" and narrate.



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Table VII A-2: Questions A through I Clarifications

Question	Clarifications
E1	Respond "NO" and narrate if significant modifications were used for the EPH, VPH, and/or APH methods. Significant modifications are summarized in the individual methods as follows: VPH by photoionization detector (PID)/flame ionization detector (FID) and VPH by GC/MS: Section 11.3.1, EPH: Section 11.3.1.1 and APH: Section 11.1.2. Leave blank if none of these methods were performed.
E2	PFAS only: Respond "NO" and narrate if aqueous sample was filtered by the laboratory prior to extraction
F	Respond "YES" only if (1) all QC and performance standard non-conformances for the CAM protocols referenced in this certification form were identified and evaluated in the laboratory narrative or (2) no QC or performance standard non-conformances were identified. Otherwise respond "NO" and narrate. Refer to Section 2.4.2 for narrative content requirements. Note: Any non-compliance with the field QC sample requirements for drinking water samples is considered a performance standard non-conformance and must be identified and discussed in the laboratory narrative.
G	Respond "YES" only if RLs/LLOQs for each analyte in each CAM protocol were at or below the CAM RLs/LLOQs. Otherwise respond "NO" and narrate. Responding "NO" to Question G does not preclude "Presumptive Certainty." PFAS only: If "NO", provide an explanation in the laboratory narrative if a reduced sample volume was used for aqueous samples.
H	Respond "YES" only if all the protocol-specific QC performance standards specified in the CAM protocol(s) were achieved. Otherwise respond "NO" and narrate. Note: If metals, cyanide, or hexavalent chromium analysis was performed on soil/sediment samples and a matrix spike was not submitted by the client, respond "NO." Responding "NO" to Question H does not preclude "Presumptive Certainty."
I	Respond "YES" only if the complete protocol-specific analyte-list was reported. Otherwise respond "NO" and narrate. Note: For the EPH Method, 17 polynuclear aromatic hydrocarbons (PAHs) are defined as "Target PAH Analytes" (see Section 3.3 and Table 2 of the EPH method). Included in this comprehensive list are a subset of 4 " Diesel PAH Analytes " (naphthalene, 2-methylnaphthalene, phenanthrene, and acenaphthene). For most sites that are known to be contaminated by a release of diesel and/or #2 fuel oil only , Diesel PAH Analytes will be the only Target PAH Analytes of interest. For purposes of CAM compliance, if only the "Diesel PAH Analytes" are requested and reported, respond "NO" and narrate. Responding "NO" to Question I does not preclude "Presumptive Certainty."



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2.4.2 Laboratory Narrative

The purpose of the laboratory narrative is to provide a means of communication (and documentation) between the laboratory and the data user. The objective of this **required** communication is to clearly and concisely inform the data user of all protocol-specific QC requirements or performance standard non-conformances associated with the reported data set. The scope of the narrative is to include all relevant information so that the data user will be able to make informed decisions concerning the accuracy, precision and sensitivity of the analytical data reported.

The laboratory narrative is to be in the form of an exception report where only the anomalies related to project- and/or protocol-specific performance standards and QC requirements are disclosed and discussed.

As applicable and appropriate, the following specific information **must** be provided in the laboratory narrative:

- Problems with sample condition, preservatives, and/or temperature on receipt;
- Lack of sampler-provided COC form;
- Qualifications regarding the identification of TICs, where required and applicable;
- Non-routine QC requirements, if provided to the laboratory;
- Description/discussion of non-conformances that resulted in a “NO” response on the MassDEP Analytical Protocol Certification Form (Exhibit VII A-2);
- Non-conformances with protocol-specific QC requirements and/or performance standards (e.g., blanks and LCS), as well as non-conformances for parameters not required to be provided in the laboratory report (e.g., calibration);
- Method modifications and corrective actions, if applicable;
- Description of procedures used for elevated total suspended solids (TSS) in aqueous samples submitted for PFAS;
- Reasons for dilutions when not due to target analyte concentrations;
- Notation if PFAS extract is over-diluted whereby additional extracted internal standard is added to the diluted extract;
- Notation if the PFAS extracted internal standard used for quantitation of a specific PFAS does not agree with the requirements from Table 10 of EPA Method 1633;
- Possible presence of weathered polychlorinated biphenyl (PCB) Aroclors or PCB congeners when reporting non-detect results for PCB Aroclors, if applicable;
- Notation that reduced analyte list requested by data user, if applicable;
- Holding time exceedances; and
- Obvious discrepancies in sample description information recorded on the COC form supplied by the sampler, as applicable.

If there are no exceptions or analytical non-conformances to report, the narrative must include (and may consist solely of) a statement that documents that there are no relevant protocol-specific QC and/or performance standard non-conformances to report.



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2.4.3 Completed Chain-of-Custody

A copy of the COC form that was submitted to the laboratory with the samples must be appended to the laboratory report. If no COC form is provided, the laboratory narrative must document this critical non-compliance.

2.5 Special Consideration for Drinking Water Samples

For the purposes of this document, drinking water samples are defined to be samples obtained from a Public Water Supply or Private Water Supply Well, as those terms are defined in the MCP at 310 CMR 40.0006. Examples that would be considered drinking water in this context include samples taken directly from a tap, as well as water collected from a private well in the delivery system prior to the tap. Conversely, examples that would ***not*** be considered “drinking water” in this context include water collected from a well that is subsequently treated prior to consumption.

The MassDEP drinking water program under the provisions of 310 CMR 22.00 directly regulates Public Water Supplies. Private Water Supply Wells are not regulated by 310 CMR 22.00, but are subject to the provisions of the MCP if and when they become contaminated by a release of oil or hazardous materials to the environment.

The CAM Protocols and provisions do not apply to drinking water samples analyzed in accordance with the provisions and requirements of 310 CMR 22.00. The CAM Protocols and provisions would apply to parties seeking “Presumptive Certainty” status for drinking water sample data being submitted to MassDEP under the provisions of the MCP.

2.5.1 Use of EPA Drinking Water Methods vs. CAM Protocols

310 CMR 22.00 generally requires the use of specific EPA drinking water methods, such as the “500” series for organics and “200” series for metals. While data from such methods may be included in an MCP submittal to MassDEP, such data will not achieve “Presumptive Certainty” status. Accordingly, MassDEP would recommend the use of the CAM Protocols for all samples obtained from Private Water Supply Wells, as well as all samples obtained from a contaminated Public Water Supply as part of an assessment being conducted under the MCP.

2.5.2 Chain-of-Custody/MCP Analytical Services Request Form

It is strongly recommended that drinking water samples be identified as such on the COC form and/or Optional MCP Analytical Services Request Form submitted with the samples, so that the laboratory will be apprised of the need for special field QC, TIC, and/or RL/LLOQ requirements.

2.5.3 Field QC for Drinking Water Samples

To achieve a “Presumptive Certainty” status, drinking water samples collected for MCP purposes must be accompanied by the field QC samples specified in Table VII A-3. Conformance with these specific field QC requirements for drinking water samples will ensure confidence in the results should an oil or hazardous material be detected in water that is used for human consumption.

For drinking water samples collected for MCP purposes, field duplicates must be collected for all samples but need only be analyzed if an oil or hazardous material is detected in the primary sample above the analyte’s RL/LLOQ. For VOCs and VPH, a trip blank must also be collected but need only be analyzed



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if an oil or hazardous material is detected in the primary or duplicate sample above the analyte's RL/LLOQ. **It should be noted that compliance with the field QC sample requirements for drinking water samples described in Table VII A-3 is considered a performance standard for "Presumptive Certainty" status.** Any non-compliance with the field QC sample requirements for drinking water samples described in Table VII A-3 **must** be identified and discussed in the laboratory narrative and data usability assessment.

2.5.4 Reporting and Evaluation of Tentatively Identified Compounds (TICs)

For drinking water samples, parties are required to instruct the laboratory to report TICs when GC/MS methods are used in the analysis of the samples. If identified, these compounds must be reported in the laboratory narrative, as described in Section 2.4.2.

All reported concentrations of TICs are by definition estimated values. The party conducting response actions may either accept the estimated TIC concentration without further qualification, or improve the identification and the accuracy of the estimated concentration by post-calibration, re-sampling and/or re-analysis with a more appropriate analytical method.

If the presence of the TIC at the concentration reported by the laboratory appreciably changes the overall risk posed by the site or the utility of the potential remedial measures under consideration, MassDEP recommends (and may require) the latter option be exercised.

2.5.5 RLs/LLOQs

In some cases, achieving CAM RLs/LLOQs for a specific analyte in a drinking water sample may not be adequate to meet site-specific data quality objectives. While such data will achieve "Presumptive Certainty" status, the submittal will likely be deemed unacceptable in a subsequent data usability evaluation. Therefore, it is essential that RL/LLOQ "needs" for drinking water samples be identified and evaluated prior to sample collection and submission, so that special sampling and/or analytical techniques can be used to achieve required RLs/LLOQs, if necessary.



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Table VII A-3: Minimum Field QC Sample Frequency for Drinking Water Samples

Analytes	Method(s)	QC Element		
		Matrix Spike (MS) ^a	Field Duplicate ^b	Trip Blank ^c
VOCs & VPH Target Analytes	8260B/CAM II A MassDEP VPH by GC/MS/CAM IV C	Not Mandatory ^d	If analyte detected	1 per cooler
SVOCs; Pesticides; PCBs; Herbicides; Nitroaromatics; EPH Target Analytes	8270D/CAM II B, 8081B/CAM V B, 8082A/CAM V A, 8151A/CAM V C, 8330A/CAM VIII A	Not Mandatory ^d	If analyte detected	Not Mandatory ^d
VPH Ranges	MassDEP VPH/CAM IV A and IV C	Not Mandatory ^d	If analyte detected	1 per cooler
EPH Ranges	MassDEP EPH/CAM IV B	Not Mandatory ^d	If analyte detected	Not Mandatory ^d
Metals	6010D, 6020B & 7000B Methods/CAM III	1 per 20 samples	If analyte detected	Not Mandatory ^d
Perchlorate	6860/CAM VIII B	1 per 20 samples	If analyte detected	Not Mandatory ^d
Hexavalent Chromium	7196A/CAM VI B	1 per 20 samples	If analyte detected	Not Mandatory ^d
Total Cyanide & Physiologically Available Cyanide (PAC)	9014 and PAC/CAM VI A	1 per 20 samples	If analyte detected	Not Mandatory ^d
PFAS	1633A/CAM X A	Not Mandatory ^d	If analyte detected	Not Mandatory ^d

^a Matrix Spikes must be selected that represent the most significant exposure points to human health and the environment.

^b Field Duplicate MUST be analyzed if one or more analytes are detected in the primary sample above the RL/LLOQ. Duplicate samples MUST be collected for every drinking water sample for such purposes. Exception: If samples are being collected as part of an on-going monitoring program, this requirement must be fulfilled only on the first round.

^c Trip Blank MUST be analyzed if one or more analytes are detected in the primary sample above the RL/LLOQ. A Trip Blank MUST accompany all drinking water samples for such purposes.

^d On a site and project-specific basis, the use of one or more of these and/or other QC elements (e.g. equipment rinsate blanks, etc.) designated "Not Mandatory" may be advisable and/or necessary to demonstrate usability of the data, and/or to determine if the data are biased high due to contamination by sampling equipment/storage conditions. See Section 3.3.



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3.0 Data Usability Requirements Under the MCP

Overall representativeness and usability of data produced using specific CAM protocols must be evaluated for compliance with project-specific data quality objectives pursuant to the requirements of 310 CMR 40.1056(2)(k) and as further described in the MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

This section provides an overview of relevant data quality evaluation guidance for all parties whether or not the “Presumptive Certainty” option as described in Section 2.0 is selected.

3.1 MCP Performance Standards for Data Quality

Under the provisions of 310 CMR 40.0017(1), “Any person undertaking response actions under the provisions of this Contingency Plan shall ensure that analytical and environmental monitoring data used in support of recommendations, conclusions, or LSP Opinions with respect to assessment, removal, or containment actions is scientifically valid and defensible, and of a level of precision and accuracy commensurate with its stated or intended use.”

The level of QA/QC for these activities should be commensurate with the complexity of the response action conducted at a disposal site, the potential risk posed to human health and the environment by the contaminants of concern, and the intended use of the data.

3.2 CAM Compliant Data, Data Representativeness and Usability

CAM-compliant analytical data (data with “Presumptive Certainty”) are of known accuracy, precision and sensitivity and possess the data quality attributes described in Section 2.1, Overview of the “Presumptive Certainty” Process.

Analytical data with “Presumptive Certainty” status may be used without reservation in a Representativeness Evaluation and Data Usability Assessment that provides:

- (1) An evaluation of whether the RL/LLOQ for the analyses are sensitive enough to support the project-specific objectives, and
- (2) A discussion of how the uncertainty associated with any identified analytical non-conformances may affect the overall accuracy, precision and sensitivity of the analytical data and the achievement of project-specific objectives.

Data that meet all method- and project-specific data quality objectives may then be used to support a Permanent or Temporary Solution pursuant to 310 CMR 40.1056(2)(k) and 40.1057(2)(k), respectively.

3.3 Summary of Field Sampling QA/QC

Considerations regarding the necessary level of field QC should be premised on the governing regulatory jurisdictions and on the intended use of the data. This evaluation is a prospective activity that should be conducted prior to the initiation of any field sampling; it is an integral component of the project planning process. Field sampling activities should incorporate methods and/or measures to allow for assessment of overall data quality objectives using appropriate data quality indicators as described in Table VII A-4



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and in MassDEP's Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

Table VII A-4: Summary of Data Quality Indicators

Data Quality Indicator	Description
Matrix Spikes	a direct measurement of matrix effects and overall measurement of data accuracy
Field Duplicates	a measure of sampling precision, representativeness, site heterogeneity, and laboratory operations (when submitted as blind samples)
Equipment Blanks	a measure of "false positive" contamination during sample acquisition and/or storage
Trip Blanks (VPH and VOC analyses only)	the assessment of field accuracy and representativeness and a measure of "false positive" contamination during sample acquisition and/or storage
Double-Blind Spikes (Prepared by Third Party)	quality control program sample with known concentration of contaminants submitted to laboratory as an overall measurement of data accuracy

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APPENDX VII A-1

SAMPLE COLLECTION, PRESERVATION AND HANDLING PROCEDURES FOR ENVIRONMENTAL SAMPLES ANALYZED IN SUPPORT OF MCP RESPONSE ACTIONS



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**Sample Collection, Preservation, and Handling Procedures for
Volatile Organic Compound Analyses**

Aqueous Samples				
Matrix	Analyte	Container ¹	Preservative ^{2,3}	Holding Time ⁵
Aqueous Samples, with no Residual Chlorine	Most Volatile Organic Compounds	(2) x 40-mL VOC vials w/ Teflon-lined septa screw caps and protect from light.	Adjust pH to < 2.0 by addition of HCl or NaHSO ₄ to container before sampling. Cool to ≤ 6°C but not frozen. No headspace.	14 days
	MTBE or other fuel oxygenates with heated purge-and-trap (≥40°C) sample introduction only	(2) x 40-mL VOC vials w/ Teflon-lined septa screw caps and protect from light.	0.7 g of trisodium phosphate dodecahydrate (TSP) per 40 ml. Verify pH >11.0. Cool to ≤ 6°C but not frozen. ⁴ No headspace.	14 days
	Volatile organics susceptible to acid hydrolysis, abiotic degradation or loss during storage	(2) x 40-mL VOC vials w/ Teflon-lined septa screw caps and protect from light.	Cool to ≤ 6°C but not frozen. No headspace.	Analyze ASAP but not more than 7 days
Aqueous, with Residual Chlorine	<u>Presence of chlorine residual is usually associated with drinking water samples.</u> Collect sample in at least two (2) x 40-mL VOC vials w/ Teflon-lined septa screw caps containing either 25 mg of Ascorbic Acid or 3 mg of Sodium Thiosulfate. If Residual Chlorine > 5 mg/L, additional dechlorination agent may be required After dechlorination is confirmed, preserve as above based on compound classes. No headspace.			
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 during shipping and/or analysis.				
² Preservation of samples by acidification to pH <2.0 and analysis within 14 days is considered a suitable preservation technique for samples not expected to contain reactive contaminants of concern.				
³ 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 6°C are acceptable.				
⁴ TSP may also be used to preserve samples for BTEX and/or VPH analysis (i.e., it would not be necessary to obtain samples in separate vials).				
⁵ As per Appendix IV of MassDEP Policy # WSC-07-350, <i>MCP Representativeness Evaluations and Data Usability Assessments</i> , 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.				



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**Sample Collection, Preservation, and Handling Procedures for
Volatile Organic Compound Analyses**

Soil, Sediment and Waste Samples

Matrix	Container ^a	Preservation ^{1,2,3}	Holding Time ^{1,4}
Soil/Sediment Samples High-Level Analysis	Extrude soil/sediment sample directly into a pre-weighed vial* w/ Teflon-lined septa screw caps: Vials must contain 1 mL purge-and-trap grade methanol for every gram soil/sediment. *(1) x 60-mL vial or (1) x 40-mL vial	1 mL methanol for every gram soil/sediment; add methanol before or at time of sampling; Cool to $\leq 6^{\circ}\text{C}$ but not frozen; protect from light.	14 days
	EnCore® samplers ⁵ or other suitable coring device	Cool to $\leq 6^{\circ}\text{C}$ (but not frozen) in field; 48 hours from date collected until methanol preservation (1 mL methanol for every gram soil/sediment).	
Soil/Sediment Samples Low-Level Analysis by Closed-System Purge-and- Trap Process (SW-846 Method 5035A)	5 g EnCore® samplers ⁵ or other suitable coring device.	Cool to $\leq 6^{\circ}\text{C}$ in field; 48 hours from date collected until extrusion in reagent water followed by freezing ($< -7^{\circ}\text{C}$) or analysis within 48 hours of sample collection (see Note 2). <u>Alternatively</u> , samples may be frozen to $< -7^{\circ}\text{C}$ in the field using gel packs.	14 days ⁷
	Extrude 5 grams of sample directly into (2) x pre-weighed 40 ml VOC vials containing 5 mL of reagent water (with or without chemical preservation; see Note 2) and a Teflon-coated magnetic stir bar ⁶ .	Cool to $\leq 6^{\circ}\text{C}$ in field and deliver to laboratory for freezing ($< -7^{\circ}\text{C}$) or analysis, both within 48 hours of sample collection. <u>Alternatively</u> , samples may be frozen to $< -7^{\circ}\text{C}$ in the field using gel packs.	
Waste Samples	Collect sample in one (1) x 500 mL amber wide mouth jar with a Teflon lined screw cap.	No special preservation required	14 days



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^aThe 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 during shipping and/or analysis. Caution: samples to be frozen should not be stored vertically. These samples should be stored horizontally or at least at a 45-degree angle to avoid breakage from expansion.

¹As per Appendix IV of MassDEP Policy # WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, if the holding time is exceeded by >2x the allowable holding time or if soil/sediment samples are not properly preserved, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias.

²A number of acceptable alternative preservation techniques requiring close communication with the receiving laboratory that require field cooling ($\leq 6^{\circ}\text{C}$) with subsequent laboratory preservation (freezing, methanol, NaHSO_4 , etc.) and/or expedited analysis (48 hours) are presented in Appendix A, "Collection and Preservation of Aqueous and Solid Samples for Volatile Organic Compounds (VOC) Analyses" of the document entitled, "Closed System Purge-and-Trap and Extraction for Volatile Organics In Soil and Waste Systems", an updated version of SW-846 Method 5035A published by US EPA In July 2002. <https://www.epa.gov/hw-sw846/validated-test-method-5035a-closed-system-purge-and-trap-and-extraction-volatile-organics>

³If 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 6°C are acceptable.

⁴Holding time is calculated from the time of sample collection and only applies to samples that have been frozen and chemically preserved.

⁵EnCore® Sampler may not be suitable for certain soil types; refer to guidance in SW-846 Method 5035A.

⁶Not required if closed system purge-and-trap device employs a means of stirring the sample other than a magnetic stirrer.

⁷Any samples which are frozen must be analyzed within 48 hours of thawing.



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Additional Sample Handling and Preservation Notes:

Aqueous Samples:

1. The most common preservation technique for aqueous samples analyzed for VOCs is the addition of HCl to the container prior to sampling (pH to < 2.0) and cooling to $\leq 6^{\circ}\text{C}$. Because of their reactivity, solubility and/or volatility, alternative preservation techniques may be required for some classes of analytes (reactive, MTBE and other fuel oxygenates, etc.). In the unusual circumstance that contaminants of concern at a disposal site require mutually exclusive preservation techniques (acid preservation/with cooling for BTEX and no acid preservation/with cooling for reactive compounds), separate sampling containers to accommodate the different preservation techniques may be required. In all cases the selection of preservation technique for samples analyzed for VOCs should be based on the data quality objectives of the sampling program.

2. If effervescence occurs upon addition of HCl, samples should be collected without the acid preservative. Where acid preservation is not used, the analysis holding time is seven (7) days from date collected to date analyzed.

Low-Level and High-Level Solid Samples:

An extra aliquot of sample must be collected in a 4 oz. glass jar with no preservative so that the laboratory can perform a percent solids analysis. If the same sample is being submitted to the laboratory for additional analyses which require no preservative, the percent solids analysis can be measured using an aliquot from these sampling containers. Otherwise, a separate bottle will be required.



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Sample Collection, Preservation, and Handling Procedures for Semivolatile Organic Compound Analyses

Matrix	Container ¹	Preservation ⁷	Holding Time ^{3,6}
Aqueous Samples, with no Residual Chlorine	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Cool to $\leq 6^{\circ}\text{C}$ but not frozen	7 days to extraction; 40 days from extraction to analysis ⁴
Aqueous Samples, with Residual Chlorine ⁵	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Add 3-mL 10% sodium thiosulfate solution per gallon (or 0.008%) ⁵ . Addition of sodium thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to $\leq 6^{\circ}\text{C}$ but not frozen.	7 days to extraction; 40 days from extraction to analysis ⁴
Soil/Sediment Samples	(1) 8-oz. amber glass jar w/ a Teflon-lined screw cap ²	Cool to $\leq 6^{\circ}\text{C}$ ²	14 days to extraction; 40 days from extraction to analysis ^{2,4}
Waste Samples	Collect sample in one (1) x 500 mL amber wide mouth jar with a Teflon-lined screw cap.	Cool to $\leq 6^{\circ}\text{C}$	14 days to extraction; 40 days from extraction to analysis ⁴

¹The 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 during shipping and/or analysis.

²Alternatively, soil/sediment samples for SVOC analyses may be held for up to one (1) year if frozen within 24 hours of collection at $<-10^{\circ}\text{C}$. 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 or extraction must be commenced within 14 days of thawing. Once the thawing process begins, samples must be kept at $0-6^{\circ}\text{C}$ until extraction.

³Holding time begins from time of sample collection or date thawed (see note #2 above).

⁴SVOC sample extracts must be stored at $\leq 6^{\circ}\text{C}$, protected from light, and stored in sealed vials (e.g., screw-cap or crimp-capped vials) with un-pierced PTFE-lined septa. See SW-846 Method 8270E, Section 8.2.

⁵Presence of chlorine residual is usually associated with drinking water samples. Confirm dechlorination. If residual chlorine $>5\text{ mg/L}$, additional dechlorination agent may be required.

⁶As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, if the holding time is exceeded by $>2\times$ the allowable holding time, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias.

⁷If 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 6°C are acceptable.



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**Sample Collection, Preservation, and Handling Procedures for
Polychlorinated Biphenyl Analyses**

Matrix	Container ¹	Preservation ⁷	Holding Time ^{3,6}
Aqueous Samples, with no Residual Chlorine	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Cool to $\leq 6^{\circ}\text{C}$ but not frozen	1 year to extraction; 40 days from extraction to analysis ⁵
Aqueous Samples, with Residual Chlorine ⁴	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Add 1-mL 10% sodium thiosulfate solution per container (or 0.008%) ⁴ . Addition of thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to $\leq 6^{\circ}\text{C}$ but not frozen.	1 year to extraction; 40 days from extraction to analysis ⁵
Soil/Sediment Samples	(1) 8-oz. amber glass jar w/ a Teflon-lined screw cap ²	Cool to $\leq 6^{\circ}\text{C}$ or freeze at <-10 $^{\circ}\text{C}$ ²	1 year to extraction; 40 days from extraction to analysis ⁵
Waste Samples	Collect sample in one (1) x 500 mL amber wide mouth jar with a Teflon-lined screw cap.	No special preservation required	1 year to extraction; 40 days from extraction to analysis ⁵

¹The 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 during shipping and/or analysis.

²If frozen, 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. Once the thawing process begins, samples must be kept at $0-6^{\circ}\text{C}$ until extraction.

³Holding time begins from time of sample collection or date thawed (see note #2 above).

⁴Presence of chlorine residual is usually associated with drinking water samples. Confirm dechlorination. If residual chlorine $>5\text{ mg/L}$, additional dechlorination agent may be required.

⁵PCB sample extracts must be stored at 4°C , protected from light, and stored in sealed vials (e.g., screw-cap or crimp-capped vials) with un-pierced PTFE-lined septa.

⁶As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, if the holding time is exceeded by $>2\times$ the allowable holding time, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias.

⁷If 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 6°C are acceptable.



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)

Sample Collection, Preservation, and Handling Procedures for Chlorinated Pesticide Analyses

Matrix	Container ¹	Preservation ⁷	Holding Time ^{3,6}
Aqueous Samples, with no Residual Chlorine	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Cool to $\leq 6^{\circ}\text{C}$ but not frozen	7 days to extraction; 40 days from extraction to analysis ⁵
Aqueous Samples, with Residual Chlorine ⁴	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Add 1-mL 10% sodium thiosulfate solution per container (or 0.008%) ⁴ . Addition of thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to $\leq 6^{\circ}\text{C}$ but not frozen.	7 days to extraction; 40 days from extraction to analysis ⁵
Soil/Sediment Samples	(1) 8-oz. amber glass jar w/ a Teflon-lined screw cap ²	Cool to $\leq 6^{\circ}\text{C}$ ²	14 days to extraction; 40 days from extraction to analysis ^{2,5}
Waste Samples	Collect sample in one (1) x 500 mL amber wide mouth jar with a Teflon-lined screw cap.	No special preservation required	14 days to extraction; 40 days from extraction to analysis ⁵

¹The 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 during shipping and/or analysis.

²Alternatively, soil/sediment samples for chlorinated pesticide analyses may be held for up to one (1) year if frozen within 24 hours of collection at $<-10^{\circ}\text{C}$. 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 or extraction must be commenced within 14 days of thawing. Once the thawing process begins, samples must be kept at $0-6^{\circ}\text{C}$ until extraction.

³Holding time begins from time of sample collection or date thawed (see note #2 above).

⁴Presence of chlorine residual is usually associated with drinking water samples. Confirm dechlorination. If residual chlorine $>5\text{ mg/L}$, additional dechlorination agent may be required.

⁵Pesticide sample extracts must be stored at $\leq 6^{\circ}\text{C}$, protected from light, and stored in sealed vials (e.g., screw-cap or crimp-capped vials) with un-pierced PTFE-lined septa.

⁶As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, if the holding time is exceeded by $>2\times$ the allowable holding time, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias.

⁷If 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 6°C are acceptable.



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Sample Collection, Preservation, and Handling Procedures for Chlorinated Herbicide Analyses

Matrix	Container ¹	Preservation ⁷	Holding Time ^{3,6}
Aqueous Samples, with no Residual Chlorine	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Cool to $\leq 6^{\circ}\text{C}$ but not frozen	7 days to extraction; 28 days to methylation; analysis immediately after methylation ⁵
Aqueous Samples, with Residual Chlorine ⁴	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Add 1-mL 10% sodium thiosulfate solution per container (or 0.008%) ⁴ . Addition of thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to $\leq 6^{\circ}\text{C}$ but not frozen.	7 days to extraction; 28 days to methylation; analysis immediately after methylation ⁵
Soil/Sediment Samples	(1) 8-oz. amber glass jar w/ a Teflon-lined screw cap ²	Cool to $\leq 6^{\circ}\text{C}$ ²	14 days to extraction; 28 days to methylation; analysis immediately after methylation ^{2,5}
Waste Samples	(1) 500-mL amber wide mouth jar with a Teflon-lined screw cap.	No special preservation required	14 days to extraction; 28 days to methylation; analysis immediately after methylation ⁵

¹The 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 during shipping and/or analysis.

²Alternatively, soil/sediment samples for chlorinated herbicide analyses may be held for up to one (1) year if frozen within 24 hours of collection at $<-10^{\circ}\text{C}$. 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 or extraction must be commenced within 14 days of thawing. Once the thawing process begins, samples must be kept at $0-6^{\circ}\text{C}$ until extraction.

³Holding time begins from time of sample collection or date thawed (see note #2 above).

⁴Presence of chlorine residual is usually associated with drinking water samples. Confirm dechlorination. If residual chlorine $>5\text{ mg/L}$, additional dechlorination agent may be required.

⁵Herbicide sample extracts must be stored at 4°C , protected from light, and stored in sealed vials (e.g., screw-cap or crimp-capped vials) with un-pierced PTFE-lined septa.

⁶As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, if the holding time is exceeded by $>2\times$ the allowable holding time, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias.

⁷If 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 6°C are acceptable.



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**Sample Collection, Preservation, and Handling Procedures for
Volatile Petroleum Hydrocarbon Analyses**

Matrix	Container ¹	Preservation ⁶	Holding Time ^{3,5}
Aqueous Samples (using ambient temperature purge)	3 x 40-mL VOC vials w/ Teflon-lined septa screw caps and protect from light	Adjust pH to < 2.0 by addition of HCl to container before sampling. Cool to $\leq 6^{\circ}\text{C}$ but not frozen.	14 days
Aqueous Samples (using heated purge [$>40^{\circ}\text{C}$]) ²	3 x 40-mL VOC vials w/ Teflon-lined septa screw caps and protect from light	0.4 to 0.44 g of trisodium phosphate dodecahydrate (TSP) per 40 mL. Verify pH > 11.0. Cool to $\leq 6^{\circ}\text{C}$ but not frozen.	14 days
Soil/Sediment Samples	Extrude soil/sediment sample directly into a pre-weighed vial* w/ Teflon-lined septa screw caps: Vials must contain 1 mL purge-and-trap grade methanol for every gram soil/sediment. *(1) x 60-mL vial or (1) x 40-mL vial	1 mL methanol for every gram soil/sediment; add methanol before or at time of sampling; methanol must cover soil/sediment sample Cool to $\leq 6^{\circ}\text{C}$ but not frozen. 60-mL vial: 25 g soil/sediment and 25 mL methanol 40-mL vial: 15 g soil/sediment and 15 mL methanol	28 days
	5 g EnCore™ samplers ⁴ or other suitable coring device	Cool to $\leq 6^{\circ}\text{C}$ (but not frozen) in field; 48 hours from date collected until methanol preservation (1 mL methanol for every gram soil/sediment).	28 days

¹The 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 during shipping and/or analysis.

²Heated purge ($>40^{\circ}\text{C}$) is considered a significant modification to the method, as per Section 11.3.1 of the VPH by GC/PID/FID and VPH by GC/MS methods.

³Holding time begins from time of sample collection.

⁴EnCore™ sampler may not be suitable for certain soil types; refer to guidance in SW-846 Method 5035A.

⁵As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, if the holding time is exceeded by >2x the allowable holding time or if soil/sediment samples are not properly preserved, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias.

⁶If 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 6°C are acceptable.



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**Sample Collection, Preservation, and Handling Procedures for
Extractable Petroleum Hydrocarbon Analyses**

Matrix	Container ¹	Preservation ⁶	Holding Time ^{3,5}
Aqueous Samples	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	1:1 HCl to pH <2; Cool to 0-6°C but not frozen	14 days to extraction; 40 days from extraction to analysis ⁴
Soil/Sediment Samples	4-oz. (120 mL) wide-mouth amber glass jar with Teflon-lined screw cap ²	Cool to 0-6°C but not frozen ²	14 days to extraction; 40 days from extraction to analysis ^{2,4}
Waste Samples	Collect sample in one (1) x 500 mL amber wide mouth jar with a Teflon-lined screw cap.	No special preservation required	14 days to extraction; 40 days from extraction to analysis ⁴

¹The 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 during shipping and/or analysis.

²Alternatively, soil/sediment samples for EPH 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. Preparation or extraction must be commenced within 14 days of thawing. Once the thawing process begins, samples must be kept at 0-6°C until extraction. Temperature must never be allowed to go below -20°C to avoid damage to seals, etc.

³Holding time begins from time of sample collection or date thawed (see note #2 above).

⁴EPH sample extracts must be stored at 4° C, protected from light, and stored in sealed vials (e .g., screw-cap or crimp-capped vials) with un-pierced PTFE-lined septa.

⁵As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, 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.

⁶If 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 6°C are acceptable.



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Sample Collection, Preservation, and Handling Procedures for Nitroaromatic and Nitramine Analyses

Matrix	Container ¹	Preservation ⁷	Holding Time ^{3,6}
Aqueous Samples, with no Residual Chlorine	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Store in dark; Cool to $\leq 6^{\circ}\text{C}$ but not frozen; pH 2.0 w/ NaHSO_4 (1.2 g/L)	7 days to extraction; 40 days from extraction to analysis ⁵
Aqueous Samples, with Residual Chlorine ⁴	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Add 1-mL 10% sodium thiosulfate solution per container (or 0.008%) ⁴ . Addition of thiosulfate solution to sample container may be performed in the laboratory prior to field use. Store in dark; Cool to $\leq 6^{\circ}\text{C}$ but not frozen.	7 days to extraction; 40 days from extraction to analysis ⁵
Soil/Sediment Samples	(1) 8-oz. amber glass jar w/ a Teflon-lined screw cap ²	Store in dark; Cool to $\leq 6^{\circ}\text{C}$ ²	14 days to extraction; 40 days from extraction to analysis ^{2,5}
Waste Samples	Collect sample in one (1) x 500 mL amber wide mouth jar with a Teflon-lined screw cap.	No special preservation required	14 days to extraction; 40 days from extraction to analysis ⁵

¹The 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 during shipping and/or analysis.

²Alternatively, soil/sediment samples for nitroaromatic or nitramine analyses may be held for up to one (1) year if frozen within 24 hours of collection at $<-10^{\circ}\text{C}$. Sampling container should only be filled to 2/3 of capacity to avoid breakage caused by expansion during freezing. Preparation or extraction must be commenced within 14 days of thawing. Once the thawing process begins, samples must be kept at $0-6^{\circ}\text{C}$ until extraction. Temperature must never be allowed to go below -20°C to avoid damage to seals, etc.

³Holding time begins from time of sample collection or date thawed (see note # 2 above).

⁴Presence of chlorine residual is usually associated with drinking water samples. Confirm dechlorination. If residual chlorine $> 5 \text{ mg/L}$, additional dechlorination agent may be required.

⁵Nitroaromatic and nitramine sample extracts must be stored at 4°C , protected from light, and stored in sealed vials (e.g., screw-cap or crimp-capped vials) with un-pierced PTFE-lined septa.

⁶As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, if the holding time is exceeded by $>2\times$ the allowable holding time, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias.

⁷If 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 6°C are acceptable.



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

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

¹The 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.

²Holding 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*, 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).

³If 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.

⁴SW-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.

⁵Alternatively, 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. 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 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.

⁶If 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 6°C are acceptable.



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Sample Collection, Preservation, and Handling Procedures for Mercury Analyses

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 ≤ 6°C	28 days ⁴
Concentrated Waste	125 mL wide-mouth glass or polyethylene bottle	Cool to ≤ 6°C	28 days

¹The 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.

²Holding 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.

³If 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.

⁴Alternatively, 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.

⁵If 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 6°C are acceptable.



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**Sample Collection, Preservation, and Handling Procedures for
Total Cyanide and PAC Analyses**

Matrix	Container ¹	Preservation ⁵	Holding Time ²
Aqueous Total Cyanide/PAC	250 mL Polyethylene or Glass bottle for micro-distillation procedure; 1 L Polyethylene or Glass bottle for macro-distillation procedure	Total Cyanide & PAC: NaOH to pH ≥ 12.0; Cool to ≤ 6°C but not frozen; 0.1N sodium arsenite or 0.6 g ascorbic acid per liter, if residual chlorine is suspected PAC: Keep out of direct light	14 days to distillation; analyze distillates within 24 hours of distillation
Aqueous Dissolved Cyanide	250 mL Polyethylene or Glass bottle for micro-distillation procedure; 1 L Polyethylene or Glass bottle for macro-distillation procedure	Filter (0.45 µm) on site or at the laboratory (prior to chemical preservation) within 24 hours of collection; then preserve with NaOH to pH ≥ 12 ³ Cool to ≤ 6°C but not frozen 0.1M sodium arsenite or 0.6 g ascorbic acid per liter, if residual chlorine is suspected	14 days to distillation; analyze distillates within 24 hours of distillation
Soil and Sediments	4-ounce glass jar with inert (Teflon) liner	Total Cyanide & PAC: Cool to ≤ 6°C but not frozen PAC: Keep out of direct light	14 days to distillation; analyze distillates within 24 hours of distillation
Concentrated Waste ⁴	250 mL amber wide-mouth jar with inert (Teflon) liner	Total Cyanide & PAC: Cool to ≤ 6°C but not frozen PAC: Keep out of direct light	As soon as possible

¹The 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.

²Holding time begins from time of sample collection. 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.

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

⁴Samples containing, or suspected of containing, cyanide or a combination of cyanide and sulfide wastes should be collected with a minimum of aeration. The sample container should be filled completely, excluding all headspace, and capped. Analysis should commence as soon as possible.

⁵If 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 6°C are acceptable.



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Sample Collection, Preservation, and Handling Procedures for Hexavalent Chromium Analyses

Matrix	Container ¹	Preservation ⁵	Holding Time ²
Concentrated Waste Cr(VI)	125 mL wide-mouth glass or polyethylene bottle	Cool to $\leq 6^{\circ}\text{C}$ but not frozen.	30 days to digestion; 7 days from digestion to analysis. Store samples and alkaline digestates at $4 \pm 2^{\circ}\text{C}$ until analyzed.
Aqueous Cr(VI): SW-846 7196A	500 mL glass or polyethylene bottle	Cool to $\leq 6^{\circ}\text{C}$ but not frozen.	24 hours
Dissolved/Filtered Cr(VI): SW-846 7196A	500 mL glass or polyethylene bottle	Filter (0.45 μm) on site or at the laboratory within 24 hours of collection, prior to analysis. Cool to $\leq 6^{\circ}\text{C}$ but not frozen.	24 hours
Aqueous Cr(VI): SW-846 7199A	125 mL polyethylene bottle	Filter (0.45 μm) on site or at the laboratory within 24 hours of collection, prior to analysis. Adjust pH of sample to 9-9.5 with buffer solution (Section 5.7 of SW-846 Method 7199A). If salts are formed after pH adjustment, the filtrate can be filtered again. Cool to $\leq 6^{\circ}\text{C}$ but not frozen.	24 hours
Soil/Sediment Cr(VI)	4-ounce glass jar with Teflon-lined cap ³	Samples should be collected with non-metallic devices and stored field-moist at $\leq 6^{\circ}\text{C}$ but not frozen.	30 days to digestion; 7 days from digestion to analysis ⁴ . Store samples and alkaline digestates at $4 \pm 2^{\circ}\text{C}$ until analyzed.
Soil/Sediment pH and ORP (Eh)	4-ounce glass jar with Teflon-lined cap	Cool to $\leq 6^{\circ}\text{C}$ but not frozen.	24 hours



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¹The collection of multiple sample containers per sample location may be required to collect enough sample for matrix QC.

²Holding time begins from time of sample collection. 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.

³If both Total Chromium and Cr(VI) are to be measured in a solid sample, separate sample jars must be collected for each analysis, such that the Cr(VI) sample container remains unopened until alkaline digestion commences.

⁴Soil/Sediment holding time as listed in SW-846 Method 3060A (December 1996).

⁵If 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 6°C are acceptable.



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**Sample Collection, Preservation, and Handling Procedures for
Perchlorate Analyses**

Matrix	Container ¹	Preservation ⁴	Holding Time ^{2,3}
Aqueous Samples	(1) 125-mL polyethylene bottle	Filter with 0.2 µm Teflon or sterile cellulose acetate filter in the field; Cool to ≤ 6°C but not frozen; Store with headspace	28 days to extraction and analysis
Soil/Sediment Samples	(1) 8-oz. amber glass jar w/ a Teflon-lined screw cap	Cool to ≤ 6°C but not frozen; Store with headspace.	28 days to extraction and analysis

¹The 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 during shipping and/or analysis.

²Holding time begins from time of sample collection.

³As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, 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.

⁴If 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 6°C are acceptable.



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**Sample Collection, Preservation, and Handling Procedures for
TO-15 and Air-Phase Petroleum Hydrocarbon Analyses**

Matrix	Container ¹	Preservation	Holding Time ^{2,3}
Air	Certified clean, leak-free, stainless steel polished or silica lined passivated air sampling canisters	None	30 days

¹The size of the canister will depend on project requirements.

²Holding time begins from time of sample collection.

³As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, 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.



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**Sample Collection, Preservation, and Handling Procedures for
PFAS Analyses**

Matrix	Container ¹	Preservation ²	Holding Time
Aqueous Samples (with exception of landfill leachates)	(2) 500-mL* high density polyethylene (HDPE) container w/ linerless HDPE or polypropylene caps (1) 125-mL* HDPE container w/ linerless HDPE or polypropylene cap ³	Cool to $\leq 6^{\circ}\text{C}$ or $\leq -20^{\circ}\text{C}$	See next page
Landfill Leachates	(3) 125-mL* HDPE container w/ linerless HDPE or polypropylene caps ⁴	Cool to $\leq 6^{\circ}\text{C}$ or $\leq -20^{\circ}\text{C}$	
Soil/Sediment/ Biosolids Samples	(1) 500-mL* HDPE container w/ linerless HDPE or polypropylene cap, no more than $\frac{3}{4}$ full	Cool to $\leq 6^{\circ}\text{C}$ or $\leq -20^{\circ}\text{C}$	
Tissue Samples	Tissue: wrap in aluminum foil or insert into resealable plastic bag or food-grade polyethylene tubing Homogenized fish: (1) 100-mL HDPE container w/ linerless HDPE or polypropylene cap	Cool to $\leq 6^{\circ}\text{C}$; must be received by laboratory within 24 hours. Freeze sample ($\leq -20^{\circ}\text{C}$) before shipping if longer transport time is necessary.	

*Smaller size sample containers can be used, as long as performance and regulatory criteria will still be achieved.

¹The 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 during shipping and/or analysis.

²If 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 6°C are acceptable.

³This container is used for determination of TSS and pre-screening analyses, if warranted.

⁴One of the three containers is used for determination of TSS and pre-screening analyses, if warranted.



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)

Holding Times*

Aqueous		Soil/Sediment/Biosolids	Tissue
Samples stored at 0-6°C and protected from light	Samples stored at ≤-20°C and protected from light	Samples stored at either 0-6°C or ≤-20°C and protected from light	Samples stored at ≤-20°C and protected from light
28 days from collection to extraction ^{1,2}	90 days from collection to extraction ¹	90 days from collection to extraction ^{1,3} Soils and Sediments: Some soils and sediments may exhibit microbial growth when stored at 0-6°C. Biosolids: Microbial activity in biosolids samples at 0-6°C may cause production of gases which can result in sample being expelled from container when opened as well as noxious odors. Therefore, USEPA Method 1633 recommends samples be stored at ≤-20°C if extraction will be delayed for a few days.	90 days from collection to extraction ^{1,3}

Extracts stored at 0-6°C or ≤-20°C and protected from light and stored in sealed polypropylene vials.

90 days from extraction to analysis⁴

***Holding time begins from time of sample collection except for fish samples. If whole fish samples are frozen within 48 hours of collection, the holding time begins when the whole fish is processed (e.g., filleted) for analysis.**

Note: According to Appendix IV of MassDEP Policy #WSC-07-350, MCP Representativeness Evaluations and Data Usability Assessments, 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. However, for PFAS, this rule does not apply as the target PFAS negatively affected by a holding time exceedance (polyfluoroalkyl PFAS) can transform and cause a potential high bias for the regulated PFAS.

¹Greater than 28 days (or 90 days, as applicable) results in potential low bias for polyfluoroalkyl PFAS and potential high bias for perfluoroalkyl PFAS.

²This is noted in Section 8.5.1 of USEPA Method 1633 and was derived from the single-laboratory validation study performed for this method (<https://www.epa.gov/system/files/documents/2022-01/pfas-slvs-report-final-with-appendices.pdf>; Appendix K, Section 5.0). MassDEP expects the 28-day holding time to be followed in this instance. However, data users should note that extractions performed greater than 7 days from collection may result in potential low bias for NMeFOSE and NEtFOSE and potential high bias for the transformation products, NMeFOSAA, and NEtFOSAA. If NMeFOSE, NEtFOSE, NMeFOSAA, and NEtFOSAA are contaminants of concern, data users may want to use the freezing option to extend the holding time.

³This exception is noted in Sections 8.5.2, 8.5.3, and 8.5.6 of USEPA Method 1633. MassDEP expects the 90-day holding time to be followed in this instance. Data users should note that extractions performed greater than 3 days from collection may result in potential low bias for NFDHA in soil, sediment, and tissue samples, regardless of storage temperature for soil/sediment and tissue. No issues are noted in the method for NFDHA in biosolids samples.

⁴This exception is noted in Sections 8.5.5 and 8.5.6 of USEPA Method 1633. MassDEP expects the 90-day holding time to be followed in this instance. Data users should note that analyses performed greater than 28 days from extraction may result in potential high bias for 9Cl-PF3ONS and 11Cl-PF3OUs.