



Massachusetts Department of Environmental
Protection Bureau of Waste Site Cleanup

WSC-CAM

Section: X B

September 30, 2026

Revision No. 0


Final

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Quality Control Requirements and Performance Standards for the ***Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Private Wells by Liquid Chromatography/Dual Mass Spectrometry (LC-MS/MS)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

WSC – CAM – X B

Quality Control Requirements and Performance Standards for
the ***Analysis of Per- and Polyfluoroalkyl Substances
(PFAS) in Private Wells by Liquid Chromatography/Dual
Mass Spectrometry (LC-MS/MS)*** in Support of Response
Actions under the Massachusetts Contingency Plan (MCP)

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X. Liquid Chromatography (LC) and Dual Mass Spectrometry (MS/MS) Methods

A. Quality Control Requirements and Performance Standards for WSC-CAM-X B (Per- and Polyfluoroalkyl Substances in Private Wells by LC-MS/MS)

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ACRONYM LIST

CAM	Compendium of Analytical Methods
CASN	Chemical Abstracts Service Number
CCC	Continuing calibration check
DF	Dilution factor
FRB	Field reagent blank
IRA	Immediate Response Action
LC-MS/MS	Liquid chromatography/dual mass spectrometry
LFB	Laboratory fortified blank
LFSM	Laboratory fortified sample matrix
LFSMD	Laboratory fortified sample matrix duplicate
LLOQ	Lower limit of quantitation
LRB	Laboratory reagent blank
MassDEP	Massachusetts Department of Environmental Protection
MCL	Maximum contaminant level
MCP	Massachusetts Contingency Plan
MD	Matrix duplicate
MDL	Method detection limit
MOHML	Massachusetts Oil and Hazardous Materials List
MRL	Minimum reporting level
MRM	Multiple reaction monitoring
MS	Matrix spike
MSD	Matrix spike duplicate
NA	Not applicable
ng/L	Nanograms per liter
PFAS	Per- and polyfluoroalkyl substances
PTFE	Polytetrafluoroethylene
QA	Quality assurance
QC	Quality control
%R	Percent recovery
RCs	Reportable Concentrations
RL	Reporting limit
RPD	Relative percent difference
RQs	Reportable Quantities
RT	Retention time
SPE	Solid phase extraction
TSS	Total suspended solids
USEPA	United States Environmental Protection Agency

Refer to Table X B-2 for PFAS acronym definitions.

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

1.1 Overview of WSC-CAM-X B

WSC-CAM-X B, *Quality Control Requirements and Performance Standards for the Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Private Wells by Liquid Chromatography/Dual Mass Spectrometry (LC-MS/MS) in Support of Response Actions under the Massachusetts Contingency Plan (MCP)*, is a component of MassDEP's Compendium of Analytical Methods (CAM). Refer to WSC-CAM-I A for an overview of the CAM process. Please note that while this protocol must be followed on and after the effective date of September 30, 2026 for the purpose of "Presumptive Certainty," the protocol may be used optionally prior to its effective date upon its publication on June 30, 2026.


This document provides quality control (QC) requirements and performance standards to be used in conjunction with the required analytical method United States Environmental Protection Agency (USEPA) 533 (the most current version), analysis for PFAS in private well samples by LC-MS/MS preceded by sample preparation methods discussed in USEPA Method 533, and described in Section 1.3 of this protocol. The QC requirements and performance standards specified in this document in Table X B-1 together with the analytical procedures described in USEPA Method 533, *Determination of Per- and Polyfluoroalkyl Substances (PFAS) in Drinking Water by Isotope Dilution Anion Exchange Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry*, constitute the WSC-CAM-X B protocol. All protocols included in the CAM are considered "methods" published by the MassDEP pursuant to the provisions of 310 CMR 40.0017(2).

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

Overall usability of data produced using this CAM protocol should be evaluated for compliance with project-specific data quality objectives, regardless of "Presumptive Certainty" status. For more guidance on data usability, refer to MassDEP Policy #WSC-25-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

1.1.1 Reporting Limits or Lower Limits of Quantitation for WSC-CAM-X B

The reporting limit (RL) or lower limit of quantitation (LLOQ) for an individual compound using WSC-CAM-X B is based on a concentration at or above the lowest non-zero calibration standard in the initial calibration, analyzed under identical conditions as the sample, with adjustments made for the sample size, extraction concentration factor, dilution factors, etc., as required. RLs/LLOQs are referred to as Minimum Reporting Levels (MRLs) in USEPA Method 533. The CAM RLs/LLOQs for WSC-CAM-X B target analytes are 2.0 nanograms per liter (ng/L).

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These values are readily achievable using LC-MS/MS. For “Presumptive Certainty” purposes, if the CAM RLs/LLOQs are not achieved, respond “NO” to Question G of the “MassDEP MCP Analytical Protocol Certification Form” and address the CAM RL/LLOQ exceedance in the laboratory narrative.


RLs/LLOQs lower than the above-referenced CAM RLs/LLOQs for WSC-CAM-X B target analytes may be required to satisfy project requirements. The RL/LLOQ (based on a concentration at or above the lowest calibration standard) for each contaminant of concern must be less than or equal to the MCP standards or criteria that the contaminant concentrations are being compared to (e.g., Maximum Contaminant Levels [MCLs], Method 1 Standards, benchmark values, background, etc.). Meeting MCP standards or criteria may require method modifications, such as increasing sample volume or reducing the volume of the final extract, to improve sensitivity. All such modifications must be described in the laboratory narrative. Regardless of the modification that is used, RLs/LLOQs for the WSC-CAM-X B target analytes will be proportionately higher for samples that require dilution, when a reduced sample size is used, or for an increased final extract volume.

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

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

Laboratories must document and have on file an Initial Demonstration of Proficiency for each combination of sample preparation and determinative method being used. These data must meet or exceed the performance standards as presented in Table X B-1 of this protocol and USEPA Method 533. Procedural requirements for performing the Initial Demonstration of Proficiency can be found in USEPA Method 533 (Section 9.1 and Table 16). The data associated with the Initial Demonstration of Proficiency must be kept on file at the laboratory and made available to potential data users on request. The data associated with the Initial Demonstration of Proficiency for WSC-CAM-X B must include the following information:

QC Element	Performance Criteria
Initial Calibration	WSC-CAM-X B, Table X B-1
Calibration Verification	WSC-CAM-X B, Table X B-1; USEPA Method 533, Section 9.1.5 and Table 16
Method Blanks	WSC-CAM-X B, Table X B-1
System Background	USEPA Method 533, Section 9.1.1 and Table 16
Average Recovery (Accuracy)	USEPA Method 533, Section 9.1.3 and Table 16
% Relative Standard Deviation (Precision)	USEPA Method 533, Section 9.1.2 and Table 16
RL/LLOQ Confirmation	USEPA Method 533, Section 9.1.4 and Table 16

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QC Element	Performance Criteria
Isotope Dilution Analogue Recovery	WSC-CAM-X B, Table X B-1
Isotope Performance Standards	WSC-CAM-X B, Table X B-1

NOTE: Because of the number of QC elements associated with the Initial Demonstration of Proficiency, it should be expected that one or more analytes may not meet the performance standard for one or more QC elements. Under these circumstances, the analyst should attempt to locate and correct the problem and repeat the analysis for all non-conforming analytes. All non-conforming analytes along with the laboratory-specific acceptance criteria should be noted in the Initial Demonstration of Proficiency documentation.

It is essential that laboratory-specific performance criteria for LFBs and isotope dilution analogue recoveries also be calculated and documented as described in USEPA Method 533, Section 9.3.2. Laboratories are encouraged to actively monitor pertinent QC performance standards described in Table X B-1 to assess analytical trends (i.e., systematic bias, etc.) and improve overall method performance by preempting potential non-conformances.

It should be noted that the performance standards listed in Table X B-1 are based on USEPA Method 533.


This protocol is restricted to use by, or under the supervision of, analysts experienced in the use of LC-MS/MS instrumentation and isotope dilution as a quantitative tool and skilled in the interpretation of data generated with these instruments.

1.2 Summary of USEPA Method 533

Private well samples are prepared using USEPA Method 533, Sections 11.3 and 11.4. After extraction, the extract is analyzed by injecting an aliquot into a ultrahigh performance liquid chromatograph (LC) equipped with a C18 column packed with 3 µm C18 solid phase particles, or equivalent, connected to tandem quadrupole mass spectrometers (MS/MS) operating in the multiple reaction monitoring (MRM) mode.

PFAS concentrations in samples are determined by isotope dilution or quantification using isotopically labeled compounds added to the sample prior to extraction. Quantitation is accomplished by using the peak areas of quantitation (product) ions and a response factor generated from a minimum five-point calibration curve or by using a second-order calibration model generated from a minimum six-point calibration curve (refer to Table X B-1). When linear and branched isomers are present in both the sample and qualitative or quantitative standards for a target PFAS, the target PFAS is reported as a combined response of the linear and branched isomers.

Isotope dilution and isotope dilution analogue quantification provide a correction for any potential losses during extraction. Isotope dilution also provides a correction for matrix effects that could lead to signal enhancement or suppression and thus can avoid measurement bias.

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Identification of target PFAS is accomplished by comparing the retention time of the target PFAS quantitation (product) ions in samples with the retention time of the target PFAS quantitation (product) ions in standards obtained under identical analytical conditions. In addition, the retention time of the target PFAS in samples is compared with the retention time of the exact corresponding isotope dilution analogue, where these exist.

1.3 Sample Extraction Methods for WSC-CAM-X B

Samples for analysis by USEPA Method 533 must be extracted using the procedures in Sections 11.3 and 11.4 of the method. In general, the following procedures are used:

- Aqueous samples are spiked with isotope dilution analogues, and then extracted using solid-phase extraction (SPE). The SPE cartridge contains weak anion exchange, mixed-mode polymeric sorbent (polymeric backbone and a diamino ligand), particle size approximately 33 µm. The SPE sorbent must have a pKa above 8 so that it remains positively charged during extraction.
- Target PFAS are eluted from the SPE cartridge with methanol containing ammonium hydroxide.
- The extract is concentrated to dryness and then adjusted to 1.0 mL with 20% water in methanol.

1.4 Method Interferences


- Refer to USEPA Method 533 (Section 4.0, in particular) for a detailed discussion of interferences and corrective actions which may be taken to eliminate contamination. Interferences co-extracted from the samples will vary considerably from matrix to matrix, and will also be dependent upon the diversity of the site being sampled.

Sources of interference in this method can be grouped into four broad categories.

- Contaminated solvents (e.g., methanol and methanolic ammonium hydroxide), reagents, preservative (e.g., ammonium acetate), or sample processing hardware,
- Disposable plasticware, glass equipment, parts of the SPE manifold, filters, and equipment used to prepare samples,
- Non-target compounds simultaneously extracted from the sample matrix which cause a detector response, and
- Co-elution of target analytes.

The most frequently encountered interferences in reagents and equipment are fluoropolymers. The equipment used by the laboratory must be demonstrated to be free of PFAS below the laboratory's method detection limit (MDL) and also must not be constructed of materials which could react with or sorb target PFAS. An in-depth discussion of the causes and corrective actions for all of these interferences is beyond the scope of this guidance document.

- If a method blank contains a contaminant, data for samples associated with that blank must **not** undergo "blank correction" (i.e., if an associated sample also contains the contaminant, subtraction of the blank amount from the sample amount is not permitted).

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- Cross-contamination may occur when any sample is analyzed immediately after a sample containing high concentrations of PFAS. After the analysis of a sample containing high concentrations of PFAS, one or more blanks should be analyzed to check for potential cross-contamination/carryover. Concentrations of PFAS which exceed the upper limit of calibration should prompt the analyst to check for potential cross-contamination/carryover. To reduce carryover, the injector must be rinsed with solvent between sample injections.
- Potential matrix interferences are as follows:
 - High levels of humic and fulvic material co-extracted during the SPE procedure may cause enhancement or suppression in the electrospray ionization source. Matrix effects due to co-extracted organic material enhanced the ionization of 4:2 FTS during the development of USEPA Method 533; total organic carbon can provide a good indication of the humic content of a sample.
 - The presence of inorganic salts may cause low recoveries during the anion-exchange SPE procedure.

1.5 Quality Control Requirements for WSC-CAM-X B

1.5.1 Specific QC Requirements and Performance Standards for WSC-CAM-X B


Specific QC requirements and performance standards for the WSC-CAM-X B protocol are presented in Table X B-1. Refer to WSC-CAM-VII A for field QC requirements. Strict compliance with the QC requirements and performance standards, as well as satisfying the CAM's other analytical and reporting requirements will provide a data user with "Presumptive Certainty" in support of Response Actions under the MCP. The concept of "Presumptive Certainty" is explained in detail in Section 2.0 of WSC-CAM-VII A.

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

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

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

- ✓ Satisfy the broad QA/QC requirements of 310 CMR 40.0017 and 40.0191 regarding the scientific defensibility, precision and accuracy, and reporting of analytical data; and
- ✓ May be used in a data usability and representativeness assessment, as required in 310 CMR 40.1056(2)(k) and 40.1057(2)(k) for Permanent and Temporary Solution submittals,


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consistent with the guidance described in MassDEP Policy #WSC-25-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

1.6 Special Analytical Considerations for WSC-CAM-X B

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

- Section 1.3 of USEPA Method 533 discusses allowable modifications to the method, including choice of LC columns, LC conditions, MRM transitions, and mass spectrometer conditions. The method also allows the laboratory to select bottle sizes for samples, ranging from 100 to 250 mL, as needed to meet project objectives. In all cases where method modifications are implemented, the laboratory must demonstrate acceptable QC and method performance via the Initial Demonstration of Proficiency and achievement of requirements in Table X B-1. Method modifications must also be discussed in the laboratory narrative. Items which cannot be modified from the method requirements include the following:
 - ✓ Ratio of SPE sorbent bed volume to aqueous sample volume (2:1)
 - ✓ Isotope dilution analogues and isotope performance standards
 - ✓ Sample preservation requirements
 - ✓ QC requirements
 - ✓ Extraction procedure
- It should be noted that field reagent blanks (FRBs) can be collected with private well samples but this CAM Protocol does not require the collection of FRBs, although required in USEPA Method 533. However, as per 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)*, field duplicates are required to be collected at each location; analysis of the field duplicates is only required when PFAS are detected in the original sample.
- Aqueous samples must not come into contact with glass containers or pipettes. Following the SPE procedure, the eluate must be collected in a polypropylene tube prior to concentration to dryness. If glass tubes are used, there could be poor recovery of target PFAS.
- Polypropylene autosampler vials and caps must be used to prevent contamination of the sample from polytetrafluoroethylene (PTFE) coated septa. However, since polypropylene caps do not reseal, there is the potential for evaporation to occur after injection. Therefore, multiple injections from the same vial are not permissible unless the cap is replaced immediately after injection. Evaporation losses can cause high-biased isotope performance standard area counts.
- Since PFAS can adhere to the walls of the sample containers, it is important that the entire volume of the aqueous sample in the container be extracted. This will allow the laboratory to be able to rinse the sides of the container (e.g., with methanolic ammonium hydroxide) to remove any potential PFAS which may adhere. If the data user suspects the aqueous samples may contain elevated concentrations of PFAS, a smaller size container can be requested from the laboratory for collection to avoid subsampling

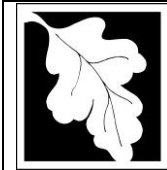
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from the container (i.e., use of a reduced volume of sample in the container). If a smaller size container is not available, subsampling may be unavoidable.

- PFAS tend to accumulate at the air/water interface and PFAS may stratify in the sample container and accumulate at the air/water interface.
 - ✓ Extraction of the entire volume of sample in the container, as discussed above, will prevent any issues from stratification in the sample container.
 - ✓ If subsampling is unavoidable, vortexing or shaking samples prior to subsampling could mitigate this stratification or accumulation at air/water interface.
- Aqueous samples with elevated levels of total suspended solids (TSS) may present challenges. It should be noted that filtering is not allowed. When elevated levels of TSS are present, there are several options for the laboratory to follow to avoid subsampling.
 - ✓ Add isotope dilution analogues to the entire sample, equilibrate for 30 minutes, decant aqueous phase through SPE, solvent (e.g., methanolic ammonium hydroxide) rinse the remaining particulates in the bottle, and add solvent rinse to SPE.
 - ✓ Add isotope dilution analogues to the entire sample, equilibrate for 30 minutes, centrifuge, put aqueous phase through SPE, solvent (e.g., methanolic ammonium hydroxide) rinse the remaining particulates in the bottle after centrifuging, and add solvent rinse to SPE.

MassDEP requires that the laboratory provide details on the procedure followed in the laboratory narrative.

- It is important that the laboratory bring all standards and sample extracts to room temperature followed by vortexing prior to analysis to ensure the homogeneity of the extracts.
- Although the method measures target PFAS as anions, MassDEP expects all target PFAS to be reported in their acid or neutral forms. In general, the conversion from anion to acid will cause a minimal change in the concentration. Some PFAS may be commercially available as salts. Although it is acceptable to use these salt standards, the concentrations of the associated target PFAS must be corrected for the salt content. The equation to perform this correction is included in Section 7.17.3 of USEPA Method 533.
- MS/MSDs are generally not required for methods that use isotope dilution quantification since this method of quantification corrects sample results for matrix effects. However, there are several target PFAS for which an exact corresponding isotope dilution analogue is not currently available. In these instances, MS/MSDs may provide useful information. As of the date of this CAM Protocol, the target PFAS which do not have an exact corresponding isotope dilution analogue are as follows: PFMPA, PFEESA, NFDHA, ADONA, 9CI-PF3ONS, and 11CI-PF3OUdS.
- Laboratories should not spike additional isotope dilution analogues in sample extracts when a sample requires a dilution. If the originally spiked isotope dilution analogue will be “diluted out” in the diluted sample, then the sample should instead be re-extracted with a reduced volume. For Presumptive Certainty purposes, if additional isotope dilution analogue is added to a sample extract which is diluted, respond



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“NO” to Question C of the “MassDEP MCP Analytical Protocol Certification Form” and address this nonconformance in the laboratory narrative.



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Table X B-1: Specific QC Requirements and Performance Standards for PFAS (USEPA 533) Using WSC-CAM-X B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-25-350 ¹	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must contain all target analytes. (3) Must follow procedure in Section 9.1 of USEPA Method 533.	No	NA	Refer to Section 9.1 and Table 16 of USEPA Method 533 and Section 1.1.2 of this protocol.	NA
Quarterly Calibration Verification	Laboratory Analytical Accuracy (Verification of Integrity of Initial Calibration Standards)	(1) Must be performed quarterly after the initial demonstration of proficiency. (2) Must be prepared using an independent dilution, preferably by a second analyst, from the same stock standard used in the initial calibration. (3) Percent recoveries (%Rs) must be within 70-130% for each target PFAS.	No	NA	(1) Prepare fresh standard dilution. (2) Repeat calibration verification standard analysis. (3) If still outside acceptance limits after re-analysis, re-calibrate.	NA
Mass Calibration	Method Sensitivity and Stability	(1) Must be performed at least annually, or as recommended by manufacturer, whichever is more frequent. (2) Use mass calibration solution specified by instrument manufacturer.	No	NA	Adjust the MS/MS if calibration masses are missing or not correctly identified.	Sample analysis cannot proceed without a valid mass calibration.
Retention Time (RT) Windows	Laboratory Analytical Accuracy	(1) Use a mid- to high-level standard in the initial calibration to establish the RT windows. Use the continuing calibration check (CCC) standard to verify the RT windows for an analytical batch. (2) Must be of sufficient width to detect earlier-eluting branched isomers.	No	NA	NA	NA
Initial Calibration	Laboratory Analytical Accuracy	(1) Must be performed at least once prior to analyzing samples, when CCC does not meet the performance standards, and when major instrument maintenance is performed. (2) Minimum of 5 standards when average response factors are used for quantitation (or 6 standards if a second-order calibration	No	NA	Recalibrate, reduce the range of calibration, or prepare new calibration standards, as required by method.	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds (outside of 50-150% or 70-130% recovery range, as



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Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-25-350 ¹	Required Corrective Action	Required Analytical Response Action
		<p>model used). Concentrations of isotope dilution analogues and isotope performance standards remain constant in all standards and must be calibrated using the average response factor approach. Standards must be prepared in solvent mix described in Section 7.17.5 of USEPA Method 533.</p> <p>NOTE: Second-order calibration models may include weighted linear regression or non-linear regression; forcing the calibration curve through the origin is required.</p> <p>(3) Low standard must be \leqRL/LLOQ.</p> <p>(4) Laboratories may use linear/non-linear regression curves for quantitation of target PFAS; forcing the calibration curve through the origin is required.</p> <p>(5) Concentration of each target PFAS from each of the initial calibration standards using the linear/non-linear regression curves must be between 50-150% of the true value for the standards that are \leqRL/LLOQ and between 70-130% of the true value for all other calibration standards.</p> <p>(6) Must contain all target PFAS, isotope dilution analogues and isotope performance standards.</p> <p>(7) Calibration must be performed under the same conditions as the samples.</p> <p>(8) Quantitative standards including linear and branched isomers must be used for target PFAS if commercially available. Currently, these include PFHxS, PFOA, PFOS, and PFNA.</p>				<p>applicable) in laboratory narrative.</p>



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Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-25-350 ¹	Required Corrective Action	Required Analytical Response Action
		(9) If qualitative standards (i.e., technical-grade standards) exist for linear and branched isomers of a target PFAS where a quantitative standard does not exist, then the qualitative standard must be included in the initial calibration for the identification of linear and branched isomers of the associated target PFAS.				
Continuing Calibration Check (CCC)	Laboratory Analytical Accuracy and Sensitivity	(1) Prior to samples, every 10 samples, and at the end of the analytical batch (24-hour period). (2) Beginning CCC must be \leq RL/LLOQ (low-level CCC). Subsequent CCCs within the analytical batch must alternate between mid- and high-level calibration standards. (3) Must contain all target PFAS, isotope dilution analogues and isotope performance standards. (4) Area count of quantitation (product) ion for the isotope performance standards must be within 50-150% of the average area count measured during the initial calibration. (5) %R must be within 70-130% for each isotope dilution analogue. (6) %R must be within 50-150% for each target PFAS in the low-level CCC (\leq RL/LLOQ) and within 70-130% for the mid- and high-level CCCs. (7) The RTs of the target PFAS, the isotope dilution analogues, and the isotope performance standards must fall within the pre-established RT windows.	No	NA	(1) Perform instrument maintenance, reanalyze CCC and/or recalibrate as required by method. (2) Reanalyze "associated samples" if beginning or ending CCC exhibited low response. (3) Reanalyze "associated samples" if beginning or ending CCC exhibited high response and associated target PFAS were detected in the "associated samples." NOTE: "Associated samples" refers to all samples analyzed since the last acceptable CCC.	If recovery is outside of 50-150% or 70-130%, as applicable, for any analyte or if the RT is outside of the acceptance window for any analyte, report non-conformances in laboratory narrative.



Quality Control Requirements and Performance Standards for the **Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Private Wells by Liquid Chromatography/Dual Mass Spectrometry (LC-MS/MS)** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table X B-1: Specific QC Requirements and Performance Standards for PFAS (USEPA 533) Using WSC-CAM-X B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-25-350 ¹	Required Corrective Action	Required Analytical Response Action
		(8) Quantitative standards including linear and branched isomers must be used for target PFAS if commercially available. Currently, these include PFHxS, PFOA, PFOS, and PFNA.				
Instrument Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Analyzed after analysis of high concentration samples or high calibration standard. (2) Must be prepared in same solution as calibration standards and contain the isotope dilution analogues and isotope performance standards. (3) Target PFAS must be <RL/LLOQ.	No	NA	(1) Analyze one or more additional instrument blanks until target PFAS not detected or locate source of contamination and correct problem. Re-analyze instrument blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	(1) If sample re-analysis is not possible, report non-conformance in laboratory narrative. (2) If contamination of instrument blanks is suspected or present, the laboratory, using a "B" or some other convention, should qualify the sample results. Instrument blank contamination should also be documented in the laboratory narrative.
Method Blank (referred to as a Laboratory Reagent Blank [LRB] in USEPA Method 533)	Laboratory Method Sensitivity (contamination evaluation)	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Matrix-specific (e.g., water). (3) Aqueous method blanks must be prepared using same volume of water as field samples in batch. (4) Target PFAS must be <RL/LLOQ.	Yes	NA	(1) If concentration of contaminant in sample is ≤10x concentration in blank locate source of contamination; correct problem; re-extract and re-analyze method blank and associated samples. (2) No corrective action required if	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" or some other convention, should



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Table X B-1: Specific QC Requirements and Performance Standards for PFAS (USEPA 533) Using WSC-CAM-X B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-25-350 ¹	Required Corrective Action	Required Analytical Response Action
					concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	qualify the sample results. Blank contamination should also be documented in the laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable method blank results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
Laboratory Fortified Blank (LFB)	Laboratory Analytical Accuracy	(1) LFB extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration of LFB must rotate between low, medium, and high concentrations from batch to batch. (3) Low-level LFB: Concentration level near or at RL/LLOQ. High-level LFB: concentration near high end of calibration range. (4) Must contain all target PFAS. (5) Matrix-specific (e.g., water). (6) Aqueous LFBs must be prepared using same volume of water as field samples in batch.	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LFB.	(1) Locate source of problem; re-extract and re-analyze LFB and associated samples if >10% of all analytes are outside of criteria. (2) If ≤10% of compounds are outside of the acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the acceptance criteria,	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of acceptance criteria for any analyte, report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LFB results, the laboratory may



Quality Control Requirements and Performance Standards for the **Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Private Wells by Liquid Chromatography/Dual Mass Spectrometry (LC-MS/MS)** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table X B-1: Specific QC Requirements and Performance Standards for PFAS (USEPA 533) Using WSC-CAM-X B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-25-350 ¹	Required Corrective Action	Required Analytical Response Action
		(7) %R must be within 50-150% for each target PFAS in the low-level LFB or an LFB within a factor of 2x the RL/LLOQ and within 70-130% for the mid- and high-level LFBs.			re-extraction is not required if affected compounds were not detected in associated samples.	report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
Isotope Performance Standards (function as internal standards)	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Isotope performance standards must consist of the three isotopically labeled compounds listed in Section 7.15 of USEPA Method 533, at a minimum. (2) Area counts of isotope performance standards in field and QC samples must be between 50-150% of the mean area count of the corresponding isotope performance standard in the most recent initial calibration. (3) Additional isotope performance standards may be used if they are isotopically labeled analytes with similar functional groups as the method analytes. Initial Demonstration of Proficiency requirements must be met if additional isotope performance standards are used.	No	Recovery <20%; affects associated nondetect results in affected sample. Rejection of affected results will be based on professional judgement. If the %R of the associated isotope dilution analogue is within the acceptance criteria, rejection may not be warranted. If the isotope performance standard %R is low due to a bad injection or significant matrix suppression,	(1) If isotope performance standard is outside of limits, re-analyze sample. (2) If isotope performance standard is outside of limits in re-analysis, re-extract the sample. (3) Instrument maintenance may be required if isotope performance standard failures persist.	(1) Report nonconformances in laboratory narrative. Include actual recovery of isotope performance standard. (2) If re-analysis and/or re-extraction yields similar isotope performance standard non-conformances, the laboratory must report results of both analyses and/or extractions. (3) If re-analysis and/or re-extraction is performed within holding time and yields acceptable isotope performance standard recoveries, the laboratory may report results of the re-analysis or re-extraction only.



Quality Control Requirements and Performance Standards for the **Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Private Wells by Liquid Chromatography/Dual Mass Spectrometry (LC-MS/MS)** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table X B-1: Specific QC Requirements and Performance Standards for PFAS (USEPA 533) Using WSC-CAM-X B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-25-350 ¹	Required Corrective Action	Required Analytical Response Action
				rejection of the associated nondetect results may be warranted.		(4) If re-analysis and/or re-extraction is performed outside of the holding time and yields acceptable isotope performance standard recoveries, the laboratory must report results of both analyses and/or extractions.
Isotope Dilution Analogues	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Isotope dilution analogues must consist of the 16 isotopically labeled compounds listed in Section 7.16 of USEPA Method 533. As noted in method, additional isotopically labeled PFAS analogues for those target PFAS without their own isotope dilution analogue can be included when commercially available. Several PFAS do not currently have an exact isotope dilution analogue match ² . (2) %Rs of each isotope dilution analogue must be within 50-200%.	Yes	Recovery <10%; affects associated nondetect results for associated PFAS in affected sample.	(1) If isotope dilution analogue is outside of limits, re-analyze sample. (2) If isotope dilution analogue is outside of limits in re-analysis, re-extract the sample. NOTE: If isotope dilution analogue recoveries are high and associated target PFAS are not detected in sample, corrective action is not required.	(1) Report recoveries outside of acceptance limits in laboratory narrative. (2) If re-analysis or re-extraction yields similar isotope dilution analogue non-conformances, the laboratory must report results of both extractions/analyses. (3) If re-analysis or re-extraction is performed within holding time and yields acceptable isotope dilution analogue recoveries, the laboratory can report results of the re-extraction only. (4) If re-analysis or re-extraction is performed outside of the holding time and yields acceptable isotope



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Table X B-1: Specific QC Requirements and Performance Standards for PFAS (USEPA 533) Using WSC-CAM-X B

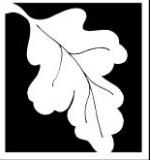
Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-25-350 ¹	Required Corrective Action	Required Analytical Response Action
						dilution analogue recoveries, the laboratory must report results of both extractions. (5) If any isotope dilution analogues used for quantitation of target PFAS vary from Table 5 of USEPA Method 533, these must be noted in the laboratory narrative.
MS/MSD (referred to as Laboratory Fortified Sample Matrix [LFSM]/LFSM Duplicate [LFSMD] in USEPA Method 533)	Method Accuracy & Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data user). (2) Matrix-specific (e.g., water). (3) Concentration level >2x RL/LLOQ or ≤2x RL/LLOQ. (4) Must contain all target PFAS. (5) %R must be within 70-130% for each target PFAS if spiked >2x RL/LLOQ and within 50-150% for each target PFAS if spiked ≤2x RL/LLOQ. (6) Relative percent differences (RPDs) ≤ 30 for each target PFAS if spiked >2x RL/LLOQ and ≤50% for each target PFAS if spiked ≤2x RL/LLOQ.	Yes ONLY when requested by the data user	Recovery <10%; affects nondetect result for affected analyte in unspiked sample only.	Check LFB; if recoveries are acceptable in LFB, narrate non-conformance.	Note non-conformances in laboratory narrative.
Matrix Duplicate (MD)	Method Precision in Sample Matrix	(1) At request of data user. (2) Prepare by extracting and analyzing an additional aliquot of the field sample. NOTE: an additional bottle is required. (3) RPDs ≤30. If concentrations ≤ 2x RL/LLOQ, RPDs ≤50.	Yes ONLY when requested by the data user	NA	Narrate.	Note non-conformances in laboratory narrative.



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Table X B-1: Specific QC Requirements and Performance Standards for PFAS (USEPA 533) Using WSC-CAM-X B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-25-350 ¹	Required Corrective Action	Required Analytical Response Action
Identification and Quantitation	NA	<p>(1) The RTs of target PFAS, isotope dilution analogues, and isotope performance standards in field and QC samples must be within the RT windows established from the initial calibration mid-to high level standard.</p> <p>(2) For PFAS where quantification includes linear and branched isomers, only the branched isomers that were identified in qualitative (technical-grade) and quantitative standards can be included in the sample quantification.</p> <p>(3) The laboratory must use the average response factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each target PFAS.</p> <p>(4) Results must be reported with 3 or more "significant figures" if \geqRL/LLOQ.³</p>	NA	NA	NA	NA
General Reporting Issues	NA	<p>(1) The laboratory must only report values \geq the sample-specific RL/LLOQ.</p> <p>Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for <u>each</u> analyte. The associated QC (e.g., method blanks, isotope dilution analogue, etc.) for each analysis must be reported.</p> <p>(2) Refer to Appendix X B-1 for chain-of-custody requirements regarding preservation, cooler temperature, and holding times.</p> <p>(3) Report all PFAS results in their acid or neutral forms.</p>	NA	NA	NA	<p>(1) Complete analytical documentation for diluted and undiluted analyses must be made available for review during an audit.</p> <p>The performance of dilutions (including samples extracted at a reduced volume) must be documented in the laboratory narrative or on the report form.</p> <p>Unless due to elevated concentrations of target</p>



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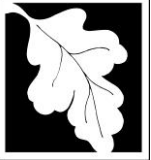
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Table X B-1: Specific QC Requirements and Performance Standards for PFAS (USEPA 533) Using WSC-CAM-X B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-25-350 ¹	Required Corrective Action	Required Analytical Response Action
		<p>(4) The laboratory must report the procedure/option used for the preparation and analysis of aqueous samples with elevated particulates (see Section 1.6 of this CAM Protocol).</p> <p>(5) The laboratory must clearly document any method modifications in the laboratory narrative (see Section 1.6 of this CAM Protocol).</p>				<p>PFAS, reasons for dilutions (including samples extracted at a reduced volume) must be explained in the laboratory narrative.</p> <p>(2) If samples are not preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory narrative.</p> <p>(3) If samples are extracted and/or analyzed outside of the holding time, note the non-conformances in the laboratory narrative.</p> <p>(4) For Presumptive Certainty purposes, if additional isotope dilution analogue is added to a sample extract which is diluted, respond "NO" to Question C of the "MassDEP MCP Analytical Protocol Certification Form" and address this nonconformance in the laboratory narrative.</p>



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Table X B-1: Specific QC Requirements and Performance Standards for PFAS (USEPA 533) Using WSC-CAM-X B

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-25-350 ¹	Required Corrective Action	Required Analytical Response Action
<p>¹As per Appendix IV of MassDEP Policy #WSC-25-350, <i>MCP Representativeness Evaluations and Data Usability Assessments</i>, if these results are observed, data users should consider nondetect results as unusable and positive results as estimated with a significant low bias.</p> <p>²Current PFAS without an exact isotope dilution analogue match: PFMPA, PFEEESA, NFDHA, ADONA, 9Cl-PF3ONS, and 11Cl-PF3OUDs.</p> <p>³Reporting protocol for “significant figures” is a policy decision included for standardization and consistency for reporting of results and is not a definition of “significant” in the scientific or mathematical sense.</p>						

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1.7 Analyte List for WSC-CAM-X B

The MCP analyte list for WSC-CAM-X B is presented in Table X B-2. The list is comprised of potential contaminants that are readily-analyzable by WSC-CAM-X B and included in USEPA Method 533.

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

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

Only six of the analytes listed in Table X B-2 have a promulgated MCP Method 1 groundwater/soil standard. The remaining analytes listed are designated “consensus contaminants” and do not have promulgated MCP Method 1 Standards as of the publication date of this revision.


1.7.1 Analyte List Reporting Requirements for WSC-CAM-X B

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

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

In 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; or

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

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

2.0 Data Usability Assessment

Specific guidance applicable to all Permanent and Temporary Solutions, including Permanent and Temporary Solutions on a portion of a disposal site, for preparation of Representativeness Evaluations and Data Usability Assessments pursuant to 310 CMR 40.1056(2)(k) and 40.1057(2)(k), respectively, of the MCP is provided in *MCP Representativeness Evaluations and Data Usability Assessments* (Policy #WSC-25-350). This document provides general information regarding the purpose and content of these required evaluations as a component of and in support of a Permanent or Temporary Solution submittal. The most current version of this document may be found at the following URL: <https://www.mass.gov/doc/wsc-25-350-mcp-representativeness-evaluations-and-data-usability-assessments-revised-october-10-2025/download>.

Overall usability of data produced using this CAM protocol should be evaluated for compliance with project-specific data objectives using MassDEP Policy #WSC-25-350, regardless of “Presumptive Certainty” status.

3.0 Reporting Requirements for WSC-CAM-X B

3.1 General Reporting Requirements for WSC-CAM-X B

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


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



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Table X B-2: Analyte List for WSC-CAM-X B (USEPA 533)

Analyte	Acronym	CASN	Analyte	Acronym	CASN
Perfluoroalkyl Carboxylic Acids			Fluorotelomer Sulfonic Acids		
Perfluorobutanoic acid	PFBA	375-22-4	1H,1H,2H,2H-Perfluorohexanesulfonic acid (4:2 fluorotelomer sulfonic acid)	4:2 FTS	757124-72-4
Perfluoropentanoic acid	PFPeA	2706-90-3	1H,1H,2H,2H-Perfluorooctanesulfonic acid (6:2 fluorotelomer sulfonic acid)	6:2 FTS	27619-97-2
Perfluorohexanoic acid	PFHxA	307-24-4	1H,1H,2H,2H-Perfluorodecanesulfonic acid (8:2 fluorotelomer sulfonic acid)	8:2 FTS	39108-34-4
Perfluoroheptanoic acid	PFHpA	375-85-9	Per- and Polyfluoroether Carboxylic Acids		
Perfluorooctanoic acid	PFOA	335-67-1	Hexafluoropropylene oxide dimer acid	HFPO-DA	13252-13-6
Perfluorononanoic acid	PFNA	375-95-1	4,8-Dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4
Perfluorodecanoic acid	PFDA	335-76-2	Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
Perfluoroundecanoic acid	PFUnA	2058-94-8	Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5
Perfluorododecanoic acid	PFDoA	307-55-1	Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6
Perfluoroalkyl Sulfonic Acids			Ether Sulfonic Acids		
Perfluorobutanesulfonic acid	PFBS	375-73-5	9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	756426-58-1
Perfluoropentanesulfonic acid	PFPeS	2706-91-4	11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9
Perfluorohexanesulfonic acid	PFHxS	355-46-4	Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7
Perfluoroheptanesulfonic acid	PFHpS	375-92-8			
Perfluorooctanesulfonic acid	PFOS	1763-23-1			
CASN – Chemical Abstracts Service Numbers					

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3.2 Specific Reporting Requirements for WSC-CAM-X B

Specific QC requirements and performance standards for WSC-CAM-X B are presented in Table X B-1. Specific reporting requirements for WSC-CAM-X B are summarized below in Table X B-3 as “Required Analytical Deliverables (YES)”. Requirements listed as “YES” must always be included as part of the laboratory deliverable for this method. It should be noted that data for those items listed as “NO” under “Required Analytical Deliverables” must be available for review during an audit and may also be requested for inclusion in the analytical deliverable on a client-specific basis.

3.2.2 Sample Dilution

Under circumstances that sample dilution is required because either the concentration of one or more of the target analytes exceed the concentration of their respective highest calibration standard or any non-target peak exceeds the dynamic range of the detector (i.e., “off scale”), the RL/LLOQ for each target PFAS must be adjusted (increased) in direct proportion to the Dilution Factor (DF).

Two options are available for dilutions:

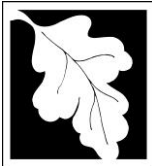
- Option #1: A dilution may be performed on the sample extract. If this is performed, the dilution must be performed with the same solvent/solution as used for sample extracts. It should be noted that refortification of the isotope dilution analogues after dilution is not permitted. The dilution factor must keep the original isotope dilution analogue concentrations within the calibration curve range and recovery must be at least 10% in order to be able to use the isotope dilution analogue for quantitation. If the %R of the isotope dilution analogue is <10%, option #2 must be performed for the target PFAS associated with the isotope dilution analogue non-conformance.
- Option #2: A reduced volume of sample can be extracted.

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

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

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


NOTE: Over dilution is an unacceptable laboratory practice. The post-dilution concentration of the target analyte with the highest concentration must be within the calibration range and 10 times higher than the RL/LLOQ. This will avoid unnecessarily high RLs/LLOQs for other target analytes which did not require dilution.



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Table X B-3 Routine Reporting Requirements for WSC-CAM-X B (USEPA 533)

Parameter	Required Analytical Deliverable
Mass Calibration	NO
Retention Time Windows	NO
Initial Calibration	NO
Continuing Calibration Check	NO
Instrument Blank	NO
Method Blank	YES
Laboratory Fortified Blank (LFB)	YES
Isotope Dilution Analogues	YES
Isotope Performance Standards	NO
Matrix Spike (MS)	YES (if requested by data user)
Matrix Spike Duplicate (MSD)	YES (if requested by data user)
Matrix Duplicate (MD)	YES (if requested by data user)
Identification and Quantitation	NO
General Reporting Issues	YES

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Appendix XB-1

Sample Collection, Preservation, and Handling Procedures for PFAS Analyses of Private Well Samples

Sample preservation, container and analytical holding time specifications for private well samples for PFAS analyzed in support of MCP decision-making are summarized below and presented in Appendix VII A-1 of WSC-CAM-VII A, *Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data Conducted in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)*.



Quality Control Requirements and Performance Standards for the ***Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Private Wells by Liquid Chromatography/Dual Mass Spectrometry (LC-MS/MS)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Matrix	Container ¹	Preservation ²	Holding Time ³
Aqueous Samples	(2) 100-250 mL polypropylene or polyethylene container w/ polypropylene screw caps	Ammonium acetate (added as solid to achieve 1 g/L concentration) Cool to ≤ 10°C after collection and transfer to laboratory within two days of collection. Store samples at ≤ 6°C at laboratory.	Extraction: within 28 days of collection ⁴ Analysis: within 28 days of extraction


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 10°C are acceptable.

³Holding time begins from time of sample collection

⁴Extracts must be stored in polypropylene vials at room temperature.

Note: According to Appendix IV of MassDEP Policy #WSC-25-350, *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. Greater than 28 days results in potential low bias for polyfluoroalkyl PFAS and potential high bias for perfluoroalkyl PFAS.

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Appendix X B-2

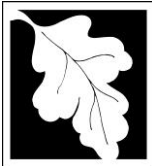
Data Deliverable Requirements for Data Audits



Quality Control Requirements and Performance Standards for the ***Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Private Wells by Liquid Chromatography/Dual Mass Spectrometry (LC-MS/MS)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

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

DELIVERABLE REQUIREMENTS FOR DATA AUDITS WSC-CAM-X B (PFAS by LC-MS/MS: USEPA 533)	
Laboratory Narrative	Must comply with the required laboratory narrative contents as described in WSC-CAM-VII A
Sample Handling Information	Chains-of-custody (external and internal), sample receipt logs (cooler temperatures), correspondences
Miscellaneous Logs	Injection logs Sample preparation logs ¹
Mass Calibration Data	Documentation that initial mass calibration performed annually, at a minimum
Initial Calibration Data	Summary of response factors for all standards in initial calibration; average response factors and linear/non-linear regression curves for all target PFAS and isotope dilution analogues Extracted ion chromatograms for all target PFAS, isotope dilution analogues, and isotope performance standards included in initial calibration Quantitation reports for all standards used in initial calibration Concentrations of target PFAS, isotope dilution analogues, and isotope performance standards used must be clearly presented Raw data for qualitative standards used for linear and branched PFAS isomers, if included Summary of %Rs for each target PFAS in each initial calibration standard using the average response factors and/or linear/non-linear regression curves, as applicable
Continuing Calibration Check Data	Summary of %Rs for all target PFAS and isotope dilution analogues Extracted ion chromatograms for all CCC standards Quantitation reports for all CCC standards Concentrations of target PFAS, isotope dilution analogues, and isotope performance standards used must be clearly presented



Quality Control Requirements and Performance Standards for the **Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Private Wells by Liquid Chromatography/Dual Mass Spectrometry (LC-MS/MS)** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

**DELIVERABLE REQUIREMENTS FOR DATA AUDITS WSC-CAM-X B
(PFAS by LC-MS/MS: USEPA 533)**

Sample Results	<p>Extracted ion chromatograms for each target PFAS, isotope dilution analogue, and isotope performance standard for all sample analyses, re-analyses, re-extractions, and dilutions</p> <p>Quantitation reports for all sample analyses, re-analyses, re-extractions, and dilutions</p> <p>Summary of results, including RLs/LLOQs for each target PFAS</p> <p>Dates of extraction and analysis</p>
Method Blank and Instrument Blank Results	<p>Extracted ion chromatograms for each target PFAS, isotope dilution analogue, and isotope performance standard for all blanks</p> <p>Quantitation reports for all blanks</p> <p>Summary of results, including RLs/LLOQs</p> <p>Summary of how method blank was prepared</p>
LFB Results	<p>Extracted ion chromatograms for each target PFAS, isotope dilution analogue, and isotope performance standard for r all LFBs</p> <p>Quantitation reports for all LFBs</p> <p>Summary of results, including concentrations detected, concentrations spiked, and %Rs</p> <p>Summary of how LFBs were prepared</p>
MS/MSD Results (if performed)	<p>Extracted ion chromatograms for each target PFAS, isotope dilution analogue, and isotope performance standard for all MS/MSDs</p> <p>Quantitation reports for all MS/MSDs</p> <p>Summary of results, including unspiked sample concentrations, concentrations detected, concentrations spiked, %Rs, and RPDs</p> <p>Summary of how MS/MSDs were prepared</p>
Matrix Duplicate Results (if performed)	<p>Extracted ion chromatograms for each target PFAS, isotope dilution analogue, and isotope performance standard for all MDs</p> <p>Quantitation reports for all MDs</p> <p>Summary of results, including concentrations detected in the original and duplicate analyses and RPDs</p>
QC Summaries	<p>Isotope dilution analogue %Rs</p> <p>Summary of isotope performance standard area count %Rs</p>

Quantitation reports must exhibit quantitation (product) ion area counts of target PFAS, isotope dilution analogues, and isotope performance standards.

¹Must clearly indicate sample volumes, solvents used, final extract volumes, extraction method used, SPE cartridge, elution solvents, procedures used for aqueous samples with elevated particulates, where appropriate