# MassDEP Drinking Water Program
## PFAS QC Guidance - Data Report Content for eDEP

MassDEP certified laboratories are required to submit PFAS results electronically to MassDEP using the eDEP portal. The eDEP data files must be complete and properly formatted to be accepted by eDEP. This format is described elsewhere ("upload02 w PFAS Aug 2021"). In addition to the eDEP data file, laboratories are required to append a full and complete laboratory report as described in this document.

The following requirements are for analytical laboratories that analyze drinking water samples for PFAS from Massachusetts Public Water Systems (PWS). The purpose of the following is to ensure that all necessary QC information associated with drinking water PFAS analyses is provided to allow for the review of data reports by clients and regulatory agencies. Note, MassDEP may request additional QC information beyond the contents listed below. Failure to properly report sampling results or QC may result in report rejection, resubmission, and/or resampling.

## eDEP Attachment: Additional PFAS Lab Data QA/QC Report Minimum Content

### General / Field Sample Specific Results

- **Primary and sub-lab lab name(s) and certification ID#(s).**
- **Sample type** (i.e., Field Sample, Field Blank) with assigned lab sample numbers.
- **Sample preservation/storage** conditions upon lab receipt.
- **Method used**: unmodified US EPA Method 537 (Version 1.1, September 2009) or unmodified EPA 537.1 (Version 1.0, November 2018). Note: Method 533 is expected to be approved for use in 2021.
- **Target Analytes.** All target analytes within the analysis method must be reported (Method 537 – 14 contaminants, Method 537.1 – 18 contaminants) with full name and acronym. **Also report analyte "PFAS6"** (sum of PFHpA, PFHxS, PFNA, PFOS, PFOA, PFDA) as though it is a target analyte. Only include results at or above the MRL (ND and J values are considered '0' when summing). Use default value of 2.0 ng/L for MDL/MRL eDEP fields.
- **MDL (required by MassDEP)** for all target analytes. The report must make it clear how Field Reagent Blanks and Lab Reagent Blanks are demonstrated to be less than the greater of 1/3 MRL or MDL.
- **MRL** for all target analytes. (note: Required for all reports except Bacteria) The MRL must be shown equal to or less than 2.0 (maximum acceptable is < 2.05) ng/L for MassDEP PFAS6 analytes.
- **Lab Sample ID** assigned by the Primary Lab as well as that assigned by any Subcontracted Lab, if the analysis was not analyzed by the Primary Lab.
- **MassDEP Location (LOC) ID#** with corresponding sample Location Name. PWS and laboratory must use the correct MassDEP Location Name (LOC) ID# and Location Name on the Chain of Custody (COC) and in lab report submissions.
- **Dates collected, extracted, and analyzed**.
- **Dilution factor**.
- **Data results** for each sample analyte (reported in ng/L or ppt).
- **Surrogates and % recoveries**, including acceptable recovery range table (70-130%)
- **Qualified results** identified/flagged (if any). Include a Result Qualifier Code with Description for each analyte associated with the estimated values or failed/suspect QC parameter.
- **Field Reagent Blank (FRB)** i.e., field blank (if necessary) analytes must be shown to be less than the greater of 1/3 MRL or MDL with associated surrogate recoveries. NOTE: The COC and lab report must make it clear which field samples are associated with each field blank.

**Batch QC**

- **Laboratory Reagent Blank (LRB)** i.e., method blank, analytes must be shown to be less than the greater of 1/3 MRL or MDL with associated surrogate recoveries.
- **Laboratory Fortified Blank (LFB)**. Report % recoveries and associated surrogate recoveries. Indicate fortified concentration used (low, medium or high) along with associated acceptable limits.
- **Lab Fortified Sample Matrix (LFSM)**, and LFSM duplicate or Field Duplicate (FD) are required in each extraction batch. Report spike recoveries, surrogate recoveries and RPD when one of the samples spiked/duplicated is conducted upon a client field sample in the report. Identify and report which sample was spiked (i.e., lab sample ID), spike levels, associated recoveries, and acceptable recovery ranges with RPD and % recovery.
- **Internal Standard (IS)** recoveries that do not meet method requirements. If IS recoveries aren't included in the body of the report, any failure to meet method requirements must be detailed in the narrative.
- **Continuing Calibration Checks (CCC)** that fail to meet method requirements. If CCC results aren't included in the body of the report, any failure to meet method requirements must be detailed in the narrative.

**Glossary**

- Define all QC parameters and qualifier codes, acronyms, or abbreviations used in the report.

**Custody Records**

- Include copy of chains of custody. If subcontracted include both chains of custody. Refer to Chain of Custody regulatory requirements [310 CMR 22.03(10)(b) and 310 CMR 42.08(5)(a)7.e.].

**Dilutions**

- If a dilution is needed to get any target analytes within the calibration range, report each target analyte once using the least diluted analysis possible. Surrogate recoveries outside of control limits for diluted analyses do not need to be qualified. Dilution analyses require a separate PFAS Report Form for each dilution.

**Reporting**

- **All PFAS report submissions must include the laboratory analysis and QC information, along with all COCs, as an attachment to the eDEP submission.**
- Field Blank information must be included in the lab QC attachment (not uploaded as a field sample).

## Reporting Qualified Results for a PWS Field Sample

- Use a unique qualifier code for each parameter outside of control limits. See below for examples.
- Detections reported ≥ MDL and <MRL must be reported as qualified (ex. 'J') with an associated qualifier description.
- **Report analyte "PFAS6"** (sum of PFHpA, PFHxS, PFNA, PFOS, PFOA, PFDA) as though it is a target analyte. Only include results at or above the MRL (ND and J values are considered '0' when summing). Use default value of 2.0 ng/L for MDL/MRL eDEP fields.

| CAS# | REGULATED PFAS CONTAMINANTS | Result[1] ng/L | Result[2] Qualifier | MCL* ng/L | MDL ng/L | MRL ng/L |
|---|---|---|---|---|---|---|
| 1763-23-1 | Perfluorooctane Sulfonic Acid (PFOS) | 6.72 | | | 0.453 | 1.84 |
| 335-67-1 | Perfluorooctanoic Acid (PFOA) | 0.773 | J | | 0.574 | 1.84 |
| 355-46-4 | Perfluorohexane Sulfonic Acid (PFHxS) | 1.36 | J | - | 0.442 | 1.84 |
| 375-95-1 | Perfluorononanoic Acid (PFNA) | ND | | | 0.438 | 1.84 |
| 375-85-9 | Perfluoroheptanoic Acid (PFHpA) | 3.74 | | | 0.239 | 1.84 |
| 335-76-2 | Perfluorodecanoic acid (PFDA) | ND | | | 0.593 | 1.84 |
| PFAS6 (sum of PFOS, PFOA, PFHxS, PFNA, PFHpA and PFDA; only include Results at or above the MRL; do not include estimated Results as described by a Result Qualifier in the next column) = | | 10.46 | -- | 20 | - | - |

- For qualified data, include a **Result Qualifier** code with Description for EACH field sample target analyte associated with the estimated values or failed/suspect QC parameter.
- Enter a **Qualifier Description** for each code used in the report. The following codes are recommended:

| Result Qualifier | Qualifier Description |
|---|---|
| **J** | = Estimated Value. (Estimated result is between the MDL and MRL.) |
| **B** | = Field Reagent Blank (FRB) or field blank (FB) criteria not met, potential result bias. |
| **SUR** | = Associated surrogate recovery criteria not met, result suspect. |
| **IS** | = Internal Standard out of limits on this result. |
| **Q** | = Associated Batch QC out of limits. This includes the Lab Reagent Blank (Method Blank), Lab Fortified Blank (Lab Control Standard), Lab Fortified Sample Matrix (Matrix Spike), Lab Fortified Sample Matrix Duplicate (Matrix Spike Duplicate), Field Duplicate, and Continuing Calibration Check, as well as the Surrogates on these Batch QC samples. |

In addition to the SUR above you must attach the results of the ongoing QC results as specified by the method for the sample's extraction batch.

☒ **Laboratory analytical report with QC attached (check one item below).**

☐ All associated QC criteria reported within control limits including Lab Reagent/Method Blank (LRB), Field Reagent Blank (FRB), Surrogate Standards (SUR), Laboratory Fortified Blank (LFB), Matrix Spike/Duplicate (LFSM/LFSMD or FD) and RPD.

☒ All associated sample and/or QC batch criteria not met. See Lab Analysis Comments below and narrative in attached report.

**Lab Analysis Comments:** (include sample/method parameters outside of or affecting QC controls/limits and result qualifiers)

| Result Qualifier | Qualifier Description |
|---|---|
| J | ESTIMATED VALUE |
| SUR | ASSOCIATED SURROGATE RECOVERY CRITERIA NOT MET, RESULT SUSPECT. |
| | |
| | |
| Other Analysis Comments: | |

- If a surrogate recovery is outside control limits and is 'qualified' place a corresponding qualifier code (ex. SUR) in the qualifier field located next to **EACH** target analyte in the field sample associated with the failed surrogate ***(see Table 1 for surrogates and associated target analytes).***
  **Ex.** If $d_5$-NEtFOSAA surrogate recovery is out of control limits (ex. 68%) in a field sample, **EACH** of the following associated target analytes in the PWS field sample must be identified with a qualifier in the corresponding eDEP (or MassDEP report form) fields along with a qualifier description: *NMeFOSAA, NEtFOSAA*

**eDEP upload example:**

| Report Type Code | AnalyteName | CAS Registry Number | Analyte Measurement Qualifier | Analyte Meas Qualifier Desc | Analyte Measurement Value | Analyte Measurement Unit | MRL Measurement Value | MDL Measurement Value |
|---|---|---|---|---|---|---|---|---|
| PFAS | N-ETHYL PERFLUOROOCTANESULFONAMIDOACETIC ACID - NETFOSAA | 2991-50-6 | SUR | ASSOCIATED SURROGATE RECOVERY CRITERIA NOT MET, RESULT SUSPECT | ND | NG/L | 1.84 | 0.876 |
| PFAS | N-METHYL PERFLUOROOCTANESULFONAMIDOACETIC ACID - NMEFOSAA | 2355-31-9 | SUR | ASSOCIATED SURROGATE RECOVERY CRITERIA NOT MET, RESULT SUSPECT | ND | NG/L | 1.84 | 0.862 |

# MassDEP QC Reference Tables for PFAS Data

| Basic QC | Criteria |
|---|---|
| **PFAS Method** | EPA 537 (Rev 1) unmodified, EPA 537.1 |
| **Lab Certification** | Lab is MassDEP certified to use Method 537, or 537.1 for PFAS<br>https://eeaonline.eea.state.ma.us/DEP/Labcert/Labcert.aspx |
| **Sample Holding Time (collection to extraction)** | ≤ 14 days between sample collection and extraction. |
| **Extract Holding Time (extraction to analysis)** | ≤ 28 days between sample extraction and analysis. |
| **Minimum Reporting Limit (MRL)** | ≤ 2.0 ng/L or ppt for PFOA, PFOS, PFHxS, PFHpA, PFNA, PFDA<br>(Note: maximum acceptable is <2.05 ppt) |
| **Method Detection Limi (MDL)** | Report submission includes the MDL for all reported method analytes. |
| **Total PFAS6** | Submission includes sum of results ≥ MRL for PFOA, PFOS, PFHxS, PFHpA, PFNA, PFDA<br>(Notes: 'J' values reported below the MRL are not added to the total. ND is equivalent to '0'.) |
| **QC Submitted** | Report submission includes minimum QC: Surrogate recoveries for Field Sample/Field Blank (if required). Batch QC includes LRB, LFB and surrogate recoveries. Matrix spike/matrix spike duplicate or field duplicate, RPDs and surrogate recoveries (if one of the client field samples was spiked). |
| **Preservation & Storage** | Samples should be received by laboratory in proper condition. *Note variations of sample receipt conditions under the **Sample Notes** field on the MassDEP report form or corresponding eDEP field.* |
| ≤ 10 °C | Samples must be chilled during shipment and must not exceed 10 °C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 10 °C when the samples are received at the laboratory. All samples shall be protected from light and refrigerated at ≤ 6 °C (but not frozen) from the time of receipt at the laboratory. Sample extracts shall be stored at room temperature from the time of the extraction completion until analysis. *(Include in narrative if samples may not have had time to cool down after a short time between sample collection and lab receipt)* |
| 7.0 pH | Preservation agent (5.0 g/L Trizma) added to each bottle as buffering agent and removal of free chlorine. (EPA 537, 537.1) |

| Field Sample QC | Criteria | Field Sample Reporting Actions | |
|---|---|---|---|
| **Surrogate Standards Method 537 & 537.1** | Surrogate recoveries reported for the specified method are within 70-130% recovery limits for the Field Sample and field blank. {EPA 537 = 3 surrogates, EPA 537.1 = 4 surrogates} *See Table 1* | **Result Qualifier** | |
| | | **Detect** | **Non-Detect** |
| Recoveries within 70-130% | Criteria met. No comments. | None | None |
| One or more recoveries outside control limits. | Report Field Sample results for method analytes associated with the failed surrogate as **suspect** (qualified) with description (ex. surrogate recovery criteria not met). *See Table 1* | SUR | SUR |
| | Exception, if recovery > 130% report associated field sample detects at or above the MRL as qualified, non-detects below the MRL do not require qualification. | SUR | None |
| **Field Reagent Blank (FRB) or Field Blank (FB)** | Analysis of a Field Blank is required only if the Field Sample associated with the blank contains method analyte detections at or above the MRL. | **Result Qualifier** | |
| | | **Detect** | **Non-Detect** |
| < 1/3 MRL or Non-Detect | Criteria met. No comments. | None | None |
| One or more detections in the blank ≥ 1/3 MRL | Field Sample results with ≥ 1/3 MRL detection of the same analyte in the associated field blank are **invalid.** Report Field Sample method analyte detections associated with the field blank detections as qualified with description (ex. Field Blank criteria not met, potential result bias). | B | None |
| | | | |

| BATCH QC | Criteria | Field Sample Reporting Actions | |
|---|---|---|---|
| **Lab Reagent Blank (LRB) or Method Blank (MB)** | A reagent water blank is prepared with the analytical batch to ensure the sample extraction and analysis procedures do not contribute contamination. | **Result Qualifier** | |
| | | **Detect** | **Non-Detect** |
| < 1/3 MRL or Non-Detect | Criteria met. No comments. | None | None |
| ≥ 1/3 MRL | Field Sample results with detections of the same analyte in the method blank ≥ 1/3 MRL are **invalid**. Report Field Sample method analyte detections associated with the Extraction Batch detections as qualified with description (ex. Method Blank criteria not met, potential result bias). | Q | None |
| | | | |
| **Laboratory Fortified Blank (LFB) or LCS or BS** | LFB is prepared with the analytical batch to evaluate the accuracy of the analytical method and laboratory performance (rotated between low, medium and high amounts). | **Result Qualifier** | |
| | | **Detect** | **Non-Detect** |
| 70-130% for med/high 50-150% for low | Recoveries within control limits. | None | None |
| Recoveries outside control limits. | Field Sample results associated for the problem analytes are considered **invalid** for all samples in the Extraction Batch. Report Field Sample results as qualified with description (ex. LFB criteria not met, potential result bias). | Q | Q |
| | Exception, Field Samples results with no detections ≥ MRL are acceptable and do not require qualification if the LFB exceeded the target recovery upper acceptance limit (**130 or 150%**). | Q | None |

| **Laboratory Fortified Sample Matrix & Duplicate (LFSM/LFSMD) or Field Duplicate (FD)** | A lab fortified matrix spike and matrix spike duplicate (or field duplicate) are prepared with the extraction batch. The percent recoveries of target analytes are calculated to measure accuracy. The relative percent difference is calculated to measure precision**.** **NOTE: Only the field sample that was actually spiked requires qualifiers for LFSM/LFSMD/FD exceeding limits.** | **Result Qualifier** | |
|---|---|---|---|
| | | **Detect** | **Non-Detect** |
| 70-130% for med/high spike<br>RPD ≦ 30% for med/high spike<br><br>50-150% for low spike (within 2x MRL)<br>RPD ≦ 50% low spike (within 2x MRL) | Recoveries and RPDs reported within control limits. | None | None |
| Outside control limits. | If the RPD or Recovery of any analyte falls outside the designated range it is considered to be matrix biased. Report Field Sample results for the problem analyte as **suspect** (qualified) with description (ex. LFSM and/or RPD recovery criteria not met, potential matrix interference) | Q | Q |
| | Exception, Field Samples with method analytes < MRL are acceptable and do not require qualification if the recovery or RPD for the problem analyte exceeded the upper acceptance limits (**130 or 150%; RPD 30 or 50%)**. | Q | None |
| **Surrogate Standards on batch QC samples** | Surrogate standards added to all field samples and batch QC. All surrogate recoveries reported for the specified method are within 70-130% recovery limits. {EPA 537 = 3 surrogates, EPA 537.1 = 4 surrogates} *See Table 1* | **Result Qualifier** | |
| | | **Detect** | **Non-Detect** |
| Recoveries within 70-130% | Criteria met. No comments. | None | None |
| Recovery outside control limits. | Report Field Sample results for method analytes associated with the failed surrogate as **suspect** (qualified) with description (ex. surrogate recovery criteria in batch QC not met, suspect). *See Table 1* | Q | Q |
| | Exception, if recovery > 130% report field sample detects at or above the MRL as qualified, non-detects do not require qualification. | Q | None |

| Table 1. Surrogate Standards and Associated Target Analytes for EPA Methods 537 and 537.1 | | |
|---|---|---|
| **EPA Method** | **Surrogate Standard** | **Associated Target Analytes** |
| 537 | $^{13}$C-PFHxA<br>$^{13}$C-PFDA | **PFOA**, PFHxA, **PFHpA**, **PFNA, PFDA**, PFUnA, PFDoA, PFTrDA, PFTA, **PFOS**, PFBS, **PFHxS** |
| | $d_5$-NEtFOSAA | NMeFOSAA, NEtFOSAA |
| 537.1 | $^{13}C_2$-PFHxA<br>$^{13}C_2$-PFDA | **PFOA**, PFHxA, **PFHpA**, **PFNA, PFDA**, PFUnA, PFDoA, PFTrDA, PFTA, **PFOS**, PFBS, **PFHxS** ADONA, 11C1-PF3OUdS, 9C1-PF3ONS |
| | $^{13}C_3$-HFPO-DA | HFPO-DA |
| | $d_5$-NEtFOSAA | NMeFOSAA, NEtFOSAA |

| Table 2. Internal Standards and Associated Target Analytes/Surrogates for EPA Methods 537 and 537.1 | | |
|---|---|---|
| **EPA Method** | **Internal Standard** | **Associated Target Analytes and Surrogates** |
| 537 | $^{13}$C-PFOA | **PFOA**, PFHxA, **PFHpA**, **PFNA**, **PFDA**, PFUnA, PFDoA, PFTrDA, PFTA, $^{13}$C-PFHxA, $^{13}$C-PFDA |
| | $^{13}$C-PFOS | **PFOS**, PFBS, **PFHxS** |
| | $d_3$-NMeFOSAA | NMeFOSAA, NEtFOSAA, $d_5$-NEtFOSAA |
| 537.1 | $^{13}C_2$-PFOA | **PFOA**, PFHxA, **PFHpA**, **PFNA**, **PFDA**, PFUnA, PFDoA, PFTrDA, PFTA, HFPO-DA, ADONA, $^{13}C_2$-PFHxA, $^{13}C_2$-PFDA, $^{13}C_3$-HFPO-DA |
| | $^{13}C_4$-PFOS | **PFOS**, PFBS, **PFHxS**, 11C1-PF3OUdS, 9C1-PF3ONS |
| | $d_3$-NMeFOSAA | NMeFOSAA, NEtFOSAA, $d_5$-NEtFOSAA |

## Abbreviations:

| | |
|---|---|
| BS | Blank Spike (also known as LCS or LFB) |
| BQC | Batch Quality Control |
| °C | Degrees Centigrade |
| CCC | Continuing Calibration Check |
| COC | Chain of Custody form |
| DEP | Massachusetts Dept of Environmental Protection |
| DL | Detection Limit |
| eDEP | DEP's electronic reporting system for drinking water lab results |
| EPA | U. S. Environmental Protection Agency |
| FB | Field Blank (also known as FRB) |
| FD | Field Duplicate (also known as Duplicate Sample) |
| FRB | Field Reagent Blank |
| FS | Field Sample (i.e., client sample) |
| ID | Identification |
| IS | Internal Standard |
| LCS | Lab Control Sample (also known as LFB) |
| LFB | Lab Fortified Blank (also known as LCS or BS) |
| LFSM | Lab Fortified Sample Matrix |
| LFSMD | Lab Fortified Sample Matrix Duplicate |
| MB | Method Blank (also known as LRB or Blank) |
| MDL | Method Detection Limit |
| MRL | Minimum Reporting Limit |
| MS | Matrix Spike (or Laboratory Fortified Sample Matrix) |
| MSD | Matrix Spike Duplicate (or LFSMD) |
| ng/L | Nanograms/liter (or parts per trillion, ppt) |
| PFAS | Per- and Poly-Fluoroalkyl Substances |
| PFAS6 | The 6 PFAS compounds regulated by MassDEP |
| ppt | Parts per trillion (or nanograms/liter, ng/L) |
| PWS | Public Water Supply |
| QC | Quality Control |
| RL | Reporting Limit |
| RPD | Relative Percent Difference |
| SUR | Surrogate Standard |