Instructions for EPA's External Multi-Laboratory Validation Study for PFAS in Soil Using Draft Method (Based on EPA Region 5 Method) by LC/MS/MS

STUDY-SPECIFIC INSTRUCTIONS FOR THE EPA AND EXTERNAL VALIDATION STUDY

Introduction

EPA has developed a liquid chromatography tandem mass spectrometry (LC/MS/MS) method to analyze for per- and poly-fluorinated alkyl substances (PFAS) in soil matrices using simple extraction techniques. The sample extracts are analyzed using direct injection (into the LC). Ten laboratories (external and EPA laboratories) have agreed to participate in this external validation study using four types of soil matrices. It is anticipated that 63 samples (60 soils, 1 Quality Control (QC) sample and 2 trip blanks) will be received by each participating laboratory for PFAS analysis using the procedures specified in the draft method (Based on EPA Region 5), Rev. 0.1, dated November 7, 2018. These instructions are for the external validation study for the 24 PFAS analytes specified in the method. Laboratory QC sample preparation required by the method will be the responsibility of each participating laboratory.

Project Manager/Point of Contact for Study

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Analyte List

Analyte		CAS RN
PFAS sulfonic acids		
Perfluorobutyl sulfonic acid	PFBS	29420-49-3
Perfluorohexyl sulfonic acid	PFHxS	3871-99-6
Perfluorooctyl sulfonic acid	PFOS	1763-23-1
1H, 1H, 2H, 2H-perfluorohexane sulfonic acid	4:2 FTS	757124-72-4
1H, 1H, 2H, 2H-perfluorooctane sulfonic acid	6:2 FTS	27619-97-2
1H, 1H, 2H, 2H-perfluorodecane sulfonic acid	8:2 FTS	39108-34-4
Perfluoro-1-pentanesulfonic acid	L-PFPeS	706-91-4
Perfluoro-1-heptanesulfonic acid	L-PFHpS	375-92-8
Perfluoro-1-nonanesulfonic acid	L-PFNS	68259-12-1
Perfluoro-1-decanesulfonic acid	L-PFDS	2806-15-7
PFAS carboxylic acids		
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9

Analyte		CAS RN
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnA	2058-94-8
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluorotridecanoic acid	PFTriA	72629-94-8
Perfluorotetradecanoic acid	PFTreA	376-06-7
PFAS sulfonamides and sulfonamidoacetic acids		
N-ethylperfluoro-1-octanesulfonamidoacetic acid	N-EtFOSAA	2991-50-6
N-methylperfluoro-1-octanesulfonamidoacetic acid	N-MeFOSAA	2355-31-9
Perfluoro-1-octanesulfonamide	FOSA	754-91-6

General Considerations

As a study participant, the laboratory agrees to follow the instructions provided below. The purpose of these instructions is to provide an overview of the timeframe, sample receipt and identification, and study-specific instructions on methodology and reporting in addition to those specified in the draft method.

1. Unless otherwise specified in these instructions, laboratories will follow the procedures *as written* in the draft method. Any planned deviations from these procedures *must be* submitted in writing to project managers/points of contact and approved for use prior to the analysis of any samples in this study. Any unanticipated deviation from these procedures *must be* described in the report narrative for each analytical batch.
2. Laboratories will be provided columns, standards, and other supplies. Instrument operating conditions specified in the draft method must be followed. Instrument settings provided in the draft method are based on the Waters Acquity UPLC® with Xevo TQ-S; other instrument models or vendor brands may require different settings and must be optimized by the laboratory. Operating conditions need to be documented and supplied with data submissions.

Laboratories must report the results for all target compounds and surrogates regardless of the results. If results are above or below the calibration ranges, the laboratory shall indicate that the result is an estimate, on both the data reports and in the report narrative. Data reporting instructions for the validation study are provided in Attachment 3. Reporting Requirements and Attachment 4, Data Qualifiers provides additional information on the use of data qualifiers (flags) for this external validation of the method.

Timeframe

Participating laboratories will receive samples the week of December 3, 2018. Partial data packages from participating laboratories (as described in Attachment 3) should be sent to EPA within four

weeks of the receipt of samples. Complete data packages should be sent to EPA (as described in Attachment 3) within eight weeks of sample receipt. See the timeline below for the analysis of samples for the validation study.

Table 1. Anticipated Schedule Summary

Task	Who	Expected Completion Date
Shipment of Validation Study Samples	EPA	12/07/2018
Submission of preliminary results	Participating Labs	01/07/2019
Submission of Analytical Data Packages to EPA	Participating Labs	02/04/2019

List of Equipment and Supplies

To ensure consistent results, EPA will provide all participating laboratories with the equipment and supplies listed in Attachment 1. All other equipment and supplies are listed in the draft method. Purchasing standard laboratory supplies is responsibility of the participating laboratory.

Instructions for Preparation of Standards and Surrogates

For the purposes of this validation study, laboratories will receive target compound and surrogate (mass-labelled) PFAS mixes from Wellington (PFAC-24PAR [target compounds] and MPFAC-24ES [surrogates}) at concentrations from of 2000 ng/mL and 1000 ng/mL, respectively. Attachment 2: *List of PFAS Calibration Standards and Concentrations for Multi-Lab Validation Study 2018* includes the target analytes, surrogates, and concentrations for the nine levels of the calibration curve. See Section 7.4 of the attached draft method for additional standard and spiking solution preparation instructions. Please note the instructions for standard preparation are examples only and may be modified as long as the final concentrations and solvent composition are achieved.

Instrument Operating Conditions:

Refer to the draft method, Appendix 2 for specific instrument operating conditions. Alternate instrument models and vendors may require different settings. Submit any modifications in advance of completing the analyses of study samples.

Use of Surrogates and Internal Standards

Refer to the draft method for surrogates (Section 7.4.1 and 11.0). No internal standards are used for the validation study.

Extraction Method

Laboratories are to follow the sample preparation/extraction procedures in Section 11.1 and 11.2 of the draft method.

Procedures for External Validation Study

A total of 61 samples and two Trip Blanks (TBs) will be shipped to each participating laboratory consisting of four matrix-types (fat clay, lean clay, silt, and sand). Table 2 provides the sample type, number of spiked and unspiked samples and the number of replicates for each type. Concentration levels will be within the specified calibration range and unknown to participating laboratories. Native soil as well as two concentration levels (low and high) will be submitted for each matrix type. Low and high concentration samples will be spiked with all 24 analytes included in this study. A statistically randomized run order for samples will be provided to laboratories in the shipment coolers and emailed to the laboratory POC; the run order must be followed. The two Trip Blanks may be added to a run either immediately prior to field samples or immediately after. Any deviations to the designated run order (other than the addition of TBs) must be noted in the report narrative for each batch. Chain-of-Custody forms using Scribe will be used to document sample identification and transfer of samples. Upon receipt, laboratories need to sign Chain-of-Custody forms and later include them in the final data package.

Table 2. Sample Types and Numbers

Matrix and Concentration	Number of samples and container specifications
Fat Clay- Native	5 X 2 gm Falcon tubes*
Fat Clay – Low Spike	5 X 2 gm Falcon tubes *
Fat Clay – High Spike	5 X 2 gm Falcon tubes *
Lean Clay – Native	5 X 2 gm Falcon tubes *
Lean Clay – Low spike	5 X 2 gm Falcon tubes *
Lean Clay – High spike	5 X 2 gm Falcon tubes *
Slit – Native	5 X 2 gm Falcon tubes *
Slit- Low Spike	5 X 2 gm Falcon tubes *
Slit- High Spike	5 X 2 gm Falcon tubes *
Sand-Native	5 X 2 gm Falcon tubes *
Sand- Low Spike	5 X 2 gm Falcon tubes *
Sand- High Spike	5 X 2 gm Falcon tubes *
Trip Blank	2 X 2 gm Falcon tubes*
QC Sample	1 X 2 gm Falcon tubes*

* Polypropylene gm = grams

Exceptions to Draft Method for the Validation Study

Unless otherwise specified in these instructions, laboratories will follow the procedures as written in the draft method. Any planned deviation from these procedures must be submitted in writing to project managers /POCs for the study before the extraction and/or analysis begins. These deviations must be approved for use in this study. Any unanticipated deviation from these procedures must be described in the report narrative for each analytical batch. Exceptions to the method as written are listed in Table 4 on the next page.

While the batch run order for samples will be analyzed according to instructions that will be sent with the sample coolers, the following overall run order including the appropriate lab QC samples must be used. Refer to the table below.

Table 3. General Batch Run Order

Run Order Batch 1	Run Order Batch 2	Run Order Batch 3
RB	RB	RB
ICAL (9 levels)	ICAL (9 Levels)	ICAL (9 Levels)
RB	RB	RB
MB1	MB1	MB1
LLOQ1	LLOQ1	LLOQ1
LLOQ2	LLOQ2	LLOQ2
LCS	LCS	LCS
LCSD	LCSD	LCSD
10 field samples	10 field samples	10 field samples
CCV	CCV	CCV
MB2	MB2	MB2
TB1	TB2	QC Sample
10 field samples	10 field samples	10 field samples
CCV	CCV	CCV

Table 4. Exceptions to Draft Method

Section	Exceptions for the Validation Study
2.2	Data for the validation study will be reported on an "as received" basis. A total solids/moisture analysis will not be required. Sample weights will be supplied to each participating laboratory.
9.6.1, 9.6.2, 11.1.2, 11.1.5	A LCS/LCSD will be prepared and analyzed in lieu of an MS/MSD. QC excursions will require the same data qualifiers applies to the MS/MSD, as described in Section 9.6.1.3.
7.4.6, 9.7	A second-source standard is not available for this study. An ICV will not be prepared and analyzed. ICAL standards will be prepared at *nine* concentrations, as described in Attachment 2: *PFAS Standards List*.
9.4	If the lab participated in the IDP for water, then a soil IDP is not required. The results of the CCV analyses from all three batches will also be used to verify that the precision and accuracy criteria required for the IDP Study are within the performance acceptance criteria over the three 24-hour sequences.
9.5.6, 11.1.1	Clarification: Two Method Blanks (MBs) will be prepared and analyzed for every 20 samples.

Section	Exceptions for the Validation Study
9.5.6, 9.5.8, 9.6.1, 9.6.2, 9.6.3, 9.8, 9.9	Clarification: Each batch of 20 samples must have one RB, two MBs, a LCS/LCSD, two LLOQ verification checks, and two CCVs after the ICAL. A RB will also be analyzed prior to the ICAL to ensure the system is clean. The laboratory may add an additional RB prior to the ICAL at their discretion.
9.6.2	Clarification: Precision and accuracy of duplicates will be determined using the LCS/LCSD. (EPA will later evaluate the precision among the five replicates of each type of matrix/concentration evaluated in the study.)
9.8	A mid-level CCV will be analyzed after every 10 field samples for a total of two in each batch of 20.
9.9.1.5	All values should be reported for which an analyte meets most or all of the qualitative identification criteria. If a compound is non-detect, report with a "U". If a compound is detected but is below the QL, qualify estimated "J". Discuss any qualifications in the case narrative for each analytical batch of 20 samples and associated QC results. (See Data Qualifiers in Attachment 4.)
7.4.6	Clarification: An initial CCV (or ICV) is not required because LCS/LCSD samples will be analyzed at a similar concentration, in addition to the two LLOQ verification checks. Each batch of 20 field samples will be reported on its own data reporting template (see Attachment 6) and have an accompanying narrative report (by batch) describing any QC excursions, data qualifiers, or other deviations from stated instrument or analysis conditions. If the CCV at the end of a batch fails (± 30% recovery), then another CCV may be analyzed. If the 2nd CCV also fails, the previous 10 samples must be qualified, and another ICAL must be analyzed prior to running any additional QC samples or validation study samples.
11.4	The laboratory is required to run a new initial calibration per batch of 20 samples within a 24-hour period. Refer to Table 3 for batch run order.
11.6.1	Follow the sample run order listed in Table 3 of these study instructions.
11.6.4	Clarification: Since an ICAL will be run for each batch, the analyte transition ion ratios will be monitored/evaluated against the average quantifier/qualifier ion ratios calculated from the ICAL standards on the day of analysis.
Appendix 1	Use the data qualifiers in Attachment 4 to these instructions, and describe any additional qualifiers applied to the data in the report narrative.

Quality Assurance and Data Review Requirements

Laboratories shall complete Attachment 5: *Data Completeness Checklist for Participating Laboratories* to document review of data. The Laboratory Manager (or person other than analyst) completes and signs the checklist and submits the checklist along with the complete data package.

Reporting Requirements

Laboratories are requested to provide the EPA project managers/points of contact with results for each of the validation study samples and supporting QC samples four weeks after receipt of samples. A full data package is required eight weeks after receipt of samples. Specific reporting requirements

are listed in Attachment 3. A list of data qualifiers along with their appropriate dilutions can be found in Attachment 4. Instructions for submitting electronic deliverables can be found in Attachment 6.

ATTACHMENTS

Attachment 1 – EPA Provided Supplies

Attachment 2 – PFAS Standards List (with concentrations)

Attachment 3 – Reporting Requirements

Attachment 4 – Data Qualifiers for Use in Reporting

Attachment 5 – Data Completeness Checklist for Participating Laboratories

Attachment 6 – Data Reporting Template (in Excel) and Instructions for Use

Attachment 1. EPA Provided Supplies

EPA is providing a set of supplies for use in the PFAS Multi-Lab External Validation Study to ensure consistency between standards and select equipment.

Item	Vendor	Quantity/lab
15 ml PP centrifuge tubes	Fisher	100
10 ml metal luer-lock all glass syringe	Supelco	30
Native PFAS Precision and Recovery Standard	Wellington (PFAC-24PAR)*	1
Labeled PFAS extraction standard	Wellington (MPFAC-24ES)**	1
CSH phenyl-hexyl column, 1.7 um, 2.1x100mm	Waters	1
BEH C18 column, 3.5 um, 2.1x50mm	Waters	1
Certified amber glass 2 ml screw cap vials with polyethylene septumless caps	Waters	2 packs (200 vials)
Acrodisc PSF GXF/GHP 02. Um filters	Pall	150 filters

POC for Supplies and Shipment Information:

Raj Singhvi - Singhvi.raj@epa.gov ; 732-321-6761

Attachment 2. List of PFAS Calibration Standards and Concentrations for Multi-Lab Validation Study 2018

PARAMETER	LV1	LV2	LV3	LV4	LV5	LV6	LV7	LV8	LV9
PFTreA*	5	10	20	40	60	80	100	150	200
PFTriA*	5	10	20	40	60	80	100	150	200
PFDoA*	5	10	20	40	60	80	100	150	200
PFUnA*	5	10	20	40	60	80	100	150	200
PFDA*	5	10	20	40	60	80	100	150	200
PFDS*	4.83	9.65	19.3	38.6	57.9	77.2	96.5	145	193
PFOS*	4.63	9.25	18.5	37	55.5	74.0	92.5	139	185
PFNA*	5	10	20	40	60	80	100	150	200
PFNS*	4.80	9.60	19.2	38.4	57.6	76.8	96.0	144	192
PFOA*	5	10	20	40	60	80	100	150	200
PFHpS*	4.75	9.50	19.0	38.0	57.0	76.0	95.0	143	190
PFHxS*	4.56	9.12	18.2	36.5	54.7	73.0	91.2	137	182
PFHpA*	5	10	20	40	60	80	100	150	200
PFHxA*	5	10	20	40	60	80	100	150	200
PFPeS*	4.7	9.4	18.8	37.6	56.4	75.2	94	141	188
PFBS*	4.43	8.85	17.7	35.4	53.1	70.8	88.5	133	177
PFPeA*	5	10	20	40	60	80	100	150	200
PFBA*	5	10	20	40	60	80	100	150	200
FOSA*	5	10	20	40	60	80	100	150	200
4:2 FTS*	4.68	9.35	18.7	37.4	56.1	74.8	93.5	140	187
6:2 FTS*	4.75	9.50	19.0	38.0	57.0	76.0	95.0	143	190
8:2 FTS*	4.80	9.60	19.2	38.4	57.6	76.8	96	144	192
NEtFOSAA*	5	10	20	40	60	80	100	150	200
NMeFOSAA*	5	10	20	40	60	80	100	150	200
M4PFBA**	5	10	20	40	60	80	100	150	200
M5PFHA**	5	10	20	40	60	80	100	150	200
M3PFHxS**	4.73	9.45	18.9	37.8	56.7	75.6	94.5	142	189
M8PFOA**	5	10	20	40	60	80	100	150	200
M9PFNA**	5	10	20	40	60	80	100	150	200
M8PFOS**	4.79	9.57	19.1	38.3	57.4	76.6	95.7	144	191
M6PFDA**	5	10	20	40	60	80	100	150	200
M7PFUn**	5	10	20	40	60	80	100	150	200
2PFDoA**	5	10	20	40	60	80	100	150	200
M2-4:2 FTS**	4.68	9.35	18.7	37.4	56.1	74.8	93.5	140	187
M2-6:2 FTS**	4.75	9.50	19.0	38.0	57.0	76.0	95.0	142	190
M2-8:2 FTS**	4.8	9.60	19.2	38.4	57.6	76.8	96.0	144	192
d5-NEtFOSAA**	5	10	20	40	60	80	100	150	200

PARAMETER	LV1	LV2	LV3	LV4	LV5	LV6	LV7	LV8	LV9
d3-NMeFOSAA**	5	10	20	40	60	80	100	150	200
M3PFBS**	4.65	9.29	18.6	37.2	55.8	74.4	93	139	186
M5PFPeA**	5	10	20	40	60	80	100	150	200
M4PFHpA**	5	10	20	40	60	80	100	150	200
M2PFTreA**	5	10	20	40	60	80	100	150	200
M8FOSA**	5	10	20	40	60	80	100	150	200

*Adjusted concentrations reflect Wellington Lot #PFAC24PAR0418.

Attachment 3. Reporting Requirements

Each participating laboratory will provide EPA with a data package for each batch of 20 samples provided by the EPA. To facilitate EPA's prompt analysis of the information derived from this method validation study, partial data packages including validation study results and corresponding QC samples from participating laboratories should be sent to EPA within four weeks of sample receipt. Complete data packages should be sent within six weeks of sample receipt.

An Excel spreadsheet and specific instructions for reporting all analytical results and associated QC data is provided in Attachment 6. Laboratories may choose to use this Data Reporting Template, or use a LIMS output to provide comparable data and metadata in an efficient format. The general content of the partial and complete data packages is described below. The complete data packages are expected to contain all of the material that was submitted in the partial data package.

Someone other than the analyst shall review components of each data package for completeness and conformance to the study requirements, for example, a Laboratory Manager, QA Manager, or other qualified individual. This person must sign-off on each data package using the Data Completeness Checklist in Attachment 5.

The partial data packages will be submitted electronically to the EPA project managers/points of contact. Any hardcopies should be scanned and provided as pdf files. Participating labs can provide electronic data deliverables (EDDs) downloaded from the LIMS to a secure site, sent via email, or submitted on CD for any of the data packages. Full data packages can be submitted electronically in .pdf format to eliminate physically storing/shipping large volumes of paper. All data retained by EPA and the participating laboratories shall be backed up by CD or external hard drive.

Partial data packages for each analytical batch to be delivered to EPA include:

- Completed data reporting template (see Attachment 6) or comparable LIMS report with results of all analyses including QC samples (e.g. blanks, LLOQ verification samples, CCVs, etc.) and draft data qualifiers (see Attachment 4)
- A list of laboratory-specific LLOQs for each analyte (i.e., actual LLOQ = lowest level calibration standard actually used in an acceptable calibration curve)
- A list of deviations from the method, both approved and unanticipated
- Initial interpretation of impact on data from any QC excursions or deviations from method requirements

Complete data packages for each analytical batch to be delivered to EPA include:

- In addition to the information contained in the partial data packages, complete data packages for each analytical batch to be delivered to EPA include:
    - Report narrative
    - Make and model number of the instrument used and a summary of the instrument operating conditions (e.g. temperature ramps, columns, detector and injection conditions). It is important to include all instrument conditions that used to acquire the data. Where possible, submit a representative acquisition method text or .xml file from the calibration data set.
    - Description of any modifications to conditions or procedures described in this set of instructions or the draft method, including those for which the laboratory may have requested and received permission to use.
    - The laboratory should identify any corrective actions that were necessary to improve method performance (e.g., ICAL failures, reanalyses performed, etc.).

- Holding time for sample extraction/analysis
- Sample analysis and extraction dates.
- Explanation of any data qualifiers applied to the data
- Interpretation of impact on data from deviations from QC or method requirements
- Instrument raw data - quantification reports and detailed chromatograms with spectra for all samples and QC samples (including detector area response and retention time);
- Calibration raw data (Initial and continuing) with curve fits, as well as, correlation coefficients of the calibration fit used (percent relative standard error [%RSE] data can be used, if the laboratory tracks calibration acceptability in this manner.)
- Instrument detector tuning verification
- Chain-of-Custody forms
- Standards preparation logs/worksheet
- Extraction log sheets.

Attachment 4. Data Qualifiers for Use in Reporting (*Application Notes on Next Page*)

Qualifier	Definitions	Method Sections
U	The analyte was analyzed for, but not detected above the LLOQ.	
J	The analyte was positively identified (possibly < LLOQ). The associated numerical value is the estimated concentration of the analyte in the sample. • The analyte is calculated to be below a reporting limit of 5 ng/L or 25 ng/kg or below the laboratory's LLOQ. • The response of a RB or MB is > half the response in the LLOQ calibration standard. • The MS/MSD recoveries are one high and one low. • The MS/MSD or duplicate relative percent difference is >30%. • LLOQ verification sample results are <50% recovery, and analyte detections are < the passing LCS level. • LLOQ verification sample results are >150% recovery, and analyte detections are < the passing LCS level. • S/N ≤ 3 for quantifier transitions. • Correlation coefficient r^2 • CCV > ± 30% recovery from true concentration. • Other QC excursions to be explained in report narrative.	9.5.7 9.6.1.3 9.6.1.3 9.6.3 9.9.1.4 11.3.4 11.4
K	High bias. • MS/MSD results high and analyte present. • Indicates reagent or method blank contamination (i.e., when the concentration of target analytes in a blank is greater than half the reporting limit). • Surrogate recovery is >130% recovery and the native analyte is present.	9.6.1.3 9.5.7, 9.5.8 9.10.3
L	Low bias. • MS/MSD results both low and analyte present in the sample above LLOQ • Surrogate recovery is <70% recovery and the native analyte is present.	9.6.1.3 9.10.3
UJ	• MS/MSD results low and analyte not present in the sample above LLOQ • Use for all field sample nondetects if LLOQ verification sample results are at < 50% recovery for the analyte. • Surrogate recovery is <70% recovery and the native analyte is not present.	9.6.1.3 9.9.1.4 9.10.3
H	The sample was analyzed more than 28 days after collection or analyzed more than 14 days after preparation.	Study-specific
I	The quantifier/qualifier MRM ion ratio is outside of ±30% of expected value.	11.6.4

Attachment 4. Data Qualifiers (Continued)

NOTES ON USAGE of Data Qualifiers:

- All QC excursions must be described in the report narrative accompanying each analytical batch.
- Any other qualifiers deemed necessary by the analyzing laboratory need to be explained in the report narrative for *each batch*.
- Use multiple data qualifiers, as applicable.
- **UJ** is used anytime an analyte is positively identified, but the concentration is below the actual lab-specific LLOQ.
- Using **U** alone indicates a nondetect (ND) without any QC excursions in the batch that would indicate a potential for low bias in the result.
- Using **J** alone would indicate that the analyte was positively identified at a concentration above the actual LLOQ, but there were QC excursions that would indicate that the result is an estimate with indeterminate bias.

Attachment 5. Data Completeness Checklist for Participating Laboratories

External Laboratory Study
Data Completeness Checklist
for Participating Laboratories
Version 08/28/18

Laboratory Name: _____

Analysis Batch: _____

Analysis Date(s): _____

Analyst Name: _____

Reviewer Name (print): _____

QA Officer (if applicable): _____

Directions: This checklist is intended to be used in conjunction with the draft method for this study, PFAS Deliverables, and Study Instructions. To complete the checklist, write **Yes**, **NA** for Not Applicable, or **No** for each line item. Provide additional explanation as necessary in the case narrative.

#	Question	Analyst	Reviewer
1	**Narrative, Summaries, COC**		
1a	Does the narrative contain all of the requested information per Section 3.1 of PFAS Deliverables document (e.g., lab info, contact, problems encountered, corrective actions, qualifiers, calculation examples, instrument conditions)?		
1b	Tabulated analytical results supplied for targets and surrogates in all samples in final concentration units (ng/L) in water, with X2 dilution factor introduced during preparation. NOTE: This info can be provided in the Excel template or as instrument output (as long as format is similar to Excel example)	1st two tabs in exact format	Others similar format
1c	Summary results supplied for all QC, including reagent blanks, method blanks, LLOQ verifications, LCSs, CCVs, surrogate recoveries, etc.? NOTE: Tabulated results preferred.		
1d	Chain of Custody (COC) Records completed according to Section 3.4 of PFAS Deliverables? Were the samples received in good condition, with ~5 g in each container?		
2	**Instrument and/or LIMS Outputs**		
2a	Are EPA supplied sample identifiers (IDs) used, or is a table provided that correlates EPA supplied IDs to laboratory assigned IDs? Are samples and associated QC clearly documented (i.e., which QC goes with which samples in which batch)? Are samples and associated calibrations also clearly documented?		
2b	Are date and times of analysis supplied either in spreadsheet or on detailed reports? Was the analytical sequence supplied?		
2c	Were the samples analyzed in the order specified in the study instructions? According to study-specific run order provided with the CoC		
2	**Instrument and/or LIMS Outputs**		

2d	Is a detailed report* included for all the following analyses? Reagent Blanks (RB) Method Blanks (MB) LLOQ verifications (LLOQ) Laboratory Control Sample (LCS) Laboratory Control Sample Duplicates (LCSD) Initial Calibrations (ICAL), all levels Continuing Calibration Verifications (CCV) * Including: quant and qual transitions for all targets and surrogates; associated responses; peak display/EICP with integration line; quant/qual response ratios; RT; scan number; and spectra		
2e	Is ICAL information supplied for every curve used in study? Must include ICAL calibration factor report with Relative Standard Deviations (RSDs) for targets and surrogates; nominal (prepared) concentration of target analyte and surrogate at each calibration level, in ng/L; curve graphics (plot of response vs. concentration showing calibration points and curve line); regression equations; weighting and forced origin options (if applicable); r^2 of fit used; refit results (ICAL standards re-calculated using final calibration model) with % error for each; relative standard error (if used)		
2f	Is CCV information supplied for every run? Must include expected concentration, and CCV evaluation (calculated concentrations using final calibration model; % recovery or deviation; responses for all targets and surrogates).		
2g	Are the specifics of the instrument, column, and run conditions documented in the data package?		
2h	Are correct peaks integrated? Are all peaks properly integrated? Are linear and branched isomers integrated/summed together (e.g., PFHxS, PFOS)? Are manual integrations marked on instrument reports? Was a clear picture of manual integration line supplied? Initialed and dated?		
2i	Are standards preparation logs included in the data package?		
2j	Are all concentrations, spike volumes, pH, volume adjustments, weights, and all preparation information correct and properly documented?		
3	**Procedural**		
3a	Was the sample preparation procedure followed according to Method for all samples, method blanks, LLOQ verifications, and LCSs (spiked with surrogates; spiked with target analytes (if applicable); diluted 1:1 with MeOH; filtered; and acidified)? Method Blanks: Sec. 11.1.1 LCS: Sec. 11.1.2 LCSD LLOQ verification: Sec. 11.1.3 Samples: Sec. 11.1.4		
3b	Is one Reagent Blank (RB) documented for every day of analysis, and do RBs meet criteria? See Section 9.5.8.		
3c	Are two Method Blanks (MBs) documented for each batch, and do MBs meet criteria? See Section 9.5.		
3	**Procedural**		

3d	Is LCS information supplied for every batch, and do LCSs meet Method criteria? Are LCS recoveries acceptable? If not, are data qualified properly and documented? See Section 9.6.2.		
3e	Is LLOQ verification information supplied for every batch, and do LLOQ verifications meet Method criteria? If LLOQ verification criteria are not met, was the LLOQ verified at a higher concentration? If so, is this information included in the narrative? See Section 9.9.		
3f	Are surrogates added to every field sample and associated QC sample (reagent blank, method blanks, LLOQ verification, CCV, LCS/LCSD)? Are surrogate recoveries acceptable (≤±30%)? If not, are data qualified properly and documented in the narrative?		
3g	Does the ICAL meet the method criteria (r^2 ≥0.99, Refit error ≤±30% for all standards, S/N ≥3)? If not, are outliers explained in the narrative and data properly qualified? See Sections 7.4.4 and 11.3		
3h	Was the CCV analyzed at the minimum frequency specified in the method (beginning [when used in lieu of ICAL], bracket every 10 samples, and end)? Are calculated CCV concentrations within ±30% of their expected values? If not, are outliers explained in the narrative and data properly qualified? See Section 11.4 and 11.6		
4	**Verifications**		
4a	Are all spiking solutions and calibration standards identified (including prepared concentration, amounts of any stocks added and total volume)?		
4b	Are reported concentrations in agreement with the raw data?		
4c	Do quantifier/qualifier ion ratios support all reported data?		
4d	Are reported results for detects and non-detects accurate?		
4e	Are all reported concentrations and percent recovery data in agreement with the quantitation reports?		
4f	Was at least one concentration verified by recalculating the reported concentration using responses, sample amounts, spike amounts, initial and final volumes, and any other needed dilutions?		
4g	Are the following data qualifiers used, when needed: H, I, J, K, L, U, and UJ?		
4h	Are raw data, acquisition and data processing methods, and tune files properly archived for long-term storage and retrievability if needed?		
4i	Were the data package and electronic deliverables to EPA verified for technical validity and completeness?		

Analyst Signature: _____
Date: _____

All concerns with this data package have been resolved to the satisfaction of the reviewer. The reviewer, by signing, verifies that that this package is complete and supports the reported results.

Reviewer Signature: _____
Date: _____

Attachment 6. Instructions for using the PFAS Data Reporting Template
External Validation Study

- **During analysis, *assign batch numbers.***
- **Follow the assigned run order provided with the validation study samples.**
- **Labs may use the Data Reporting Template in Attachment 6, or they may use instrument output or a LIMS if they can submit the first two worksheets to EPA with the data in an identical format.**
- **Name data reporting files in this manner: labname_batch ID_1 (2, 3, etc.).**
- Use one batch per spreadsheet file (1 calibration curve, 20 field samples, plus QC) per spreadsheet.
    - *If a mid-level CCV is analyzed in lieu of a full calibration curve (see clarification of the method in Table 3 of the study instructions), it will be obvious on the Batch Run List sheet, and the sheet that is titled "Optional CCVs in lieu of cal" should be used instead of the one titled "Calibration". Be sure to notate the batch number associated with the previous full calibration curve. (Note that a CCV cannot be used in lieu of ICAL if the ending CCV for the previous batch failed and one subsequent reanalysis of the CCV also fails (% recovery > ±30%).*

Sheets included in the Excel workbook:

1. Batch Run List – List all of the samples analyzed in the single batch in order.
(MUST use this worksheet or identically formatted electronic data deliverable (EDD), output from an instrument or a LIMS.)

2. Results – Input the sample results, and add any data qualifiers in the Qual column.
(MUST use this worksheet or identically formatted electronic data deliverable (EDD), output from an instrument or a LIMS.)

3. Calibration – Input information associated with the batch calibration curve and QC.

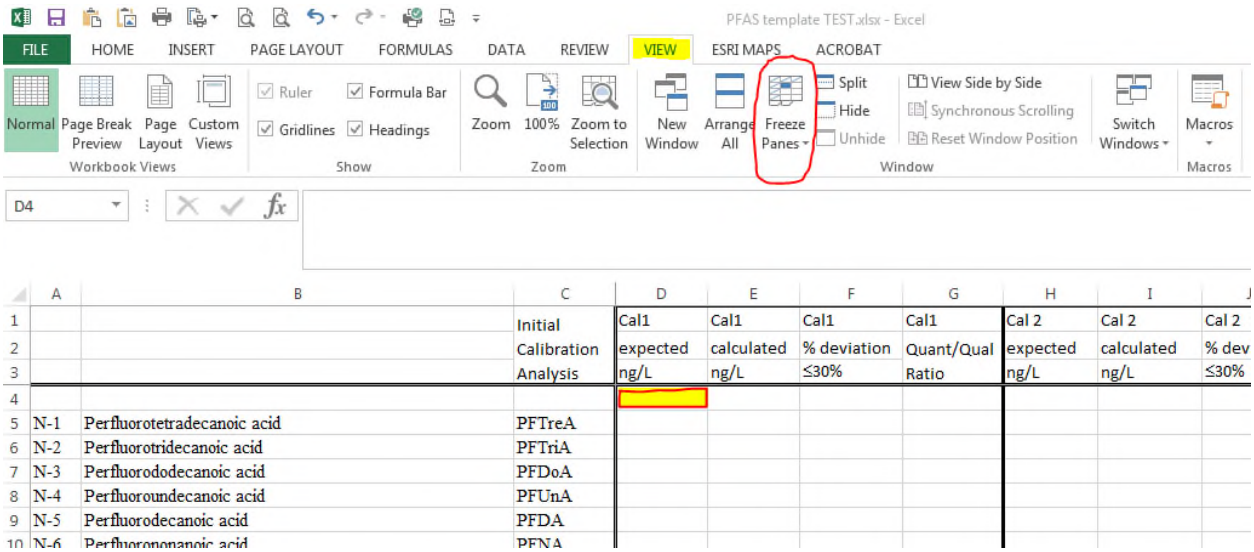
4. CCV – Two CCVs are required per 24-hour sample sequence, but insert additional columns if another was analyzed for any reason.

5. LLOQ verification – Two LLOQ verification check sample (at the QL, which should be at the concentration of the lowest calibration standard to be used) is required per batch.
-
6. Blanks – This sheet includes Reagent Blanks (RBs, aka instrument blanks) and Method Blanks (MBs). Two MBs and one RB are required for each batch, but insert additional columns if others are analyzed.

7. LCS_LCSD - Input information associated with the LCS and the LCSD.

If you lose pane freezing in the screen views:

- Click in the first space that you want to scroll (crosshairs of the rows/columns you want to freeze)
- Go to the "View" menu
- Click on "Freeze Panes" (shown below)



	A	B	C	D	E	F	G	H	I	J
1			Initial	Cal1	Cal1	Cal1	Cal1	Cal 2	Cal 2	Cal 2
2			Calibration	expected	calculated	% deviation	Quant/Qual	expected	calculated	% dev
3			Analysis	ng/L	ng/L	≤30%	Ratio	ng/L	ng/L	≤30%
4										
5	N-1	Perfluorotetradecanoic acid	PFTreA							
6	N-2	Perfluorotridecanoic acid	PFTriA							
7	N-3	Perfluorododecanoic acid	PFDoA							
8	N-4	Perfluoroundecanoic acid	PFUnA							
9	N-5	Perfluorodecanoic acid	PFDA							
10	N-6	Perfluorononanoic acid	PFNA							

To print or preview an entire workbook:

- Select the 1st worksheet tab, "Batch Run List".
- Right click and select "Select All Sheets".
- Select printing or previewing options as you normally would.

Abbreviations/ Acronyms

Cal – Calibration Standard

CALx – Calibration Standards x = 1-9

CCV – Continuing Calibration Verification

LCS/LCSD – Laboratory Control Sample/Laboratory Control Sample Duplicate

LLOQ – Lower Limit of Quantification

MB – Method Blank

mid-cal RT – Retention Time of mid-level calibration standard

RB – Reagent Blank

Quant/Qual Ratio – Ratio of Quantification Ion to Qualification Ion

QC – Quality Control Samples (e.g., CCV, LLOQ, MB, etc.)

RT – Retention Time

SOP – Standard Operating Procedure

Definition of Terms for the External Validation Study

- Instrument ID – Manufacturer and Model/Type of Instrumentation Used

- Laboratory Control Sample & Duplicate – ASTM type 1 water or equivalent PFAS-free reagent water spiked with surrogates and targets, processed through the procedure as a sample

- Method Blank – a sample consisting of ASTM type 1 water equivalent PFAS-free reagent water and surrogates, processed through the procedure as a sample

- Reagent Blank – a sample consisting of the reagents used to process the samples, used to check for contamination

- Time of Injection – the time of day that the CCV was injected to ensure it was < 24 hrs after the calibration curve was analyzed