

## **REQUIREMENTS FOR SERDP & ESTCP PROJECTS ADDRESSING PFAS-RELATED ISSUES**

This document defines the requirements for SERDP or ESTCP projects addressing PFAS-related issues. It also contains recommendations for the types of information needed at various stages of the project timeline so that the Technical Committee can accurately evaluate proposal and project quality, and determine the level of Quality Assurance/Quality Control (QA/QC) required. ***All SERDP and ESTCP projects addressing PFASs will be directed as to which of the documents below must be submitted.***

### **1. ANALYTE LIST**

Provide a list of the analytes to be studied during the project. The project analyte list is dependent on the goals of the proposal; however, the analytes (Table 1) currently included in the EPA's method development initiatives should be considered a baseline, unless a technical justification can be provided. Provide the technical justification as an attachment to the Analyte List for any deviations from the analytes listed in Table 1.

### **2. ANALYTICAL CONFIRMATION WHITE PAPER**

All investigators conducting in-house PFAS analyses will be required to submit an Analytical Confirmation White Paper within four months of contract award. The white paper should summarize the means utilized for analytical confirmation. Acceptable methods for analytical confirmation are summarized below.

- Results of PFAS analyses must be compared to a DoD Environmental Laboratory Accreditation Program (ELAP)-accredited laboratory within three months of contract award. The DoD ELAP-accredited laboratory must be accredited for preparation and analysis of the project analytes by *PFAS by LCMSMS Compliant with DoD QSM 5.3 (or latest version) Table B-15* method for all non-drinking water media and EPA Method 527 for drinking water. Both the accredited and non-accredited laboratory must run at least  $n = 3$  replicates to determine if the average results are statistically different. Results should be within 30% of the ELAP-accredited laboratory, with any major discrepancies evaluated in the Analytical Confirmation White Paper.
- Investigators may also use a Standard Reference Material (SRM) (once available) to demonstrate the validity of their analysis method. All reported results should be within the published tolerance of the certified value. If values fall outside the published tolerance an explanation must be provided in the Analytical Confirmation White Paper.
- Analytical confirmation should be repeated periodically to ensure continued compliance. Annual confirmation is recommended, but investigators may propose a different schedule dependent on analytical frequency. The schedule for analytical confirmation should be provided in the Analytical Confirmation White Paper, with brief white papers submitted for each confirmation that occurs during the project.
- For ESTCP projects demonstrating PFAS removal in the field, a minimum of 20% of initial and final samples for PFAS analyses must be sent to a DoD-ELAP accredited laboratory for confirmatory analyses. Any major discrepancies should be presented and discussed in the Final Report. The Analytical Confirmation White Paper should define the schedule and plan for such sampling.

**Table 1. EPA Method Development Analyte List**

<b>Analyte Name</b>	<b>Acronym</b>	<b>CAS Number</b>
Perfluorotetradecanoic acid	PFTreA	376-06-7
Perfluorotridecanoic acid	PFTriA	72629-94-8
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluoroundecanoic acid	PFUnA	2058-94-8
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorononanoic acid	PFNA	375-95-1
Perfluorooctanoic acid	PFOA	335-67-1
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorobutanoic acid	PFBA	375-22-4
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorooctanesulfonamide	PFOSA	754-91-6
Fluorotelomer sulfonic acid 8:2	FtS 8:2	39108-34-4
Fluorotelomer sulfonic acid 6:2	FtS 6:2	27619-97-2
Fluorotelomer sulfonic acid 4:2	FtS 4:2	757124-72-4
2-(N-Ethylperfluorooctanesulfonamido)acetic acid	NEtFOSAA	2991-50-6
2-(N-Methylperfluorooctanesulfonamido)acetic acid	NMeFOSAA	2355-31-9

### **3. PERFORMANCE OBJECTIVES WHITE PAPER**

All ESTCP projects and some SERDP projects must provide a white paper as an initial deliverable that delineates the performance objectives and provides the performance objectives table as shown in the ESTCP Demonstration Plan guidance and in Table 2 in this document. The Program Office will assign this white paper to you if applicable. Please see the ESTCP Demonstration Plan guidance for more information as to how to define the performance objectives.

Performance objectives may be related to qualitative or quantitative parameters (i.e., reduction in mass flux, reduction in point source contaminant concentrations, etc.). These should include, but are not limited to, such things as end-point criteria, remediation time, and analytical sensitivity. Details on the methods for collecting and analyzing the data required to assess the quantitative performance objectives will be required in the Demonstration Plan and in the Standard Operating Procedures (SOPs). The following information should be included in the detailed description of each performance objective:

- A full explanation of the objective
- A statement as to what data are required to evaluate the performance objectives
- A statement as to how the data will be interpreted and a measure of what signifies success.

### **4. STANDARD OPERATING PROCEDURES**

All projects must submit the SOPs for sample collection, preparation, and analysis. Table 3 provides a list of the required elements for each of these SOPs. If the project analytes are included in Table 1, the project media are aqueous, AFFF, tissue, and/or solid; and the data are to be used in quantitative determinations, then the project must ensure that either:

- The laboratory performing the preparation and analysis is accredited to the latest version of the DoD QSM (currently 5.3) and their scope of accreditation includes a method designated as compliant the current version of the DoD QSM's PFAS sample preparation and analysis requirements (currently DoD QSM 5.3 Table B-15) for the project-specified PFAS/media combinations included in the project that are not drinking water. For drinking water samples, the laboratory performing the preparation analysis must be DoD ELAP and/or EPA accredited for the current published EPA drinking water method for the specific PFAS to be analyzed. Currently, there are two EPA drinking water methods (537.1 and 533), each with different analyte lists; or
- Where appropriate, the project's proposed SOPs meet the PFAS sampling and analysis requirements (currently Table B-15) of the latest version (currently 5.3) of the DoD QSM for all other applicable media (aqueous, solid, tissue, and AFFF) and the requirement of the published EPA method (currently EPA Method 537.1 or 533, depending on the analyte) for the preparation and analysis of drinking water samples. The SOP must clearly articulate how the samples are to be prepared and analyzed. The SOP must articulate how each criteria listed in Table B-15 will either be met, or, if not appropriate due to the use of a less standard technique (such as LC-HRMS), the reason why that criterion is considered not appropriate must be explained. For these cases, additional information should be provided to demonstrate the project team's approach for meeting the intent of each criterion specified in Table B-15.

If the medium (e.g., blood serum) is not included in scope of Table B-15, the data are to be used for screening purposes, and/or the data are to be considered qualitative, the requirements in Table 4 must be met.

**Table 2. Performance Objectives**  
**[SAMPLE ONLY–Performance objectives must be specific to the technology being demonstrated.]**

<b>Performance Objective</b>	<b>Data Requirements</b>	<b>Success Criteria</b>
<b>Quantitative Performance Objectives</b>		
Determine remediation effectiveness	Pre- and post-treatment contaminant concentrations in soil and groundwater	<ul style="list-style-type: none"> <li>• &gt;90% reduction considered successful</li> <li>• Student t-test or ANOVA for statistical analysis</li> </ul>
Analytical field sensitivity	Matrix-specific field samples	Concentrations between 2x-5x reporting limit are detected
<b>Qualitative Performance Objectives</b>		
Ease of use	Feedback from field technician on usability of technology and time required	A single field technician able to effectively take measurements

S A M P L E

**3.1 PERFORMANCE OBJECTIVE: DETERMINE REMEDIATION EFFECTIVENESS**

The effectiveness of the technology for soil and groundwater remediation is a function of the degree to which the target contaminants are removed. The success in remediating the test area depends on the residual contamination after application of the process.

**3.1.1 Data Requirements**

The technology remedial effectiveness will be evaluated on the basis of contaminant concentration reductions in soil and groundwater within the zone of treatment. Data required for the remedial effectiveness assessment include pre- and post-treatment contaminant concentrations in the treated media. Soil and groundwater samples for contaminant concentration characterization will be collected and analyzed both before and after the process implementation.

Post-treatment soil and groundwater sampling results will indicate the residual contamination remaining within the treatment zone. These concentrations will be compared with initial concentrations to determine if significant removal has occurred.

**3.1.2 Success Criteria**

The objective will be considered to be met if >90% reduction in contaminant mass is achieved. A standard student t-test will be used to evaluate the statistical significance of the data. Other statistical tests such as ANOVA or other nonparametric tests may be applied as appropriate to test the significance of the data.

**Table 3. SOP Required Elements**

<b>Required SOPs</b>	<b>Required elements</b>
Sample Collection	Media and analytes
	Equipment/supplies
	Collection process
	Decontamination procedures
	Bottle type
	Field QC types, frequency and criteria
	Sample preservation, shipping and hold time requirements
Sample Preparation	Media and analytes
	Equipment/supplies
	Homogenization and subsampling processes
	Preparation technique (i.e., dilution, SPE)
	Clean-up procedures
	Sample preparation QC types, frequency and criteria
	Sample extract storage and hold time requirements
Sample Analysis	Media and analytes
	Equipment/supplies
	Type of standards utilized (e.g., analytical/technical, reference, isomeric blends (linear/branched))
	Calibration procedure (e.g., single point or curve, external, internal, isotope dilution)
	Instrument cleanliness requirements (instrument blank criteria)
	Calibration verification procedure (e.g., type, frequency and criteria)
	Known interferences
	Verification of numeric detection/quantitation value if numeric values provided for sample results

**Table 4. Qualitative Analysis Requirements**

<b>QC Element</b>	<b>Criteria</b>
Sampling Precision (e.g., Field Sample Duplicate)	%RPD or correlation of detection (both positive) or within same range (both > a specified limit)
Sampling Bias (e.g., Certificates of Cleanliness of Sample Containers, Field Blank, Decon Blanks)	Not detected or < a specified limit
Sample Preparation Precision (e.g., Sample Duplicate, Laboratory Control Sample Duplicate (LCSDup), Matrix Spike Duplicate (MSD))	%RPD or correlation of detection (both positive) or within same range (both > a specified limit)
Sample Preparation Bias (e.g., Method Blank(MB), Laboratory Control Sample (LCS), Matrix Spike (MS), isotope dilution standards, internal standards)	% Recovery range or correlation of detection (both positive) or within same range (both > a specified limit)
Detection and/or Quantitation Limit	Define elements which are required to be met to indicate presence of target analyte (e.g., S/N ratio, confirmation ion transition, ion transition ratio, confirmation ion, peak area threshold, intensity threshold). If numeric value is associated with detection and/or quantitation, derivation and validation of limits reported to for samples.
Calculations	If numeric values are provided for sample results, derivation of those values must be provided

## **5.0 QA/QC DOCUMENTATION**

Final reports and presentations must include supplemental information that evaluate the data that is presented relative to the QC requirements that were included in the proposal and/or SOPs. Raw data should be provided as needed sufficient to recreate the results of the research and/or demonstration. Examples of all calculations used to derive the results are required, including information on analytical standards (type and traceability).

## **6.0 INFORMATION FOR PROPOSAL EVALUATION**

Investigators should familiarize themselves with the requirements specified in this document for a SERDP or ESTCP project in order to properly cost the proposal. The following information should be included at the pre-proposal or full proposal stage to allow for technical evaluation and appropriate cost estimates.

### **Pre-Proposals**

Preproposals must provide the following information:

- Types of media (e.g., drinking water, surface water, soil, sediment, biota) to be collected
- PFAS analyte list for each media type to be collected

### **Full proposals**

Full proposals must provide the information listed below and a draft of the performance objectives table shown in Section 2 of this document.

- Identification of types of media (e.g., drinking water, surface water, soil, sediment, biota) to be collected
- Identification of PFAS analyte list for each media type to be collected
- Identification of sample collection, sample preparation and analytical procedures to be utilized for each media type
- Identification of class of result (quantitative or qualitative) to be reported for each media/analyte combination
- Identification of intended use of data sets (e.g., definitive, screening)