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Statistical Analyses for Single Laboratory Validation Study of Novel PFAS Measurement Method (Presentation)

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About This Publication

This work was conducted by the Institute for Defense Analyses under contract HQ0034-19-D-0001, Project AM-2-1528, "Validation of PFAS Detection Methods," for the Executive Director, Strategic Environmental Research and Development Program (SERPD) and Environmental Security Technology Certification Program (ESTCP), under the Office of the Deputy Under Secretary of Defense (Installations and Environment). The views, opinions, and findings should not be construed as representing the official position of either the Department of Defense or the sponsoring organization.

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Executive Summary

The Strategic Environmental Research and Development Program (SERDP) and the Environmental Security Technology Certification Program (ESTCP) are funding the development of novel methods for measuring trace contamination of per- and polyFluoroalkyl Substances (PFAS), a broad category of manmade chemicals that are environmentally persistent and associated with health problems in humans. PFAS are resistant to heat, oil and water and because of this property have been used widely throughout the country, in particular, by the Department of Defense (DoD) as fire extinguishing agents. In Fiscal Year 2021, IDA, NAVSEA, and AFCEC performed statistical analyses to assist SERDP/ESTCP in the validation of these novel PFAS mass spectrometry detection methods, as part of the DoD's Single Laboratory Validation study. We processed the data reported by the laboratory to calculate the average native concentrations, spike concentrations, and percent recoveries of dozens of PFAS analytes in 8 types of environmental samples. An IDA-coded Python computer script produced over 100 tables that illustrate the performance of the PFAS analytical method across various aqueous, solids, and tissues samples.

Statistical Analyses for Single Laboratory Validation Study of Novel PFAS Measurement Method (374)

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Janice Willey, Naval Sea Systems Command (NAVSEA),
Hunter Anderson, Air Force Civil Engineer Center (AFCEC),
Tim Thompson, Strategic Environmental Research and Development Program (SERDP) & Environmental Security Technology Certification Program (ESTCP)

Objectives

Bottom Line Up Front

- SERDP/ESTCP sponsored the validation of a novel PFAS measurement method for 8 environmental sample types: groundwater, landfill leachate, surface water, wastewater; sediment, biosolids, soil; and tissue.
- DoD tasked an independent organization (IDA) to analyze data from the single laboratory validation (SLV) study, producing summary statistics of the novel PFAS method.

IDA calculated summary statistics and automatically generated over 100 data tables in a systematic and reproducible manner using a coded Python computer script to eliminate human error. The data can now be used to support the government's SLV of the novel PFAS measurement method.

Summary of Data from *Sample Native Concentration Tables* Generated for the SLV Study of 40 PFAS Analytes in 8 Types of Environmental Samples

Technical Approach

Data Collected with PFAS Method

- 40 PFAS analytes and 24 PFAS isotope standards from 8 types of environmental samples: groundwater, landfill leachate, surface water, wastewater; sediment, biosolids, soil; and tissue, were measured.
- Dataset from single laboratory validation (SLV) study provided to IDA consisted of 30,000+ subsample measurements from 32 unique environmental samples.

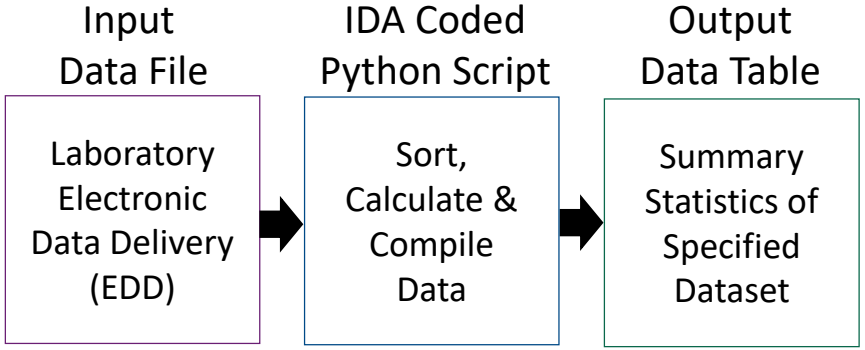
Summary Data Tables for SLV study

- IDA developed a code to calculate summary statistics and compiled data into several categories of data tables.
- IDA verified the calculation in every column in each of the tables generated by the coded script by comparing values to manually calculated values in Excel.

Schedule:

- First set of tables delivered (May 2021)
- All 100+ tables delivered (Aug 2021)
- Final report completed (Sept 2021)

Task Structure



Summary Statistic Calculations

Percent Recovery	$\left(\frac{\text{Measured spike [ ]} - \text{Meausured native [ ]}}{\text{Known spike [ ] added}}\right) \cdot 100$
Mean	$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$
Standard Deviation	$\frac{\sqrt{\sum(x_i - \mu)^2}}{n-1}$
Percent Relative Standard Deviation	$\left(\frac{SD}{\text{Mean}}\right) \cdot 100$

[ ] = concentration, n = number of quantities, x_i = ith quantity, μ = sample mean

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Results to Date

- **Data Tables & Categories Generated**
  - Sample Native Concentration (35 tables)
  - Sample Matrix Recovery (32 tables)
  - Media Type Matrix Recovery (11 tables)
  - Extracted Internal Standard (EIS) Spike Recovery (11 tables)
  - Injected Internal Standard (IIS) Spike Recovery (11 tables)
  - Ongoing Precision & Recovery (OPR) Spike Recovery (4 tables)
  - Limit of Quantification Verification (LOQVER) Spike Recovery (4 tables)
  - Method Blank (MB) Spike Recovery (4 tables)
- Each table used a prescribed template to format the output CSV data file

Example Sample Matrix Recovery Table

Analyte
PFBA
PFPeA
PFHxA
PFHpA
PFOA
⋮

Low Spike Recovery

Number Detects	Native Mean PFAS Concentration (µg/kg)	Mean PFAS Spike Concentration (µg/kg)	% Recovery	Native> Spike (Yes/No)
0	NA	2.62	104.07	No
1	0.027	1.31	112.7	No
2	0.0235	0.656	95.71	No
0	NA	0.656	105.34	No
2	0.0565	0.656	99.67	No
⋮	⋮	⋮	⋮	⋮

Medium Spike Recovery

Number Detects	Native Mean PFAS Concentration (µg/kg)	Mean PFAS Spike Concentration (µg/kg)	% Recovery	Native> Spike (Yes/No)
0	NA	3.94	108.55	No
1	0.027	1.97	111	No
2	0.0235	0.984	104.02	No
0	NA	0.984	104.38	No
2	0.0565	0.984	101.68	No
⋮	⋮	⋮	⋮	⋮

High Spike Recovery

Number Detects	Native Mean PFAS Concentration (µg/kg)	Mean PFAS Spike Concentration (µg/kg)	% Recovery	Native> Spike (Yes/No)
0	NA	6.57	104.41	No
1	0.027	3.28	109.63	No
2	0.0235	1.64	102.23	No
0	NA	1.64	105.49	No
2	0.0565	1.64	97.98	No
⋮	⋮	⋮	⋮	⋮

ALL SPIKE RECOVERIES

n =	% Mean Recovery	RSD
3	105.68	2.36
3	111.11	1.38
3	100.65	4.35
3	105.07	0.57
3	99.78	1.86
⋮	⋮	⋮

First 5 Rows from 1 of the 32 *Sample Matrix Recovery Tables* Produced for Every Environmental Sample for All 40 PFAS Analytes

Lessons Learned and Next Steps

- For 92% of the analyte-environmental sample combinations in the *Sample Matrix Recovery Data Tables*, the average percent recovery surpassed 75%.
- The multi-laboratory validation (MLV) study of the PFAS method should continue refining evaluation metrics for the analyte-sample combinations.

Resources:

IDA Document D-22794, *Data Compilation in Support of Single Laboratory Validation of a Novel Per- and Polyfluoroalkyl Substances (PFAS)Detection Method for Environmental Matrices,*

EPA Draft Method 1633, *Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS*

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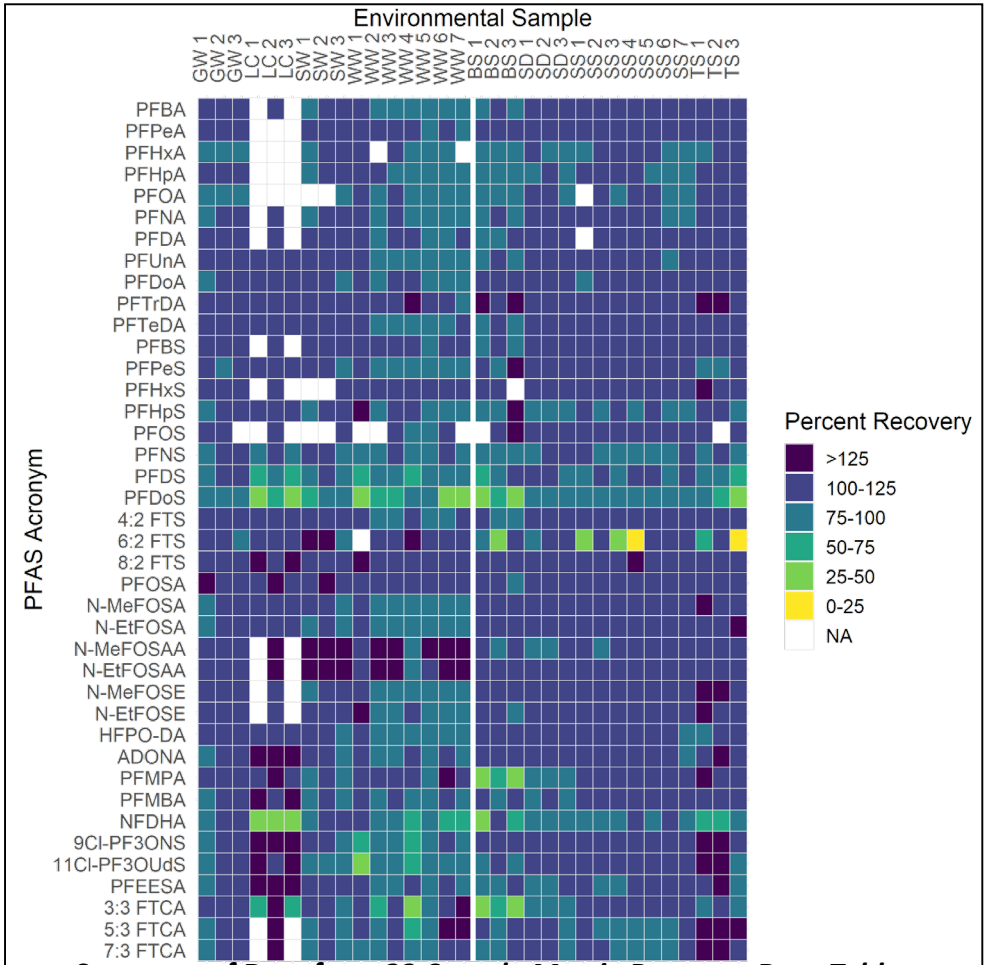

Summary of Data from 32 *Sample Matrix Recovery Data Tables* Generated for the SLV Study of 40 PFAS Analytes in 8 Types of Environmental Samples

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14. ABSTRACT

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