Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

Proficiency Test Report
AQA 19-19
PFAS TOP Assay in Water

August 2020

ACKNOWLEDGMENTS

This study was conducted by the National Measurement Institute (NMI). Support funding was provided by the Australian Government Department of Industry, Science, Energy and Resources.

I would like to thank the management and staff of the participating laboratories for supporting the study. It is only through widespread participation that we can provide an effective service to laboratories.

The assistance of the following NMI staff members in the planning, conduct and reporting of the study is acknowledged.

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Accredited for compliance with ISO/IEC 17043

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SUMMARY

This report presents the results of the proficiency test AQA 19-19 PFAS TOP Assay in Water. The study focused on the measurement of per-and poly-fluoroalkyl substances (PFAS) in water before and after total oxidisable precursor (TOP) assay treatment.

Twenty-two laboratories participated and eighteen submitted results.

Six test samples were prepared at the NMI North Ryde laboratory and consisted of:

- two identical MilliQ water samples labelled S1 (pre-oxidation) and S2 (post-oxidation) spiked with 6:2 diPAP, 8:2 diPAP, PFDA and PFDS;
- two identical MilliQ water samples labelled S3 (pre-oxidation) and S4 (post-oxidation) spiked with Alcoseal foam (30,000 x dilution), PFDA and PFDS;
- two identical river water samples labelled S5 (pre-oxidation) and S6 (post-oxidation) spiked with Alcoseal foam (30,000 x dilution), a commercially available fluorine free foam and PFDA and PFDS.

The assigned values were the robust average of participants' results. The associated uncertainties were estimated from the robust standard deviation of the participants' results.

The assessment of the oxidative methods used by participants is based on the criteria stipulated in PFAS National Environmental Management Plan, Version 2.0 (NEMP), published in January 2020 for aqueous samples.[1]

The outcomes of the study were assessed against the aims as follows:

- *compare the performances of participant laboratories and to assess their accuracy in the measurement of PFAS analytes in water before and after oxidation pre-treatment;*

Laboratory performance was assessed using both z-scores and E_n-scores.

Of 369 z-scores, 337 (91%) were satisfactory with $|z| \leq 2.0$.

Of 369 E_n-scores, 270 (73%) were satisfactory with $|En| \leq 1.0$.

Laboratory 13 returned both satisfactory z and E_n-scores for all reported results.

Laboratories 1, 9, 13, 15, 16 and 18 returned satisfactory z-scores for all reported results.

- *to evaluate the laboratories' methods for TOP assay oxidative pre-treatment;*

All laboratories based their TOP assay procedure on the Houtz and Sedlak method published in 2012.[2] Most laboratories: used extra doses of oxidant (2 to 500 times the amount of oxidant used by Houtz and Sedlak per mL of sample), had an extended oxidation time of up to 18 hours, diluted the samples before oxidation and/or increased the number of oxidation of cycles to up to 3.

Although the majority of participants complied with NEMP QA requirements, the consistent low results reported for PFDA in the post-oxidation samples indicates that the methods employed biased carboxylic acid concentration low, for which there is no numerical quality assurance criteria in NEMP.

- *to develop the practical application of traceability and measurement uncertainty and provide participants with information that will be useful in assessing their uncertainty estimates.*

Of 896 results, 873 were reported with an associated estimate of expanded measurement uncertainty. The magnitude of the reported expanded uncertainties was within the range 1.3% to 500% of the reported value

1 INTRODUCTION

1.1 NMI Proficiency Testing Program

The National Measurement Institute (NMI) is responsible for Australia's national measurement infrastructure, providing a range of services including a chemical proficiency testing program.

Proficiency testing (PT) is: 'evaluation of participant performance against pre-established criteria by means of interlaboratory comparison.'[3] NMI PT studies target chemical testing in areas of high public significance such as trade, environment, law enforcement and food safety. NMI offers studies in:

- pesticide residues in fruit and vegetables, soil and water;
- per- and polyfluorinated alkyl substances (PFAS);
- petroleum hydrocarbons in soil and water;
- inorganic analytes in soil, water, food and pharmaceuticals;
- controlled drug assay.

1.2 Study Background

The per- and poly-fluoroalkyl substances (PFAS) total oxidisable precursor (TOP) assay is an oxidative sample pre-treatment method aimed at converting perfluoroalkyl acids (PFAA) precursors within a sample into measurable perfluorocarboxylic acids (PFCA) products. The method is usually used where the PFAS product composition extends beyond the US EPA suite or where the precursors are unknown and therefore is providing a better understanding of the overall PFAS contamination present within the sample.

1.3 Study Aims

The aims of the study were to:

- compare and assess laboratories' accuracy in the measurement of PFAS before and after the oxidative pre-treatment;
- evaluate the laboratories' methods for TOP assay oxidative pre-treatment; and
- develop the practical application of traceability and measurement uncertainty and provide participants with information that will be useful in assessing their uncertainty estimates.

1.4 Study Conduct

The conduct of NMI proficiency tests is described in the NMI Chemical Proficiency Testing Study Protocol.[4] The statistical methods used are described in the NMI Chemical Proficiency Statistical Manual.[5] These documents have been prepared with reference to ISO 17043[3] and The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.[6] The acceptability for oxidation step has been assessed using criteria from 2020 PFAS National Environmental Management Plan.[1]

The study falls within the scope of NMI's accreditation as a proficiency testing provider.

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of the study was:

Invitation issued	20 November 2019
Samples dispatched	29 February 2020
Results due	13 March 2020

Interim report issued 18 March 2020

2.2 Participation

Twenty-two laboratories participated and eighteen submitted results.

2.3 Test Material Specification

The study design was aimed to allow participants to evaluate their oxidative methods for foam products in clean (interference free) matrices and in more complex matrices with high total organic carbon content.

Six test samples were prepared:

- Two identical samples labelled S1 (pre-oxidation) and S2 (post-oxidation)
- Two identical samples labelled S3 (pre-oxidation) and S4 (post-oxidation); and
- Two identical samples labelled S5 (pre-oxidation) and S6 (post oxidation**).**

Participants were asked to perform:

- A pre-TOP assay analysis using their routine method for PFAS measurements on samples labelled: S1 (pre-oxidation), S3 (pre-oxidation) and S5 (pre-oxidation); and
- A post-TOP assay analysis using their routine method for PFAS measurements after using an oxidative pre-treatment on samples labelled: S2 (post-oxidation), S4 (post-oxidation) and S6 (post-oxidation).

Sample S1 and S2 were identical MilliQ water samples spiked with 6:2 diPAP (50µg/L), 8:2 diPAP (50 µg/L), PFDA (6.42 µg/L) and PFDS (3.14 µg/L).

Expected:

S1 (PRE TOP)	S2 (POST TOP)
6:2 diPAP*	PFBA to PFHpA
8:2 diPAP*	PFBA to PFNA
PFDS	PFDS
PFDA	PFDA

*Not analysed by participating laboratories

Samples S3 and S4 were identical MilliQ water samples spiked with the aqueous film forming foam (AFFF) Alcoseal (30000 x dilution), PFDA (9.08 µg/L) and PFDS (6.51 µg/L).

Expected:

S3 (PRE-TOP)	S4 (POST-TOP)
Alcoseal*	PFCAs
PFDA	PFDA
PFDS	PFDS

*Not analysed by participating laboratories

Sample S5 and S6 were identical autoclaved river water samples spiked with the same amount of Alcoseal (30000 x dilution), PFDA (9.08 µg/L) and PFDS (6.51 µg/L) as for S3 and S4. These samples were also fortified with a fluorine free AFFF for a total organic carbon content of 100 mg/L.

Expected:

S5 (PRE-TOP)	S6 (POST TOP)
Alcoseal*	PFCAs
PFDA	PFDA
PFDS	PFDS

*Not analysed by participating laboratories

2.4 Test Material Homogeneity and Stability Testing

The preparation of the study samples is described in Appendix 2. No homogeneity or stability testing was conducted on the study samples. They were prepared and packaged using a process that has been demonstrated to produce homogeneous and stable samples for previous NMI PFAS TOP Assay interlaboratory comparison study[7] and participants' results gave no reason to question the homogeneity and stability. However, there were stability issues in this study with regards to PFDS. Possible reasons for instability of this compound are presented in section 6.5. A reasonable consensus was found between participants' results and consequently an assigned value was set for this analyte.

2.5 Laboratory Code

All laboratories that agreed to participate were assigned a confidential code number.

2.6 Sample Storage, Dispatch and Receipt

Prior to dispatch, samples were refrigerated at 4ºC. Participants were sent two 60 mL water in HDPE bottles for each of the six samples. The samples were packed in a foam box with a cooler brick and sent by courier on 29 February 2020. The following items were packaged with the samples:

- a covering letter which included a description of the test samples and instructions for participants; and
- a form for participants to confirm the receipt and condition of the samples.

An Excel spreadsheet for the electronic reporting of results was e-mailed to participants.

2.7 Instructions to Participants

Participants were instructed as follows:

- To use their normal TOP assay method
- To report results in units of µg/L.
- For each analyte in each sample to report one result for pre-oxidation and one for post-oxidation.
- For analytes that contain linear and branched isomers to report the sum of linear and branched.
- Report results as you would to a client, applying the limit of reporting of the method used for analysis.
- To complete the method details as required in the Methodology sheet.
- To return the completed results sheet by e-mail (proficiency@measurement.gov.au).

2.8 Interim Report

An interim report was emailed to participants on 18 March 2020.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Methods Reported by Participants

Table 1 Oxidative Treatment – Samples S2, S4 and S6

Lab Code	Sample	Sample Amount (mL)	Dilution Factor	No. of Cycles	$K_2S_2O_8$ (g/cycle)	NaOH (mL/cycle)	Heating Time (hr)	Temperature (°C)	pH before Oxidation	pH after Oxidation	pH before Extraction	Additional Information
HOUTZ & SEDLAK		125	No	1	2 (60mM)	1.9 (150mM)	6	85		n/a	5-9	
1	S2	61	No	3	4	3.8	6	85	13	NA	6.01	S2: Total vol was 250mL. S4: 60 mL sample- rinsed bottle - oxidation not completed used diluted sample (10) S6: 0.03ug/L 6:2 FTS were detected (<LOR)
	S4	6	10									
	S6	62	No									
3	S2	61.4	4.08	1	8.192	7.5	16.73	86	5	13	2	
	S4	60.64	4.13									
	S6	62.04	4.04									
4	S2	25	No	1	0.811	25	8	85	5.9	12	2	
	S4								5.9			
	S6								7.9			
5	S2, S4, S6	50	No	3	5, 3, 3	5, 3, 3	2	85	14	12	6 to 7	All: Base was 10M NaOH
7	S2, S4, S6	5	No	1	0.24	0.228 (of 10N)	6	85-95	>11	Neutral	Neutral	All: Direct injection after oxidation
8	S2, S4, S6	5	No	1	0.5	0.475 (10M in water)	16.5	85			Approx. 7	
9	S2, S4, S6	20	No	2	1	1	3 (overnight)	85	14	14	4	All: Current method is to shake sample then take 20mL aliquot
10	S2	50	1 in 10	1	2	1.9	6	85	13	13	6	
	S4		1 in 100									
	S6		1 in 100									
11	S2, S4, S6	5	10	1	0.08	0.076	6	85				All: The samples were diluted ten fold in a 60mM potassium persulfate and 150 mM NaOH
12	S2	0.5	10	1	0.15	0.375	16	85				
	S4	0.25	20									

Lab Code	Sample	Sample Amount (mL)	Dilution Factor	No. of Cycles	$K_2S_2O_8$ (g/cycle)	NaOH (mL/cycle)	Heating Time (hr)	Temperature (°C)	pH before Oxidation	pH after Oxidation	pH before Extraction	Additional Information
HOUTZ & SEDLAK		125	No	1	2 (60mM)	1.9 (150mM)	6	85		n/a	5-9	
	S6	0.25	20									
13	S2, S4, S6	30	various	1	4	4 (of 4N)	>6	85	>12	>12	>6	
14	S2	5	10,50	1	0.4	0.38 (10M)	6	90	13	11	4 to 7	
	S4		10,50,100									
	S6		10,50,100									
15	S2	5	10	1	0.08	5	18	85	13	13	5 to 9	
	S4		20									
	S6		20									
16	S2	5	No	1	0.24	0.228	6	85		14		All: NaOH used is 10M solution
	S4				0.48	0.456						
	S6				0.48	0.456						
18	S2	10	No	1	Single Dose	Single Dose	6	85	14	14	4	All: NaOH used is 10M solution
	S4		10		Double Dose	Double Dose						
	S6		10		Double Dose	Double Dose						
19	S2, S4, S6	50	No	1	4.365	5	6	85	Over 12	Over 12	Between 6 & 8	
20	S2, S4, S6	20	No	1	0.81	0.32 g	6	80	7	NR	7	
21	S2, S4, S6	5	20X	1	2	1.9	6	85	Not determined	>7	7	

Table 2 Test Methods Pre and Post Oxidation

Lab Code	Pre/ Post	Sample	Sample Size (mL)	Bottle Rinsed	Sample Pretreatment	Extraction Technique	Extraction Solvent(s)	Extraction Process	Extraction Temperature	Extraction Clean Up	Instrument	Column Type	Column Specifications	Delay Column	Blank Correction	Standard Method
1	Pre	S1	61	Yes	pH adjustment	SPE (Strata X-AW 33um polymeric Weak Anion)					Orbitrap	Kinetex C18	100x3mm 2.6um	No	Yes	No: Method 537 modified
		S3	61	Yes												
		S5	6 (x10 dilution)	No												
	Post	S2	61	Yes												
		S4		No												
		S6		Yes												
3	Pre	S1	61.28	Yes	Homogenisation	SPE (Strata-X-AW, 33um, 200mg/ 6mL	MeOH/ NH4OH		Room (30 min)	SPE	LC-MSMS or LCQQQ (Waters XEVO-TQS-micro)	C18	1.7µm 2.1x100mm	Yes	No	No
		S3	61.54													
		S5	60.04													
	Post	S2	250.28													
		S4	250.3													
		S6	250.4													
4	Pre/ Post	All	8	No	Homogenisation	Direct Injection	Acetonitrile	Shaking	Room		LC-MSMS or LCQQQ	C18	10 cm x 2.1 mm x 1.7 µm	Yes	No	No
5	Pre	S1,S3,S5	50	Yes	No Pretreatment	SPE (DVB Cartirdge)	Methanol/ Water				LC-MSMS or LCQQQ	C18	150mm x 2.1 mm x 2.7 µm	Yes	No	Yes: EPA 537.1 (Not accredited for TOP)
	Post	S2,S4,S6	120													
7	Pre	S1,S3,S5	2.5	No		Direct Injection	Methanol	Vortex		Filter with 0.2um RC	LC-MSMS or LCQQQ	Phenyl-Hexyl	100mm x 2.1mm x 3um	Yes	No	No
	Post	S2,S4,S6	5		pH adjustment (pH to neutral)											
8	Pre	S1,S3,S5	5	No		Direct Injection					LC-MSMS or LCQQQ	Poroshell EC-C18	100mm x 2.1mm x 2.7 um	Yes	Yes	No
	Post	S2,S4,S6								Centrifugation						
9	Pre	S1,S3,S5	60	Yes	pH adjustment	SPE (STRATA XL-AW)	NH4-MeOH	SPE	Room (20 min)	Strata XL-AW	LC-MSMS or LCQQQ	C18	50 x 2.1 mm	No	No	Yes: EPA 537
	Post	S2,S4,S6	20	No												
10	Pre	S1,S3,S5	50	Yes	Homogenisation	SPE (WAX)	0.2% acetic acid in methanol	Shaking	Room (quickly)		LC-MSMS or LCQQQ	HPH-C18	2.1 x 50 mm x 2.7 micron	Yes	No	No
	Post	S2	50	No												
		S4	5													
		S6	5													
11	Pre	S1,S3,S5	0.5	No	Homogenisation	Direct Injection					LC-MSMS or LCQQQ	C18, Fluoro	2.0 mm ID, 50 mm L, 1.6 µm	Yes	Yes	No
	Post	S2,S4,S6						Vortex								Yes: EPA

Lab Code	Pre/ Post	Sample	Sample Size (mL)	Bottle Rinsed	Sample Pretreatment	Extraction Technique	Extraction Solvent(s)	Extraction Process	Extraction Temperature	Extraction Clean Up	Instrument	Column Type	Column Specifications	Delay Column	Blank Correction	Standard Method
12	Pre/ Post	All	10	Yes	pH adjustment	SPE		Sonication	Room		Orbitrap	C18		Yes		
13	Pre	S1,S3,S5	50	No	pH adjustment	SPE (WAX)	Methanol/ basic Methanol	Vortex	Room	Centrifuge	LC-MSMS or LCQQQ	C18	50cm x 2.1 x 1.7 um	Yes	No	Yes: based on USEPA 537
	Post	S2,S4,S6	10			Direct Injection	Methanol	Sonication								No
14	Pre	S1,S3,S5	5	No	Dilution 1:1 MeOH	Direct Injection					LC-MSMS or LCQQQ	C18	100 x 3.0 x 1.8um	Yes	No	No
	Post	S2,S4,S6			pH adjustment (dilute methanol 1:1)											
15	Pre	S1,S3,S5	10	Yes	Homogenisation	SPE (Waters Oasis HLB)	Methanol	Sonication	Room	SPE	Orbitrap	C18	50 cm x 2.1 cm x 2.7 um	Yes	No	No
	Post	S2,S4,S6	5					Shaking								
16	Pre	S1	60.99	Yes	Homogenisation	SPE (Polymeric reverse phase (styrene divinylbenzene)	Acetonitrile /Methanol	SPE	Room	SPE	LC-MSMS or LCQQQ	C18	1.6µm, 2.0mm x 50mm	Yes	No	No
		S3	61.59													
		S5	61.99													
	Post	S2,S4,S6	5	No	pH adjustment	Direct Injection										
18	Pre	S1	58.61	Yes	Homogenisation	SPE (Polymeric reverse phase styrene divinylbenzene)	Acetonitrile /Methanol	SPE	Room	SPE	LC-MSMS or LCQQQ	C18	1.6µm, 2.0mm x 50mm	Yes	No	No
		S3	60.65													
		S5	59.44													
	Post	S2,S4,S6	5	No	pH adjustment	Direct Injection										
19	Pre/ Post	All	50	Yes		SPE (WAX)	MeOH				LC-MSMS or LCQQQ	C18	100x2 mm	Yes	No	No
20	Pre	S1,S3,S5	10	No	pH adjustment	SPE (StrataX-AW 33um)	Acetonitrile with 1% ammonium hydroxide				LC-MSMS or LCQQQ	C18	50x2.1mm, 1.6um	No	Yes	
	Post	S2,S4,S6	20											No	No	
21	Pre/ Post	All	5	No		SPE (WAX 150mg/6cc)	NH4OH/ methanol		Room		LC-MSMS or LCQQQ	C18	10cm x 3.0mm x 3um	Yes	No	No

Table 3 Labelled Internal Standards Pre and Post Oxidation

Lab Code	Pre/ Post	Labelled Standard Added Before Extraction	Labelled Standard Source	Recovery Correction	Labelled Standard Added Before Instrument Analysis
1	Pre/ Post	PFBS: Perfluoro-n-13C4butanoic acid MPFBA; PFPeS: Perfluoro-n-13C5pentanoic acid M5PFPeA; PFHxS: Perfluoro-n-1,2,3,4,6-13C5hexanoic acid M5PFHxA; PFHpS: Perfluoro-n-1,2,3,4-13C4heptanoic acid M4PFHpA; PFOS: Perfluoro-n-13C8octanoic acid M8PFOA; PFNS: Perfluoro-n-13C9nonanoic acid M9PFNA; PFDS: Perfluoro-n-1,2,3,4,6-13C6decanoic acid M6PFDA; PFBA: Perfluoro-n-1,2,3,4,6,7-13C7undecanoic acid M7PFUdA; PFPeA: Perfluoro-n-1,2 13C2dodecanoic acid MPFDoA; PFHpA: Perfluoro-n-1,2 13C2tetradecanoic acid M2PFTeDA; PFOA: Sodium perfluoro-1-2,3,4 13C3 butanesulfonate M3PFBS; PFDA: Sodium perfluoro-1-1,2,3 13C3 hexanesulfonate M3PFHxS; PFDoA: Sodium perfluoro-1- 13C8 ocatanesulfonate M8PFOS; 6:2 FTS: Perfluoro-1-13C8otanesulfonamide; 8:2 FTS: N-methyl-d3-perfluoro-1-octancesulfonamide; PFOSA: N-ethyl-d5-perfluoro-1-octanesulfonamide; N-EtFOSA: N-ethyl-d5-perfluoro-1-octanesulfonamide; N-EtFOSAA: d5-NEtFOSAA	Wellington	Yes	
3	Pre	PFBS: 13C3-PFBS; PFPeS: 13C3-PFBS; PFHxS: 13C3-PFHxS; PFHpS: 13C8-PFOS; PFOS: 13C8-PFOS; PFNS: 13C8-PFOS; PFDS: 13C8-PFOS; PFBA: 13C3-PFBA; PFPeA: 13C3-PFPeA; PFHxA: 13C2-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C2-PFOA; PFNA: 13C5-PFNA; PFDA: 13C2-PFDA; PFUdA: 13C2-PFUdA; PFDoA: 13C2-PFDoA; PFTrDA: 13C2-PFDoA; PFTeDA: 13C2-PFTeDA; 6:2 FTS: 13C2-6:2 FTS; 8:2 FTS: 13C2-8:2 FTS; PFOSA: 13C8-PFOSA; N-MeFOSA: d3-N-MeFOSA; N-EtFOSA: d5-N-EtFOSA; N-MeFOSAA: d3-N-MeFOSAA; N-EtFOSAA: d5-N-EtFOSAA; N-MeFOSE: d7-N-MeFOSE; N-EtFOSE: d9-N-EtFOSE	Wellington	Yes	PFBS: 18O2-PFHxS; PFPeS: 18O2-PFHxS; PFHxS: 18O2-PFHxS; PFHpS: 13C4-PFOS; PFOS: 13C4-PFOS; PFNS: 13C4-PFOS; PFDS:13C4-PFOS; PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C5-PFHxA; PFOA: 13C8-PFOA; PFNA: 13C9-PFNA; PFDA: 13C2-PFDA; PFUdA: 13C7-PFUdA; PFDoA: 13C2-PFDA; PFTrDA: 13C7-PFUdA; PFTeDA: 13C7-PFUdA; 6:2 FTS: 13C4-PFOS; 8:2 FTS: 13C4-PFOS; PFOSA: 13C7-PFUdA; N-MeFOSA: 13C7-PFUdA; N-EtFOSA: 13C7-PFUdA; N-MeFOSAA: 13C7-PFUdA; N-EtFOSAA: 13C7-PFUdA; N-MeFOSE: 13C7-PFUdA; N-EtFOSE: 13C7-PFUdA
	Post				As above except PFOA: 13C4-PFOS
4	Pre	PFBS: MPFBS; PFPeS: MPFBS; PFHxS: MPHxS; PFHpS: MPFOS; PFOS: MPFOS; PFNS: MPFOS; PFDS: MPFOS; PFBA: MPFBA; PFPeA: MPFHxA; PFHxA: MPFHxA; PFHpA: MPFHpA; PFOA: MPFOA; PFNA: MPFNA; PFDA: MPFDA; PFUdA: MPFUdA; PFDoA: MPFDoA; PFTrDA: MPFDoA; PFTeDA: MPFTeDA; 6:2 FTS: M6:2 FTS; 8:2 FTS: M8:2 FTS; PFOSA: MPFOS; N-MeFOSA: MEtFOSA; N-EtFOSA: MEtFOSA; N-MeFOSE: MEtFOSA; N-EtFOSE: MEtFOSA	Wellington	Yes	As per Labelled Standard Added Before Extraction
	Post	As above except PFBS: MPFOS; PFPeS: MPFOS; PFNS: -; PFHpA: MPFHxA; PFTeDA: MPFDoA			
5	Pre/ Post	PFBS: 13C3-PFBS; PFHxS: 13C3-PFHxS; PFOS: 13C8-PFOS; PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C8-PFOA; PFNA: 13C9-PFNA; PFDA: 13C6-PFDA; PFUdA: 13C7-PFUdA; PFDoA: 13C2-PFDoA; PFTeDA: 13C2-PFTeDA; 6:2 FTS: 13C2-	Wellington	Yes	PFOS: 13C4-PFOS; PFBA: 13C3-PFBA; PFOA: 13C2-PFOA; PFDA: 13C2-PFDA

Lab Code	Pre/ Post	Labelled Standard Added Before Extraction	Labelled Standard Source	Recovery Correction	Labelled Standard Added Before Instrument Analysis
		6:2FtS; 8:2 FTS: 13C2-8:2FtS; PFOSA: 13C8-FOSA; N-MeFOSAA: 2H3-N-MeFOSAA; N-EtFOSAA: 2H5-N-EtFOSAA			
7	Pre/ Post	PFBS: 13C4-PFOS; PFPeS: 13C4-PFOS; PFHxS: 13C4-PFOS; PFHpS: 13C4-PFOS; PFOS: 13C4-PFOS; PFDS: 13C4-PFOS; PFBA: 13C4 PFBA; PFPeA: 13C4-PFOS; PFHxA: 13C4-PFOS; PFHpA: 13C4-PFOS; PFOA: 13C4-PFOS; PFNA: 13C4-PFOS; PFDA: 13C4-PFOS; PFUdA: 13C2-PFDoA; PFDoA: 13C2-PFDoA; PFTrDA: 13C2-PFDoA; PFTeDA: 13C2-PFDoA; 6:2 FTS: 13C2-4-2 FTS; 8:2 FTS: 13C2-4-2 FTS; PFOSA: 13C4-PFOS; N-MeFOSA: d5-EtFOSA; N-EtFOSA: d5-EtFOSA; N-MeFOSAA: d5-EtFOSA; N-EtFOSAA: d5-EtFOSA; N-MeFOSE: d5-EtFOSA; N-EtFOSE: d5-EtFOSA	Wellington	Yes	
8	Pre/ Post	PFOS: PFOS-13C8; PFOA: PFOA-13C8; N-EtFOSA: N-EtFOSA-d5	Wellington	No	PFHxS: PFHxS-18O2; PFOS: PFOS-13C4; PFBA: PFBA-13C4; PFHxA: PFHxA-13C2; PFOA: PFOA-13C4; PFNA: PFNA-13C5; PFDA: PFDA-13C2; PFUdA: PFUdA-13C2; PFDoA: PFDoA-13C2; N-MeFOSA: N-MeFOSA-d3; N-EtFOSAA: N-EtFOSAA-d5
9	Pre/ Post	PFBS: M3PFBS; PFPeS: M3PFBS; PFHxS: M3PFHxS; PFHpS: M3PFHxS; PFOS: M8PFOS; PFNS: M8PFOS; PFDS: M8PFOS; PFBA: MPFBA; PFPeA: M5PFPeA; PFHxA: M5PFHxA; PFHpA: M4PFHpA; PFOA: M8PFOA; PFNA: M9PFNA; PFDA: M6PFDA; PFUdA: M7PFUdA; PFDoA: MPFDoA; PFTrDA: MPFDoA; PFTeDA: M2PFTeDA; 6:2 FTS: M2-6:2 FTS; 8:2 FTS: M2-8:2 FTS; PFOSA: M8FOSA-I; N-MeFOSA: d-N-MeFOSA-M; N-EtFOSA: d-N-EtFOSA-M; N-MeFOSAA: d3-N-MeFOSAA; N-EtFOSAA: d5-N-EtFOSAA; N-MeFOSE: d7-N-MeFOSE-M; N-EtFOSE: d9-N-EtFOSE-M	Wellington	Yes	PFOS: MPFOS; PFBA: M3PFBA; PFOA: M2PFOA; PFDA: MPFDA
10	Pre/ Post	PFBS: PFBS - 13C3; PFPeS: PFHxS - 13C3; PFHxS: PFHxS - 13C3; PFHpS: PFHxS - 13C3; PFOS: PFOS - 13C8; PFNS: PFOS - 13C8; PFDS: PFOS - 13C8; PFBA: PFBA - 13C4; PFPeA: PFPeA - 13C5; PFHxA: PFHxA - 13C5; PFHpA: PFHpA - 13C4; PFOA: PFOA - 13C4; PFNA: PFNA - 13C9; PFDA: PFDA - 13C6; PFUdA: PFUdA - 13C7; PFDoA: PFDoA - 13C2; PFTrDA: PFTeDA - 13C2; PFTeDA: PFTeDA - 13C2; 6:2 FTS: 6:2 FTS - 13C2; 8:2 FTS: 8:2 FTS - 13C2; PFOSA: PFOSA - 13C8; N-MeFOSA: N-MeFOSA - 2H3; N-EtFOSA: N-EtFOSA - D5; N-MeFOSAA: N-MeFOSAA - 2H3; N-EtFOSAA: N-EtFOSA - 2H5; N-MeFOSE: N-MeFOSE - D7; N-EtFOSE: N-EtFOSE - 2H9	Wellington	No	PFBS: PFHxS-18O2; PFPeS: PFHxS-18O2; PFHxS: PFHxS-18O2; PFHpS: PFHxS-18O2; PFOS: PFOS-13C4; PFNS: PFOS-13C4; PFDS: PFOS-13C4; PFBA: PFBA-13C3; PFPeA: PFBA-13C3; PFHxA: PFOA-13C2; PFHpA: PFOA-13C2; PFOA: PFOA-13C2; PFNA: PFNA-13C5; PFDA: PFDA-13C2; PFUdA: PFDA-13C2; PFDoA: PFDA-13C2; PFTrDA: PFDA-13C2; PFTeDA: PFDA-13C2; 6:2 FTS: PFHxS-18O2; 8:2 FTS: PFHxS-18O2; PFOSA: PFOS-13C4; N-MeFOSA: PFOS-13C4; N-EtFOSA: PFOS-13C4; N-MeFOSAA: PFOS-13C4; N-EtFOSAA: PFOS-13C4; N-MeFOSE: PFOS-13C4; N-EtFOSE: PFOS-13C4
11	Pre/ Post	PFBS: 18O2 PFHxS ISTD; PFHxS: 18O2 PFHxS ISTD; PFHpS: 13C4 PFOS ISTD; PFOS: 13C4 PFOS ISTD; PFNS: 13C4 PFOS ISTD; PFDS: 13C4 PFOS ISTD; PFBA: 13C4 PFBA ISTD; PFPeA: 13C4 PFBA ISTD; PFHxA: 13C2 PFHxA ISTD; PFHpA: 13C2 PFHxA ISTD; PFOA: 13C4 PFOA ISTD; PFNA: 13C5 PFNA ISTD; PFDA: 13C2 PFDA ISTD; PFUdA: 13C2 PFUnDA ISTD; PFDoA: 13C2 PFDoDA ISTD; PFTrDA: 13C2 PFDoDA ISTD; PFTeDA: 13C2 PFDoDA ISTD; 6:2 FTS: 13C2 12C6 62FTS ISTD; 8:2 FTS: 13C2 12C6 62FTS ISTD; PFOSA: D5-EtFOSAA ISTD; N-MeFOSA: D5-EtFOSAA ISTD;	Wellington	Yes	As per Labelled Standard Added Before Extraction

Lab Code	Pre/ Post	Labelled Standard Added Before Extraction	Labelled Standard Source	Recovery Correction	Labelled Standard Added Before Instrument Analysis
		N-EtFOSA: D5-EtFOSAA ISTD; N-MeFOSAA: D5-EtFOSAA ISTD; N-EtFOSAA: D5-EtFOSAA ISTD			
12	Pre/ Post	PFBS: PFBS-13C3; PFPeS: PFHxS-18O2; PFHxS: PFHxS-18O2; PFHpS: PFOS-13C4; PFOS: PFOS-13C4; PFNS: PFOS-13C4; PFDS: 6:2 FTS-13C2; PFBA: PFBA-13C4; PFPeA: PFPeA-13C3; PFHxA: PFHxA-13C2; PFHpA: PFHpA-13C4; PFOA: PFOA-13C4; PFNA: PFNA-13C5; PFDA: PFDA-13C2; PFUdA: PFUnDA-13C2; PFDoA: PFDoDA-13C2; PFTrDA: PFDoDA-13C2; PFTeDA: PFTeDA-13C2; 6:2 FTS: 6:2 FTS-13C2; 8:2 FTS: 6:2 FTS-13C2; PFOSA: FOSA-13C8; N-MeFOSA: MeFOSA-D3; N-EtFOSA: EtFOSA-D5; N-MeFOSAA: MeFOSAA-D3; N-EtFOSAA: EtFOSAA-D5; N-MeFOSE: MeFOSE-D7; N-EtFOSE: EtFOSE-D9	Wellington	Yes	
13	Pre/ Post	PFBS: 13 C3 PFBS; PFPeS: 13 C3 PFBS; PFHxS: 18 O2 PFHxS; PFHpS: 13 C4 PFOS; PFOS: 13 C4 PFOS; PFNS: 13 C4 PFOS; PFDS: 13 C4 PFOS; PFBA: 13 C4 PFBA; PFPeA: 13 C3 PFPeA; PFHxA: 13 C2 PFHxA; PFHpA: 13 C4 PFHpA; PFOA: 13 C4 PFOA; PFNA: 13 C5 PFNA; PFDA: 13 C2 PFDA; PFUdA: 13 C2 PFUnDA; PFDoA: 13 C2 PFDoA; PFTrDA: 13 C2 PFTeDA; PFTeDA: 13 C2 PFTeDA; 6:2 FTS: 13 C2 6:2FTS; 8:2 FTS: 13 C2 8:2FTS; PFOSA: 13 C8 FOSA; N-MeFOSA: d3 N MeFOSA; N-EtFOSA: d5 N EtFOSA; N-MeFOSAA: d3 N MeFOSAA; N-EtFOSAA: d5 N EtFOSAA; N-MeFOSE: d7 N MeFOSE; N-EtFOSE: d9 N EtFOSE	Wellington	Yes	All Added Before Extraction
14	Pre/ Post	PFBS: 13C3-PFBS; PFPeS: 13C3-PFBS; PFHxS: 13C3-PFHxS; PFHpS: 13C3-PFHxS; PFOS: 13C8-PFOS; PFNS: 13C8-PFOS; PFDS: 13C8-PFOS; PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C8-PFOA; PFNA: 13C9-PFNA; PFDA: 13C6-PFDA; PFUdA: 13C7-PFUdA; PFDoA: 13C2-PFDoA; PFTrDA: 13C2-PFTeDA; PFTeDA: 13C2-PFTeDA; 6:2 FTS: 13C8-PFOA; 8:2 FTS: 13C2-8:2FTS; PFOSA: d3-N-MeFOSA; N-MeFOSA: d3-N-MeFOSA; N-EtFOSA: d5-EtFOSA; N-MeFOSAA: d3-N-MeFOSAA; N-EtFOSAA: d5-N-EtFOSAA; N-MeFOSE: d9-EtFOSE; N-EtFOSE: d9-EtFOSE	Wellington	No	
15	Pre/ Post	PFBS: PFBS-13C3; PFPeS: PFHxS-18O2; PFHxS: PFHxS-18O2; PFHpS: PFOS-13C4; PFOS: PFOS-13C4; PFNS: PFBA-13C4; PFDS: PFHxS-18O2; PFBA: PFBA-13C4; PFPeA: PFPeA-13C3; PFHxA: PFHxA-13C2; PFHpA: PFHpA-13C4; PFOA: PFOA-13C4; PFNA: PFNA-13C5; PFDA: PFDA-13C2; PFUdA: PFUnDA-13C2; PFDoA: PFDoDA-13C2; PFTrDA: PFDoDA-13C2; PFTeDA: PFTeDA-13C2; 6:2 FTS: 6:2 FTS-13C2; 8:2 FTS: 8:2 FTS-13C2; PFOSA: FOSA-13C8; N-MeFOSA: MeFOSA-D3; N-EtFOSA: EtFOSA-D5; N-MeFOSAA: MeFOSAA-D3; N-EtFOSAA: EtFOSAA-D5; N-MeFOSE: MeFOSE-D7; N-EtFOSE: EtFOSE-D9	Wellington	Yes	All: PFOS-13C8
16	Pre/ Post	PFBS: 13C3-PFBS; PFPeS: 16O2-PFHxS; PFHxS: 16O2-PFHxS; PFHpS: 13C8-PFOS; PFOS: 13C8-PFOS; PFDS: 13C8-PFOS; PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA:	Wellington	Yes	

Lab Code	Pre/ Post	Labelled Standard Added Before Extraction	Labelled Standard Source	Recovery Correction	Labelled Standard Added Before Instrument Analysis
		13C4-PFOA; PFNA: 13C5-PFNA; PFDA: 13C6-PFDA; PFUdA: 13C2-PFUnDA; PFDoA: 13C2-PFDoDA; PFTrDA: 13C2-PFTeDA; PFTeDA: 13C2-PFTeDA; 6:2 FTS: 13C2-6:2 FTS; 8:2 FTS: 13C2-8:2 FTS; PFOSA: 13C8-FOSA; N-MeFOSA: d3-MeFOSA; N-EtFOSA: d5-EtFOSA; N-MeFOSAA: d3-MeFOSAA; N-EtFOSAA: d5-EtFOSAA; N-MeFOSE: d7-MeFOSE; N-EtFOSE: d3EtFOSE			
18	Pre	PFBS: 13C3-PFBS; PFPeS: 16O2-PFHxS; PFHxS: 16O2-PFHxS; PFHpS: 13C8-PFOS; PFOS: 13C8-PFOS; PFDS: 13C8-PFOS; PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C4-PFOA; PFNA: 13C5-PFNA; PFDA: 13C6-PFDA; PFUdA: 13C2-PFUnDA; PFDoA: 13C2-PFDoDA; PFTrDA: 13C2-PFTeDA; PFTeDA: 13C2-PFTeDA; 6:2 FTS: 13C2-6:2 FTS; 8:2 FTS: 13C2-8:2 FTS; PFOSA: 13C8-FOSA; N-MeFOSA: d3-MeFOSA; N-EtFOSA: d5-EtFOSA; N-MeFOSAA: d3-MeFOSAA; N-EtFOSAA: d5-EtFOSAA; N-MeFOSE: d7-MeFOSE; N-EtFOSE: d3EtFOSE	Wellington	Yes	
	Post				As per Labelled Standard Added Before Extraction - Pre
19	Pre/ Post	PFBS: M3PFBS; PFHxS: M3PFHxS; PFOS: M8PFOS; PFBA: MPFBA; PFPeA: M5PFPeA; PFHxA: M5PFHxA; PFHpA: M4PFHpA; PFOA: M8PFOA; PFNA: M9PFNA; PFDA: M6PFDA; PFUdA: M7PFUdA; PFDoA: MPFDoA; PFTeDA: M2PFTeDA; 6:2 FTS: M2-6:2FTS; 8:2 FTS: M2-8:2FTS; PFOSA: M8FOSA-1; N-MeFOSA: D-N-MeFOSA-M; N-EtFOSA: D-N-EtFOSA-M; N-MeFOSAA: D3-N-MeFOSAA; N-EtFOSAA: D5-N-EtFOSAA; N-MeFOSE: D7-N-MeFOSE-M; N-EtFOSE: D9-N-EtFOSE-M	Wellington	Yes (Approximate as normally don't calculate internal standard recovery)	
20	Pre/ Post	PFHxS: 18O2-PFHxS; PFOS: 13C8-PFOS; PFBA: 13C4-PFBA; PFHxA: 13C2-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C8-PFOA; PFNA: 13C5-PFNA; PFDA: 13C6-PFDA; PFUdA: 13C2-PFUnA; PFDoA: 13C2-PFDoA; 6:2 FTS: 13C2-62FTS; 8:2 FTS: 13C2-82FTS; PFOSA: 13C8-FOSA; N-MeFOSA: d3-MeFOSA; N-EtFOSA: d3-MeFOSA; N-MeFOSAA: d3-N-MeFOSAA; N-EtFOSAA: d5-N-EtFOSAA; N-MeFOSE: d7-MeFOSE; N-EtFOSE: d9-EtFOSE		Yes	
21	Pre/ Post	PFBS: 13C3 PFBS; PFPeS: 13C3 PFBS; PFHxS: 18O2 PFHxS; PFHpS: 13C4 PFOS; PFOS: 13C4 PFOS; PFNS: 13C4 PFOS; PFDS: 13C4 PFOS; PFBA: 13C4 PFBA; PFPeA: 13C5 PFPeA; PFHxA: 13C2 PFHxA; PFHpA: 13C4 PFHpA; PFOA: 13C4 PFOA; PFNA: 13C5 PFNA; PFDA: 13C2 PFDA; PFUdA: 13C2 PFUnA; PFDoA: 13C2 PFDoA; PFTrDA: 13C2 PFDoA; PFTeDA: 13C2 PFTeDA; 6:2 FTS: M2-6:2 FTS; 8:2 FTS: M2-8:2 FTS; PFOSA: 13C8 PFOSA; N-MeFOSA: d-N-MeFOSA-M; N-EtFOSA: d-N-EtFOSA-M; N-MeFOSAA: d3-NMeFOSAA; N-EtFOSAA: d7-NEtFOSAA; N-MeFOSE: d7-N-MeFOSE-M; N-EtFOSE: d9-N-EtFOSA-M	Wellington	Yes	All: 13C2-PFOA: solely used to quantitate extracted standards recoveries

3.2 Basis of Participants' Measurement Uncertainty Estimates

Table 4 Basis of Participants' Uncertainty Estimate

Lab Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
1	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis Instrument calibration	Laboratory bias from PT studies Recoveries of SS	NATA - Estimating and reporting MU of chemical test results
3	Uncertainty as a function of standard error	Control samples - SS	Instrument calibration Recoveries of SS Standard purity	Eurochem / CITAC Guide CG 4
4	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	Recoveries of SS	Eurachem/CITAC Guide
5	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS	Internal SOP
7	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	Recoveries of SS	Eurachem/CITAC Guide
8	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis Instrument calibration	Recoveries of SS	NATA - Estimating and reporting MU of chemical test results
9	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis	CRM	NATA - Estimating and reporting MU of chemical test results
10	Standard deviation of replicate analyses multiplied by 2 or 3	Duplicate analysis	CRM Instrument calibration	NATA - Estimating and reporting MU of chemical test results
11	if other please type	Control samples - SS Duplicate analysis		
12	Standard deviation of replicate analyses multiplied by 2 or 3	Standard deviation from PT studies		NATA - Estimating and reporting MU of chemical test results
		Control samples - SS Duplicate analysis	CRM Recoveries of SS Standard purity	
13	Top Down - precision and estimates of the method and laboratory bias	Control samples	CRM Instrument calibration Recoveries of SS	NATA - Estimating and reporting MU of chemical test results
14	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Instrument calibration	Instrument calibration Recoveries of SS Standard purity	Eurachem/CITAC Guide
15		Standard deviation from PT studies		NATA - Estimating and reporting MU of chemical test results
		Control samples - SS Duplicate analysis	CRM Recoveries of SS Standard purity	
16	Top Down - precision and estimates of the method and laboratory bias using the historic QC data	Control samples - SS Duplicate analysis	Recoveries of SS	Eurachem/CITAC Guide
18	Top Down - precision and estimates of the method and laboratory bias using the historic QC data	Control samples - SS Duplicate analysis		Eurachem/CITAC Guide
19	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis Instrument calibration	Instrument calibration Standard purity	NATA - Estimating and reporting MU of chemical test results
20	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM Duplicate analysis Instrument calibration	CRM Instrument calibration Laboratory bias from PT studies	ISO/GUM

Lab Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
			Recoveries of SS Standard purity	
21	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS	Recoveries of SS	USEPA SW-846

*SS = Spiked Samples, RM = Reference Material, CRM = Certified Reference Material

3.3 Participants' Comments

The study co-ordinator welcomes comments or suggestions from participants about this study or possible future studies. Participants' comments are reproduced in Table 5.

Table 5 Participants' Comments

Lab Code	Participants' Comments	Study manager response
16	Study: Suggest study for PFAS in Biosolids	We will consider this matrix in a future TOP assay study if there is sufficient interest from our participants.
18	Study: Suggest study for PFAS in Biosolids	

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 6 to 57 with the summary statistics: robust average, mean, median, maximum, minimum, robust standard deviation (SD_{rob}) and robust coefficient of variation (CV_{rob}). Bar charts of results and performance scores are presented in Figures 2 to 53.

An example chart with interpretation guide is shown in Figure 1.

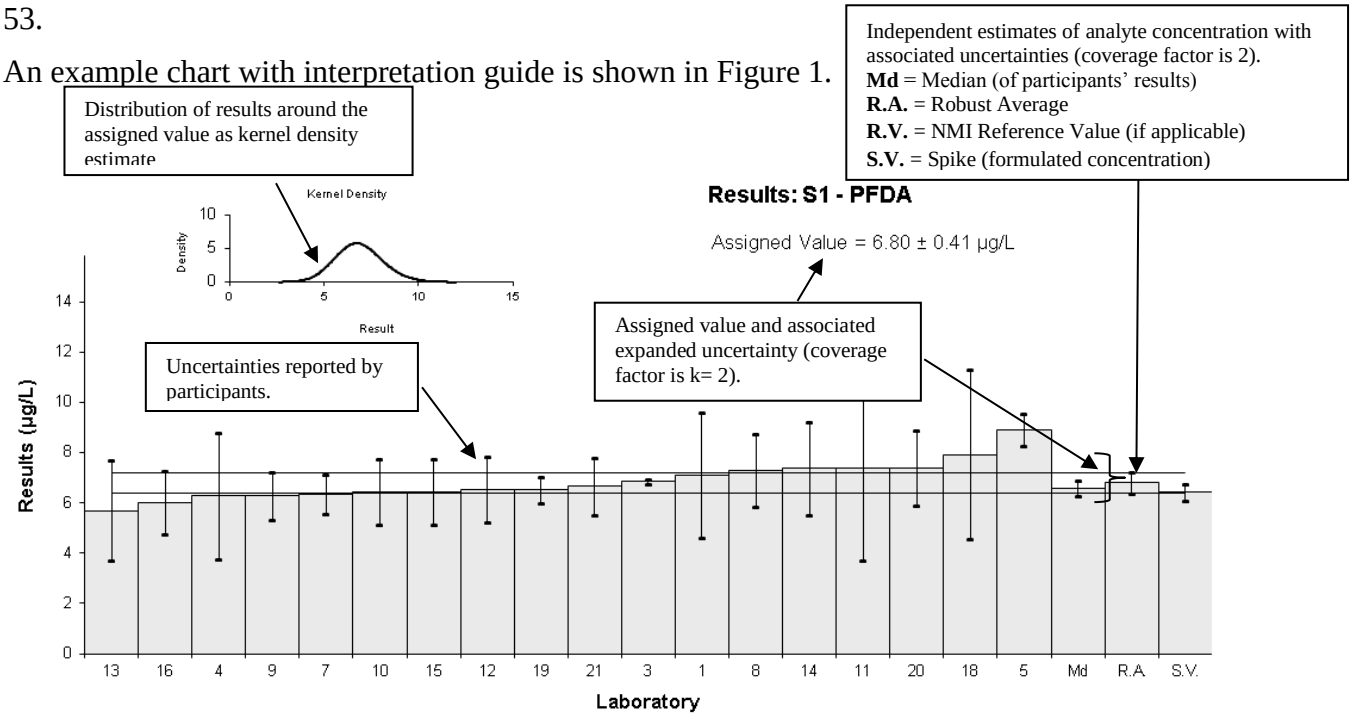


Figure 1 Guide to Presentation of Results

4.2 Assigned Value

An example of the assigned value calculation using data from the present study is given in Appendix 3. The assigned value is defined as: 'value attributed to a particular property of a proficiency test item.'[3] In this study assigned values were the robust average of participants' results; the expanded uncertainties were estimated from the associated robust standard deviations.

4.3 Robust Average

The robust averages and associated expanded measurement uncertainties were calculated using the procedure described in 'Statistical methods for use in proficiency testing by inter-laboratory comparisons, ISO13528:2015(E)'.[8]

4.4 Robust Between-Laboratory Coefficient of Variation

The robust between-laboratory coefficient of variation (robust CV) is a measure of the variability of participants' results and was calculated using the procedure described in ISO13528:2015(E).[8]

4.5 Performance Coefficient of Variation (PCV)

The performance coefficient of variation (PCV) is a measure of the between laboratory variation that in the judgement of the study coordinator would be expected from participants. It is important to note that is not the coefficient of variation of participant results. The fixed value set for PCV is based on the existing regulation, the acceptance criteria indicated by the methods, the matrix, the concentration level of analyte and on experience from previous studies. It is backed up by mathematical models such as Thompson Horwitz equation.[9] By setting a fixed and realistic value for the PCV, the participant's performance does not depend on other participants' performance and can be compared from study to study and against achievable performance

4.6 Target Standard Deviation

The target standard deviation (σ) is the product of the assigned value (X) and the performance coefficient of variation (PCV) as presented in Equation 1. This value is used for calculation of each participant z-score.

$$\sigma = X * PCV \qquad \textit{Equation 1}$$

4.7 z-Score

An example of z-score calculation using data from the present study is given in Appendix 3.

For each participant's result a z-score is calculated according to Equation 2 below:

$$z = \frac{(\chi - X)}{\sigma} \qquad \textit{Equation 2}$$

where:

z is z-score
χ is a participant's result
X is the assigned value
σ is the target standard deviation from equation 1

A z-score with absolute value (|z|):

- $|z| \leq 2.0$ is satisfactory;
- $2.0 < |z| < 3.0$ is questionable;
- $|z| \geq 3.0$ is unsatisfactory.

4.8 E_n-Score

An example of E_n-score calculation using data from the present study is given in Appendix 3.

The E_n-score is complementary to the z-score in assessment of laboratory performance. E_n-score includes measurement uncertainty and is calculated according to Equation 3 below:

$$E_n = \frac{(\chi - X)}{\sqrt{U_\chi^2 + U_X^2}} \qquad \textit{Equation 3}$$

where:

E_n is E_n-score
χ is a participant's result
X is the assigned value
U_χ is the expanded uncertainty of the participant's result
U_X is the expanded uncertainty of the assigned value

An E_n-score with absolute value ($|E_n|$):

- $|E_n| \leq 1.0$ is satisfactory;
- $|E_n| > 1.0$ is unsatisfactory.

4.9 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025:2017[10] must establish and demonstrate the traceability and measurement uncertainty associated with their test results. Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.[11]

5 TABLES AND FIGURES

Table 6

Sample Details

Sample No.	S1
Matrix.	MilliQ water, 6:2 diPAP, 8:2 diPAP, PFDA, PFDS
Analyte.	PFDS
Units	µg/L

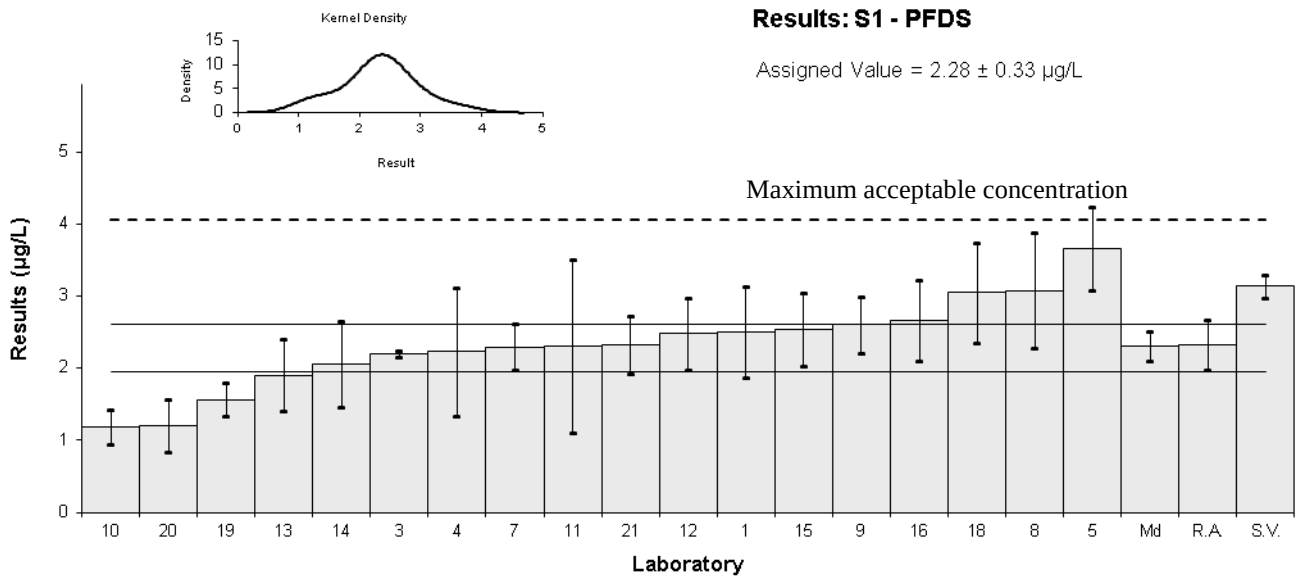
Participant Results

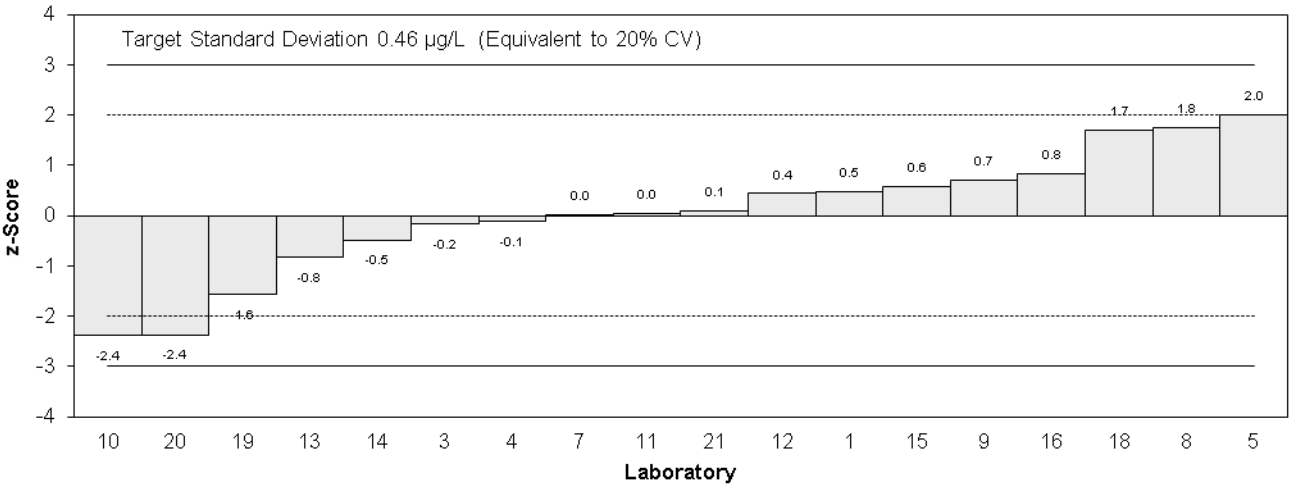
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	2.5	0.63	NR	0.48	0.31
3	2.20	0.0469	107	-0.18	-0.24
4	2.231	0.892	86	-0.11	-0.05
5*	3.66	0.58	NR	2.00	1.00
7	2.29	0.32	100.33	0.02	0.02
8	3.08	0.80	NR	1.75	0.92
9	2.6	0.39	104	0.70	0.63
10	1.19	0.24	99	-2.39	-2.67
11	2.3	1.2	110	0.04	0.02
12	2.48	0.496	83	0.44	0.34
13	1.9	0.5	96	-0.83	-0.63
14	2.06	0.60	86	-0.48	-0.32
15	2.54	0.508	76	0.57	0.43
16	2.66	0.555	NR	0.83	0.59
18	3.05	0.695	170	1.69	1.00
19	1.566	0.23	NR	-1.57	-1.78
20	1.2	0.36	NR	-2.37	-2.21
21	2.32	0.40	95	0.09	0.08

Statistics

Assigned Value**	2.28	0.33
Spike	3.14	0.16
Robust Average	2.33	0.35
Median	2.31	0.20
Mean	2.32	
N	18	
Max.	3.66	
Min.	1.19	
Robust SD	0.59	
Robust CV	25%	

*z-Score adjusted to 2 and E_n-score adjusted to 1 (see Section 6.3).
**Robust Average excluding Laboratory 5.


Kernel Density
Density
Result
Results: S1 - PFDS
Assigned Value = 2.28 ± 0.33 µg/L
Maximum acceptable concentration
Results (µg/L)
10 20 19 13 14 3 4 7 11 21 12 1 15 9 16 18 8 5 Md R.A S.V.
Laboratory


z-Scores: S1 - PFDS
Target Standard Deviation 0.46 µg/L (Equivalent to 20% CV)
z-Score
-2.4 -2.4 -1.6 -0.8 -0.5 -0.2 -0.1 0.0 0.0 0.1 0.4 0.5 0.6 0.7 0.8 1.7 1.8 2.0
10 20 19 13 14 3 4 7 11 21 12 1 15 9 16 18 8 5
Laboratory

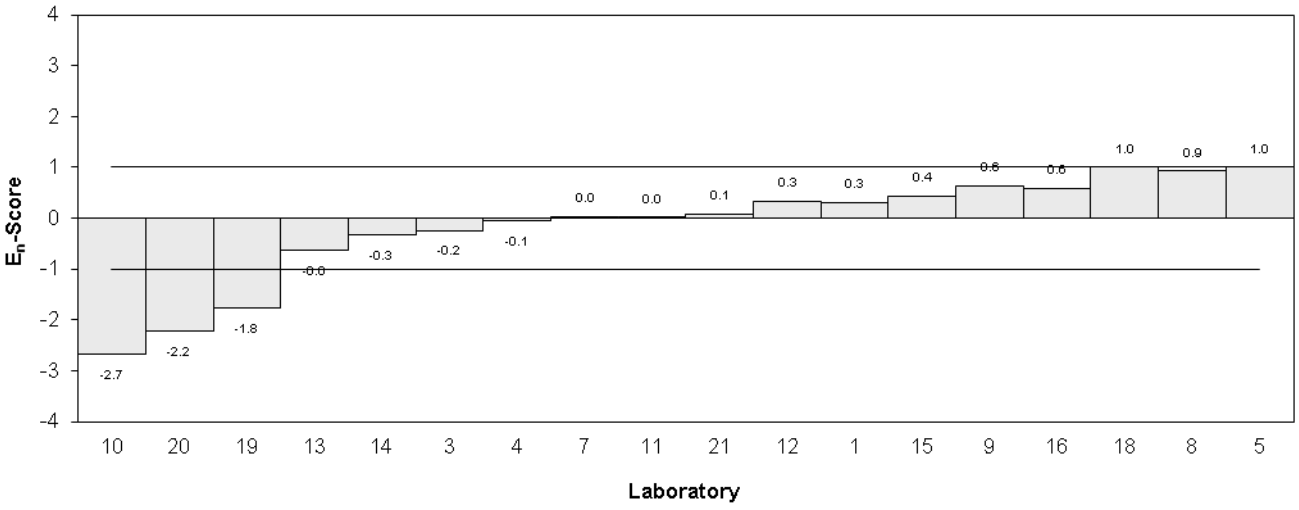

En-Scores: S1 - PFDS
En-Score
-2.7 -2.2 -1.8 -0.6 -0.3 -0.2 -0.1 0.0 0.0 0.1 0.3 0.3 0.4 0.6 0.6 1.0 0.9 1.0
10 20 19 13 14 3 4 7 11 21 12 1 15 9 16 18 8 5
Laboratory

Figure 2

Table 7

Sample Details

Sample No.	S1
Matrix.	MilliQ water, 6:2 diPAP, 8:2 diPAP, PFDA, PFDS
Analyte.	PFNA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	0.035	0.010	96
3	0.0261	0.000538	90.1
4	0.0383	0.0115	101
5	0.041	0.004	98
7	0.032	0.007	100.33
8	0.057	0.012	NR
9	0.031	0.0047	147
10	<0.04	0.01	103
11	0.03	0.015	90
12	0.0321	0.00963	78
13	0.03	0.02	100
14	0.03	0.01	101
15	0.0351	0.0105	90
16	0.03	0.009	98
18	0.05	0.004	169
19	0.038	0.00228	73
20	0.03	0.0075	NR
21	< 0.10	NR	97

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Robust Average	0.034	0.004
Median	0.032	0.002
Mean	0.035	
N	16	
Max.	0.057	
Min.	0.026	
Robust SD	0.006	
Robust CV	17%	

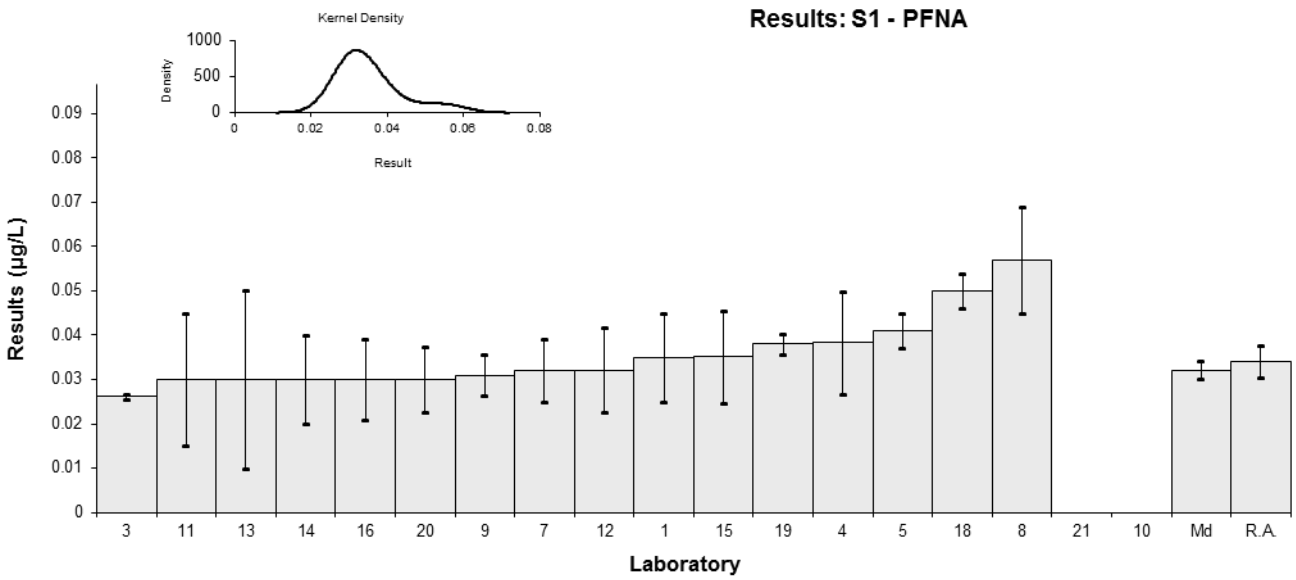

Results: S1 - PFNA
Kernel Density
Density
1000
500
0
0
0.02
0.04
0.06
0.08
Result
Results (µg/L)
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
3
11
13
14
16
20
9
7
12
1
15
19
4
5
18
8
21
10
Md
R.A.
Laboratory

Figure 3

Table 8

Sample Details

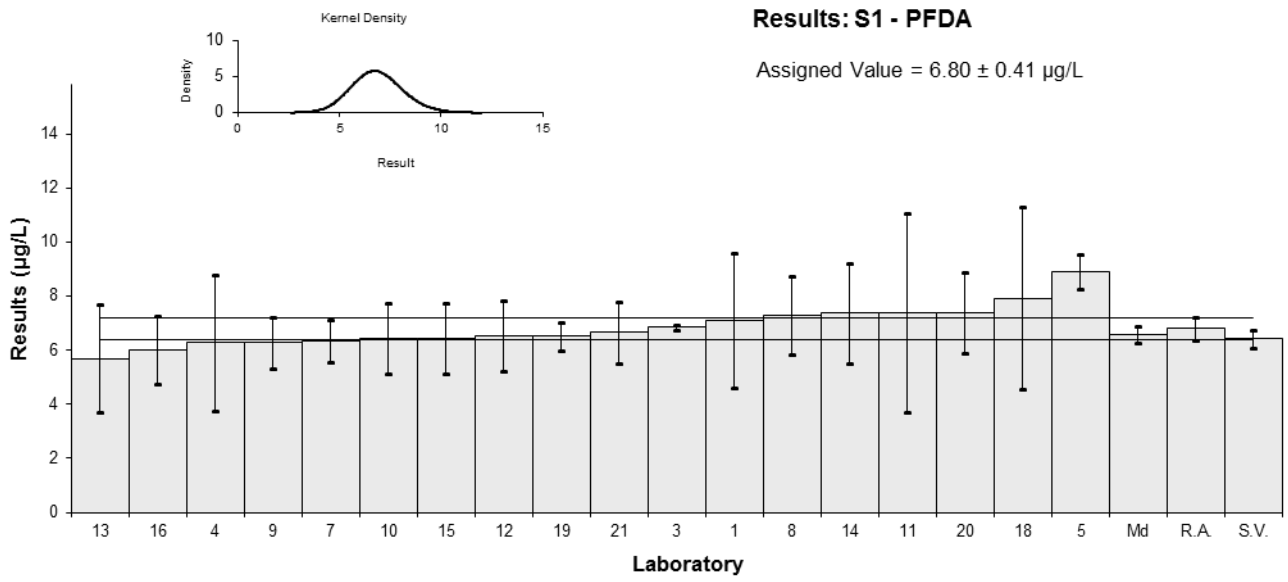
Sample No.	S1
Matrix.	MilliQ water, 6:2 diPAP, 8:2 diPAP, PFDA, PFDS
Analyte.	PFDA
Units	µg/L

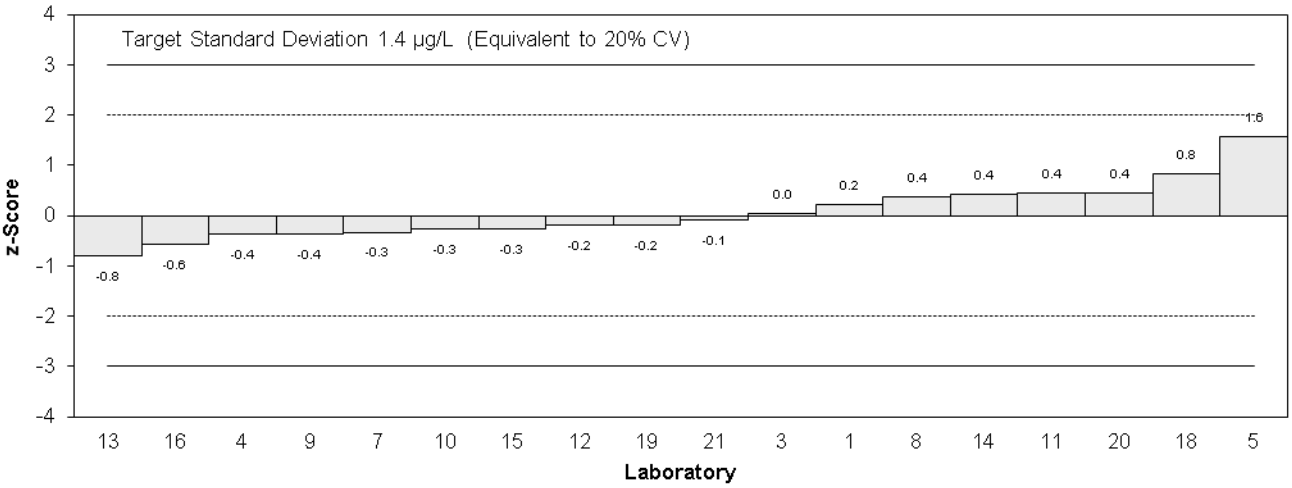
Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	7.1	2.5	88	0.22	0.12
3	6.86	0.0871	84.4	0.04	0.14
4	6.287	2.515	106	-0.38	-0.20
5	8.93	0.64	82	1.57	2.80
7	6.35	0.80	100.33	-0.33	-0.50
8	7.30	1.46	NR	0.37	0.33
9	6.3	0.95	103	-0.37	-0.48
10	6.44	1.29	93	-0.26	-0.27
11	7.4	3.7	100	0.44	0.16
12	6.53	1.31	79	-0.20	-0.20
13	5.7	2	91	-0.81	-0.54
14	7.38	1.85	77	0.43	0.31
15	6.447	1.2895	94	-0.26	-0.26
16	6.03	1.267	84	-0.57	-0.58
18	7.94	3.382	142	0.84	0.33
19	6.534	0.52272	70	-0.20	-0.40
20	7.4	1.48	NR	0.44	0.39
21	6.67	1.14	98	-0.10	-0.11

Statistics

Assigned Value	6.80	0.41
Spike	6.42	0.32
Robust Average	6.80	0.41
Median	6.60	0.30
Mean	6.87	
N	18	
Max.	8.93	
Min.	5.7	
Robust SD	0.7	
Robust CV	10%	


Kernel Density
Density
Result
Results: S1 - PFDA
Assigned Value = 6.80 ± 0.41 µg/L
Results (µg/L)
Laboratory
13 16 4 9 7 10 15 12 19 21 3 1 8 14 11 20 18 5 Md R.A. S.V.


z-Scores: S1 - PFDA
Target Standard Deviation 1.4 µg/L (Equivalent to 20% CV)
z-Score
Laboratory
13 16 4 9 7 10 15 12 19 21 3 1 8 14 11 20 18 5
-0.8 -0.6 -0.4 -0.4 -0.3 -0.3 -0.3 -0.2 -0.2 -0.1 0.0 0.2 0.4 0.4 0.4 0.4 0.8 1.6

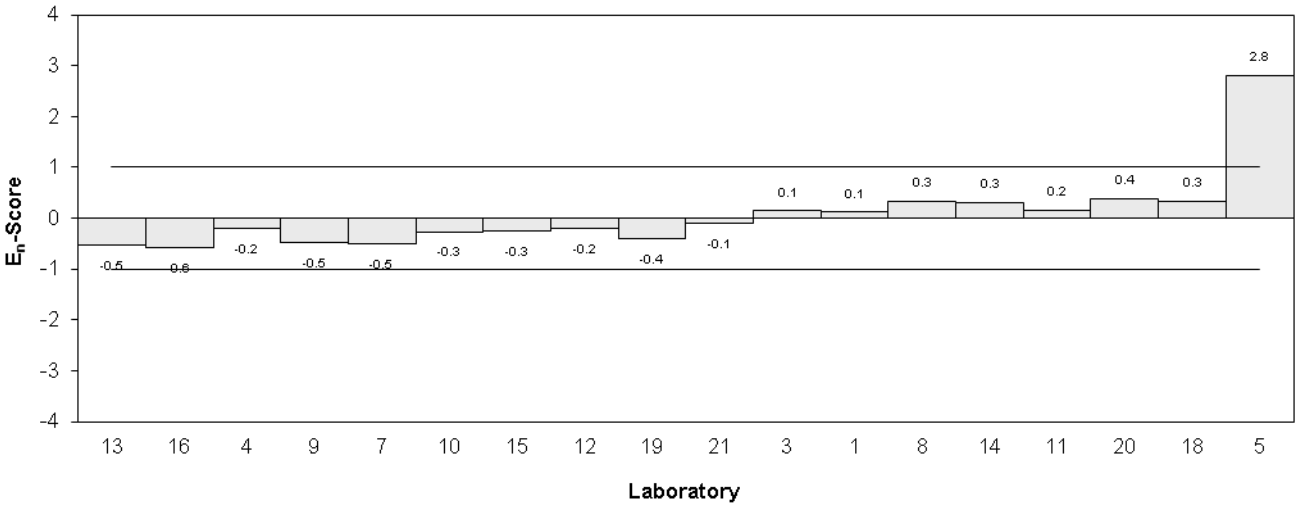

En-Scores: S1 - PFDA
En-Score
Laboratory
13 16 4 9 7 10 15 12 19 21 3 1 8 14 11 20 18 5
-0.5 -0.6 -0.2 -0.5 -0.5 -0.3 -0.3 -0.2 -0.4 -0.1 0.1 0.1 0.3 0.3 0.2 0.4 0.3 2.8

Figure 4

Table 9

Sample Details

Sample No.	S2
Matrix.	MilliQ water, 6:2 diPAP, 8:2 diPAP, PFDA, PFDS
Analyte.	PFDS
Units	µg/L

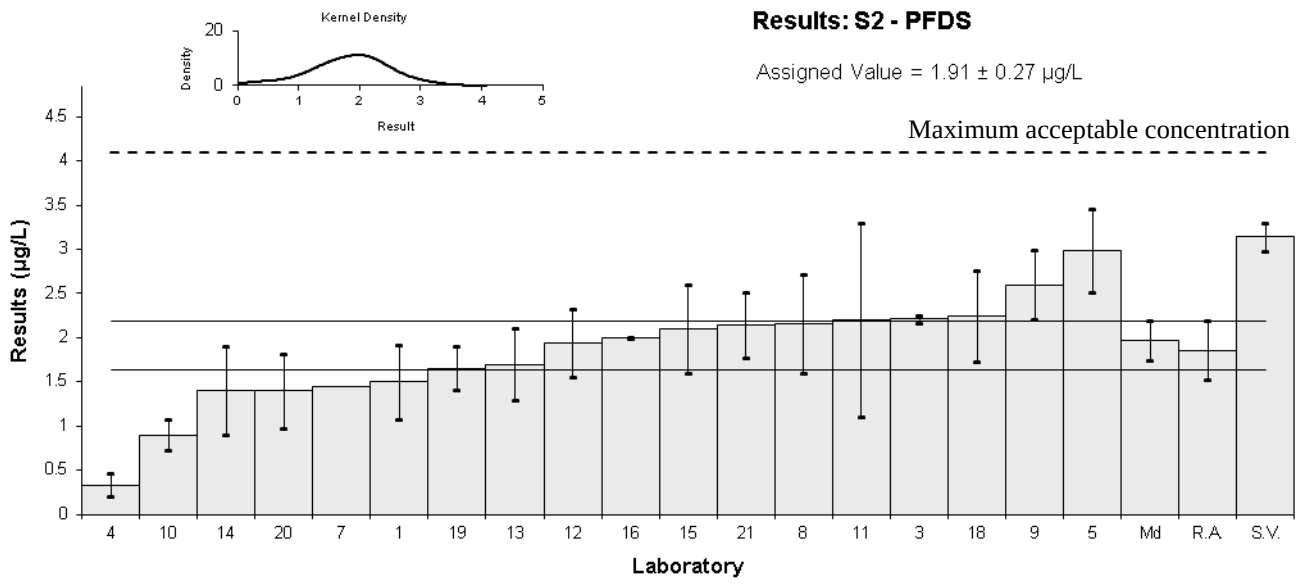
Participant Results

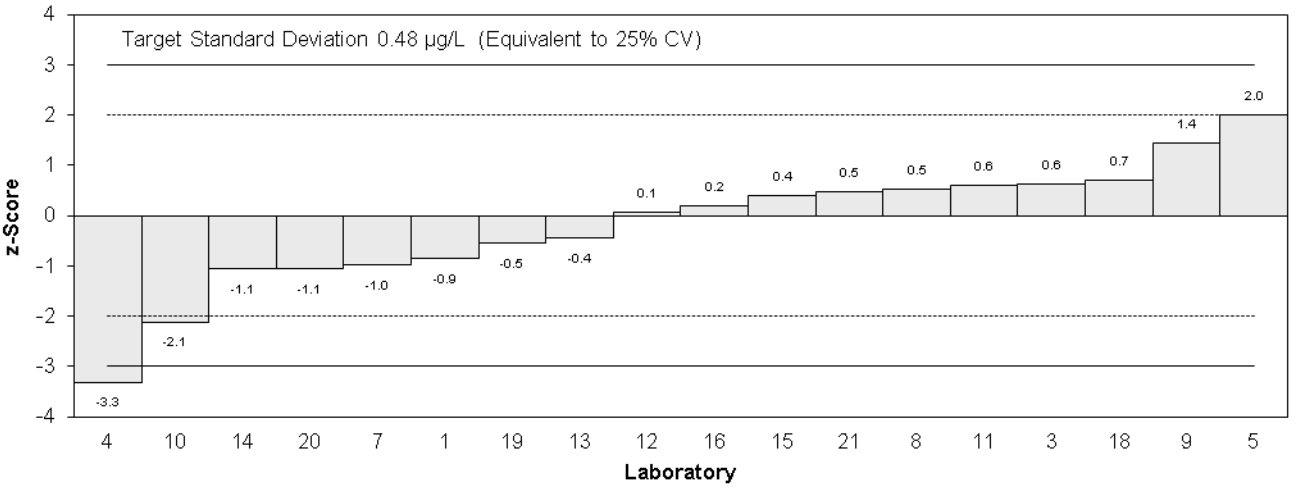
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	1.5	0.42	NR	-0.86	-0.82
3	2.21	0.0471	92.9	0.63	1.09
4	0.329	0.132	80	-3.31	-5.26
5*	2.99	0.47	NR	2.00	1.00
7	1.44	NR	98.21	-0.98	-1.74
8	2.16	0.56	NR	0.52	0.40
9	2.6	0.39	103	1.45	1.45
10	0.90	0.18	99	-2.12	-3.11
11	2.2	1.1	110	0.61	0.26
12	1.94	0.388	72	0.06	0.06
13	1.7	0.4	93	-0.44	-0.44
14	1.40	0.50	88	-1.07	-0.90
15	2.1	0.495	76	0.40	0.34
16	2.00	0.006	NR	0.19	0.33
18	2.245	0.512	120	0.70	0.58
19	1.651	0.24765	NR	-0.54	-0.71
20	1.4	0.42	NR	-1.07	-1.02
21	2.14	0.37	105	0.48	0.50

Statistics

Assigned Value**	1.91	0.27
Spike	3.14	0.16
Robust Average	1.86	0.33
Median	1.97	0.22
Mean	1.83	
N	18	
Max.	2.99	
Min.	0.329	
Robust SD	0.55	
Robust CV	30%	

*z-Score adjusted to 2 and E_n-score adjusted to 1 (see Section 6.3).
**Robust average excluding Laboratories 4, 5 and 10.


Kernel Density
Density
Result
Results: S2 - PFDS
Assigned Value = 1.91 ± 0.27 µg/L
Maximum acceptable concentration
Results (µg/L)
Laboratory
4 10 14 20 7 1 19 13 12 16 15 21 8 11 3 18 9 5 Md R.A S.V.


z-Scores: S2 - PFDS
Target Standard Deviation 0.48 µg/L (Equivalent to 25% CV)
z-Score
Laboratory
4 10 14 20 7 1 19 13 12 16 15 21 8 11 3 18 9 5
-3.3 -2.1 -1.1 -1.1 -1.0 -0.9 -0.5 -0.4 0.1 0.2 0.4 0.5 0.5 0.6 0.6 0.7 1.4 2.0

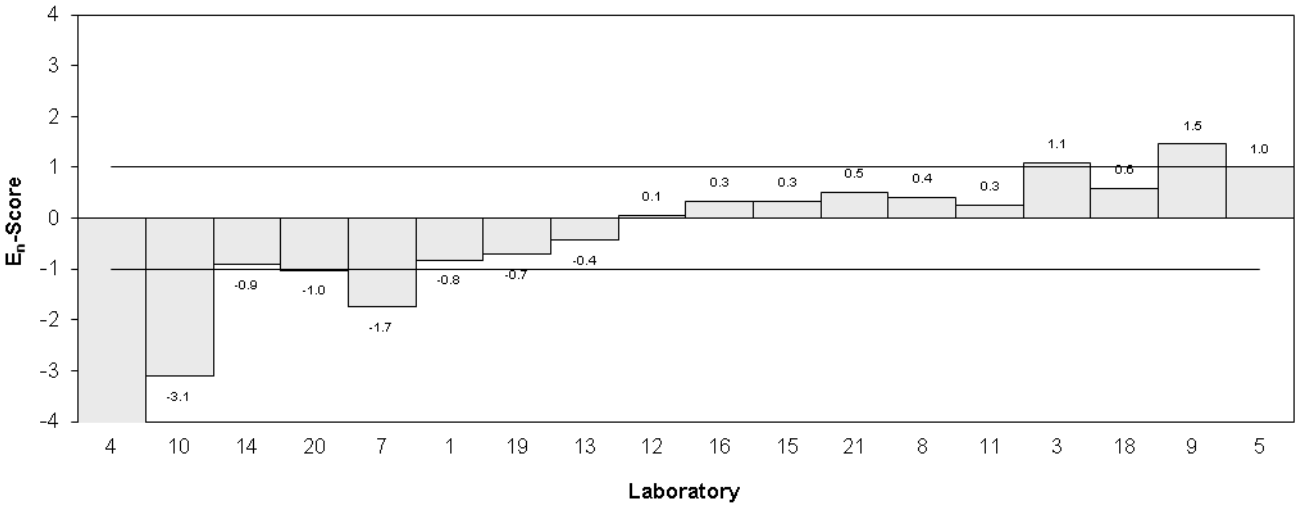

En-Scores: S2 - PFDS
En-Score
Laboratory
4 10 14 20 7 1 19 13 12 16 15 21 8 11 3 18 9 5
-3.1 -0.9 -1.0 -1.7 -0.8 -0.7 -0.4 0.1 0.3 0.3 0.5 0.4 0.3 1.1 0.6 1.5 1.0

Figure 5

Table 10

Sample Details

Sample No.	S2
Matrix.	MilliQ water, 6:2 diPAP, 8:2 diPAP, PFDA, PFDS
Analyte.	PFBA
Units	µg/L

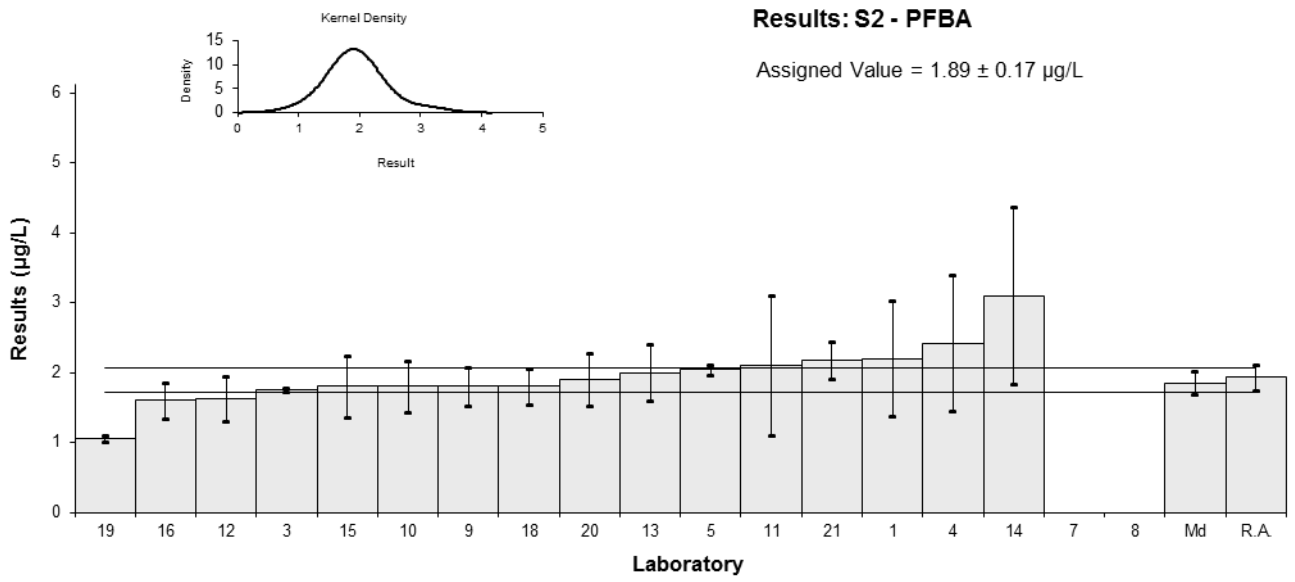
Participant Results

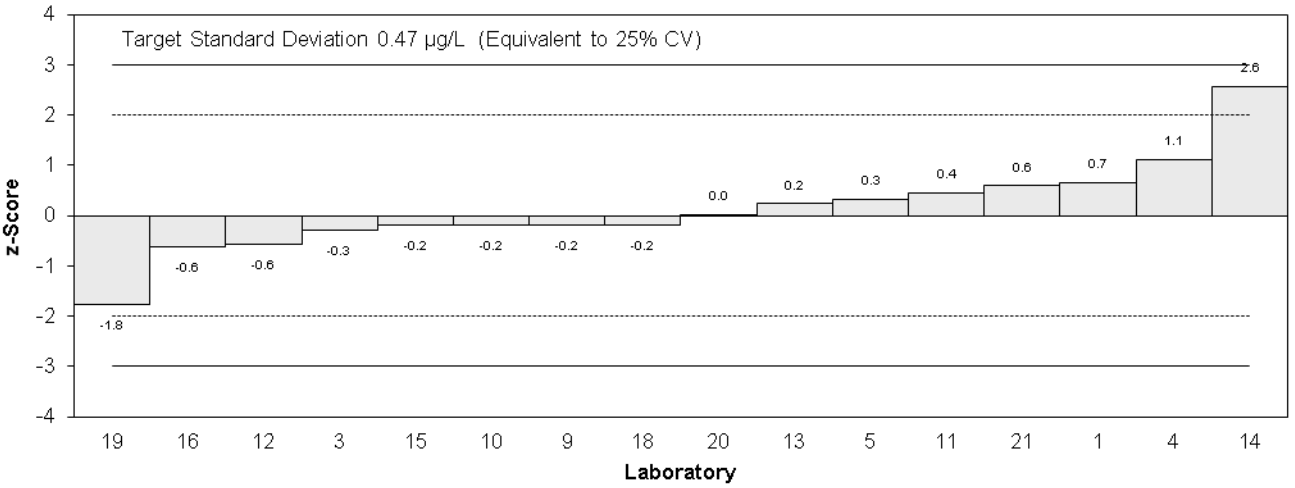
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	2.2	0.82	50	0.66	0.37
3	1.75	0.0236	99.4	-0.30	-0.82
4	2.419	0.968	62	1.12	0.54
5	2.04	0.08	63	0.32	0.80
7	<5.0	NR	96.00		
8	NR	NR	NR		
9	1.8	0.27	100	-0.19	-0.28
10	1.80	0.36	100	-0.19	-0.23
11	2.1	1.0	90	0.44	0.21
12	1.62	0.324	79	-0.57	-0.74
13	2.0	0.4	64	0.23	0.25
14	3.10	1.27	83	2.56	0.94
15	1.799	0.449	85	-0.19	-0.19
16	1.60	0.254	104	-0.61	-0.95
18	1.801	0.262	11	-0.19	-0.28
19	1.057	0.04228	110	-1.76	-4.76
20	1.9	0.38	NR	0.02	0.02
21	2.17	0.27	96	0.59	0.88

Statistics

Assigned Value*	1.89	0.17
Robust Average	1.93	0.19
Median	1.85	0.17
Mean	1.95	
N	16	
Max.	3.1	
Min.	1.057	
Robust SD	0.31	
Robust CV	16%	

* Robust average excluding Laboratory 14.


Kernel Density
Density
Result
Results: S2 - PFBA
Assigned Value = 1.89 ± 0.17 µg/L
Results (µg/L)
19 16 12 3 15 10 9 18 20 13 5 11 21 1 4 14 7 8 Md R.A
Laboratory


z-Scores: S2 - PFBA
Target Standard Deviation 0.47 µg/L (Equivalent to 25% CV)
z-Score
-1.8 -0.6 -0.6 -0.3 -0.2 -0.2 -0.2 -0.2 0.0 0.2 0.3 0.4 0.6 0.7 1.1 2.6
19 16 12 3 15 10 9 18 20 13 5 11 21 1 4 14
Laboratory

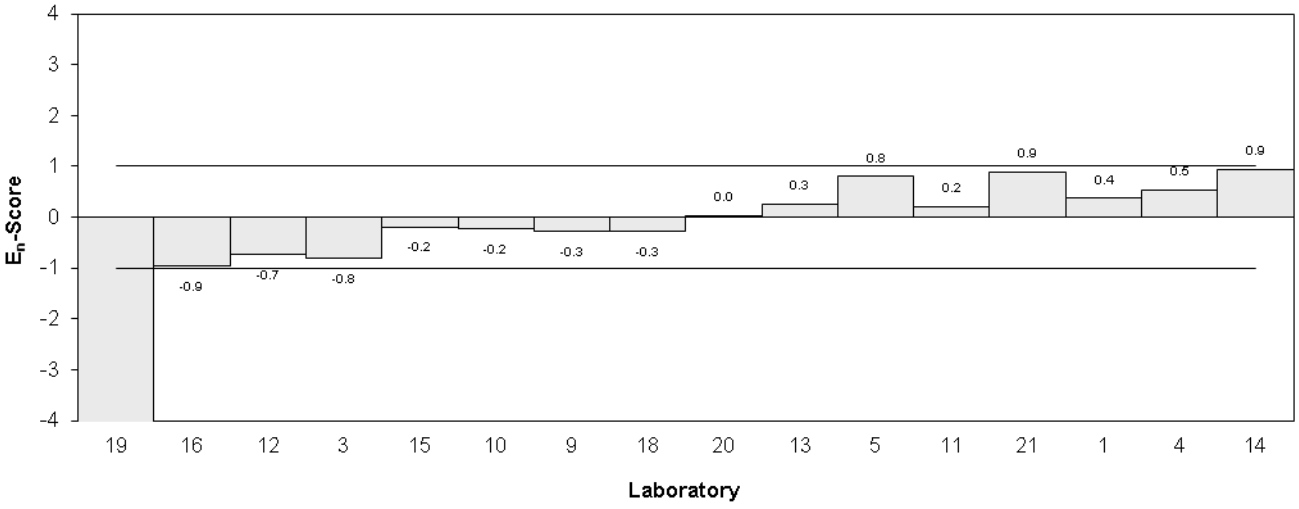

En-Scores: S2 - PFBA
En-Score
-0.9 -0.7 -0.8 -0.2 -0.2 -0.3 -0.3 0.0 0.3 0.8 0.2 0.9 0.4 0.5 0.9
19 16 12 3 15 10 9 18 20 13 5 11 21 1 4 14
Laboratory

Figure 6

Table 11

Sample Details

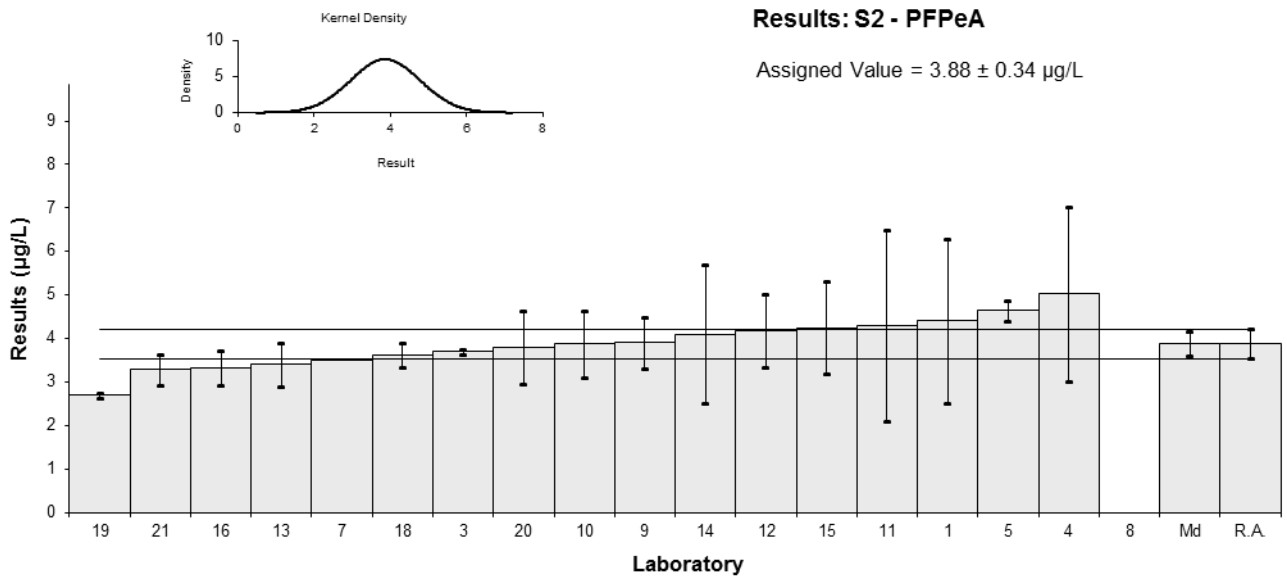
Sample No.	S2
Matrix.	MilliQ water, 6:2 diPAP, 8:2 diPAP, PFDA, PFDS
Analyte.	PFPeA
Units	µg/L

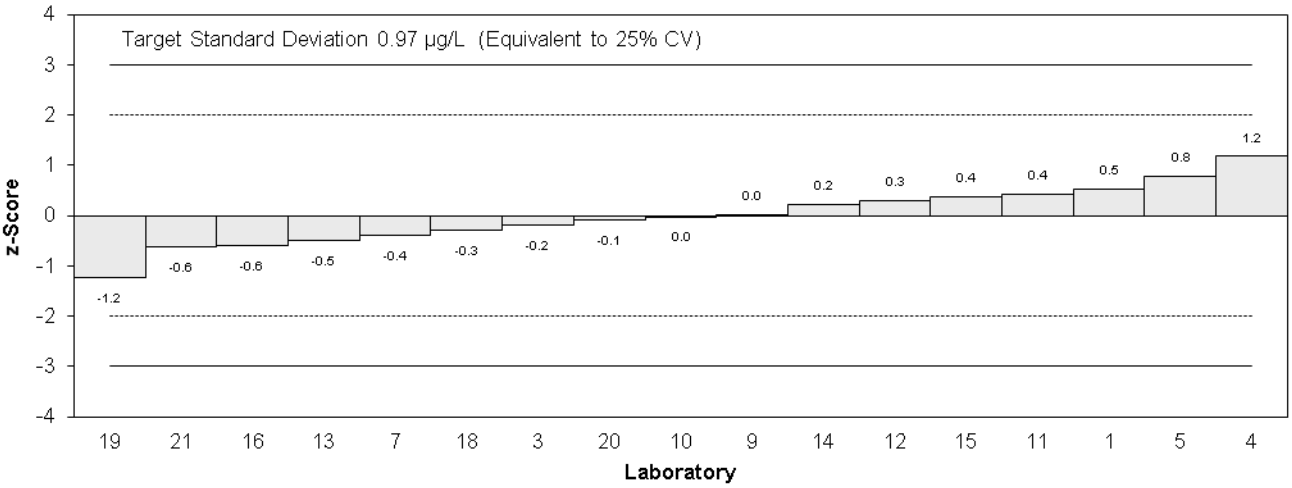
Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	4.4	1.9	53	0.54	0.27
3	3.70	0.0596	106	-0.19	-0.52
4	5.020	2.008	40	1.18	0.56
5	4.64	0.23	65	0.78	1.85
7	3.5	NR	93.62	-0.39	-1.12
8	NR	NR	NR		
9	3.9	0.59	83	0.02	0.03
10	3.87	0.77	88	-0.01	-0.01
11	4.3	2.2	100	0.43	0.19
12	4.17	0.834	78	0.30	0.32
13	3.4	0.5	63	-0.49	-0.79
14	4.10	1.60	112	0.23	0.13
15	4.243	1.061	96	0.37	0.33
16	3.31	0.401	113	-0.59	-1.08
18	3.603	0.281	60	-0.29	-0.63
19	2.689	0.05378	123	-1.23	-3.46
20	3.8	0.836	NR	-0.08	-0.09
21	3.28	0.35	87	-0.62	-1.23

Statistics

Assigned Value	3.88	0.34
Robust Average	3.88	0.34
Median	3.87	0.28
Mean	3.88	
N	17	
Max.	5.02	
Min.	2.689	
Robust SD	0.56	
Robust CV	14%	


Kernel Density
Density
Result
Results: S2 - PFPeA
Assigned Value = 3.88 ± 0.34 µg/L
Results (µg/L)
19 21 16 13 7 18 3 20 10 9 14 12 15 11 1 5 4 8 Md R.A
Laboratory


z-Scores: S2 - PFPeA
Target Standard Deviation 0.97 µg/L (Equivalent to 25% CV)
z-Score
-1.2 -0.6 -0.6 -0.5 -0.4 -0.3 -0.2 -0.1 0.0 0.0 0.2 0.3 0.4 0.4 0.5 0.8 1.2
19 21 16 13 7 18 3 20 10 9 14 12 15 11 1 5 4
Laboratory

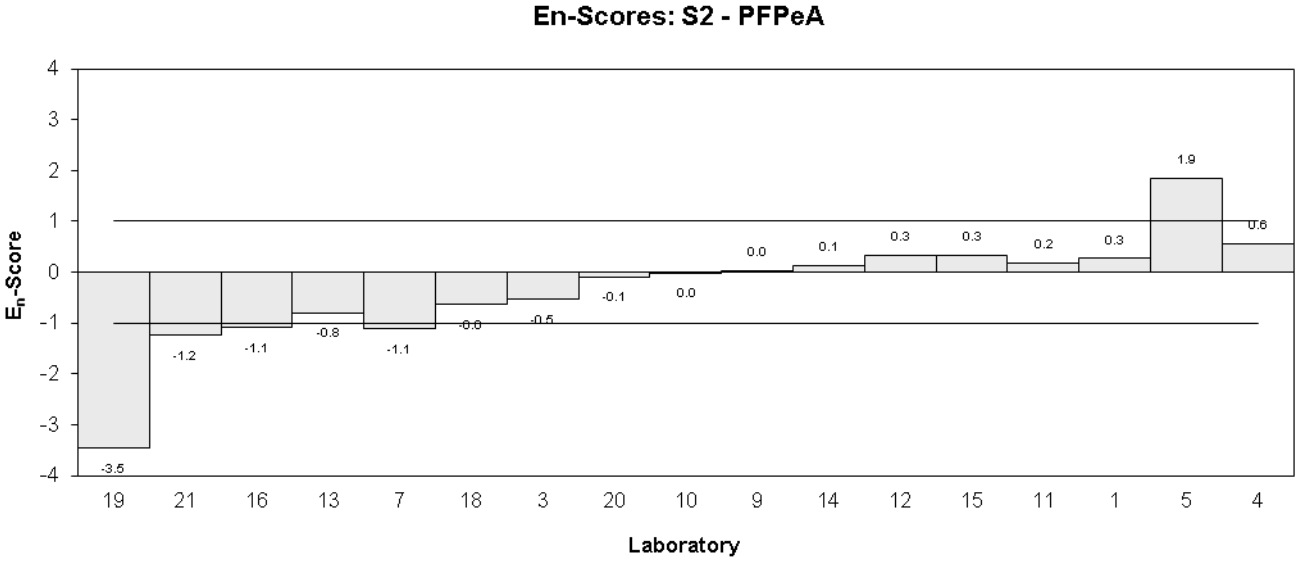

En-Scores: S2 - PFPeA
En-Score
-3.5 -1.2 -1.1 -0.8 -1.1 -0.6 -0.5 -0.1 0.0 0.0 0.1 0.3 0.3 0.2 0.3 1.9 0.6
19 21 16 13 7 18 3 20 10 9 14 12 15 11 1 5 4
Laboratory

Figure 7

Table 12

Sample Details

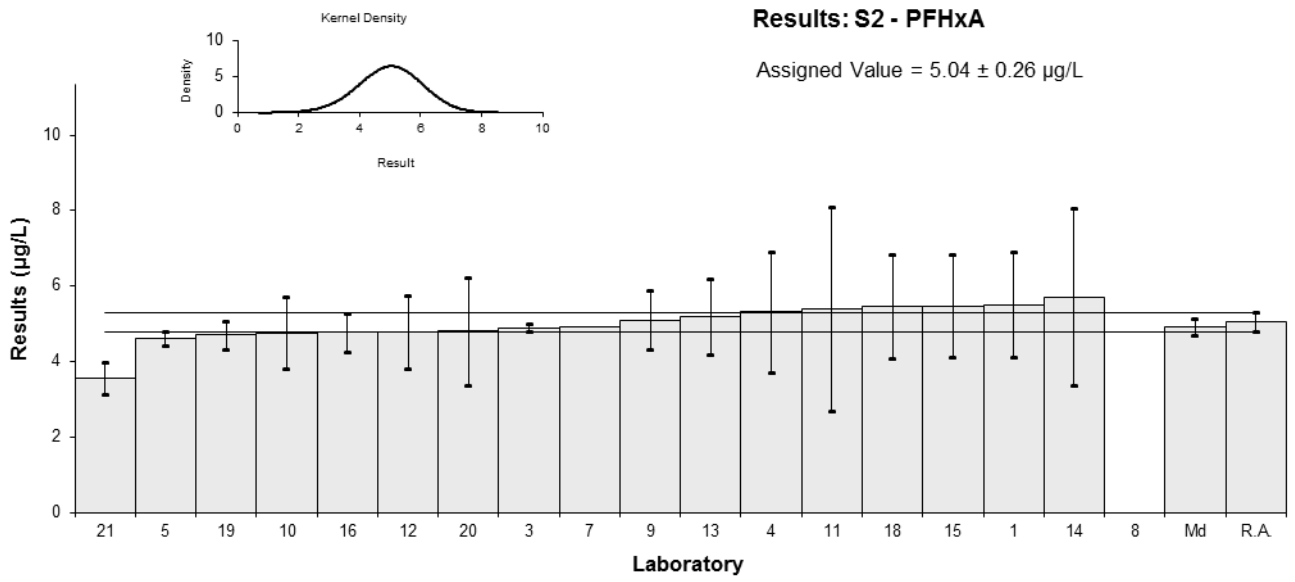
Sample No.	S2
Matrix.	MilliQ water, 6:2 diPAP, 8:2 diPAP, PFDA, PFDS
Analyte.	PFHxA
Units	µg/L

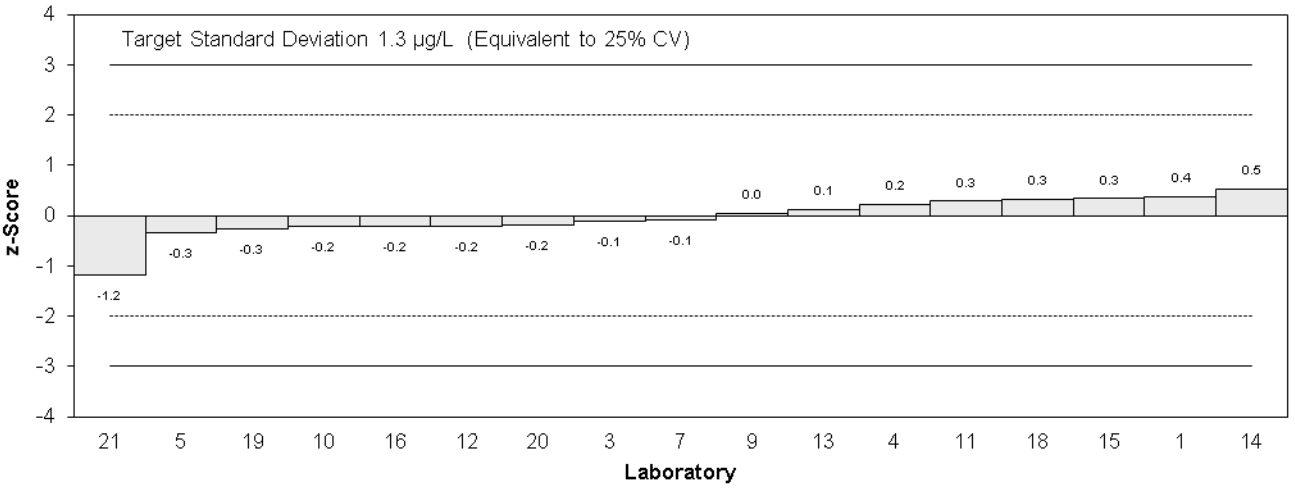
Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	5.5	1.4	52	0.37	0.32
3	4.89	0.0988	97.6	-0.12	-0.54
4	5.310	1.593	40	0.21	0.17
5	4.61	0.19	64	-0.34	-1.34
7	4.92	NR	95.98	-0.10	-0.46
8	NR	NR	NR		
9	5.1	0.77	97	0.05	0.07
10	4.76	0.95	94	-0.22	-0.28
11	5.4	2.7	90	0.29	0.13
12	4.78	0.956	75	-0.21	-0.26
13	5.2	1	73	0.13	0.15
14	5.71	2.34	77	0.53	0.28
15	5.462	1.36	97	0.33	0.30
16	4.77	0.513	105	-0.21	-0.47
18	5.452	1.385	90	0.33	0.29
19	4.701	0.37608	94	-0.27	-0.74
20	4.8	1.44	NR	-0.19	-0.16
21	3.56	0.42	95	-1.17	-3.00

Statistics

Assigned Value	5.04	0.26
Robust Average	5.04	0.26
Median	4.92	0.21
Mean	5.00	
N	17	
Max.	5.71	
Min.	3.56	
Robust SD	0.42	
Robust CV	8.3%	


Kernel Density
Density
Result
Results: S2 - PFHxA
Assigned Value = 5.04 ± 0.26 µg/L
Results (µg/L)
Laboratory
21 5 19 10 16 12 20 3 7 9 13 4 11 18 15 1 14 8 Md R.A.


z-Scores: S2 - PFHxA
Target Standard Deviation 1.3 µg/L (Equivalent to 25% CV)
z-Score
Laboratory
21 5 19 10 16 12 20 3 7 9 13 4 11 18 15 1 14
-1.2 -0.3 -0.3 -0.2 -0.2 -0.2 -0.2 -0.1 -0.1 0.0 0.1 0.2 0.3 0.3 0.3 0.4 0.5

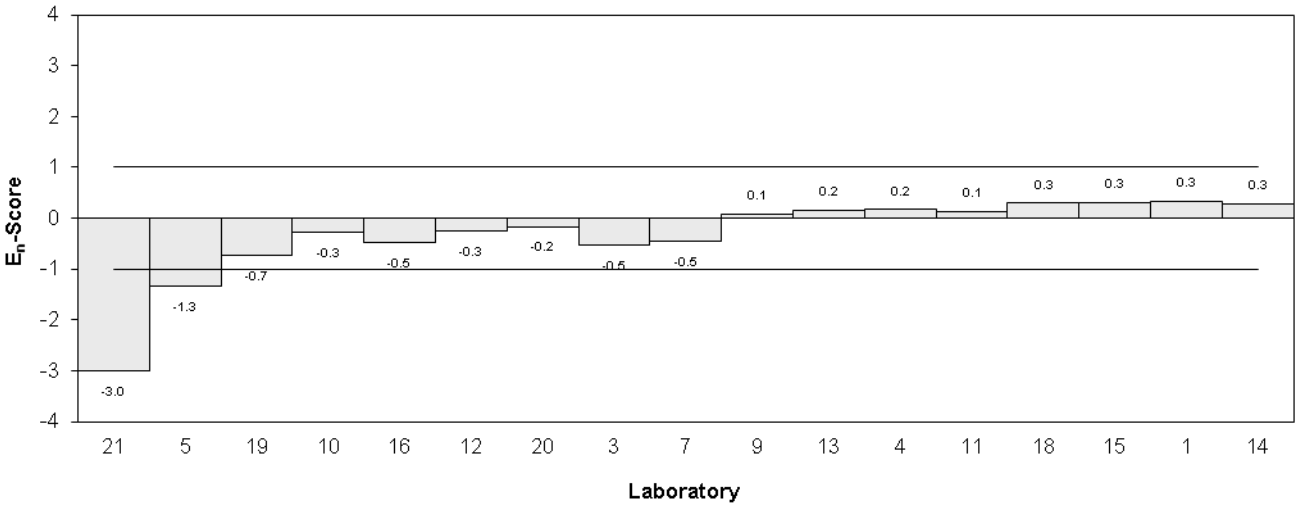

En-Scores: S2 - PFHxA
En-Score
Laboratory
21 5 19 10 16 12 20 3 7 9 13 4 11 18 15 1 14
-3.0 -1.3 -0.7 -0.3 -0.5 -0.3 -0.2 -0.5 -0.5 0.1 0.2 0.2 0.1 0.3 0.3 0.3 0.3

Figure 8

Table 13

Sample Details

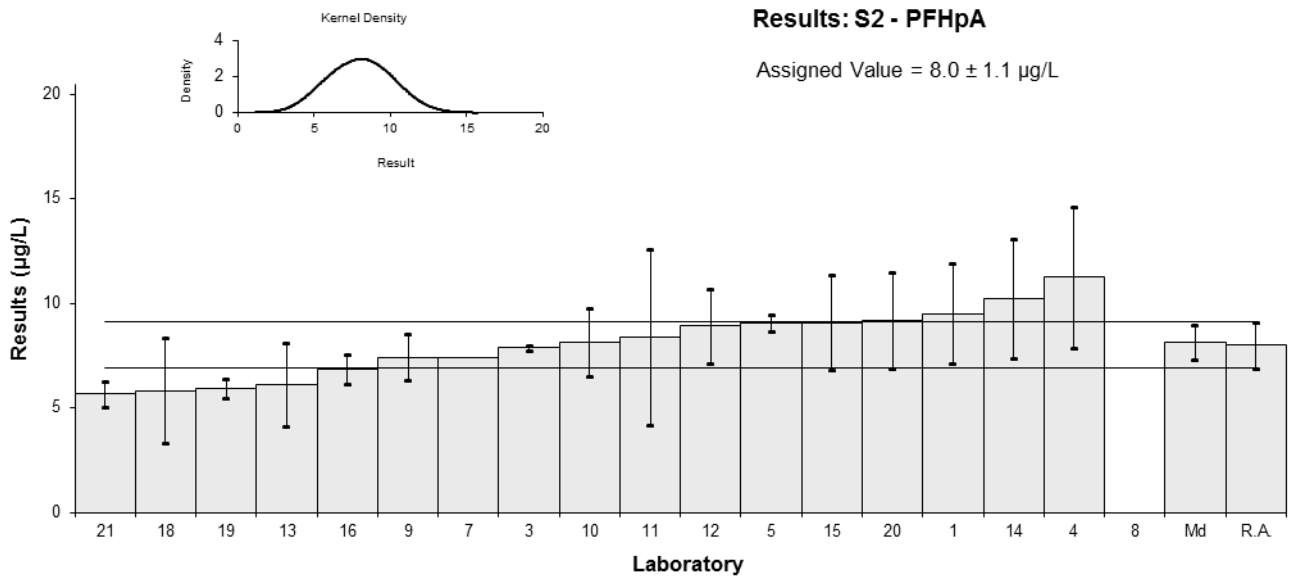
Sample No.	S2
Matrix.	MilliQ water, 6:2 diPAP, 8:2 diPAP, PFDA, PFDS
Analyte.	PFHpA
Units	µg/L

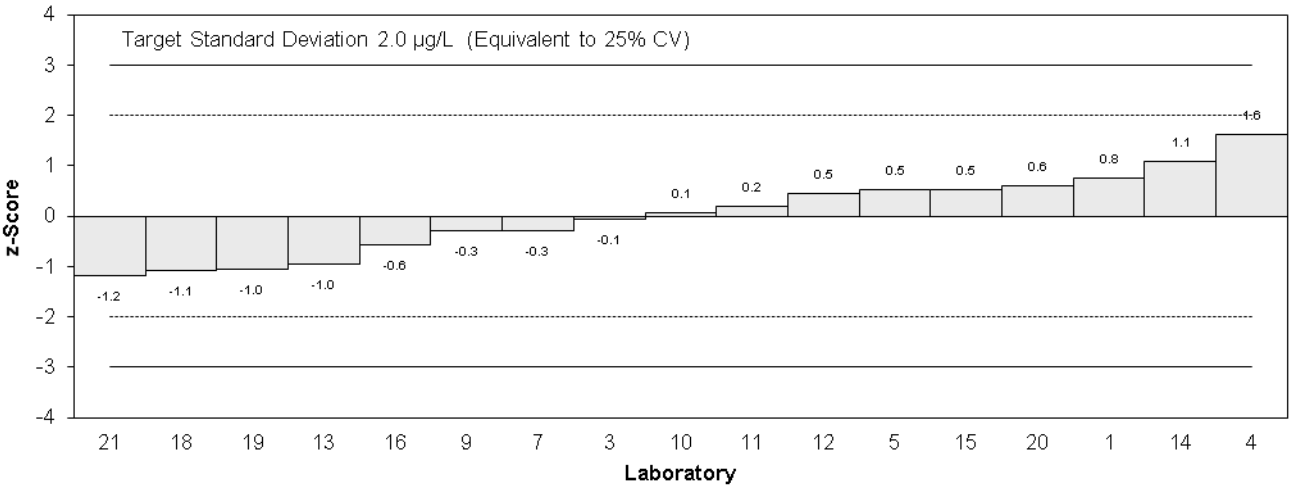
Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	9.5	2.4	53	0.75	0.57
3	7.86	0.123	95.8	-0.07	-0.13
4	11.24	3.372	40	1.62	0.91
5	9.06	0.40	57	0.53	0.91
7	7.42	NR	95.98	-0.29	-0.53
8	NR	NR	NR		
9	7.4	1.1	106	-0.30	-0.39
10	8.12	1.62	88	0.06	0.06
11	8.4	4.2	100	0.20	0.09
12	8.91	1.782	76	0.46	0.43
13	6.1	2	70	-0.95	-0.83
14	10.19	2.85	78	1.10	0.72
15	9.067	2.27	85	0.53	0.42
16	6.84	0.730	103	-0.58	-0.88
18	5.824	2.519	92	-1.09	-0.79
19	5.909	0.47272	87	-1.05	-1.75
20	9.2	2.3	NR	0.60	0.47
21	5.65	0.63	97	-1.18	-1.85

Statistics

Assigned Value	8.0	1.1
Robust Average	8.0	1.1
Median	8.12	0.82
Mean	8.04	
N	17	
Max.	11.24	
Min.	5.65	
Robust SD	1.8	
Robust CV	22%	


Kernel Density
Density
Result
Results: S2 - PFHpA
Assigned Value = 8.0 ± 1.1 µg/L
Results (µg/L)
Laboratory
21 18 19 13 16 9 7 3 10 11 12 5 15 20 1 14 4 8 Md R.A


z-Scores: S2 - PFHpA
Target Standard Deviation 2.0 µg/L (Equivalent to 25% CV)
z-Score
-1.2 -1.1 -1.0 -1.0 -0.6 -0.3 -0.3 -0.1 0.1 0.2 0.5 0.5 0.5 0.6 0.8 1.1 1.6
Laboratory
21 18 19 13 16 9 7 3 10 11 12 5 15 20 1 14 4

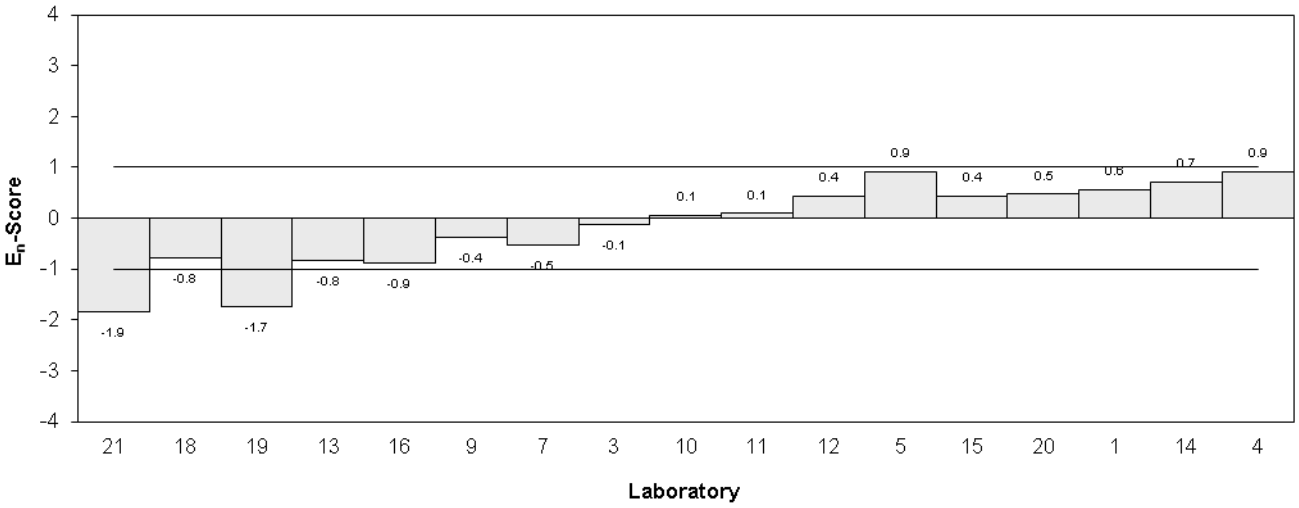

En-Scores: S2 - PFHpA
En-Score
-1.9 -0.8 -1.7 -0.8 -0.9 -0.4 -0.5 -0.1 0.1 0.1 0.4 0.9 0.4 0.5 0.6 0.7 0.9
Laboratory
21 18 19 13 16 9 7 3 10 11 12 5 15 20 1 14 4

Figure 9

Table 14

Sample Details

Sample No.	S2
Matrix.	MilliQ water, 6:2 diPAP, 8:2 diPAP, PFDA, PFDS
Analyte.	PFOA
Units	µg/L

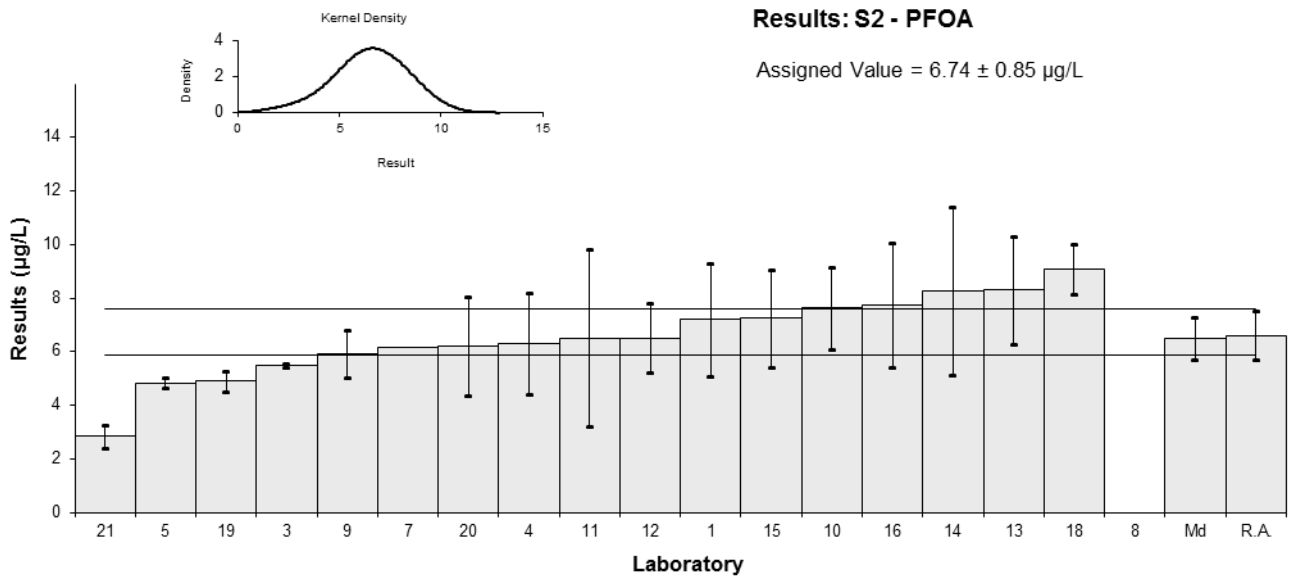
Participant Results

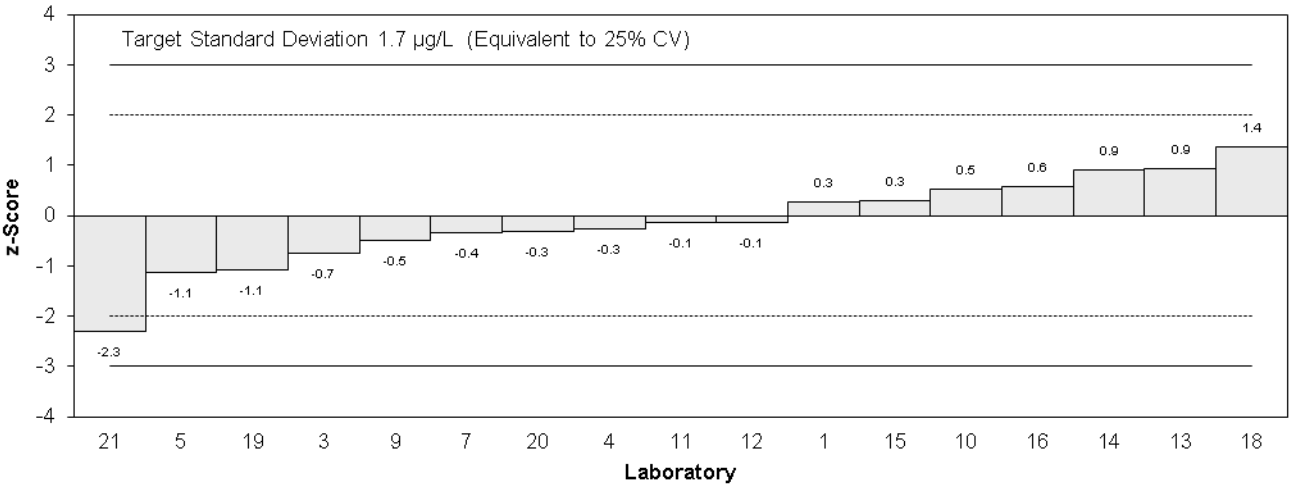
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	7.2	2.1	56	0.27	0.20
3	5.48	0.0833	76.7	-0.75	-1.48
4	6.307	1.892	62	-0.26	-0.21
5	4.84	0.19	60	-1.13	-2.18
7	6.15	NR	95.98	-0.35	-0.69
8	NR	NR	87.9		
9	5.9	0.89	102	-0.50	-0.68
10	7.62	1.52	99	0.52	0.51
11	6.5	3.3	90	-0.14	-0.07
12	6.50	1.3	71	-0.14	-0.15
13	8.3	2	64	0.93	0.72
14	8.27	3.14	77	0.91	0.47
15	7.238	1.81	89	0.30	0.25
16	7.73	2.335	99	0.59	0.40
18	9.06	0.931	103	1.38	1.84
19	4.903	0.39224	98	-1.09	-1.96
20	6.2	1.86	NR	-0.32	-0.26
21	2.84	0.42	101	-2.31	-4.11

Statistics

Assigned Value*	6.74	0.85
Robust Average	6.61	0.89
Median	6.50	0.78
Mean	6.53	
N	17	
Max.	9.06	
Min.	2.84	
Robust SD	1.5	
Robust CV	23%	

*Robust Average excluding Lab 21.


Kernel Density
Density
Result
Results: S2 - PFOA
Assigned Value = 6.74 ± 0.85 µg/L
Results (µg/L)
Laboratory
21 5 19 3 9 7 20 4 11 12 1 15 10 16 14 13 18 8 Md R.A.


z-Scores: S2 - PFOA
Target Standard Deviation 1.7 µg/L (Equivalent to 25% CV)
z-Score
Laboratory
21 5 19 3 9 7 20 4 11 12 1 15 10 16 14 13 18
-2.3 -1.1 -1.1 -0.7 -0.5 -0.4 -0.3 -0.3 -0.1 -0.1 0.3 0.3 0.5 0.6 0.9 0.9 1.4

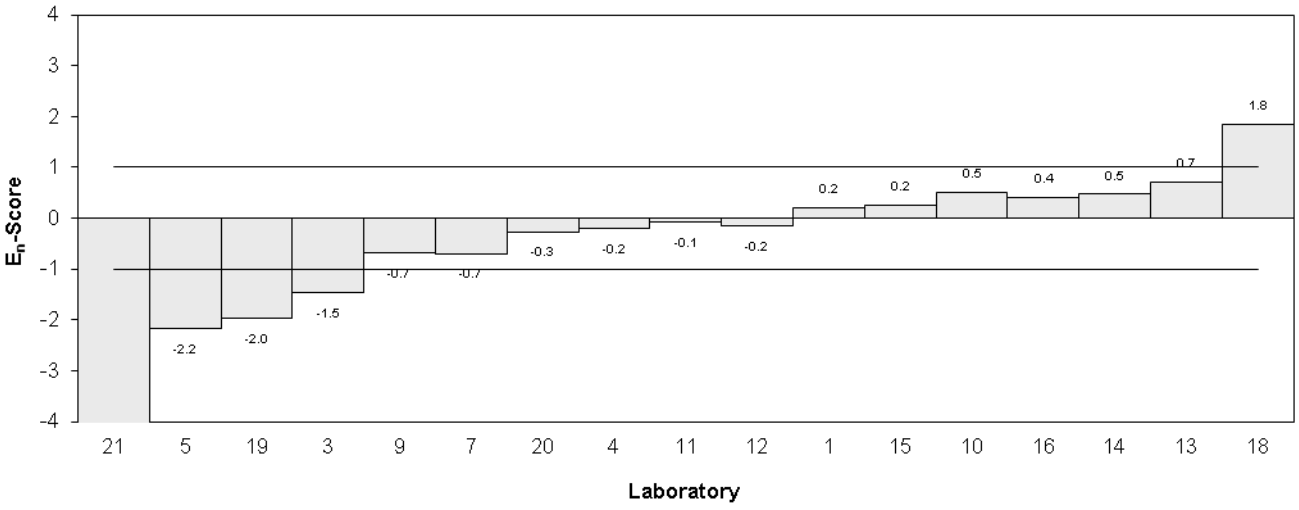

En-Scores: S2 - PFOA
En-Score
Laboratory
21 5 19 3 9 7 20 4 11 12 1 15 10 16 14 13 18
-2.2 -2.0 -1.5 -0.7 -0.7 -0.3 -0.2 -0.1 -0.2 0.2 0.2 0.5 0.4 0.5 0.7 1.8

Figure 10

Table 15

Sample Details

Sample No.	S2
Matrix.	MilliQ water, 6:2 diPAP, 8:2 diPAP, PFDA, PFDS
Analyte.	PFNA
Units	µg/L

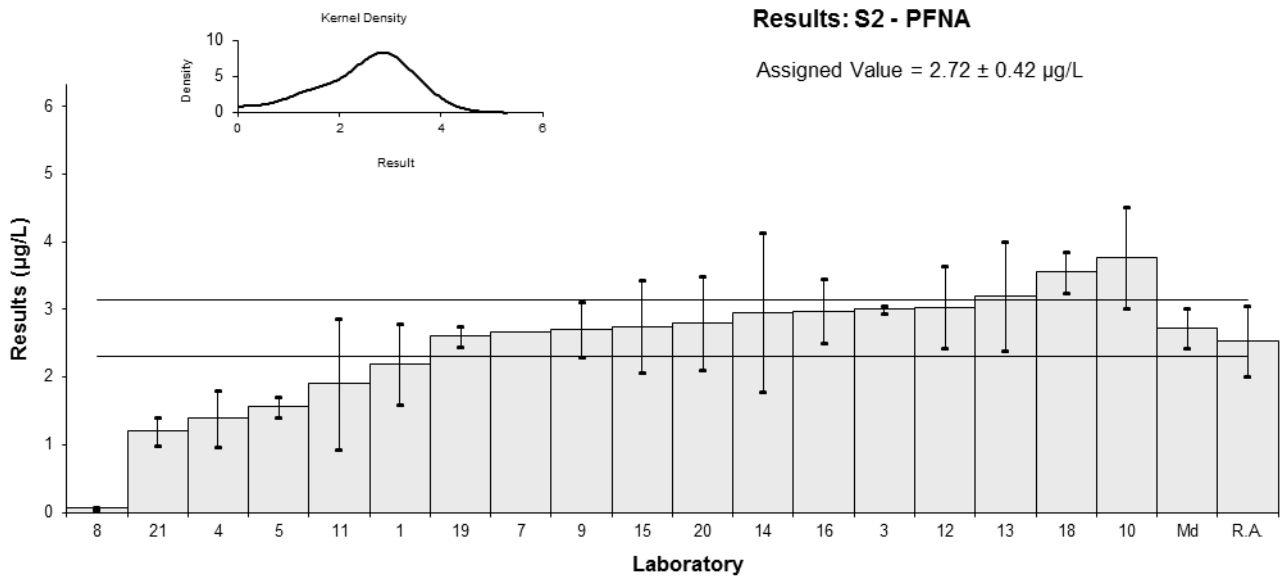
Participant Results

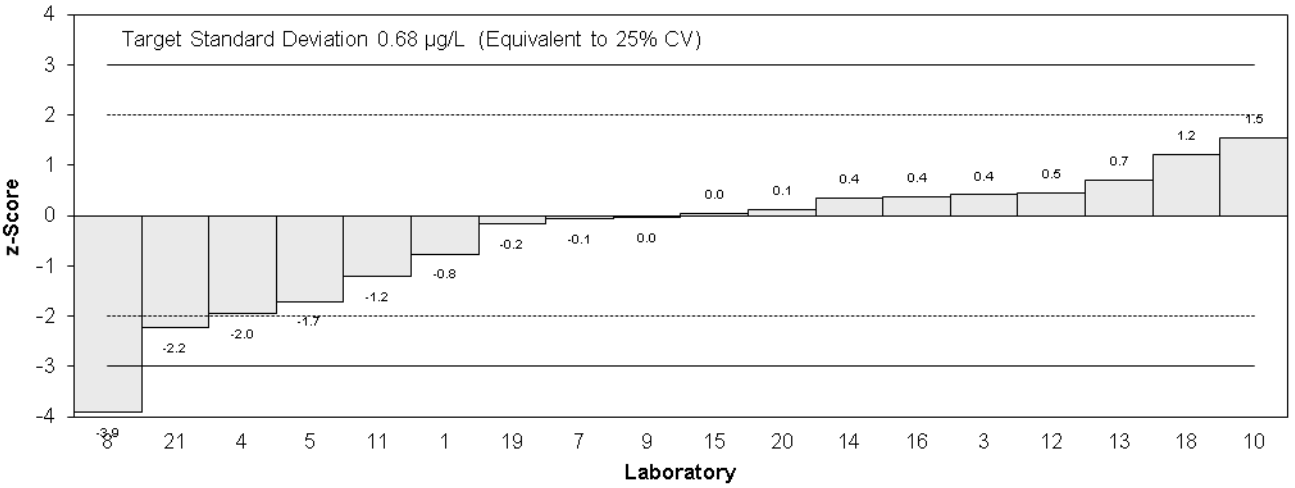
Lab Code	Result	Uncertainty	Recovery	z-Score	En-Score
1	2.2	0.60	65	-0.76	-0.71
3	3.00	0.0618	96.3	0.41	0.66
4	1.391	0.417	80	-1.95	-2.25
5	1.56	0.15	68	-1.71	-2.60
7	2.67	NR	98.21	-0.07	-0.12
8	0.061	0.013	NR	-3.91	-6.33
9	2.7	0.41	98	-0.03	-0.03
10	3.77	0.75	89	1.54	1.22
11	1.9	0.96	90	-1.21	-0.78
12	3.03	0.606	73	0.46	0.42
13	3.2	0.8	71	0.71	0.53
14	2.96	1.18	80	0.35	0.19
15	2.748	0.686	84	0.04	0.03
16	2.98	0.479	109	0.38	0.41
18	3.553	0.298	122	1.23	1.62
19	2.604	0.15624	83	-0.17	-0.26
20	2.8	0.7	NR	0.12	0.10
21	1.20	0.21	104	-2.24	-3.24

Statistics

Assigned Value*	2.72	0.42
Robust Average	2.53	0.52
Median	2.72	0.29
Mean	2.46	
N	18	
Max.	3.77	
Min.	0.061	
Robust SD	0.88	
Robust CV	35%	

*Robust Average excluding Laboratories 8 and 21.


Kernel Density
Density
Result
Results: S2 - PFNA
Assigned Value = 2.72 ± 0.42 µg/L
Results (µg/L)
Laboratory
8 21 4 5 11 1 19 7 9 15 20 14 16 3 12 13 18 10 Md R.A.


z-Scores: S2 - PFNA
Target Standard Deviation 0.68 µg/L (Equivalent to 25% CV)
z-Score
Laboratory
8 21 4 5 11 1 19 7 9 15 20 14 16 3 12 13 18 10
-3.9 -2.2 -2.0 -1.7 -1.2 -0.8 -0.2 -0.1 0.0 0.0 0.1 0.4 0.4 0.4 0.5 0.7 1.2 1.5

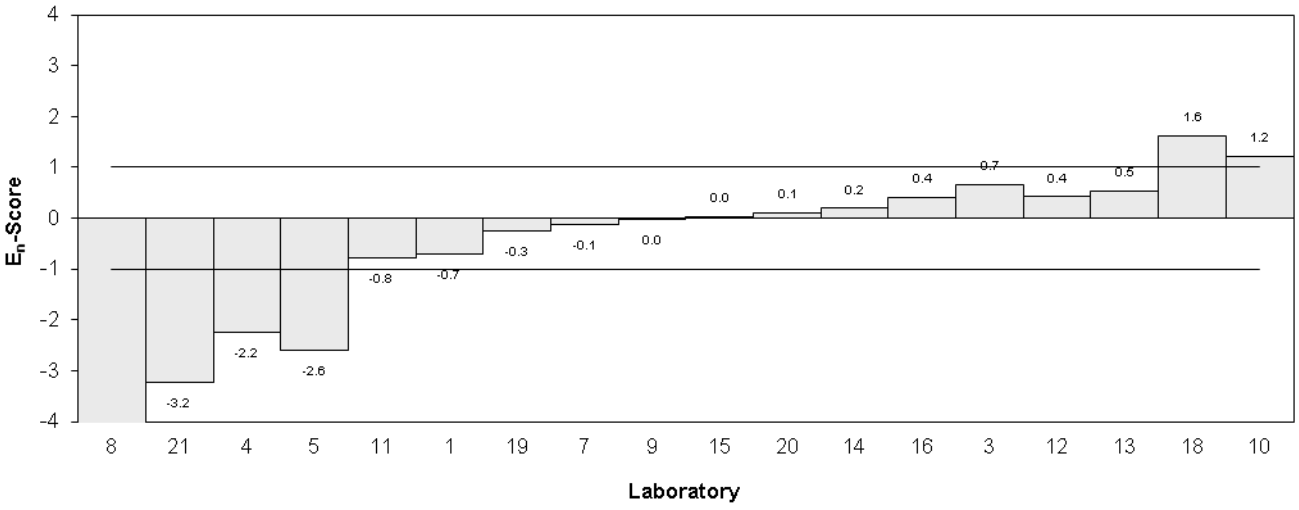

En-Scores: S2 - PFNA
En-Score
Laboratory
8 21 4 5 11 1 19 7 9 15 20 14 16 3 12 13 18 10
-3.2 -2.2 -2.6 -0.8 -0.7 -0.3 -0.1 0.0 0.0 0.1 0.2 0.4 0.7 0.4 0.5 1.6 1.2

Figure 11

Table 16

Sample Details

Sample No.	S2
Matrix.	MilliQ water, 6:2 diPAP, 8:2 diPAP, PFDA, PFDS
Analyte.	PFDA
Units	µg/L

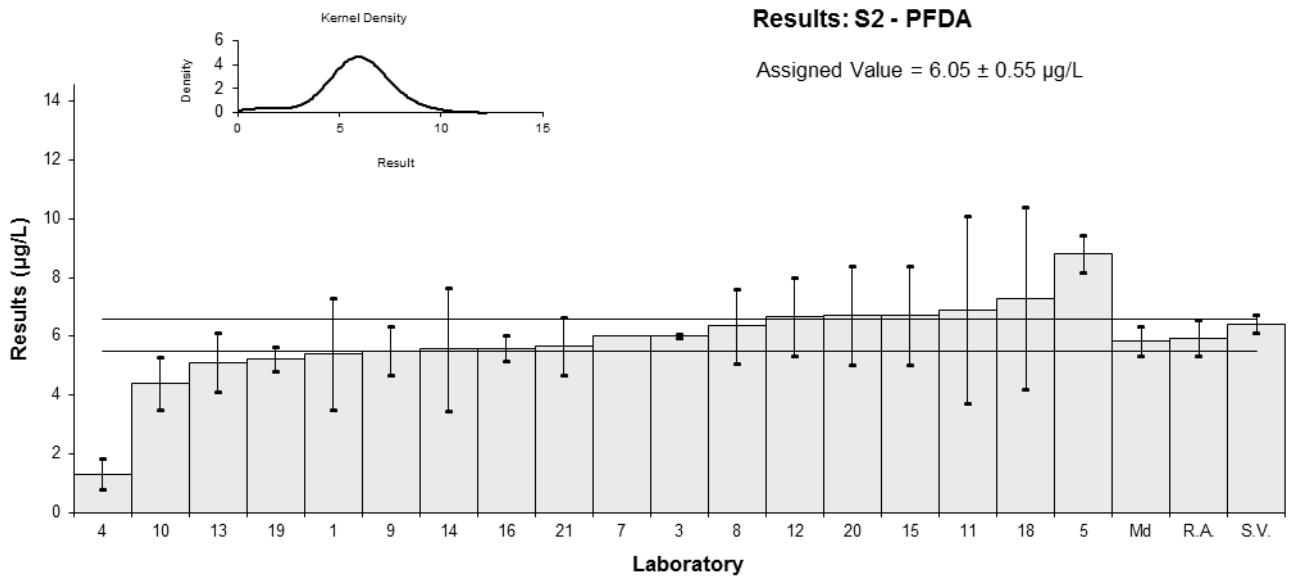
Participant Results

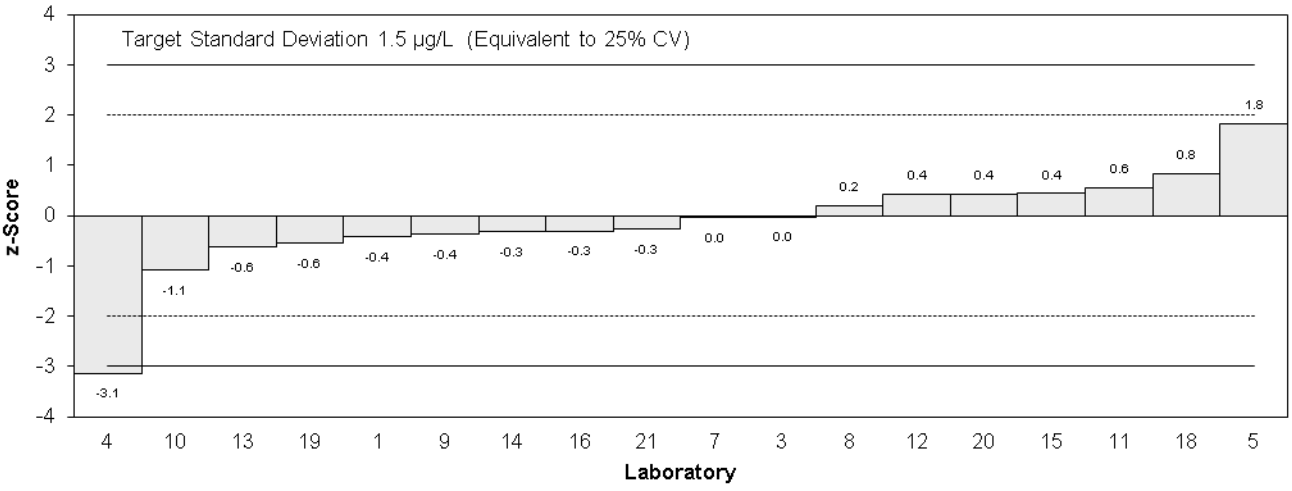
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	5.4	1.9	67	-0.43	-0.33
3	6.02	0.0765	79.7	-0.02	-0.05
4	1.310	0.524	86	-3.13	-6.24
5	8.81	0.63	56	1.82	3.30
7	6	NR	98.21	-0.03	-0.09
8	6.35	1.27	NR	0.20	0.22
9	5.5	0.83	111	-0.36	-0.55
10	4.40	0.88	106	-1.09	-1.59
11	6.9	3.2	100	0.56	0.26
12	6.68	1.34	75	0.42	0.43
13	5.1	1	78	-0.63	-0.83
14	5.56	2.11	78	-0.32	-0.22
15	6.713	1.67	83	0.44	0.38
16	5.59	0.429	109	-0.30	-0.66
18	7.301	3.110	102	0.83	0.40
19	5.210	0.4168	76	-0.56	-1.22
20	6.7	1.675	NR	0.43	0.37
21	5.66	0.97	111	-0.26	-0.35

Statistics

Assigned Value*	6.05	0.55
Spike	6.42	0.32
Robust Average	5.94	0.60
Median	5.83	0.50
Mean	5.84	
N	18	
Max.	8.81	
Min.	1.31	
Robust SD	1.0	
Robust CV	17%	

* Robust Average excluding Laboratory 4.


Kernel Density
Density
Result
Results: S2 - PFDA
Assigned Value = 6.05 ± 0.55 µg/L
Results (µg/L)
Laboratory
4 10 13 19 1 9 14 16 21 7 3 8 12 20 15 11 18 5 Md R.A. S.V.


z-Scores: S2 - PFDA
Target Standard Deviation 1.5 µg/L (Equivalent to 25% CV)
z-Score
Laboratory
4 10 13 19 1 9 14 16 21 7 3 8 12 20 15 11 18 5
-3.1 -1.1 -0.6 -0.6 -0.4 -0.4 -0.3 -0.3 -0.3 0.0 0.0 0.2 0.4 0.4 0.4 0.6 0.8 1.8

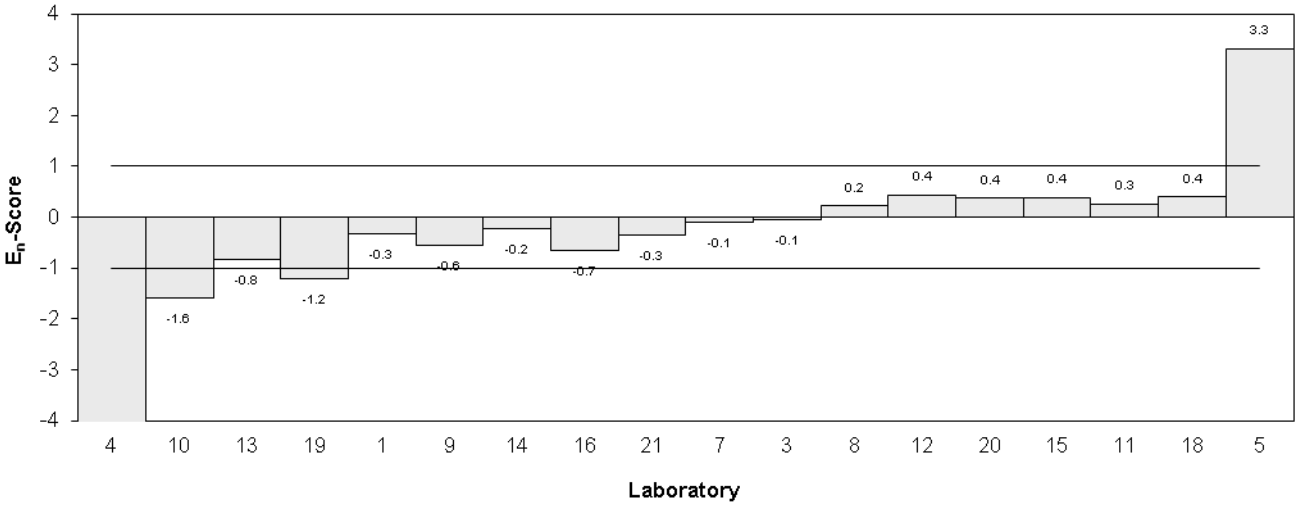

En-Scores: S2 - PFDA
En-Score
Laboratory
4 10 13 19 1 9 14 16 21 7 3 8 12 20 15 11 18 5
-1.6 -0.8 -1.2 -0.3 -0.6 -0.2 -0.7 -0.3 -0.1 -0.1 0.2 0.4 0.4 0.4 0.3 0.4 3.3

Figure 12

Table 17

Sample Details

Sample No.	S2
Matrix.	MilliQ water, 6:2 diPAP, 8:2 diPAP, PFDA, PFDS
Analyte.	Total PFCA
Units	µg/L

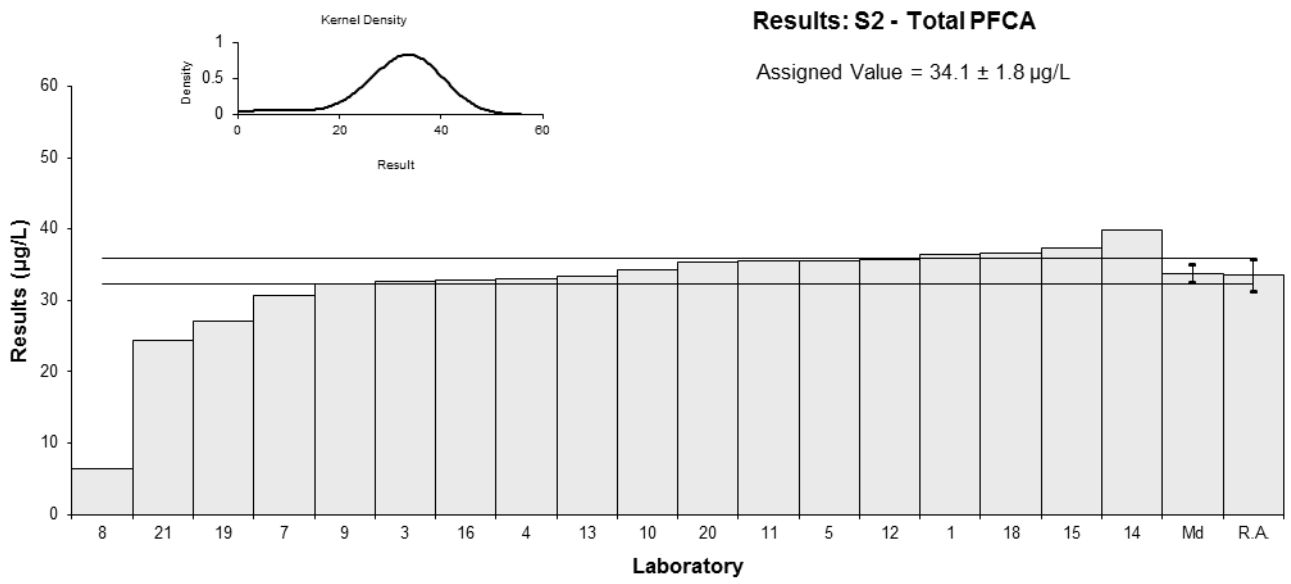
Participant Results

Lab Code	Result	z-Score
1	36.4	0.27
3	32.70913	-0.16
4	32.997	-0.13
5	35.568	0.17
7	30.66	-0.40
8	6.411	-3.25
9	32.3	-0.21
10	34.34	0.03
11	35.53	0.17
12	35.69622	0.19
13	33.3	-0.09
14	39.89	0.68
15	37.2752	0.37
16	32.82	-0.15
18	36.594	0.29
19	27.073	-0.82
20	35.4	0.15
21	24.36	-1.14

Statistics

Assigned Value*	34.1	1.8
Robust Average	33.6	2.2
Median	33.8	1.3
Mean	32.2	
N	18	
Max.	39.89	
Min.	6.411	
Robust SD	3.7	
Robust CV	11%	

*Robust Average excluding Laboratory 8


Kernel Density
Density
Result
Results: S2 - Total PFCA
Assigned Value = 34.1 ± 1.8 µg/L
Results (µg/L)
Laboratory
8 21 19 7 9 3 16 4 13 10 20 11 5 12 1 18 15 14 Md R.A.

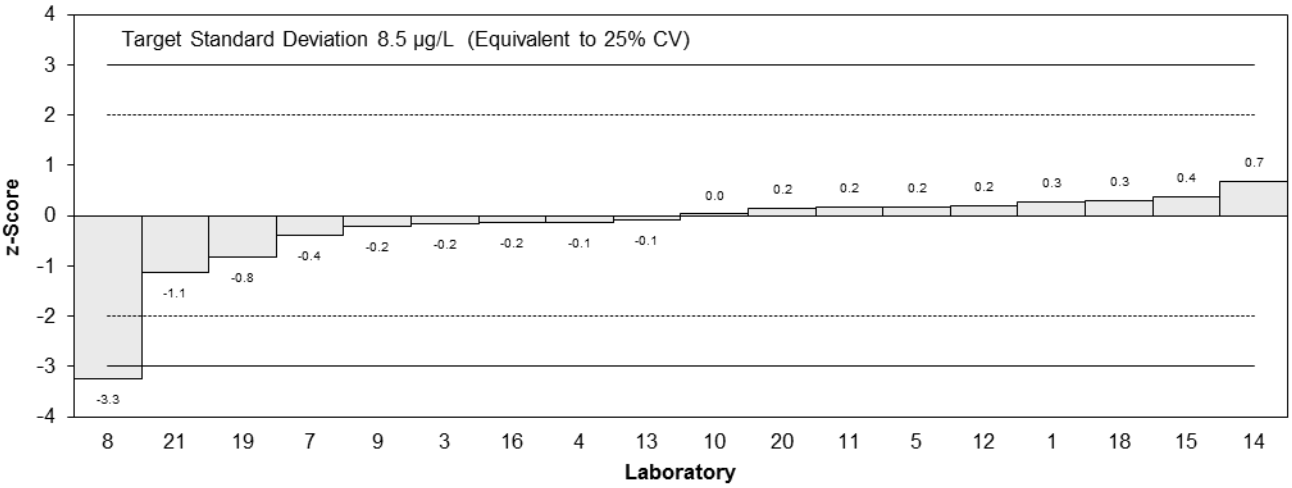

z-Scores: S2 - Total PFCA
Target Standard Deviation 8.5 µg/L (Equivalent to 25% CV)
z-Score
Laboratory
8 21 19 7 9 3 16 4 13 10 20 11 5 12 1 18 15 14
-3.3 -1.1 -0.8 -0.4 -0.2 -0.2 -0.2 -0.1 -0.1 0.0 0.2 0.2 0.2 0.2 0.3 0.3 0.4 0.7

Figure 13

Table 18

Sample Details

Sample No.	S3
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFDS
Units	µg/L

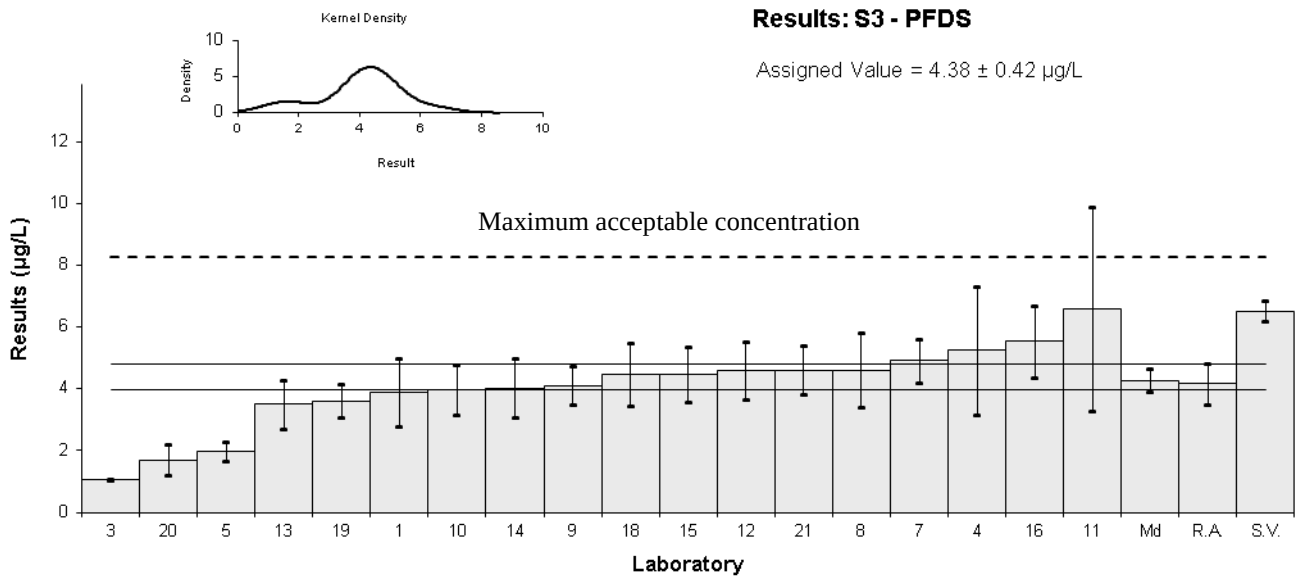
Participant Results

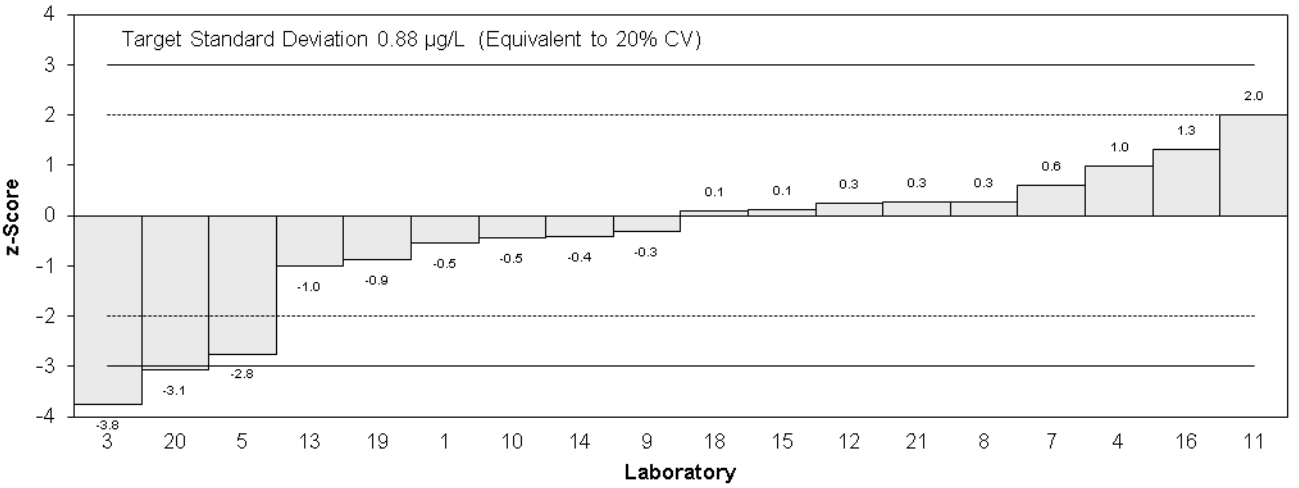
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	3.9	1.1	NR	-0.55	-0.41
3	1.08	0.0230	96.8	-3.77	-7.85
4	5.240	2.096	85	0.98	0.40
5	1.97	0.31	NR	-2.75	-4.62
7	4.91	0.69	118.56	0.61	0.66
8	4.61	1.20	NR	0.26	0.18
9	4.1	0.62	104	-0.32	-0.37
10	3.98	0.80	104	-0.46	-0.44
11*	6.6	3.3	110	2.00	0.67
12	4.60	0.921	71	0.25	0.22
13	3.5	0.8	103	-1.00	-0.97
14	4.02	0.95	106	-0.41	-0.35
15	4.48101	0.896	76	0.12	0.10
16	5.54	1.154	NR	1.32	0.94
18	4.46	1.017	138	0.09	0.07
19	3.605	0.54075	NR	-0.88	-1.13
20	1.7	0.51	NR	-3.06	-4.06
21	4.61	0.79	95	0.26	0.26

Statistics

Assigned Value**	4.38	0.42
Spike	6.51	0.33
Robust Average	4.16	0.65
Median	4.28	0.37
Mean	4.05	
N	18	
Max.	6.6	
Min.	1.08	
Robust SD	1.1	
Robust CV	26%	

*z-Score adjusted to 2 (see Section 6.3).
**Robust Average excluding Laboratories 3, 5, 11 and 20.


Kernel Density
Density
Result
Results: S3 - PFDS
Assigned Value = 4.38 ± 0.42 µg/L
Maximum acceptable concentration
Results (µg/L)
Laboratory
3 20 5 13 19 1 10 14 9 18 15 12 21 8 7 4 16 11 Md R.A S.V.


z-Scores: S3 - PFDS
Target Standard Deviation 0.88 µg/L (Equivalent to 20% CV)
z-Score
Laboratory
3 20 5 13 19 1 10 14 9 18 15 12 21 8 7 4 16 11
-3.8 -3.1 -2.8 -1.0 -0.9 -0.5 -0.5 -0.4 -0.3 0.1 0.1 0.3 0.3 0.3 0.6 1.0 1.3 2.0

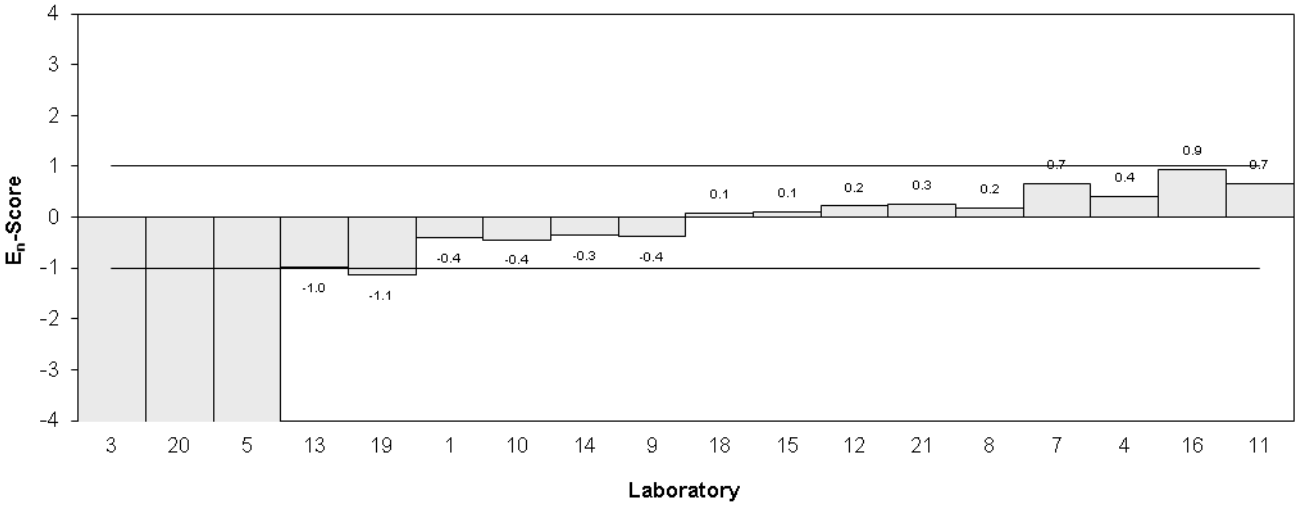

En-Scores: S3 - PFDS
En-Score
Laboratory
3 20 5 13 19 1 10 14 9 18 15 12 21 8 7 4 16 11
-1.0 -1.1 -0.4 -0.4 -0.3 -0.4 0.1 0.1 0.2 0.3 0.2 0.7 0.4 0.9 0.7

Figure 14

Table 19

Sample Details

Sample No.	S3
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFNA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	0.046	0.013	103
3	0.0405	0.000834	91.9
4	0.0469	0.0141	97
5	0.055	0.006	100
7	0.032	0.007	118.56
8	0.067	0.014	NR
9	0.049	0.0074	112
10	0.049	0.01	100
11	0.04	0.02	90
12	0.0411	0.0123	70
13	0.04	0.02	112
14	0.03	0.011	110
15	0.04120	0.0103	84
16	0.04	0.010	99
18	0.06	0.005	110
19	0.045	0.0027	92
20	0.05	0.0125	NR
21	< 0.10	NR	98

Statistics

Assigned Value	Not Set	
Robust Average	0.0449	0.0054
Median	0.0450	0.0038
Mean	0.0455	
N	17	
Max.	0.067	
Min.	0.03	
Robust SD	0.0089	
Robust CV	20%	

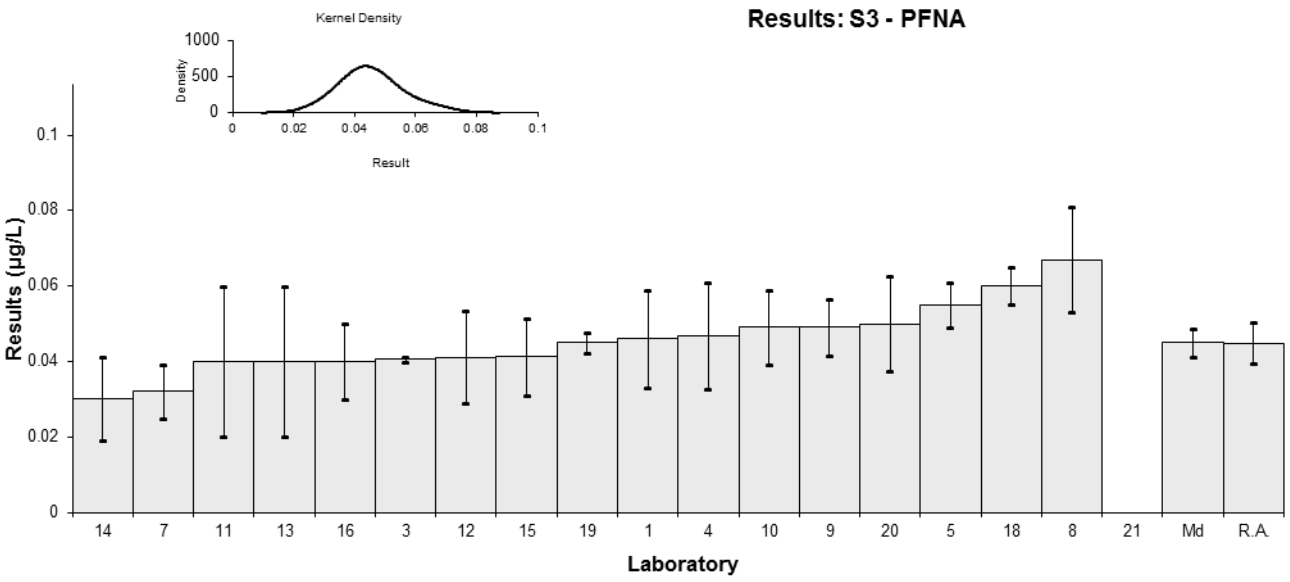

Results: S3 - PFNA
Kernel Density
Density
1000
500
0
0
0.02
0.04
0.06
0.08
0.1
Result
Results (µg/L)
0.1
0.08
0.06
0.04
0.02
0
14
7
11
13
16
3
12
15
19
1
4
10
9
20
5
18
8
21
Md
R.A.
Laboratory

Figure 15

Table 20

Sample Details

Sample No.	S3
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFDA
Units	µg/L

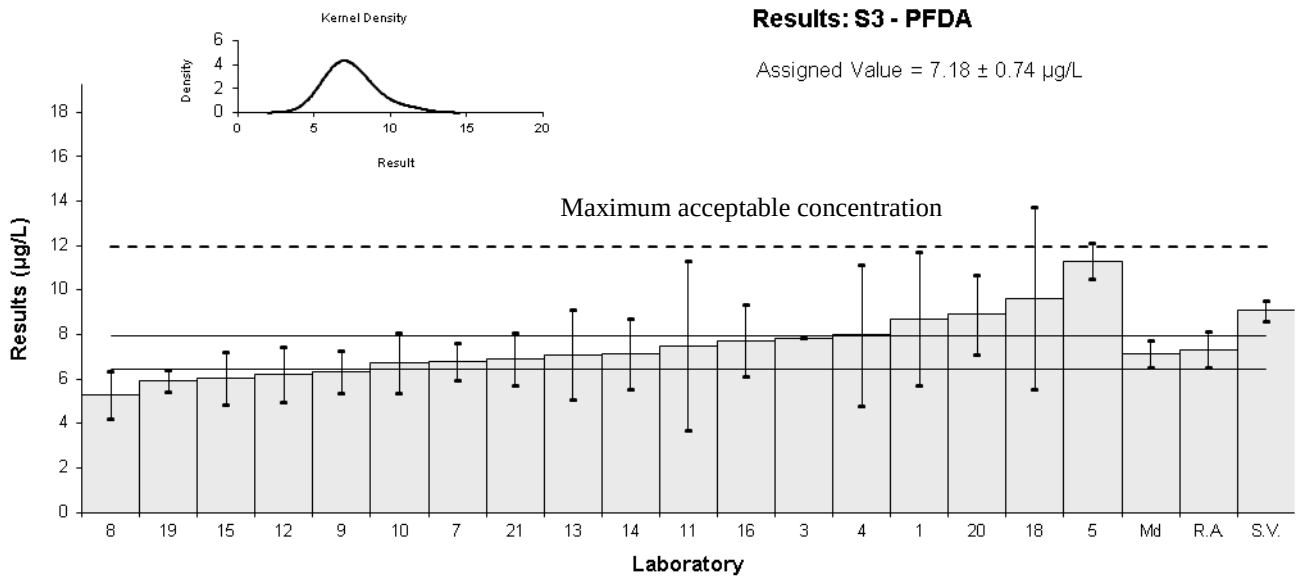
Participant Results

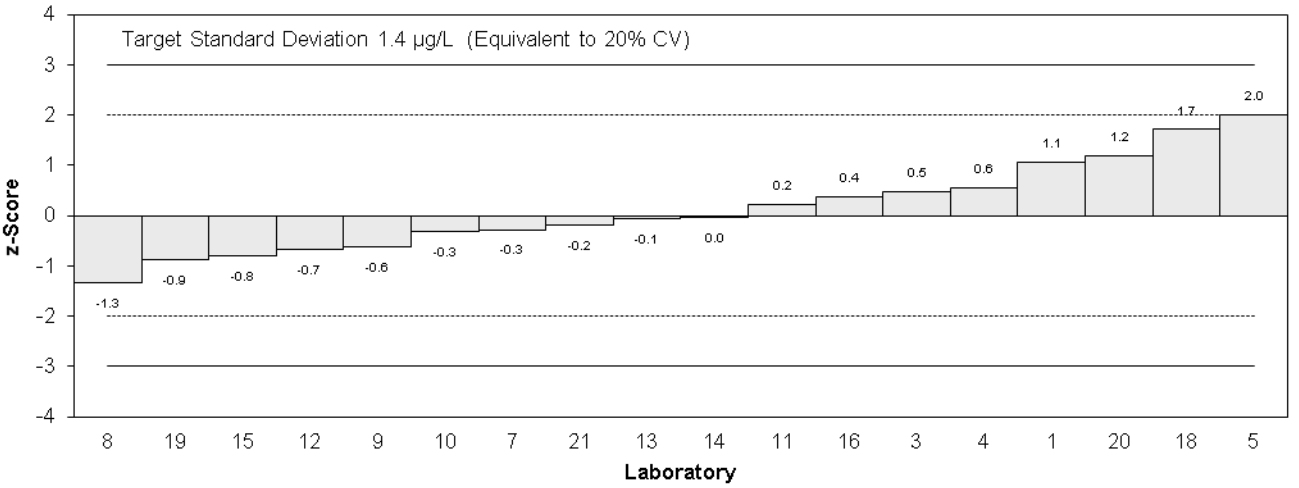
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	8.7	3.0	91	1.06	0.49
3	7.85	0.0100	97.4	0.47	0.91
4	7.979	3.192	90	0.56	0.24
5*	11.3	0.8	75	2.00	1.00
7	6.78	0.86	118.56	-0.28	-0.35
8	5.27	1.06	NR	-1.33	-1.48
9	6.3	0.95	96	-0.61	-0.73
10	6.72	1.34	83	-0.32	-0.30
11	7.5	3.8	100	0.22	0.08
12	6.22	1.24	77	-0.67	-0.66
13	7.1	2	96	-0.06	-0.04
14	7.13	1.57	111	-0.03	-0.03
15	6.034	1.206	83	-0.80	-0.81
16	7.72	1.622	80	0.38	0.30
18	9.64	4.107	19	1.71	0.59
19	5.926	0.47408	59	-0.87	-1.43
20	8.9	1.78	NR	1.20	0.89
21	6.91	1.18	96	-0.19	-0.19

Statistics

Assigned Value**	7.18	0.74
Spike	9.08	0.45
Robust Average	7.32	0.80
Median	7.12	0.62
Mean	7.44	
N	18	
Max.	11.3	
Min.	5.27	
Robust SD	1.4	
Robust CV	19%	

*z-Score adjusted to 2 and E_n-score adjusted to 1 (see Section 6.3).
**Robust Average excluding Laboratory 5.


Kernel Density
Density
Result
Results: S3 - PFDA
Assigned Value = 7.18 ± 0.74 µg/L
Maximum acceptable concentration
Results (µg/L)
8 19 15 12 9 10 7 21 13 14 11 16 3 4 1 20 18 5 Md R.A S.V.
Laboratory


z-Scores: S3 - PFDA
Target Standard Deviation 1.4 µg/L (Equivalent to 20% CV)
z-Score
-1.3 -0.9 -0.8 -0.7 -0.6 -0.3 -0.3 -0.2 -0.1 0.0 0.2 0.4 0.5 0.6 1.1 1.2 1.7 2.0
8 19 15 12 9 10 7 21 13 14 11 16 3 4 1 20 18 5
Laboratory

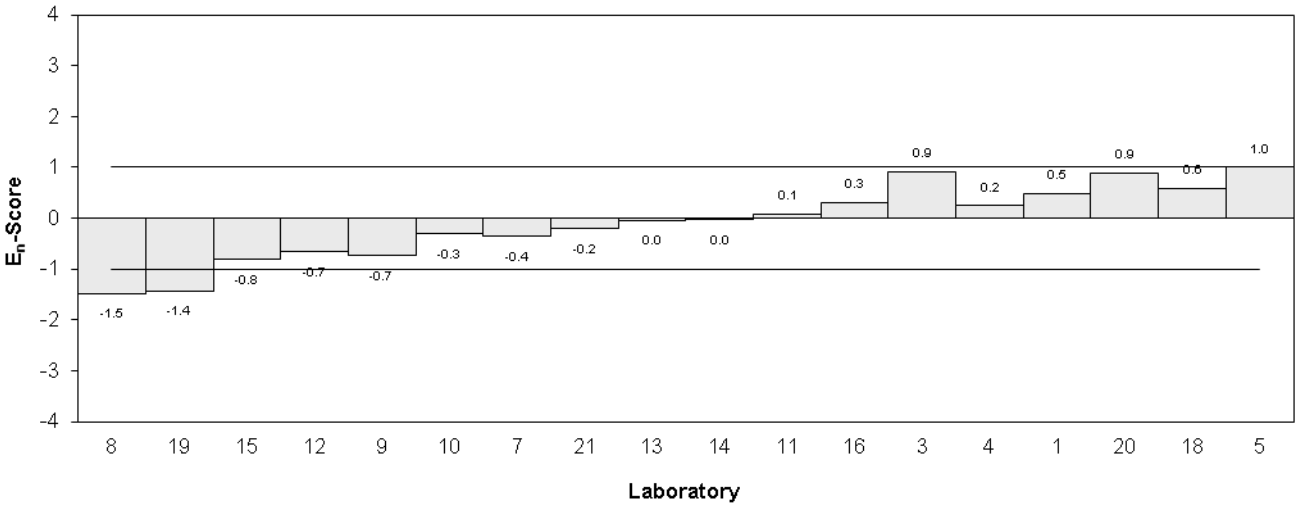

En-Scores: S3 - PFDA
En-Score
-1.5 -1.4 -0.8 -0.7 -0.7 -0.3 -0.4 -0.2 0.0 0.0 0.1 0.3 0.9 0.2 0.5 0.9 0.6 1.0
8 19 15 12 9 10 7 21 13 14 11 16 3 4 1 20 18 5
Laboratory

Figure 16

Table 21

Sample Details

Sample No.	S3
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	6:2FTS
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	3.8	0.95	95
3	4.63	0.0708	83.2
4	4.567	1.827	396
5	3.46	0.18	125
7	4.86	0.61	119.29
8	6.41	2.24	NR
9	3.9	0.59	98
10	4.48	0.9	97
11	3.5	1.8	100
12	4.21	0.842	71
13	4.6	1	154
14	3.95	1.03	110
15	4.055	0.8109	84
16	3.81	0.818	89
18	3.11	1.319	79
19	3.385	0.23695	90
20	2.9	0.87	NR
21	3.92	2.28	110

Statistics

Assigned Value	Not Set	
Robust Average	4.01	0.39
Median	3.94	0.38
Mean	4.09	
N	18	
Max.	6.41	
Min.	2.9	
Robust SD	0.62	
Robust CV	16%	

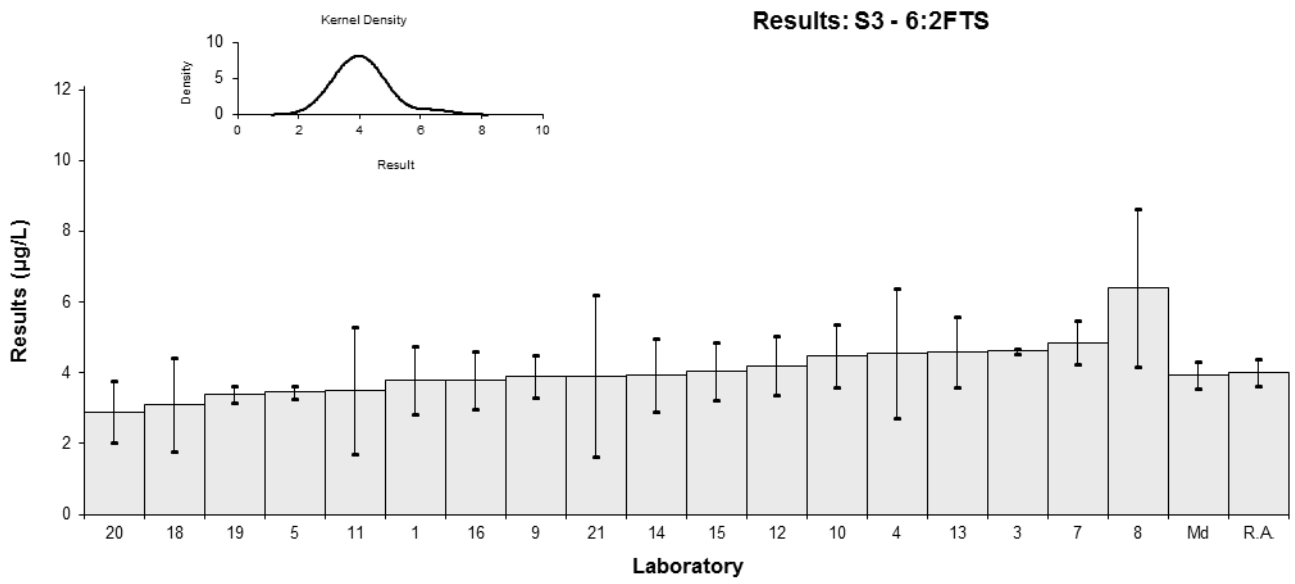

Results: S3 - 6:2FTS
Kernel Density
Density
Result
Results (µg/L)
Laboratory
20
18
19
5
11
1
16
9
21
14
15
12
10
4
13
3
7
8
Md
R.A.

Figure 17

Table 22

Sample Details

Sample No.	S3
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	8:2FTS
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 0.02	0.005	NR
3	NR	NR	60.5
4	0.0115	0.0046	161
5	0.019	0.004	56
7	<0.015	NR	119.29
8	NR	NR	NR
9	<0.02	NR	NR
10	<0.04	0.01	64
11	<0.01	NR	90
12	0.00981	0.00490	71
13	0.01	0.01	109
14	<0.02	NR	97
15	0.00754	0.00264	75
16	<0.05	0.018	80
18	<0.05	NR	137
19	0.049	0.0049	59
20	0.01	0.003	NR
21	< 1.00	NR	100

Statistics

Assigned Value	Not Set	
Robust Average	0.0129	0.0059
Median	0.0100	0.0021
Mean	0.0167	
N	7	
Max.	0.049	
Min.	0.00754	
Robust SD	0.0026	
Robust CV	20%	

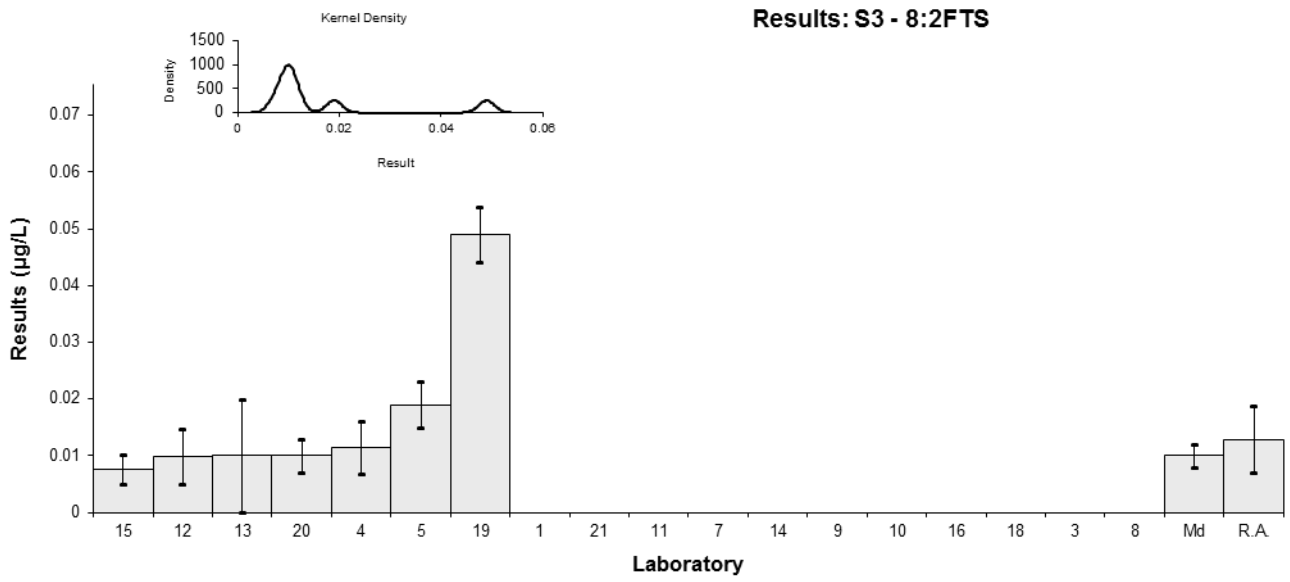

Results: S3 - 8:2FTS
Kernel Density
Density
1500
1000
500
0
0
0.02
0.04
0.06
Result
Results (µg/L)
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
15
12
13
20
4
5
19
1
21
11
7
14
9
10
16
18
3
8
Md
R.A.
Laboratory

Figure 18

Table 23

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFDS
Units	µg/L

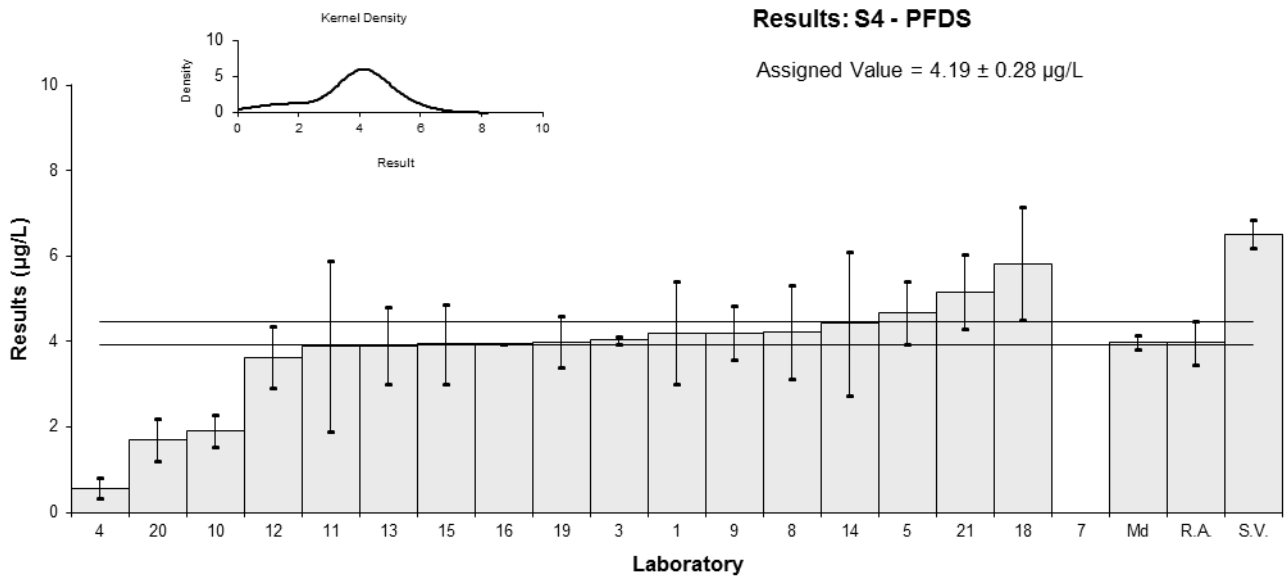
Participant Results

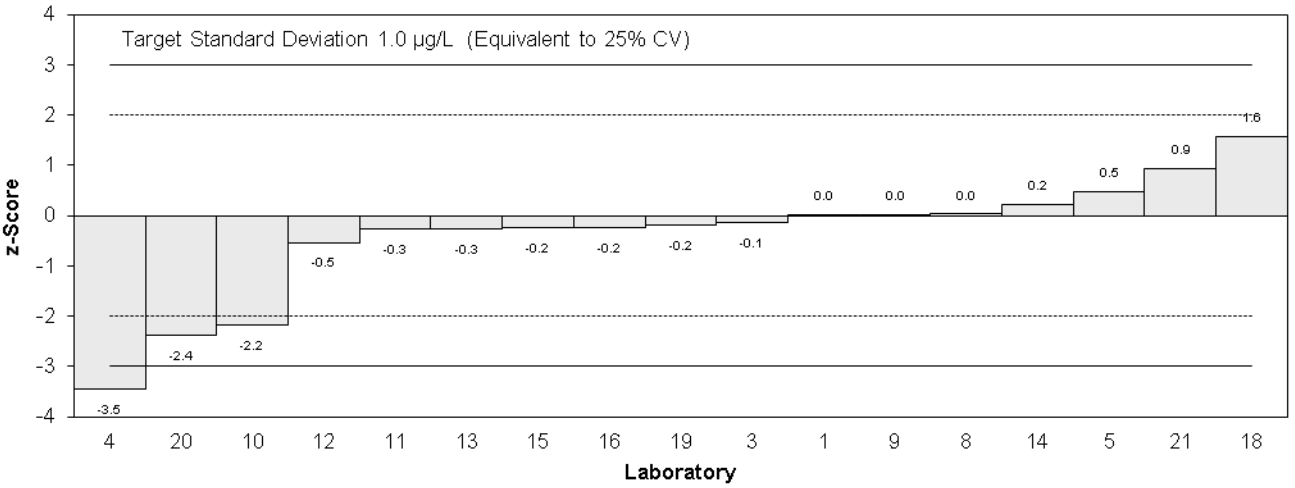
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	4.2	1.2	79	0.01	0.01
3	4.04	0.0861	89.3	-0.14	-0.51
4	0.572	0.229	81	-3.45	-10.00
5	4.68	0.74	NR	0.47	0.62
7	<5.0	NR	122.93		
8	4.22	1.10	NR	0.03	0.03
9	4.2	0.63	101	0.01	0.01
10	1.90	0.38	99	-2.19	-4.85
11	3.9	2.0	110	-0.28	-0.14
12	3.63	0.726	69	-0.53	-0.72
13	3.9	0.9	91	-0.28	-0.31
14	4.43	1.68	78	0.23	0.14
15	3.94	0.936	76	-0.24	-0.26
16	3.94	0.006	NR	-0.24	-0.89
18	5.83	1.329	112	1.57	1.21
19	3.993	0.59895	NR	-0.19	-0.30
20	1.7	0.51	NR	-2.38	-4.28
21	5.17	0.88	100	0.94	1.06

Statistics

Assigned Value*	4.19	0.28
Spike	6.51	0.33
Robust Average	3.98	0.51
Median	3.99	0.17
Mean	3.78	
N	17	
Max.	5.83	
Min.	0.572	
Robust SD	0.83	
Robust CV	21%	

*Robust Average excluding Laboratories 4, 10 and 20.


Kernel Density
Density
Result
Results: S4 - PFDS
Assigned Value = 4.19 ± 0.28 µg/L
Results (µg/L)
Laboratory
4 20 10 12 11 13 15 16 19 3 1 9 8 14 5 21 18 7 Md R.A. S.V.


z-Scores: S4 - PFDS
Target Standard Deviation 1.0 µg/L (Equivalent to 25% CV)
z-Score
Laboratory
4 20 10 12 11 13 15 16 19 3 1 9 8 14 5 21 18
-3.5 -2.4 -2.2 -0.5 -0.3 -0.3 -0.2 -0.2 -0.2 -0.1 0.0 0.0 0.0 0.2 0.5 0.9 1.6

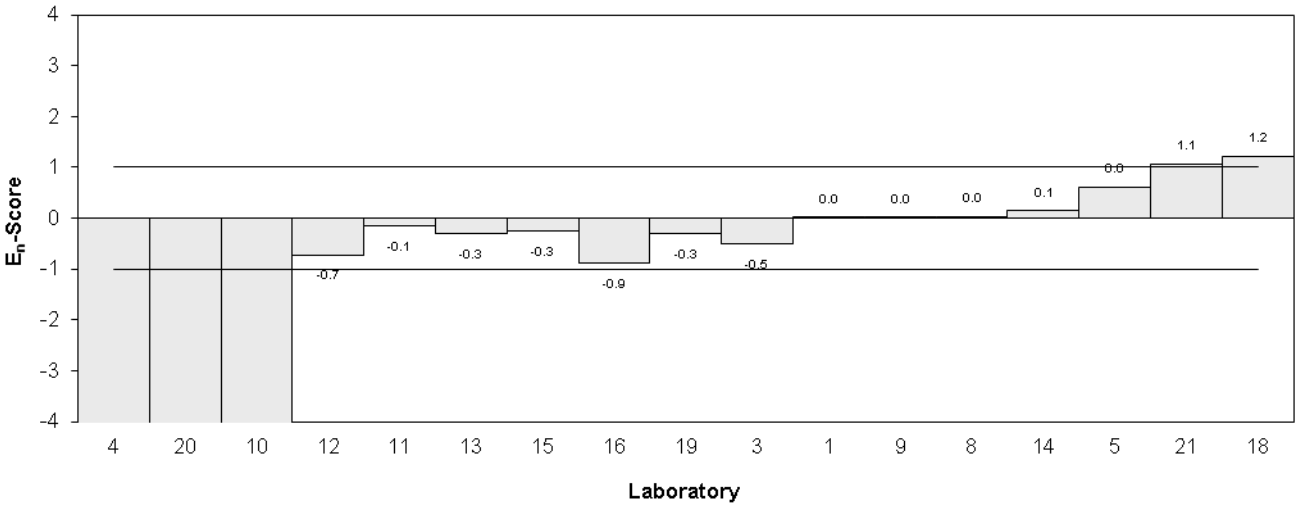

En-Scores: S4 - PFDS
En-Score
Laboratory
4 20 10 12 11 13 15 16 19 3 1 9 8 14 5 21 18
-0.7 -0.1 -0.3 -0.3 -0.9 -0.3 -0.5 0.0 0.0 0.0 0.1 0.6 1.1 1.2

Figure 19

Table 24

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFBA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	35	13	61
3	26.0	0.351	90.7
4	39.73	15.89	71
5	21.2	0.9	59
7	<100	NR	117.38
8	0.84	0.17	NR
9	26	3.9	97
10	32.6	6.52	101
11	37	19	90
12	33.2	6.64	72
13	30	8	50
14	44.5	16.0	80
15	27.3	6.84	83
16	20.3	3.16	93
18	25.06	3.644	11
19	29.248	1.16992	67
20	36	7.2	NR
21	21.7	2.67	100

Statistics

Assigned Value*	Not Set	
Robust Average	29.4	4.9
Median	29.2	4.4
Mean	28.6	
N	17	
Max.	44.5	
Min.	0.84	
Robust SD	6.9	
Robust CV	24%	

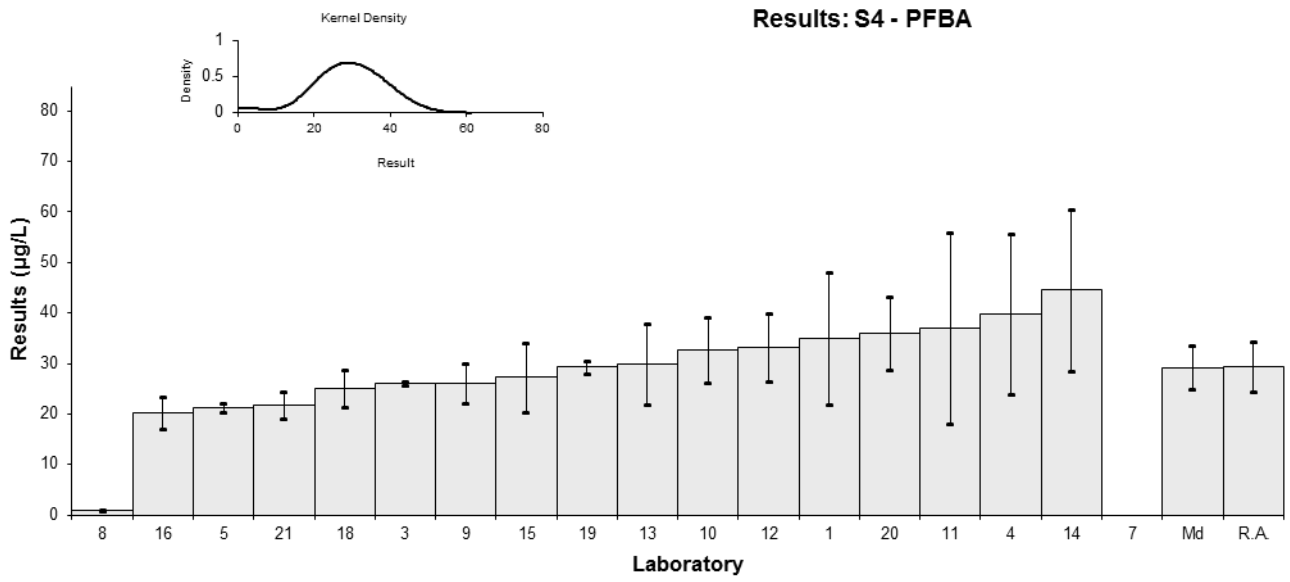

Results: S4 - PFBA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
8
16
5
21
18
3
9
15
19
13
10
12
1
20
11
4
14
7
Md
R.A.

Figure 20

Table 25

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFPeA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	82	35	62
3	40.8	0.657	85.4
4	92.58	37.03	53
5	62.2	3.2	51
7	58.3	NR	122.93
8	0.66	0.13	NR
9	61	9.2	126
10	81.3	16.3	103
11	87	44	100
12	85.1	17.0	69
13	55	10	42
14	65.6	20.1	71
15	82.3	20.57	88
16	46.6	5.60	85
18	55.17	4.189	51
19	72.981	1.45962	75
20	76	16.72	NR
21	43.1	4.65	92

Statistics

Assigned Value*	Not Set	
Robust Average	66	12
Median	64	13
Mean	64	
N	18	
Max.	92.58	
Min.	0.66	
Robust SD	20	
Robust CV	30%	

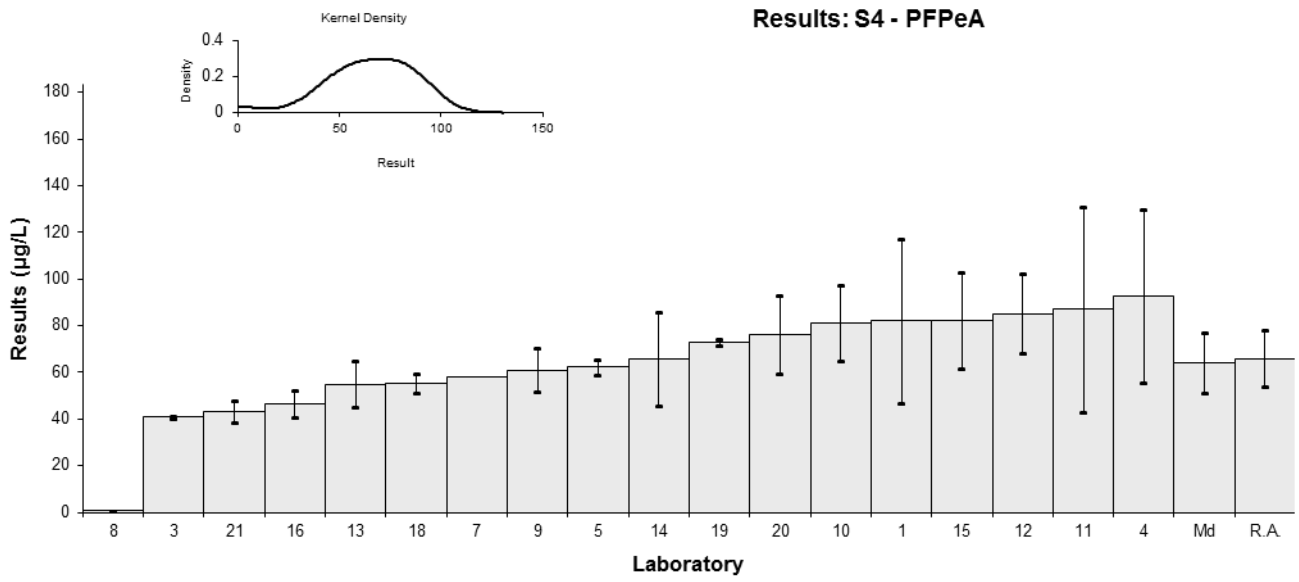

Results: S4 - PFPeA
Kernel Density
Density
0.4
0.2
0
0
50
100
150
Result
Results (µg/L)
180
160
140
120
100
80
60
40
20
0
8
3
21
16
13
18
7
9
5
14
19
20
10
1
15
12
11
4
Md
R.A.
Laboratory

Figure 21

Table 26

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFHxA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	26	6.5	64
3	19.6	0.396	132
4	25.51	7.653	53
5	14.2	0.6	58
7	17.6	NR	122.93
8	0.68	0.14	NR
9	20	3.0	101
10	40.8	8.2	99
11	17	9	90
12	22.1	4.42	73
13	40	10	72
14	22.9	8.0	70
15	21.1	5.28	94
16	20.6	2.21	95
18	33.17	8.425	84
19	74.805	5.9844	56
20	18	5.4	NR
21	10.7	1.25	100

Statistics

Assigned Value	Not Set	
Robust Average	22.8	6.1
Median	20.9	3.1
Mean	24.7	
N	18	
Max.	74.8	
Min.	0.68	
Robust SD	10	
Robust CV	44%	

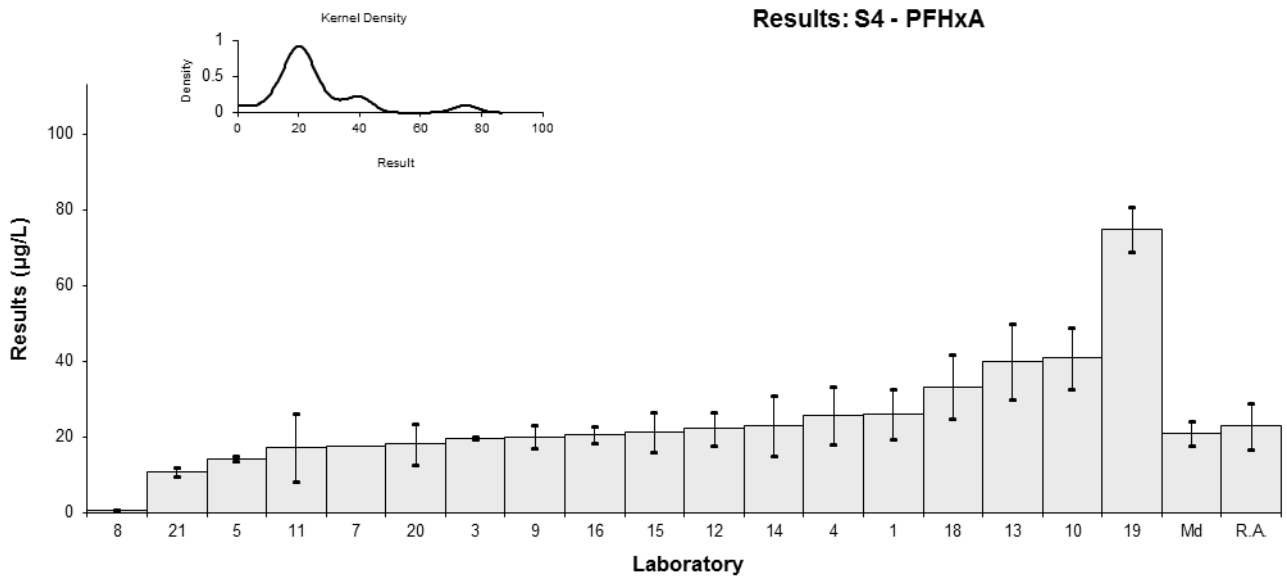

Results: S4 - PFHxA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
8
21
5
11
7
20
3
9
16
15
12
14
4
1
18
13
10
19
Md
R.A.

Figure 22

Table 27

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFHpA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	11	2.8	65
3	6.52	0.1024	115
4	10.76	3.228	53
5	5.29	0.23	54
7	4.1	NR	122.93
8	0.012	0.004	NR
9	4.4	0.66	89
10	10.6	2.1	94
11	7	4	100
12	9.92	1.98	75
13	6.3	1	72
14	7.27	2.47	76
15	8.75	2.18	78
16	3.97	0.424	93
18	6.15	2.660	93
19	16.756	1.34048	76
20	7.1	1.775	NR
21	5.32	0.59	99

Statistics

Assigned Value	Not Set	
Robust Average	7.2	1.9
Median	6.8	1.6
Mean	7.29	
N	18	
Max.	16.756	
Min.	0.012	
Robust SD	3.1	
Robust CV	43%	

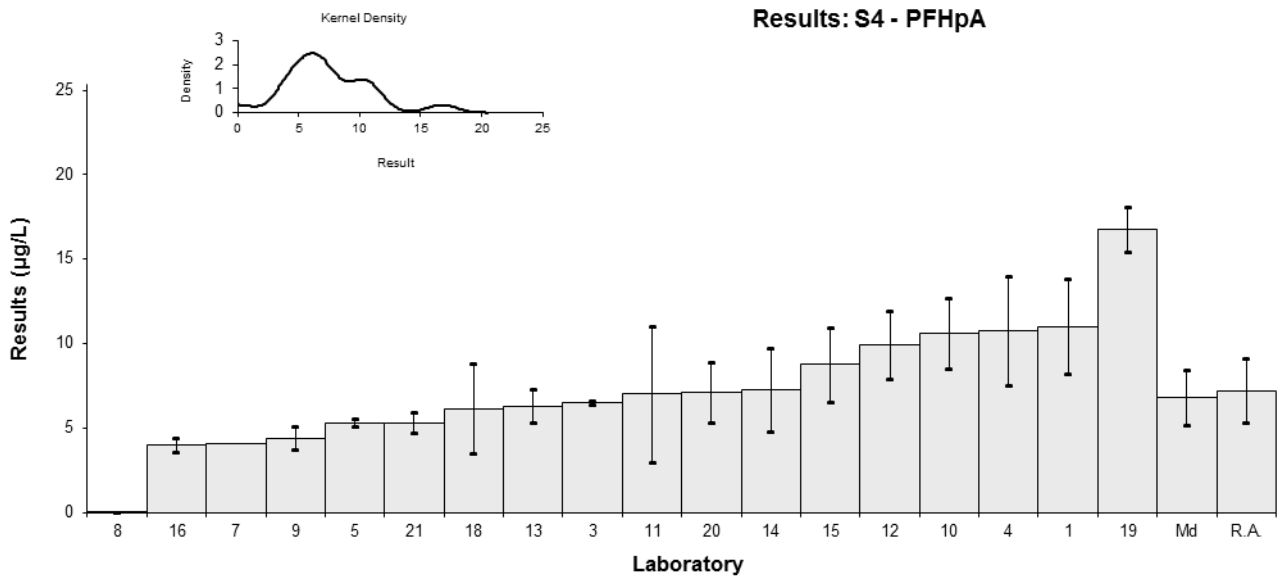

Results: S4 - PFHpA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
8
16
7
9
5
21
18
13
3
11
20
14
15
12
10
4
1
19
Md
R.A.

Figure 23

Table 28

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFOA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	1.9	0.54	69
3	2.15	0.0327	74.7
4	1.20	0.360	70
5	1.50	0.06	55
7	1.5	NR	122.93
8	NR	NR	101.7
9	1.8	0.27	101
10	2.52	0.50	102
11	1.6	0.8	90
12	1.63	0.326	71
13	2.8	0.7	64
14	2.20	0.79	82
15	1.512	0.377	89
16	1.98	0.600	96
18	2.4	0.248	100
19	3.708	0.29664	89
20	2.6	0.78	NR
21	0.981	0.144	99

Statistics

Assigned Value	Not Set	
Robust Average	1.95	0.38
Median	1.90	0.30
Mean	2.0	
N	17	
Max.	3.708	
Min.	0.981	
Robust SD	0.58	
Robust CV	30%	

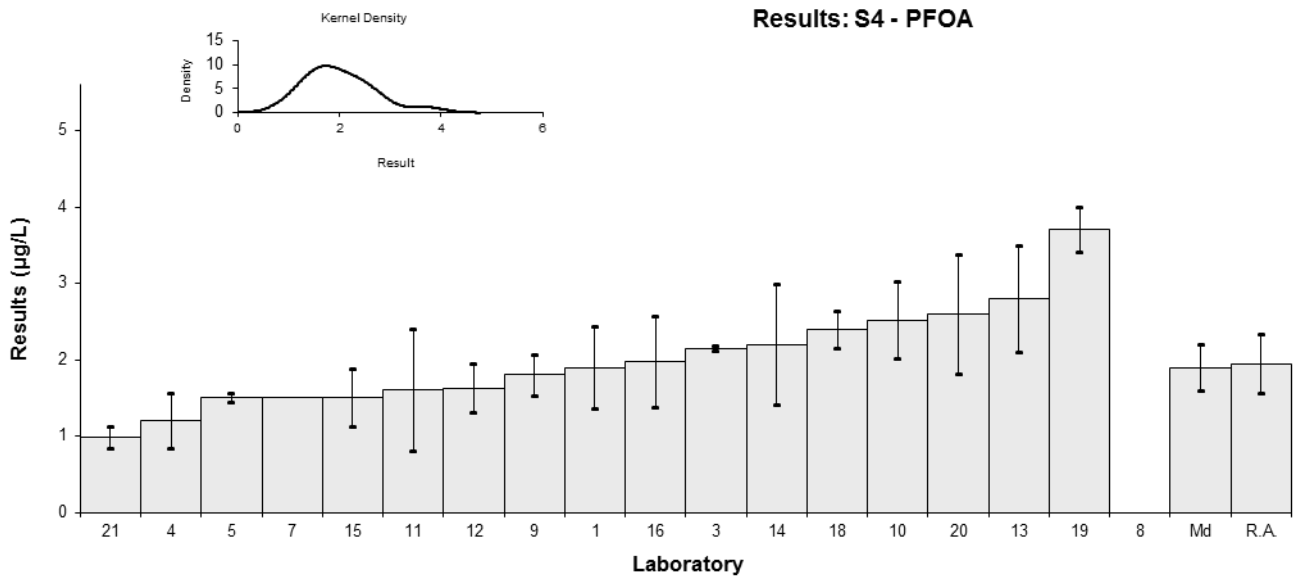

Results: S4 - PFOA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
21
4
5
7
15
11
12
9
1
16
3
14
18
10
20
13
19
8
Md
R.A.

Figure 24

Table 29

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFNA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	0.98	0.27	73
3	0.984	0.0203	84.5
4	0.443	0.133	84
5	1.01	0.10	65
7	<1.0	NR	122.93
8	0.063	0.013	NR
9	0.86	0.13	100
10	0.77	0.15	97
11	0.9	0.5	90
12	1.15	0.230	72
13	0.83	0.3	73
14	1.75	0.60	82
15	1.107	0.276	84
16	0.60	0.097	100
18	0.85	0.071	116
19	2.150	0.129	93
20	1.3	0.325	NR
21	0.884	0.156	101

Statistics

Assigned Value	Not Set	
Robust Average	0.94	0.20
Median	0.900	0.099
Mean	0.978	
N	17	
Max.	2.15	
Min.	0.063	
Robust SD	0.170	
Robust CV	18%	

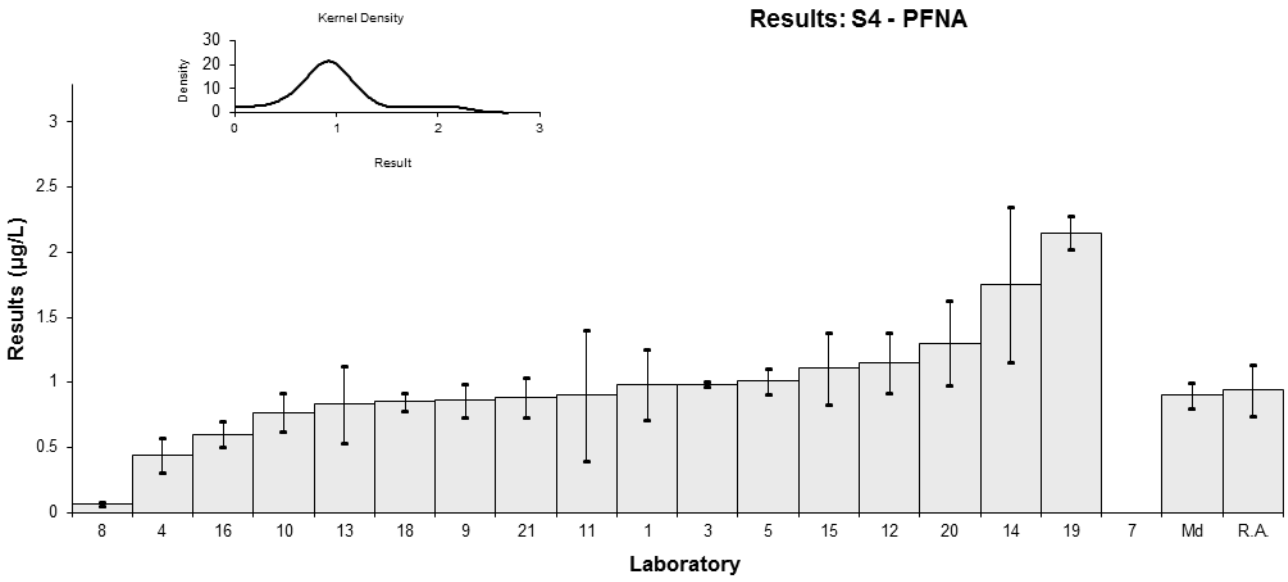

Results: S4 - PFNA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
8
4
16
10
13
18
9
21
11
1
3
5
15
12
20
14
19
7
Md
R.A.

Figure 25

Table 30

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFDA
Units	µg/L

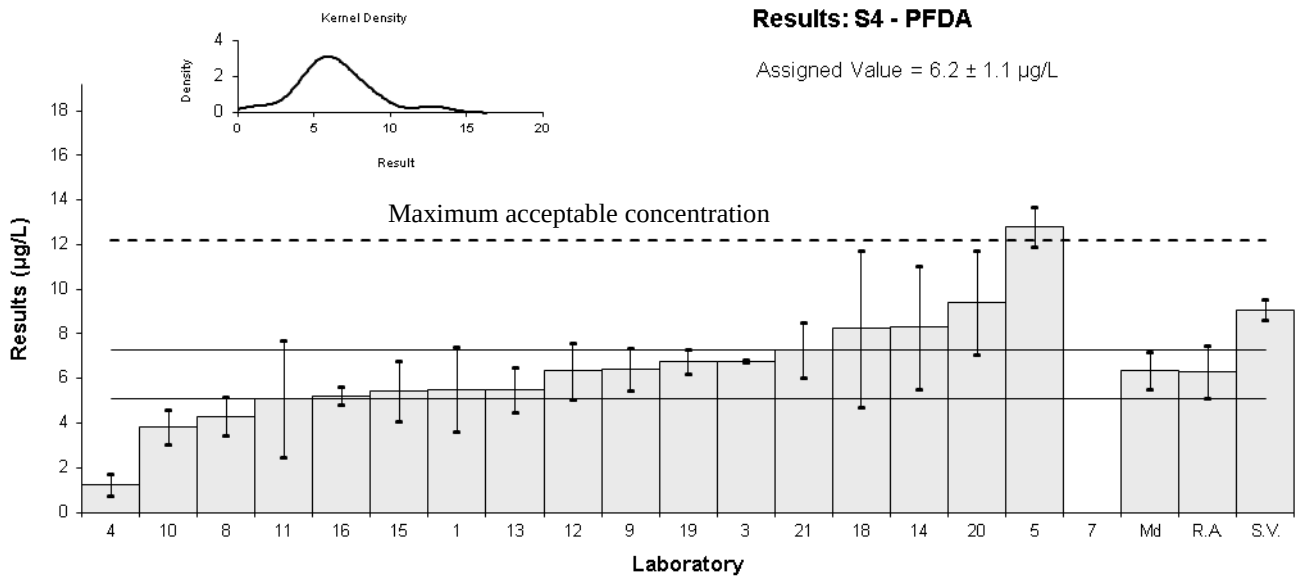
Participant Results

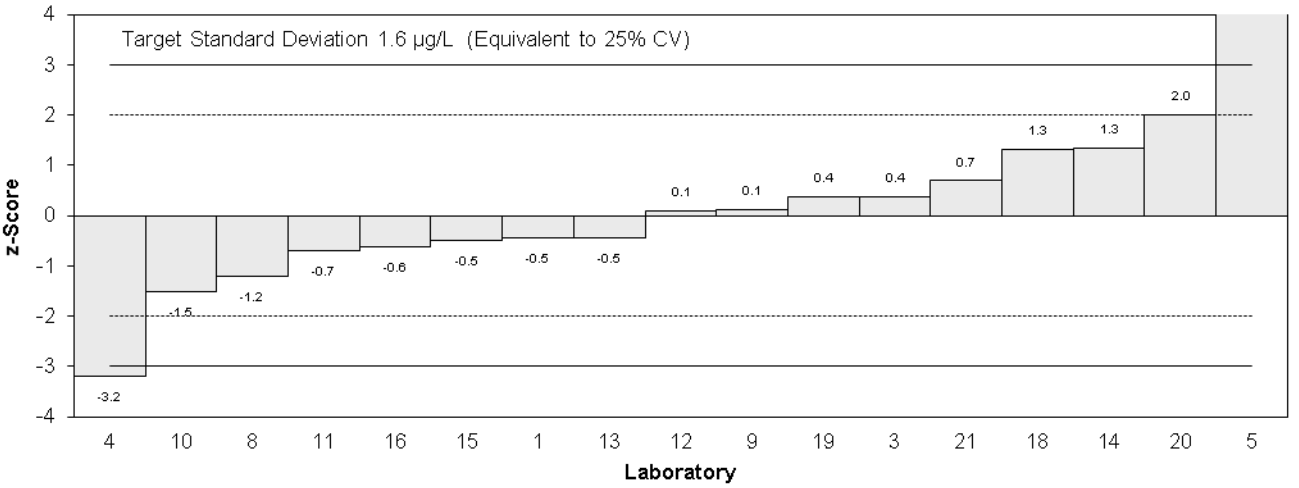
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	5.5	1.9	88	-0.45	-0.32
3	6.79	0.0862	83.0	0.38	0.53
4	1.231	0.492	81	-3.21	-4.12
5	12.8	0.9	53	4.26	4.64
7	<5.0	NR	122.93		
8	4.31	0.86	NR	-1.22	-1.35
9	6.4	0.96	98	0.13	0.14
10	3.85	0.77	105	-1.52	-1.75
11	5.1	2.6	100	-0.71	-0.39
12	6.35	1.27	74	0.10	0.09
13	5.5	1	75	-0.45	-0.47
14	8.29	2.74	74	1.35	0.71
15	5.42	1.36	83	-0.50	-0.45
16	5.23	0.402	103	-0.63	-0.83
18	8.25	3.515	101	1.32	0.56
19	6.767	0.54136	76	0.37	0.46
20*	9.4	2.35	NR	2.00	1.00
21	7.27	1.24	100	0.69	0.65

Statistics

Assigned Value**	6.2	1.1
Spike	9.08	0.45
Robust Average	6.3	1.2
Median	6.35	0.85
Mean	6.38	
N	17	
Max.	12.8	
Min.	1.23	
Robust SD	1.6	
Robust CV	25%	

*z-score adjusted to 2 and En score adjusted to 1(see Section 6.3).
**Robust Average excluding Laboratories 4 and 5.


Kernel Density
Density
Result
Results: S4 - PFDA
Assigned Value = 6.2 ± 1.1 µg/L
Maximum acceptable concentration
Results (µg/L)
Laboratory
4 10 8 11 16 15 1 13 12 9 19 3 21 18 14 20 5 7 Md R.A S.V.


z-Scores: S4 - PFDA
Target Standard Deviation 1.6 µg/L (Equivalent to 25% CV)
z-Score
Laboratory
4 10 8 11 16 15 1 13 12 9 19 3 21 18 14 20 5
-3.2 -1.5 -1.2 -0.7 -0.6 -0.5 -0.5 -0.5 0.1 0.1 0.4 0.4 0.7 1.3 1.3 2.0

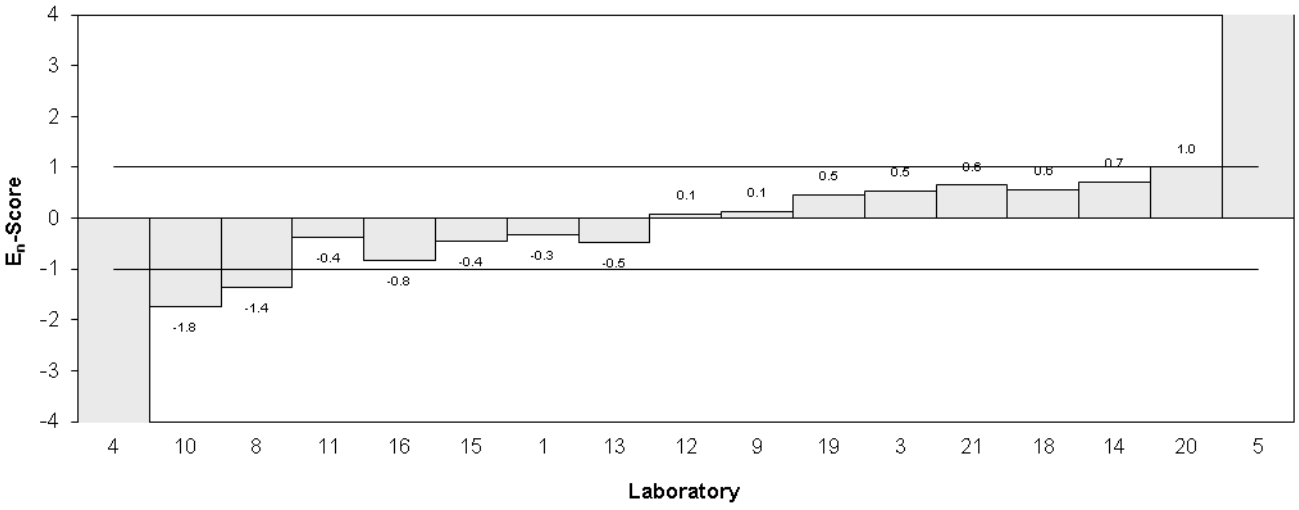

En-Scores: S4 - PFDA
En-Score
Laboratory
4 10 8 11 16 15 1 13 12 9 19 3 21 18 14 20 5
-1.8 -1.4 -0.4 -0.8 -0.4 -0.3 -0.5 0.1 0.1 0.5 0.5 0.6 0.6 0.7 1.0

Figure 26

Table 31

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFUdA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 0.42	0.11	86
3	0.307	0.00488	78.5
4	< 0.1	NR	NR
5	0.237	0.020	51
7	<1.0	NR	131.04
8	NR	NR	NR
9	0.26	0.039	115
10	<0.40	0.10	96
11	0.3	0.15	120
12	0.383	0.0766	79
13	0.20	0.08	77
14	<0.02	NR	73
15	0.327	0.0818	80
16	0.21	0.010	102
18	0.34	NR	108
19	0.621	0.03105	80
20	0.39	0.0975	NR
21	0.211	0.040	106

Statistics

Assigned Value	Not Set	
Robust Average	0.300	0.064
Median	0.304	0.069
Mean	0.316	
N	12	
Max.	0.621	
Min.	0.2	
Robust SD	0.078	
Robust CV	26%	

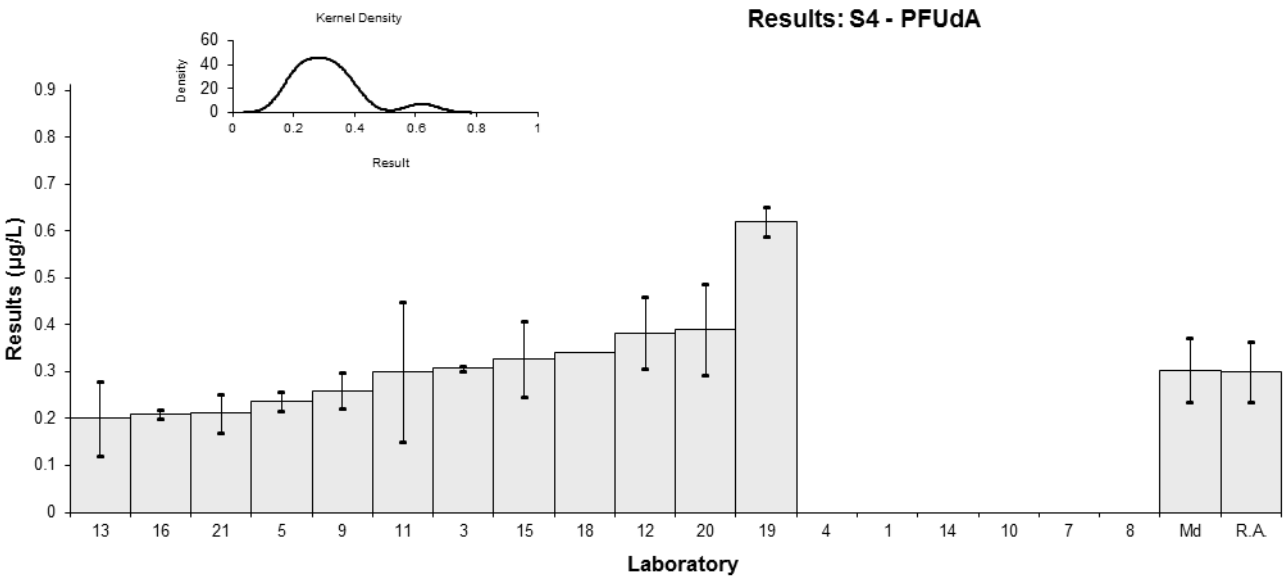

Results: S4 - PFUdA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
13
16
21
5
9
11
3
15
18
12
20
19
4
1
14
10
7
8
Md
R.A.

Figure 27

Table 32

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFDoA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 0.83	0.21	NR
3	0.269	0.00422	93.3
4	< 0.1	NR	NR
5	0.180	0.012	41
7	<5.0	NR	131.04
8	NR	NR	NR
9	0.20	0.030	109
10	<0.40	0.10	107
11	0.2	0.1	130
12	0.222	0.0444	77
13	0.25	0.1	81
14	<0.02	NR	70
15	0.220	0.0549	75
16	0.26	0.008	104
18	0.33	NR	117
19	0.683	0.12977	74
20	0.29	0.087	NR
21	0.126	0.024	120

Statistics

Assigned Value	Not set	
Robust Average	0.242	0.050
Median	0.236	0.034
Mean	0.269	
N	12	
Max.	0.683	
Min.	0.126	
Robust SD	0.057	
Robust CV	24%	

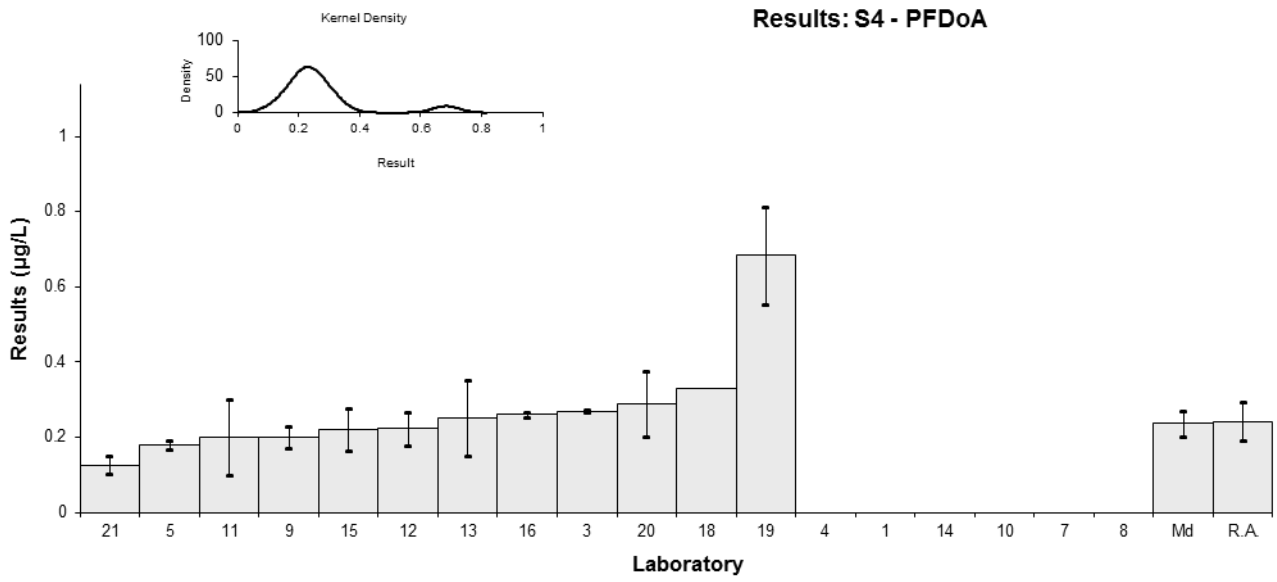

Results: S4 - PFDoA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
21
5
11
9
15
12
13
16
3
20
18
19
4
1
14
10
7
8
Md
R.A.

Figure 28

Table 33

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFTrDA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 2.1	1.4	NR
3	0.143	0.00265	93.3
4	< 0.1	NR	NR
5	0.118	0.021	NR
7	<5.0	NR	131.04
8	NR	NR	NR
9	0.12	0.018	98
10	<0.40	0.10	103
11	0.47	0.24	90
12	0.141	0.0282	77
13	<0.1	NR	78
14	<0.02	NR	69
15	0.112	0.0336	73
16	0.09	0.007	NR
18	<0.2	NR	130
19	<0.005	NR	NR
20	0.14	0.07	NR
21	< 0.10	NR	120

Statistics

Assigned Value	Not Set	
Robust Average	0.129	0.025
Median	0.130	0.015
Mean	0.167	
N	8	
Max.	0.47	
Min.	0.09	
Robust SD	0.028	
Robust CV	22%	

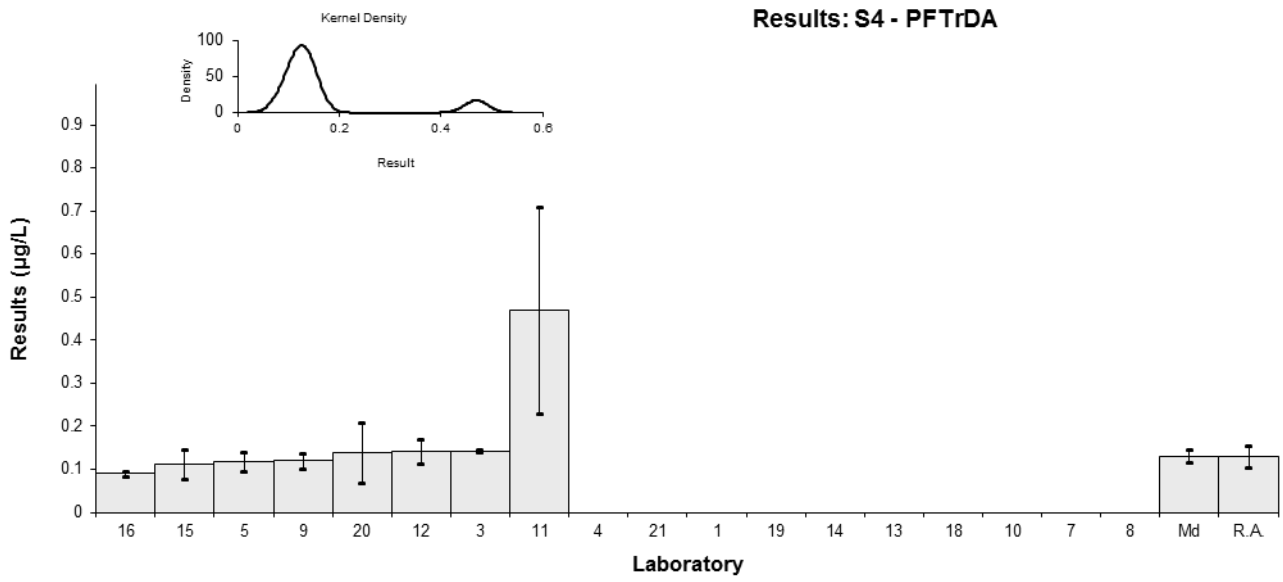

Results: S4 - PFTrDA
Kernel Density
Density
100
50
0
0
0.2
0.4
0.6
Result
Results (µg/L)
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
16
15
5
9
20
12
3
11
4
21
1
19
14
13
18
10
7
8
Md
R.A.
Laboratory

Figure 29

Table 34

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	PFTeDA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 0.83	0.47	80
3	0.164	0.00244	88.2
4	< 0.1	NR	NR
5	0.093	0.012	29
7	<5.0	NR	131.04
8	NR	NR	NR
9	0.15	0.023	112
10	<0.40	0.10	103
11	0.33	0.17	140
12	0.114	0.0228	75
13	<0.5	NR	78
14	<0.02	NR	69
15	0.121	0.0361	73
16	0.18	0.019	110
18	<0.2	NR	130
19	<0.005	NR	65
20	0.15	0.06	NR
21	< 0.10	NR	133

Statistics

Assigned Value	Not Set	
Robust Average	0.148	0.039
Median	0.150	0.037
Mean	0.163	
N	8	
Max.	0.33	
Min.	0.093	
Robust SD	0.044	
Robust CV	30%	

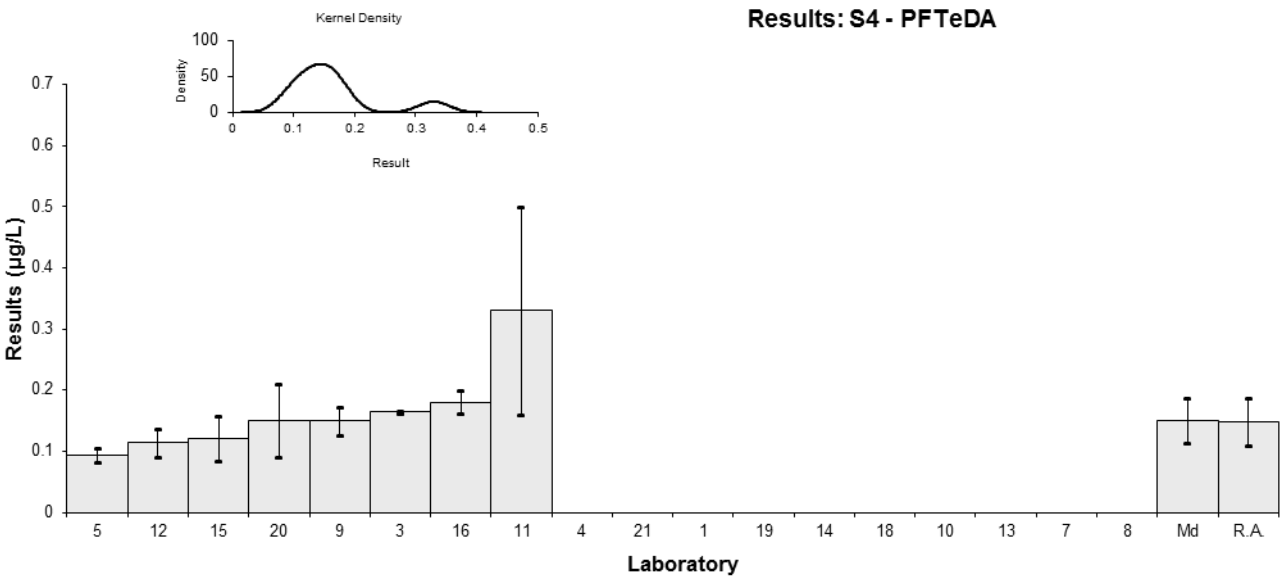

Results: S4 - PFTeDA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
5
12
15
20
9
3
16
11
4
21
1
19
14
18
10
13
7
8
Md
R.A.

Figure 30

Table 35

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	Total PFCA
Units	µg/L

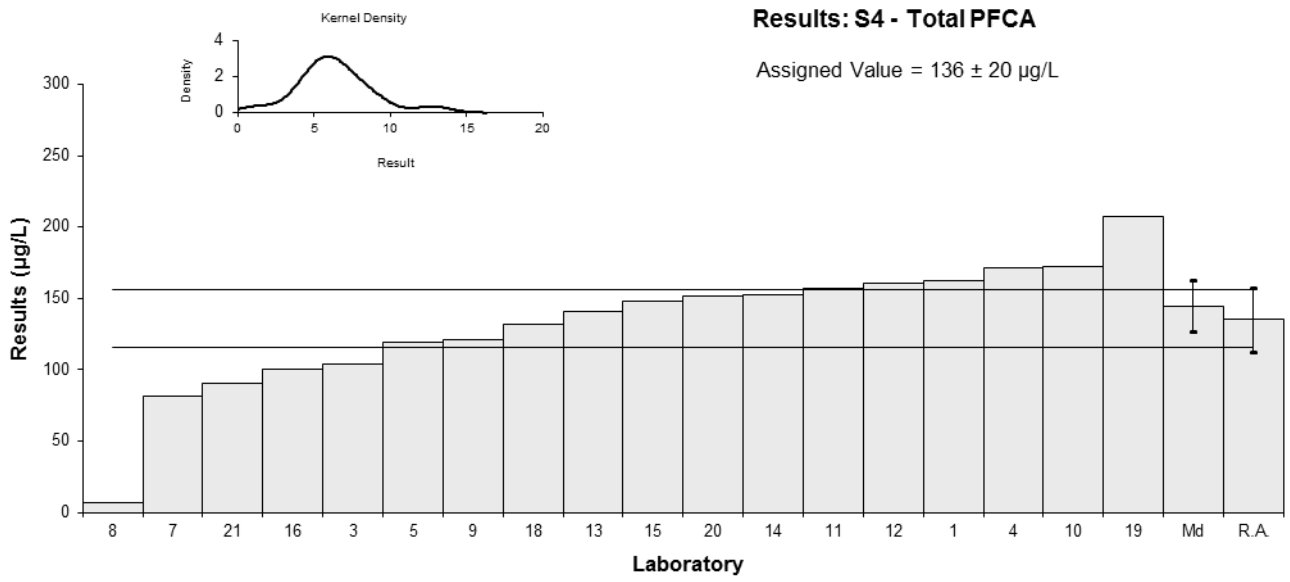
Participant Results

Lab Code	Result	z-Score
1	162.38	0.78
3	103.727	-0.95
4	171.454	1.04
5	118.828	-0.51
7	81.5	-1.60
8	6.565	-3.81
9	121.19	-0.44
10	172.44	1.07
11	156.9	0.61
12	160.31	0.72
13	140.88	0.14
14	152.51	0.49
15	148.269	0.36
16	100.02	-1.06
18	131.72	-0.13
19	207.719	2.11
20	151.37	0.45
21	90.292	-1.34

Statistics

Assigned Value*	136	20
Robust Average	135	23
Median	145	18
Mean	132	
N	18	
Max.	207.719	
Min.	6.565	
Robust SD	38	
Robust CV	28%	

*Robust Average excluding Laboratories 8 and 19


Kernel Density
Density
Result
Results: S4 - Total PFCA
Assigned Value = 136 ± 20 µg/L
Results (µg/L)
Laboratory
8 7 21 16 3 5 9 18 13 15 20 14 11 12 1 4 10 19 Md R.A.

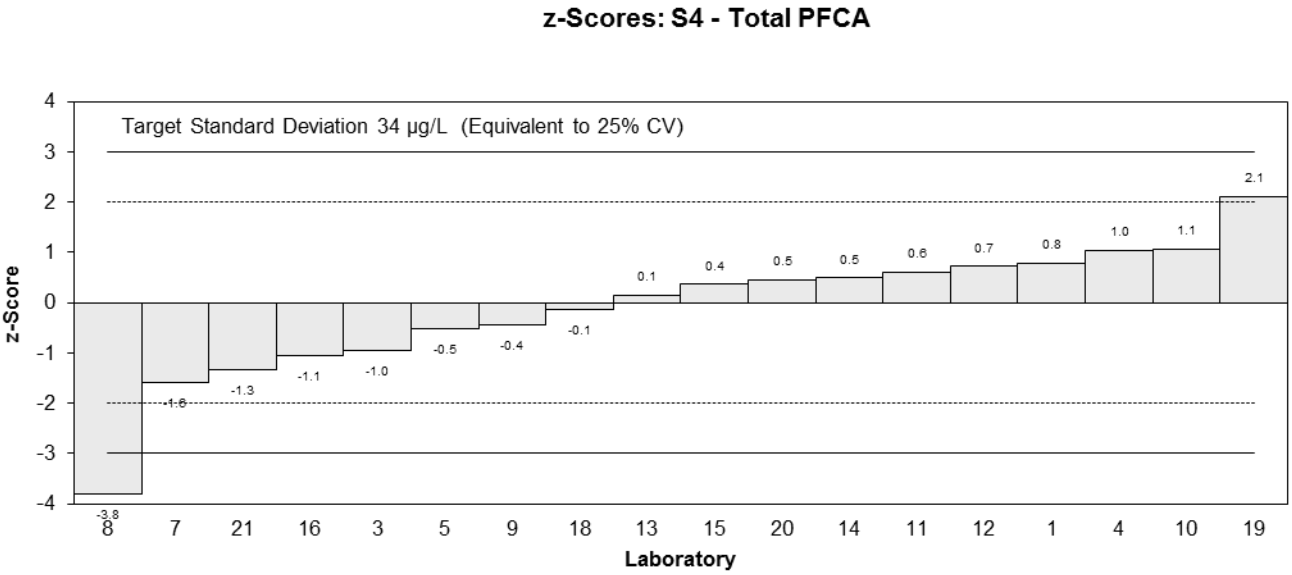

z-Scores: S4 - Total PFCA
Target Standard Deviation 34 µg/L (Equivalent to 25% CV)
z-Score
Laboratory
8 7 21 16 3 5 9 18 13 15 20 14 11 12 1 4 10 19
-3.8 -1.6 -1.3 -1.1 -1.0 -0.5 -0.4 -0.1 0.1 0.4 0.5 0.5 0.6 0.7 0.8 1.0 1.1 2.1

Figure 31

Table 36

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	6:2FTS
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 0.42	0.11	91
3	0.326	0.00499	101
4	< 0.1	NR	NR
5	0.031	0.003	48
7	<1.0	NR	128.42
8	29.8	10.5	NR
9	0.11	0.017	87
10	<5.0	1.0	132
11	<0.01	NR	100
12	NR	NR	NR
13	<0.2	NR	51
14	<0.02	NR	82
15	NR	NR	NR
16	0.08	0.033	96
18	<0.5	NR	246
19	<0.005	NR	97
20	< 0.05	0.015	NR
21	< 1.00	NR	108

Statistics

Assigned Value	Not Set	
Median	0.11	0.15
Mean	6.07	
N	5	
Max.	29.8	
Min.	0.031	

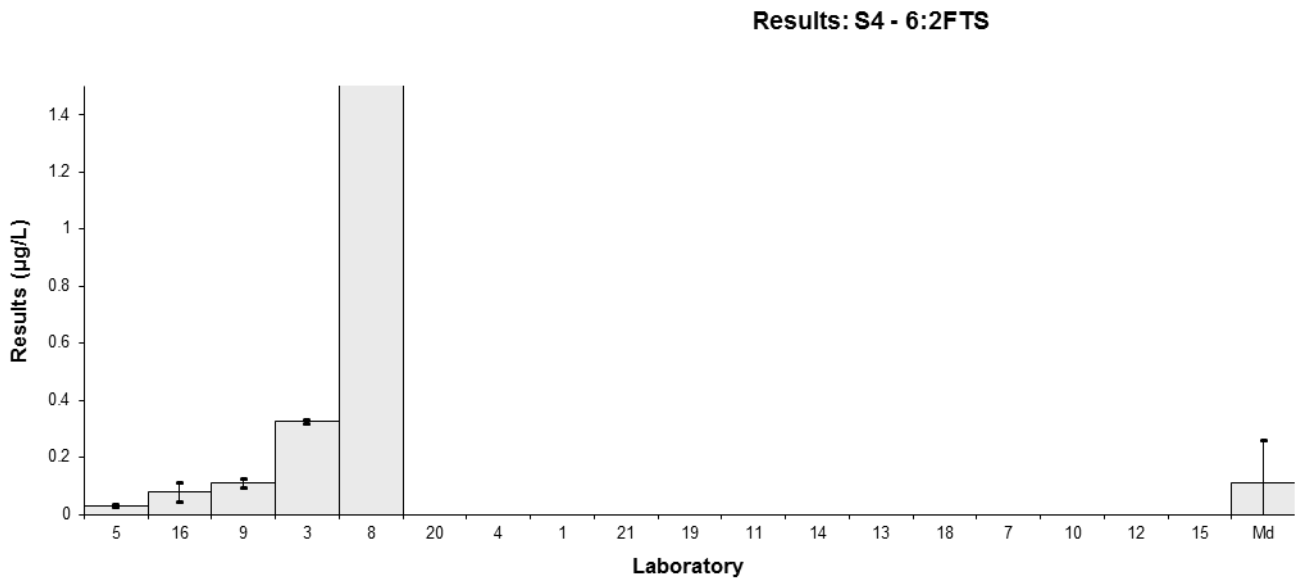

Results: S4 - 6:2FTS
Results (µg/L)
1.4
1.2
1
0.8
0.6
0.4
0.2
0
5
16
9
3
8
20
4
1
21
19
11
14
13
18
7
10
12
15
Md
Laboratory

Figure 32

Table 37

Sample Details

Sample No.	S4
Matrix.	MilliQ water, Alcoseal, PFDA, PFDS
Analyte.	8:2FTS
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 0.21	0.06	NR
3	0.0933	0.00160	96.3
4	< 0.1	NR	NR
5	<0.002	0.002	30
7	<1.0	NR	128.42
8	7.25	2.39	NR
9	<0.02	NR	NR
10	<0.40	0.10	131
11	<0.01	NR	90
12	NR	NR	NR
13	<0.2	NR	54
14	<0.02	NR	NR
15	NR	NR	NR
16	<0.05	0.015	98
18	<0.5	NR	95
19	<0.005	NR	82
20	< 0.01	0.003	NR
21	< 1.00	NR	96

Statistics*

Assigned Value	Not Set	
Mean	3.67	
N	2	

*Insufficient data to calculate statistics

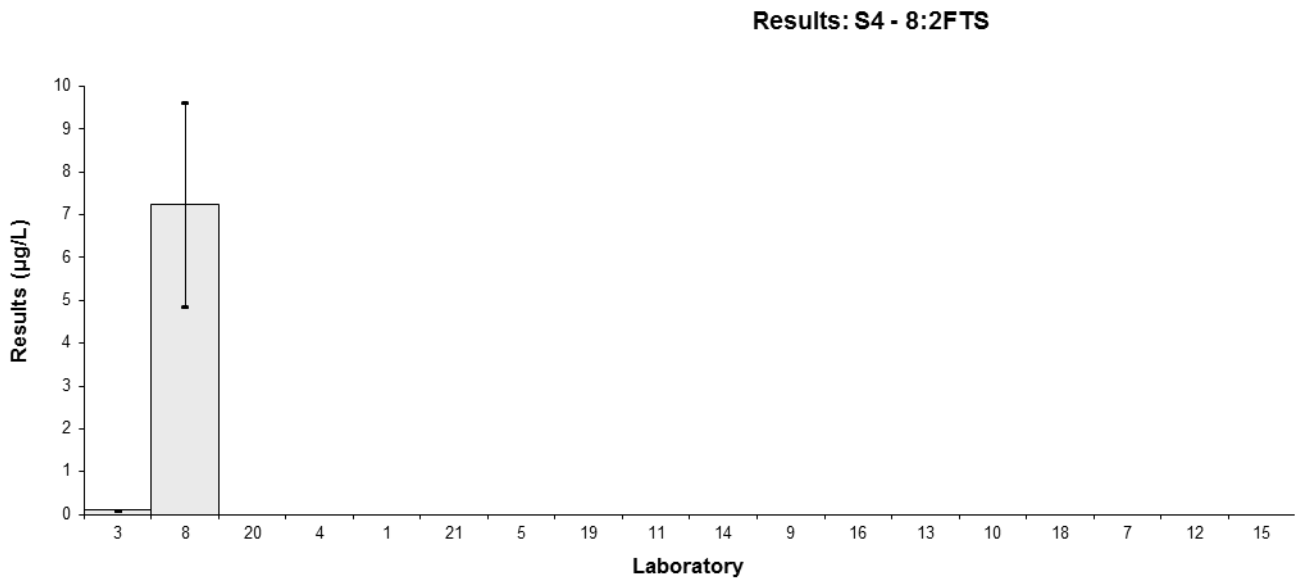

Results: S4 - 8:2FTS
Results (µg/L)
10
9
8
7
6
5
4
3
2
1
0
3
8
20
4
1
21
5
19
11
14
9
16
13
10
18
7
12
15
Laboratory

Figure 33

Table 38

Sample Details

Sample No.	S5
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFDS
Units	µg/L

Participant Results

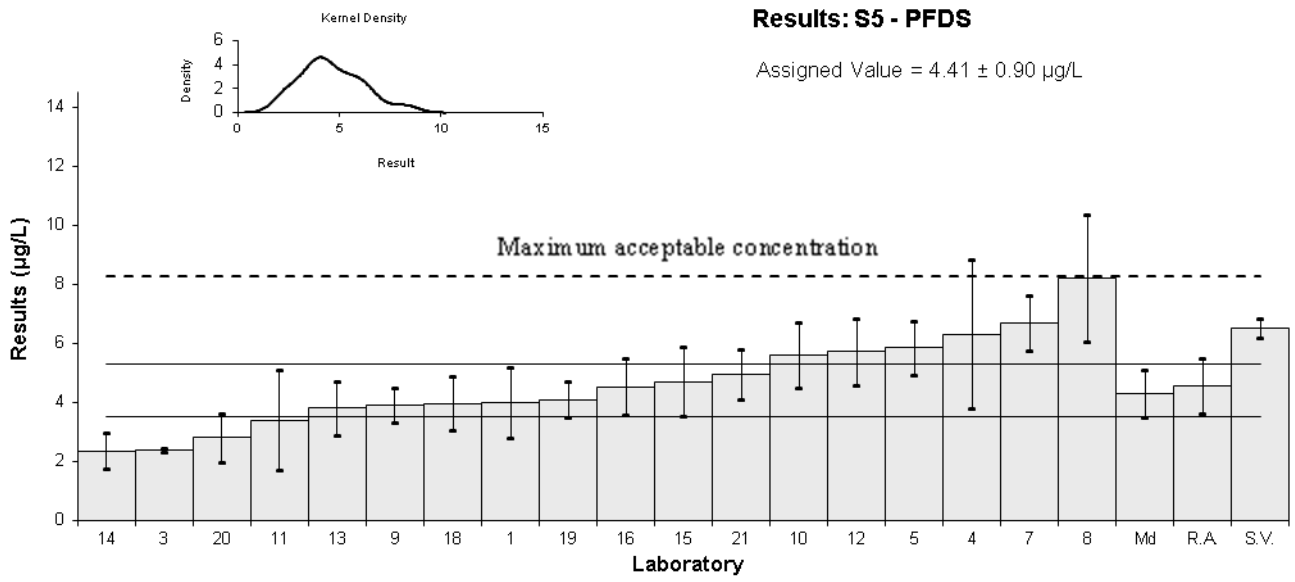
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	4.0	1.2	NR	-0.46	-0.27
3	2.38	0.0507	103	-2.30	-2.25
4*	6.303	2.521	84	2.00	0.71
5	5.85	0.92	NR	1.63	1.12
7*	6.68	0.94	74.23	2.00	1.00
8*	8.22	2.14	NR	2.00	1.00
9	3.9	0.59	94	-0.58	-0.47
10	5.60	1.1	99	1.35	0.84
11	3.4	1.7	110	-1.15	-0.53
12	5.72	1.14	81	1.49	0.90
13	3.8	0.9	102	-0.69	-0.48
14	2.35	0.61	116	-2.34	-1.89
15	4.71	1.175	76	0.34	0.20
16	4.53	0.944	NR	0.14	0.09
18	3.96	0.903	83	-0.51	-0.35
19	4.087	0.61305	NR	-0.37	-0.30
20	2.8	0.84	NR	-1.83	-1.31
21	4.95	0.85	94	0.61	0.44

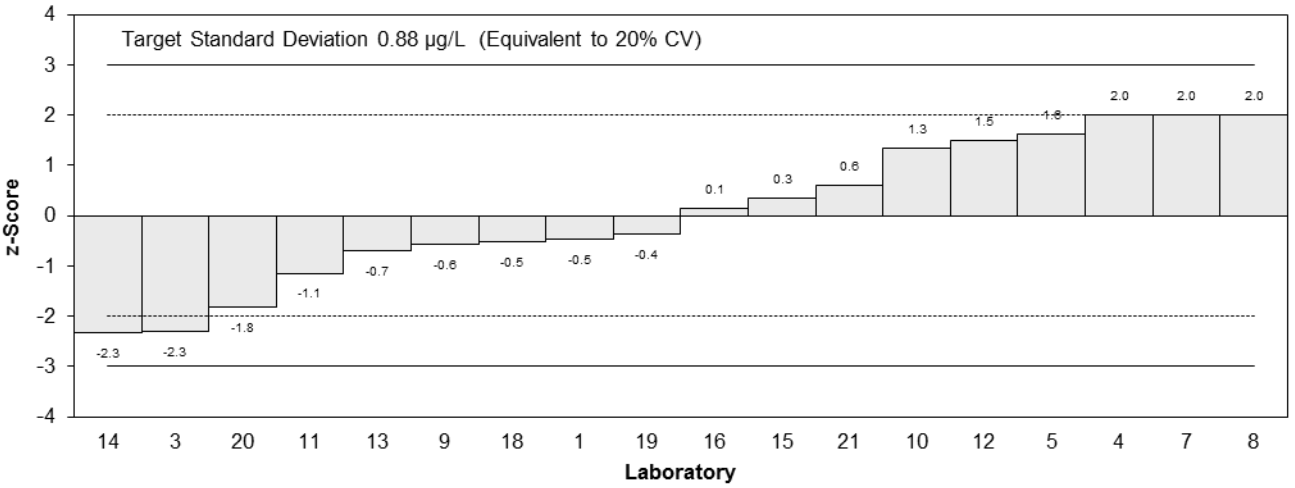
Statistics

Assigned Value**	4.41	0.90
Spike	6.50	0.33
Robust Average	4.55	0.94
Median	4.31	0.81
Mean	4.62	
N	18	
Max.	8.22	
Min.	2.35	
Robust SD	1.6	
Robust CV	35%	

*z-Score adjusted to 2 and E_n score adjusted to 1 (see Section 6.3).

**Robust Average excluding Laboratory 8.


Kernel Density
Density
Result
Results: S5 - PFDS
Assigned Value = 4.41 ± 0.90 µg/L
Maximum acceptable concentration
Results (µg/L)
Laboratory
14 3 20 11 13 9 18 1 19 16 15 21 10 12 5 4 7 8 Md R.A. S.V.


z-Scores: S5 - PFDS
Target Standard Deviation 0.88 µg/L (Equivalent to 20% CV)
z-Score
Laboratory
14 3 20 11 13 9 18 1 19 16 15 21 10 12 5 4 7 8
-2.3 -2.3 -1.8 -1.1 -0.7 -0.6 -0.5 -0.5 -0.4 0.1 0.3 0.6 1.3 1.5 1.6 2.0 2.0 2.0

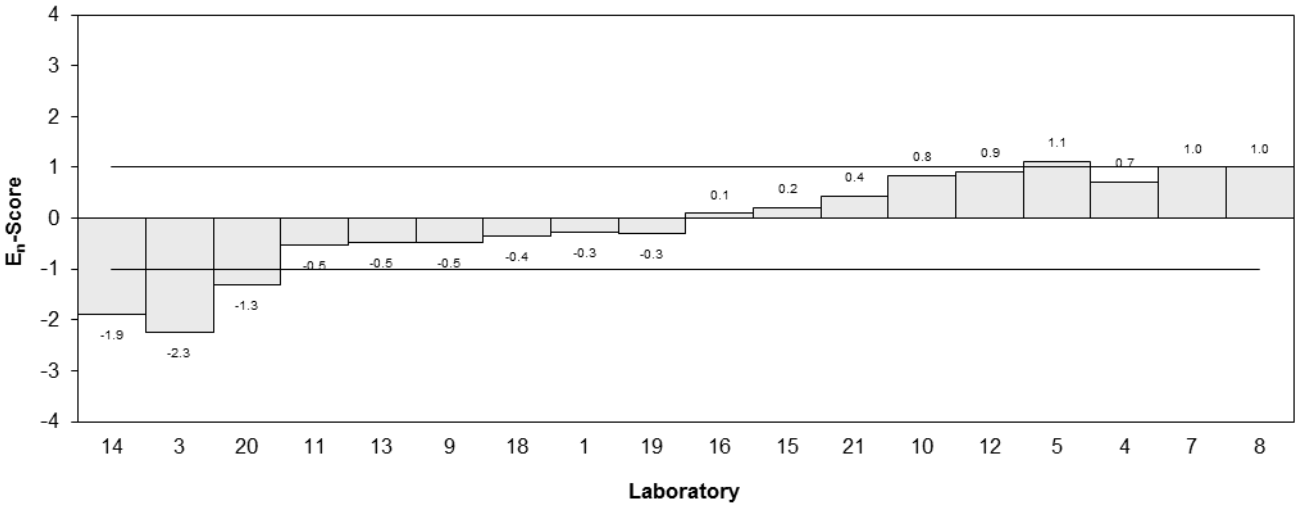

En-Scores: S5 - PFDS
En-Score
Laboratory
14 3 20 11 13 9 18 1 19 16 15 21 10 12 5 4 7 8
-1.9 -2.3 -1.3 -0.5 -0.5 -0.5 -0.4 -0.3 -0.3 0.1 0.2 0.4 0.8 0.9 1.1 0.7 1.0 1.0

Figure 34

Table 39

Sample Details

Sample No.	S5
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFNA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 0.29	0.08	101
3	0.0495	0.00102	100
4	0.0706	0.0212	75
5	0.069	0.006	106
7	0.046	0.009	74.23
8	0.087	0.018	NR
9	0.058	0.0087	127
10	0.07	0.01	97
11	0.06	0.03	90
12	0.0497	0.0149	77
13	0.06	0.04	109
14	<0.02	NR	110
15	0.0562	0.017	82
16	0.06	0.013	73
18	0.08	0.007	68
19	0.064	0.00384	75
20	0.13	0.0325	NR
21	< 0.10	NR	99

Statistics

Assigned Value	No Set	
Robust Average	0.0642	0.0091
Median	0.0600	0.0082
Mean	0.0673	
N	15	
Max.	0.13	
Min.	0.046	
Robust SD	0.014	
Robust CV	22%	

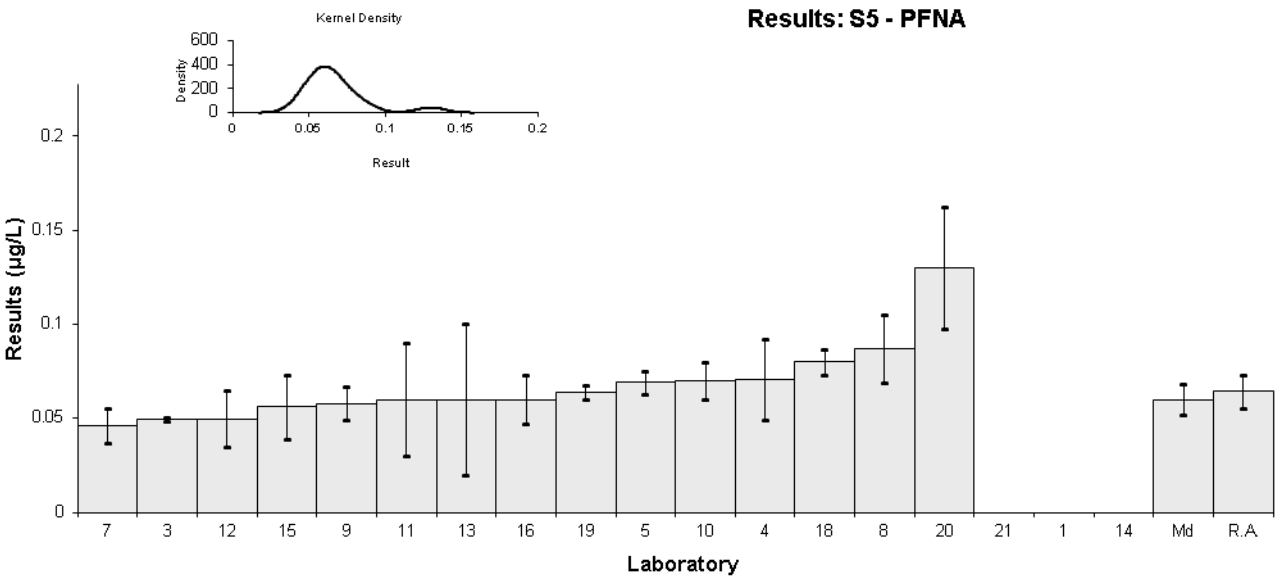

Results: S5 - PFNA
Kernel Density
Density
600
400
200
0
0
0.05
0.1
0.15
0.2
Result
Results (µg/L)
0.2
0.15
0.1
0.05
0
7
3
12
15
9
11
13
16
19
5
10
4
18
8
20
21
1
14
Md
R.A
Laboratory

Figure 35

Table 40

Sample Details

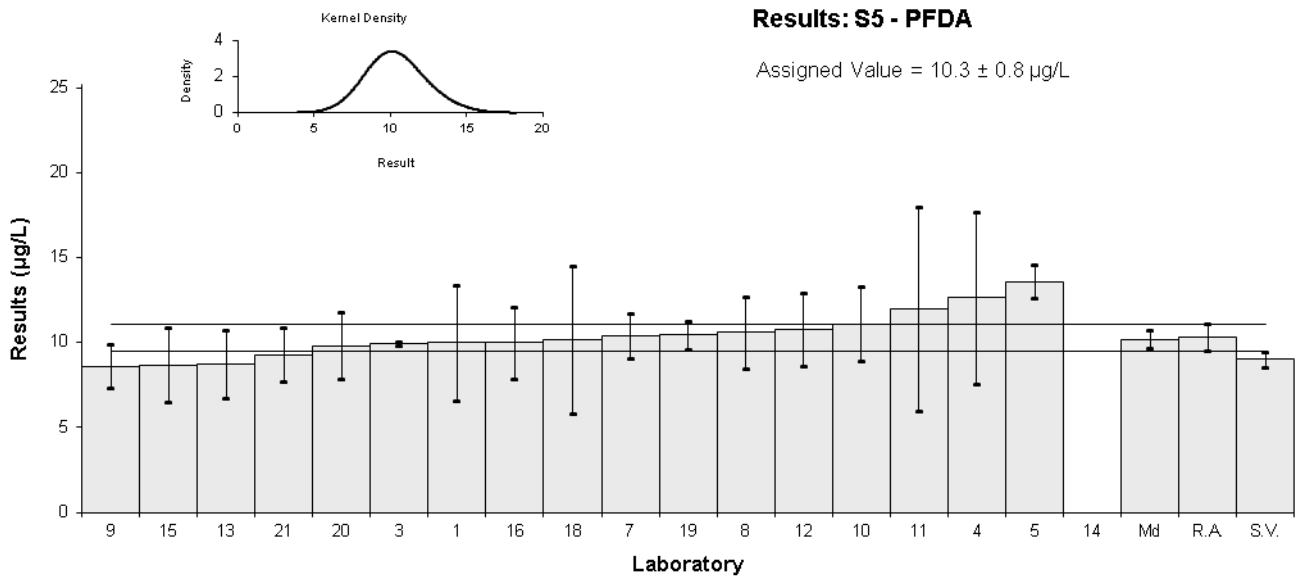
Sample No.	S5
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFDA
Units	µg/L

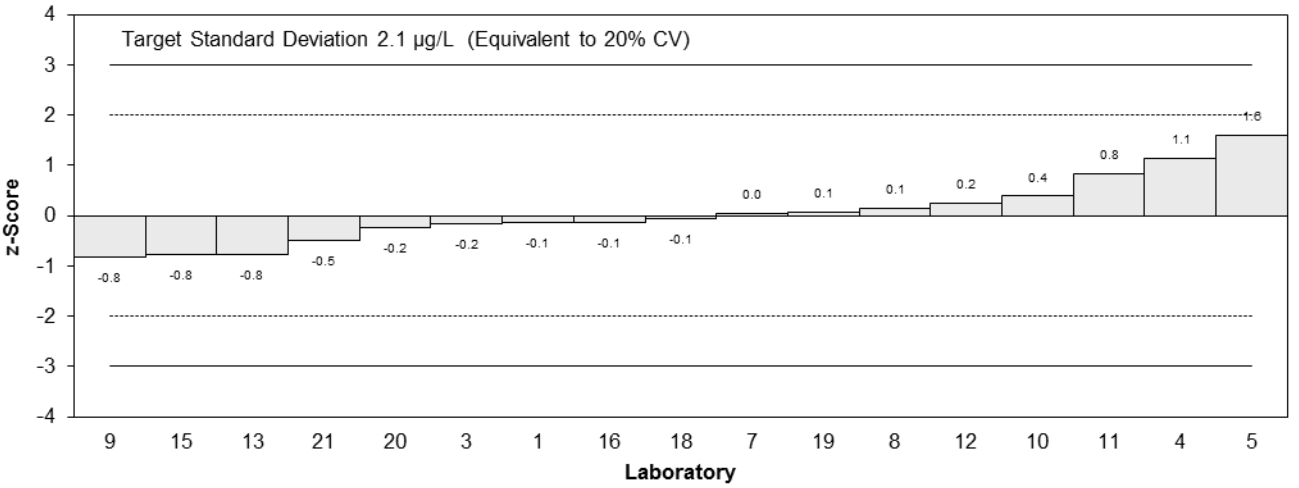
Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	10	3.4	103	-0.15	-0.09
3	9.95	0.126	110	-0.17	-0.43
4	12.64	5.056	100	1.14	0.46
5	13.6	1.0	83	1.60	2.58
7	10.4	1.3	74.23	0.05	0.07
8	10.6	2.1	NR	0.15	0.13
9	8.6	1.3	97	-0.83	-1.11
10	11.1	2.2	94	0.39	0.34
11	12	6	100	0.83	0.28
12	10.8	2.16	79	0.24	0.22
13	8.7	2	109	-0.78	-0.74
14	<0.02	NR	91		
15	8.6900	2.173	83	-0.78	-0.70
16	10.0	2.10	82	-0.15	-0.13
18	10.18	4.337	12	-0.06	-0.03
19	10.445	0.8356	60	0.07	0.13
20	9.8	1.96	NR	-0.24	-0.24
21	9.29	1.59	100	-0.49	-0.57

Statistics

Assigned Value	10.3	0.8
Spike	9.01	0.45
Robust Average	10.3	0.8
Median	10.2	0.5
Mean	10.4	
N	17	
Max.	13.6	
Min.	8.6	
Robust SD	1.3	
Robust CV	13%	


Kernel Density
Density
Result
Results: S5 - PFDA
Assigned Value = 10.3 ± 0.8 µg/L
Results (µg/L)
Laboratory
9 15 13 21 20 3 1 16 18 7 19 8 12 10 11 4 5 14 Md R.A S.V.


z-Scores: S5 - PFDA
Target Standard Deviation 2.1 µg/L (Equivalent to 20% CV)
z-Score
Laboratory
9 15 13 21 20 3 1 16 18 7 19 8 12 10 11 4 5
-0.8 -0.8 -0.8 -0.5 -0.2 -0.2 -0.1 -0.1 -0.1 0.0 0.1 0.1 0.2 0.4 0.8 1.1 1.6

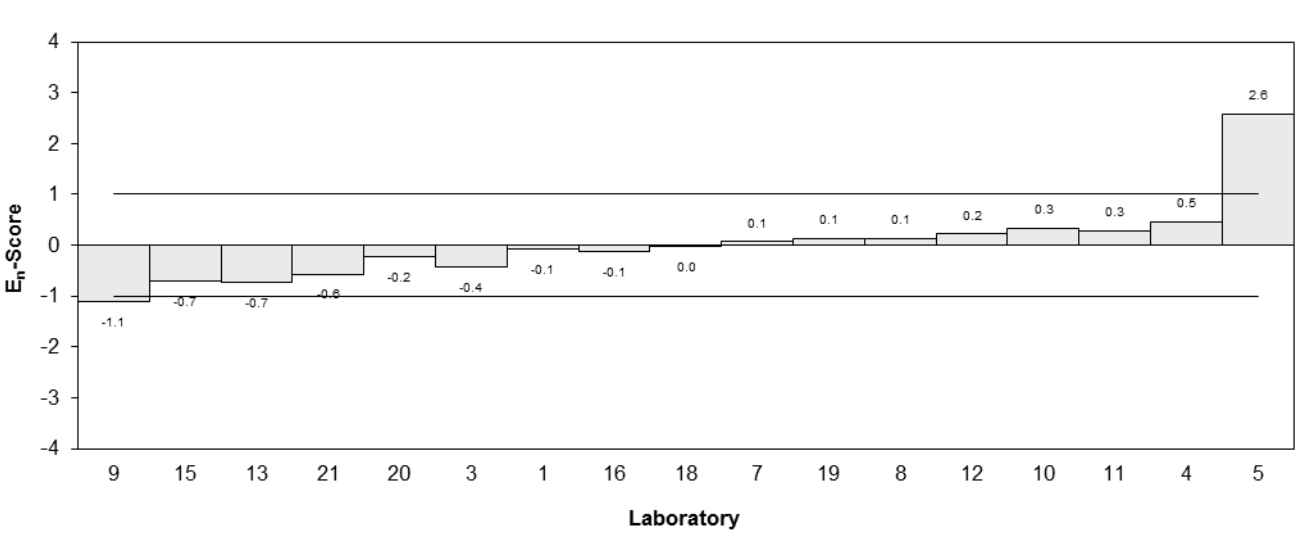

En-Scores: S5 - PFDA
En-Score
Laboratory
9 15 13 21 20 3 1 16 18 7 19 8 12 10 11 4 5
-1.1 -0.7 -0.7 -0.6 -0.2 -0.4 -0.1 -0.1 0.0 0.1 0.1 0.1 0.2 0.3 0.3 0.5 2.6

Figure 36

Table 41

Sample Details

Sample No.	S5
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	6:2 FTS
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	3.8	0.95	193
3	4.37	0.0669	83.8
4	4.992	1.997	338
5	2.82	0.15	143
7	4.26	0.53	72.60
8	6.49	2.27	NR
9	3.8	0.57	86
10	4.51	0.90	97
11	3.6	1.8	100
12	4.32	0.864	81
13	4.3	0.7	158
14	16.4	5.41	100
15	4.31	0.86	77
16	3.83	0.822	45
18	3.57	1.514	45
19	4.009	0.28063	70
20	4.6	1.38	NR
21	3.67	2.14	102

Statistics

Assigned Value	Not set	
Robust Average	4.19	0.36
Median	4.28	0.34
Mean	4.87	
N	18	
Max.	16.4	
Min.	2.82	
Robust SD	0.62	
Robust CV	15%	

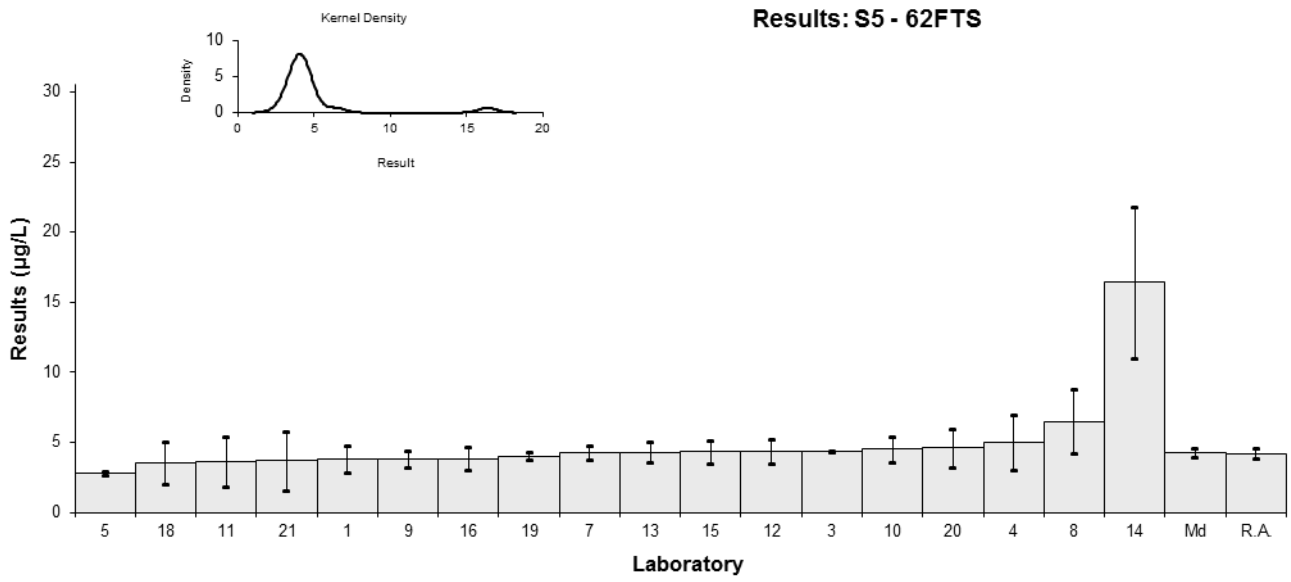

Results: S5 - 62FTS
Kernel Density
Density
Result
Results (µg/L)
Laboratory
5
18
11
21
1
9
16
19
7
13
15
12
3
10
20
4
8
14
Md
R.A.

Figure 37

Table 42

Sample Details

Sample No.	S5
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	8:2FTS
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 0.21	0.06	NR
3	NR	NR	55.4
4	< 0.10	NR	NR
5	0.019	0.004	111
7	<0.015	NR	72.75
8	NR	NR	NR
9	<0.02	NR	NR
10	<0.04	0.01	77
11	0.03	0.015	90
12	0.0120	0.00600	81
13	0.01	0.01	119
14	<0.02	NR	92
15	0.0075	0.00264	NR
16	<0.05	0.018	66
18	<0.05	NR	87
19	0.028	0.0028	89
20	< 0.1	0.03	NR
21	< 1.00	NR	98

Statistics

Assigned Value	Not set	
Robust Average	0.018	0.011
Median	0.016	0.011
Mean	0.018	
N	6	
Max.	0.03	
Min.	0.0075	
Robust SD	0.011	
Robust CV	61%	

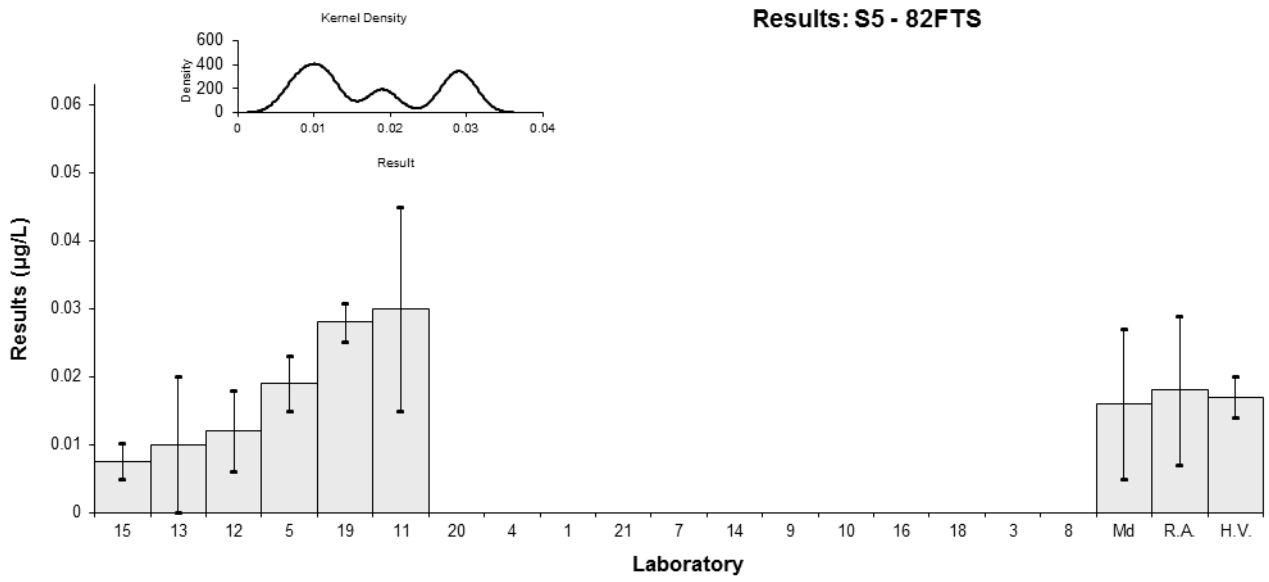

Results: S5 - 82FTS
Kernel Density
Density
600
400
200
0
0
0.01
0.02
0.03
0.04
Result
Results (µg/L)
0.06
0.05
0.04
0.03
0.02
0.01
0
15
13
12
5
19
11
20
4
1
21
7
14
9
10
16
18
3
8
Md
R.A.
H.V.
Laboratory

Figure 38

Table 43

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFDS
Units	µg/L

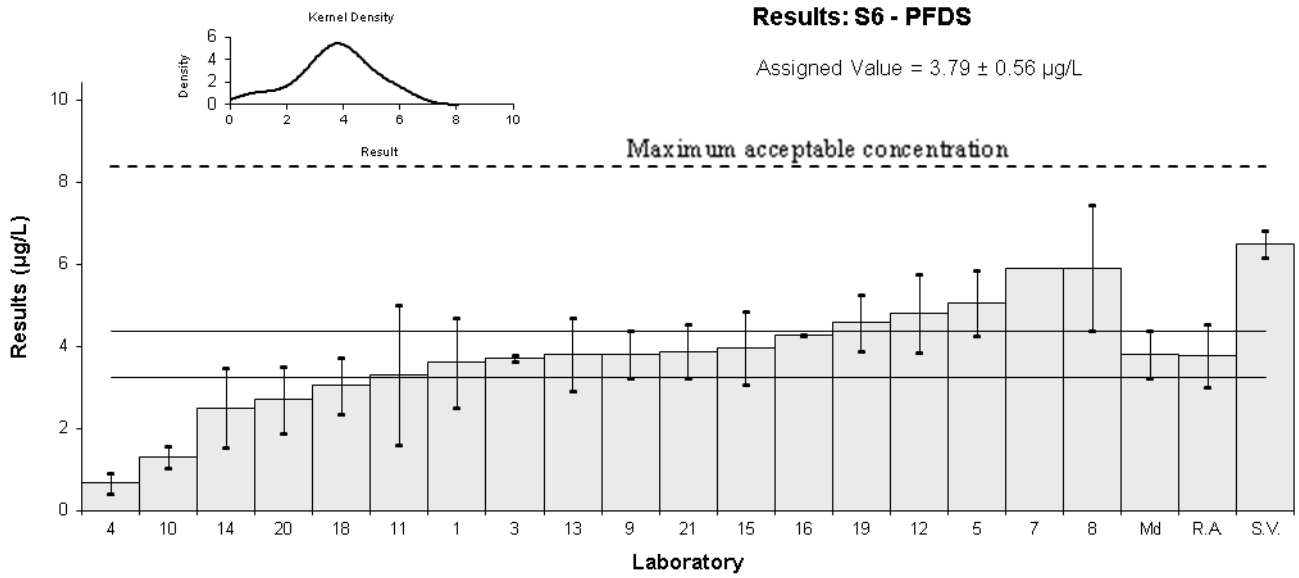
Participant Results

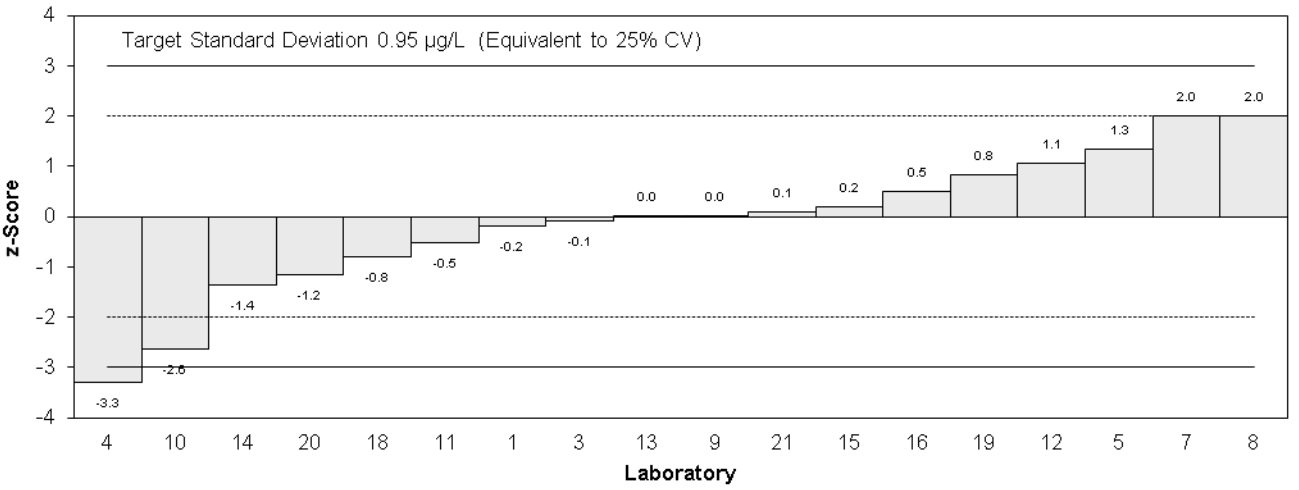
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	3.6	1.1	NR	-0.20	-0.15
3	3.70	0.0788	97.3	-0.09	-0.16
4	0.660	0.264	79	-3.30	-5.06
5	5.06	0.80	NR	1.34	1.30
7*	5.9	NR	NR	2.00	1.00
8*	5.91	1.54	NR	2.00	1.00
9	3.8	0.57	101	0.01	0.01
10	1.30	0.26	104	-2.63	-4.03
11	3.3	1.7	110	-0.52	-0.27
12	4.8	0.961	72	1.07	0.91
13	3.8	0.9	95	0.01	0.01
14	2.50	0.98	87	-1.36	-1.14
15	3.9720	0.893	76	0.19	0.17
16	4.27	0.006	NR	0.51	0.86
18	3.04	0.693	92	-0.79	-0.84
19	4.573	0.68595	NR	0.83	0.88
20	2.7	0.81	NR	-1.15	-1.11
21	3.87	0.66	107	0.08	0.09

Statistics

Assigned Value**	3.79	0.56
Spike	6.50	0.33
Robust Average	3.78	0.77
Median	3.80	0.57
Mean	3.71	
N	18	
Max.	5.91	
Min.	0.66	
Robust SD	1.3	
Robust CV	34%	

*z-Score adjusted to 2 and E_n score adjusted to 1 (see Section 6.3).
**Robust Average excluding Laboratories 4, 7, 8 and 10.


Kernel Density
Density
Result
Results: S6 - PFDS
Assigned Value = 3.79 ± 0.56 µg/L
Maximum acceptable concentration
Results (µg/L)
Laboratory
4 10 14 20 18 11 1 3 13 9 21 15 16 19 12 5 7 8 Md R.A S.V.


z-Scores: S6 - PFDS
Target Standard Deviation 0.95 µg/L (Equivalent to 25% CV)
z-Score
Laboratory
4 10 14 20 18 11 1 3 13 9 21 15 16 19 12 5 7 8
-3.3 -2.6 -1.4 -1.2 -0.8 -0.5 -0.2 -0.1 0.0 0.0 0.1 0.2 0.5 0.8 1.1 1.3 2.0 2.0

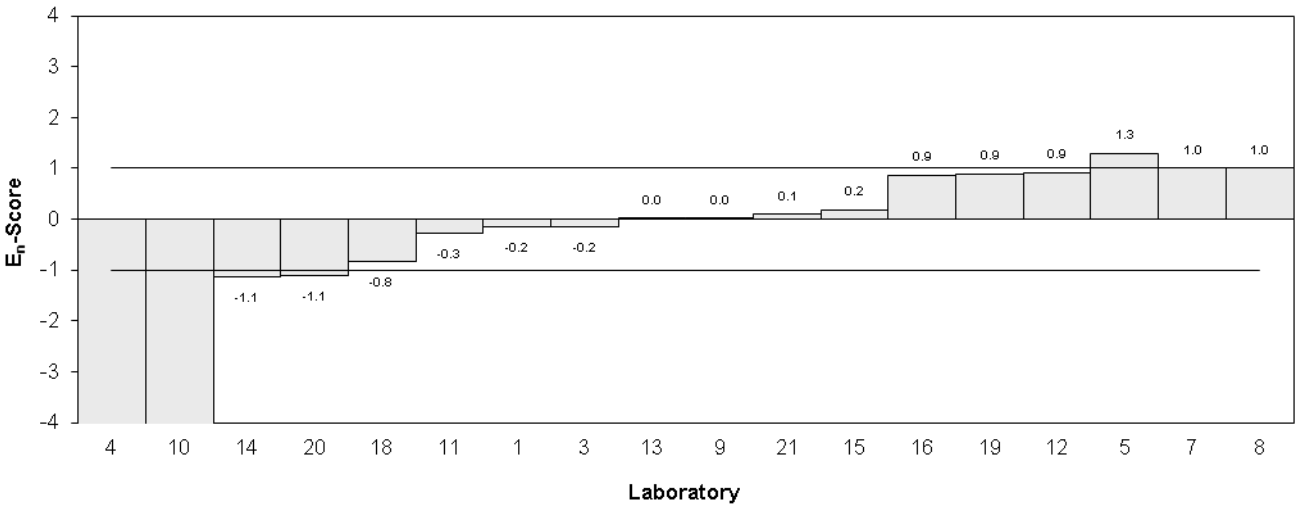

En-Scores: S6 - PFDS
En-Score
Laboratory
4 10 14 20 18 11 1 3 13 9 21 15 16 19 12 5 7 8
-1.1 -1.1 -0.8 -0.3 -0.2 -0.2 0.0 0.0 0.1 0.2 0.9 0.9 0.9 1.3 1.0 1.0

Figure 39

Table 44

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFBA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	29	11	70
3	23.8	0.321	102
4	48.57	19.43	71
5	22.7	0.9	71
7	<110	NR	NR
8	0.89	0.18	NR
9	21	3.2	102
10	36.4	7.28	100
11	28	14	90
12	28.2	5.64	68
13	24	7	51
14	36.4	13.8	68
15	28.096	7.024	83
16	17.4	2.71	92
18	15.49	2.252	11
19	36.721	1.46884	68
20	24	4.8	NR
21	16.8	2.07	96

Statistics

Assigned Value	Not Set	
Robust Average	25.9	5.6
Median	24.0	3.8
Mean	25.7	
N	17	
Max.	48.57	
Min.	0.89	
Robust SD	9.2	
Robust CV	36%	

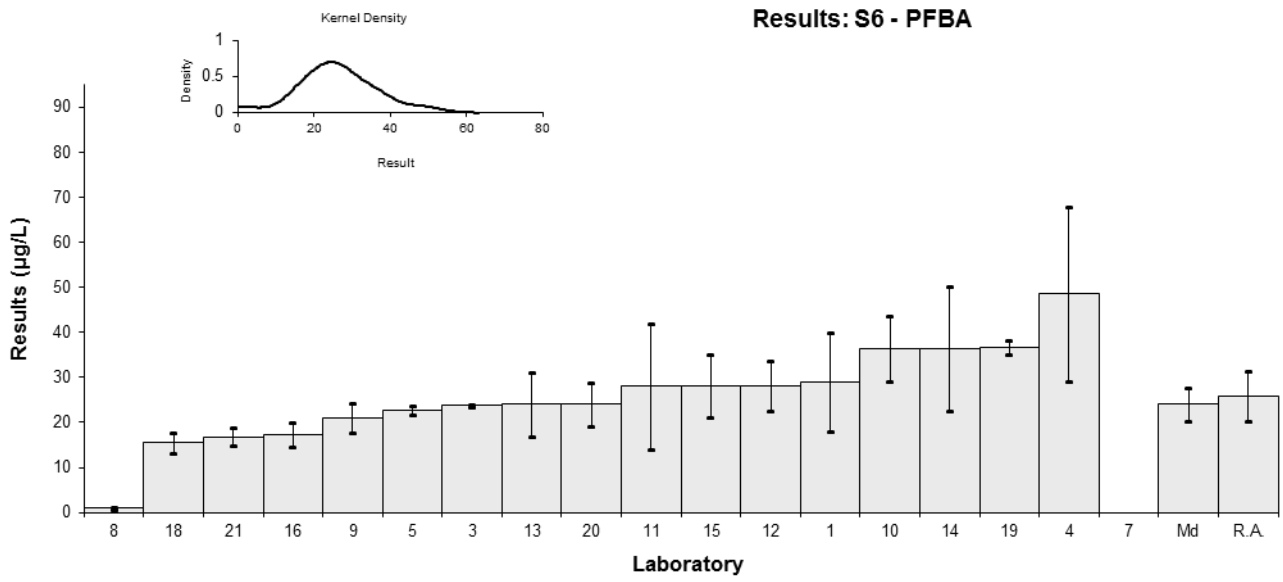

Results: S6 - PFBA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
8
18
21
16
9
5
3
13
20
11
15
12
1
10
14
19
4
7
Md
R.A.

Figure 40

Table 45

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFPeA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	54	23	74
3	33.3	0.5361	87.2
4	103.1	41.24	57
5	57.7	2.9	63
7	77.2	NR	NR
8	0.81	0.17	NR
9	41	6.2	117
10	89.9	18.0	104
11	67	34	100
12	84.3	16.9	65
13	51	10	46
14	55.5	20.5	100
15	71.841	17.960	81
16	31.4	3.78	88
18	35.78	2.720	63
19	91.629	1.83258	79
20	40	8.8	NR
21	35.9	3.88	91

Statistics

Assigned Value	Not Set	
Robust Average	57	16
Median	55	14
Mean	56.7	
N	18	
Max.	103.1	
Min.	0.81	
Robust SD	27	
Robust CV	47%	

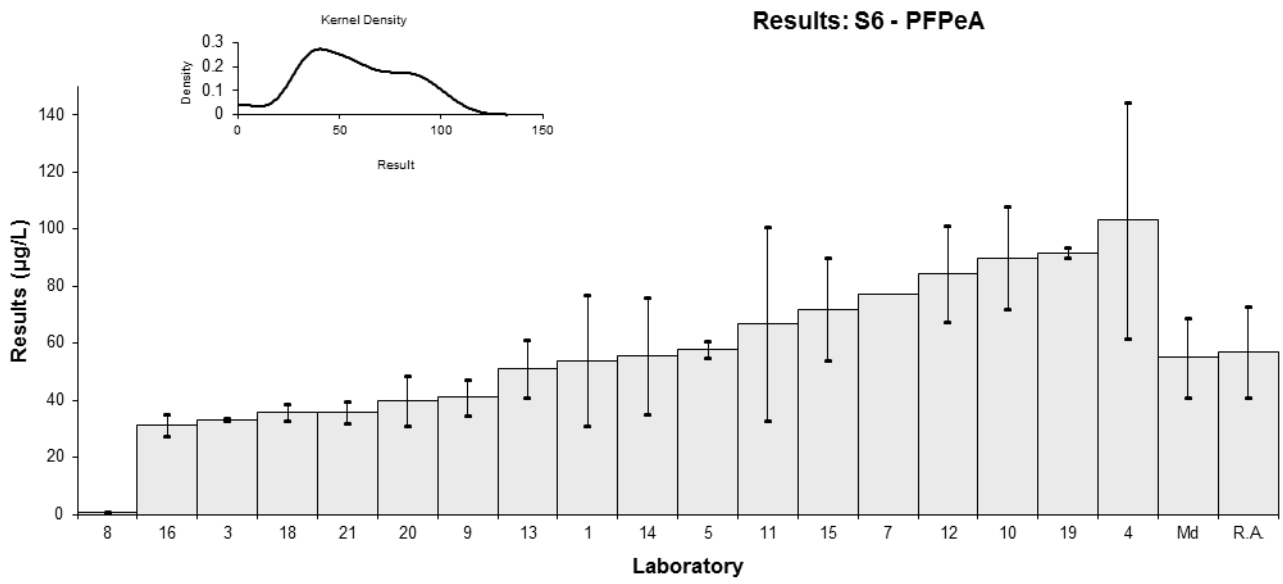

Results: S6 - PFPeA
Kernel Density
Density
0.3
0.2
0.1
0
0
50
100
150
Result
Results (µg/L)
140
120
100
80
60
40
20
0
8
16
3
18
21
20
9
13
1
14
5
11
15
7
12
10
19
4
Md
R.A.
Laboratory

Figure 41

Table 46

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFHxA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	12	3.0	90
3	11.5	0.232	101
4	27.16	8.148	57
5	14.0	0.6	60
7	20.1	NR	NR
8	1.16	0.26	NR
9	10	1.5	103
10	36.9	7.38	98
11	11	6	90
12	24.0	4.80	68
13	27	8	63
14	15.4	5.4	75
15	23.178	5.794	94
16	11.1	1.19	95
18	15.59	3.960	88
19	80.643	6.45144	60
20	10	0.3	NR
21	8.67	1.01	95

Statistics

Assigned Value	Not Set	
Robust Average	17.0	5.7
Median	14.7	3.7
Mean	20.0	
N	18	
Max.	80.6	
Min.	1.16	
Robust SD	9.7	
Robust CV	57%	

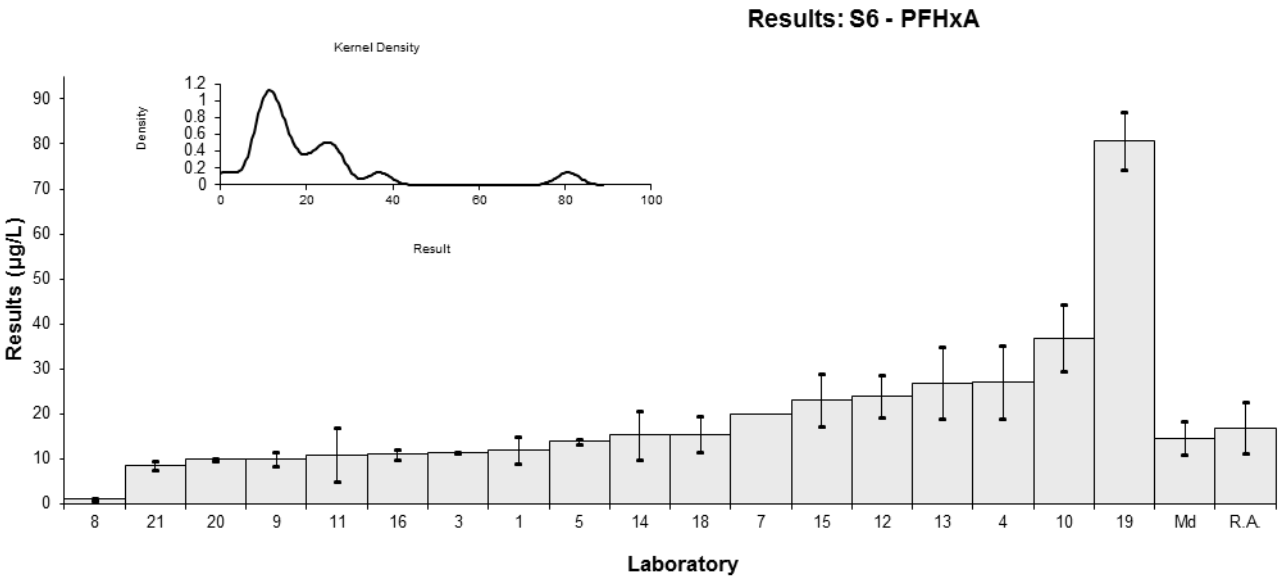

Results: S6 - PFHxA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
8
21
20
9
11
16
3
1
5
14
18
7
15
12
13
4
10
19
Md
R.A.

Figure 42

Table 47

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFHpA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	4.6	1.2	100
3	4.87	0.0765	121
4	12.72	3.816	57
5	5.06	0.22	59
7	7.3	NR	NR
8	0.12	0.03	NR
9	3.7	0.56	99
10	12.5	2.5	95
11	5.00	2.5	100
12	10.7	2.14	71
13	4.2	1	76
14	4.51	1.71	90
15	10.369	2.592	78
16	3.03	0.324	99
18	3.33	1.442	93
19	23.326	1.86608	81
20	3.7	0.925	NR
21	4.11	0.47	98

Statistics

Assigned Value	Not Set	
Robust Average	6.3	2.6
Median	4.74	0.90
Mean	6.84	
N	18	
Max.	23.326	
Min.	0.12	
Robust SD	4.4	
Robust CV	70%	

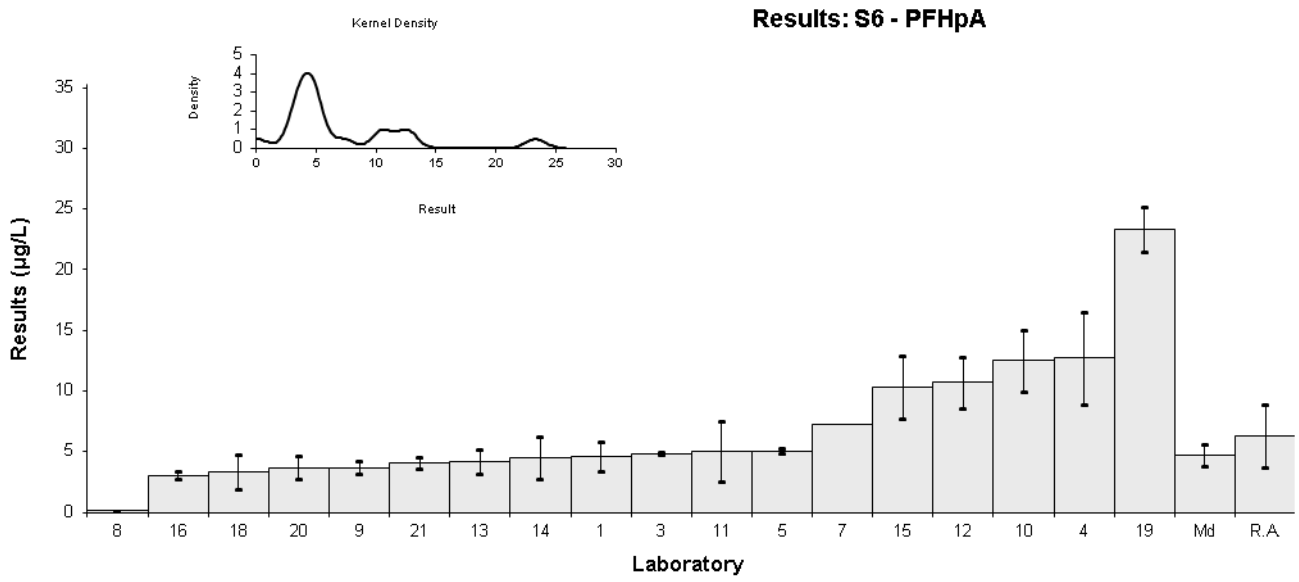

Results: S6 - PFHpA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
8
16
18
20
9
21
13
14
1
3
11
5
7
15
12
10
4
19
Md
R.A

Figure 43

Table 48

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFOA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	1.9	0.54	100
3	1.58	0.0240	78.7
4	1.897	0.569	69
5	1.63	0.06	64
7	2.6	NR	NR
8	0.22	0.05	104.3
9	1.8	0.27	94
10	1.93	0.39	97
11	1.6	0.8	90
12	2.33	0.466	75
13	2.6	0.8	69
14	1.61	0.58	90
15	2.481	0.620	84
16	2.37	0.718	104
18	1.72	0.525	109
19	7.331	0.58648	84
20	2	0.6	NR
21	0.977	0.144	102

Statistics

Assigned Value	Not Set	
Robust Average	1.95	0.33
Median	1.90	0.23
Mean	2.14	
N	18	
Max.	7.331	
Min.	0.22	
Robust SD	0.56	
Robust CV	28%	

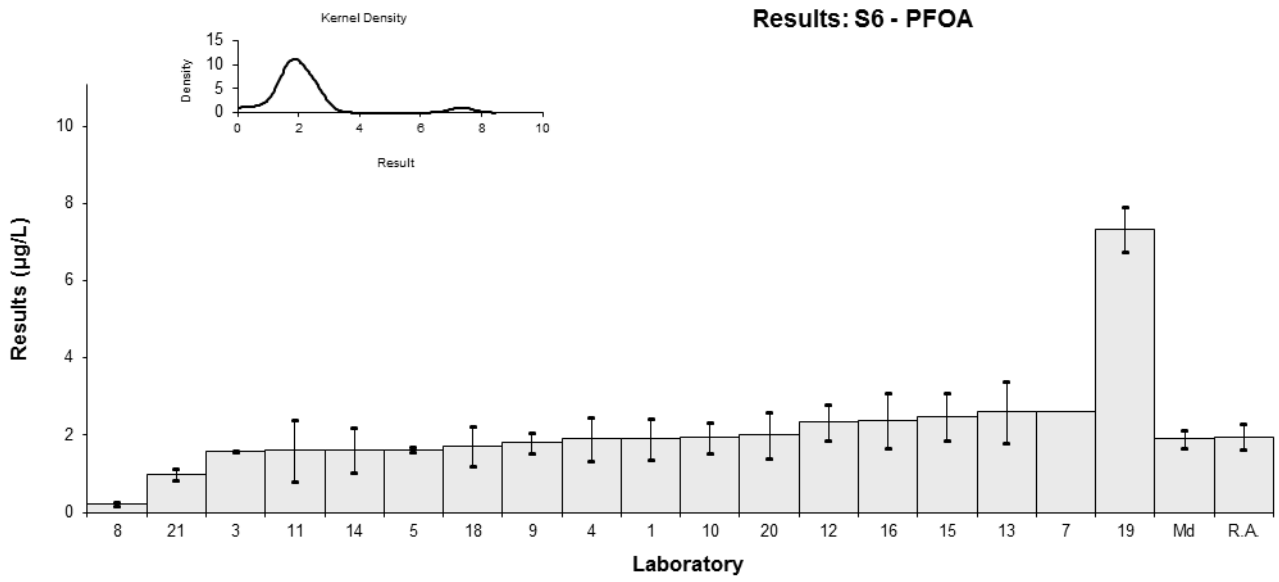

Results: S6 - PFOA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
8
21
3
11
14
5
18
9
4
1
10
20
12
16
15
13
7
19
Md
R.A.

Figure 44

Table 49

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFNA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	1.3	0.36	93
3	1.54	0.0317	93.8
4	0.864	0.259	82
5	1.29	0.12	76
7	1.4	NR	NR
8	0.11	0.03	NR
9	1.1	0.17	118
10	1.26	0.25	88
11	1.1	0.6	90
12	2.58	0.516	74
13	1.1	0.4	70
14	1.12	0.37	90
15	2.291	0.573	82
16	1.06	0.171	105
18	1.03	0.087	126
19	3.485	0.2091	87
20	1.2	0.3	NR
21	0.951	0.168	113

Statistics

Assigned Value	Not Set	
Robust Average	1.24	0.20
Median	1.16	0.10
Mean	1.38	
N	18	
Max.	3.485	
Min.	0.11	
Robust SD	0.34	
Robust CV	28%	

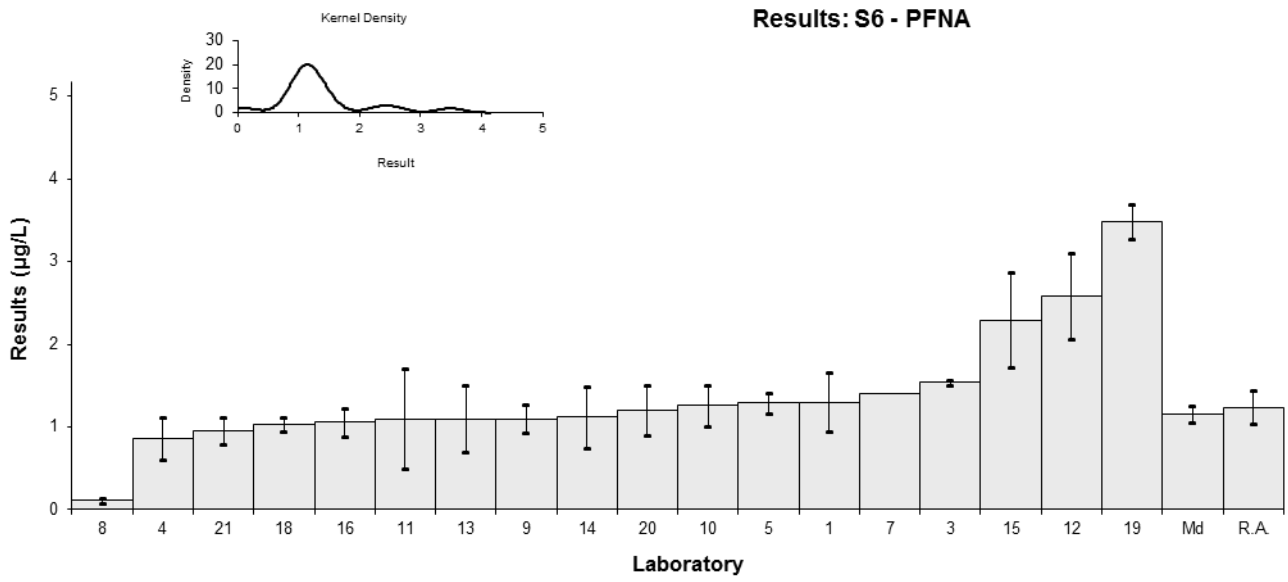

Results: S6 - PFNA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
8
4
21
18
16
11
13
9
14
20
10
5
1
7
3
15
12
19
Md
R.A.

Figure 45

Table 50

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFDA
Units	µg/L

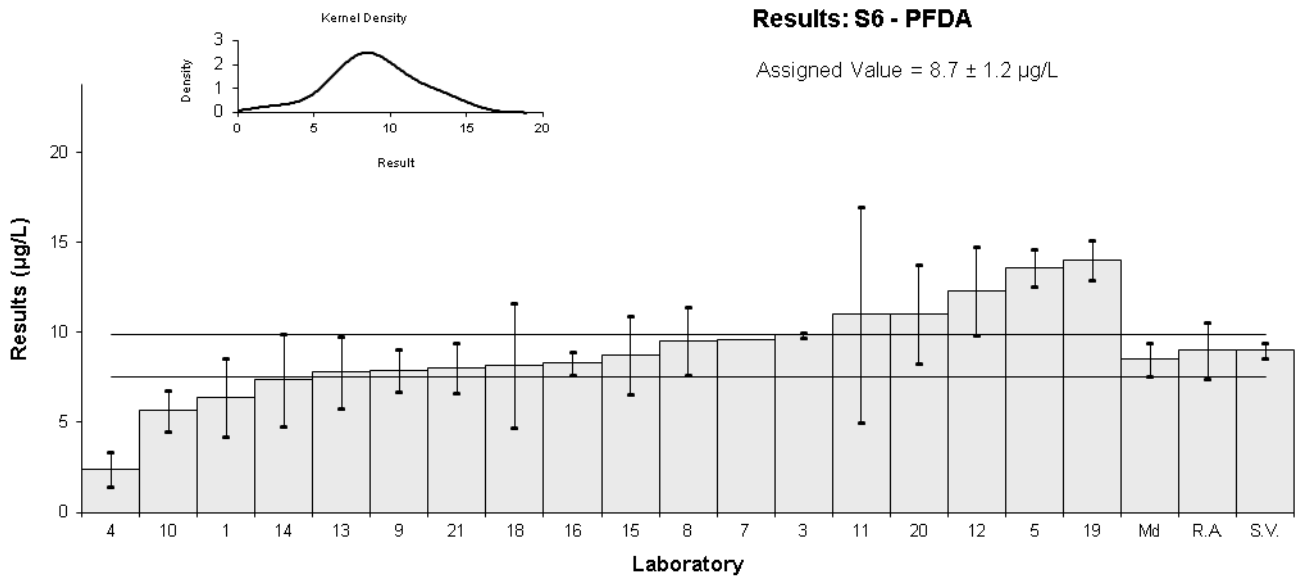
Participant Results

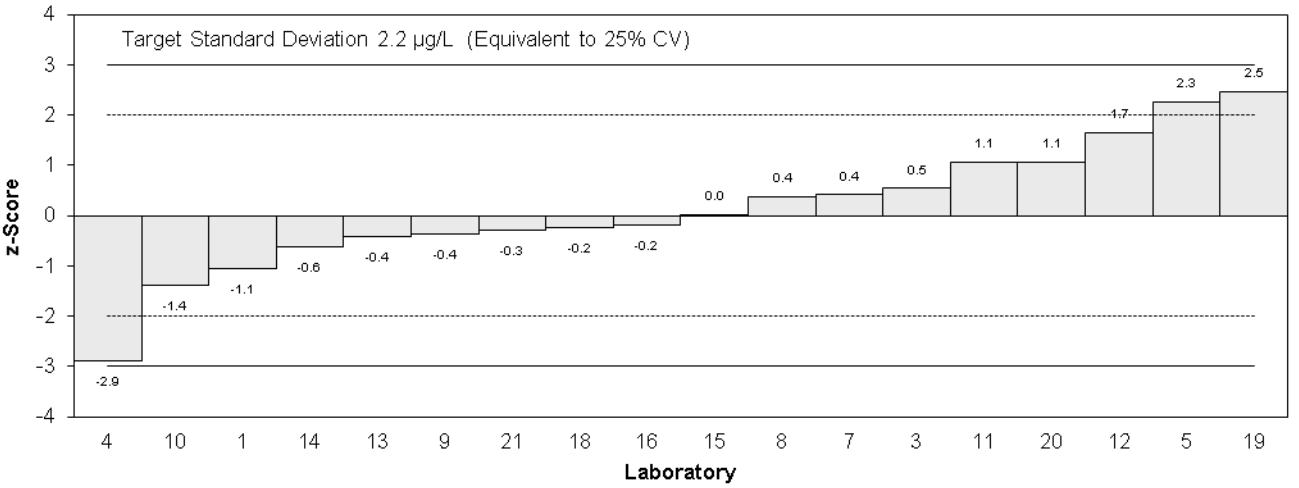
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	6.4	2.2	86	-1.06	-0.92
3	9.87	0.125	102	0.54	0.97
4	2.392	0.957	86	-2.90	-4.11
5	13.6	1.0	62	2.25	3.14
7	9.6	NR	NR	0.41	0.75
8	9.51	1.90	NR	0.37	0.36
9	7.9	1.2	116	-0.37	-0.47
10	5.66	1.13	105	-1.40	-1.84
11	11	6	100	1.06	0.38
12	12.3	2.46	69	1.66	1.32
13	7.8	2	76	-0.41	-0.39
14	7.36	2.58	81	-0.62	-0.47
15	8.723	2.181	83	0.01	0.01
16	8.30	0.637	106	-0.18	-0.29
18	8.16	3.476	105	-0.25	-0.15
19	14.046	1.12368	68	2.46	3.25
20	11	2.75	NR	1.06	0.77
21	8.05	1.38	104	-0.30	-0.36

Statistics

Assigned Value*	8.7	1.2
Spike	9.01	0.45
Robust Average	9.0	1.6
Median	8.51	0.93
Mean	8.98	
N	18	
Max.	14.046	
Min.	2.392	
Robust SD	2.7	
Robust CV	30%	

*Robust Average excluding Laboratories 4, 5 and 19.


Kernel Density
Density
Result
Results: S6 - PFDA
Assigned Value = 8.7 ± 1.2 µg/L
Results (µg/L)
Laboratory
4 10 1 14 13 9 21 18 16 15 8 7 3 11 20 12 5 19 Md R.A S.V.


z-Scores: S6 - PFDA
Target Standard Deviation 2.2 µg/L (Equivalent to 25% CV)
z-Score
Laboratory
4 10 1 14 13 9 21 18 16 15 8 7 3 11 20 12 5 19
-2.9 -1.4 -1.1 -0.6 -0.4 -0.4 -0.3 -0.2 -0.2 0.0 0.4 0.4 0.5 1.1 1.1 1.7 2.3 2.5

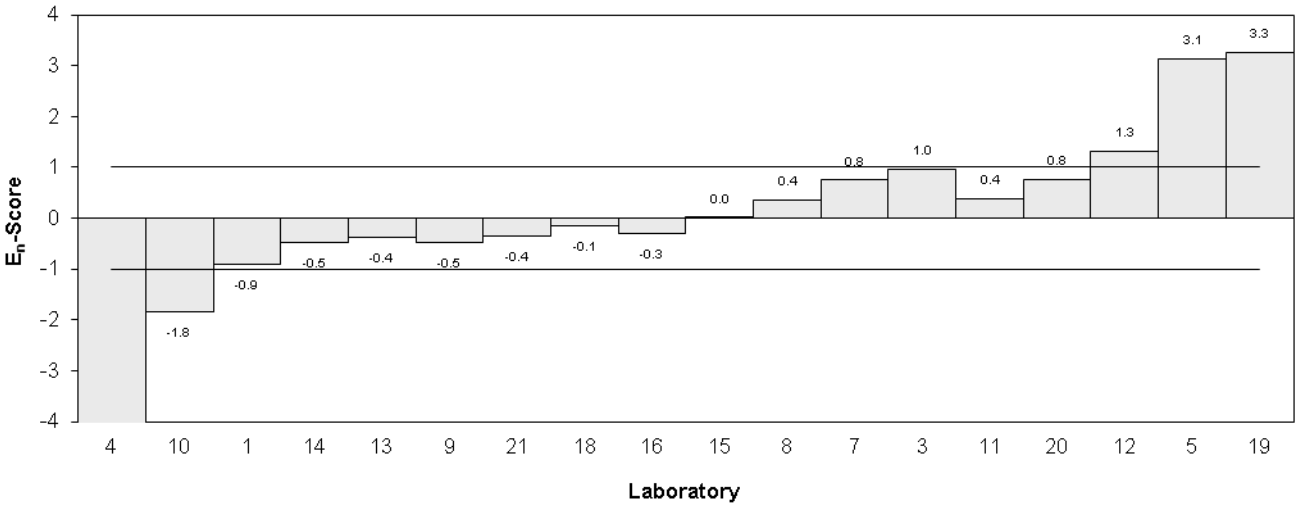

En-Scores: S6 - PFDA
En-Score
Laboratory
4 10 1 14 13 9 21 18 16 15 8 7 3 11 20 12 5 19
-1.8 -0.9 -0.5 -0.4 -0.5 -0.4 -0.1 -0.3 0.0 0.4 0.8 1.0 0.4 0.8 1.3 3.1 3.3

Figure 46

Table 51

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFUdA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	0.41	0.11	91
3	0.562	0.00894	82.1
4	< 0.1	NR	NR
5	0.424	0.035	61
7	<1.0	NR	NR
8	NR	NR	NR
9	0.36	0.054	104
10	<0.4	0.10	95
11	0.7	0.35	120
12	0.761	0.152	70
13	0.29	0.09	74
14	0.34	0.14	75
15	0.608	0.152	81
16	0.39	0.013	107
18	0.51	NR	118
19	1.076	0.0538	80
20	0.46	0.115	NR
21	0.257	0.0478	111

Statistics

Assigned Value	Not Set	
Robust Average	0.49	0.13
Median	0.442	0.095
Mean	0.511	
N	14	
Max.	1.076	
Min.	0.257	
Robust SD	0.19	
Robust CV	39%	

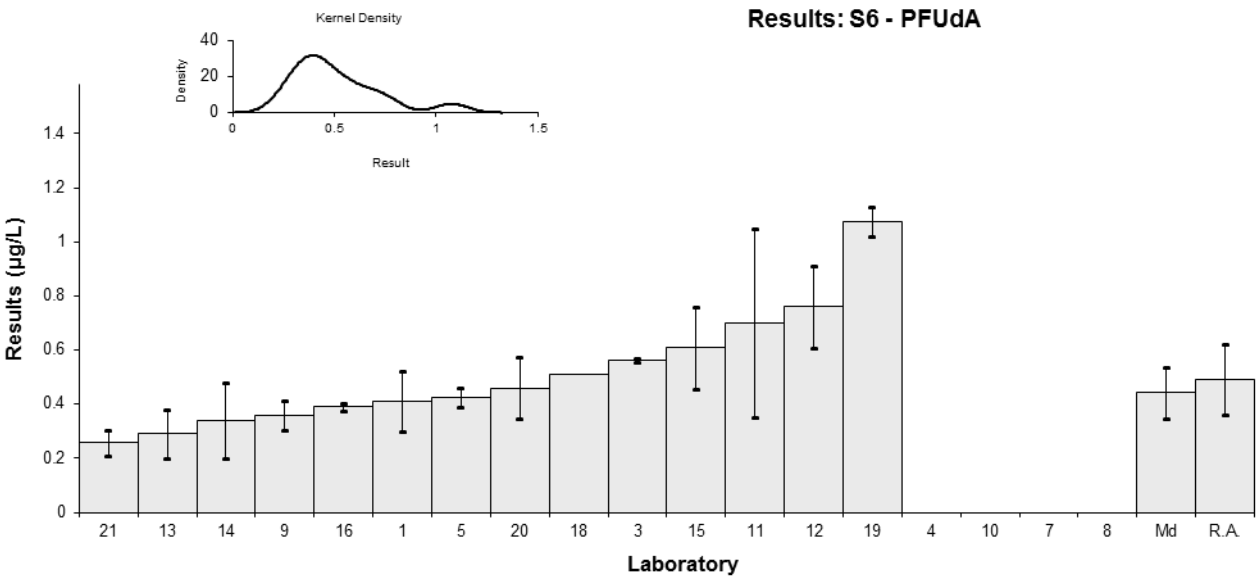

Results: S6 - PFUdA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
21
13
14
9
16
1
5
20
18
3
15
11
12
19
4
10
7
8
Md
R.A.

Figure 47

Table 52

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFDoA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	0.24	0.06	94
3	0.342	0.00537	103
4	< 0.1	NR	NR
5	0.343	0.024	52
7	<5.0	NR	NR
8	NR	NR	NR
9	0.24	0.036	113
10	<0.4	0.10	106
11	0.9	0.45	130
12	0.430	0.0860	79
13	0.34	0.09	77
14	<0.02	NR	75
15	0.452	0.113	94
16	0.32	0.008	122
18	0.47	NR	92
19	0.879	0.16701	74
20	0.29	0.087	NR
21	0.179	0.034	115

Statistics

Assigned Value	Not Set	
Robust Average	0.37	0.10
Median	0.342	0.091
Mean	0.417	
N	13	
Max.	0.879	
Min.	0.179	
Robust SD	0.37	
Robust CV	39%	

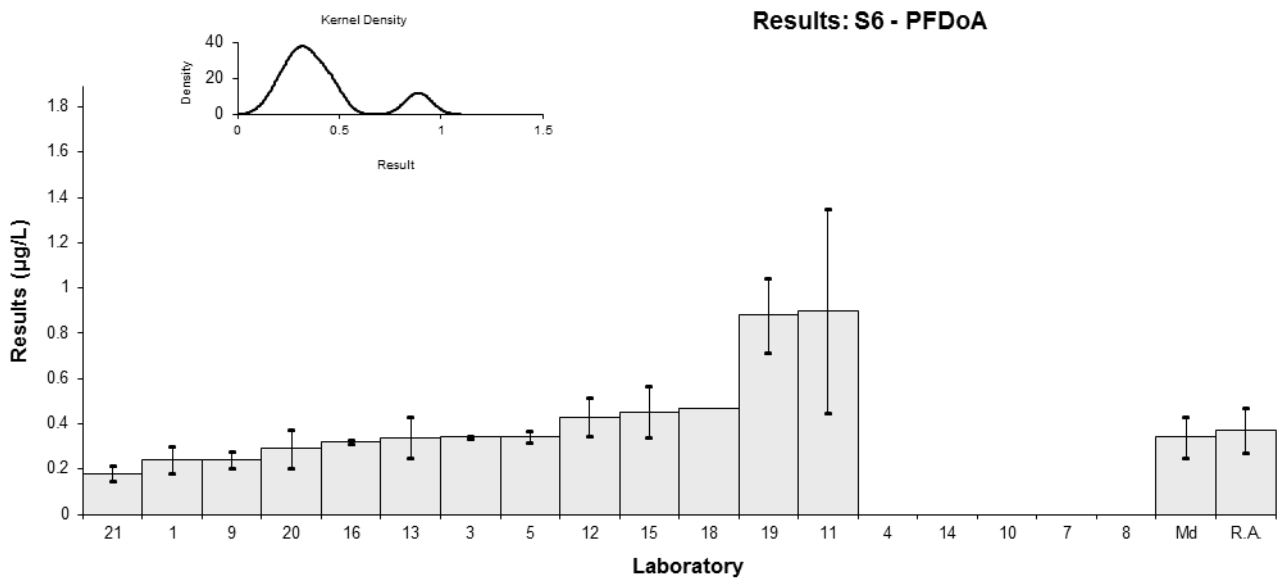

Results: S6 - PFDoA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
21
1
9
20
16
13
3
5
12
15
18
19
11
4
14
10
7
8
Md
R.A.

Figure 48

Table 53

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFTrDA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 0.20	0.13	NR
3	0.148	0.00274	103
4	< 0.1	NR	NR
5	0.147	0.027	NR
7	<5.0	NR	NR
8	NR	NR	NR
9	0.11	0.017	113
10	<0.4	0.10	100
11	0.49	0.25	90
12	0.183	0.0366	79
13	<0.1	NR	50
14	<0.02	NR	75
15	0.152	0.0455	73
16	0.13	0.007	NR
18	<0.2	NR	118
19	<0.005	NR	NR
20	0.1	0.05	NR
21	< 0.10	NR	115

Statistics

Assigned Value	Not Set	
Robust Average	0.147	0.036
Median	0.148	0.033
Mean	0.183	
N	8	
Max.	0.49	
Min.	0.1	
Robust SD	0.04	
Robust CV	27%	

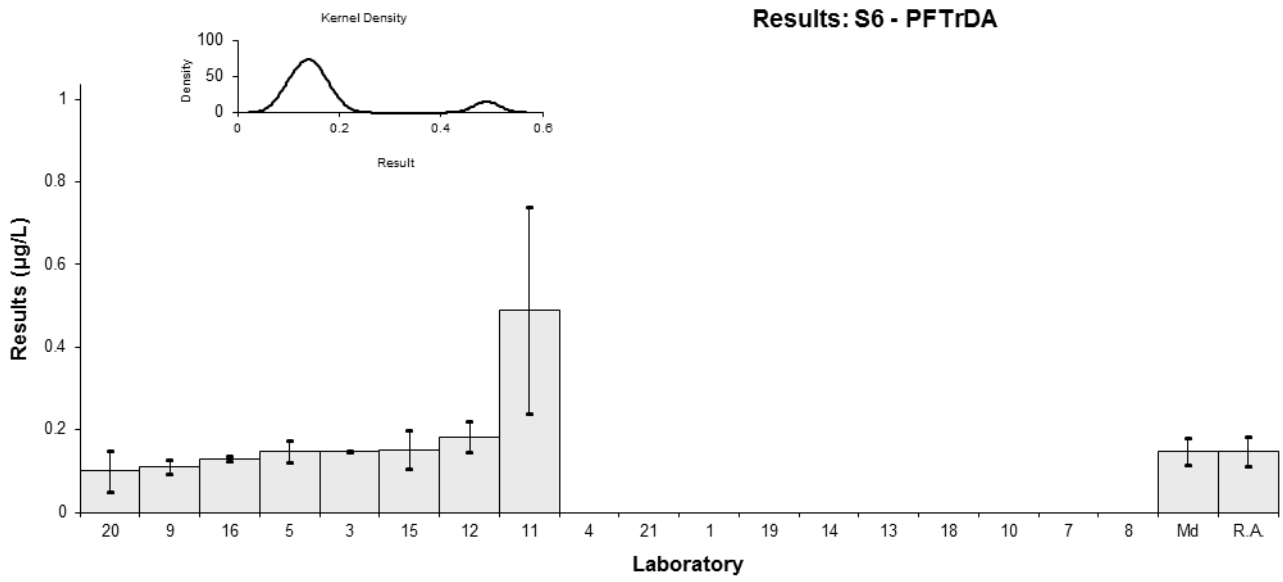

Results: S6 - PFTrDA
Kernel Density
Density
100
50
0
0
0.2
0.4
0.6
Result
Results (µg/L)
1
0.8
0.6
0.4
0.2
0
20
9
16
5
3
15
12
11
4
21
1
19
14
13
18
10
7
8
Md
R.A.
Laboratory

Figure 49

Table 54

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	PFTeDA
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	0.09	0.06	74
3	0.127	0.00189	90.4
4	< 0.1	NR	NR
5	0.122	0.015	41
7	<5.0	NR	NR
8	NR	NR	NR
9	0.11	0.017	107
10	<0.4	0.10	100
11	0.43	0.22	140
12	0.122	0.0244	72
13	<0.5	NR	50
14	<0.02	NR	75
15	0.126	0.0378	71
16	0.16	0.019	126
18	<0.2	NR	118
19	<0.005	NR	71
20	0.07	0.028	NR
21	< 0.10	NR	132

Statistics

Assigned Value	Not Set	
Robust Average	0.123	0.031
Median	0.122	0.014
Mean	0.151	
N	9	
Max.	0.43	
Min.	0.07	
Robust SD	0.04	
Robust CV	30%	

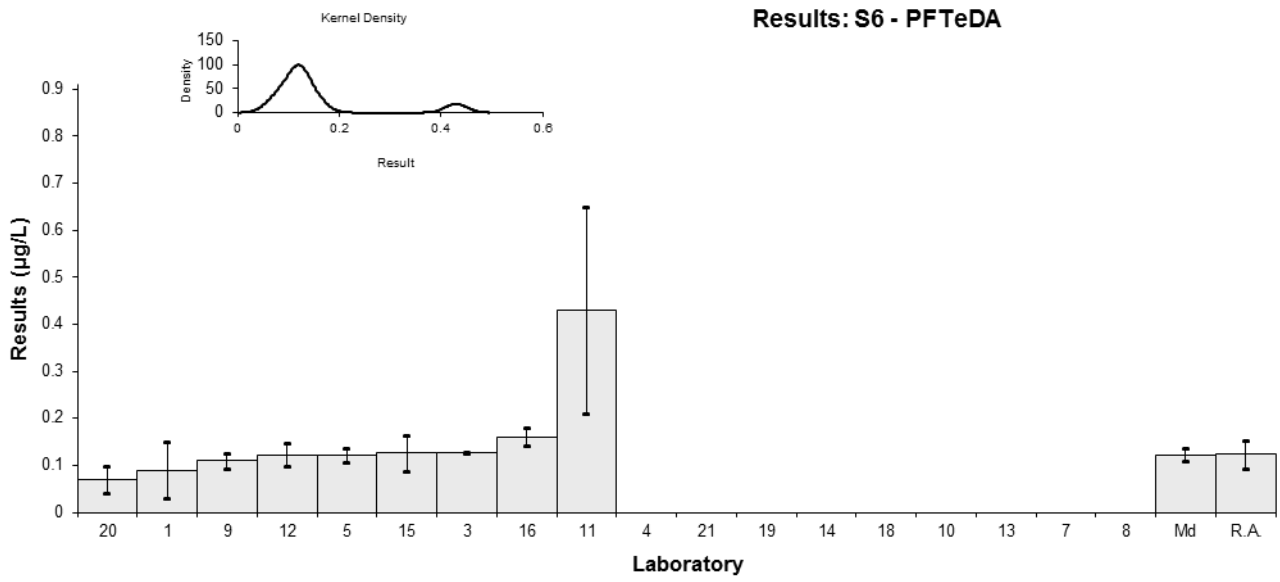

Results: S6 - PFTeDA
Kernel Density
Density
Result
Results (µg/L)
Laboratory
20
1
9
12
5
15
3
16
11
4
21
19
14
18
10
13
7
8
Md
R.A.

Figure 50

Table 55

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	Total PFCA
Units	µg/L

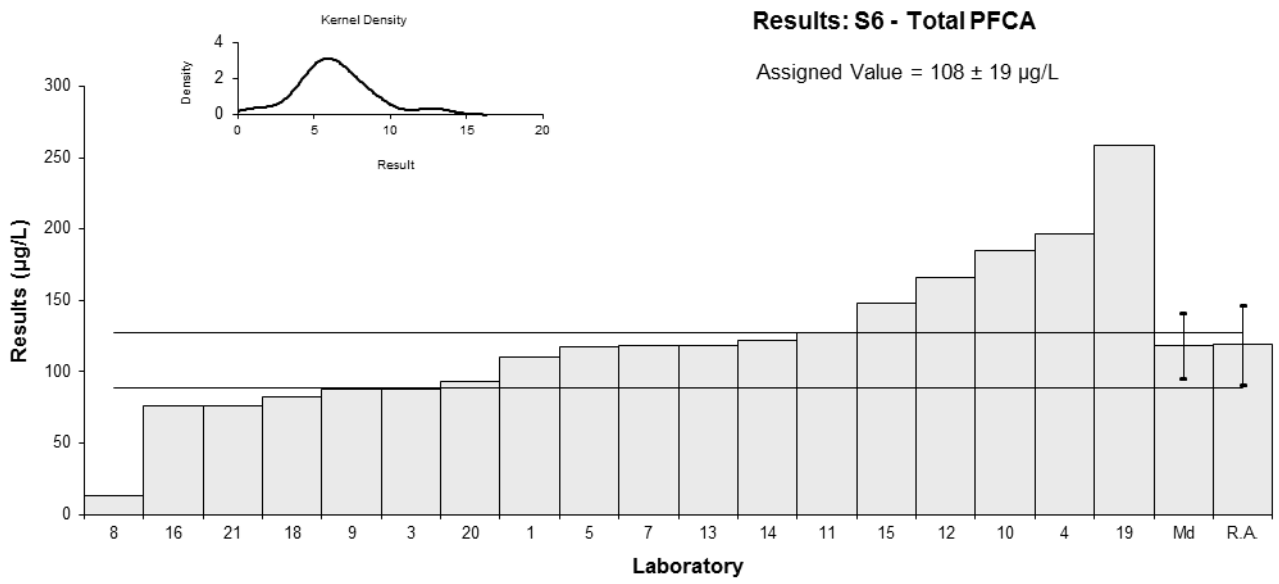
Participant Results

Lab Code	Result	z-Score
1	109.94	0.07
3	87.639	-0.75
4	196.703	3.29
5	117.016	0.33
7	118.2	0.38
8	12.82	-3.53
9	87.32	-0.77
10	184.55	2.84
11	127.22	0.71
12	165.906	2.14
13	118.33	0.38
14	122.24	0.53
15	148.317	1.49
16	75.66	-1.20
18	82.08	-0.96
19	259.136	5.60
20	92.82	-0.56
21	75.894	-1.19

Statistics

Assigned Value*	108	19
Robust Average	119	29
Median	118	22
Mean	121	
N	18	
Max.	259.136	
Min.	12.82	
Robust SD	49	
Robust CV	41%	

*Robust Average excluding Laboratories 4, 8, 10 and 19.


Kernel Density
Density
Result
Results: S6 - Total PFCA
Assigned Value = 108 ± 19 µg/L
Results (µg/L)
Laboratory
8 16 21 18 9 3 20 1 5 7 13 14 11 15 12 10 4 19 Md R.A.

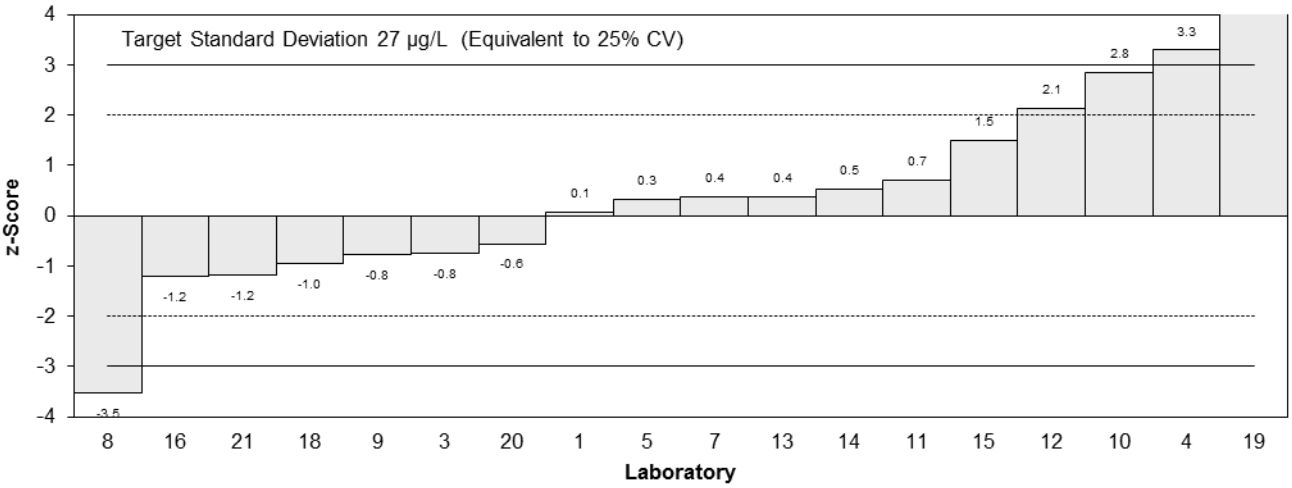

z-Scores: S6 - Total PFCA
Target Standard Deviation 27 µg/L (Equivalent to 25% CV)
z-Score
Laboratory
8 16 21 18 9 3 20 1 5 7 13 14 11 15 12 10 4 19
-3.5 -1.2 -1.2 -1.0 -0.8 -0.8 -0.6 0.1 0.3 0.4 0.4 0.5 0.7 1.5 2.1 2.8 3.3

Figure 51

Table 56

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	6:2FTS
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 0.04	0.01	111
3	0.0940	0.00144	109
4	< 0.1	NR	NR
5	0.024	0.002	51
7	<1.0	NR	NR
8	31.8	11.2	NR
9	<0.02	NR	NR
10	<2.0	0.4	121
11	<0.01	NR	100
12	NR	NR	NR
13	<0.2	NR	57
14	<0.02	NR	90
15	NR	NR	NR
16	0.09	0.034	111
18	<0.5	NR	299
19	<0.005	NR	97
20	< 0.05	0.015	NR
21	< 1.00	NR	106

Statistics

Assigned Value	Not Set	
Median	0.09	0.08
Mean	8.00	
N	4	
Max.	31.8	
Min.	0.024	
Robust SD	18	
Robust CV	220%	

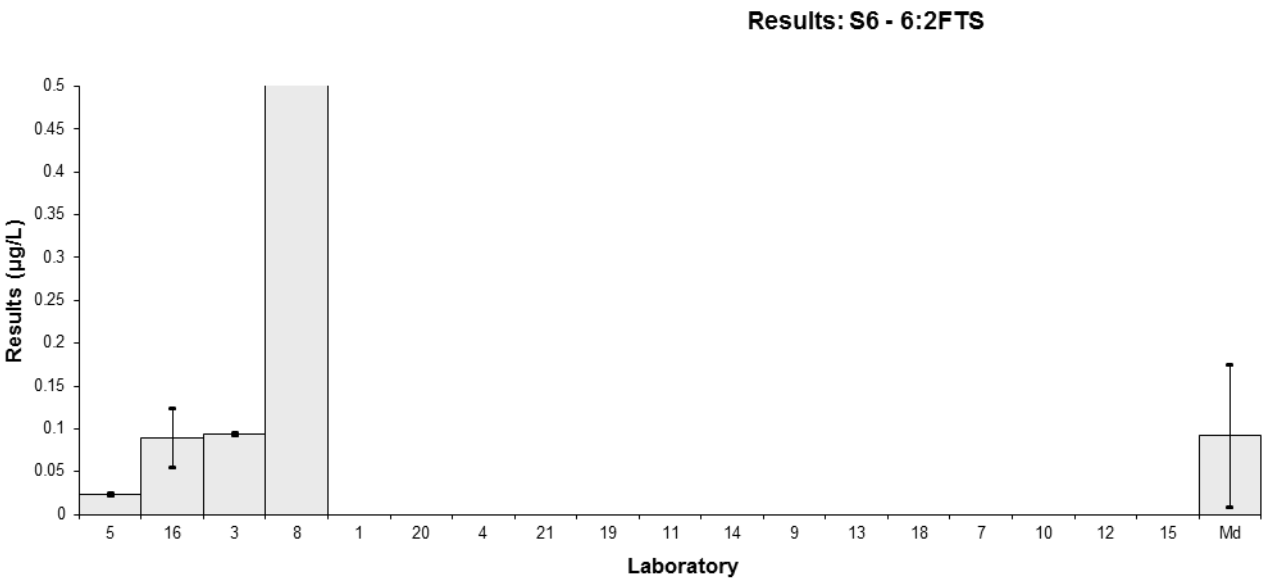

Results: S6 - 6:2FTS
Results (µg/L)
0.5
0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
5
16
3
8
1
20
4
21
19
11
14
9
13
18
7
10
12
15
Md
Laboratory

Figure 52

Table 57

Sample Details

Sample No.	S6
Matrix.	River water, Alcoseal, PFDA, PFDS, Fluorine Free AFFF
Analyte.	8:2FTS
Units	µg/L

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 0.02	0.005	NR
3	0.099	0.00170	87.9
4	< 0.1	NR	NR
5	<0.002	0.002	39
7	<1.0	NR	NR
8	14.0	4.6	NR
9	<0.02	NR	NR
10	<0.4	0.10	118
11	<0.01	NR	90
12	NR	NR	NR
13	<0.2	NR	61
14	<0.02	NR	78
15	NR	NR	NR
16	0.13	0.019	93
18	<0.5	NR	110
19	<0.005	NR	79
20	< 0.01	0.003	NR
21	< 1.00	NR	99

Statistics

Assigned Value	Not Set	
Mean	4.74	
N	3	
Max.	14	
Min.	0.099	

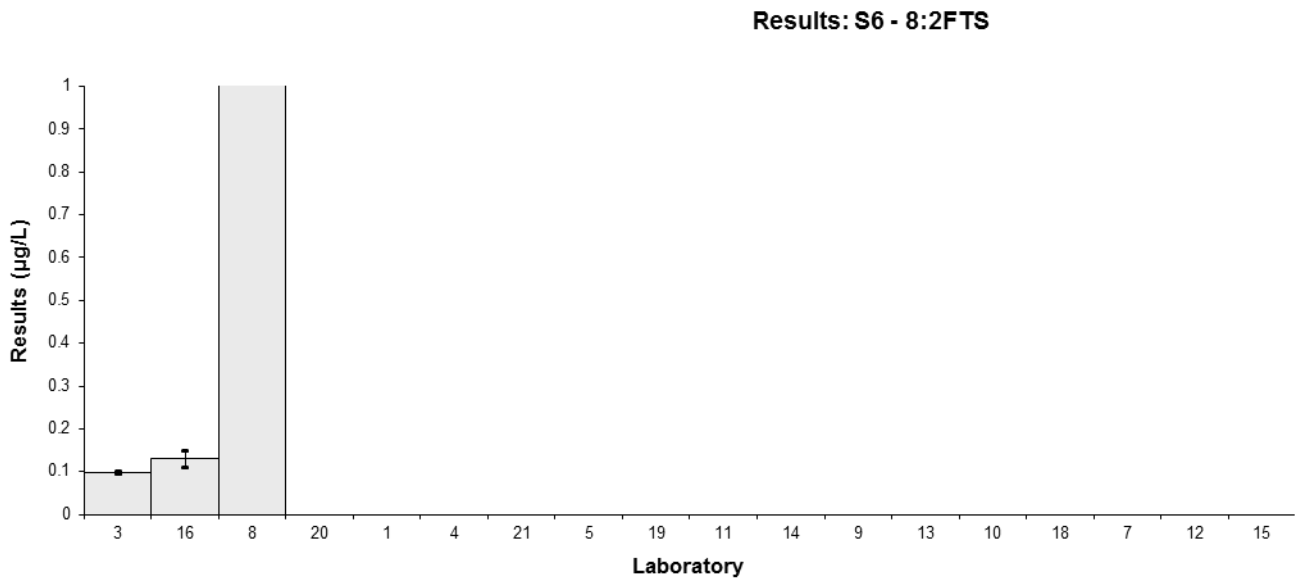

Results: S6 - 8:2FTS
Results (µg/L)
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
3
16
8
20
1
4
21
5
19
11
14
9
13
10
18
7
12
15
Laboratory

Figure 53

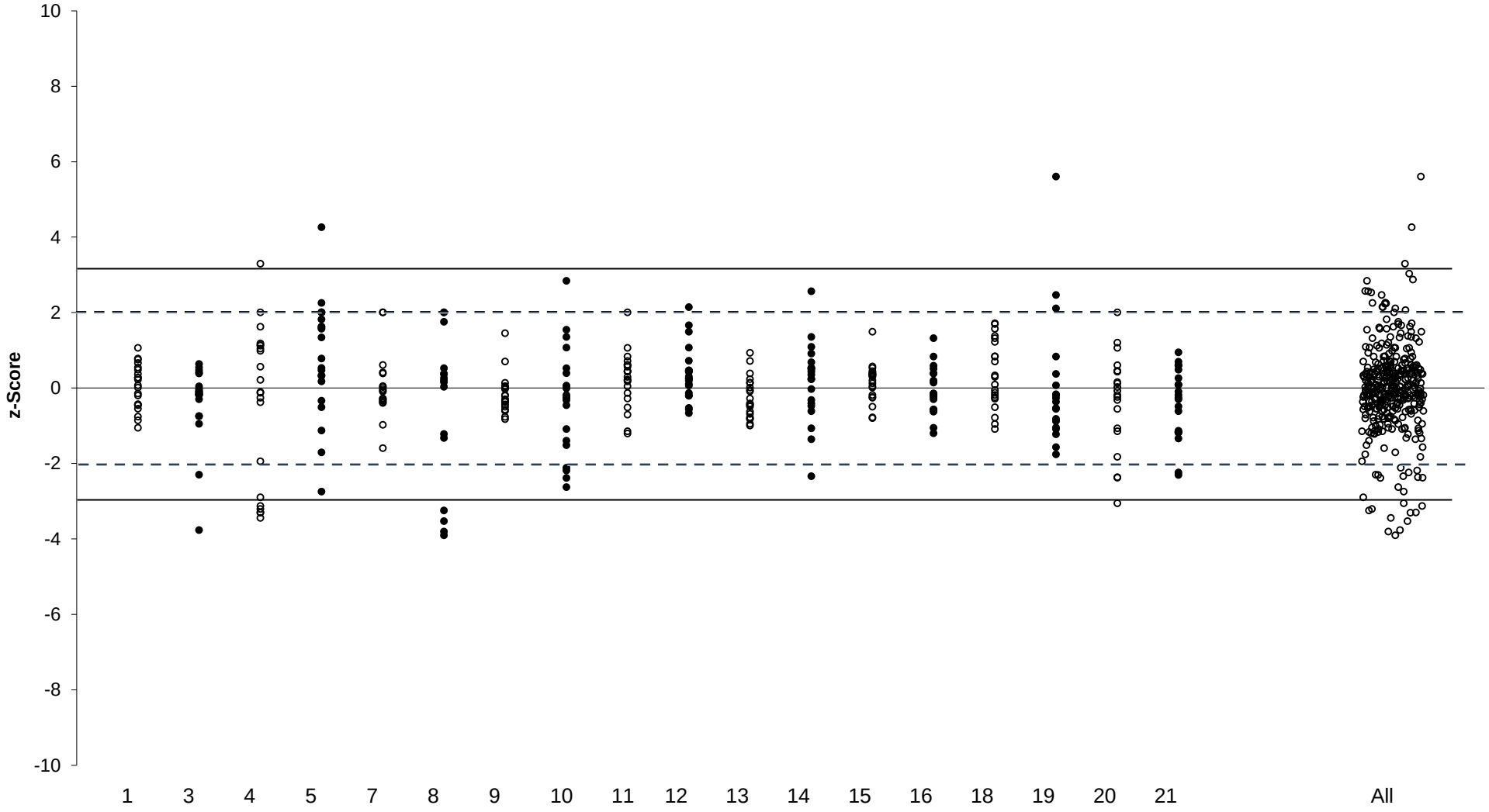


Figure 54 z-Score Dispersal by Laboratory

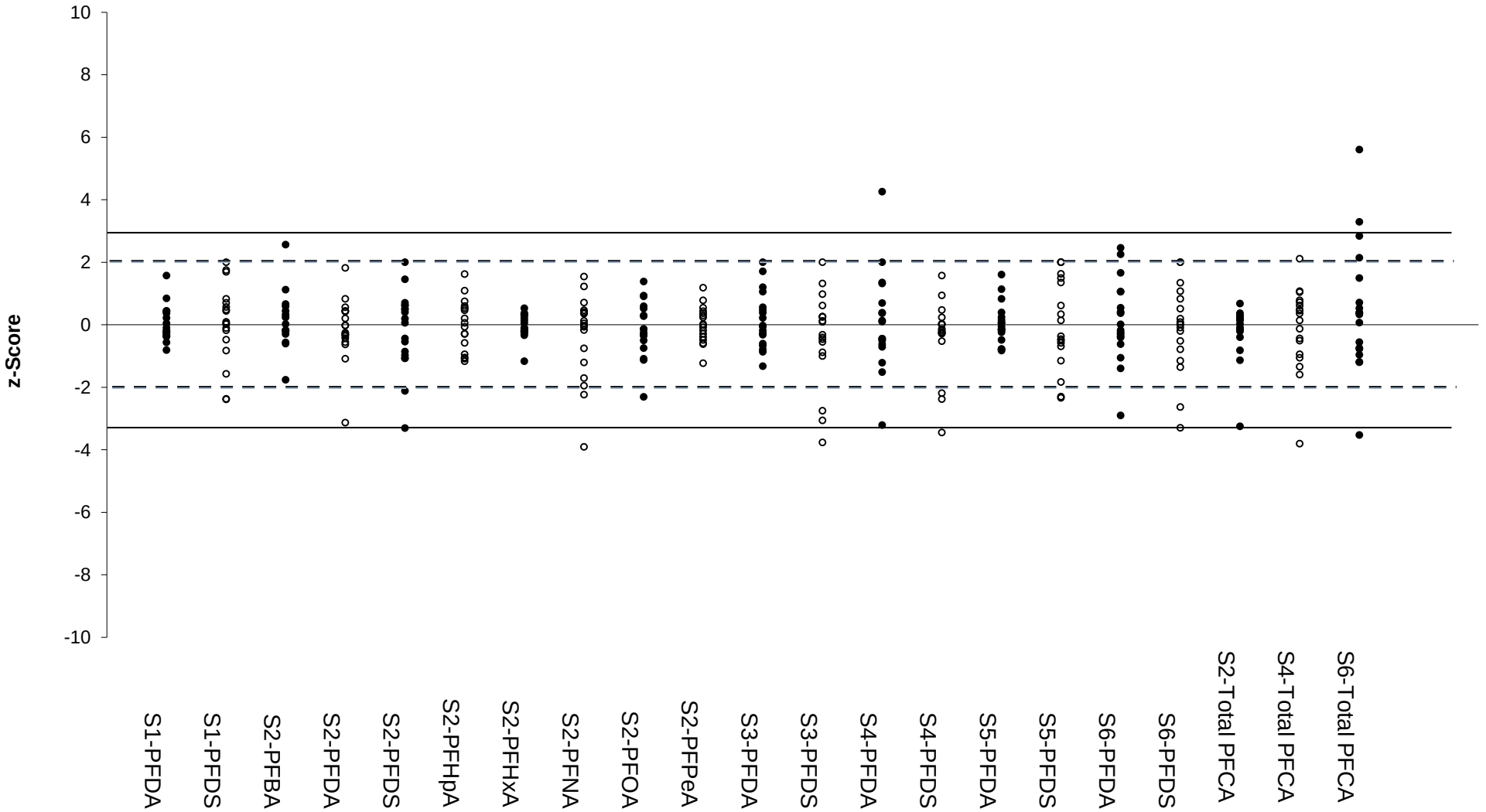
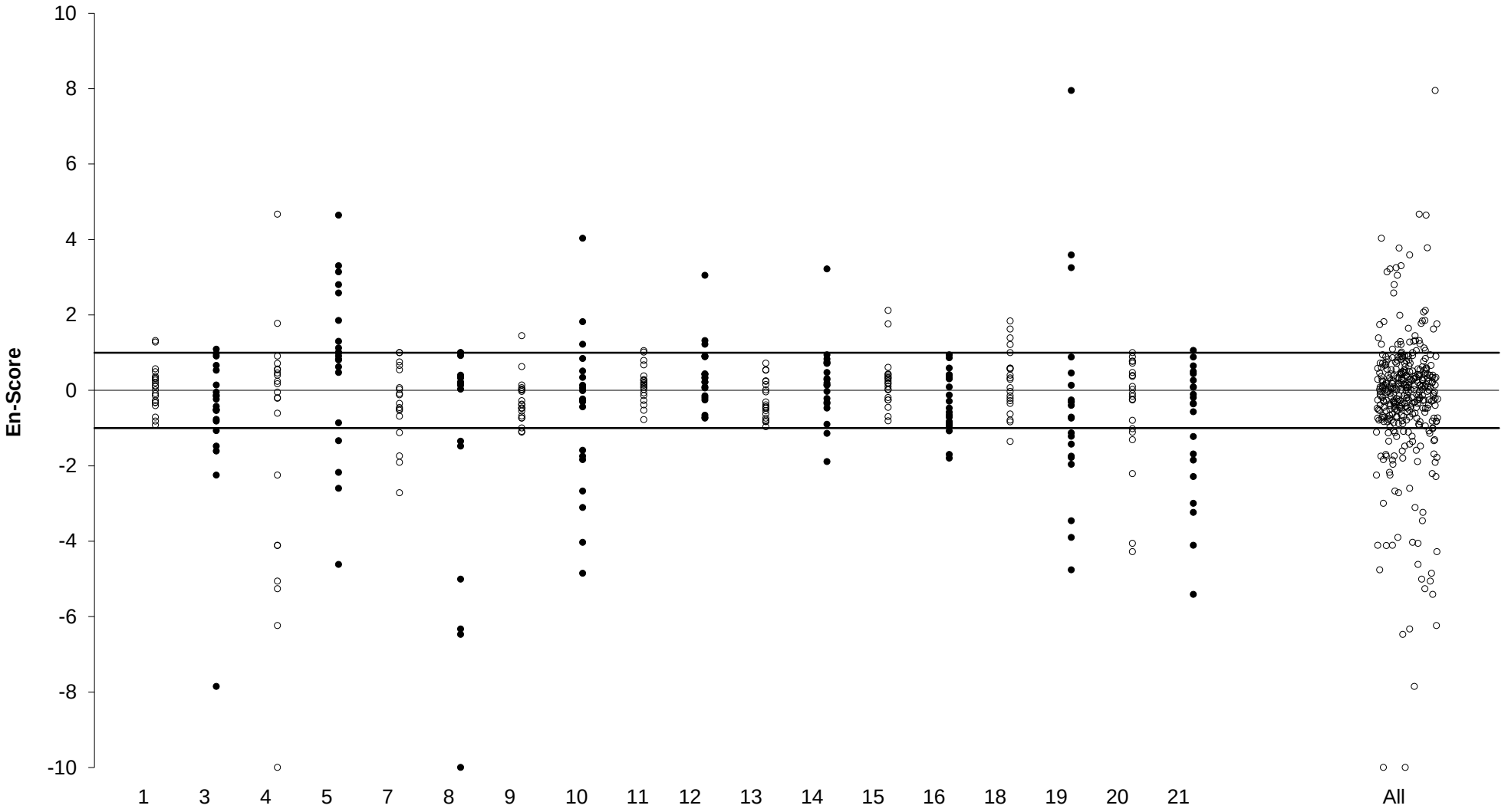


Figure 55 z-Score Dispersal by Analyte



Scores greater than 10 have been plotted as 10.

Figure 56 E_n-Score Dispersal by Laboratory

6 DISCUSSION OF RESULTS

6.1 Assigned Value

Assigned values for the tests in the study samples were the robust averages of participants' results. The robust averages and their associated expanded uncertainties were calculated using the procedures described in ISO 13528:2015(E). Results less than 50% and more than 150% were removed before the calculation of assigned value.[8] Appendix 3 sets out the calculation for the expanded uncertainty of the robust average of PFDA in S1.

Although a large number of results were reported for PFNA in S1, S3 and S5, no assigned value was set for this analyte because it was not added to the test samples.

Participants' reported results for Total PFCA content in S4 and S6 were assessed instead of the results reported for individual PFCA, because there is not enough information available on Alcoseal with regards to its chemical composition or expected oxidation products. With the exception of PFDA, no assigned value was calculated for individual PFCA or for 6:2 FTS, and 8:2 FTS in S4 and S6. S3 and S5 were also spiked with Alcoseal and no assigned value was set for 6:2 FTS and 8:2 FTS in these samples either. Descriptive statistics for these tests are presented in Chapter 5.

The PFAA results PRE Top assay are likely impurities from Alcoseal foam and 6:2 diPAP and 8:2 diPAP standards. These results are presented in Appendix 1.

Assigned values for spiked analytes were within the range 58% – 73% of the spiked concentration for PFDS and 68% -114% for PFDA (Table 58). Although a low spiked recovery was found for PFDS in the study samples, an assigned value was still set for this analyte because there was reasonable consensus between the reported results (CV from 11% to 33%).

Traceability: The consensus of participants' results is not traceable to any external reference, so although expressed in SI units, metrological traceability has not been established.

Table 58 Comparison of Assigned Value and Spiked Concentration.

Sample	Matrix	Analyte	Units	Spiked Concentration	Assigned Value	Assigned/ Spike
S1	MilliQ water	PFDS	µg/L	3.14	2.28	73%
S2	MilliQ water	PFDS	µg/L	3.14	1.91	61%
S3	MilliQ water	PFDS	µg/L	6.51	4.38	67%
S4	MilliQ water	PFDS	µg/L	6.51	4.19	64%
S5	River Water	PFDS	µg/L	6.50	4.41	68%
S6	River Water	PFDS	µg/L	6.50	3.79	58%
S1	MilliQ water	PFDA	µg/L	6.42	6.80	106%
S2	MilliQ water	PFDA	µg/L	6.42	6.05	94%
S3	MilliQ water	PFDA	µg/L	9.08	7.18	79%
S4	MilliQ water	PFDA	µg/L	9.08	6.2	68%
S5	River Water	PFDA	µg/L	9.01	10.3	114%
S6	River Water	PFDA	µg/L	9.01	8.7	97%

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded measurement uncertainty associated with their results. Of 896 numerical results, 873 were reported with an expanded measurement uncertainty, indicating that not all laboratories have addressed this requirement of ISO 17025.[10] The participants used a wide variety of procedures to estimate the expanded measurement uncertainty. These are presented in Table 5.

Proficiency tests allow a check of participants' uncertainty estimates. Results and the expanded MU are presented in the bar charts for each analyte (Figure 2 to 53). In this study, the magnitude of the reported expanded uncertainties was within the range 1% to 500% of the reported value. 162 were less than 10% relative, which the study coordinator believes are unrealistically small for a routine PFAS measurement and 39 were larger than 50% relative.

Results returning a satisfactory z-score but an unsatisfactory E_n-score may have underestimated the uncertainty.

Some participants attached an estimate of the expanded measurement uncertainty to a result reported as less than their limit of reporting. An estimate of uncertainty expressed as a numerical value cannot be attached to a result expressed as a range.[11]

In some cases the results were reported with an inappropriate number of significant figures. The recommended format is to write uncertainty to no more than two significant figures and then to write the result with the corresponding number of decimal places (for example instead of 12.808 ± 2.818 µg/L, it is better to report 12.8 ± 2.8 µg/L).[10]

6.3 z-Score

A target standard deviation equivalent to the 20% coefficient of variation (CV) was used to calculate z-scores in S1, S3 and S5 and equivalent to the 25% CV in S2, S4, and S6.

The between-laboratory coefficient of variation predicted by the modified Horwitz equation[7] and the between laboratories CV are presented for comparison in Table 59.

Table 59 Performance Target Standard Deviation, Modified Horwitz Values and Between Laboratories CV

Sample	Analyte	Assigned value	Unit	Target SD (as PCV, %)	Modified Horwitz CV (%)	Between laboratories' CV* (%)
S1	PFDS	2.28	µg/L	20	22	24%
S1	PFDA	6.80	µg/L	20	22	10%
S2	PFDS	1.91	µg/L	25	22	22%
S2	PFBA	1.89	µg/L	25	22	14%
S2	PFPeA	3.88	µg/L	25	22	14%
S2	PFHxA	5.04	µg/L	25	22	8.3%
S2	PFHpA	8.0	µg/L	25	22	23%
S2	PFOA	6.74	µg/L	25	22	21%
S2	PFNA	2.72	µg/L	25	22	27%
S2	PFDA	6.05	µg/L	25	22	15%
S2	Total PFCA	34.1	µg/L	25	22	8.7%
S3	PFDS	4.38	µg/L	20	22	15%

Sample	Analyte	Assigned value	Unit	Target SD (as PCV, %)	Modified Horwitz CV (%)	Between laboratories' CV* (%)
S3	PFDA	7.18	µg/L	20	22	16%
S4	PFDS	4.19	µg/L	25	22	11%
S4	PFDA	6.2	µg/L	25	22	25%
S4	Total PFCA	136	µg/L	25	22	24%
S5	PFDS	4.41	µg/L	20	22	33%
S5	PFDA	10.3	µg/L	20	22	9.7%
S6	PFDS	3.79	µg/L	25	22	22%
S6	PFDA	8.7	µg/L	25	22	21%
S6	Total PFCA	108	µg/L	25	22	26%

*Between laboratories' CV outliers removed

Note: Shaded cells are between participant laboratories' CV which were higher than the target SD established by the study coordinator and the coefficient of variation from predictive mathematical model (modified Horwitz equation).

To account for possible bias in the consensus values due to laboratories using inefficient analytical/extraction techniques, z-scores were adjusted for PFBS in S1, S2, S3, S4, S5 and S6 and for PFDA in S3 and S4. A maximum acceptable concentration was set to two target standard deviations more than the spiked level. For results higher than the maximum acceptable concentration, z-scores were not adjusted. This ensured that laboratories reporting results close to the spiked concentration were not penalised. z-Scores of less than 2 were left unaltered.

The dispersal of participants' z-scores is graphically presented by laboratory in Figure 54 and by analyte in Figure 55.

Of the 369 results for which z-scores were calculated, 337 (91%) returned a satisfactory z-score of $|z| \leq 2.0$.

6.4 E_n-Score

E_n-score should be interpreted only in conjunction with z-scores. The E_n-score indicates how closely a result agrees with the assigned value taking into account the respective uncertainties. An unsatisfactory E_n score for an analyte can either be caused by an inappropriate measurement, an inappropriate estimation of measurement uncertainty, or both.

The dispersal of participants' E_n-scores is graphically presented in Figure 56. Where a laboratory did not report an expanded uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the E_n-score.

Of 369 results for which E_n-scores were calculated, 270 (73%) returned a satisfactory score of $|E_n| \leq 1.0$ indicating agreement of the participants' results with the assigned values within their respective expanded measurement uncertainties.

6.5 Participants' Methods and Analytical Results

Participants were requested to analyse the samples using their normal test method and to report a single result as they would normally do for a client. A summary of the methods used for oxidative treatment together with the test methods used pre- and post-oxidative treatment are presented in Tables 1, 2 and 3.

All participants developed their oxidative methods based on that published by Houtz and Sedlack (H&S) in 2012.[2] An evaluation of the methods used by participants for TOP Assay oxidative treatment is presented in Chapter 7.

With the exception of three participants, all laboratories reported using LC-MSMS for PFAS measurements. Laboratories 1, 12 and 15 used Orbitrap.

A large majority of laboratories attempted to undertake analysis on the entire sample, however this was not possible with post-oxidation samples for some participants. Laboratory 9 commented: *"Some of the longer chain PFAS may have lower water solubility and could be sticking on to the sides of the bottle. Trouble is we can't oxidise the entire sample!...tricky…. Analyst just shook the sample as best she could prior to taking an aliquot before oxidation."*

Laboratories 1, 3 and 5 used the entire pre- and post-oxidation samples for analysis; they also reported rinsing the bottle and using SPE extraction.

Laboratory 13 used SPE extraction for pre-oxidation samples and direct injection (DI) for post-oxidation samples

The majority of participants reported results for the long chain carboxylic acids in the post-oxidation samples S4 and S6. Plots of participants' results for **PFUdA, PFDoA, PFTrDA** and **PFTeDA** in S4 and S6 versus testing method are presented in Figures 57 to 60.

Losses in long chain carboxylic acids can occur during the sample extraction process, as they can be absorbed onto the wall of the container. However, losses in the precursors before sample oxidation (some have been found to stick onto container walls) and small chain carboxylic acids formation during oxidation can also affect the results for these acids.[12, 13, 14]

PFDS was spiked at 3.14 µg/L in water samples S1/S2 and at 6.51 µg/L in the two pairs of water samples S3/S4 and S5/S6. The results reported for PFDS in the six study samples were consistently low: within 58% to 73% of the spiked value. Although there was a slight decrease in PFDS between pre- and post-oxidation samples, this decrease is not substantial. This is more an indication that the low recovery might be due to absorption of the analyte into the walls of the sample bottles before the oxidation treatment, rather than during the oxidation process. There is also a possibility that PFDS may have been lost during PT test samples preparation prior to sample dispatch.

Figure 61 show the normalised PFDS results (against the spiked concentration) versus the amount of sample taken for analyses and extraction technique for each sample. Participants used a variety of methods for extraction. No correlation between results and method was evident.

PFDA was spiked at 6.42 µg/L in water samples S1/S2 and at 9.08 µg/L in the two pairs of water samples S3/S4 and S5/S6. While the PFDA results in the pre-oxidation samples S1, S3 and S5 were within 79% to 114% of the spiked value, the PFDA results in the post-oxidation samples were within 68% to 97% of the spiked value. Plots of participants' results normalised to the spiked value versus sample size and extraction technique are presented in Figure 62. No correlation was evident between the results and method used.

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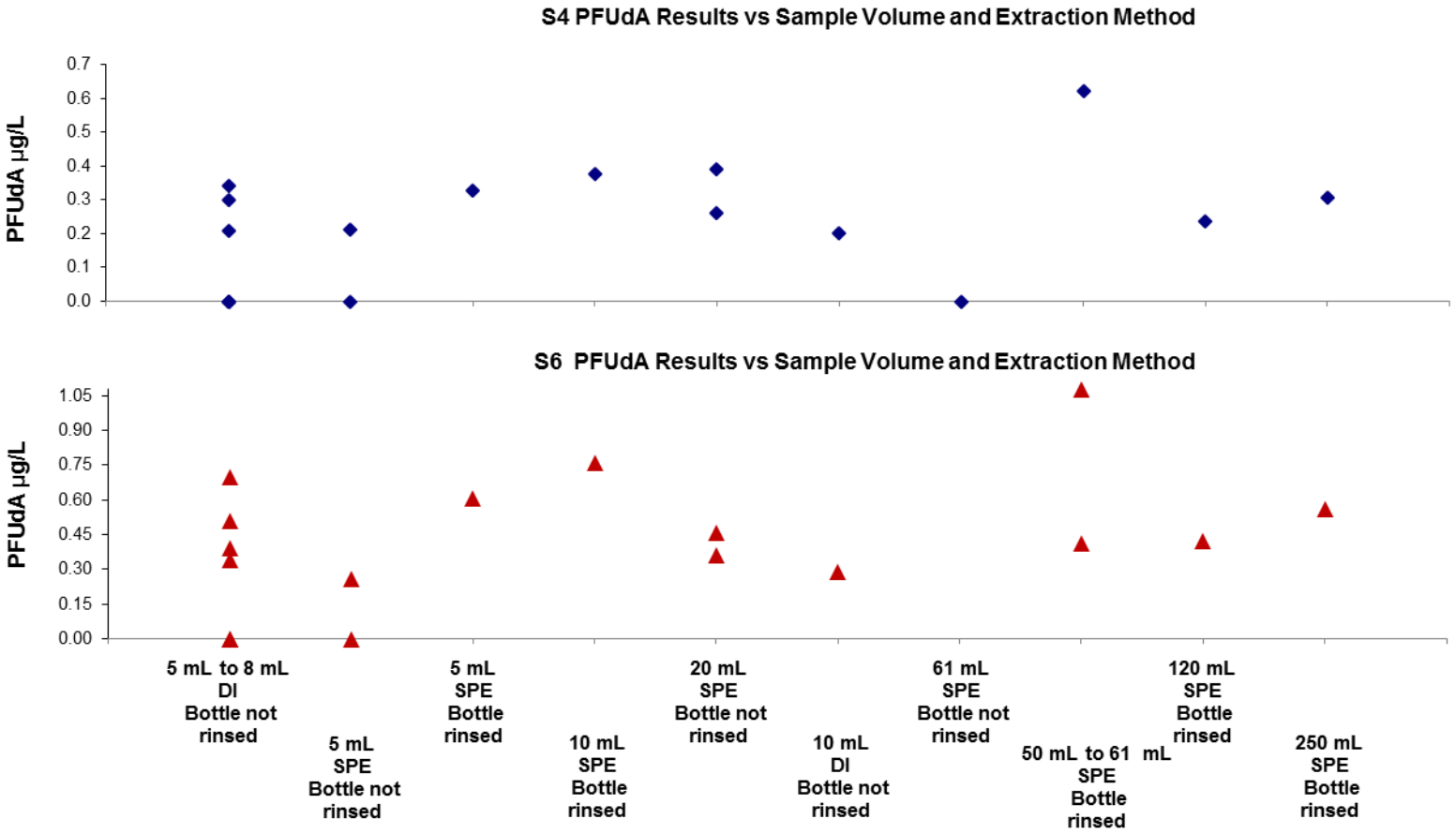


Figure 57 PFUdA Results in S4 and S6 versus Sample Volume and Extraction Method

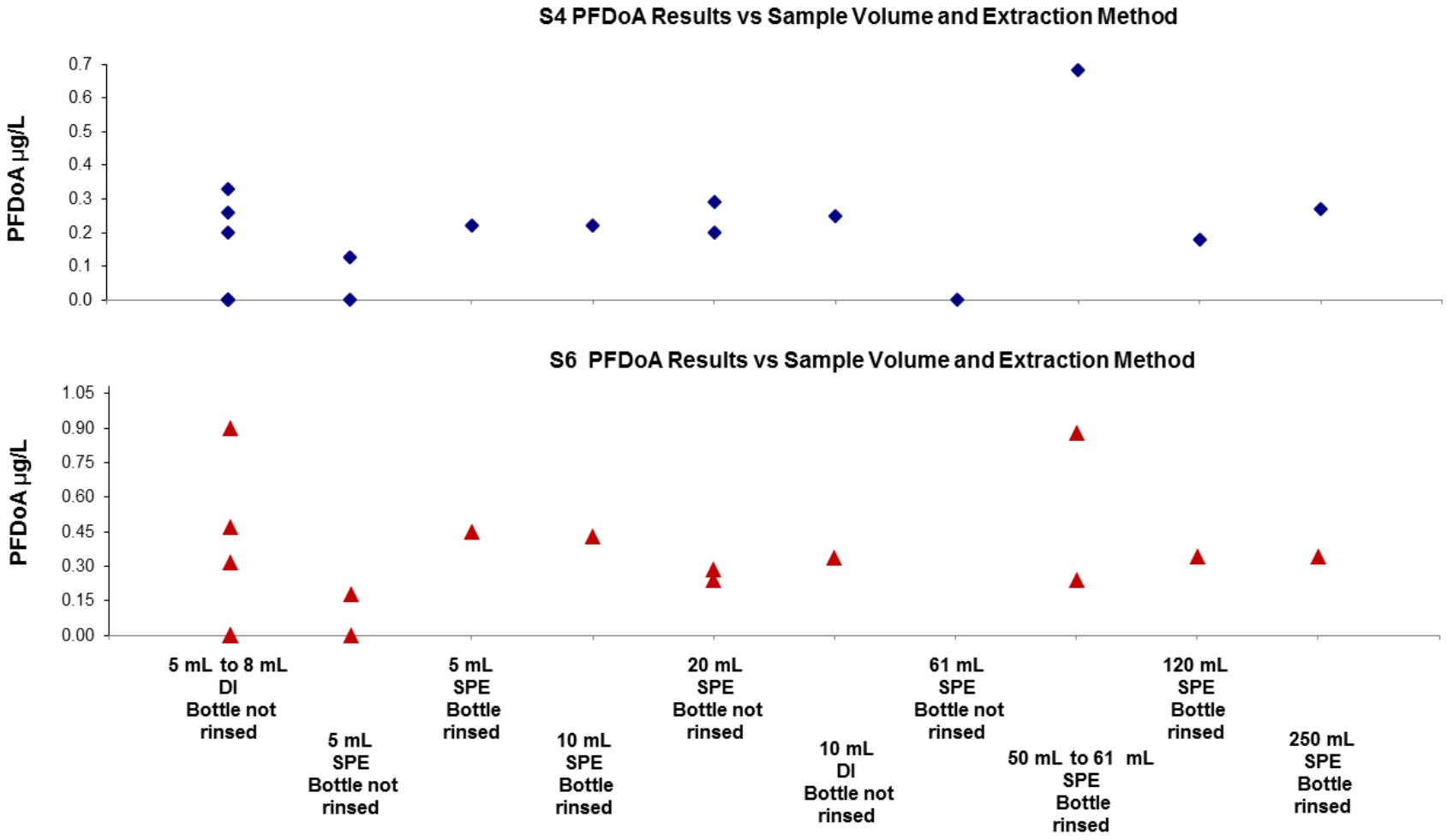


Figure 58 PFDoA Results in S4 and S6 versus Sample Volume and Extraction Method

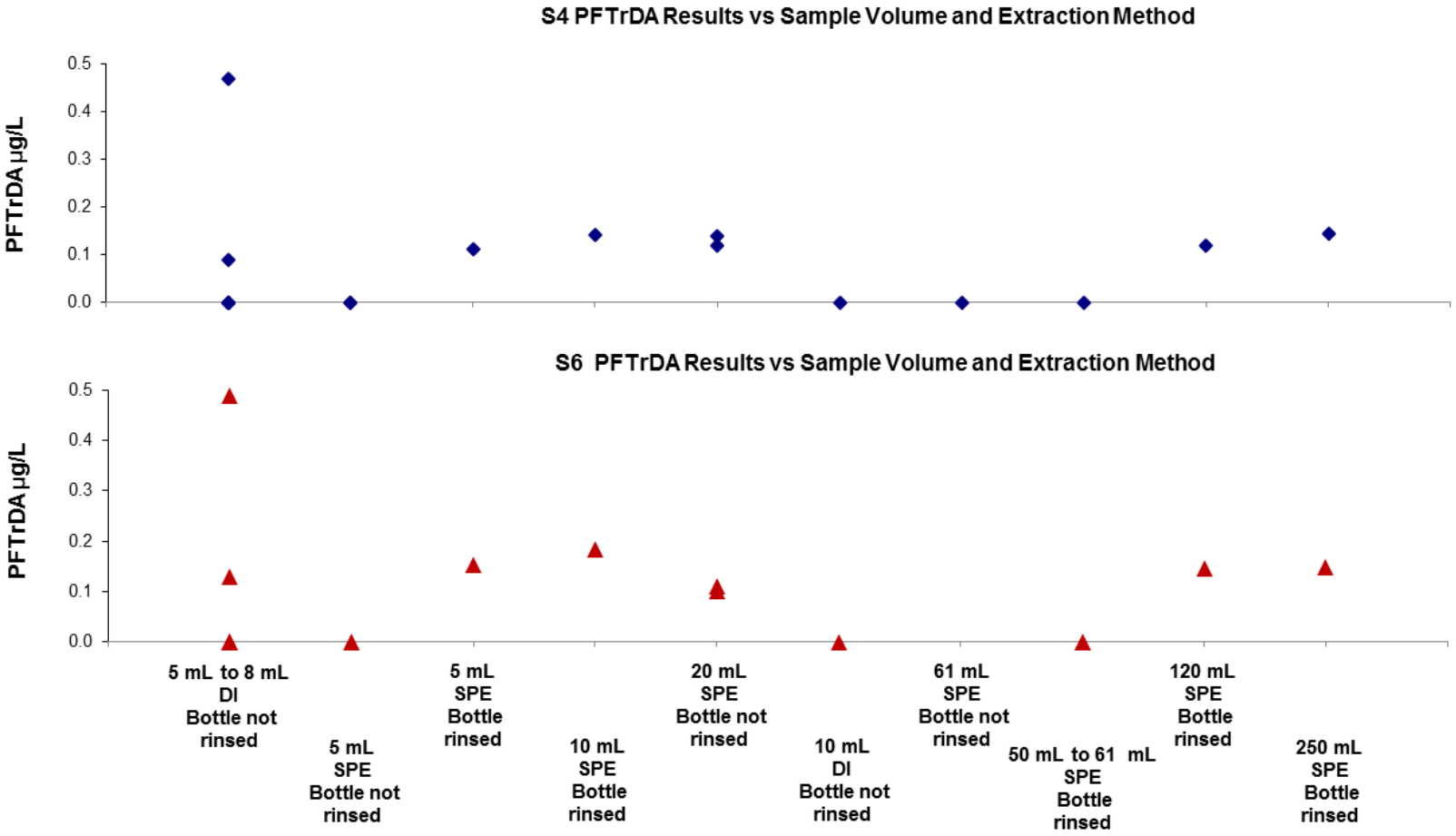


Figure 59 PFTrDA Results in S4 and S6 versus Sample Volume and Extraction Method

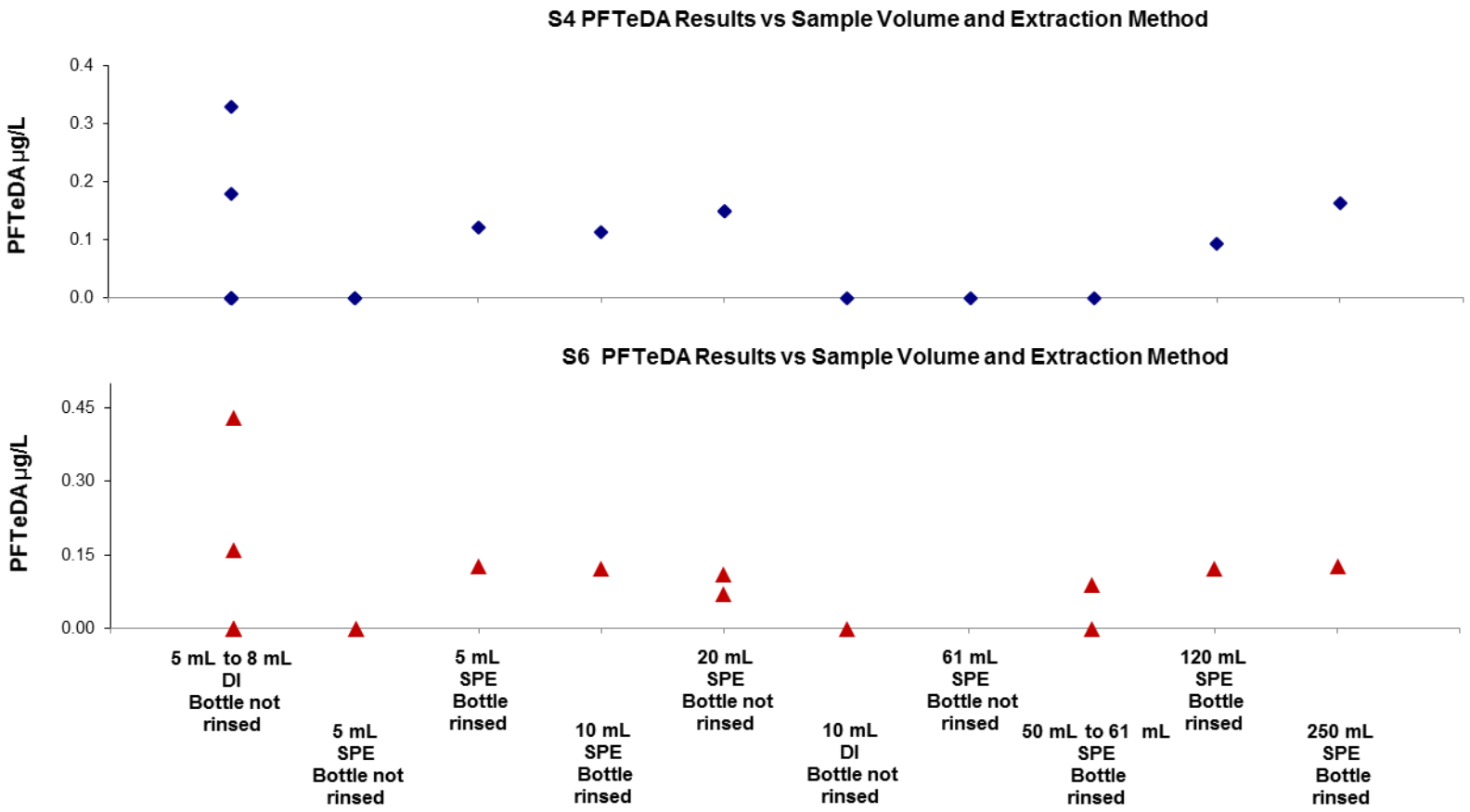


Figure 60 PFTeDA Results in S4 and S6 versus Sample Volume and Extraction Method

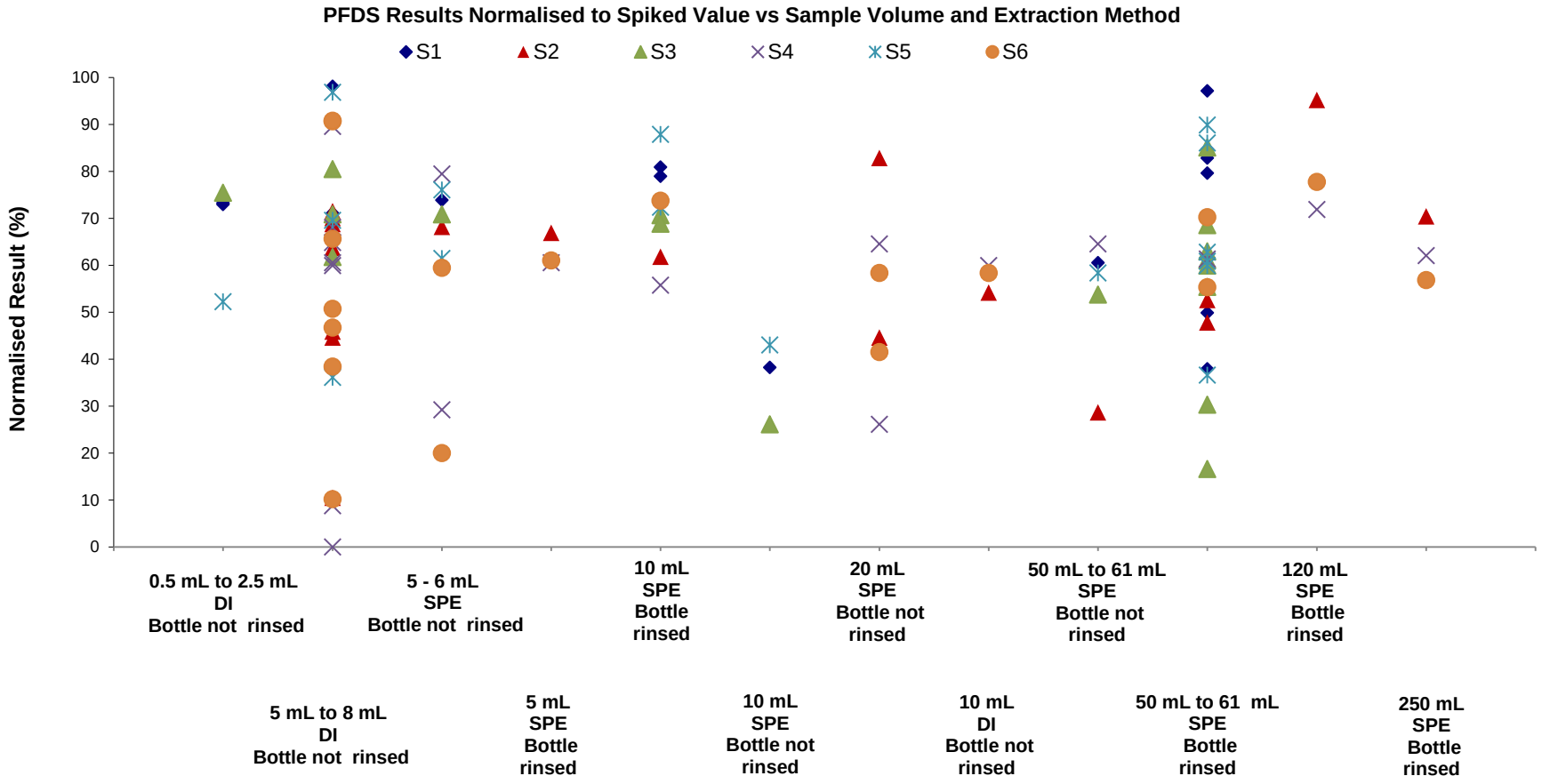


Figure 61 PFDS Results Normalised to Spiked Value versus Sample Volume and Extraction Method

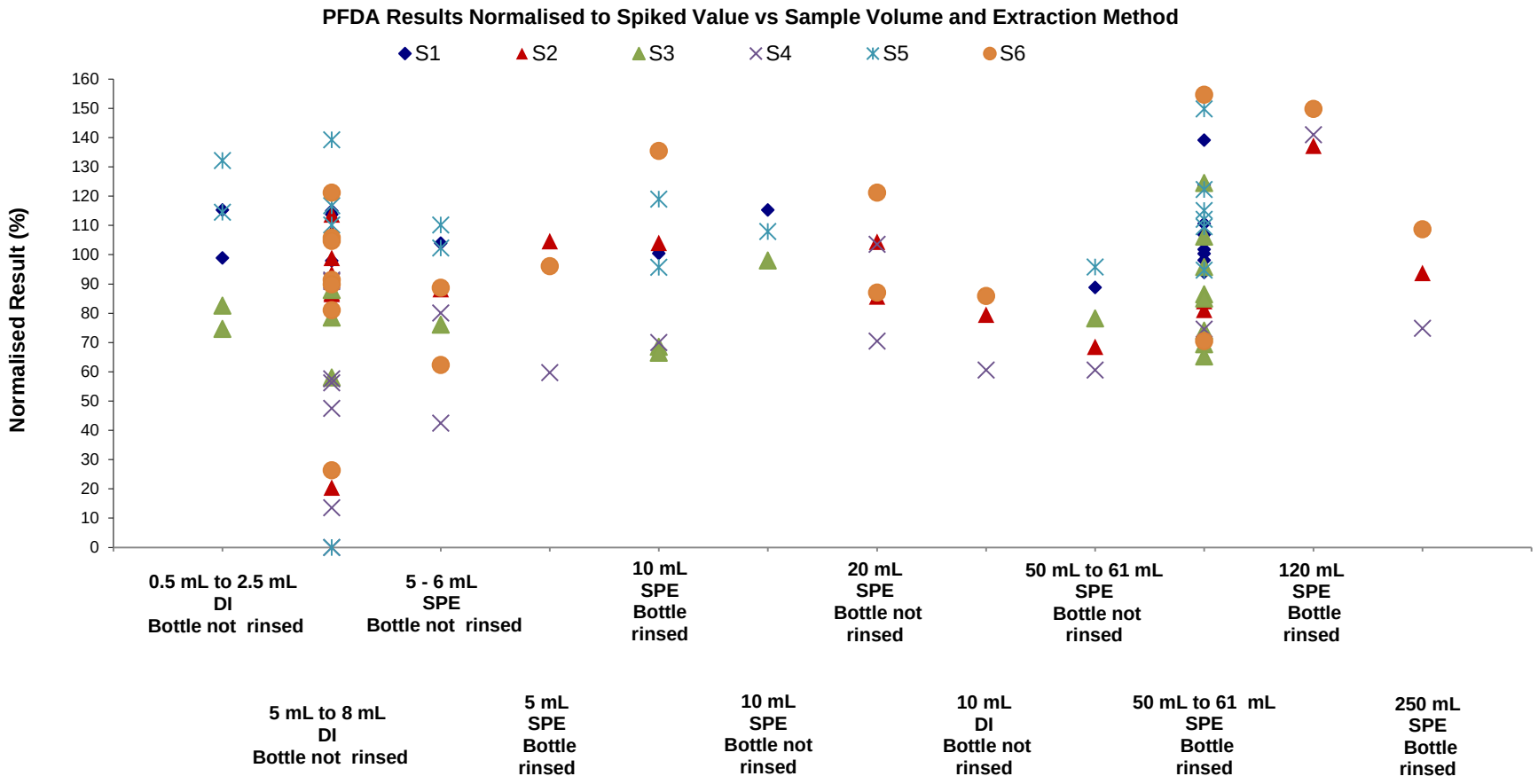


Figure 62 PFDA Results Normalised to Spiked Value versus Sample Volume and Extraction Method

7 EVALUATION OF THE METHODS USED FOR TOP ASSAY OXIDATIVE PRE-TREATMENT

7.1 Study Design

The study included three duplicate Samples S1/S2, S3/S4 and S5/S6. Laboratories were instructed to use their routine methods for PFAS measurement but to analyse S1, S3 and S5 pre-oxidative treatment and S2, S4 and S6 post-oxidative treatment.

This study was designed to assess participants' oxidative methods for:

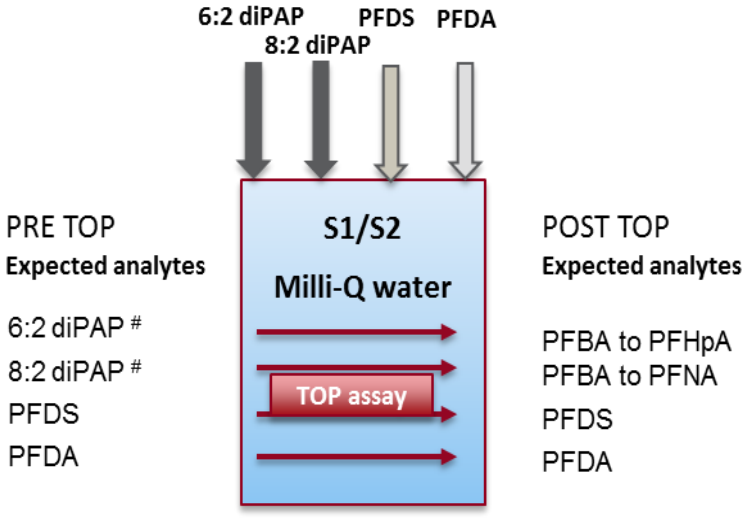
- method capability in converting precursors to PFCA;
- analyte losses during preparation;
- precursors conversion to PFSA; and
- complete or near complete oxidation.

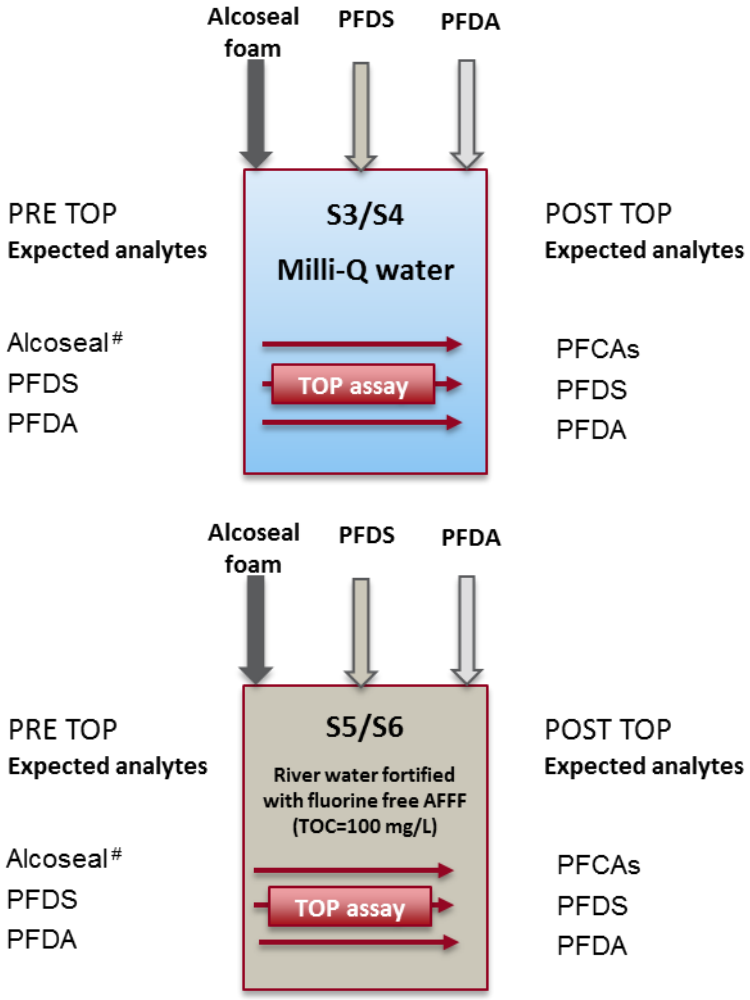
6:2 diPAP and 8:2 diPAP in S1/S2 and Alcoseal foam in S3/S4 and S5/S6 were used to assess method capability in converting precursors to PFCAs with complete or near complete precursor oxidation.

PFDS and PFDA were used in this study as monitoring compounds. Where there are no precursors which can oxidise to PFDA or PFDS, then the respective pre- and post-oxidation concentrations should be similar. In S1/S2, there were no precursors added which were expected to form PFDA or PFDS on oxidation, and in S3/S4 and S5/S6, while participants reported the formation of long chain PFCAs wich suggests formation of PFDA is likely, the amount of PFDA formed on oxidation is expected to be small compared to the spiked amount. In addition, as no participants reported an increase in other PFSA post-oxidation, it is assumed the precursors in these samples are unable to produce PFDS during oxidation.

Samples S3/S4 were MilliQ water while Samples S5/S6 were river water, further fortified with a fluorine free AFFF for a TOC content of 100 mg/L. The concentration of PFCA precursors in the MilliQ water and river water was intended to be similar in order to evaluate the effects caused by differences in matrices. However, this was not possible for all compounds as some were present in the river water used to prepare the samples.

The design of the study is graphically presented in Figure 63.





Not analysed by participating laboratories

Figure 63 Study Design

7.2 Test for Acceptability of Oxidative Methods

The assessment of the oxidative methods used by participants is based on the criteria stipulated in PFAS NEMP published in January 2020 for aqueous samples (Table 60).[5]

Table 60 Criteria for Oxidative Methos Acceptability as per PFAS NEMP

Method Assessment	Criteria
Analyte losses during preparation process	Total PFAS post-TOP ≥ Total PFAS pre-TOP*
Precursors conversion to PFCA products	Sum PFCA post-TOP ≥ Sum of PFCA pre-TOP
Precursor conversion to PFSA products	Sum of PFSA post-TOP = Sum of PFSA pre-TOP
Oxidation completeness	No PFAA precursor
Near complete oxidation	Sum of PFAA precursors post-TOP divided by sum of Total PFAS post-TOP < 5%

*A decrease of up to 10% might be expected due to normal analytical variability

Laboratory 1

Table 61

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	2.2
			PFPeA	4.4
			PFHxA	5.5
			PFHpA	9.5
			PFOA	7.2
			PFNA	2.2
PFDA 6.42 µg/L	PFDA	7.1	PFDA	5.4
PFDS 3.14 µg/L	PFDS	2.5	PFDS	1.5
	PFCA	7.14	PFCA	36.4
	PFSA	2.5	PFSA	1.5
	*PFAA precursor	ND	PFAA precursor	ND
	PFAS	9.64	PFAS	37.9

Table 62

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	8.80	PFCA	162
	PFSA	3.9	PFSA	4.2
	PFAA precursor	3.8	PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	8.7	PFDA	5.5
PFDS 6.51 µg/L	PFDS	3.9	PFDS	4.2
	PFAS	16.5	PFAS	167

Table 63

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	10	PFCA	110
	PFSA	4	PFSA	3.69
	PFAA precursor	3.8	PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	10	PFDA	6.4
PFDS 6.51 µg/L	PFDS	4.0	PFDS	3.6
	PFAS	17.8	PFAS	114

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP / SUM PFSA pre-TOP	-40%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP<5%	Pass

ND= Not Detected

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP / SUM PFSA pre-TOP	7.7%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP / SUM PFSA pre-TOP	-7.7%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

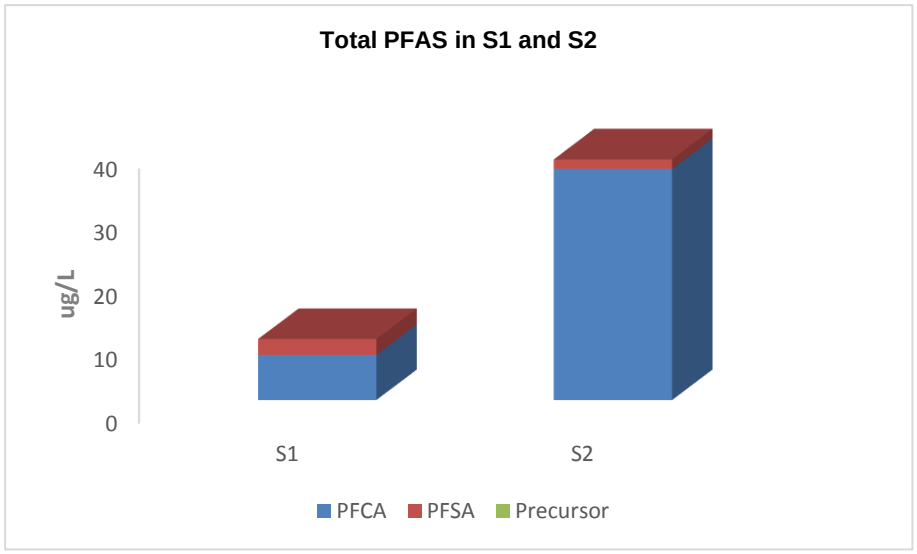


Figure 64 Laboratory 1 Results for Total PFAS in S1 and S2

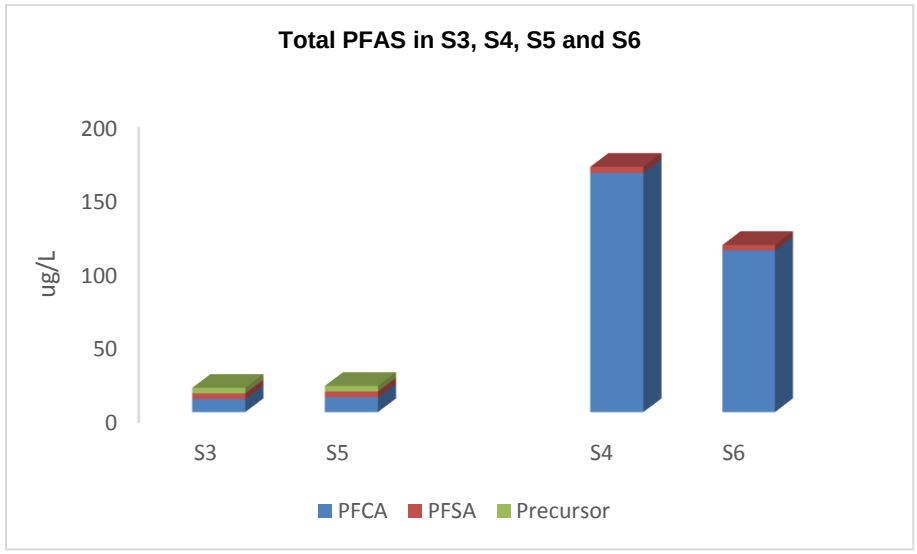


Figure 65 Laboratory 1 Results for Total PFAS in S3, S4, S5 and S6

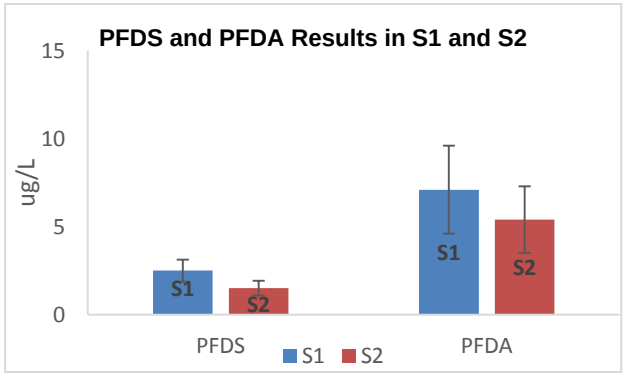


Figure 66 Laboratory 1 Results for PFDS and PFDA in S1 and S2

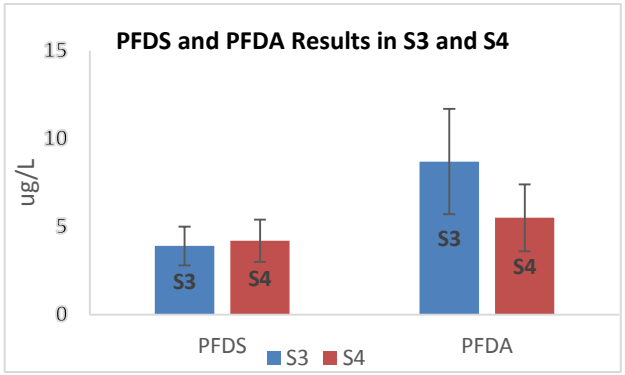


Figure 67 Laboratory 1 Results for PFDS and PFDA in S3 and S4

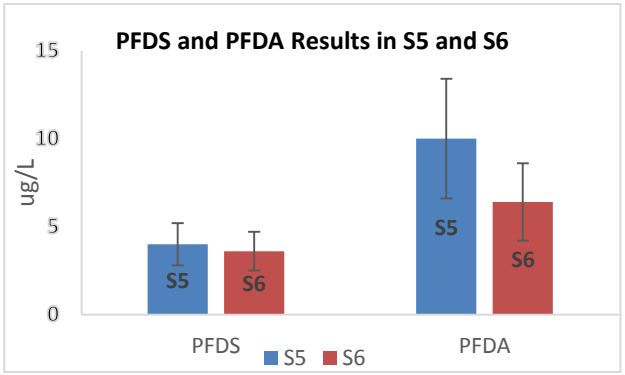


Figure 68 Laboratory 1 Results for PFDS and PFDA in S5 and S6

PFDS Change: -40%; **PFDA Change:** -24%

PFDS Change: 7.7%; **PFDA Change:** -37%

PFDS Change: -10%; **PFDA Change:** -36%

Laboratory 3[#]

Table 64

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	1.75
			PFPeA	3.7
			PFHxA	4.89
			PFHpA	7.86
			PFOA	5.48
			PFNA	3
PFDA 6.42 µg/L	PFDA	6.86	PFDA	6.02
PFDS 3.14 µg/L	PFDS	2.2	PFDS	2.21
	PFCA	6.89	PFCA	32.7
	PFSA	2.2	PFSA	2.21
	PFAA precursor	ND	PFAA precursor	0.00447
	PFAS	9.09	PFAS	34.9

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP / SUM PFSA pre-TOP	0.6%
No PFAA precursor in post oxidation	Fail
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 65

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	7.96	PFCA	104
	PFSA	1.08	PFSA	4.04
	PFAA precursor	4.63	PFAA precursor	0.419
PFDA 9.08 µg/L	PFDA	7.85	PFDA	6.79
PFDS 6.51 µg/L	PFDS	1.08	PFDS	4.04
	PFAS	13.7	PFAS	108

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP / SUM PFSA pre-TOP	274%
No PFAA precursor in post oxidation	Fail
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 66

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	10.2	PFCA	87.6
	PFSA	2.39	PFSA	3.93
	PFAA precursor	4.37	PFAA precursor	0.193
PFDA 9.08 µg/L	PFDA	9.95	PFDA	9.87
PFDS 6.51 µg/L	PFDS	2.38	PFDS	3.7
	PFAS	17.0	PFAS	91.8

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP /SUM PFSA pre-TOP	64%
No PFAA precursor in post oxidation	Fail
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

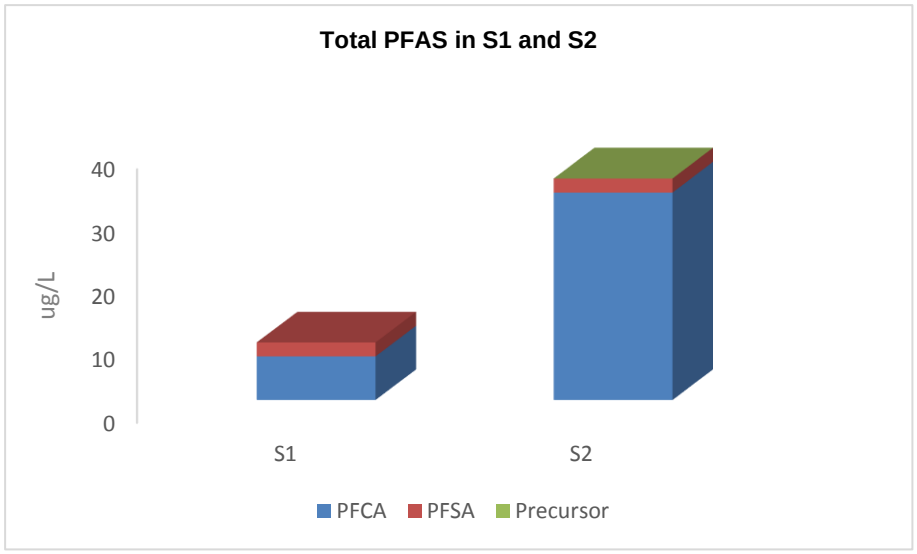


Figure 69 Laboratory 3 Results for Total PFAS in S1 and S2

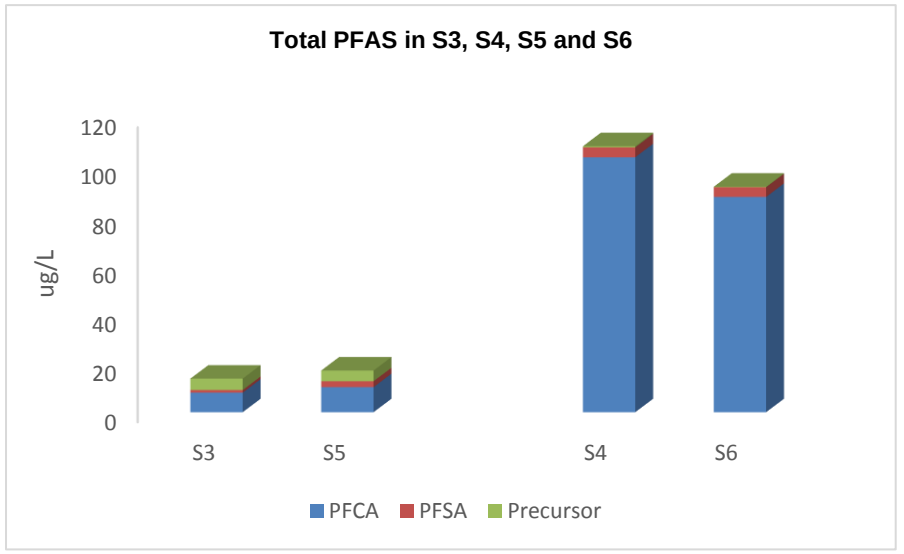


Figure 70 Laboratory 3 Results for Total PFAS in S3, S4, S5 and S6

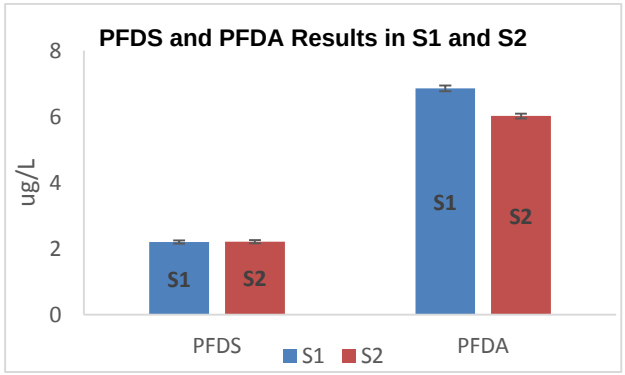


Figure 71 Laboratory 3 Results for PFDS and PFDA in S1 and S2

PFDS Change: 0.5%; **PFDA Change:** -12%

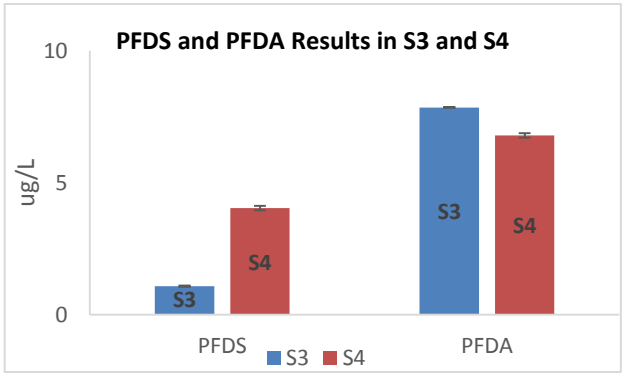


Figure 72 Laboratory 3 Results for PFDS and PFDA in S3 and S4

PFDS Change: 270%; **PFDA Change:** -14%

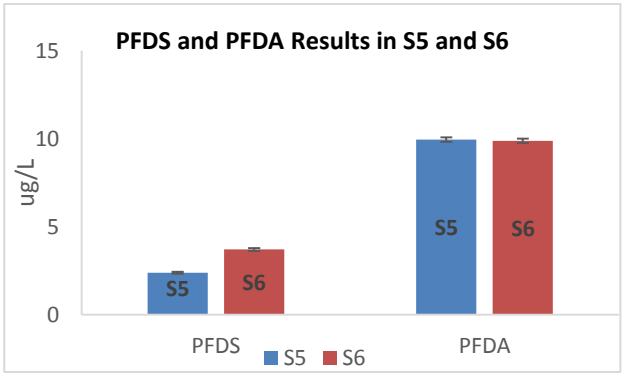


Figure 73 Laboratory 3 Results for PFDS and PFDA in S5 and S6

PFDS Change: 55%; **PFDA Change:** -0.8%

[#]The study coordinator considers the change in PFDS in S4 and S6 to be excessive, and such the oxidative method should be reviewed.

Laboratory 4

Table 67

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	2.42
			PFPeA	5.02
			PFHxA	5.31
			PFHpA	11.2
			PFOA	6.31
			PFNA	1.39
PFDA 6.42 µg/L	PFDA	6.29	PFDA	1.31
PFDS 3.14 µg/L	PFDS	2.23	PFDS	0.329
	PFCA	6.35	PFCA	33.0
	PFSA	2.23	PFSA	0.329
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	8.58	PFAS	33.3

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-85.3%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND = Not Detected

Table 68

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	8.11	PFCA	171
	PFSA	5.24	PFSA	0.572
	PFAA precursor	4.58	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	7.98	PFDA	1.23
PFDS 6.51 µg/L	PFDS	5.24	PFDS	0.57
	PFAS	17.9	PFAS	172

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-89.1%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 69

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	12.9	PFCA	197
	PFSA	6.30	PFSA	0.66
	PFAA precursor	4.99	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	12.6	PFDA	2.39
PFDS 6.51 µg/L	PFDS	6.30	PFDS	0.66
	PFAS	24.2	PFAS	197

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-89.5%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

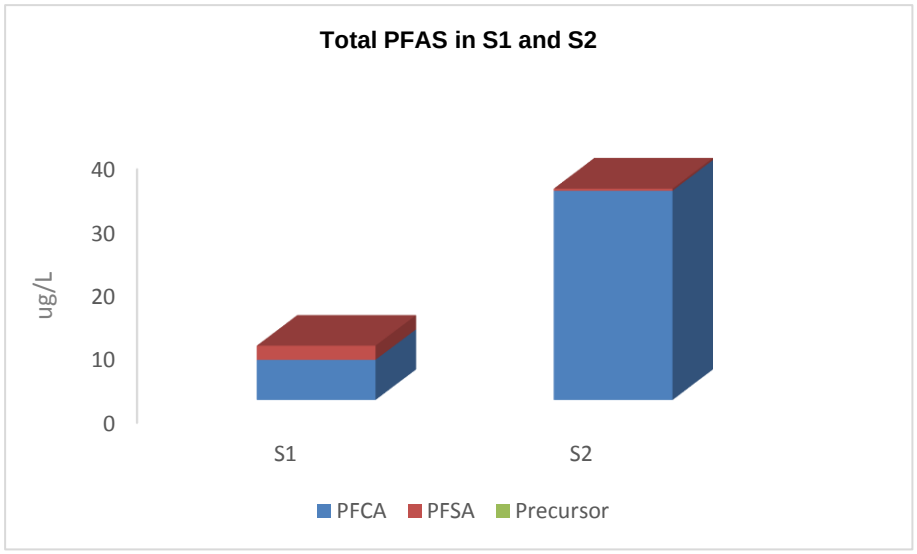


Figure 74 Laboratory 4 Results for Total PFAS in S1 and S2

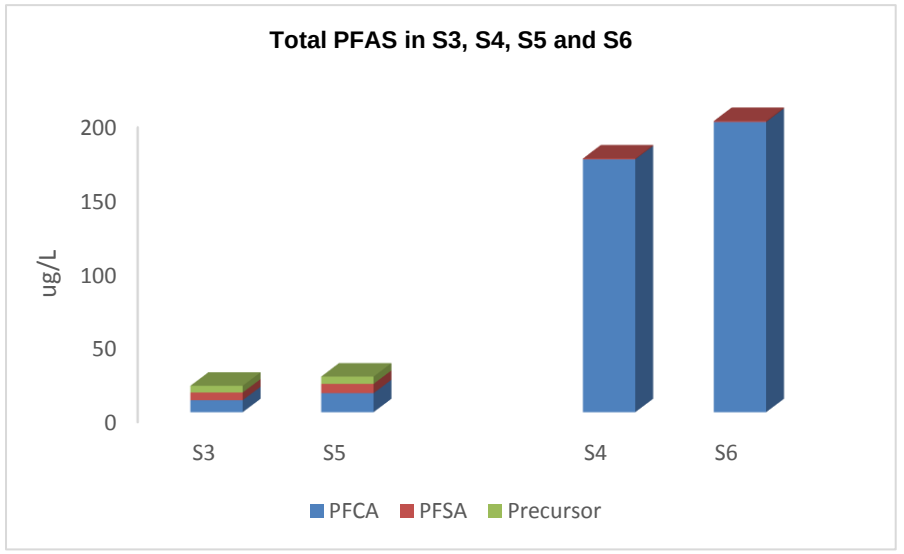


Figure 75 Laboratory 4 Results for Total PFAS in S3, S4, S5 and S6

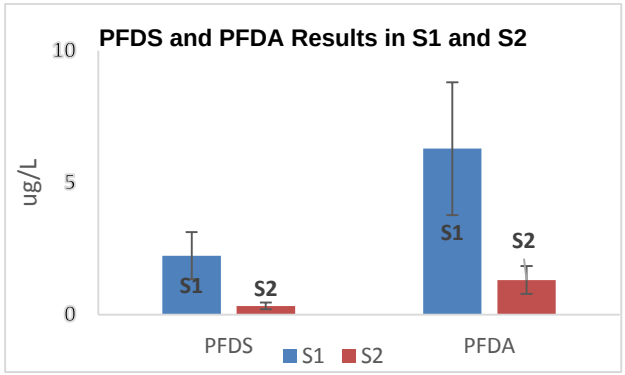


Figure 76 Laboratory 4 Results for PFDS and PFDA in S1 and S2

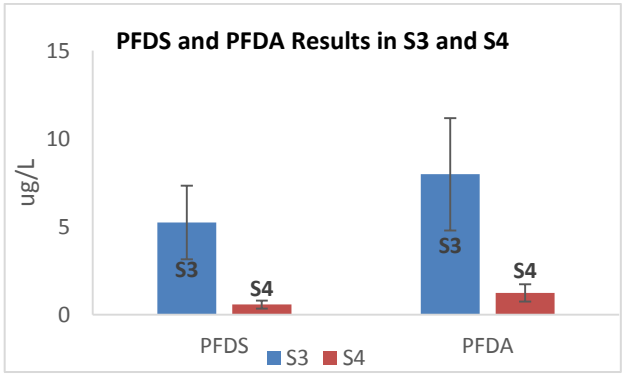


Figure 77 Laboratory 4 Results for PFDS and PFDA in S3 and S4

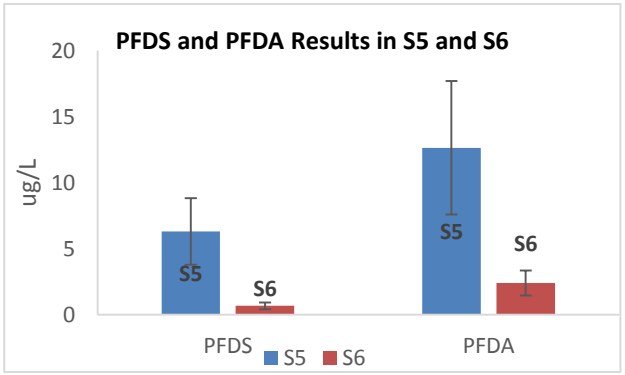


Figure 78 Laboratory 4 Results for PFDS and PFDA in S5 and S6

PFDS Change: -85%; **PFDA Change:** -79% **;**

PFDS Change: -89%; **PFDA Change:** -85%

PFDS Change: -90%**; PFDA Change:** -81% **;**

[#]The study coordinator considers the change in PFDA and PFDS in S2, S4 and S6 to be excessive, and such the oxidative method should be reviewed.

Laboratory 5[#]

Table 70

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	2.04
			PFPeA	4.64
			PFHxA	4.61
			PFHpA	9.06
			PFOA	4.84
			PFNA	1.56
PFDA 6.42 µg/L	PFDA	8.93	PFDA	8.81
PFDS 3.14 µg/L	PFDS	3.66	PFDS	2.99
	PFCA	9.00	PFCA	35.6
	PFSA	3.66	PFSA	2.99
	PFAA precursor	0.014	PFAA precursor	0.003
	PFAS	12.7	PFAS	38.6

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-18.3%
No PFAA precursor in post oxidation	Fail
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 71

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	11.5	PFCA	119
	PFSA	1.97	PFSA	4.70
	PFAA precursor	3.48	PFAA precursor	0.031
PFDA 9.08 µg/L	PFDA	11.3	PFDA	12.8
PFDS 6.51 µg/L	PFDS	1.97	PFDS	4.68
	PFAS	16.9	PFAS	124

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	139%
No PFAA precursor in post oxidation	Fail
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 72

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	14.0	PFCA	117
	PFSA	5.89	PFSA	5.12
	PFAA precursor	2.84	PFAA precursor	0.024
PFDA 9.08 µg/L	PFDA	13.6	PFDA	13.6
PFDS 6.51 µg/L	PFDS	5.85	PFDS	5.06
	PFAS	22.7	PFAS	122

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-13.0%
No PFAA precursor in post oxidation	Fail
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

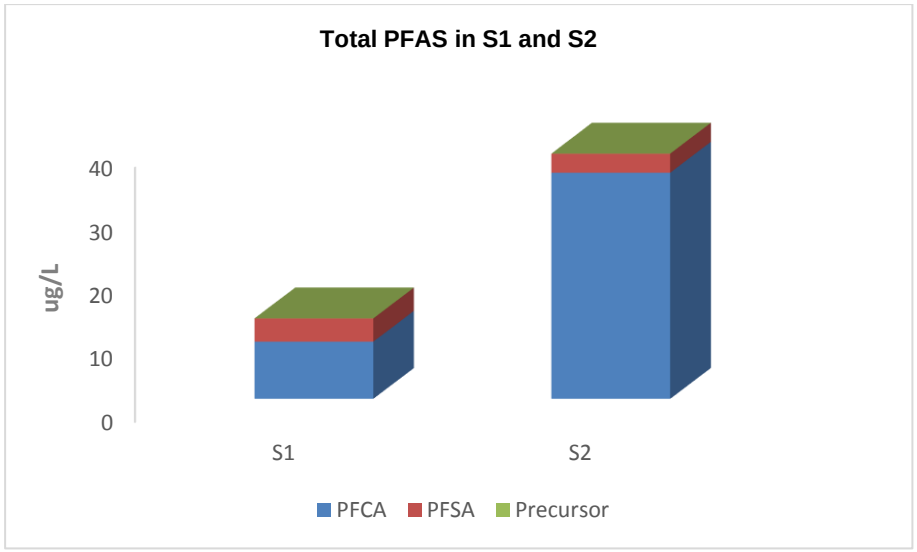


Figure 79 Laboratory 5 Results for Total PFAS in S1 and S2

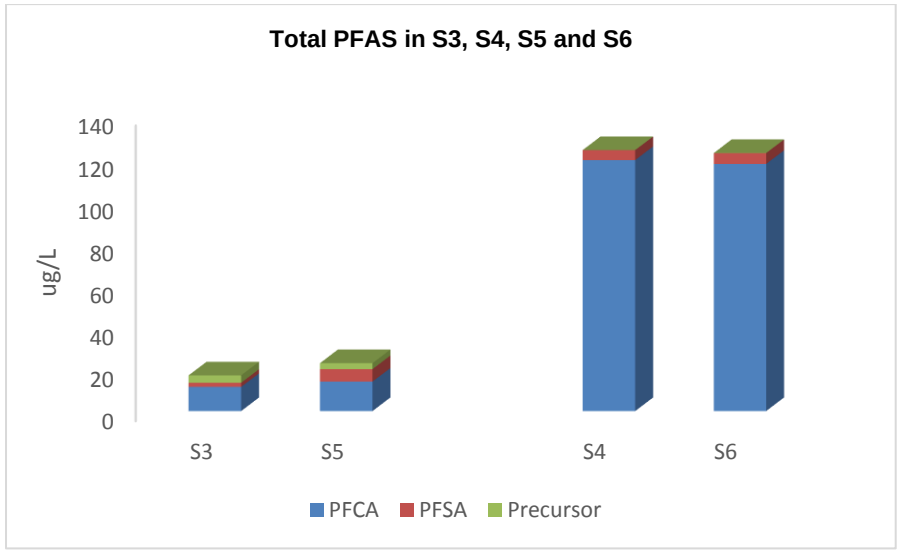


Figure 80 Laboratory 5 Results for Total PFAS in S3, S4, S5 and S6

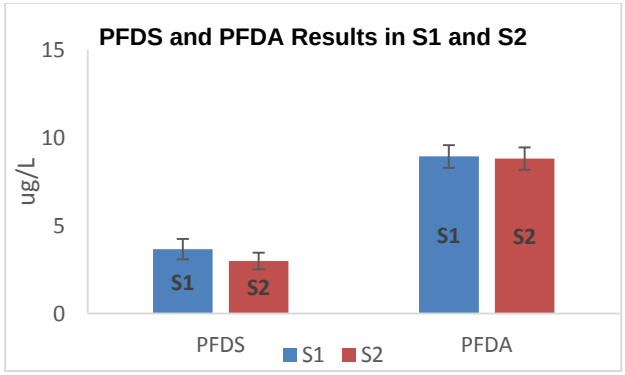


Figure 81 Laboratory 5 Results for PFDS and PFDA in S1 and S2

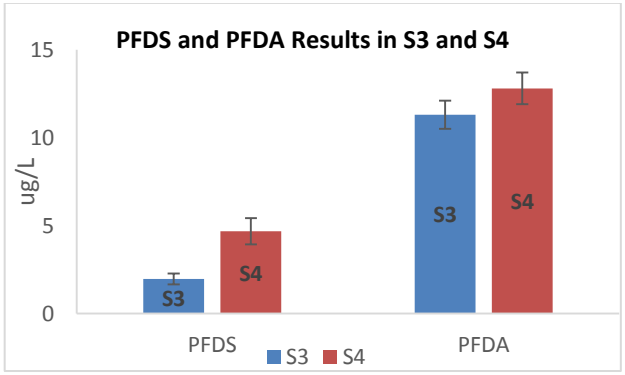


Figure 82 Laboratory 5 Results for PFDS and PFDA in S3 and S4

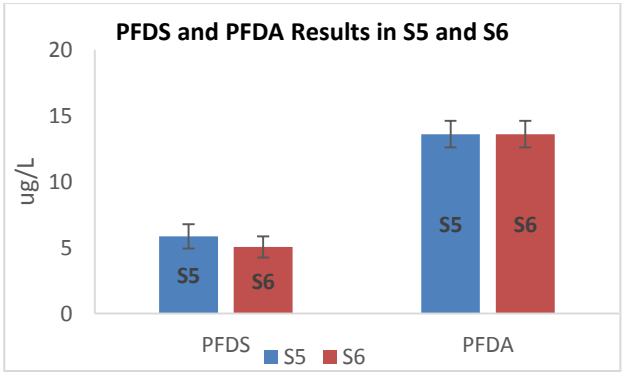


Figure 83 Laboratory 5 Results for PFDS and PFDA in S5 and S6

PFDS Change: -18%**; PFDA Change:** -1.3%

PFDS Change: 140%; **PFDA Change:** 13%

PFDS Change: -14%; **PFDA Change:** 0%

[#]The study coordinator considers the change in PFDS in S4 to be excessive, and such the oxidative method should be reviewed.

Laboratory 7[#]

Table 73

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	<5.0
			PFPeA	3.5
			PFHxA	4.92
			PFHpA	7.42
			PFOA	6.15
			PFNA	2.67
PFDA 6.42 µg/L	PFDA	6.35	PFDA	6
PFDS 3.14 µg/L	PFDS	2.29	PFDS	1.44
	PFCA	6.38	PFCA	30.7
	PFSA	2.29	PFSA	1.44
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	8.67	PFAS	32.1

Table 74

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	6.86	PFCA	81.5
	PFSA	4.91	*PFSA	ND
	PFAA precursor	4.86	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	6.78	PFDA	<5.0
PFDS 6.51 µg/L	PFDS	4.91	PFDS	<5.0
	PFAS	16.6	PFAS	81.5

Table 75

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	10.6	PFCA	118
	PFSA	6.69	PFSA	5.9
	PFAA precursor	4.26	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	10.4	PFDA	9.6
PFDS 6.51 µg/L	PFDS	6.68	PFDS	5.9
	PFAS	21.6	PFAS	124

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-37.1%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND=Not Dtected

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-100%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-11.8%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

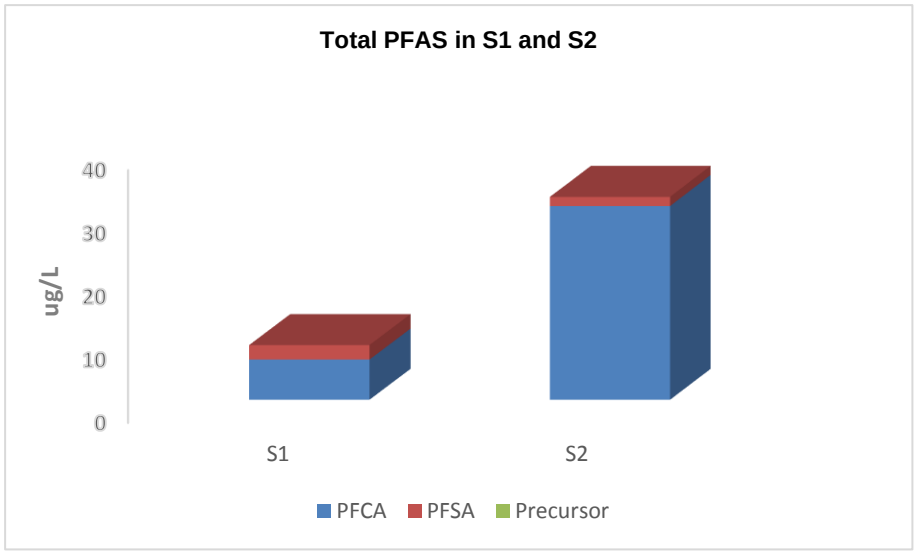


Figure 84 Laboratory 7 Results for Total PFAS in S1 and S2

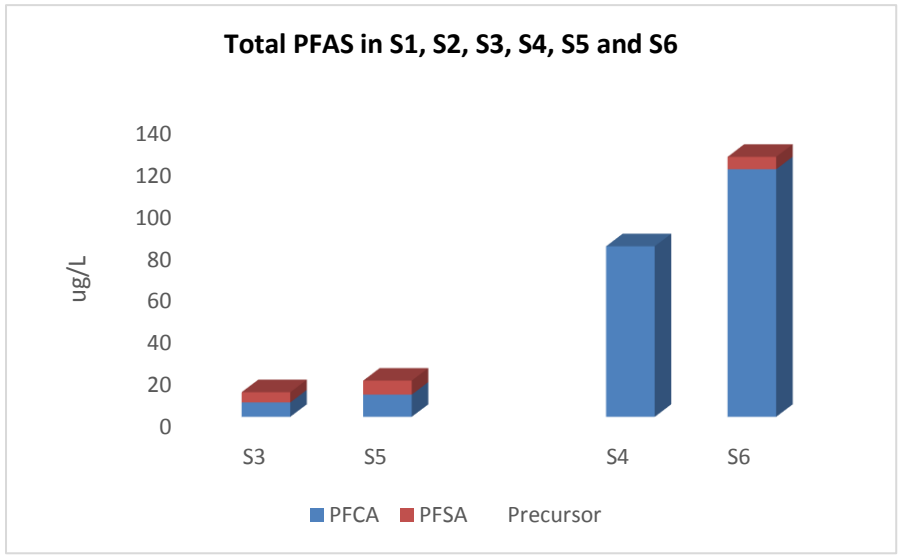


Figure 85 Laboratory 7 Results for Total PFAS in S3, S4, S5 and S6

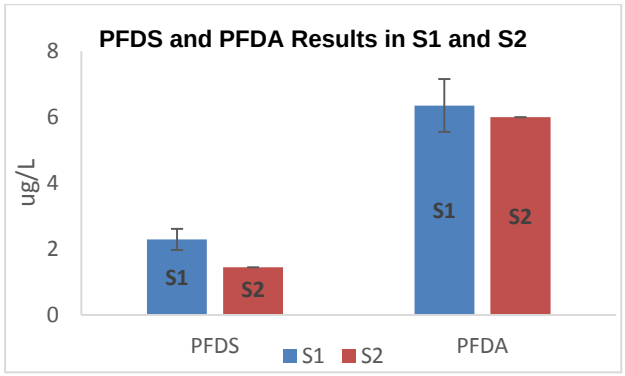


Figure 86 Laboratory 7 Results for PFDS and PFDA in S1 and S2

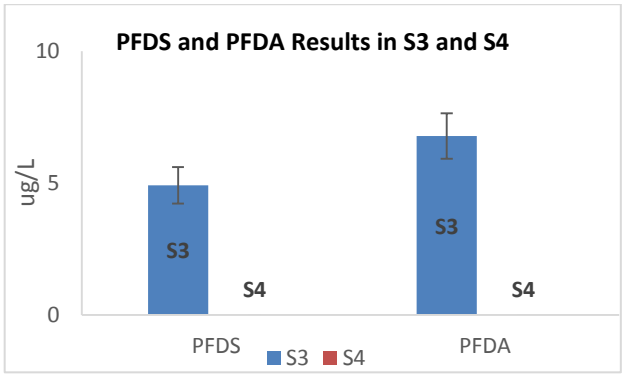


Figure 87 Laboratory 7 Results for PFDS and PFDA in S3 and S4

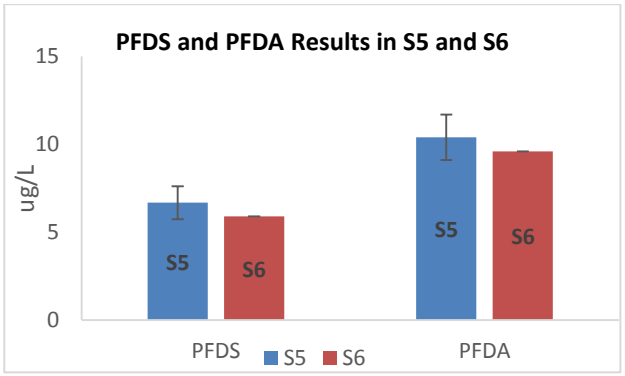


Figure 88 Laboratory 7 Results for PFDS and PFDA in S5 and S6

PFDS Change: -37%; **PFDA Change:** -5.5% **;**

PFDS Change: -100%**; PFDA Change:** -100%

PFDS Change: -12%; **PFDA Change:** -7.7%

#The study coordinator considers the change in PFDA and PFDS in S4 to be excessive, and such the oxidative method should be reviewed.

Laboratory 8

Table 76

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	NR
			PFPeA	NR
			PFHxA	NR
			PFHpA	NR
			PFOA	NR
			PFNA	0.061
PFDA 6.42 µg/L	PFDA	7.3	PFDA	6.35
PFDS 3.14 µg/L	PFDS	3.08	PFDS	2.16
	PFCA	7.36	PFCA	6.41
	PFSA	3.08	PFSA	2.16
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	10.4	PFAS	8.57

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Fail
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Fail
SUM PFSA post-TOP = SUM PFSA pre-TOP	-29.9%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND = Not Detected

Table 77

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	5.39	PFCA	6.57
	PFSA	4.61	PFSA	4.22
	PFAA precursor	6.41	PFAA precursor	37.1
PFDA 9.08 µg/L	PFDA	5.27	PFDA	4.31
PFDS 6.51 µg/L	PFDS	4.61	PFDS	4.22
	PFAS	16.4	PFAS	47.8

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-8.5%
No PFAA precursor in post oxidation	Fail
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Fail

Table 78

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	10.9	PFCA	12.8
	PFSA	8.22	PFSA	5.91
	PFAA precursor	6.49	PFAA precursor	45.8
PFDA 9.08 µg/L	PFDA	10.6	PFDA	9.51
PFDS 6.51 µg/L	PFDS	8.22	PFDS	5.91
	PFAS	25.6	PFAS	64.5

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-28.1%
No PFAA precursor in post oxidation	Fail
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Fail

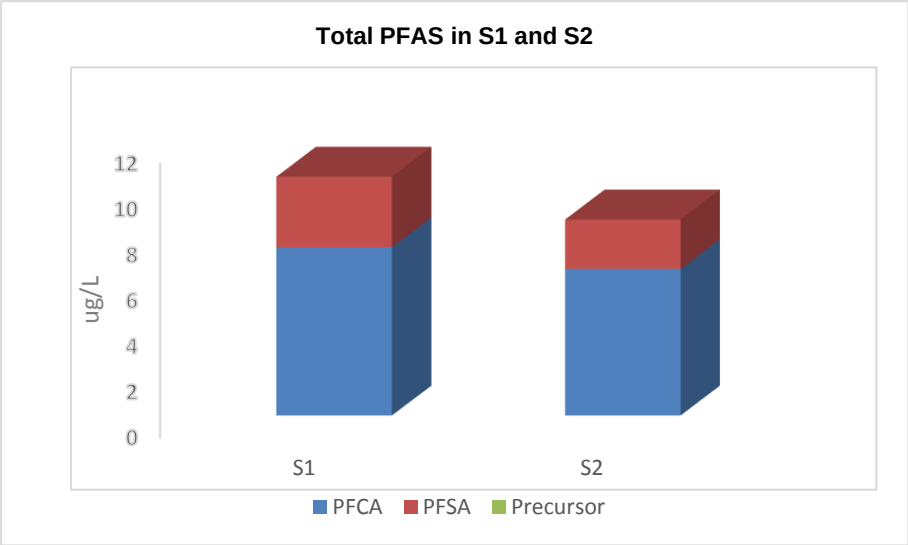


Figure 89 Laboratory 8 Results for Total PFAS in S1 and S2

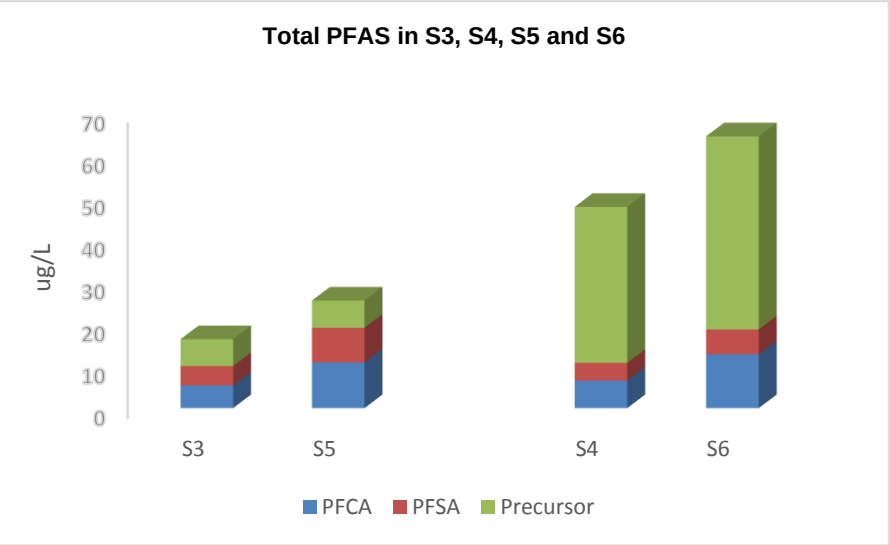


Figure 90 Laboratory 8 Results for Total PFAS in S3, S4, S5 and S6

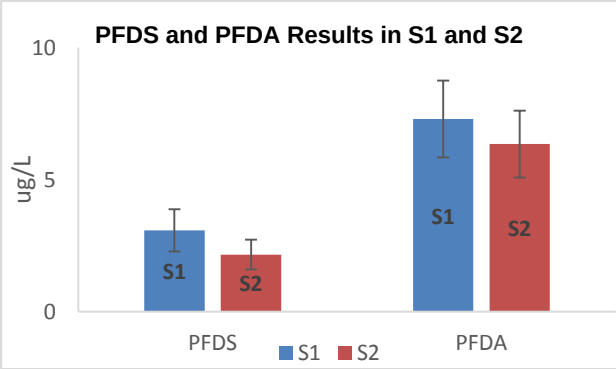


Figure 91 Laboratory 8 Results for PFDS and PFDA in S1 and S2

PFDS Change: -30%**; PFDA Change:** -13%

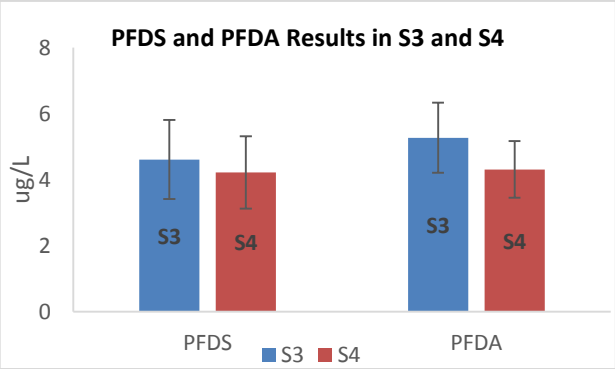


Figure 92 Laboratory 8 Results for PFDS and PFDA in S3 and S4

PFDS Change: -8.5%; **PFDA Change:** -18%

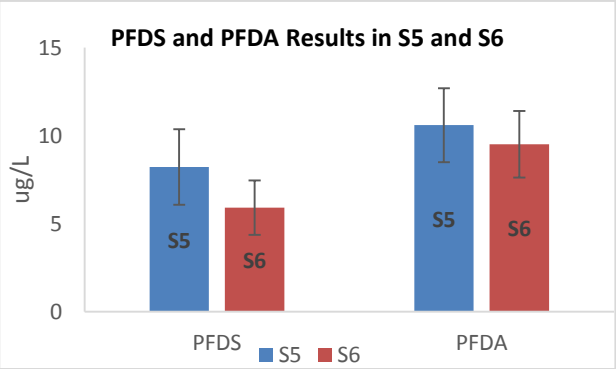


Figure 93 Laboratory 8 Results for PFDS and PFDA in S5 and S6

PFDS Change: -28%**; PFDA Change:** -10% **;**

Laboratory 9

Table 79

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	1.8
			PFPeA	3.9
			PFHxA	5.1
			PFHpA	7.4
			PFOA	5.9
			PFNA	2.7
PFDA 6.42 µg/L	PFDA	6.3	PFDA	5.5
PFDS 3.14 µg/L	PFDS	2.6	PFDS	2.6
	PFCA	6.33	PFCA	32.3
	PFSA	2.6	PFSA	2.6
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	8.93	PFAS	34.9

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	0.0%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND = Not Detected

Table 80

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	6.42	PFCA	121
	PFSA	4.1	PFSA	4.2
	PFAA precursor	3.9	PFAA precursor	0.11
PFDA 9.08 µg/L	PFDA	6.3	PFDA	6.4
PFDS 6.51 µg/L	PFDS	4.1	PFDS	4.2
	PFAS	14.4	PFAS	126

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	2.4%
No PFAA precursor in post oxidation	Fail
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 81

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	8.85	PFCA	87.3
	PFSA	3.9	PFSA	4.00
	PFAA precursor	3.8	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	8.6	PFDA	7.9
PFDS 6.51 µg/L	PFDS	3.9	PFDS	3.8
	PFAS	16.6	PFAS	91.3

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	2.7%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

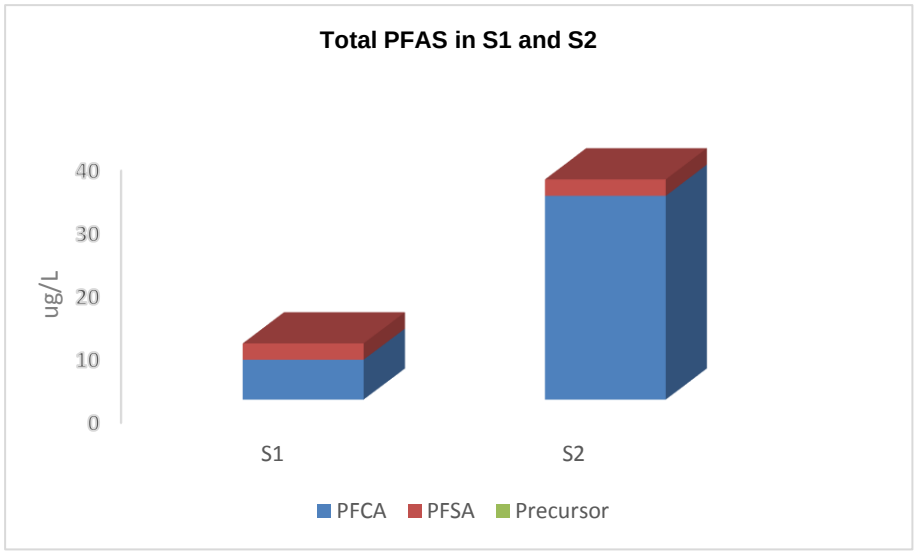


Figure 94 Laboratory 9 Results for Total PFAS in S1 and S2

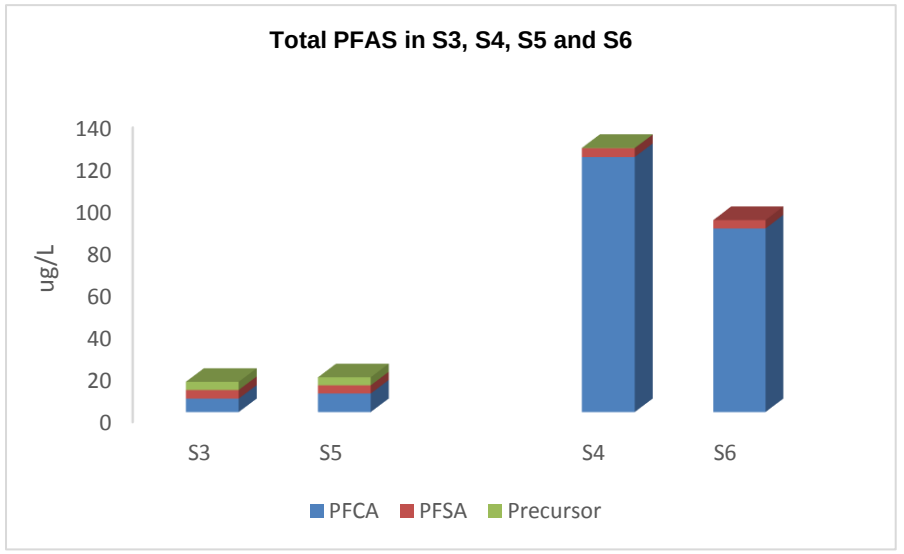


Figure 95 Laboratory 9 Results for Total PFAS in S3, S4, S5 and S6

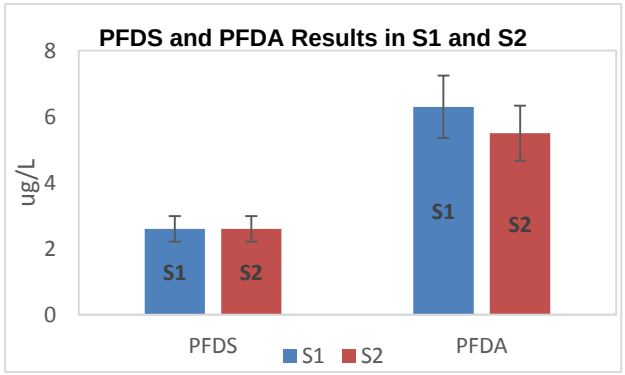


Figure 96 Laboratory 9 Results for PFDS and PFDA in S1 and S2

PFDS Change: 0%; **PFDA Change:** -13%

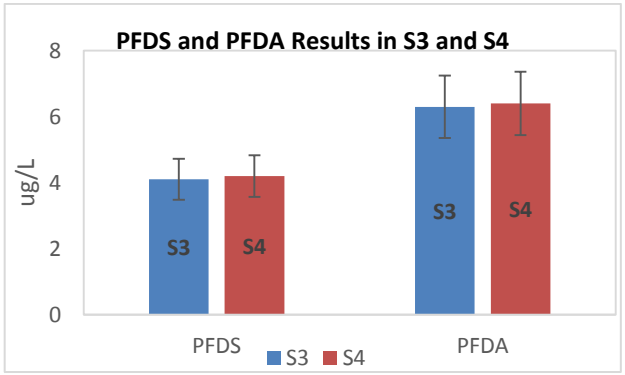


Figure 97 Laboratory 9 Results for PFDS and PFDA in S3 and S4

PFDS Change: 2.4%; **PFDA Change:** 1.6%

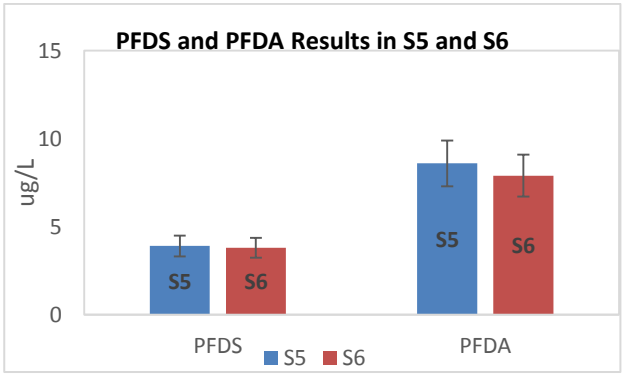


Figure 98 Laboratory 9 Results for PFDS and PFDA in S5 and S6

PFDS Change: -2.6%; **PFDA Change:** -8.1%

Laboratory 10[#]

Table 82

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	1.8
			PFPeA	3.87
			PFHxA	4.76
			PFHpA	8.12
			PFOA	7.62
			PFNA	3.77
PFDA 6.42 µg/L	PFDA	6.44	PFDA	4.4
PFDS 3.14 µg/L	PFDS	1.19	PFDS	0.9
	PFCA	6.44	PFCA	34.3
	PFSA	1.19	PFSA	0.9
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	7.63	PFAS	35.2

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-24.4%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND = Not Detected

Table 83

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	6.77	PFCA	172
	PFSA	3.98	PFSA	1.9
	PFAA precursor	4.48	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	6.72	PFDA	3.85
PFDS 6.51 µg/L	PFDS	3.98	PFDS	1.9
	PFAS	15.2	PFAS	174

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-52.3%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 84

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	11.5	PFCA	185
	PFSA	5.66	PFSA	1.3
	PFAA precursor	4.51	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	11.1	PFDA	5.66
PFDS 6.51 µg/L	PFDS	5.6	PFDS	1.3
	PFAS	21.6	PFAS	186

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-77.0%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

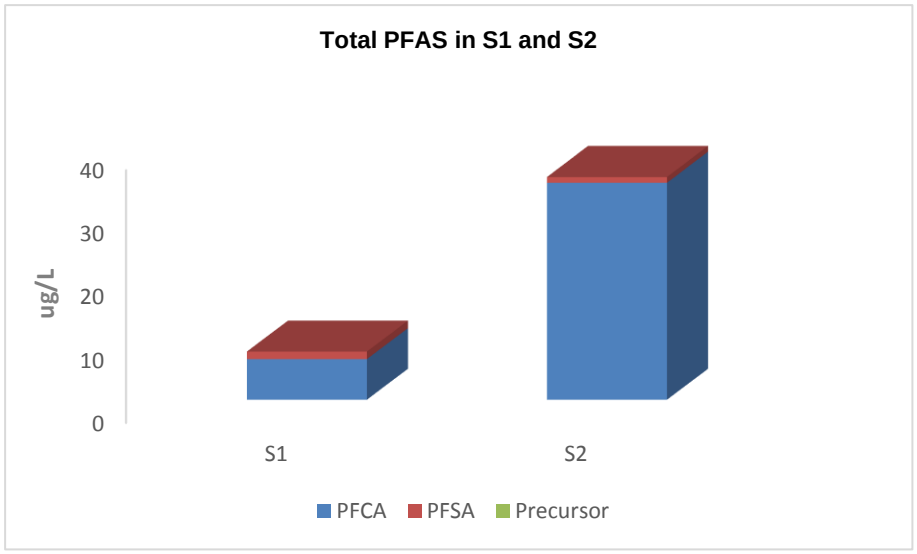


Figure 99 Laboratory 10 Results for Total PFAS in S1 and S2

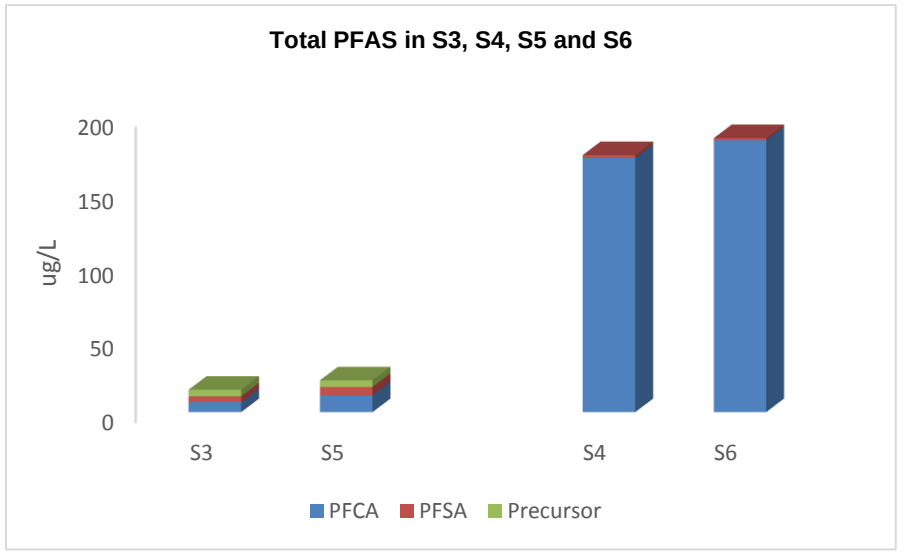


Figure 100 Laboratory 10 Results for Total PFAS in S3, S4, S5 and S6

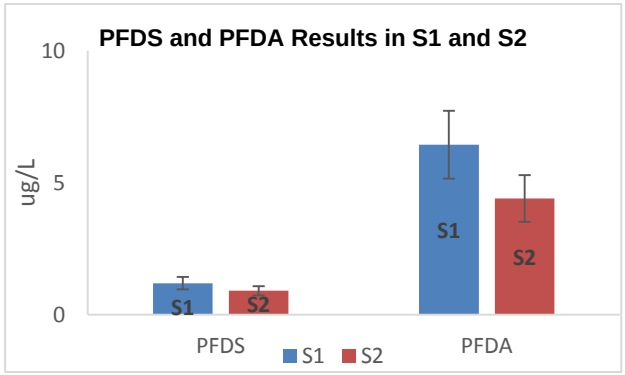


Figure 101 Laboratory 10 Results for PFDS and PFDA in S1 and S2

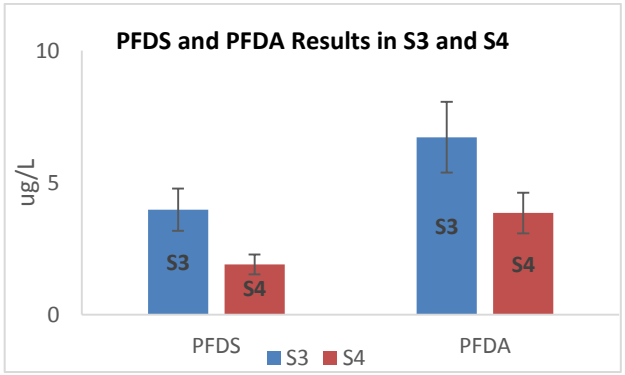


Figure 102 Laboratory 10 Results for PFDS and PFDA in S3 and S4

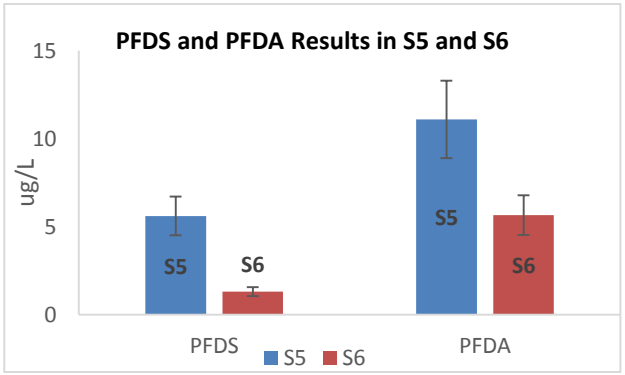


Figure 103 Laboratory 10 Results for PFDS and PFDA in S5 and S6

PFDS Change: -24%; **PFDA Change:** -32%

PFDS Change: -52%; **PFDA Change:** -43%

PFDS Change: -77%; **PFDA Change:** -49% **;**

#The study coordinator considers the change in PFDS in S4 and S6 to be excessive, and such the oxidative method should be reviewed.

Laboratory 11

Table 85

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	2.1
			PFPeA	4.3
			PFHxA	5.4
			PFHpA	8.4
			PFOA	6.5
			PFNA	1.9
PFDA 6.42 µg/L	PFDA	7.4	PFDA	6.9
PFDS 3.14 µg/L	PFDS	2.3	PFDS	2.2
	PFCA	7.45	PFCA	35.5
	PFSA	2.3	PFSA	2.2
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	9.75	PFAS	37.7

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-4.3%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND = Not Detected

Table 86

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	7.68	PFCA	157
	PFSA	6.6	PFSA	3.9
	PFAA precursor	3.54	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	7.5	PFDA	5.1
PFDS 6.51 µg/L	PFDS	6.6	PFDS	3.9
	PFAS	17.8	PFAS	161

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-40.9%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 87

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	12.4	PFCA	127
	PFSA	3.43	PFSA	3.33
	PFAA precursor	3.63	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	12	PFDA	11
PFDS 6.51 µg/L	PFDS	3.4	PFDS	3.3
	PFAS	19.5	PFAS	131

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-2.9%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

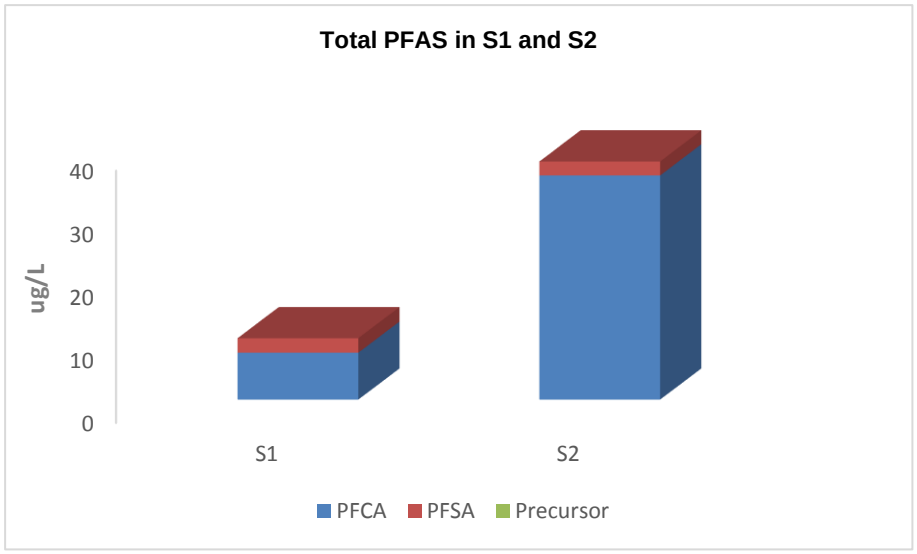


Figure 104 Laboratory 11 Results for Total PFAS in S1 and S2

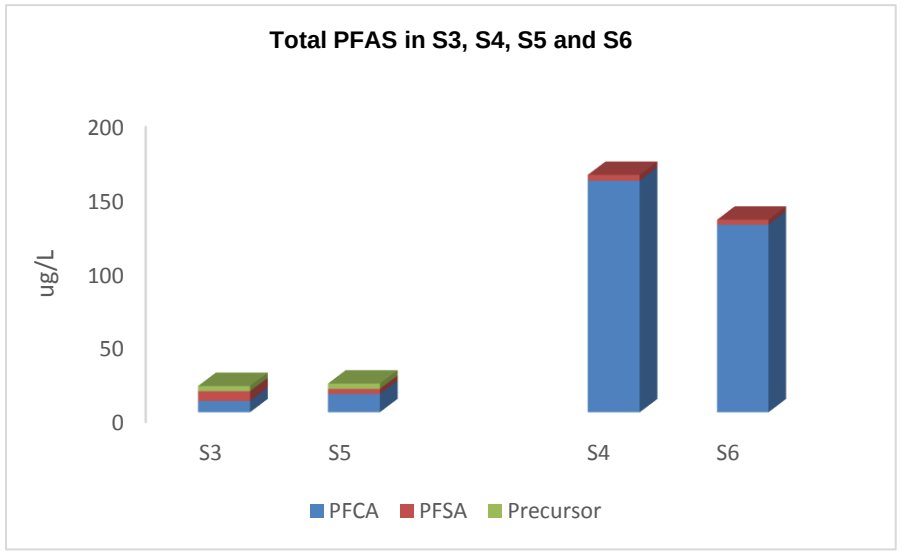


Figure 105 Laboratory 11 Results for Total PFAS in S3, S4, S5 and S6

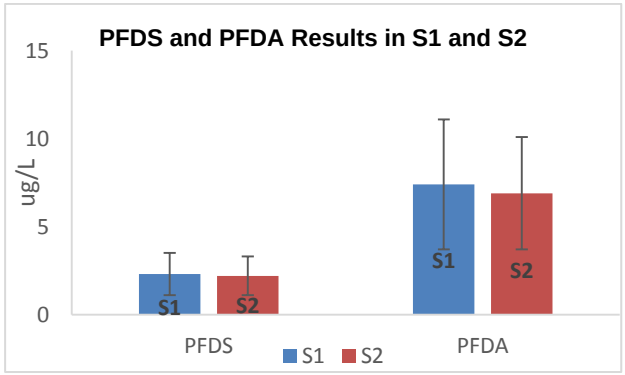


Figure 106 Laboratory 11 Results for PFDS and PFDA in S1 and S2

PFDS Change: -4.3%; **PFDA Change:** -6.8%

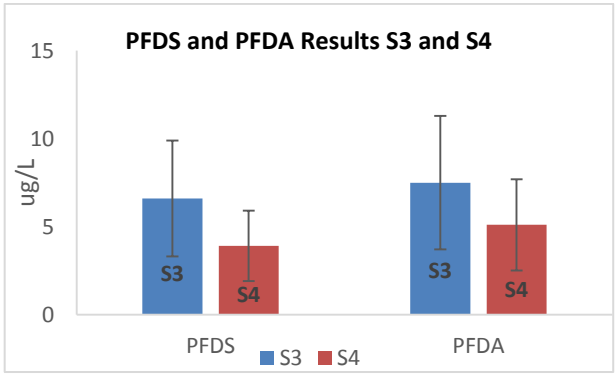


Figure 107 Laboratory 11 Results for PFDS and PFDA in S3 and S4

PFDS Change: -41%; **PFDA Change:** -32%

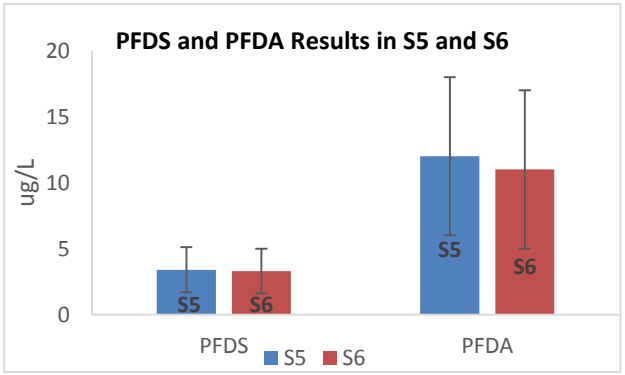


Figure 108 Laboratory 11 Results for PFDS and PFDA in S5 and S6

PFDS Change: -2.9%; **PFDA Change:** -8.3%

Laboratory 12

Table 88

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	1.62
			PFPeA	4.17
			PFHxA	4.78
			PFHpA	8.91
			PFOA	6.5
			PFNA	3.03
PFDA 6.42 µg/L	PFDA	6.53	PFDA	6.68
PFDS 3.14 µg/L	PFDS	2.48	PFDS	1.94
	PFCA	6.58	PFCA	35.7
	PFSA	2.48	PFSA	1.94
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	9.06	PFAS	37.6

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-21.8%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND = Not Detected

Table 89

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	6.40	PFCA	160
	PFSA	4.6	PFSA	3.63
	PFAA precursor	4.22	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	6.22	PFDA	6.35
PFDS 6.51 µg/L	PFDS	4.6	PFDS	3.63
	PFAS	15.2	PFAS	164

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-21.1%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 90

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	11.1	PFCA	166
	PFSA	5.73	PFSA	4.83
	PFAA precursor	4.33	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	10.8	PFDA	12.3
PFDS 6.51 µg/L	PFDS	5.72	PFDS	4.8
	PFAS	21.1	PFAS	171

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-15.8%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

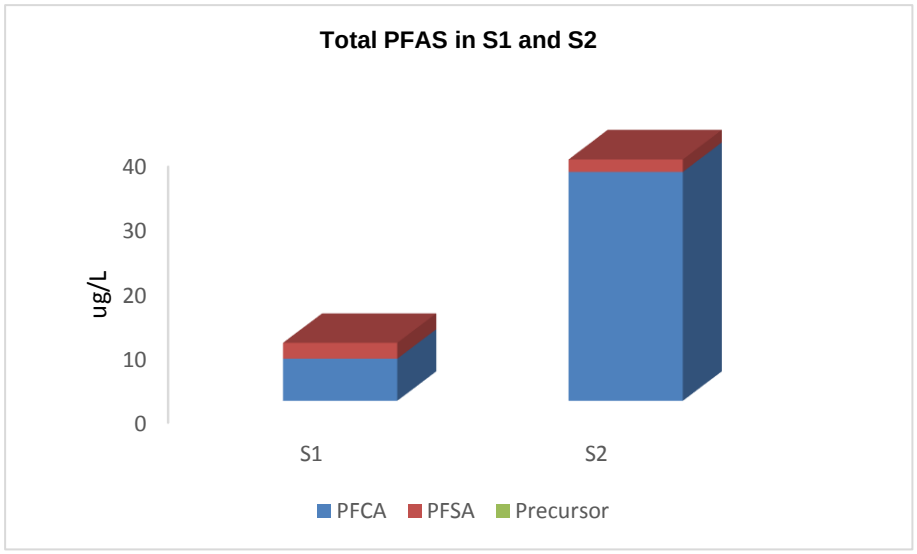


Figure 109 Laboratory 12 Results for Total PFAS in S1 and S2

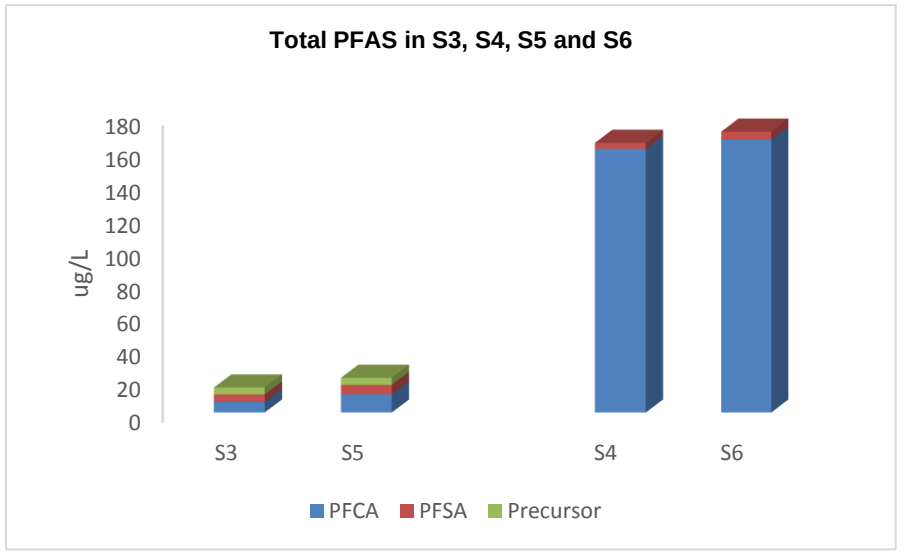


Figure 110 Laboratory 12 Results for Total PFAS in S3, S4, S5 and S6

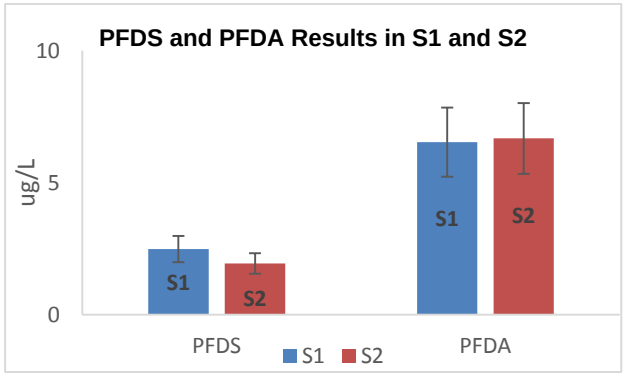


Figure 111 Laboratory 12 Results for PFDS and PFDA in S1 and S2

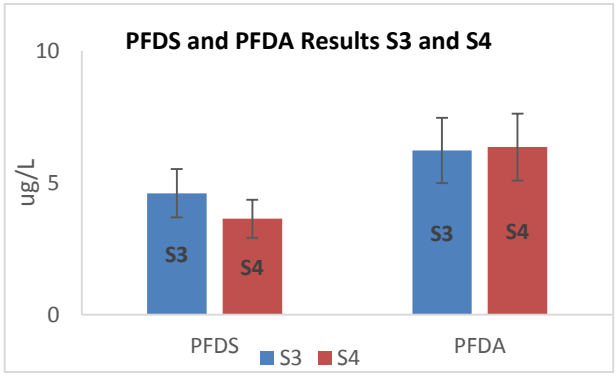


Figure 112 Laboratory 12 Results for PFDS and PFDA in S3 and S4

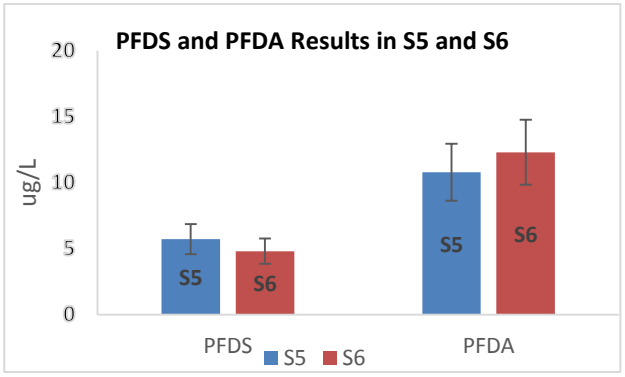


Figure 113 Laboratory 12 Results for PFDS and PFDA in S5 and S6

PFDS Change: -22%; **PFDA Change:** 2.3%

PFDS Change: -21%; **PFDA Change:** 2.1%

PFDS Change: -16%; **PFDA Change:** 14% **;**

Laboratory 13

Table 91

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	2
			PFPeA	3.4
			PFHxA	5.2
			PFHpA	6.1
			PFOA	8.3
			PFNA	3.2
PFDA 6.42 µg/L	PFDA	5.7	PFDA	5.1
PFDS 3.14 µg/L	PFDS	1.9	PFDS	1.7
	PFCA	5.73	PFCA	33.3
	PFSA	1.9	PFSA	1.7
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	7.63	PFAS	35

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-10.5%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 92

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	7.24	PFCA	141
	PFSA	3.5	PFSA	3.9
	PFAA precursor	4.61	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	7.1	PFDA	5.5
PFDS 6.51 µg/L	PFDS	3.5	PFDS	3.9
	PFAS	15.4	PFAS	145

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	11.4%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 93

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	9.03	PFCA	118
	PFSA	3.8	PFSA	3.8
	PFAA precursor	4.31	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	8.7	PFDA	7.8
PFDS 6.51 µg/L	PFDS	3.8	PFDS	3.8
	PFAS	17.1	PFAS	122

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	0.0%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

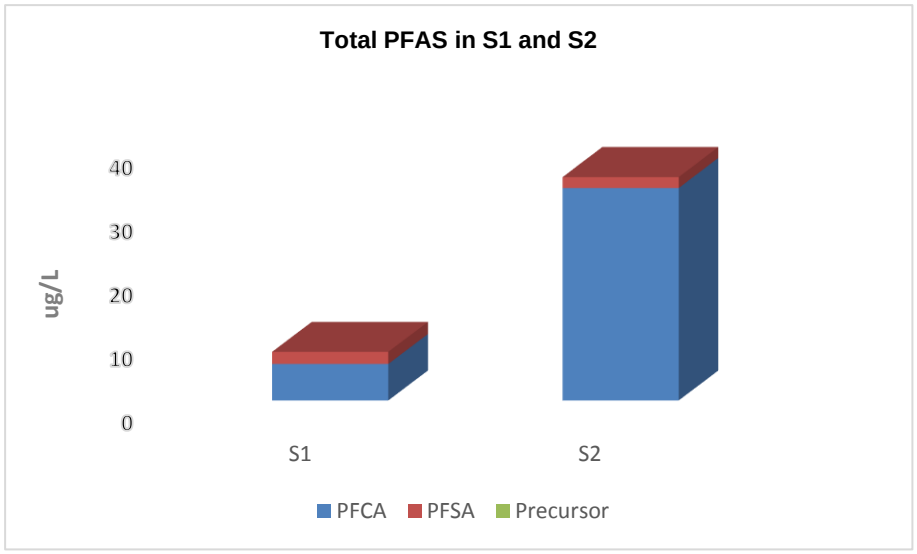


Figure 114 Laboratory 13 Results for Total PFAS in S1 and S2

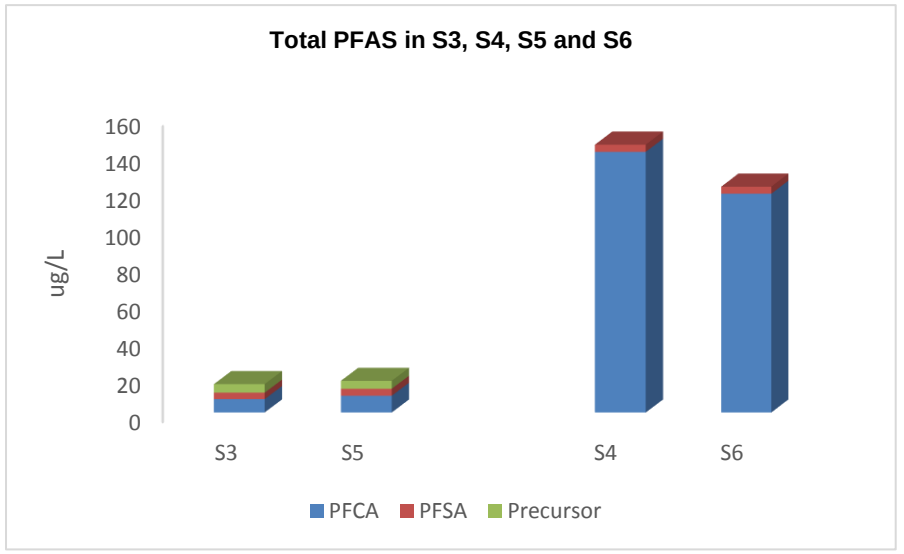


Figure 115 Laboratory 13 Results for Total PFAS in S3, S4, S5 and S6

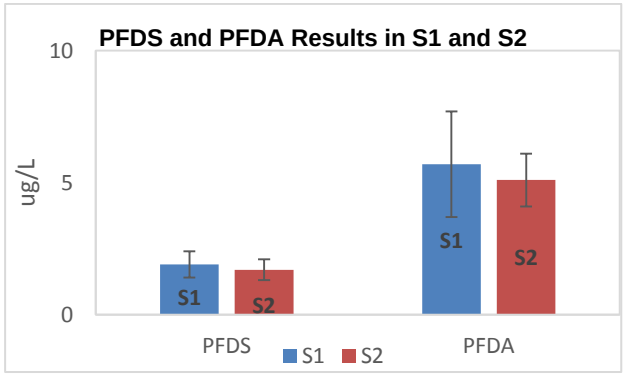


Figure 116 Laboratory 13 Results for PFDS and PFDA in S1 and S2

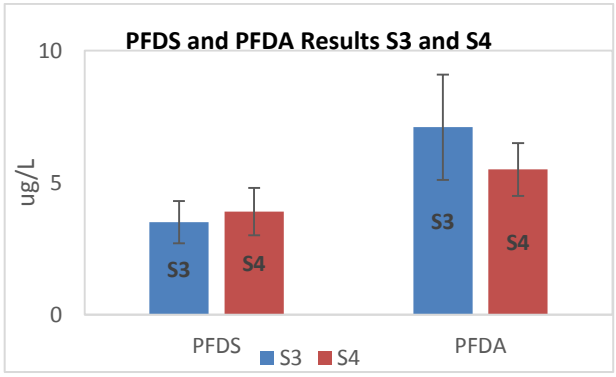


Figure 117 Laboratory 13 Results for PFDS and PFDA in S3 and S4

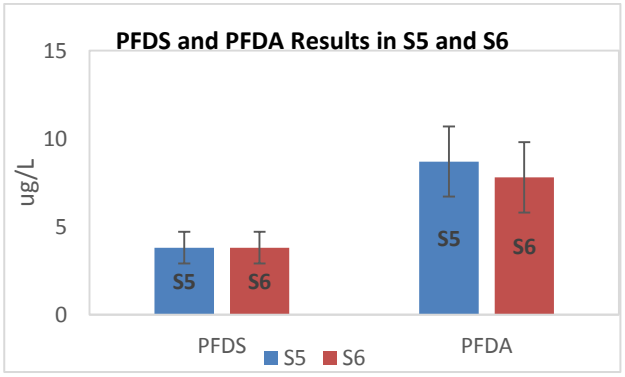


Figure 118 Laboratory 13 Results for PFDS and PFDA in S5 and S6

PFDS Change: -11%; **PFDA Change:** -11% ;

PFDS Change: 11%; **PFDA Change:** -23% ;

PFDS Change: 0%**; PFDA Change:** -10% ;

Laboratory 14[#]

Table 94

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	3.1
			PFPeA	4.1
			PFHxA	5.71
			PFHpA	10.2
			PFOA	8.27
			PFNA	2.96
PFDA 6.42 µg/L	PFDA	7.38	PFDA	5.56
PFDS 3.14 µg/L	PFDS	2.06	PFDS	1.4
	PFCA	7.41	PFCA	39.9
	PFSA	2.06	PFSA	1.4
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	9.47	PFAS	41.3

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-32.0%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND = Not Detected

Table 95

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	7.22	PFCA	153
	PFSA	4.02	PFSA	4.43
	PFAA precursor	3.95	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	7.13	PFDA	8.29
PFDS 6.51 µg/L	PFDS	4.02	PFDS	4.43
	PFAS	15.2	PFAS	157

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	10.2%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 96

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	0.02	PFCA	122
	PFSA	2.35	PFSA	2.5
	PFAA precursor	16.4	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	<0.02	PFDA	7.36
PFDS 6.51 µg/L	PFDS	2.35	PFDS	2.5
	PFAS	18.8	PFAS	125

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	6.4%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

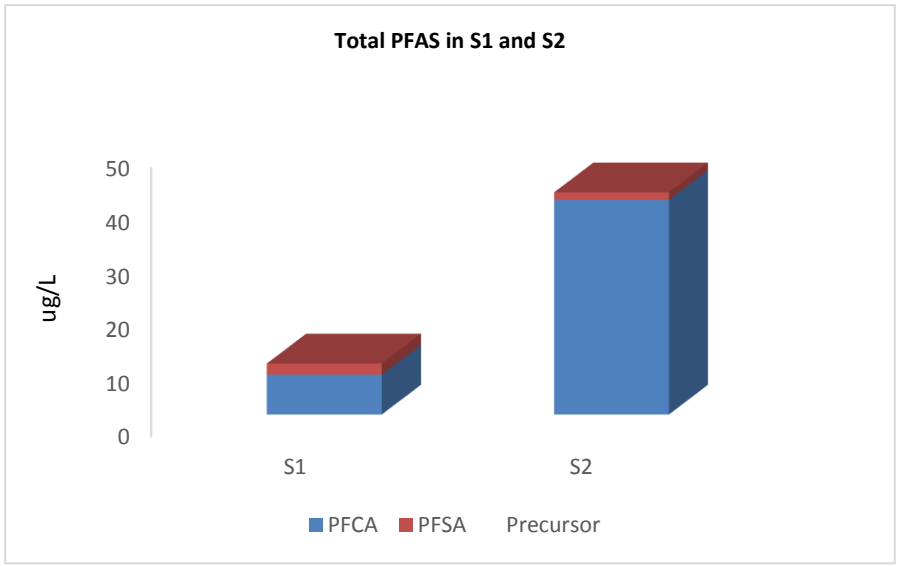


Figure 119 Laboratory 14 Results for Total PFAS in S1 and S2

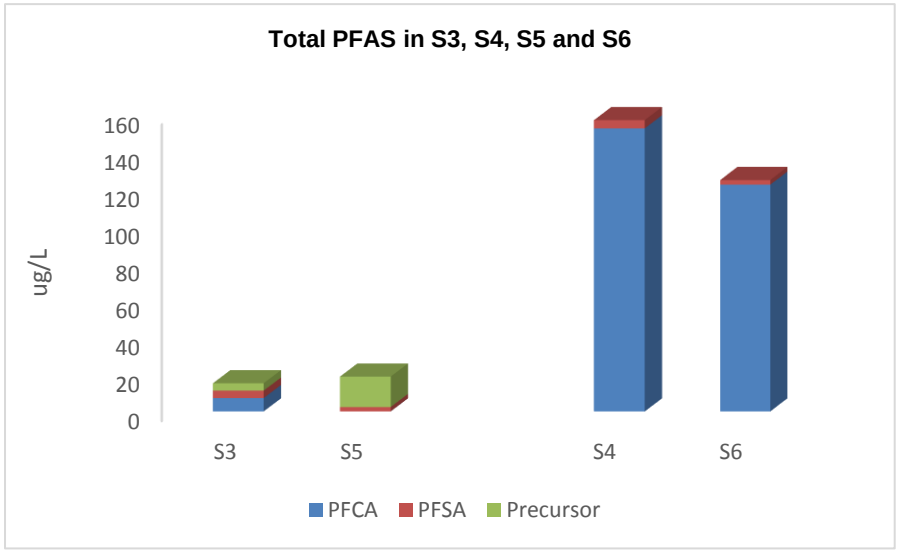


Figure 120 Laboratory 14 Results for Total PFAS in S3, S4, S5 and S6

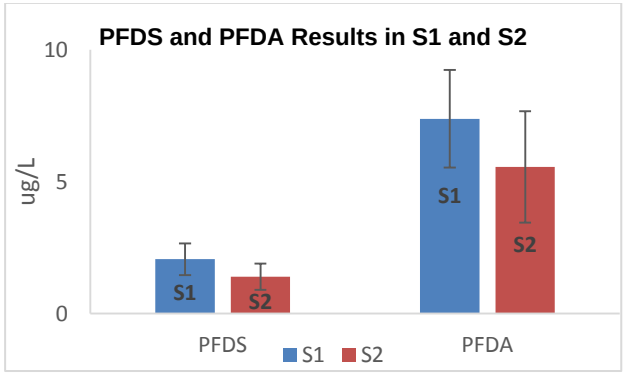


Figure 121 Laboratory 14 Results for PFDS and PFDA in S1 and S2

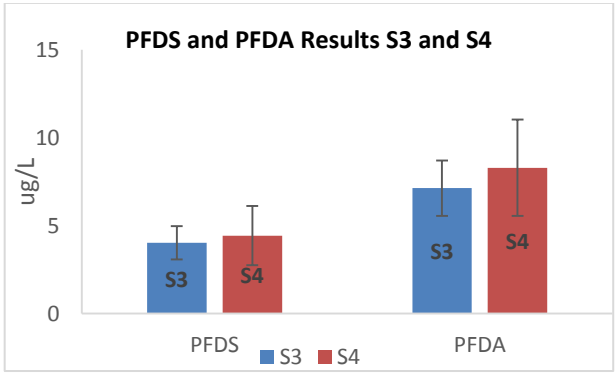


Figure 122 Laboratory 14 Results for PFDS and PFDA in S3 and S4

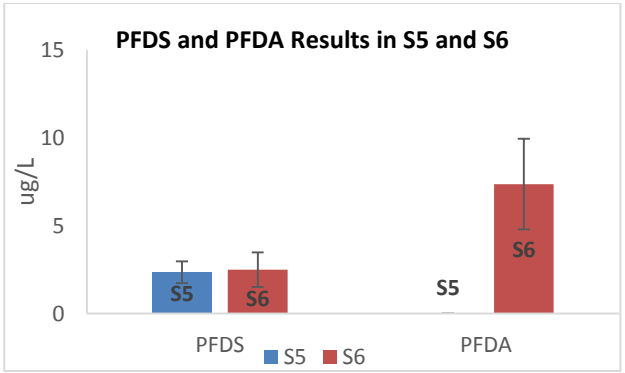


Figure 123 Laboratory 14 Results for PFDS and PFDA in S5 and S6

PFDS Change: -32%; **PFDA Change:** -25%

PFDS Change: 10%; **PFDA Change:** 16%

PFDS Change: 6.4%; **PFDA Change:** 100%

[#]The study coordinator considers the change in PFDA in S6 to be excessive, and such the oxidative method should be reviewed.

Laboratory 15

Table 97

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	1.80
			PFPeA	4.24
			PFHxA	5.46
			PFHpA	9.07
			PFOA	7.24
			PFNA	2.75
PFDA 6.42 µg/L	PFDA	6.45	PFDA	6.71
PFDS 3.14 µg/L	PFDS	2.54	PFDS	2.1
	PFCA	6.50	PFCA	37.3
	PFSA	2.54	PFSA	2.1
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	9.04	PFAS	39.4

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-17.3%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND = Not Detected

Table 98

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	6.22	PFCA	148
	PFSA	4.48	PFSA	3.94
	PFAA precursor	4.06	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	6.03	PFDA	5.42
PFDS 6.51 µg/L	PFDS	4.48	PFDS	3.94
	PFAS	14.8	PFAS	152

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-12.1%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 99

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	8.99	PFCA	148
	PFSA	4.72	PFSA	3.99
	PFAA precursor	4.32	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	8.69	PFDA	8.72
PFDS 6.51 µg/L	PFDS	4.71	PFDS	3.97
	PFAS	18.0	PFAS	152

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-15.5%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

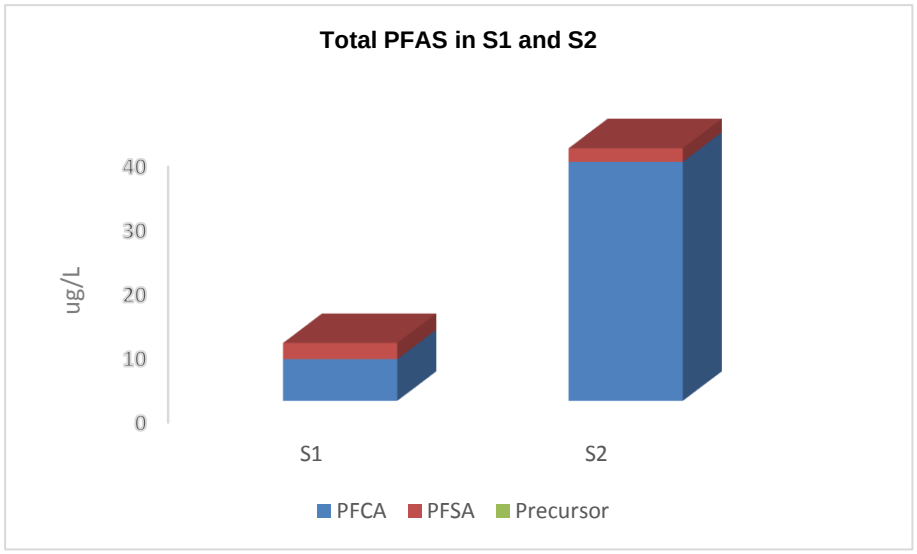


Figure 124 Laboratory 15 Results for Total PFAS in S1 and S2

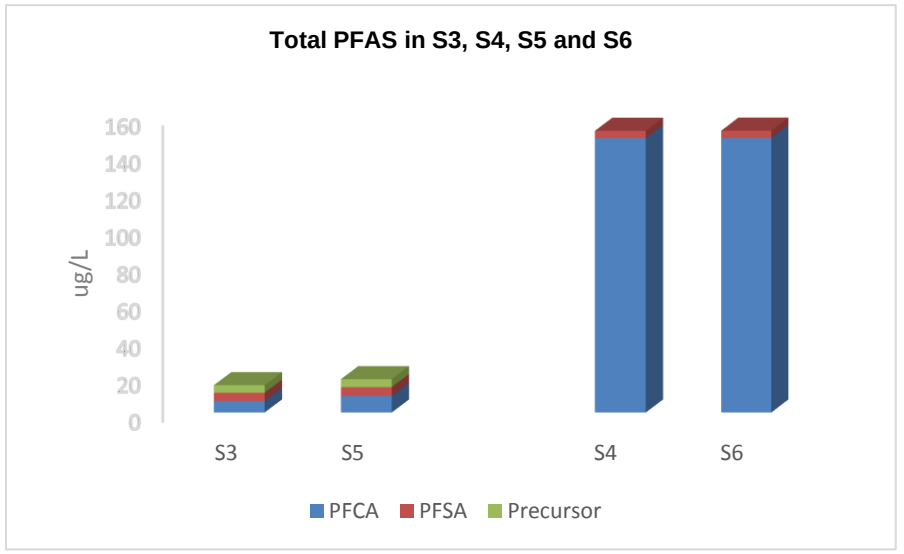


Figure 125 Laboratory 15 Results for Total PFAS in S3, S4, S5 and S6

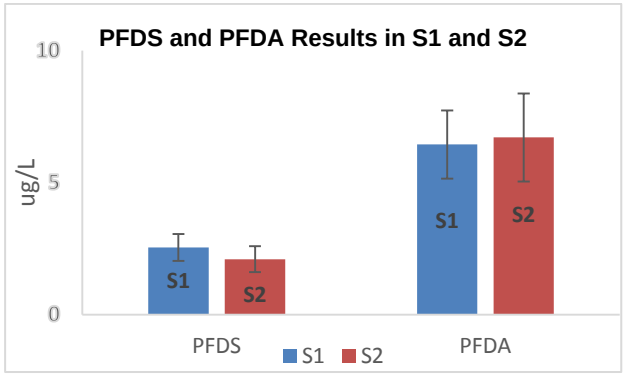


Figure 126 Laboratory 15 Results for PFDS and PFDA in S1 and S2

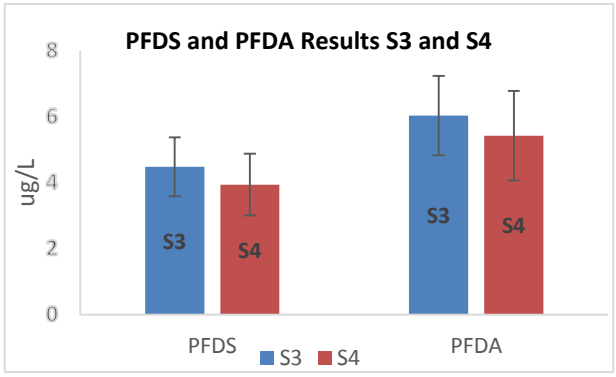


Figure 127 Laboratory 15 Results for PFDS and PFDA in S3 and S4

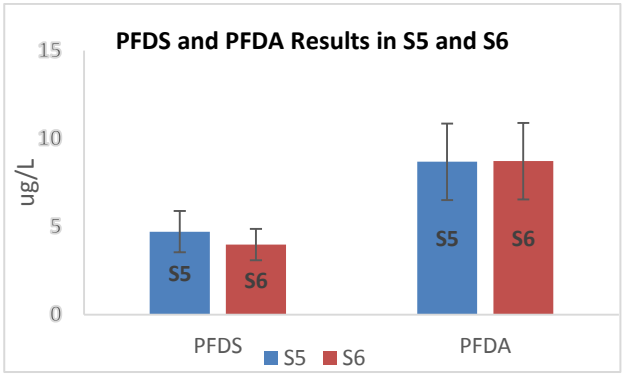


Figure 128 Laboratory 15 Results for PFDS and PFDA in S5 and S6

PFDS Change: -17%; **PFDA Change:** 4.1%

PFDS Change: -12%; **PFDA Change:** -10%

PFDS Change: -16%; **PFDA Change:** 0.4%

Laboratory 16

Table 100

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	1.6
			PFPeA	3.31
			PFHxA	4.77
			PFHpA	6.84
			PFOA	7.73
			PFNA	2.98
PFDA 6.42 µg/L	PFDA	6.03	PFDA	5.59
PFDS 3.14 µg/L	PFDS	2.66	PFDS	2
	PFCA	6.06	PFCA	32.8
	PFSA	2.66	PFSA	2
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	8.72	PFAS	34.8

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-24.8%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND = Not Detected

Table 101

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	7.81	PFCA	100
	PFSA	5.54	PFSA	3.94
	PFAA precursor	3.81	PFAA precursor	0.08
PFDA 9.08 µg/L	PFDA	7.72	PFDA	5.23
PFDS 6.51 µg/L	PFDS	5.54	PFDS	3.94
	PFAS	17.2	PFAS	104

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-28.9%
No PFAA precursor in post oxidation	Fail
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 102

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	10.3	PFCA	75.7
	PFSA	4.53	PFSA	4.5
	PFAA precursor	3.83	PFAA precursor	0.22
PFDA 9.08 µg/L	PFDA	10	PFDA	8.3
PFDS 6.51 µg/L	PFDS	4.53	PFDS	4.27
	PFAS	18.6	PFAS	80.4

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-0.7%
No PFAA precursor in post oxidation	Fail
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

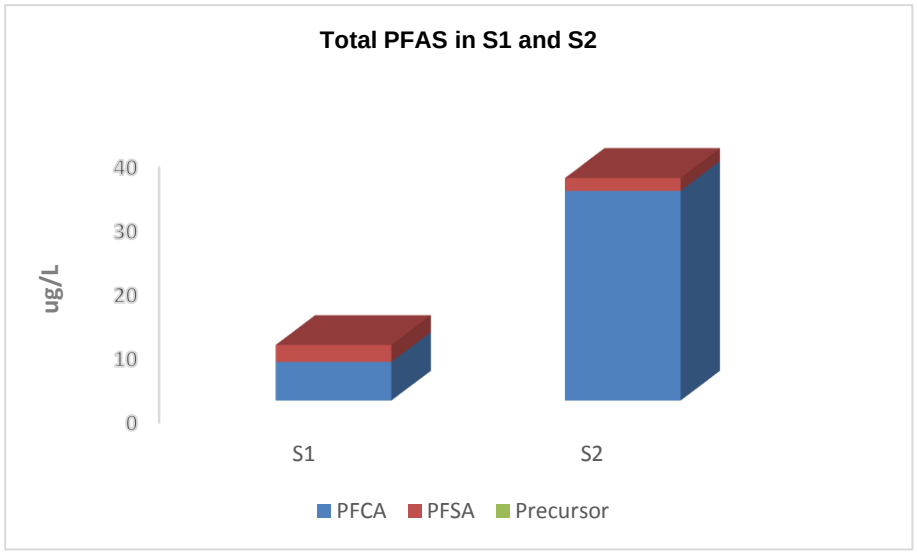


Figure 129 Laboratory 16 Results for Total PFAS in S1 and S2

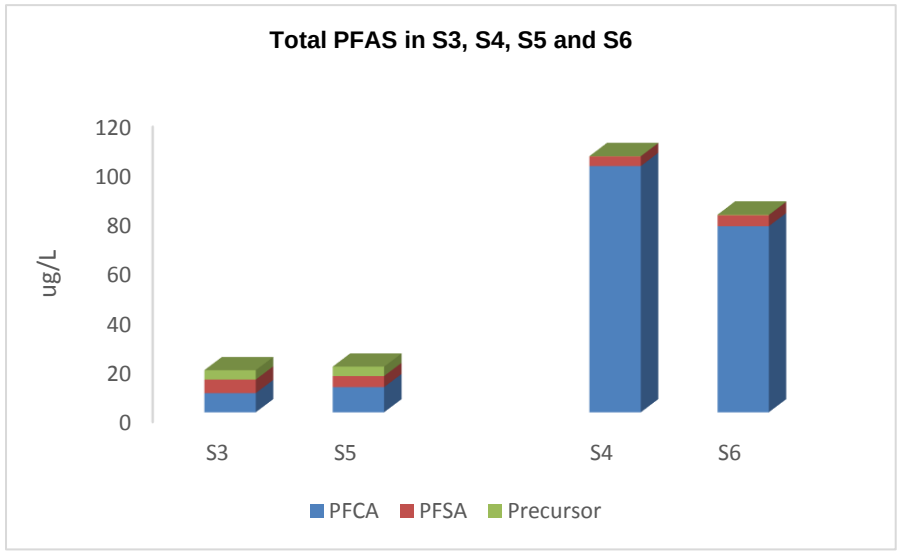


Figure 130 Laboratory 16 Results for Total PFAS in S3, S4, S5 and S6

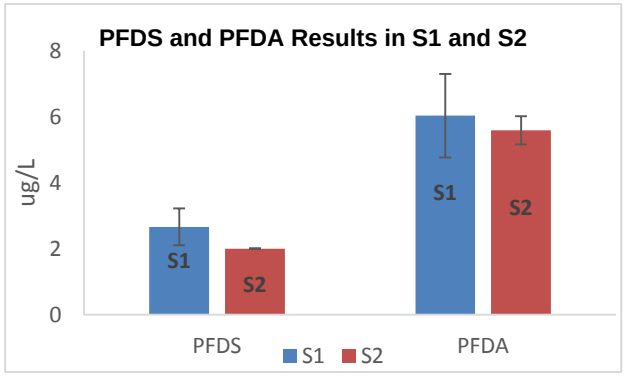


Figure 131 Laboratory 16 Results for PFDS and PFDA in S1 and S2

PFDS Change: -25%; **PFDA Change:** -7.3%

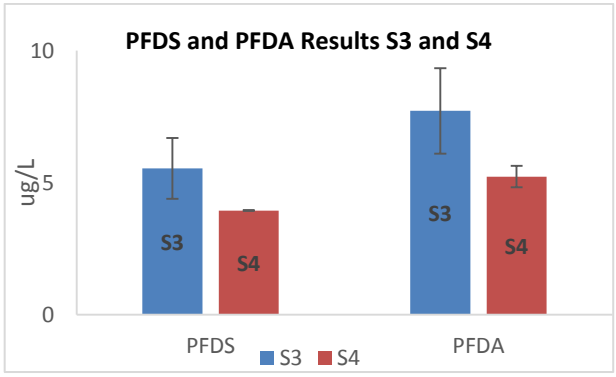


Figure 132 Laboratory 16 Results for PFDS and PFDA in S3 and S4

PFDS Change: -29%; **PFDA Change:** -32%

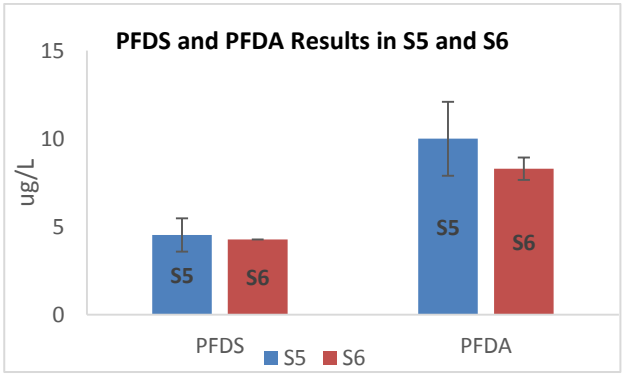


Figure 133 Laboratory 16 Results for PFDS and PFDA in S5 and S6

PFDS Change: -5.7%; **PFDA Change:** -17%

Laboratory 18

Table 103

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	1.80
			PFPeA	3.60
			PFHxA	5.45
			PFHpA	5.82
			PFOA	9.06
			PFNA	3.55
PFDA 6.42 µg/L	PFDA	7.94	PFDA	7.30
PFDS 3.14 µg/L	PFDS	3.05	PFDS	2.25
	PFCA	7.99	PFCA	36.6
	PFSA	3.05	PFSA	2.25
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	11.0	PFAS	38.8

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-26.4%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND = Not Detected

Table 104

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	9.77	PFCA	132
	PFSA	4.46	PFSA	5.83
	PFAA precursor	3.11	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	9.64	PFDA	8.25
PFDS 6.51 µg/L	PFDS	4.46	PFDS	5.83
	PFAS	17.3	PFAS	138

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	30.7%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 105

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	10.4	PFCA	82.1
	PFSA	3.96	PFSA	3.04
	PFAA precursor	3.57	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	10.2	PFDA	8.16
PFDS 6.51 µg/L	PFDS	3.96	PFDS	3.04
	PFAS	17.9	PFAS	85.1

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-23.2%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

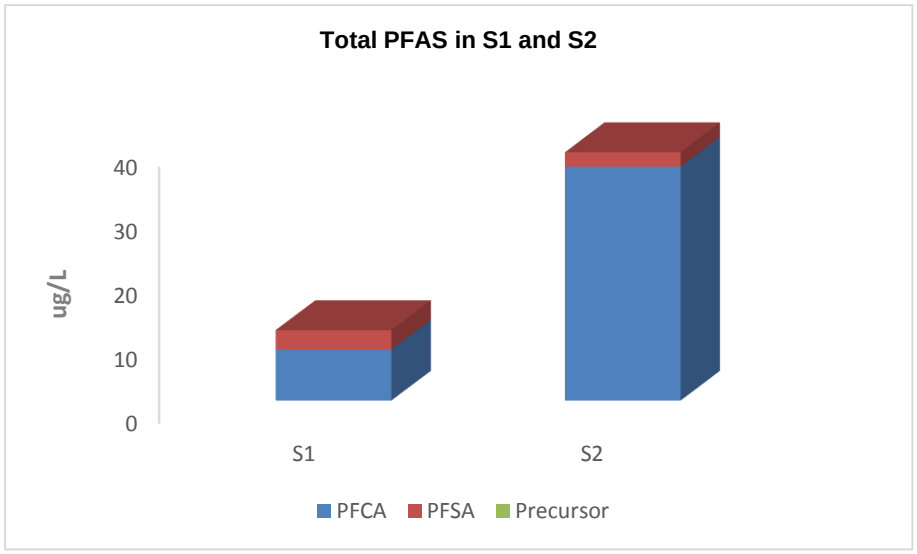


Figure 134 Laboratory 18 Results for Total PFAS in S1 and S2

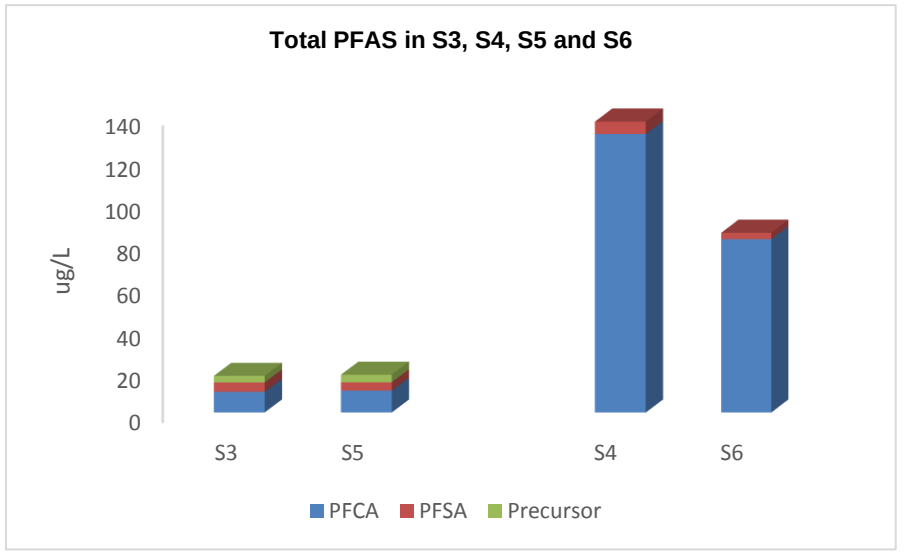


Figure 135 Laboratory 18 Results for Total PFAS in S3, S4, S5 and S6

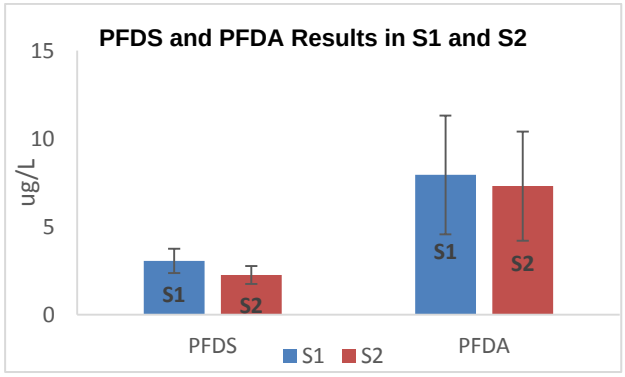


Figure 136 Laboratory 18 Results for PFDS and PFDA in S1 and S2

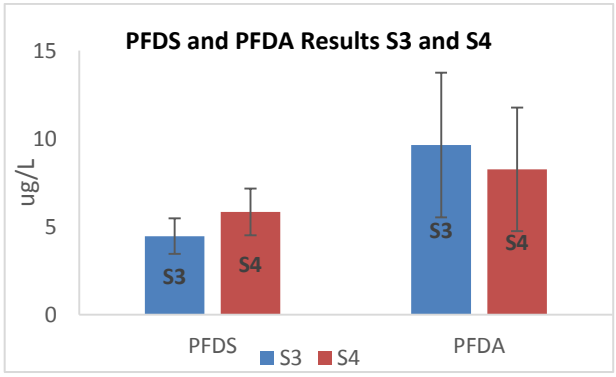


Figure 137 Laboratory 18 Results for PFDS and PFDA in S3 and S4

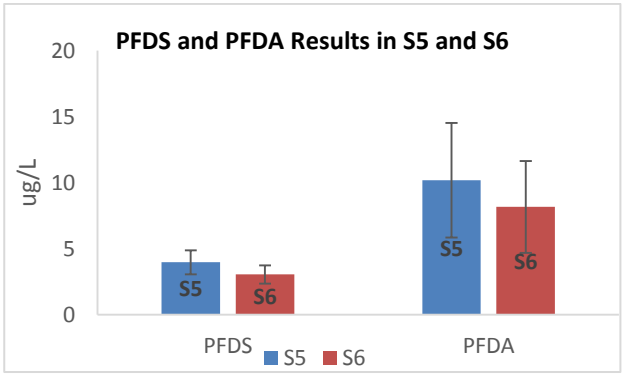


Figure 138 Laboratory 18 Results for PFDS and PFDA in S5 and S6

PFDS Change: -26%; **PFDA Change:** -8.1%

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PFDS Change: 31%;**PFDA Change:** -14% **;**

PFDS Change: -23%; **PFDA Change:** -20%

Laboratory 19

Table 106

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	1.06
			PFPeA	2.69
			PFHxA	4.70
			PFHpA	5.91
			PFOA	4.90
			PFNA	2.60
PFDA 6.42 µg/L	PFDA	6.53	PFDA	5.21
PFDS 3.14 µg/L	PFDS	1.57	PFDS	1.65
	PFCA	6.60	PFCA	27.1
	PFSA	1.57	PFSA	1.65
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	8.16	PFAS	28.7

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	5.4%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND = Not Detected

Table 107

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	6.09	PFCA	208
	PFSA	3.61	PFSA	3.99
	PFAA precursor	3.43	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	5.93	PFDA	6.77
PFDS 6.51 µg/L	PFDS	3.61	PFDS	3.99
	PFAS	13.1	PFAS	212

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	10.8%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 108

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	10.8	PFCA	259
	PFSA	4.09	PFSA	4.57
	PFAA precursor	4.04	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	10.4	PFDA	14.0
PFDS 6.51 µg/L	PFDS	4.09	PFDS	4.57
	PFAS	18.9	PFAS	264

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	11.9%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

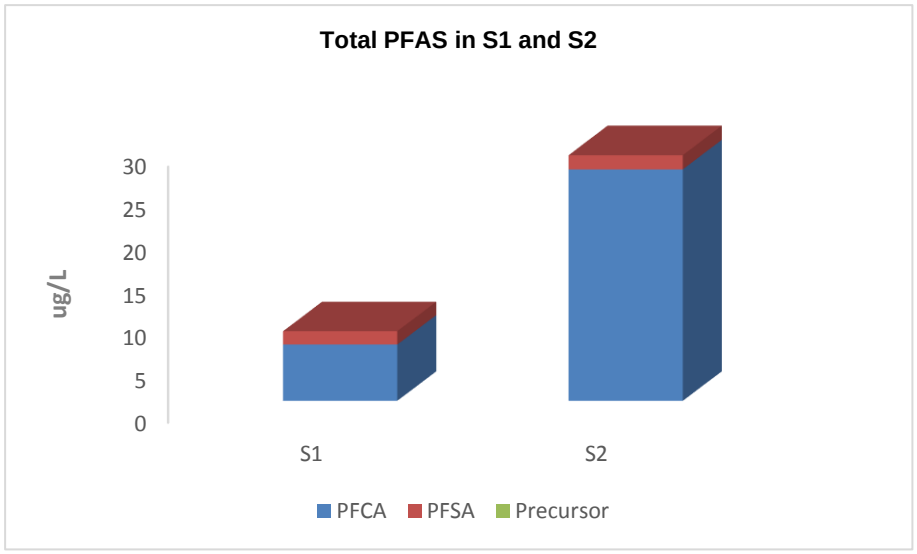

Figure 139 Laboratory 19 Results for Total PFAS in S1 and S2

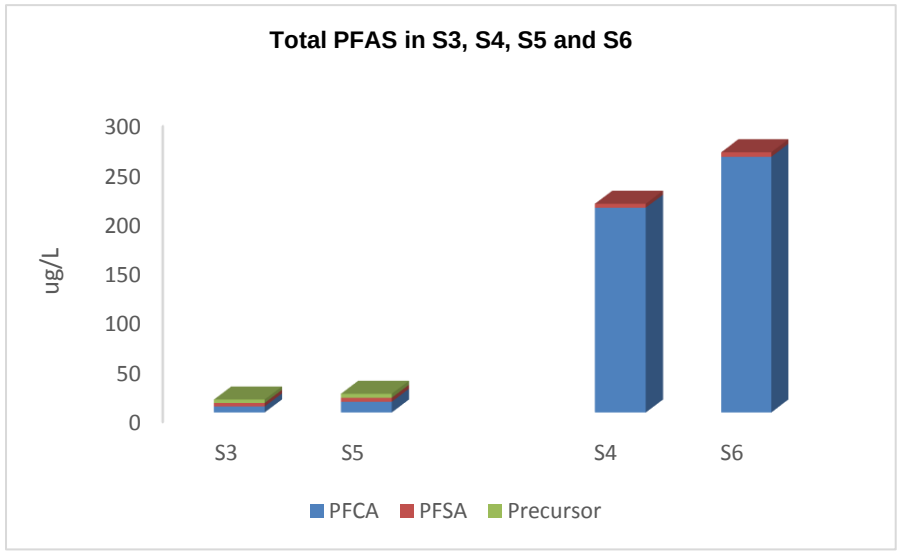

Figure 140 Laboratory 19 Results for Total PFAS in S3, S4, S5 and S6

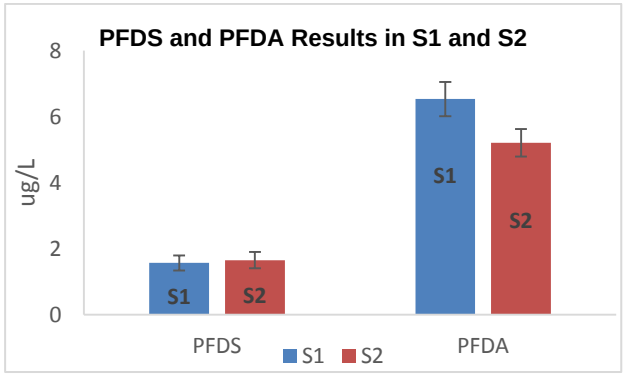

Figure 141 Laboratory 19 Results for PFDS and PFDA in S1 and S2

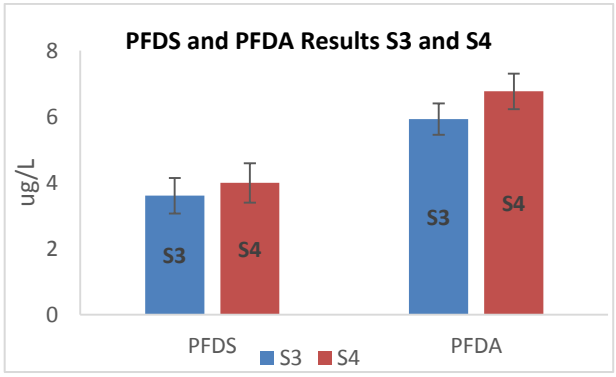

Figure 142 Laboratory 19 Results for PFDS and PFDA in S3 and S4

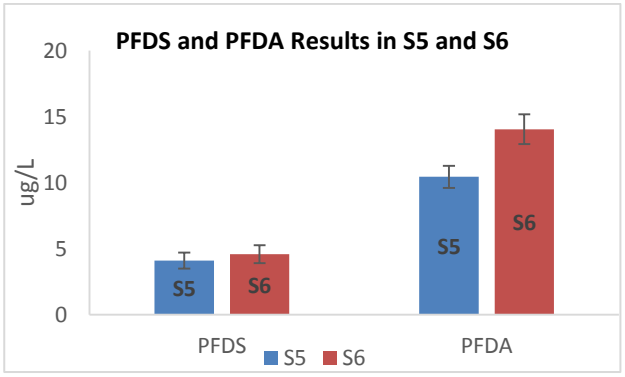

Figure 143 Laboratory 19 Results for PFDS and PFDA in S5 and S6

PFDS Change: 5.4%; **PFDA Change:** -20%

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PFDS Change: 11%; **PFDA Change:** 14%

PFDS Change: 12%; **PFDA Change:** 34%

Laboratory 20

Table 109

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	1.9
			PFPeA	3.8
			PFHxA	4.8
			PFHpA	9.2
			PFOA	6.2
			PFNA	2.8
PFDA 6.42 µg/L	PFDA	7.4	PFDA	6.7
PFDS 3.14 µg/L	PFDS	1.2	PFDS	1.4
	PFCA	7.43	PFCA	35.4
	PFSA	1.2	PFSA	1.4
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	8.63	PFAS	36.8

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	16.7%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND = Not Detected

Table 110

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	9	PFCA	151
	PFSA	1.7	PFSA	1.7
	PFAA precursor	2.91	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	8.9	PFDA	9.4
PFDS 6.51 µg/L	PFDS	1.7	PFDS	1.7
	PFAS	13.6	PFAS	153

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	0.0%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 111

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	10.6	PFCA	92.8
	PFSA	2.91	PFSA	2.79
	PFAA precursor	4.6	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	9.8	PFDA	11
PFDS 6.51 µg/L	PFDS	2.8	PFDS	2.7
	PFAS	18.1	PFAS	95.6

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-4.1%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

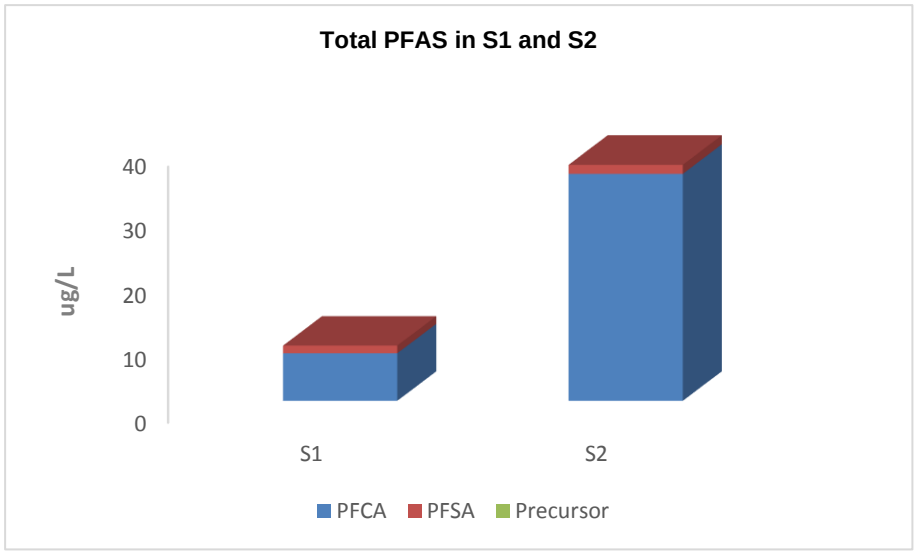


Figure 144 Laboratory 20 Results for Total PFAS in S1 and S2

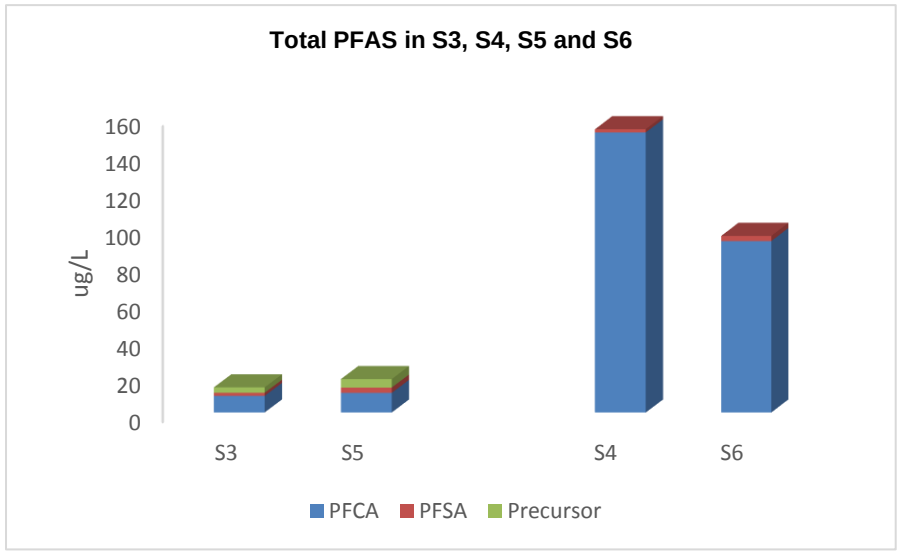


Figure 145 Laboratory 20 Results for Total PFAS in S3, S4, S5 and S6

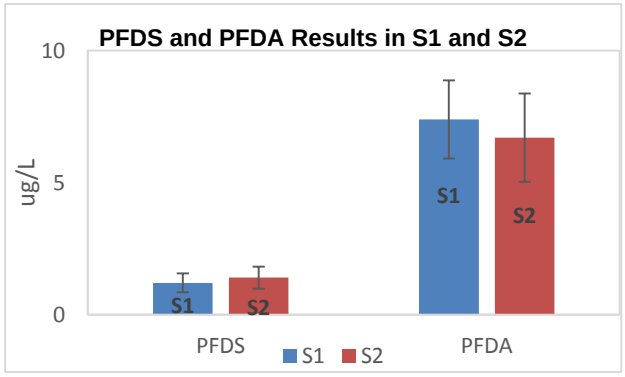


Figure 146 Laboratory 20 Results for PFDS and PFDA in S1 and S2

PFDS Change: 17%; **PFDA Change:** -9.5%

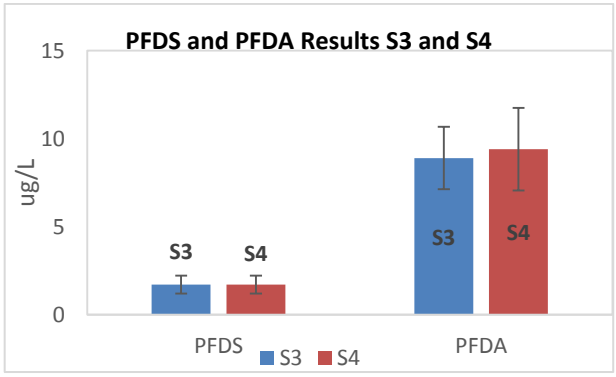


Figure 147 Laboratory 20 Results for PFDS and PFDA in S3 and S4

PFDS Change: 0%; **PFDA Change:** 5.6%

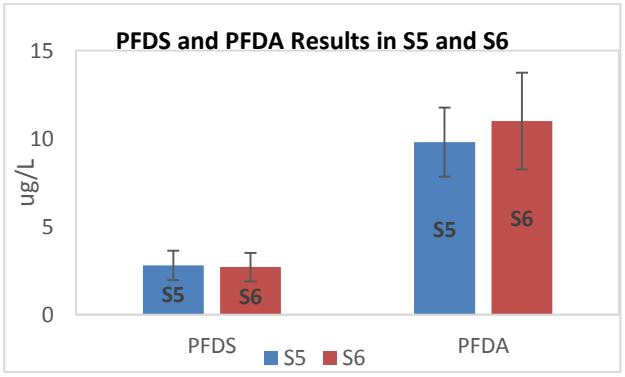


Figure 148 Laboratory 20 Results for PFDS and PFDA in S5 and S6

PFDS Change: -3.6%; **PFDA Change:** 12%

Laboratory 21

Table 112

Spike	S1		S2	
	Analyte	Result µg/L	Analyte	Result µg/L
6:2diPAP 50 µg/L 8:2diPAP 50 µg/L	Nil	Nil	PFBA	2.17
			PFPeA	3.28
			PFHxA	3.56
			PFHpA	5.65
			PFOA	2.84
			PFNA	1.2
PFDA 6.42 µg/L	PFDA	6.67	PFDA	5.66
PFDS 3.14 µg/L	PFDS	2.32	PFDS	2.14
	PFCA	6.67	PFCA	24.4
	PFSA	2.32	PFSA	2.14
	*PFAA precursor	ND	*PFAA precursor	ND
	PFAS	8.99	PFAS	26.5

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-7.8%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

*ND = Not Detected

Table 113

Spike	S3		S4	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	6.91	PFCA	90.3
	PFSA	4.61	PFSA	5.17
	PFAA precursor	3.92	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	6.91	PFDA	7.27
PFDS 6.51 µg/L	PFDS	4.61	PFDS	5.17
	PFAS	15.4	PFAS	95.5

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	12.1%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

Table 114

Spike	S5		S6	
	Analyte	Result µg/L	Analyte	Result µg/L
Alcoseal 30000 dilution	PFCA	9.29	PFCA	75.9
	PFSA	4.95	PFSA	3.87
	PFAA precursor	3.67	*PFAA precursor	ND
PFDA 9.08 µg/L	PFDA	9.29	PFDA	8.05
PFDS 6.51 µg/L	PFDS	4.95	PFDS	3.87
	PFAS	17.9	PFAS	79.8

Test for Acceptability of the Oxidation Method

Test	Result
TOTAL PFAS post-TOP ≥TOTAL PFAS pre TOP x 90%	Pass
SUM PFCA post-TOP ≥ SUM PFCA pre-TOP	Pass
SUM PFSA post-TOP = SUM PFSA pre-TOP	-21.8%
No PFAA precursor in post oxidation	Pass
SUM PFAA precursors post-TOP / SUM Total PFAS post-TOP <5%	Pass

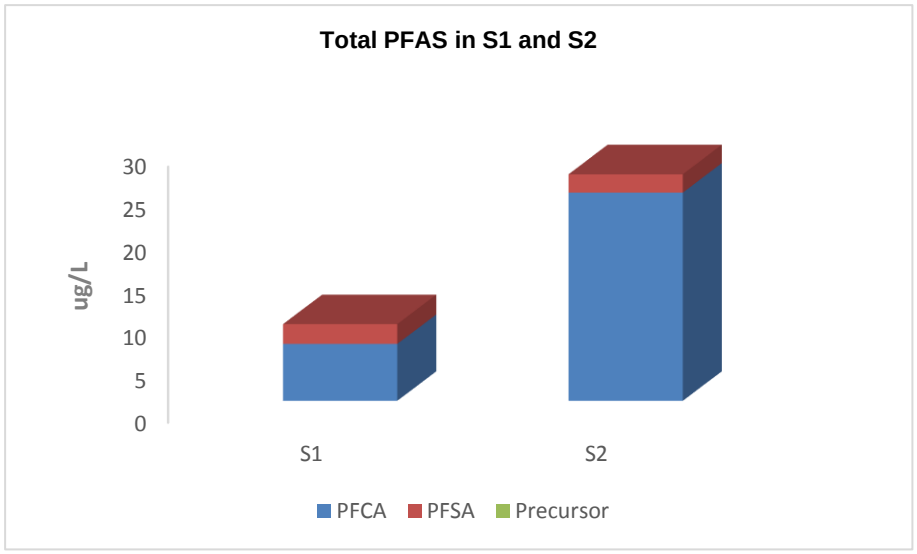


Figure 149 Laboratory 21 Results for Total PFAS in S1 and S2

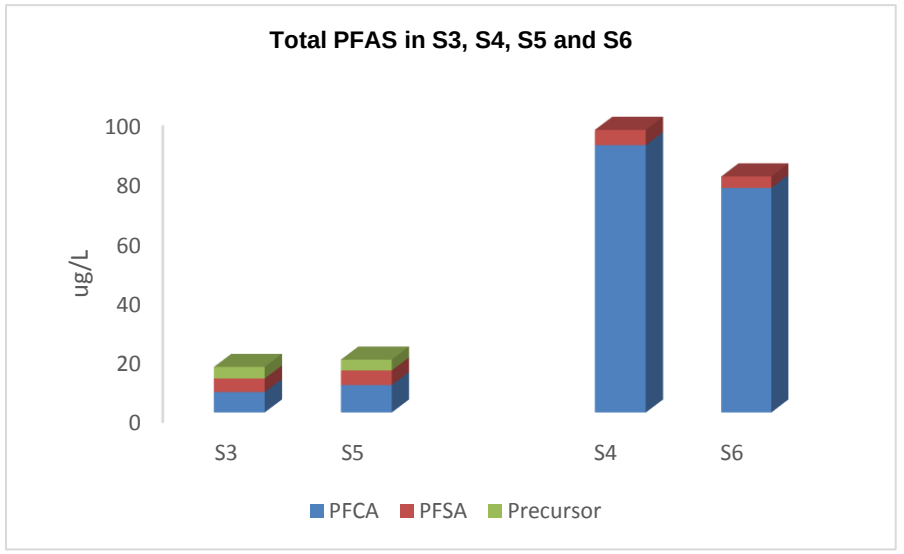


Figure 150 Laboratory 21 Results for Total PFAS in S3, S4, S5 and S6

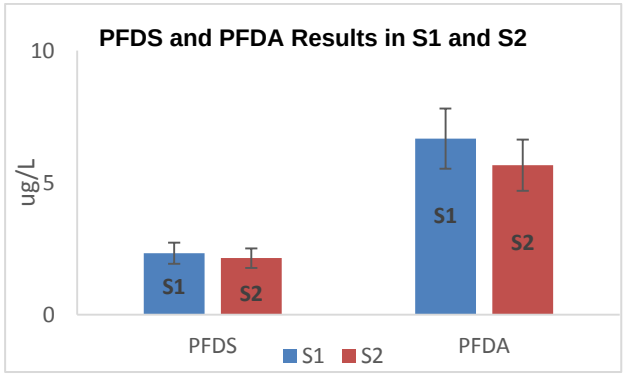


Figure 151 Laboratory 21 Results for PFDS and PFDA in S1 and S2

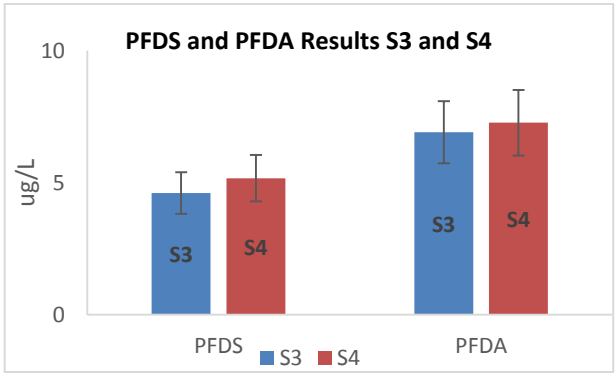


Figure 152 Laboratory 21 Results for PFDS and PFDA in S3 and S4

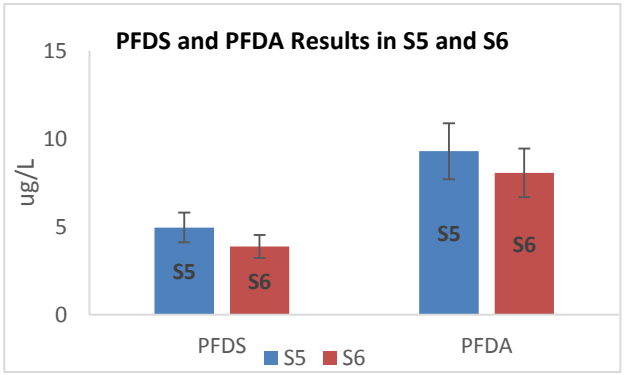


Figure 153 Laboratory 21 Results for PFDS and PFDA in S5 and S6

PFDS Change: -7.8%; **PFDA Change:** -15% **;**

PFDS Change: 12%; **PFDA Change:** 5.2% **;**

PFDS Change: -22%; **PFDA Change:** -13% **;**

7.3 Participants' Oxidative Methods

A summary of participants' test methods for oxidative treatment is presented in Table 2 and a summary of PFAS analysis in Table 3. All laboratories used modified versions of the method developed by Houtz and Sedlak (H&S) in 2012 .[1]

In this study "H&S oxidant dosage" refers to a dosage of 0.016 g/mL which is the total amount of $K_2S_2O_8$ used by H&S (2 g) per mL of sample taken for analysis (125 mL).

Of 18 laboratories, 10 used the same sample amount, dilution factor, number of cycles and oxidant dosage across all three samples. All 18 laboratories used the same oxidation time and temperature for all three samples: S2, S4 and S6.

Below are some of the most notable departures from H&S method:

- Laboratory 21 used 500 times the H&S oxidant dosage (8 g/mL) and reported using 5 mL of sample, a dilution factor of 20 and 2 g of $K_2 S_2O_8$;
- Laboratory 10 used 2 g of $K_2S_2O_8$ but diluted Sample S2 10 times and Samples S4 and S6 100 times;
- Laboratory 13 used 4 g of $K_2S_2O_8$ and 4 mL of 4N NaOH solution.
- Laboratory 20 used 0.81 g of $K_2S_2O_8$ and 0.32 g of NaOH.

Comparison of Participants' Methods

Figures 154, 155 and 156 presents a summary of the oxidative methods used by participants for the treatment of Samples S2, S4 and S6 respectively. In cases when multiple dilution factors were provided, the largest was used to calculate the amount of sample taken for analysis (sample size).

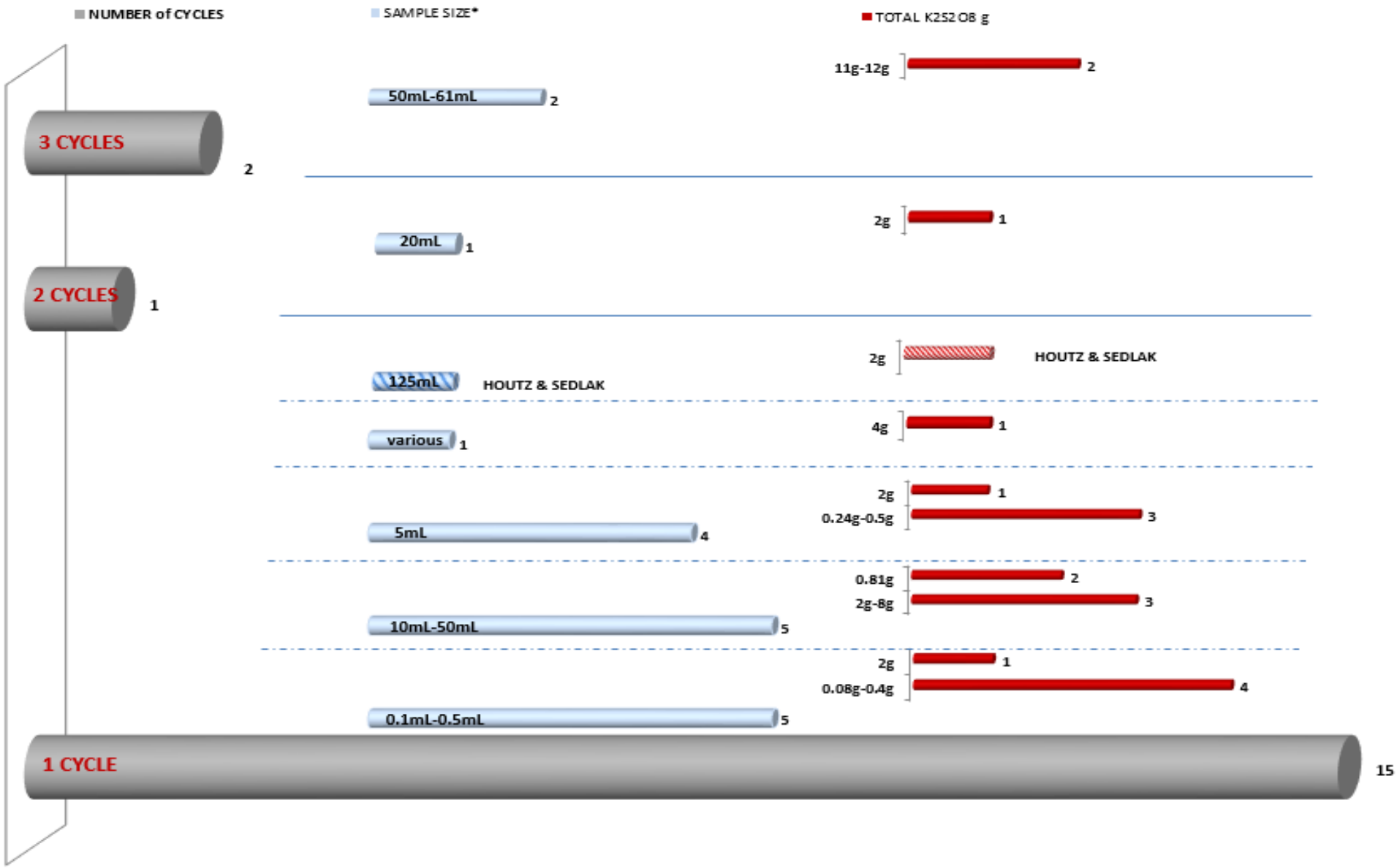
Of 18 participants, 15 used one oxidation cycle, one used two oxidation cycles, and two used three oxidation cycles. The sample size taken for analysis (after application of the dilution factor) varied from 0.05 mL to 62 mL, while the total amount of oxidant used varied from 0.08 g to 15 g.

The most popular oxidative treatment consisted of one oxidation cycle, a sample size of 5 mL and 0.24 g to 0.5 g of $K_2S_2O_8$.

The total amount of oxidant used per mL of sample was calculated for each participant and further normalised to the dosage used by H&S per mL of sample in order to able to assess participants' results in the context of the methods used.
Participants used between 2 and 500 times the dosage of oxidant used by H&S per mL of sample. Most participants also used a molar ratio of $K_2S_2O_8$ (60mM) to NaOH (150mM) of approximately 0.8.

All participants used an oxidation temperature close to 85°C as used by H&S, and an oxidation time of 6 hours or longer.

With the exception of one laboratory, all reported a pH >7 after oxidation.

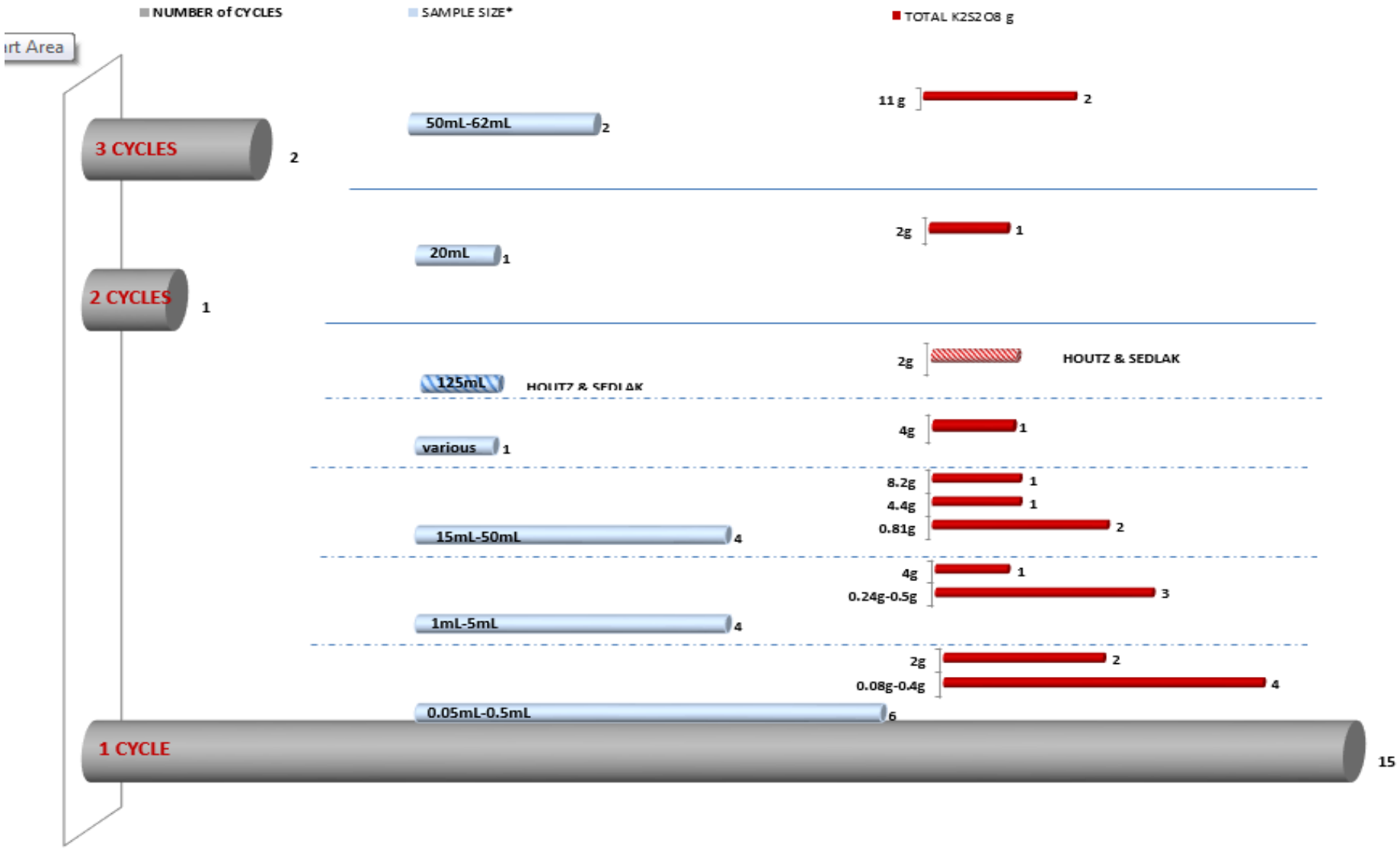


*Sample size, dilution factor applied. In cases when multiple dilution factors were provided, the largest was used to calculate the sample size.
In this chart, the figure at the end of each bar shows the number of laboratories that are using the number of cycles, sample size and total amount of $K_2S_2O_8$

Figure 154 Summary of Participants' Oxidative Methods Used for Sample S2

*Sample size, dilution factor applied. In cases when multiple dilution factors were provided, the largest was used to calculate the sample size.
In this chart, the figure at the end of each bar shows the number of laboratories that are using the number of cycles, sample size and total amount of $K_2S_2O_8$

Figure 155 Summary of Participants' Oxidative Methods Used for Sample S4



*Sample size, dilution factor applied. In cases when multiple dilution factors were provided, the largest was used to calculate the sample size.
In this chart, the figure at the end of each bar shows the number of laboratories that are using the number of cycles, sample size and total amount of $K_2S_2O_8$

Figure 156 Summary of Participants' Oxidative Methods Used for Sample S6

7.4 Discussion of Methods

7.4.1. Analyte Losses During Preparation

According to PFAS NEMP a total PFAS concentration post-TOP Assay greater than or equal to the total PFAS concentration pre-TOP Assay is an indication of no analyte loss during preparation. As the amount of precursors spiked in all study samples was high, it is unlikely for this criteria not to be fulfilled.

PFCA presented in samples or resulting from precursor oxidation are assumed not to undergo further oxidation or other transformations under oxidation condition. In this study, PFDA was spiked into the samples to monitor any possible losses of PFCAs. In S1/S2, there were no precursors added which would form PFDA on oxidation. In S3/S4 and S5/S6, while participants reported the formation of long chain PFCAs, the amount of PFDA formed on oxidation is expected to be small compared to the spiked amount. PFDA was expected to have a similar concentration pre-oxidation compared to post-oxidation in all 3 pairs of samples. In PFAS NEMP a decrease of less than 10% in PFAS concentration is considered acceptable, however there is no specific numerical criteria given for the assessment of carboxylic acid losses during oxidation treatment. As a result, in this study, a variation of 25% in PFDA concentration between the pair of samples was considered as an acceptable analytical variability by the study coordinator.

Bar charts of the differences in PFDA concentration across the three pairs, together with each laboratory's pH values pre- and post- oxidation, are presented in Figure 157. Many participant results indicate loss of PFDA during oxidation.

Laboratory 1 had losses in PFDA of 24%, 37% and 36% in samples S2, S4 and S6, respectively. They used three oxidation cycles and 12 times the amount of oxidant used by H&S per mL of sample. They also reported using a dilution factor of 10 for S4, but not for S2 and S6.

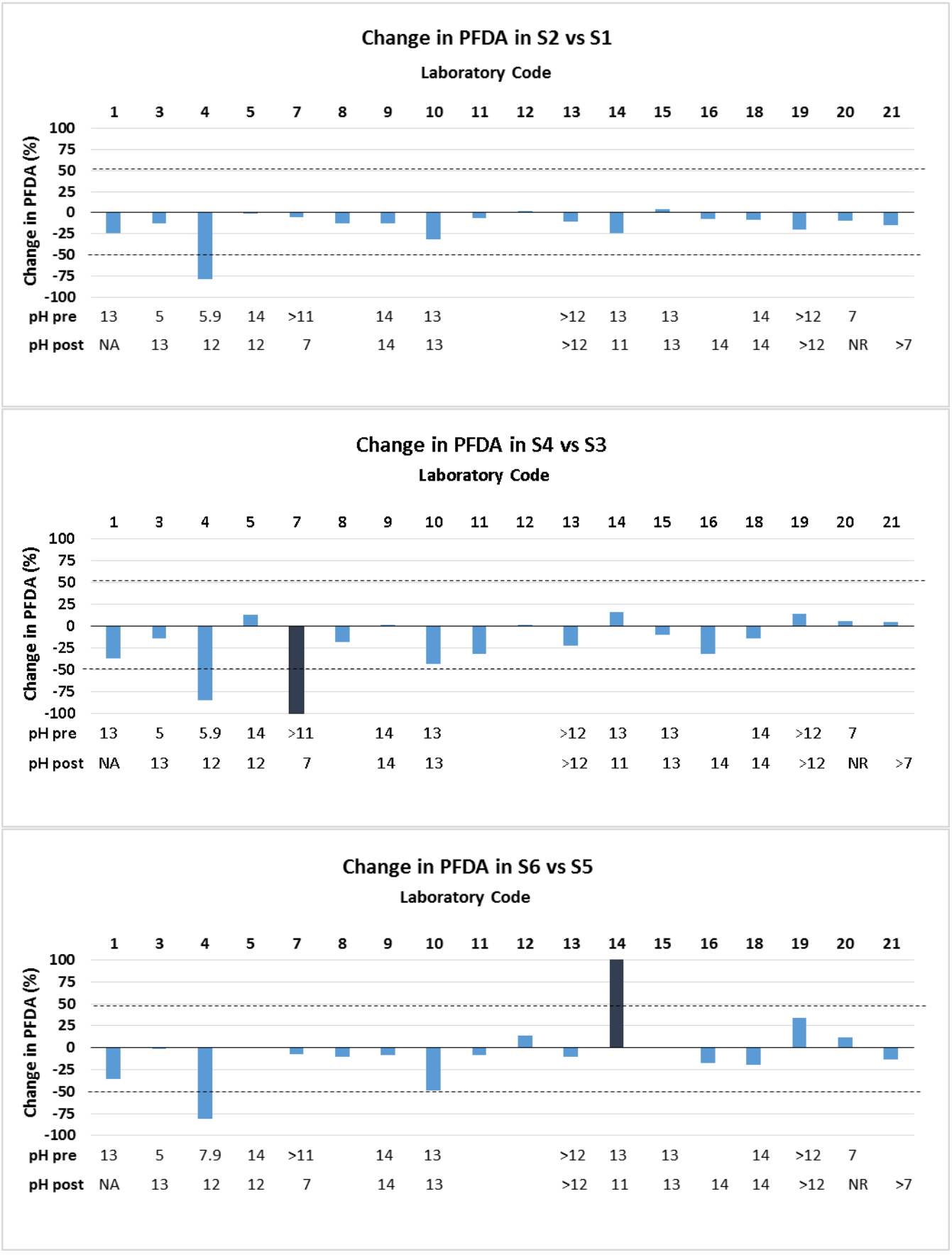
Laboratory 4 lost approximately the same amount of PFDA across the three-post oxidation samples, losing 79%, 85% and 81% in S2, S4 and S6 respectively. They also reported a similar loss of 88% in PFDS across the three samples. They used the same oxidative treatment for all three samples: one oxidation cycle, twice the amount of $K_2S_2O_8$ used by H&S per mL of sample, but 65.8 times the amount of NaOH used by H&S per mL of sample. They also reported a pH of 5.9 before oxidation for S2 and S4, and a pH of 7.9 before oxidation for S6, but reported an alkaline pH after oxidation (12) for S2, S4 and S6.

Although they used the same oxidative method for all three samples, Laboratory 7 did not detect any measurable amounts of PFDA and PFDS in S4, but did detect them in S2 and S6. The reported results for PFDA and PFDS in S4 were below their level of reporting.

Laboratory 10's losses of PFDA were 32%, 43% and 49% for S2, S4 and S6 respectively. For oxidation in S2, they used 25 times the amount of oxidant used by H&S per mL of sample, and 250 times that amount for S4 and S6.

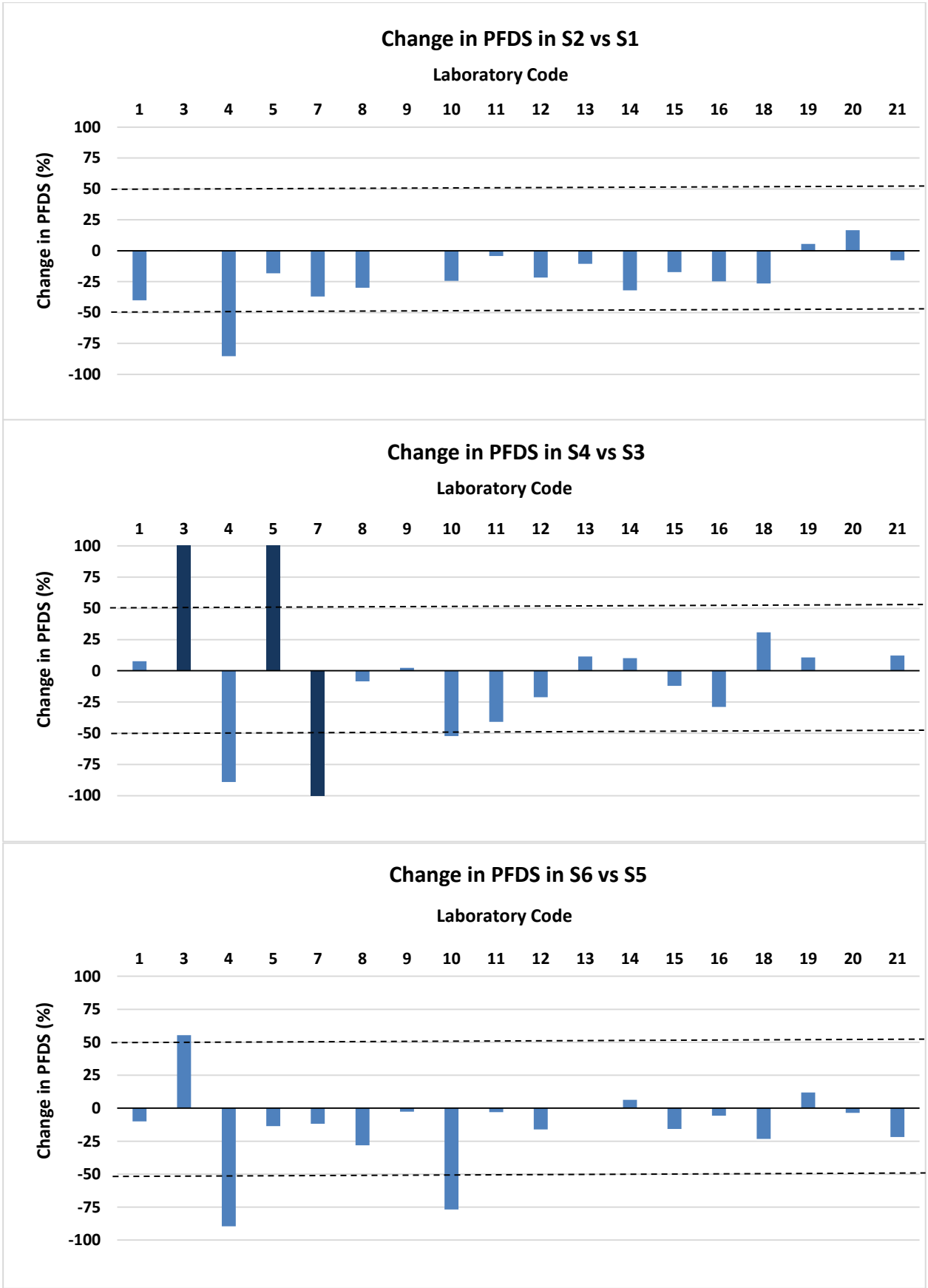
Laboratory 14 registered a 25% loss of PFDA in S2 only. They used the same oxidative regime for all three samples but diluted S2 up to 50 times, and S4 and S6 up to 100 times. They also reported in S5 a PFDA result below their level of reporting.

Laboratories' results for PFDA in the pre-oxidation samples were within 79% to 114% of the spiked value, and in the post-oxidation samples were within 68% to 97% of the spiked value (Table 58). The decrease in PFDA by approximately 14% was consistent among the three sample pairs. This is an indication of possible losses during the oxidation process.



The dark blue bars represent PFDA results which were reported below the laboratory level of reporting and were replaced with a value of zero (0) to calculate the change.

Figure 157 Change in PFDA Pre and Post Oxidation



The dark blue bars represent PFDS results which were reported below the laboratory level of reporting and were replaced with a value of zero (0) in order to calculate the change.

Figure 158 Change in PFDS Pre and Post Oxidation

Low recoveries were also registered for PFDS results in both pre- and post-oxidation types of samples. The results reported for PFDS in the three pairs of samples were consistently low: within 67% to 73% of the spiked value for pre-oxidation samples, and within 58% to 64% of the spike value for post-oxidation samples. The low PFDS recovery might be due to absorption of the analyte into the walls of the container, rather than the oxidation process. There is also a possibility that PFDS may have been lost during the preparation of the PT test samples prior to dispatch. Although there was a slight decrease in PFDS between pre- and post-oxidation samples, this decrease is neither substantial nor consistent (Figure 158).

Laboratories should consider reviewing their oxidation method when there is a *decrease* exceeding 50% (25% when a confidence interval of 95% is considered) between the pre-oxidation and post-oxidation PFDA concentration. It is important to ensure that the pH remains strongly alkaline during the oxidation process so as to avoid perfluorinated alkyl chain shortening. [2] According to H&S, the sulphate radical can react directly with PFCA, however its reaction with the hydroxide ion is much faster than its reaction with PFOA at pH values above 12.

7.4.2. Method Capability in Converting Precursors to PFCA

Samples S1/S2 were MilliQ water samples spiked with 6:2 diPAP and 8:2 diPAP. A mixture of C4 - C9 PFCAs were expected to be produced after the oxidation of the fluorotelomer containing precursors.

Participants' performance in measuring PFBA, PFPeA, PFHxA, PFHpA, PFOA and PFNA after oxidation was assessed in S2. The z-score for these analytes in S2 is not only a reflection of participants' ability to measure PFCA in water but also of the efficiency of their oxidation method. The between-laboratory coefficient of variation for these analytes (from 8.3% to 27%) was in good agreement with the set performance coefficient of variation of 25% .The results reported for Total PFCA in S2 were also in excellent agreement (CV 8.7%) indicating a comparable conversion of the spiked precursors to PFCAs by participating laboratories (Table 59).

A commercial supply of Alcoseal was used to spike the MilliQ water samples S3/S4 and river water samples S5/S6 respectively. No assessment was conducted for individual PFCAs in these samples because there is not enough information available on Alcoseal with regards to its chemical composition or expected oxidation products. The major PFAA precursor identified by laboratories in pre-oxidation samples was: 6:2 FTS. Traces of 8:2 FTS and of carboxylic acids (possible impurities from Alcoseal) were also identified by most participants (Appendix 1).

A reasonable consensus was found between the results reported by participants for Total PFCA in S4 and S6: the between-laboratory CV was 24% and 26% respectively.

With the exception of Laboratory 8, all participants reported results for Total PFCA in post-oxidation samples which were greater than or equal to the results reported for Total PFCA in the pre-oxidation sample (Figure 159).

7.4.3. Precursors Conversion to PFSA

According to the PFAS NEMP QA for equivalence of sulfonate concentrations, no precursor conversion to PFSA should take place for AFFF samples: "The sum of PFAS post-TOP Assay should approximate the sum of PFSA pre-TOP Assay".

Results reported by participants for PFSA pre and post oxidation are presented in Figure 160.

Figure 159 Total PFCA Pre and Post Oxidation

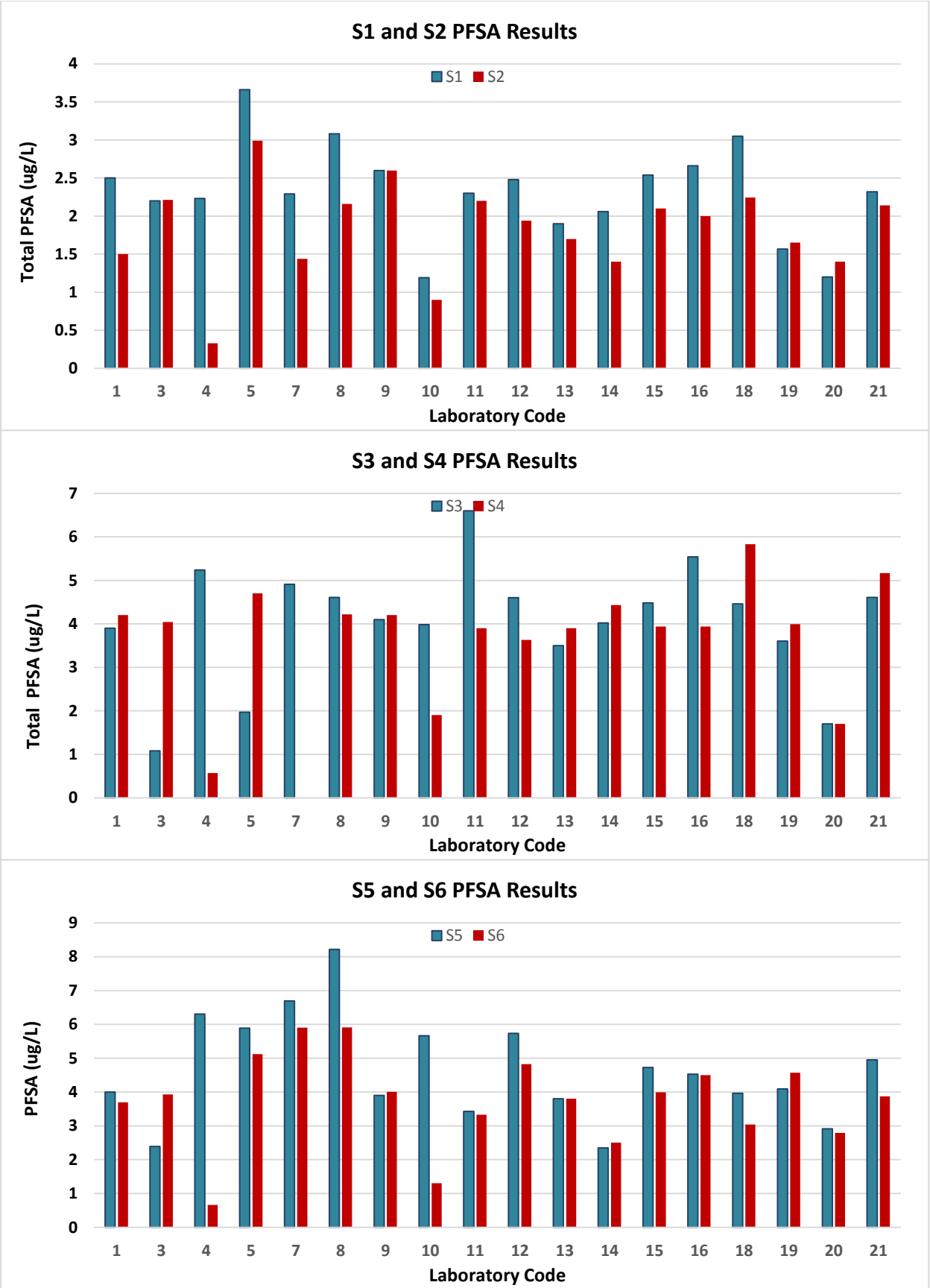
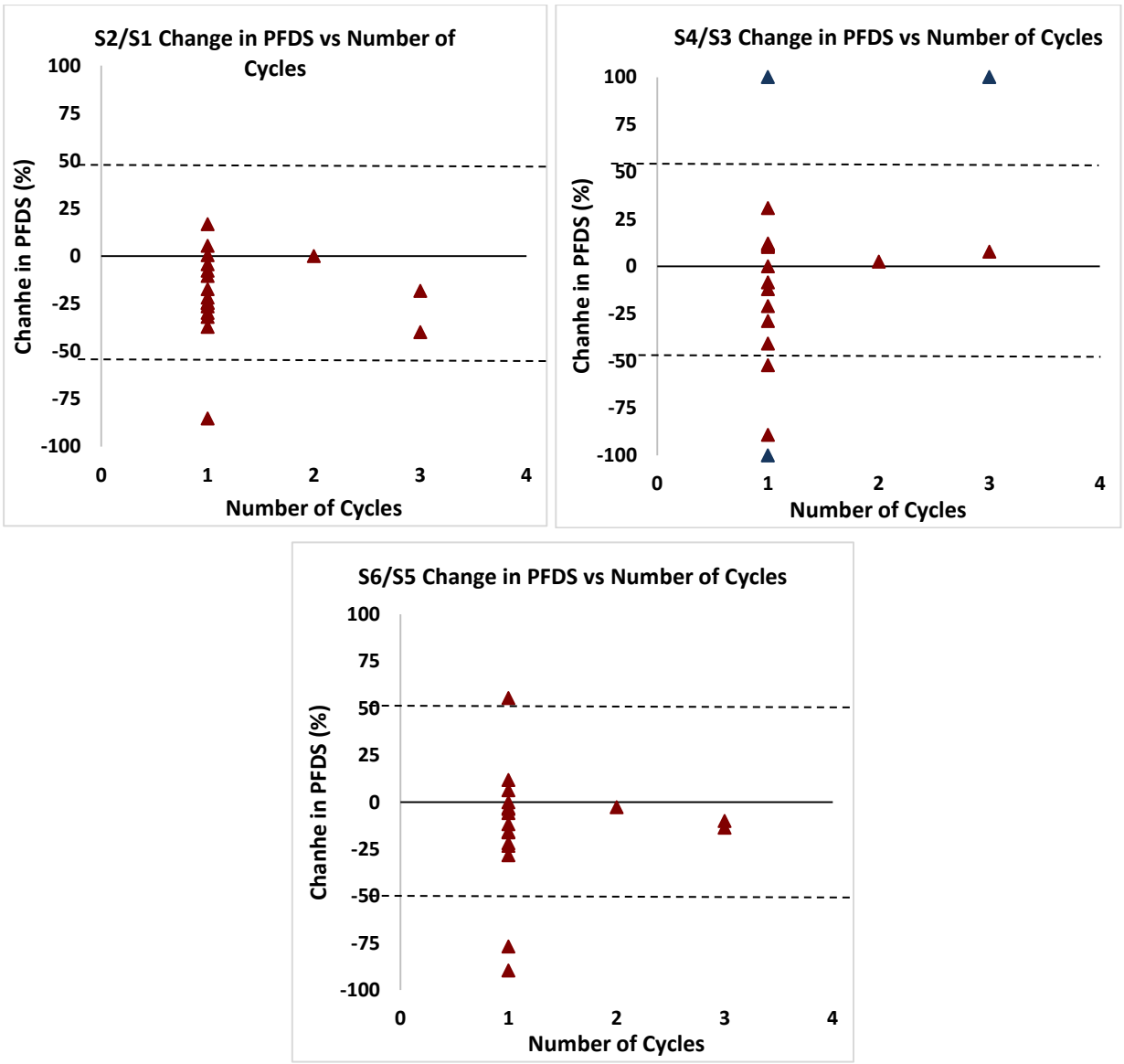


Figure 160 PFSA Results Pre and Post Oxidation



The dark blue plotted points represent PFDS results which were reported below the laboratory level of reporting and were replaced with a value of zero (0) in order to calculate the change.

Figure 161 Change in PFDS Pre and Post Oxidation vs Number of Oxidation Cycles

PFDS was added to the study samples in order to be able to monitor PFSA loss during the oxidation process. PFDS concentration was expected not to change during the oxidative treatment. As there is no specific numerical criteria given for the assessment of sulfonic acids recovery during oxidation treatment in PFAS NEMP a variation of 25% in PFDS concentration between the pair of samples was considered as an acceptable analytical variability by the study coordinator. Bar charts with changes in PFDS pre- and post-oxidation are presented in Figure 158.

Laboratory 3's increase in PFDS in S2 was insignificant at 0.5%, but was large in S4 and S6 samples spiked with Alcoseal at 270% and 55% respectively. They used only one oxidation cycle but their dosage was 34 times that used by H&S.

Laboratory 5 reported an increase of 140% in PFDS in S4, but not in S2 and S6. Their oxidative regime involved three oxidative cycles, 50 mL of sample and 5 g, 3 g and 3 g respectively of $K_2S_2O_8$ for each oxidative cycle. They also used 5 mL, 3 mL and 3 mL respectively of NaOH for each oxidative cycle.

Laboratory 19 reported an increase in PFDS in all three post-oxidation samples, ranging from 5.4% to 12%. They used the same oxidative treatment for all 3 samples: one oxidation cycle, 5.5 times the amount of $K_2S_2O_8$ used by H&S per mL of samples, and 6.6 times the amount of NaOH used by H&S per mL of sample.

Figure 161 presents scatter plots of PFDS differences pre- and post-oxidation versus number of cycles used. No trend was evident in changes in PFDS between those laboratories that used reduced oxidation reagent over 2 or 3 cycles and those laboratories who used a large dose in one cycle.

Increased oxidation reagent doses ($K_2S_2O_8$ and NaOH) might create alkaline conditions that can favour precursor conversion to PFSAs via hydrolysis rather than PFCA. Laboratories should consider reviewing their oxidation method when there is *a change* exceeding 50% (25% when a confidence interval of 95% is used) between the pre-oxidation and post-oxidation PFDS concentration.

7.4.4. Oxidation Completeness and Near Completeness

In order to be able to effectively assess the extent of PFAS contamination in samples, TOP Assay relies on complete oxidation/conversion of suspected precursors to C4 – C14 PFAS compounds. However, when this cannot be achieved, PFAS NEMP considers a near-complete oxidation acceptable when "for aqueous samples, sum of PFAA precursors divided by sum of Total PFAS < 5%."Participants' results reported for precursors, PFCA and PFSA pre- and post-TOP Assay are presented in Figure 162.

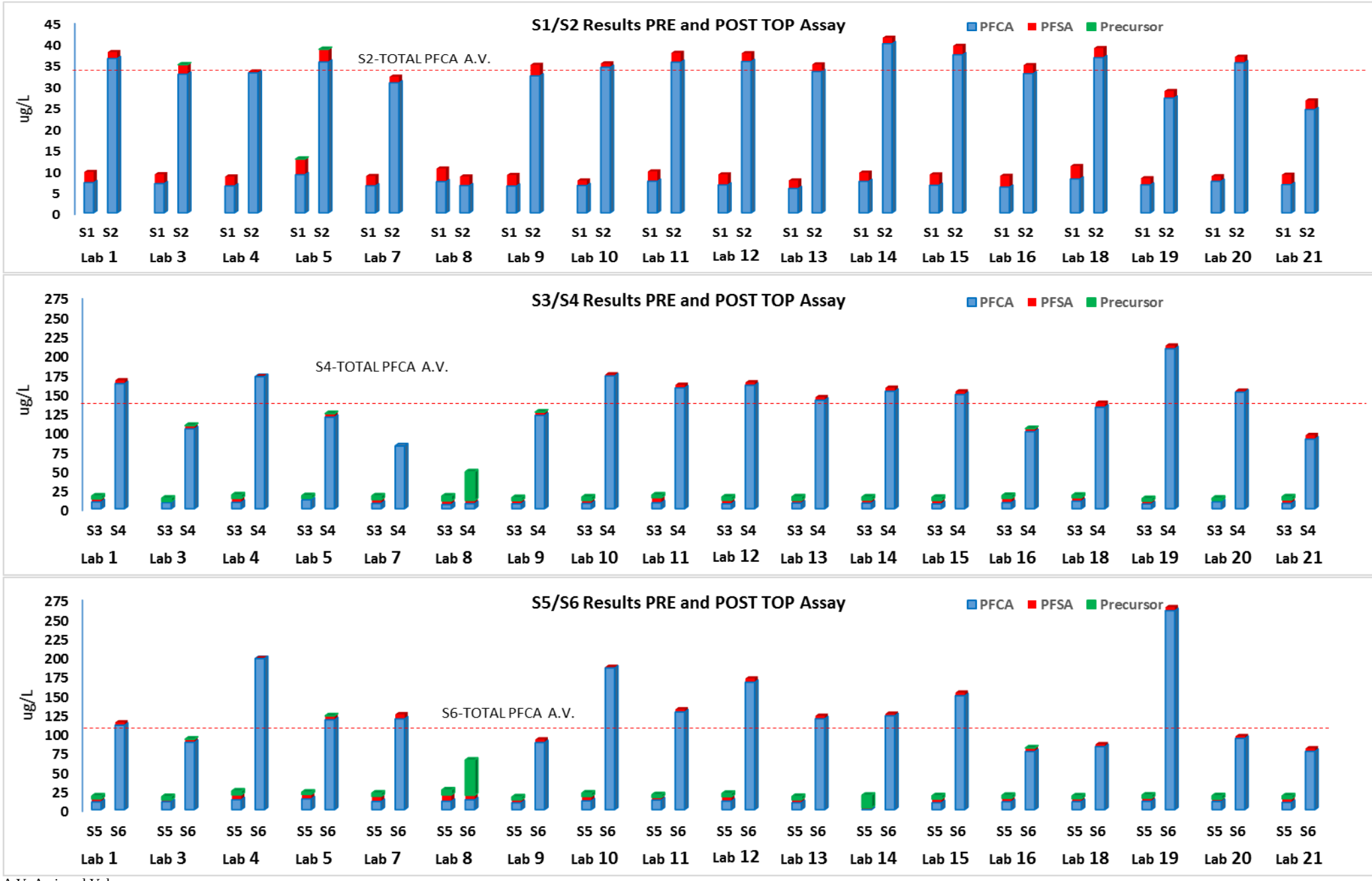
The MilliQ water samples S1/S2 were spiked with 6:2 diPAP and 8:2 diPAP. These compounds are usually not within the laboratories' analytical suite and so oxidation completion was difficult to assess directly. However, the results for C4 - C9 PFCA in S2 were in good agreement, indicating comparable conversion of the added fluorotelomer precursors to the expected PFCA by participating laboratories (Table 59).

Samples S3/S4 and S5/S6 were spiked with the same amount of Alcoseal. The major precursor identified by laboratories in pre-oxidation samples was 6:2 FTS. The oxidative pre-treatment used by laboratories 3, 5, 9 and 16 did not fully convert the identified precursors to PFCA. However, all participants complied with the NEMP criteria for acceptability of 'near complete' oxidation.

Laboratory 8 reported a larger amount of precursor in post-oxidation samples than in pre-oxidation samples.

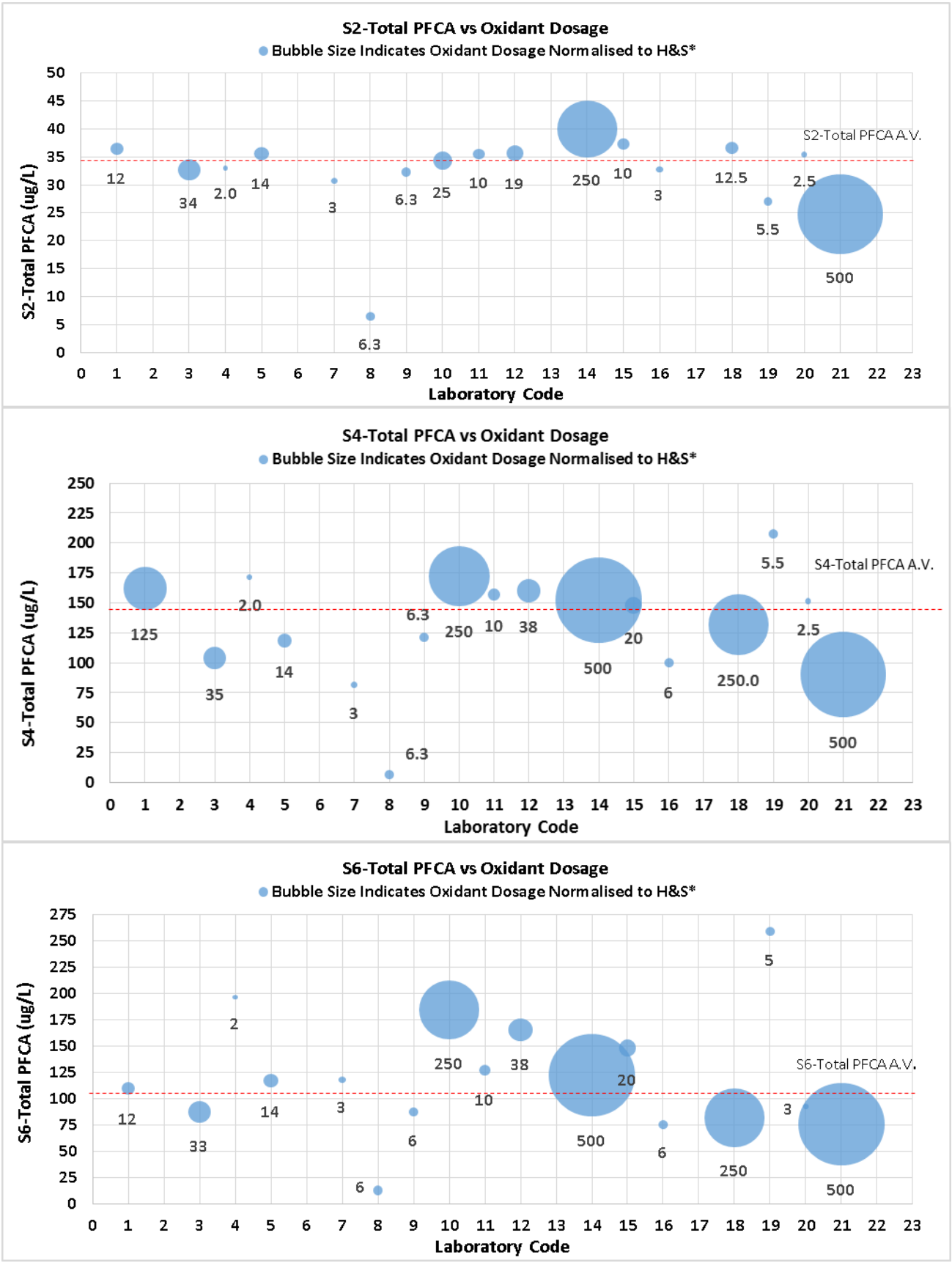
To meet PFAS NEMP QA requirements for aqueous samples, laboratories needed to increase oxidant dosage, dilute the samples and/or employ extra oxidative cycles relative to the H&S method (Figures 154, 155 and 156). Plots of participants' results for Total PFCAs versus amount of oxidant used per mL of sample normalised to the H&S dosage, are presented in Figure 163. Laboratories used between 2 to 500 times the dosage employed by Houtz and Sedlack. Most of the Total PFCA results they reported were in good agreement with each other, regardless of the amount of dosage used.
Figure 164 presents plots of participants' results for Total PFCA versus oxidation time. No significant trend with the extension of oxidation time was identified.

Laboratories have generally complied with PFAS NEMP requirements. However, this is not necessarily an accurate evaluation of their assay efficiency, as the *"Sum of Total PFAS"* used in the NEMP formula depends on the scope of PFAS analytes reported by laboratories, while for *" Sum of PFAA precursors* "only a narrow selection of precursors form part of their analytical suite.



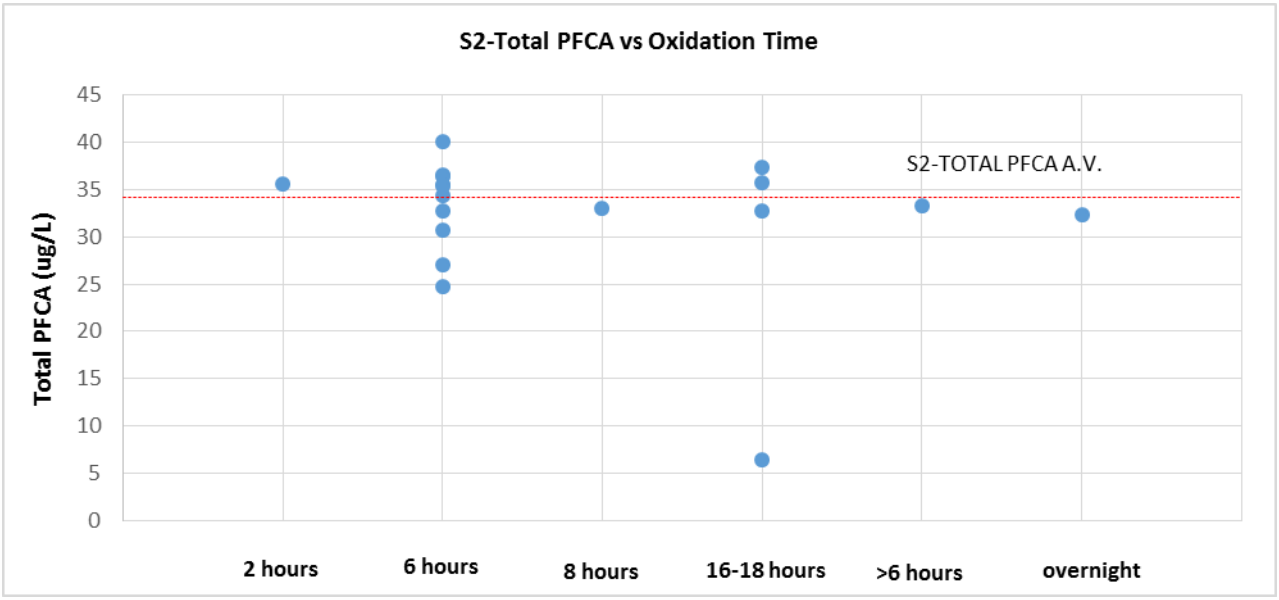
A.V. Assigned Value

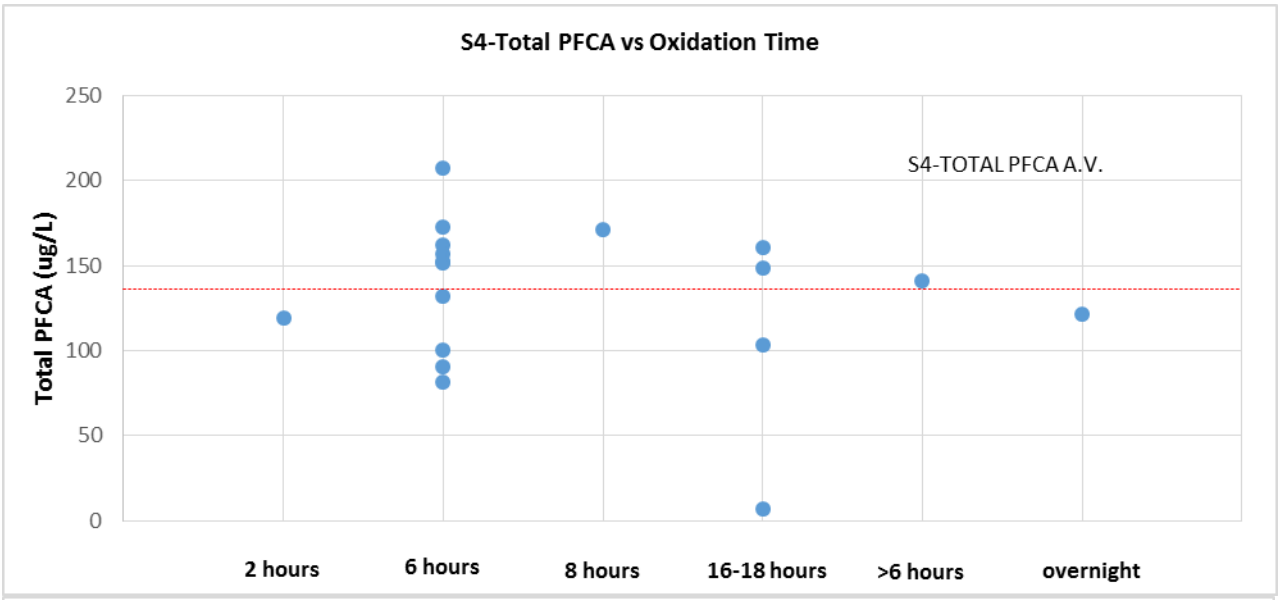
Figure 162 PFCA, PFSA and Precursors Results Pre and Post Oxidation

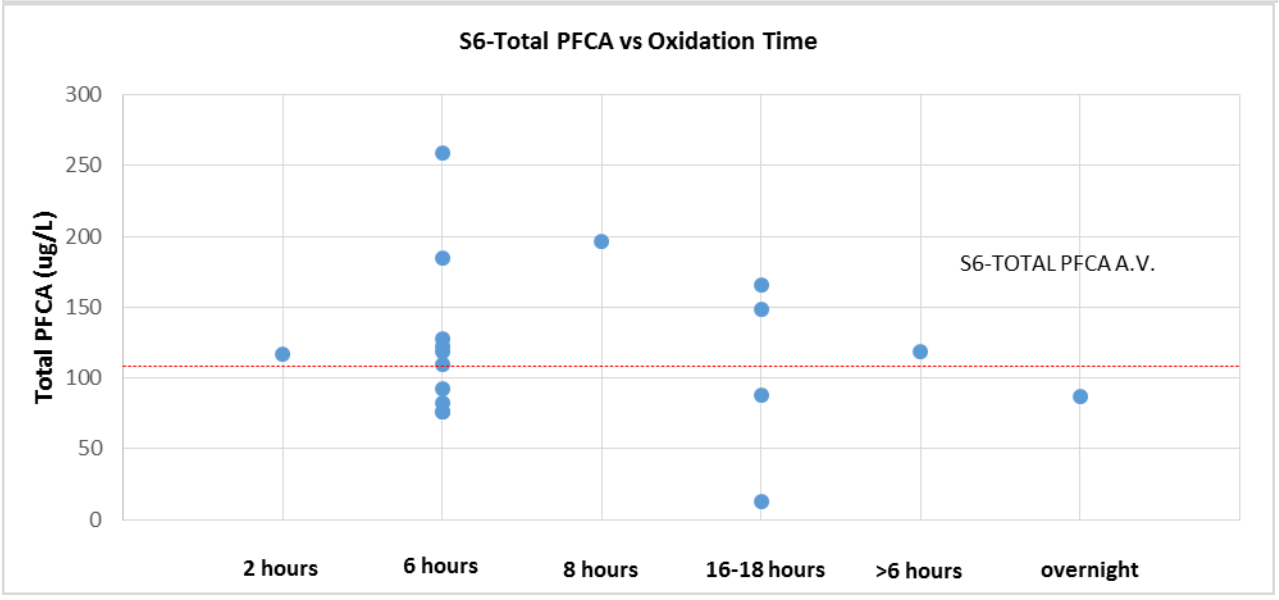


For Example: The normalised dosage of $K_2S_2O_8$ used by Laboratory 1 (12) is the total amount of $K_2S_2O_8$ used per mL of sample, (4 g) multiplied by the number of cycles (3) divided by samples size (61 mL) and further divided by the total amount of $K_2S_2O_8$ used by Houtz and Sedlak per mL of sample (2g/125mL) - where the sample size is the amount of sample taken for analysis (61 mL) divided by the dilution factor (1).

Figure 163 Total PFCA vs Dosage Normalised to H&S







A.V.-Assigned Value

Figure 164 Total PFCA vs Oxidation Time

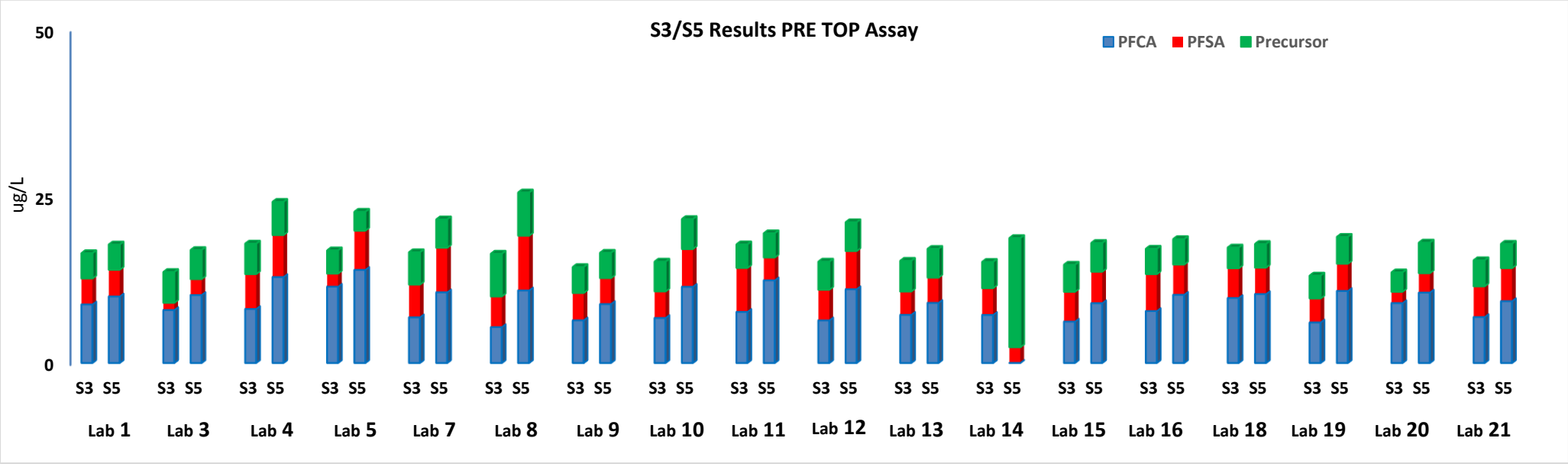


Figure 165 Results Pre TOP Assay in S3 and S5

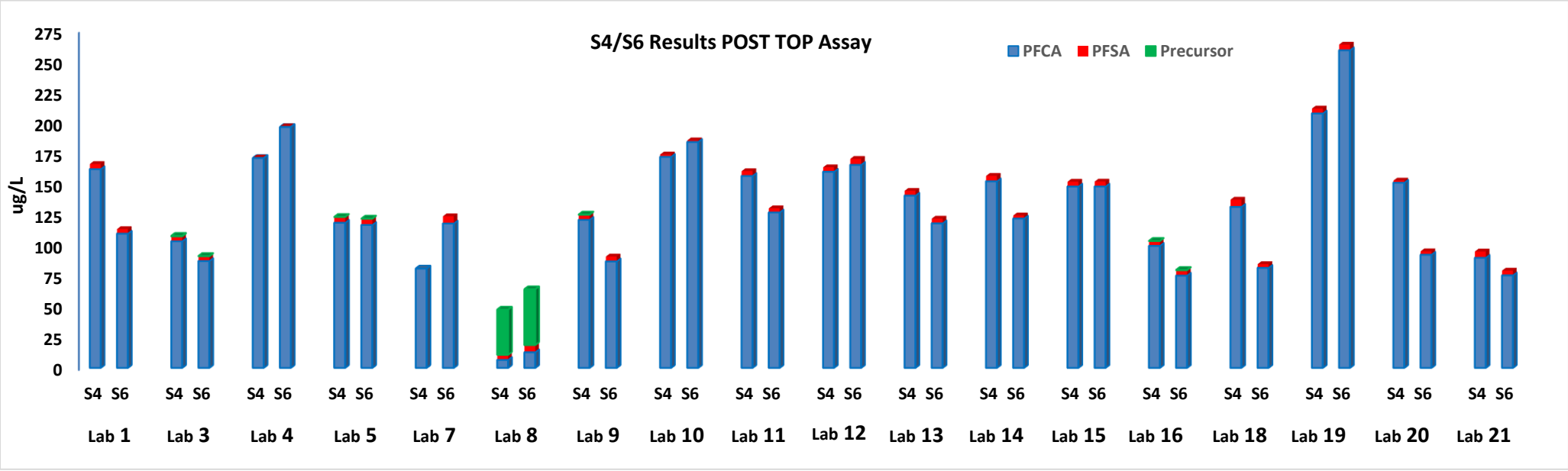


Figure 166 Results Post TOP Assay in S4 and S6

7.4.5. Oxidative Method Performance in Complex Matrices

Samples S1/S2 were MilliQ water samples spiked with fluorotelomer precursors (6:2 diPAP and 8:2 diPAP) while samples S3/S4 were also MilliQ water samples but spiked with Alcoseal foam. Laboratories 1, 10, 12, 14, 15 and 18 used a higher dilution factor for S4 than for S2; all other participants used the same regime across all three samples.

Samples S5/S6 were river water samples also spiked with Alcoseal but further fortified with a fluorine-free AFFF for a TOC content of 100 mg/L. The same amount of Alcoseal was added to the two pairs of samples S3/S4 and S5/S6. The concentration of Total PFCA in the post-oxidative Samples S4 and S6 was expected to be similar. The assigned value calculated for Total PFCA in S4 was 136 µg/L, while that of the more complex sample S6 was lower at 108 µg/L. With the exception of Laboratory 1, all participants used the same oxidation regime for both samples. Laboratory 1 diluted Sample S4 10 times, but did not do so for the more complex sample S6.

Figure 165 presents participants' results pre-TOP Assay in MilliQ water sample S3 and river water sample S5, while Figure 166 presents results post-TOP Assay in S4 and S6. Overall, there was no substantial difference between participants' oxidative method performance in the MilliQ water sample S4 compared to the river water sample S6.

The between laboratory coefficient of variation in the 3 types of post-oxidation samples was found to increase with matrix complexity: 8.7% in the MilliQ water sample S2 spiked with fluorotelomer precursors, 24% in the MilliQ water sample S4 spiked with Alcoseal, and 26% in the river water sample spiked with Alcoseal and further fortified with TOC (Figure 55 and Table 59).

7.5 Conclusions

TOP Assay is a method designed to assess the extent of overall PFAS contamination in a sample. It can indirectly measure the precursors by comparing PFCA concentration before and after oxidation. The maximum chain length of the oxidation products after TOP Assay application is a reflection of the maximum possible perfluorinated chain length of the precursors.

Based on PFAS NEMP QA requirements, the following criteria was used to assess participants' application of TOP Assay to study samples: material loss during preparation, method capability in converting precursors to PFCA, precursor conversion to PFSA, and complete or near-complete oxidation.

All laboratories based their TOP assay procedure on the H&S method. However, most laboratories: used extra doses of oxidant (2 to 500 times the amount of oxidant used by H&S per mL of sample), had an extended oxidation time of up to 18 hours, diluted the samples before oxidation and/or increased the number of oxidation of cycles to up to 3.

With very few exceptions, laboratories fulfilled the NEMP QA requirements. The results for individual PFCAs in S2 and for Total PFCA in S2, S4 and S6 were in reasonable consensus with each other. This indicates a comparable conversion of the added precursors to the expected PFCAs by participating laboratories. A bias towards low results was noticed among the PFDA results reported by laboratories in post-oxidation samples. The decrease was consistent among the three pairs of samples by approximately 14%. This is an indication of possible losses during the oxidation process employed by laboratories. Low spike recoveries were also registered for PFDS results in both pre- and post-oxidation types of samples, but the low PFDS recovery might be due to absorption of the analyte into the walls of the container rather than the oxidation process. Possible losses in PFDS could also have been registered during the preparation of the study samples, before samples' dispatch.

Although the majority of participants complied with NEMP QA requirements, the consistent low results reported for PFDA in the post-oxidation samples indicates that the methods employed biased carboxylic acid concentration low, for which there is no numerical quality assurance criteria in NEMP.

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APPENDIX 1 – PRE TOP ASSAY PERFLUOROALKYL ACIDS INCURRED

Table 115 Participants' Results for PFAA in Sample S1

Lab	PFBA µg/L	PFPeA µg/L	PFHxA µg/L	PFOA µg/L	PFUdA µg/L
1	< 0.04	< 0.03	< 0.02	< 0.03	< 0.04
3	0.00887	NR	NR	NR	NR
4	0.0107	< 0.01	< 0.01	0.0103	< 0.01
5	0.017	0.002	0.001	0.01	0.001
7	<0.10	<0.010	<0.010	<0.010	<0.010
8	NR	NR	NR	NR	NR
9	<0.05	<0.02	<0.01	<0.01	<0.01
10	<0.04	<0.04	<0.04	<0.04	<0.04
11	0.01	<0.01	<0.01	0.01	<0.01
12	0.0141	NR	NR	0.00721	NR
13	<0.02	< 0.02	< 0.01	<0.01	<0.02
14	<0.02	<0.02	<0.01	<0.02	<0.02
15	0.0132	NR	NR	0.0081	NR
16	<0.1	<0.02	<0.02	<0.01	<0.02
18	<0.1	<0.02	<0.02	<0.01	<0.02
19	0.011	<0.005	<0.005	0.012	<0.005
20	< 0.05	< 0.01	< 0.01	< 0.01	< 0.01
21	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10

Table 116 Participants' Results for PFAA in Sample S3

Lab	PFBA µg/L	PFPeA µg/L	PFHxA µg/L	PFHpA µg/L	PFOA µg/L	PFUdA µg/L	PFDoA µg/L
1	< 0.04	< 0.03	0.05	< 0.02	< 0.03	< 0.04	< 0.08
3	0.0234	NR	0.0338	NR	0.0106	NR	NR
4	0.0229	< 0.01	0.0521	< 0.01	0.0136	< 0.01	< 0.01
5	0.032	0.004	0.058	0.001	0.015	0.001	<0.002
7	<0.15	<0.010	0.042	<0.010	0.01	<0.010	<0.010
8	NR	NR	0.054	NR	NR	NR	NR
9	<0.05	<0.02	0.053	<0.01	0.014	<0.01	<0.01
10	<0.04	<0.04	<0.04	<0.04	<0.04	<0.04	<0.04
11	0.03	<0.01	0.07	<0.01	0.01	0.02	0.01
12	0.0887	NR	0.0435	NR	0.0101	NR	NR
13	0.03	< 0.02	0.06	< 0.01	0.01	<0.02	<0.05
14	0.02	<0.02	0.03	<0.02	0.011	<0.02	<0.02
15	0.08027	NR	0.04937	NR	0.0152	NR	NR
16	<0.1	<0.02	0.05	<0.02	<0.01	<0.02	<0.02
18	<0.1	<0.02	0.06	<0.02	0.01	<0.02	<0.02
19	0.026	<0.005	0.078	<0.005	0.015	<0.005	<0.005
20	< 0.05	< 0.01	0.04	< 0.01	0.01	< 0.01	< 0.01
21	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10

Table 117 Participants' Results for PFAA in Sample S5

Lab	PFBS µg/L	PFHxS µg/L	PFOS µg/L	PFNS µg/L	PFBA µg/L	PFPeA µg/L	PFHxA µg/L	PFHpA µg/L	PFOA µg/L	PFUdA µg/L	PFDoA µg/L	PFTeDA µg/L
1	< 0.21	< 0.21	< 0.21	< 0.21	< 0.42	< 0.29	< 0.21	< 0.21	< 0.29	< 0.42	< 0.83	< 0.83
3	NR	NR	0.0128	NR	0.0395	NR	0.106	NR	0.0672	NR	NR	NR
4	< 0.10	< 0.10	< 0.10	< 0.10	< 0.15	< 0.10	0.109	< 0.10	0.0885	< 0.10	< 0.10	< 0.10
5	0.005	0.01	0.021	<0.002	0.063	0.04	0.111	0.019	0.081	0.003	<0.002	<0.002
7	<0.010	<0.010	0.01	NT	<0.40	0.026	0.074	0.014	0.058	<0.010	<0.010	<0.025
8	NR	NR	NR	NT	0.051	0.034	0.11	NR	0.016	NR	NR	NR
9	<0.01	<0.01	<0.02	<0.01	<0.05	0.028	0.087	0.017	0.064	<0.01	<0.01	<0.02
10	<0.04	<0.04	<0.04	0.06	0.04	0.06	0.11	<0.04	0.07	<0.04	<0.04	<0.04
11	<0.01	0.01	0.02	<0.02	0.06	0.05	0.09	<0.01	0.12	<0.01	0.04	<0.2
12	0.00321	0.0029	0.0069	NR	0.0462	0.0234	0.0867	0.0114	0.0612	NR	NR	NR
13	< 0.05	< 0.01	<0.02	NT	0.05	0.03	0.1	0.02	0.07	<0.02	<0.05	<0.5
14	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	0.02	<0.02	<0.02	<0.02
15	0.0034	0.0045	0.0057	NR	0.0403	0.0274	0.0976	0.0137	0.0638	NR	NR	NR
16	<0.02	<0.02	<0.02	NT	<0.1	<0.04	0.11	<0.02	0.08	<0.02	<0.02	<0.05
18	<0.02	<0.02	<0.02	NT	<0.1	0.02	<0.02	<0.02	0.074	<0.02	<0.02	<0.05
19	<0.005	<0.005	<0.005	NT	0.044	0.037	0.134	0.026	0.072	<0.005	<0.005	<0.005
20	< 0.1	< 0.1	< 0.1	0.11	< 0.05	< 0.1	0.1	< 0.1	0.11	0.12	0.17	0.15
21	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10

APPENDIX 2 - SAMPLE PREPARATION AND HOMOGENEITY TESTING

A2.1 Sample Preparation

Analytical standards used for spiking samples in AQA 19-19 were purchased from HPC Standards GmbH, Toronto Research Chemicals, Sigma-Aldrich and Wellington Laboratories Canada. On the analytical reports provided with the standards, all analytes have a stated purity of >95%.

Sample S1 and S2: 11452.1 g of MilliQ water was spiked with four individual solutions in methanol and stirred with an IKA stirrer for approximately 4 hours. A peristaltic pump was then used to dispense the water into labelled 60 mL HDPE bottles, the first half being Sample S1 and the second Sample S2.

Sample S3 and S4: 11331.8 g of MilliQ water was spiked with three individual solutions. The water was then stirred for approximately 4 hours before being dispensed into labelled 60 mL HDPE bottles using a peristaltic pump. The first portion was labelled Sample S3 and the second portion labelled S4.

Sample S5 and S6: 1808.8 g of MilliQ water was added to 9611.8 g of river water collected from Brown's Water Hole Turramurra which had been autoclaved. This mixture was stirred using an IKA stirrer for approximately 2 hours to ensure homogeneity, prior to being spiked with four individual solutions. The spiked water was then further stirred for approximately 4 hours prior to dispensing with a peristaltic pump. The first half of the spiked water was Sample S5 and the second half Sample S6, dispensed into labelled 60 mL HDPE bottles.

All six water samples were stored at 4°C prior to dispatch to participants.

A2.2 Homogeneity Testing

The same validated preparation procedure was followed as in a similar study previous conducted by NMI. The test samples from the previous study were demonstrated to be sufficiently homogeneous for evaluation of participants' performance. [7]

APPENDIX 3 - ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY

The robust average was calculated using the procedure described in 'ISO 13528:2015(E), Statistical methods for use in proficiency testing by interlaboratory comparisons – Annex C'.[8]

The uncertainty was estimated as:

$$u_{rob\ av} = 1.25 * S_{rob\ av} / \sqrt{p} \qquad \text{Equation 4}$$

where:

$u_{rob\ av}$	robust average standard uncertainty
$S_{rob\ av}$	robust average standard deviation
p	number of results

The expanded uncertainty ($U_{rob\ av}$) is the standard uncertainty multiplied by a coverage factor of 2 at approximately 95% confidence level.

A worked example is set out below in Table 118.

Table 118 Uncertainty Estimate for PFDA in Sample S1

No. results (p)	18
Robust Average	6.80 µg/L
$S_{rob\ av}$	0.70 µg/L
$u_{rob\ av}$	0.20 µg/L
k	2
$U_{rob\ av}$	0.41 µg/kL

The robust average for PFDA in Sample S1 is **6.80 ± 0.41 µg/L**.

z-Score and E_n-score

For each participant's result a z-score and E_n-score are calculated according to Equation 2 and Equation 3 respectively (see page 17).

A worked example is set out below in Table 119.

Table 119 z-Score and E_n-score for PFDA result reported by Laboratory 8 in S1

PFDA Result µg/L	Assigned Value µg/L	Set Target Standard Deviation	z-Score	E_n-Score
7.30±1.46	6.80±0.41	20% as CV or 0.2x6.80= =1.36 µg/L	$z = \frac{(7.30 - 6.80)}{1.36}$ z = 0.368	$En = \frac{(7.30 - 6.80)}{\sqrt{1.46^2 + 0.41^2}}$ E_n= 0.33

APPENDIX 4- ACRONYMS AND ABBREVIATIONS

6:2 FTS	1H, 1H, 2H, 2H-perfluorooctane sulfonate
6:2 diPAP	6:2/6:2 polyfluoroalkyl phosphate ester
8:2 FTS	1H, 1H, 2H, 2H-perfluorodecane sulfonate
8:2 diPAP	8:2/8:2 polyfluoroalkyl phosphate ester
AFFF	Aqueous Film Forming Foam
AV	Assigned Value
CRM	Certified Reference Material
CV	Coefficient of Variation
DI	Direct Injection
EPA	Environment Protection Authority
GUM	Guide for Uncertainty Measurement
H&S	Houtz and Sedlak
ID-LC-MSMS	Isotope Dilution – Liquid Chromatography – Tandem Mass Spectrometry
ISO	International Standards Organisation
LC	Liquid Chromatography
LOR	Limit Of Reporting
Max	Maximum value in a set of results
Md	Median
Min	Minimum value in a set of results
MS	Mass Spectrometry
NATA	National Association of Testing Autority
NMI	National Measurement Institute (of Australia)
NEMP	National Environment Management Plan
NR	Not Reported
NT	Not Tested
PFAA	Perfluoroalkyl acids
PFAS	Per- and poly fluorinated alkyl substances
PFBA	Perfluoro-n-butanoic acid
PFBS	Potassium perfluoro-1-butanesulfonate
PFCA	Perfluorocarboxylic acid
PFDA	Perfluoro-n-decanoic acid
PFDoA	Perfluorododecanoic acid
PFDS	Perfluorodecane sulfonate
PFHpA	Perfluoro-n-heptanoic acid
PFHpS	Perfluoroheptane sulfonate
PFHxA	Perfluoro-n-hexanoic acid
PFHxS	Potassium perfluorohexanesulfonate
PFNA	Perfluoro-n-nonanoic acid

PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctane sulfonate
PFOSA	Perfluoro-1-octanesulfonamide
PFPeA	Perfluoro-n-pentanoic acid
PFPeS	Perfluoropentane sulfonate
PFTeDA	Perfluorotetradecanoic acid
PFTrA	Perfluorotridecanoic acid
PFUdA	Perfluoroundecanoic acid
PT	Proficiency Test
QA	Quality Assurance
QTOF	Quadrupole Time-of-Flight
QQQ	Triple Quadrupole (mass spectrometry)
Robust CV	Robust Coefficient of Variation
Robust SD	Robust Standard Deviation
SV	Spiked or formulated concentration of a PT sample (Spike Value)
SPE	Solid Phase Extraction
Target SD	Target standard deviation
TOC	Total Organic Carbon
TOP	Total Oxidisable Precursor
σ	Target standard deviation

END OF REPORT