



Australian Government
Department of Industry,
Science and Resources

National
Measurement
Institute

Proficiency Test Final Report AQA 25-18 TF and PFAS in Packaging Materials

September 2025

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ACKNOWLEDGMENTS

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

Luminita Antin

Mark Lewin

Jasmine Duong

Jenny Xu

Hamish Lenton

Sofia Racomelara

Sam Falvey

Patricia Apostol

Todd Rattanakone

Chunhai Yu

Gavin Stevenson

I would also like to thank Bob Symons from Eurofins Environment Testing Australia for reviewing this report.

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SUMMARY

This report presents the results of the proficiency testing (PT) study, AQA 25-18 TF and PFAS in Packaging Materials. This study was a NMI Special Study designed based on participants' requests and is focused on the measurement of total fluorine (TF) and 16 per- and polyfluoroalkyl substances (PFAS): PFBA, PFPeA, PFHxA, PFOA, PFNA, PFDA, PFDoA, PFBS, total PFHxS, linear PFHxS, PFHpS, total PFOS, linear PFOS, 6:2FTS, 6:2diPAP and 8:2diPAP in cardboard packaging material. The study also included a pilot sample, a plastic food packaging material to be analysed for TF.

This is the first time a cardboard packaging material matrix has been introduced in our PFAS program, and possibly the first time some laboratories have participated in a study involving this type of sample. For this initial study, a spiked sample was selected to allow the spike values to serve as supporting evidence for the assigned values. A packaging material sample containing PFAS analytes as incurred compounds will also be included in our next PT study for PFAS in consumables.

Eighteen laboratories from Australia, Bulgaria, Canada, France, Japan, the Netherlands, the UK, the USA and Vietnam enrolled in this study and seventeen reported results.

Three test samples were provided:

- two cardboard packaging material samples: Sample S1 with incurred TF, and Sample S2 with 12 spiked and 4 incurred individual PFAS analytes; and
- one plastic food packaging material Sample S3, with incurred TF.

The assigned values were the robust averages of participants' results. The associated uncertainties were evaluated from the robust standard deviations of the participants' results.

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

- i. *assess laboratory capability in measuring TF and PFAS in cardboard packaging material;*

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

Of 175 z-scores, 144 (82%) returned $|z| \leq 2.0$, indicating an acceptable performance.

Of 169 E_n -scores, 119 (70%) returned $|E_n| < 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Laboratory 12 had the highest number of acceptable z-scores and E_n -scores (14).

- ii. *evaluate the laboratories' methods used in determination of total fluorine and PFAS in packaging material;*

83% of the results reported for PFAS in Sample S2 returned acceptable z-scores, but challenges remain with packaging matrices compared to environmental samples.

Eight laboratories reported at least one PFAS analyte that was not spiked into test sample S2.

The most popular sample preparation method used for PFAS in packaging materials was a SLE extraction based on the method that involved a sample size of 3 g, base modified methanol as extraction solvent and no extract concentration and cleanup. LC-MS/MS (triple quadrupole, QQQ) was the instrumental technique of choice for all participants.

- iii. *develop the practical application of measurement uncertainty and provide participants with information that will be useful in evaluating their uncertainties;*

Laboratories should review their uncertainties evaluation procedure as some laboratories have reported unrealistically small or large relative uncertainties for routine PFAS. The magnitude

of these expanded uncertainties was within the range 1.2% to 90% of the reported value. Additionally, some laboratories have reported numeric evaluations of uncertainties for non-numeric results.

iv. produce materials that can be used in method validation and as control samples.

Surplus test samples from the present study are available for sale. The samples are homogeneous and well characterised, both by in-house testing and from the results of the proficiency round.

1 INTRODUCTION

1.1 NMI Proficiency Testing Program

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

Proficiency testing (PT) is the: 'evaluation of participant performance against pre-established criteria by means of interlaboratory comparison.'¹ 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:

- per- and polyfluoroalkyl substances in soil, biosolid, water, biota, food, and consumables;
- hydrocarbons, phenols and other organic compounds in soil and water;
- pesticide residues in soil, water, fruit, vegetables, and herbs;
- metals in soil, water, food, filters, and paint;
- nutrients and anions in water and soil;
- chlorophyll a in water; and
- controlled drug assay, drugs in wipes, and clandestine laboratory.

1.2 Study Background

This study was initiated to support laboratories in developing and/or assessing their analytical methods for measuring PFAS in packaging materials — an emerging area of concern that is driving significant regulatory changes.

Under the European Union's Packaging and Packaging Waste Regulation (PPWR), strict limits on PFAS in food contact materials will come into effect from 12 August 2026. These measures aim to address the environmental persistence and serious health risks associated with PFAS, including carcinogenicity and reproductive toxicity. The regulation sets concentration thresholds at 25 ppb for individual PFAS, 250 ppb for the sum of PFAS, and 50 ppm for total fluorine content (including polymeric PFAS).²

In the United States, the Food and Drug Administration (FDA) has reached voluntary agreements with manufacturers to phase out the use of certain PFAS in food contact substances, particularly those used as grease-proofing agents.^{3,4}

Canada regulates PFAS in food packaging through Health Canada's safety guidelines and inspections protocol. Meanwhile, Japan's the Ministry of Health, Labour and Welfare proposed a ban in 2022 on 56 substances related to perfluorooctanoic acid (PFOA), aligning with global efforts to restrict hazardous PFAS compounds.^{3,4}

Australia introduced its first nationwide PFAS restriction through the Industrial Chemicals Environmental Management Standard (IChEMS). This regulation prohibits the import, export, and manufacture of certain PFAS substances—whether on their own or within articles—although it does not specifically target food packaging.⁵

1.3 Study Aims

The aims of the study were to:

- assess laboratory capability in measuring TF and PFAS in cardboard packaging material;
- evaluate the laboratories' methods used in determination of TF and PFAS in packaging material;

- develop the practical application of measurement uncertainty and provide participants with information that will be useful in evaluating their uncertainties; and
- produce materials that can be used in method validation and as control samples.

1.4 Study Conduct

The conduct of NMI proficiency tests is described in the NMI Study Protocol for Proficiency Testing.⁶ The statistical methods used are described in the NMI Chemical Proficiency Testing Statistical Manual.⁷ These documents have been prepared with reference to ISO/IEC 17043¹ and The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.⁸

NMI is accredited by the National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043:2023 as a provider of proficiency testing schemes.¹ This study is not within the scope of NMI's accreditation.

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of the study was:

Invitation issued	22 April 2025
Samples dispatched	02 June 2025
Results due	04 July 2025
Interim report issued	09 July 2025
Preliminary report issued	21 July 2025

2.2 Test Material Preparation

Three test samples were provided for analysis.

- Sample S1 consisted of 3 g cardboard packaging material
- Sample S2 consisted of two identical containers of 3 g cardboard packaging material, spiked with 12 individual PFAS components.
- Sample S3 consisted of 1 g plastic food packaging material

Details of the spiked analytes and levels for Sample S2 are presented in Table 1 and sample preparation details for all three samples are collated in Appendix 1.

Table 1 Formulated Mass Fraction of Test Sample S2

PFAS	S2 Cardboard Spike Value µg/kg
PFBA	25.3**
PFPeA	5.98
PFHxA	264**
PFOA	20.1**
PFDA	1.50
PFDoA	15.0
PFBS*	4.12**
PFHxS*	60.1
PFHxS L*	60.1
PFOS*	14.3**
PFOS L*	9.66**
6:2FTS*	50.1

*Values for these analytes are the anion concentration. **The spike value includes the incurred value.

2.3 Participation

Eighteen laboratories participated in this study, and seventeen submitted results. A confidential laboratory code number was assigned to each of these eighteen participants.

2.4 Test Material Homogeneity and Stability Testing

The preparation of the study samples is described in Appendix 1. Full homogeneity and stability assessment was conducted on the study samples except for PFNA, PFDA, 6:2diPAP and 8:2diPAP. However, no relationship was evident between the results reported for these analytes and the date when the sample was received (Appendix 1).

2.5 Sample Storage, Dispatch and Receipt

Before dispatch, Samples S1 and S3 were stored at room temperature, whilst Sample S2 was refrigerated at 4°C.

The samples were packed in a foam box with cooler bricks and sent by courier on 2 June 2025.

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.

2.6 Instructions to Participants

Participants were instructed as follows:

- Quantitatively analyse the samples using your routine test method.
- Report results on an as received basis in units of mg/kg for Samples S1 and S3 (TF), and on an as received basis in units of µg/kg for Sample S2 (PFAS).
- If analyses cannot be commenced on the day of receipt, please store Sample S2 refrigerated. Store Samples S1 and S3 at room temperature in a dry place, covered.
- For Sample S2, participants will be asked to use the entire sample and to rinse the sample container with the reagent used for extraction. For the calculation of the results the sample weight should be assumed to be 3.00 g. Two identical containers are provided for this sample so that laboratories can repeat the measurement if required.
- PFAS analytes that may be present in the samples were given in a list. Participants could elect not to test for all listed analytes.
- For analytes that contain linear and branched isomers you will be requested to report the sum of linear and branched isomers. For PFOS and PFHxS, participants are asked to report results for both total and linear isomers.
- The concentration range for TF in Sample S1 is >10 mg/kg and in Sample S3 is <50 mg/kg. The analyte range of PFAS in Sample S2 is 0-500 µg/kg.
- Report results using the electronic results sheet emailed to you.
- For each analyte, report a single result expressed as if reporting to a client (i.e. corrected for recovery or not, according to your standard procedure, but state if results are corrected on the result sheet). This figure will be used in all statistical analysis in the study report.
- For each analyte report the associated expanded measurement uncertainty as mg/kg for Samples S1 and S3 or µg/kg for Sample S2 e.g., 0.532 ± 0.021 mg/kg or 0.532 ± 0.021 µg/kg, if determined.
- No limit of reporting has been set for this study. Report results as you would to a client, applying the limit of reporting of the method used for analysis.

- Report any listed analyte not tested as NT.
- Please complete the method details and report the basis of your uncertainty evaluations as required by the results sheet.
- If determined, report your internal standard percentage recovery. This will be presented in the report for information only

2.7 Interim Report and Preliminary Report

An Interim Report was emailed to all participants on 9 July 2025.

A Preliminary Report was emailed to all participants on 21 July 2025. This report included: a summary of results reported by all laboratories, assigned values, performance coefficients of variation, z-scores and E_n -scores for each analyte tested by participants.

No data from the Preliminary Report has been changed in the present Final Report.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Methods Reported by Participants

Participants were requested to provide methodology information. Responses are presented in Appendix 5 for TF and Appendix 6 for PFAS analytes. The study coordinator thanks participants for completing the questionnaire.

3.2 Basis of Participants' Measurement Uncertainty Evaluations

Participants were requested to provide information about their basis of measurement uncertainty (MU). This is presented in Tables 2 and 3 (some responses have been modified so that participants cannot be identified).

Table 2 Basis of Participants' Uncertainty Evaluations

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
1	Top Down - precision and evaluates of the method and laboratory bias k = 2	Control samples - RM Duplicate analysis	CRM Laboratory bias from PT studies Recoveries of SS	Nordtest Report TR537
2 ^a	Standard deviation of replicate analyses multiplied by 2 or 3 k = 2	Duplicate analysis	CRM Instrument calibration	ISO/GUM
3	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control samples - SS Duplicate analysis Instrument calibration		Eurachem/CITAC Guide
4 ^a	Coverage factor not reported			
5	Top Down - precision and evaluates of the method and laboratory bias k = 2	Duplicate analysis Instrument calibration	CRM Instrument calibration Recoveries of SS Standard purity	ISO/GUM
6	Standard deviation of replicate analyses multiplied by 2 or 3 k = 2	Control samples - SS	Recoveries of SS	NMI Uncertainty Course
7	Top Down - precision and evaluates of the method and laboratory bias Coverage factor not reported	Duplicate analysis	Recoveries of SS	Eurachem/CITAC Guide
8	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS	
9	Top Down - precision and evaluates of the method and laboratory bias k = 2	Control samples - RM Duplicate analysis		ISO/GUM
10	Top Down - precision and evaluates of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis Instrument calibration	CRM Recoveries of SS	NMI Uncertainty Course
11	Top Down - precision and evaluates of the method and laboratory bias Coverage factor not reported	Control samples - SS	Recoveries of SS	

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
12	Top Down - precision and evaluates of the method and laboratory bias Coverage factor not reported	Control samples - SS Duplicate analysis Instrument calibration	CRM	NMI Uncertainty Course
13	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control samples - SS	Recoveries of SS	standard deviations from the mean
14	Top Down - precision and evaluates of the method and laboratory bias Coverage factor not reported	Control samples - SS	Laboratory bias from PT studies	ISO 11352
15	Top Down - precision and evaluates of the method and laboratory bias Coverage factor not reported	Duplicate analysis Instrument calibration	Recoveries of SS	ISO/GUM
16	Top Down - precision and evaluates of the method and laboratory bias Coverage factor not reported	Control samples - CRM Duplicate analysis Instrument calibration		Eurachem/CITAC Guide
17 ^a	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control samples - SS	Recoveries of SS	USEPA SW-846

*SS = Spiked Samples, RM = Reference Material, CRM = Certified Reference Material. ^aAdditional Information in Table 4

Table 3 Uncertainty Evaluation Additional Information

Lab Code	Approach to Evaluating MU
2	ISO 17025 certified, In house SOP described the method for uncertainty evaluation S1: Sample measured in triplicate (values: 61 - 63 - 66 mg/kg) S3: Analysis in triplicate (all values were below Limit of detection; this was determined using multiple blank measurements). Therefore, no uncertainty reported
4	Method under validation - no MU available yet.
17	Standard Practice for laboratories utilizing US EPA's SW-846 document

3.3 Participants' Comments

Participants were invited to make comments for this PT study. Such feedback allows for the improvement of future studies. Participants' comments are presented in Table 4, along with the study coordinator's response where appropriate.

Table 4 Participants' Comments

Lab Code	Participants' Comments	Study Coordinator's Response
8	S1: White part of the sample had the majority of the Total Fluorine (<100 mg/kg), whilst the brown part was less than reporting limit (>5mg/kg). Due to the technique of using C-IC only a small amount of sample was able to be weighed out (between 10-30 mg). Depending on the size of the precut cardboard square, different ratios of White and Brown cardboard could be present.	The aim of this study was to provide laboratories feedback on the method employed for TF measurements in packaging material, including subsampling procedure. A full homogeneity test was conducted on Sample S1 for TF in the cardboard material. The data is provided in Appendix 1.

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 5 to 22 with resultant summary statistics: robust average, median, mean, number of numeric results, 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 19. An example chart with interpretation guide is shown in Figure 1.

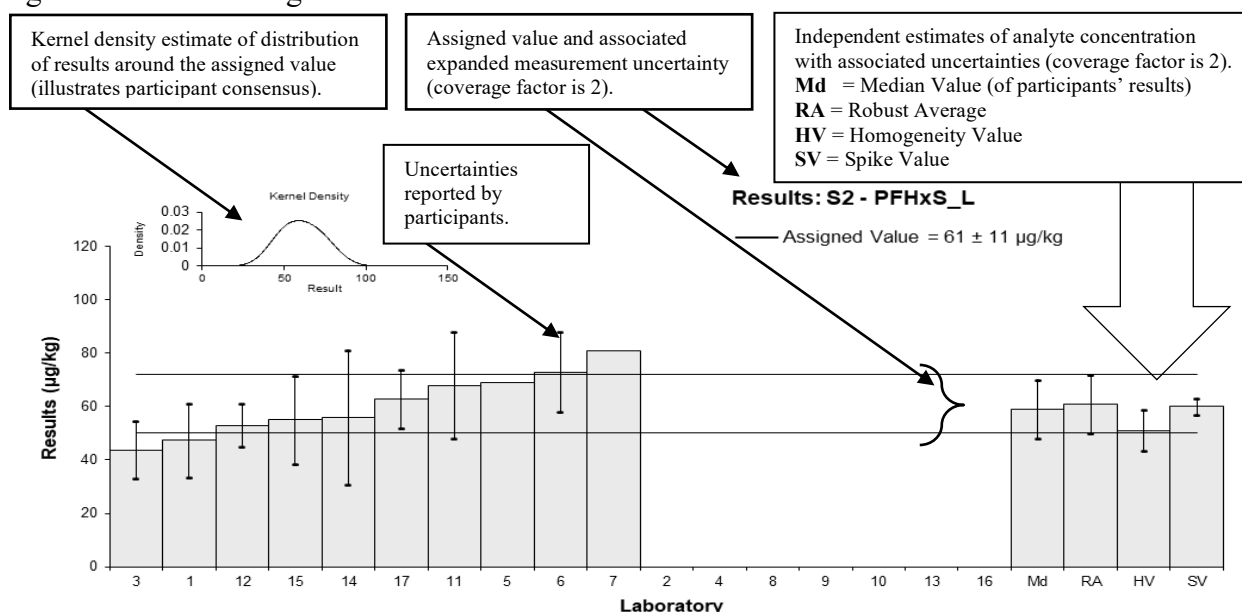


Figure 1 Guide to Presentation of Results

4.2 Outliers and Extreme Outliers

Outliers were results less than 50% and greater than 150% of the robust average and were removed before assigned value calculation. Extreme outliers (gross errors) were obvious blunders, such as those with incorrect units, decimal errors, or results from a different proficiency test item and were removed for calculation of summary statistics.^{8,9}

4.3 Assigned Value

An example of the assigned value calculation using data from the present study is given in Appendix 2. The assigned value is defined as 'the value attributed to a particular property of a proficiency test item'.¹ In this study the property is the mass fraction of analyte. Assigned values were the robust average of participants' results; outliers removed; the expanded uncertainties were evaluated from the associated robust standard deviations.^{8,9}

4.4 Robust Average and Robust Between-Laboratory Coefficient of Variation

The robust averages and associated expanded measurement uncertainties were calculated using the procedure described in 'Statistical methods for use in proficiency testing by interlaboratory comparisons, ISO13528'.⁹ 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 ISO13528.⁹

4.5 Standard Deviation for Proficiency Assessment

The standard deviation for proficiency assessment ($SDPA, \sigma$) is the product of the assigned value (X) and the performance coefficient of variation (PCV). This value is used for

calculation of participant z-score and provides scaling for laboratory deviation from the assigned value.

$$\sigma = (X) \times \text{PCV} \quad \text{Equation 1}$$

It is important to note that the PCV is a fixed value and is not the standard deviation of participants' 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.¹⁰

4.6 z-Score

An example of z-score calculation using data from the present study is given in Appendix 2. For each participants' result a z-score is calculated according to Equation 2 below:

$$z = \frac{(\chi - X)}{\sigma} \quad \text{Equation 2}$$

where:

- z is z-score;
- χ is participants' result;
- X is the assigned value;
- σ is the standard deviation for proficiency assessment

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

- $|z| \leq 2.0$ is acceptable;
- $2.0 < |z| < 3.0$ is questionable;
- $|z| \geq 3.0$ is unacceptable.

4.7 E_n-Score

An example of E_n-score calculation using data from the present study is given in Appendix 2. The E_n-score is complementary to the z-score in assessing laboratory performance. E_n-score includes measurement uncertainty and is calculated according to Equation 3 below:

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

where:

- E_n is E_n-score;
- χ is a participants' result;
- X is the assigned value;
- U_χ is the expanded uncertainty of the participants' result;
- U_X is the expanded uncertainty of the assigned value.

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

- $|E_n| < 1.0$ is acceptable;
- $|E_n| \geq 1.0$ is unacceptable.

4.8 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025¹¹ 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.¹²

5 TABLES AND FIGURES

Table 5

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	Total Fluorine
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NR	NR		
2	63	5	-0.62	-0.74
3	NS	NS		
4	42	NR	-2.08	-2.73
5	NS	NS		
6	NS	NS		
7	79	15.8	0.49	0.36
8	75.5	10.21198	0.24	0.23
9	NS	NS		
10	81	13	0.62	0.53
11	68	20	-0.28	-0.18
12	NS	NS		
13	<0.140	0.01848		
14	80.44	16.08	0.59	0.43
15*	22.2	4.31	-3.46	-4.22
16	NS	NS		
17	NT	NT		

* Outlier, see Section 4.2

Statistics

Assigned Value	72	11
Spike Value	Not Spiked	
Homogeneity Value	87	14
Robust Average	66	18
Median	72	11
Mean	64	
N	8	
Max	81	
Min	22.2	
Robust SD	20	
Robust CV	31%	

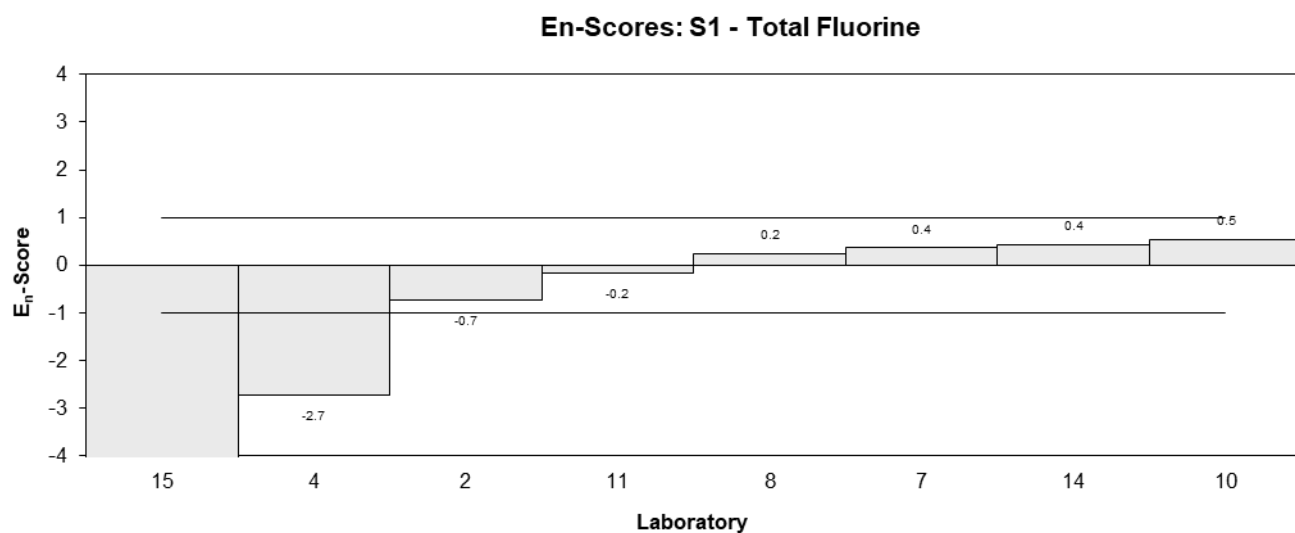
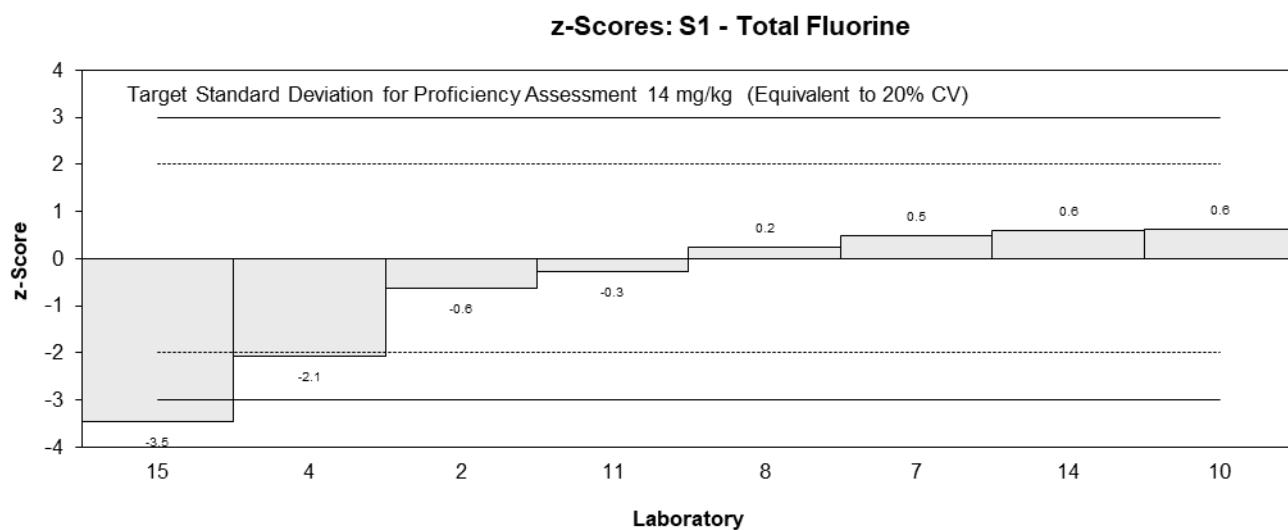
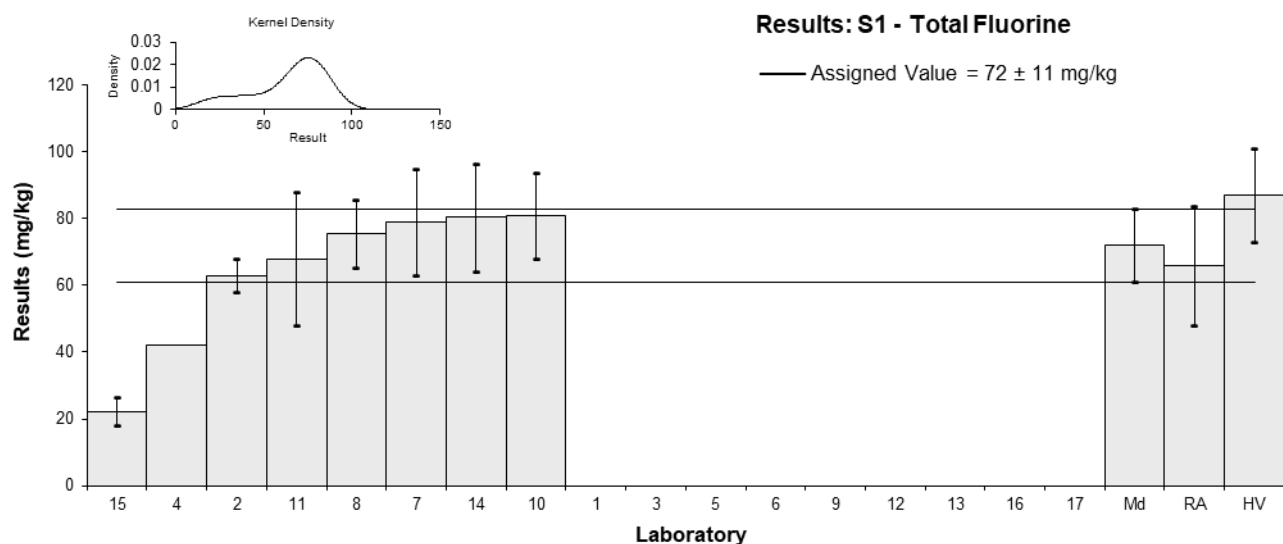


Figure 2

Table 6

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	PFBA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	17.656	3.98	31.4	-1.11	-0.95
2	NS	NS	NS		
3	17.3	6.51	35	-1.19	-0.73
4	NR	NR	NR		
5	19	NR	104	-0.81	-1.06
6	23	4.6	NR	0.07	0.05
7	<10	NR	NR		
8**	15248.61	NR	NR	3,353.72	4,350.26
9	NR	NR	NR		
10	NS	NS	NS		
11	27	9	88	0.95	0.45
12	23	3.5	121	0.07	0.06
13	19.2	4.224	84.2	-0.77	-0.64
14	26	11.7	NR	0.73	0.27
15	21.5	7.53	64	-0.26	-0.14
16	26.626	NR	21	0.86	1.12
17	29.0	5.2	72	1.39	1.01

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	22.7	3.5
Spike Value	25.3	1.2
Homogeneity Value	22.9	3.4
Robust Average	22.7	3.5
Median	23.0	4.2
Mean	22.7	
N	11	
Max	29	
Min	17.3	
Robust SD	4.6	
Robust CV	20%	

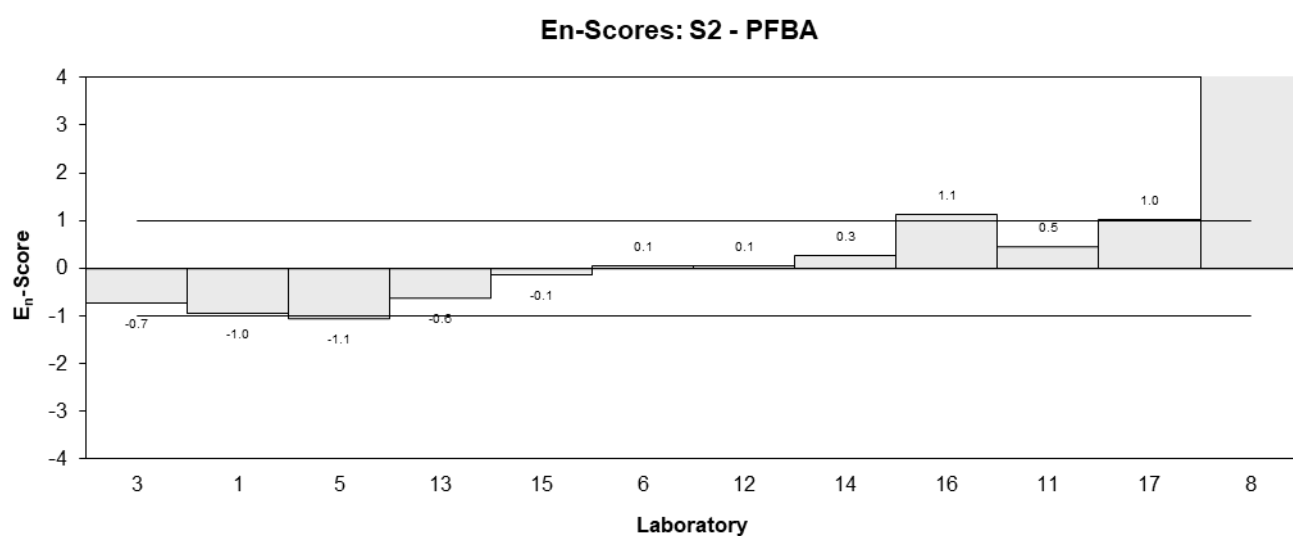
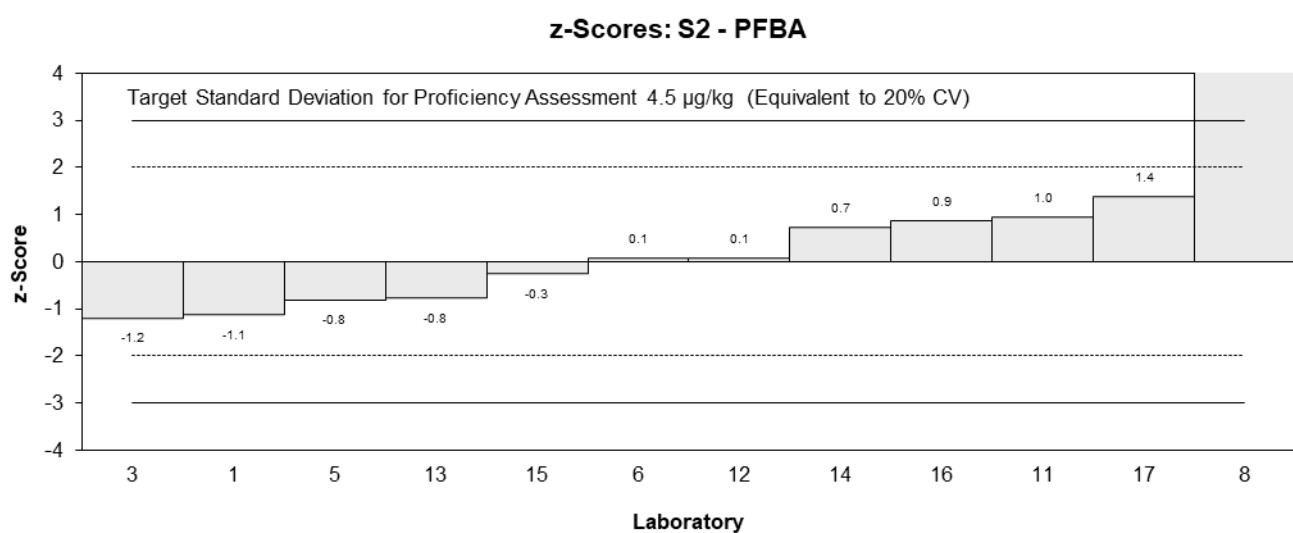
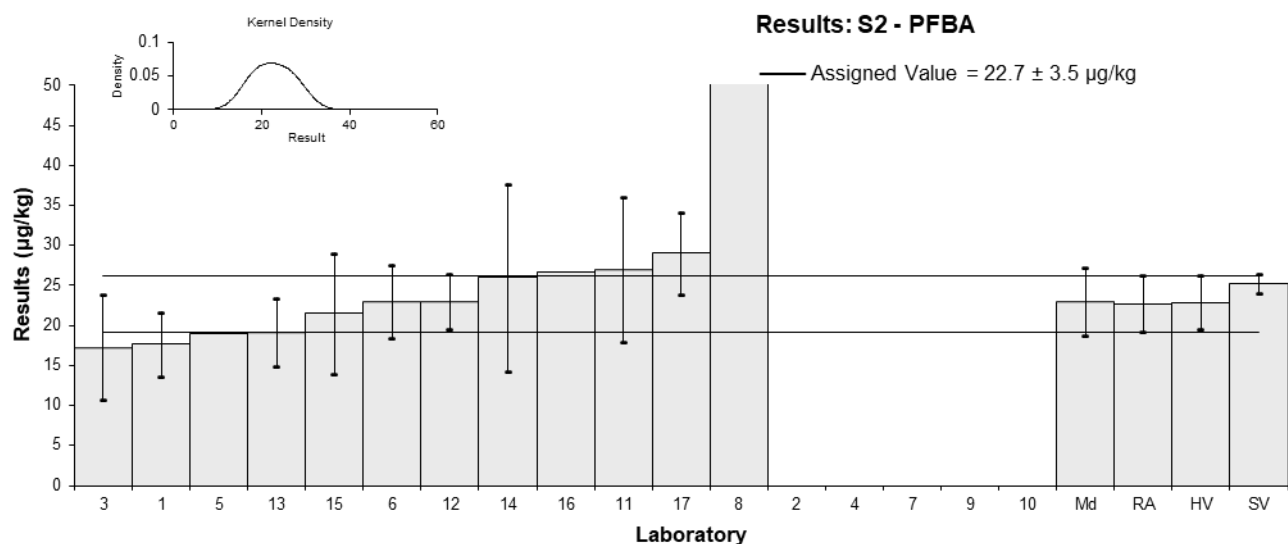


Figure 3

Table 7

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	PFPeA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	<0.584	NR	11.5		
2	NS	NS	NS		
3	4.84	1.3	33	-1.19	-0.95
4	NR	NR	NR		
5	6.3	NR	80	-0.04	-0.05
6	5.7	1.1	NR	-0.51	-0.46
7	<10	NR	NR		
8**	5694.58	NR	NR	4,478.92	6,250.80
9	NR	NR	NR		
10	NS	NS	NS		
11	7.3	3	99	0.75	0.30
12	6.4	0.96	88	0.04	0.04
13	3.99	0.9177	121	-1.86	-1.83
14	6.9	3.1	NR	0.43	0.17
15	6.57	2.30	62	0.17	0.09
16	7.561	NR	19	0.95	1.33
17	7.27	1.81	59	0.72	0.45

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	6.35	0.91
Spike Value	5.98	0.30
Homogeneity Value	5.73	0.86
Robust Average	6.35	0.91
Median	6.49	0.92
Mean	6.28	
N	10	
Max	7.561	
Min	3.99	
Robust SD	1.1	
Robust CV	18%	

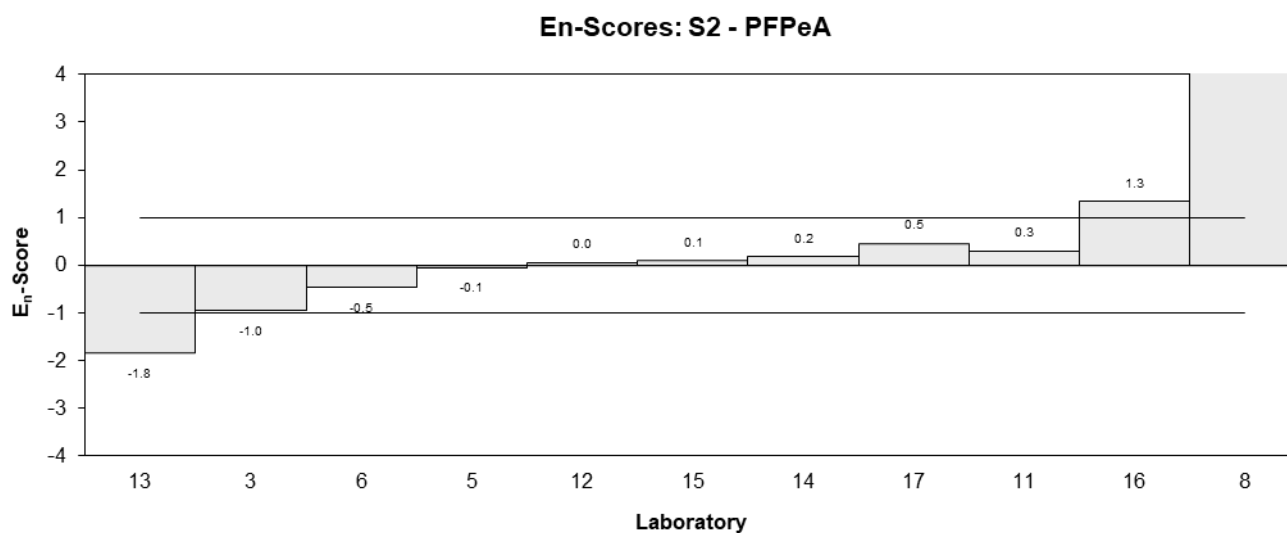
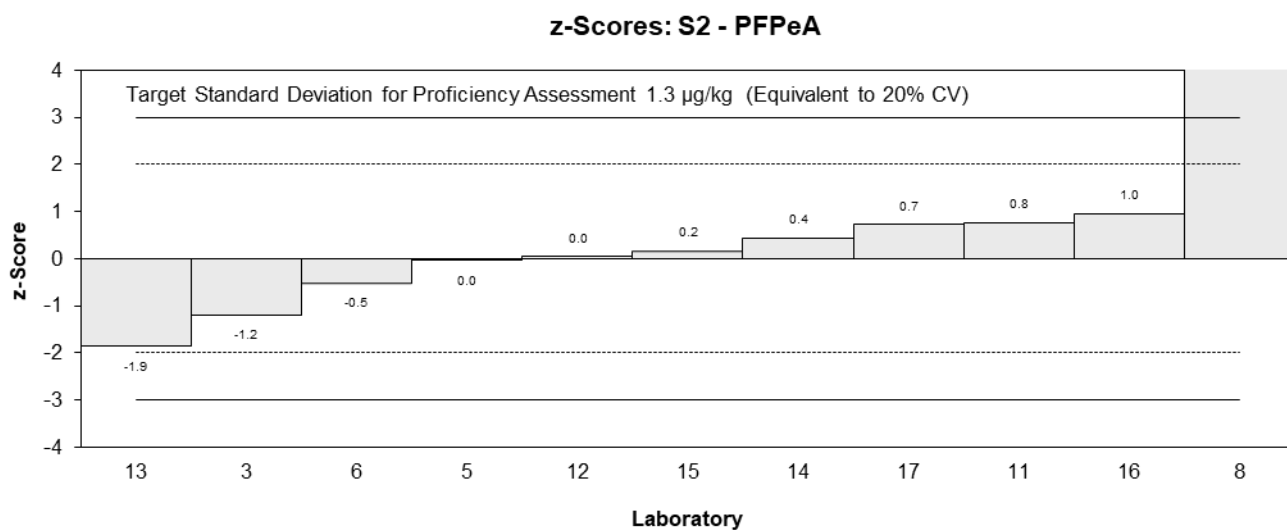
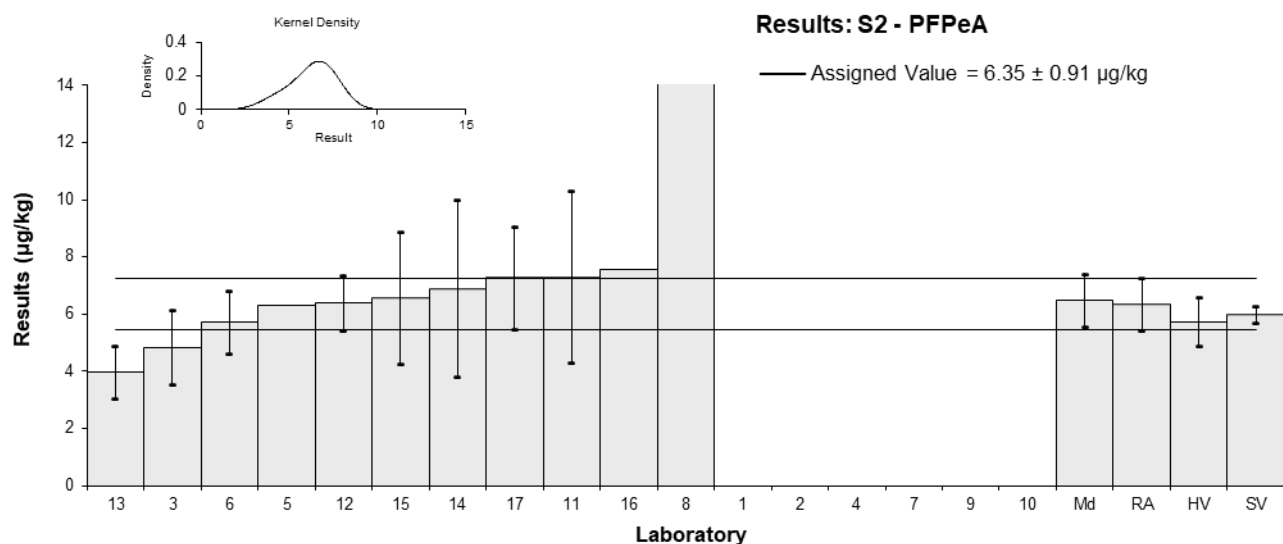


Figure 4

Table 8

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	PFHxA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	128	28.5	50.1	-2.25	-1.94
2	NS	NS	NS		
3	177.4	16.5	19	-1.19	-1.14
4	NR	NR	NR		
5	220	NR	69	-0.28	-0.28
6	240	48	NR	0.15	0.11
7*	390	14	83	3.37	3.27
8**	117353.8	NR	NR	2,513.32	2,546.11
9	251	58	NR	0.39	0.24
10	NS	NS	NS		
11	330	100	89	2.00▼	
12	235	35	59	0.04	0.03
13	189	41.58	85.2	-0.94	-0.71
14	207	93	NR	-0.56	-0.25
15	222	88.6	75	-0.24	-0.11
16	331.12	NR	15	2.00▼	
17	264	46.7	77	0.67	0.47

* Outlier, ** Extreme Outlier, see Section 4.2; ▼ Adjusted Score, see Section 6.3

Statistics

Assigned Value	233	46
Spike Value	264	13
Homogeneity Value	253	38
Robust Average	242	50
Max Acceptable Result	369	
Median	235	30
Mean	245	
N	13	
Max	390	
Min	128	
Robust SD	72	
Robust CV	30%	

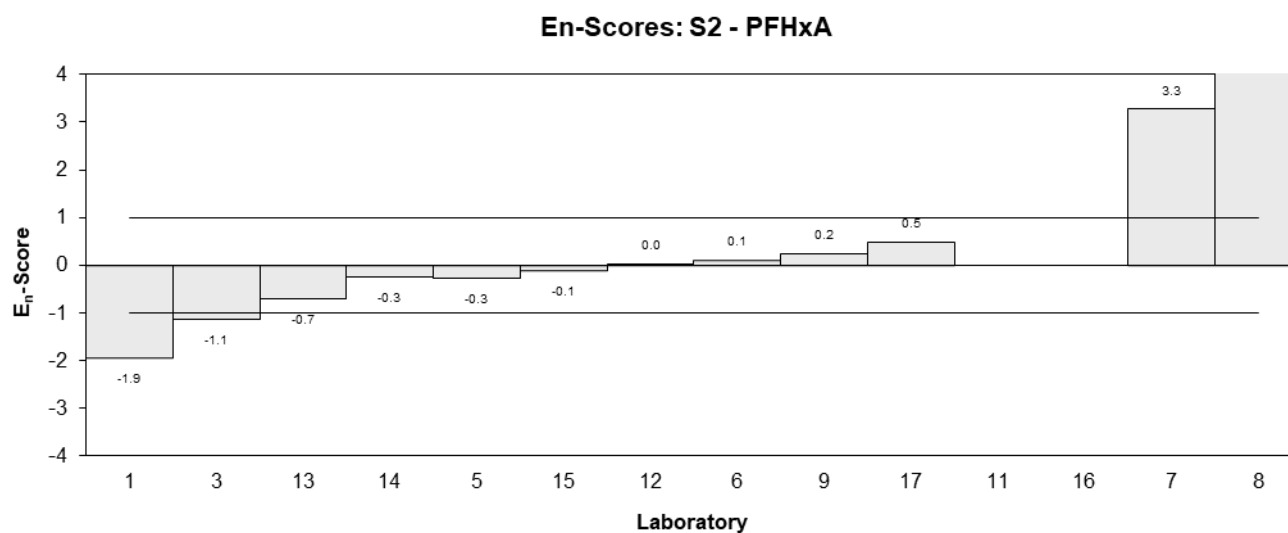
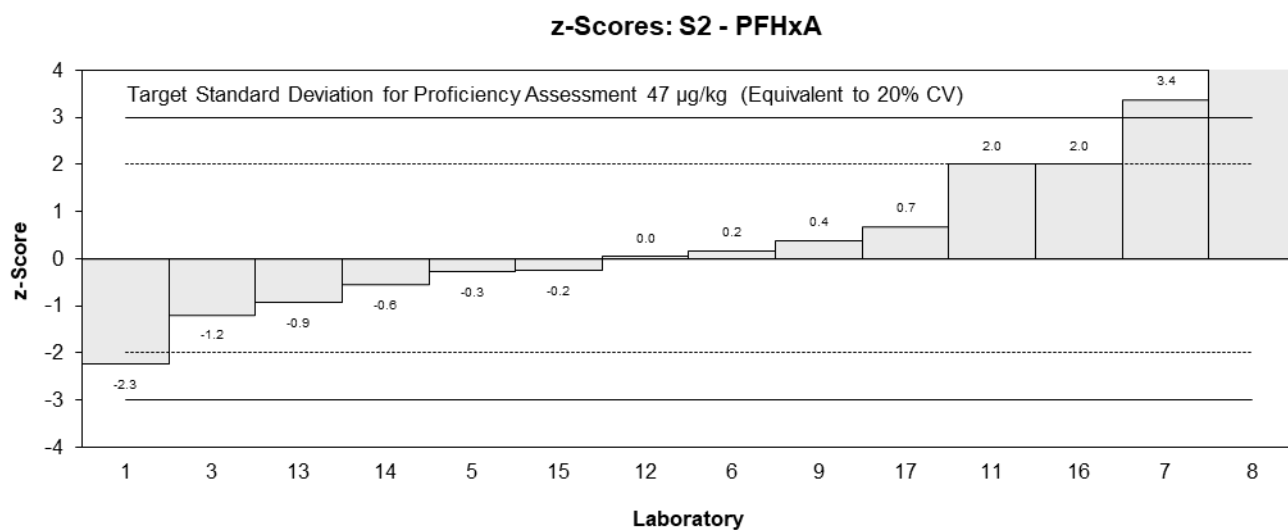
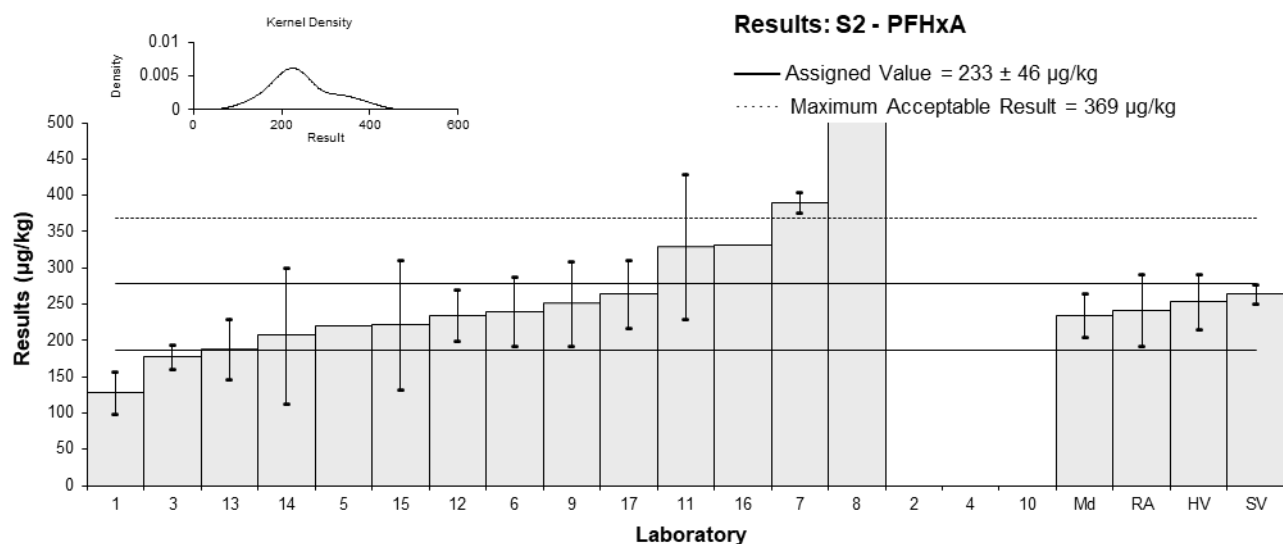


Figure 5

Table 9

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	PFOA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	14.2	2.59	68	-1.30	-1.36
2	NS	NS	NS		
3	15.6	0.18	33	-0.94	-1.38
4	NR	NR	NR		
5	29	11	102	2.55	0.87
6	19	3.8	NR	-0.05	-0.04
7*	42	20	116	5.94	1.13
8**	14677.70	NR	NR	3,817.32	5,637.88
9	20	5	NR	0.21	0.14
10	NS	NS	NS		
11	23	8	105	0.99	0.45
12	18	2.7	129	-0.31	-0.32
13	18.6	5.952	86.1	-0.16	-0.09
14	16.5	7.42	NR	-0.70	-0.34
15	18.4	6.45	70	-0.21	-0.12
16	18.621	NR	30	-0.15	-0.22
17	23.7	4.38	78	1.17	0.88

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	19.2	2.6
Spike Value	20.1	1.0
Homogeneity Value	18.1	2.7
Robust Average	19.9	3.1
Median	18.6	2.2
Mean	21.3	
N	13	
Max	42	
Min	14.2	
Robust SD	4.5	
Robust CV	22%	

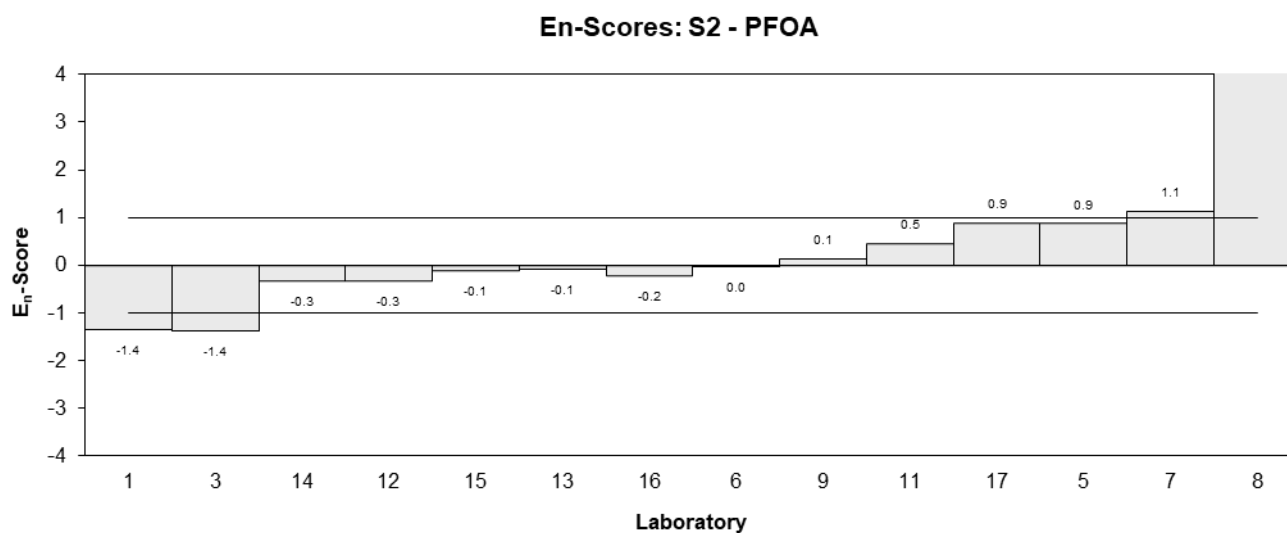
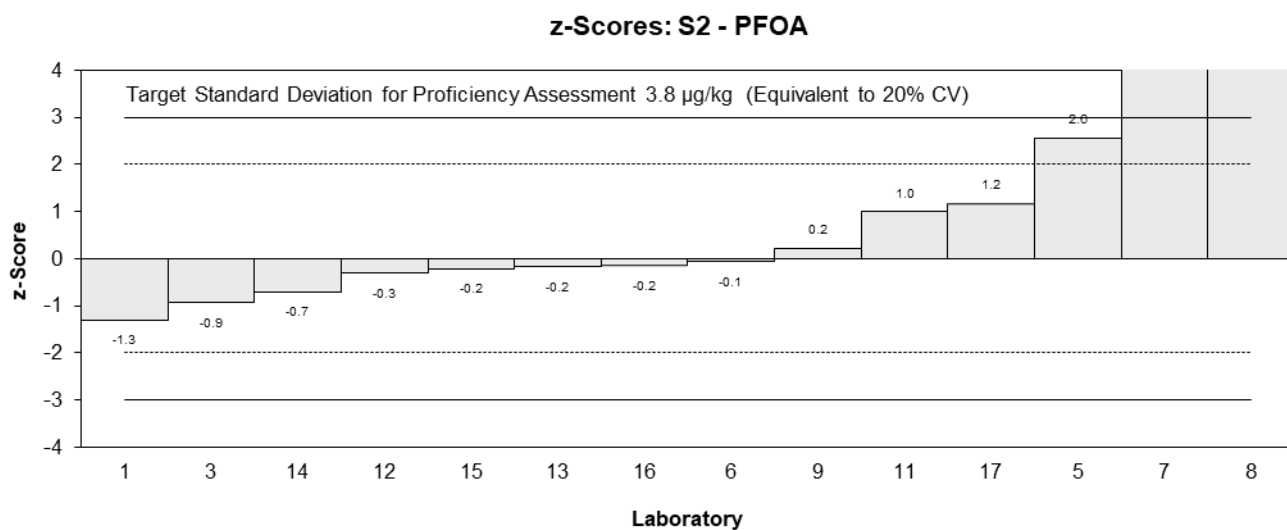
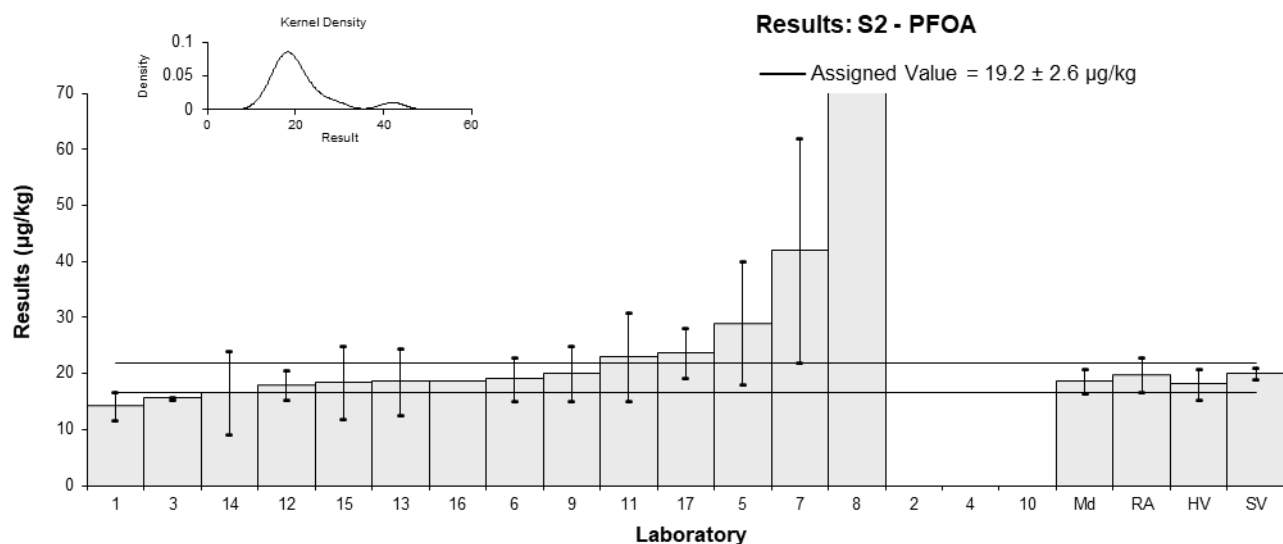


Figure 6

Table 10

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	PFNA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	0.139	0.038	65.6
2	NS	NS	NS
3	0.161	0.072	31
4	NR	NR	NR
5	0.19	0.04	102
6	<0.5	NR	NR
7	<10	NR	NR
8**	163.49	NR	NR
9	NR	NR	NR
10	NS	NS	NS
11	< 0.5	NR	112
12	0.14	0.038	143
13	0.162	0.0486	94.4
14	<0.1	NR	NR
15	<1	NR	67
16	<0.2	NR	19
17	<0.332	NR	87

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	Not Set	0.035
Spike Value	Not Spiked	
Robust Average	NA (N<6)	
Median	0.161	
Mean	0.158	
N	5	
Max	0.19	
Min	0.139	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

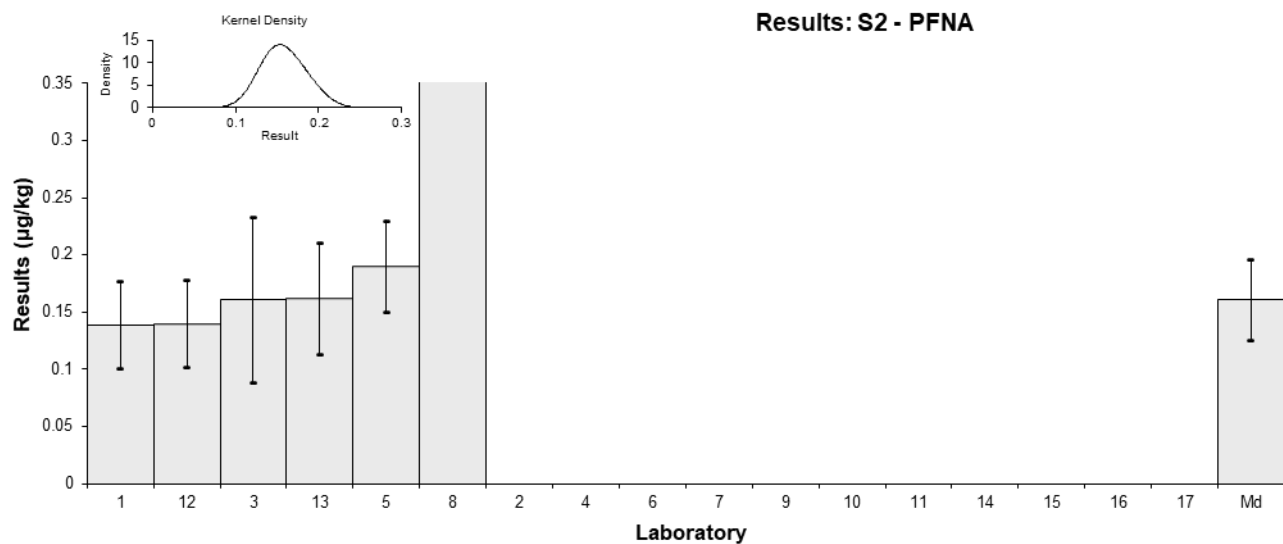


Figure 7

Table 11

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	PFDA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E _n
1	0.596	0.147	69	-2.19	-1.39
2	NS	NS	NS		
3	0.872	0.168	21	-0.89	-0.55
4	NR	NR	NR		
5	1.5	0.6	108	2.00▼	
6	<1	NR	NR		
7	<10	NR	NR		
8**	855.11	NR	NR	4,028.54	2,846.83
9	NR	NR	NR		
10	NS	NS	NS		
11	1.5	1	113	2.00▼	
12	0.82	0.12	160	-1.13	-0.74
13	0.84	0.2184	93.9	-1.04	-0.59
14	0.75	0.33	NR	-1.46	-0.70
15	1.02	0.358	93	-0.19	-0.09
16	1.359	NR	11	1.41	1.00
17	1.31	0.213	74	1.18	0.68

** Extreme Outlier, see Section 4.2; ▼ Adjusted Score, see Section 6.3

Statistics

Assigned Value	1.06	0.30
Spike Value	1.50	0.08
Robust Average	1.06	0.30
Max Acceptable Result	2.11	
Median	0.95	0.32
Mean	1.06	
N	10	
Max	1.5	
Min	0.596	
Robust SD	0.38	
Robust CV	36%	

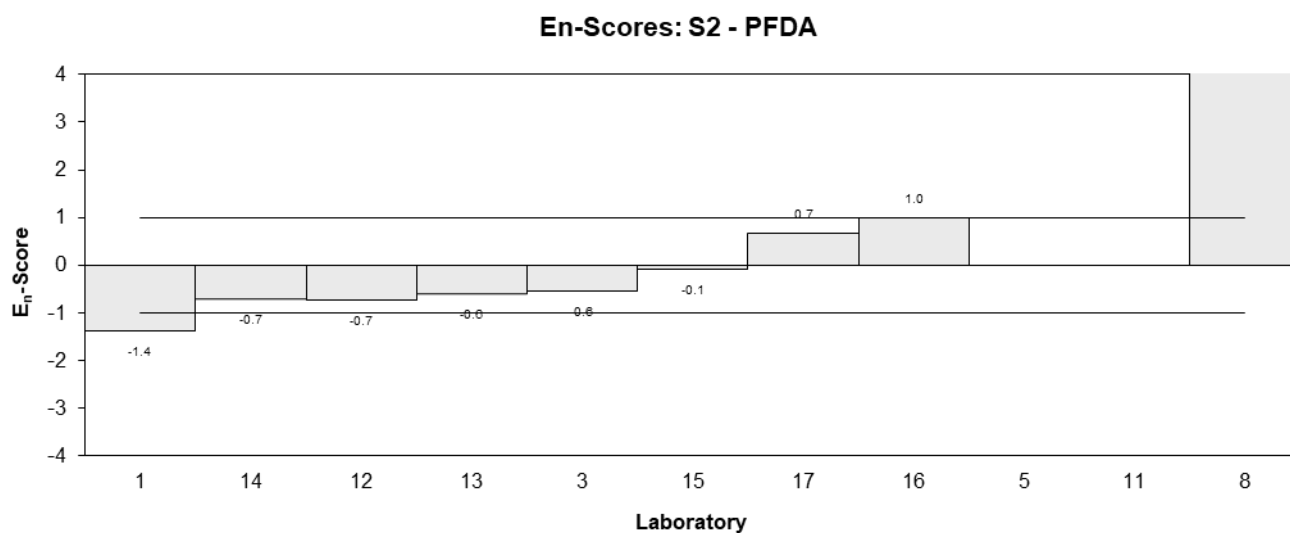
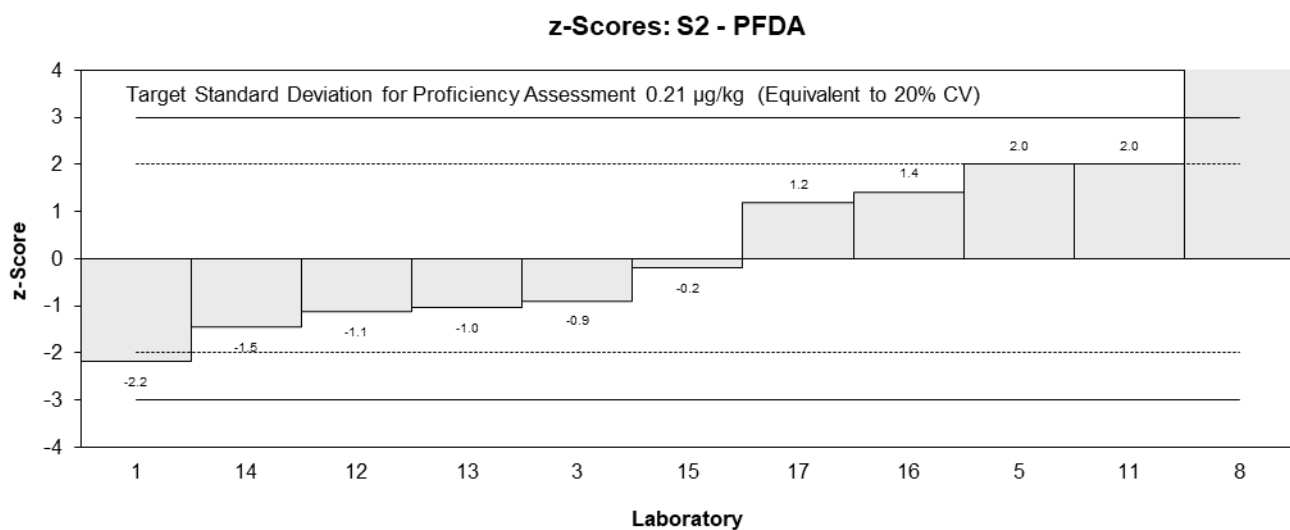
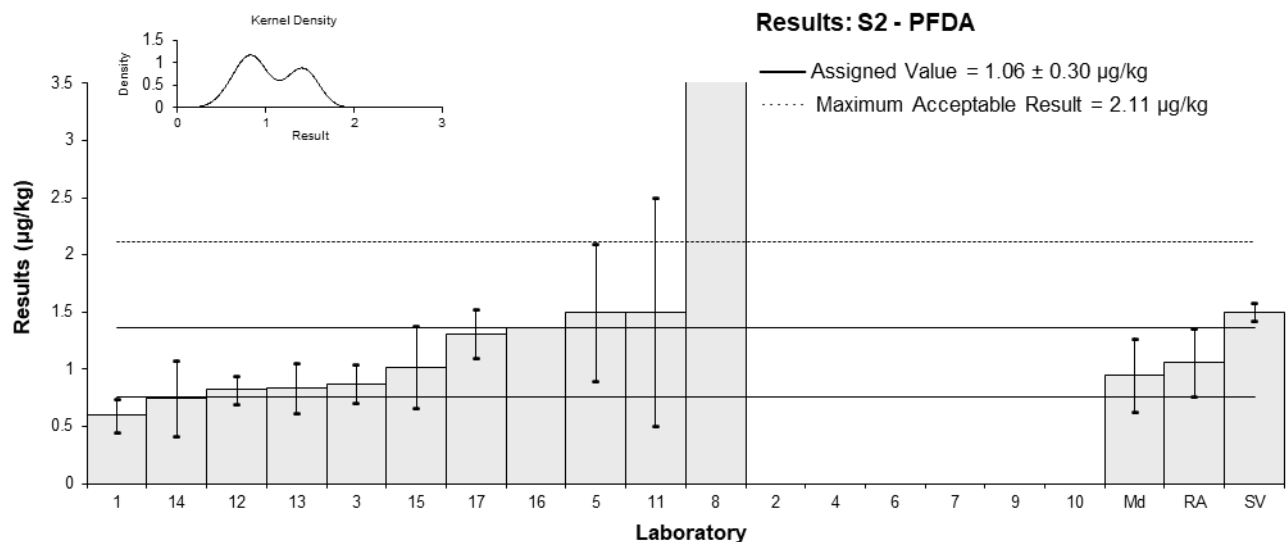


Figure 8

Table 12

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	PFD _o A
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E _n
1	4.66	1	19.8	-2.16	-1.58
2	NS	NS	NS		
3	8.99	1.56	9	0.48	0.31
4	NR	NR	NR		
5*	3.9	2.7	104	-2.62	-1.28
6	7	1.4	NR	-0.73	-0.49
7	<10	NR	NR		
8**	7195.12	NR	NR	4,382.27	3,593.46
9	NR	NR	NR		
10	NS	NS	NS		
11	12	4	122	2.00▼	
12	8.3	1.2	69	0.06	0.04
13	6.55	1.5065	72.7	-1.01	-0.66
14*	16.5	7.42	NR	2.00▼	
15	8.84	3.98	64	0.39	0.14
16	7.13	NR	5	-0.65	-0.53
17	10.2	1.28	65	1.22	0.84

* Outlier, ** Extreme Outlier, see Section 4.2; ▼ Adjusted Score, see Section 6.3

Statistics

Assigned Value	8.2	2.0
Spike Value	15.0	0.8
Homogeneity Value	7.3	1.1
Robust Average	8.2	2.4
Max Acceptable Result	21.1	
Median	8.3	2.0
Mean	8.6	
N	11	
Max	16.5	
Min	3.9	
Robust SD	3.2	
Robust CV	39%	

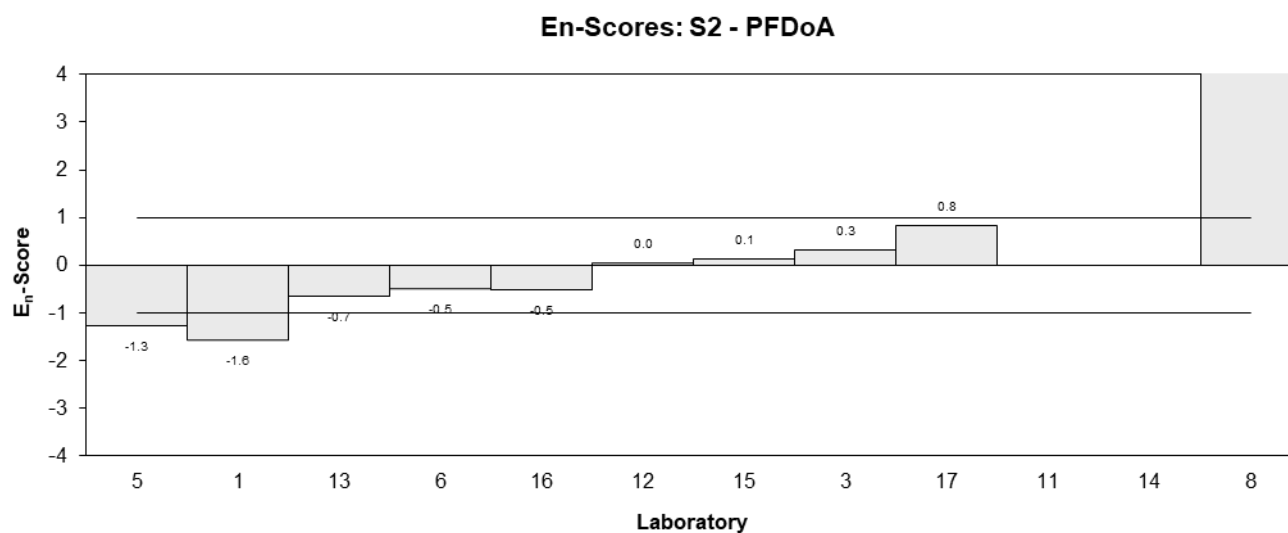
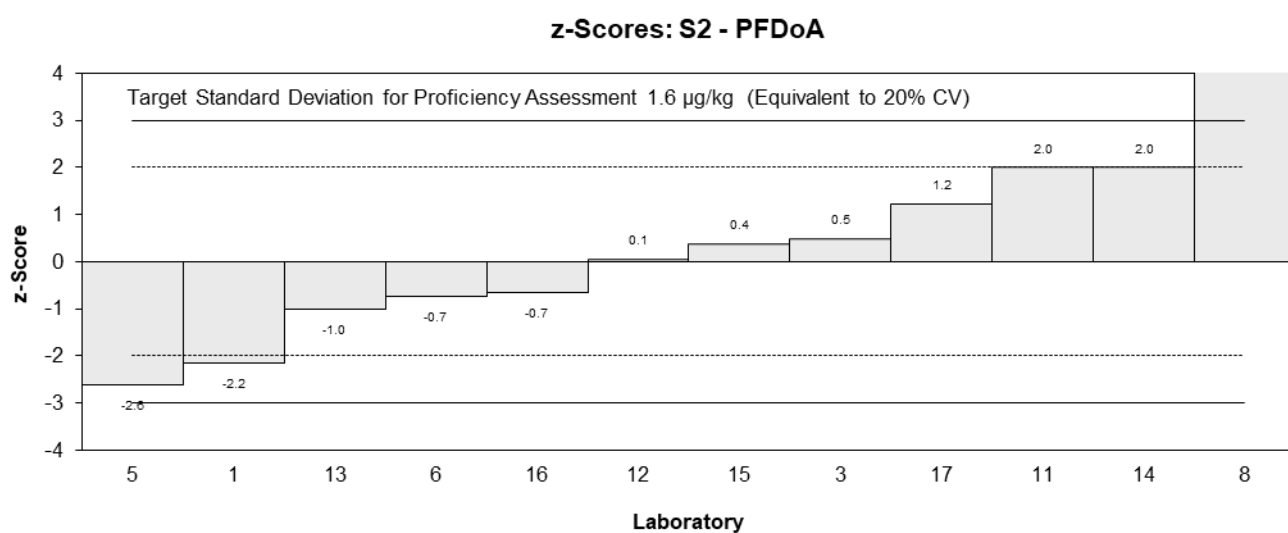
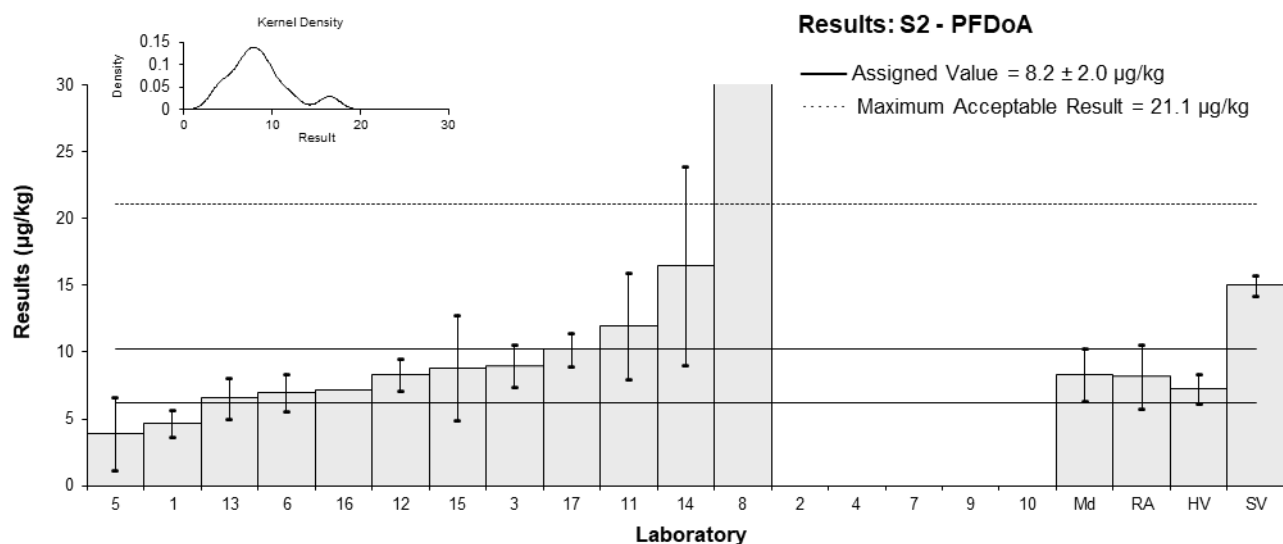


Figure 9

Table 13

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	PFBS
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	2.1	0.427	54.7	-2.15	-2.00
2	NS	NS	NS		
3	2.96	0.613	43	-0.99	-0.80
4	NR	NR	NR		
5	4.3	NR	69	0.83	0.91
6	4.3	0.86	NR	0.83	0.56
7	<10	NR	NR		
8**	4146.26	NR	NR	5,613.24	6,182.94
9	NR	NR	NR		
10	NS	NS	NS		
11	4.5	2	90	1.10	0.38
12	3.7	0.56	148	0.01	0.01
13	2.89	0.7514	90.6	-1.08	-0.79
14	3.9	1.755	NR	0.28	0.11
15	3.39	1.02	71	-0.41	-0.25
16*	1.19	NR	38	-3.39	-3.73
17	4.50	1.28	82	1.10	0.56

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	3.69	0.67
Spike Value	4.12	0.20
Homogeneity Value	3.52	0.53
Robust Average	3.50	0.79
Median	3.70	0.83
Mean	3.43	
N	11	
Max	4.5	
Min	1.19	
Robust SD	1.1	
Robust CV	30%	

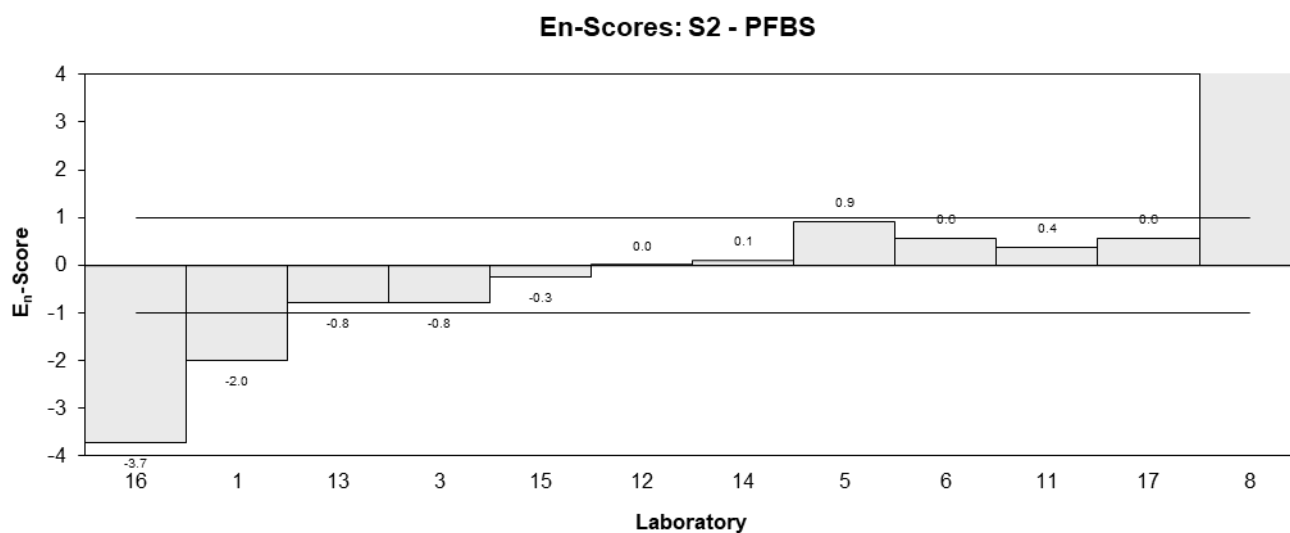
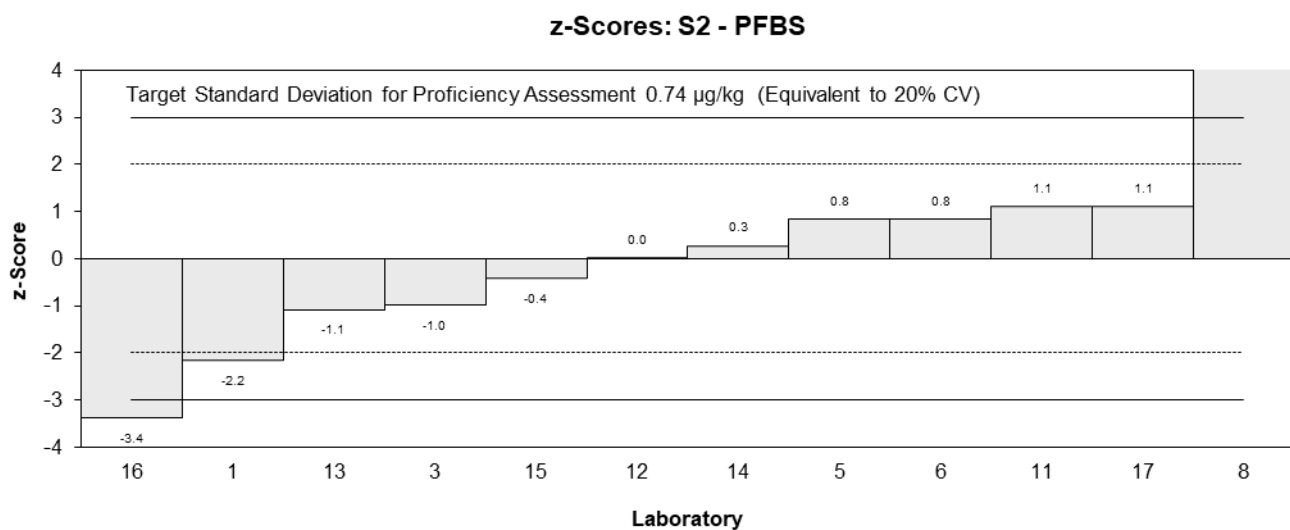
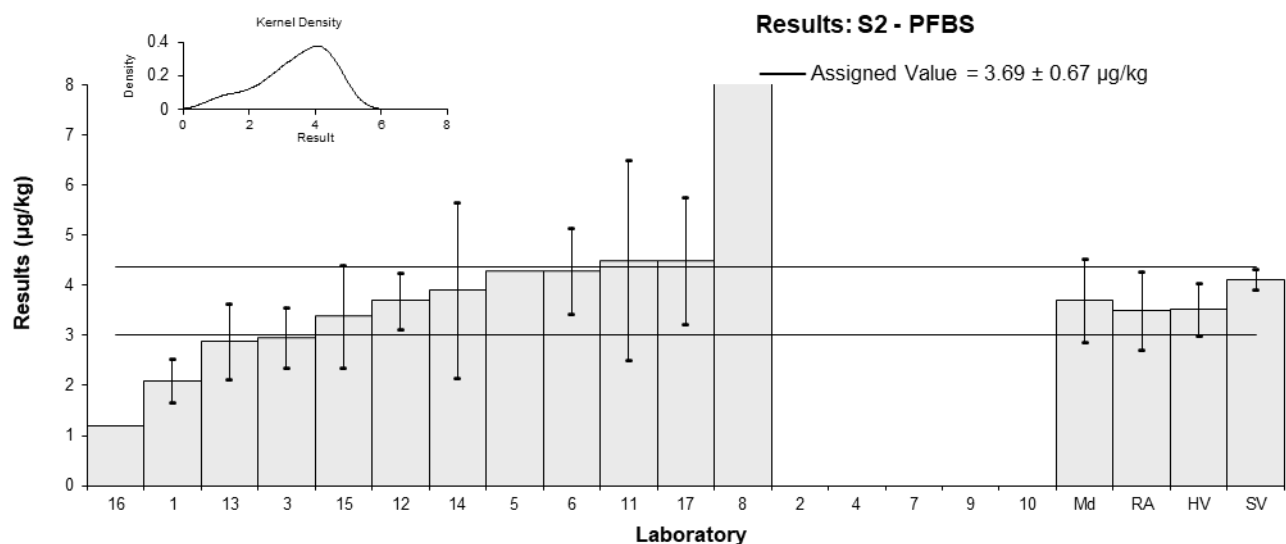


Figure 10

Table 14

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	PFHxS
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	NT	NT	NT		
2	NS	NS	NS		
3	44.9	10	24	-1.26	-1.07
4	NR	NR	NR		
5	69	NR	96	0.75	0.90
6	73	15	NR	1.08	0.72
7	81	22	109	1.75	0.87
8**	37365.19	NR	NR	3,108.77	3,730.52
9	52	12	NR	-0.67	-0.51
10	NS	NS	NS		
11	68	20	93	0.67	0.36
12	53	8.0	182	-0.58	-0.55
13	41.1	10.275	91.2	-1.58	-1.32
14	56	25.2	NR	-0.33	-0.15
15	55.1	16.5	58	-0.41	-0.25
16*	97.845	NR	34	3.15	3.78
17	63.6	NR	NR	0.30	0.36

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	60	10
Spike Value	60.1	3.0
Homogeneity Value	51.0	7.7
Robust Average	62	11
Median	59.8	9.3
Mean	62.9	
N	12	
Max	97.845	
Min	41.1	
Robust SD	16	
Robust CV	25%	

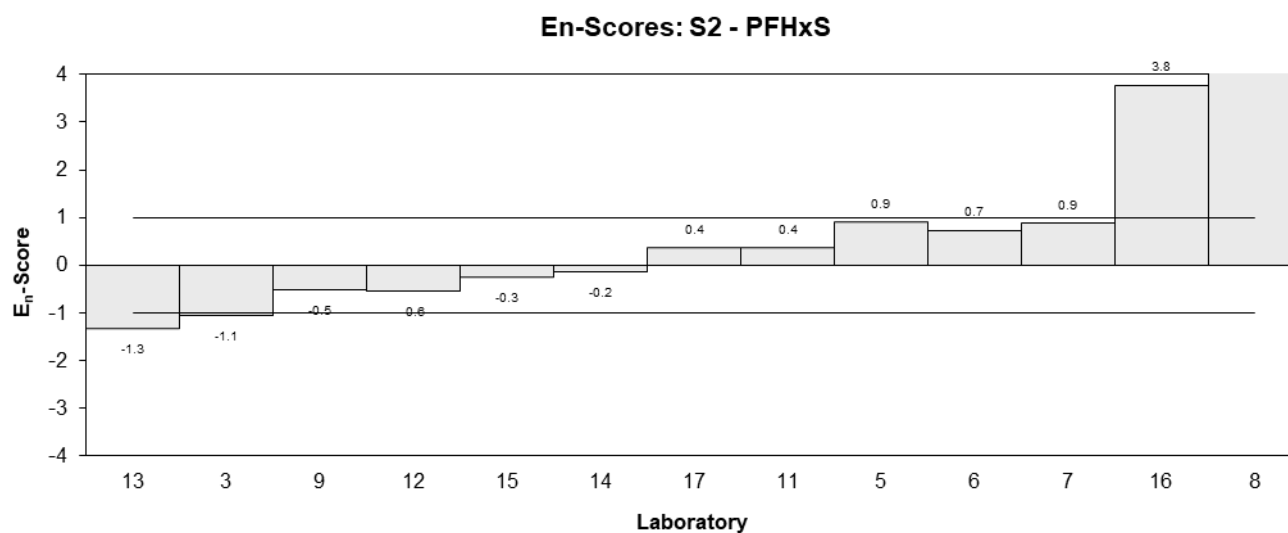
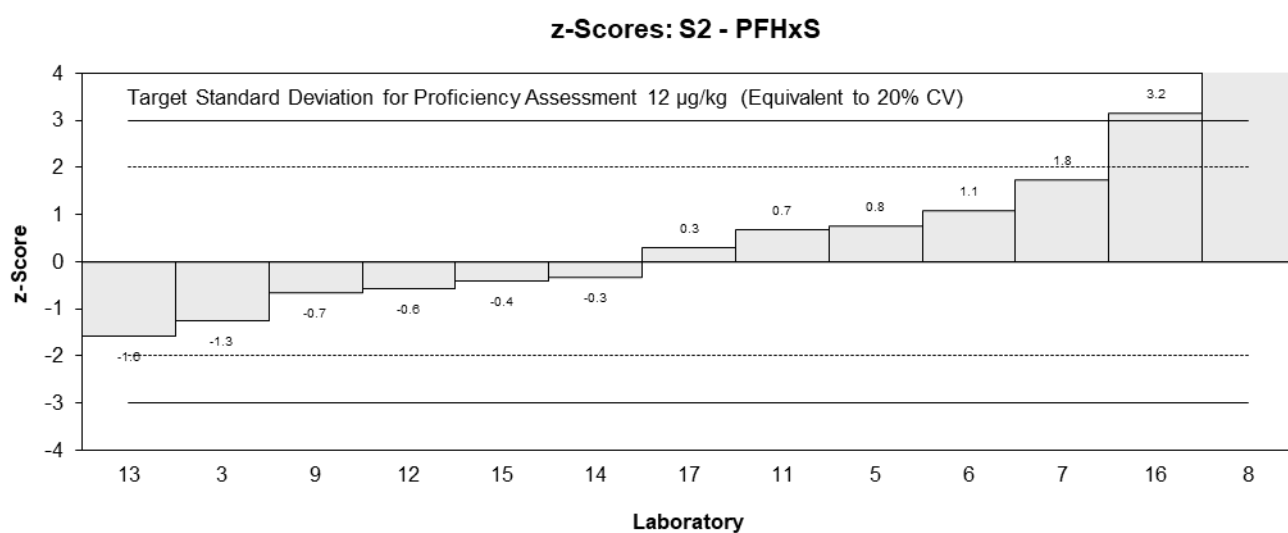
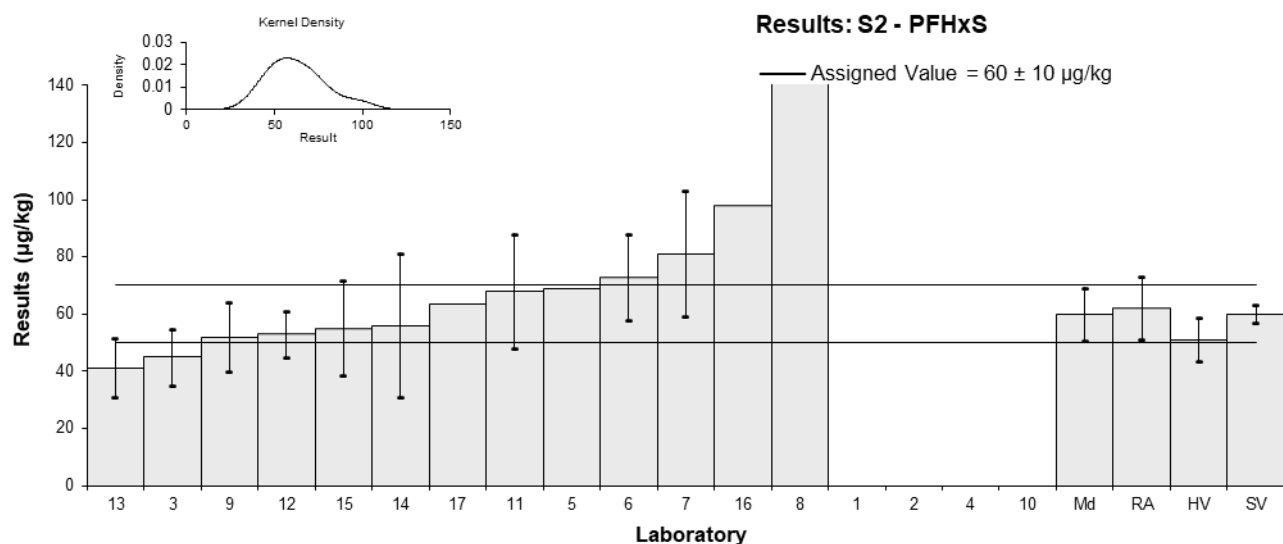


Figure 11

Table 15

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	PFHxS_L
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	47.4	13.8	54.7	-1.11	-0.77
2	NS	NS	NS		
3	43.7	10.8	24	-1.42	-1.12
4	NR	NR	NR		
5	69	NR	96	0.66	0.73
6	73	15	NR	0.98	0.65
7	81	NR	NR	1.64	1.82
8	NT	NT	NT		
9	NR	NR	NR		
10	NS	NS	NS		
11	68	20	93	0.57	0.31
12	53	8.0	NR	-0.66	-0.59
13	NT	NT	NT		
14	56	25.2	NR	-0.41	-0.18
15	55.1	16.5	58	-0.48	-0.30
16	NT	NT	NT		
17	62.8	10.9	89	0.15	0.12

Statistics

Assigned Value	61	11
Spike Value	60.1	3.0
Homogeneity Value	51.0	7.7
Robust Average	61	11
Median	59	11
Mean	60.9	
N	10	
Max	81	
Min	43.7	
Robust SD	13	
Robust CV	22%	

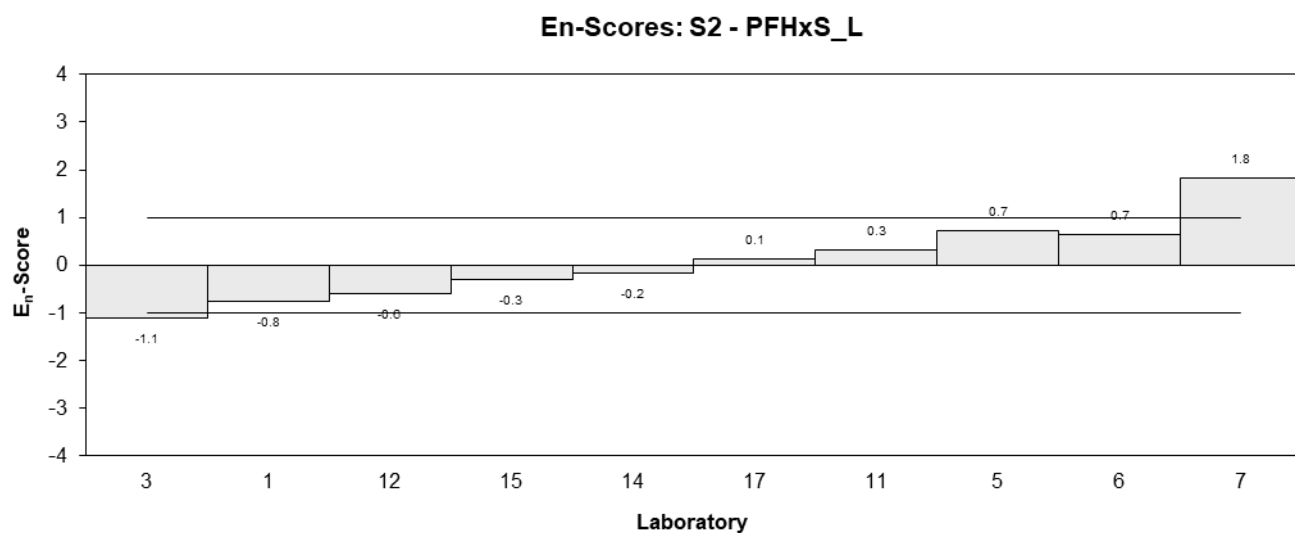
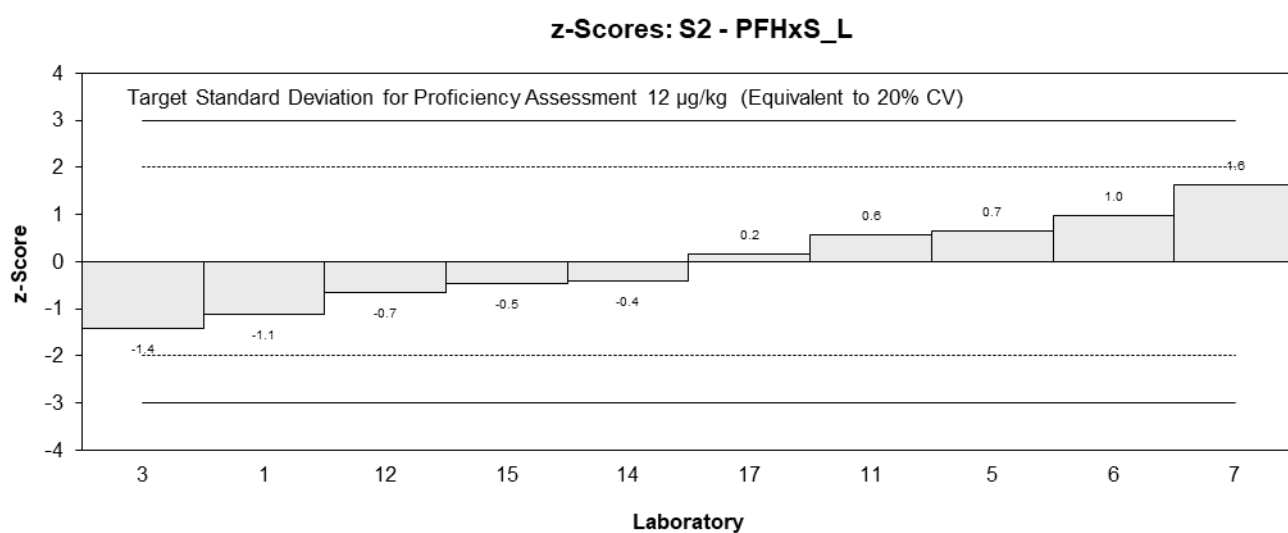
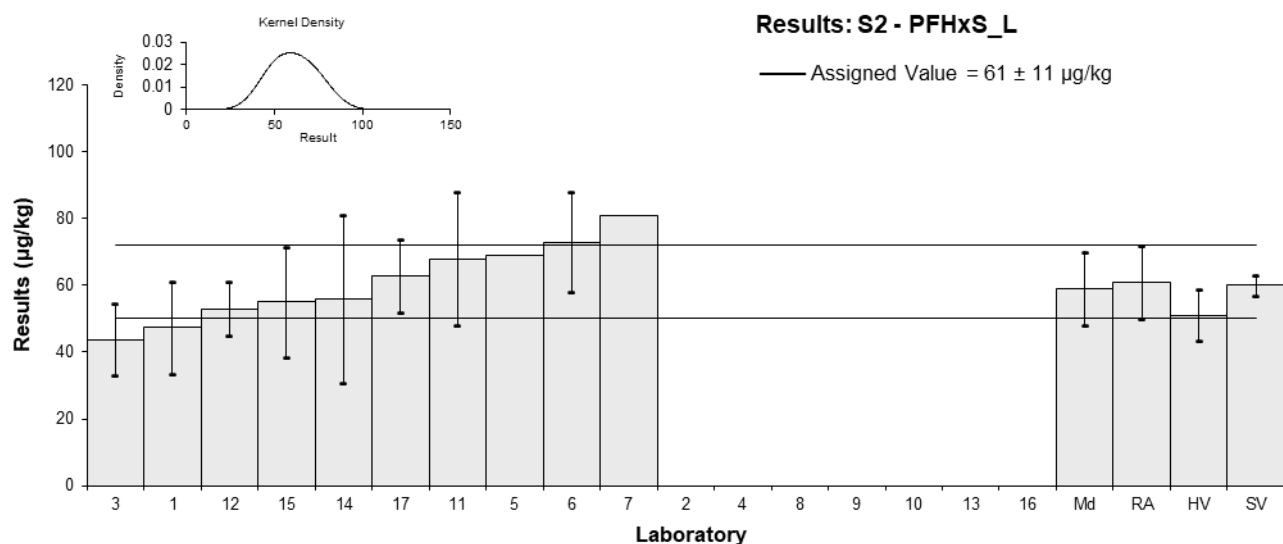


Figure 12

Table 16

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	PFHpS
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	1.86	0.445	54.7	-1.40	-1.00
2	NS	NS	NS		
3	2.79	1.21	24	0.41	0.16
4	NR	NR	NR		
5	3.0	NR	NR	0.81	0.74
6*	4.1	0.82	NR	2.95	1.52
7	<10	NR	NR		
8**	1081.61	NR	NR	2,091.14	1,893.04
9	NR	NR	NR		
10	NS	NS	NS		
11	3.1	1	93	1.01	0.45
12	2.1	0.34	NR	-0.93	-0.72
13	1.59	0.4134	91.1	-1.92	-1.41
14*	1.2	0.52	NR	-2.67	-1.79
15	2.73	1.09	58	0.29	0.12
16	3.393	NR	37	1.58	1.43
17	2.70	0.534	75	0.23	0.15

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	2.58	0.57
Spike Value	Not Spiked	
Homogeneity Value	1.97	0.30
Robust Average	2.61	0.70
Median	2.73	0.70
Mean	2.60	
N	11	
Max	4.1	
Min	1.2	
Robust SD	0.93	
Robust CV	36%	

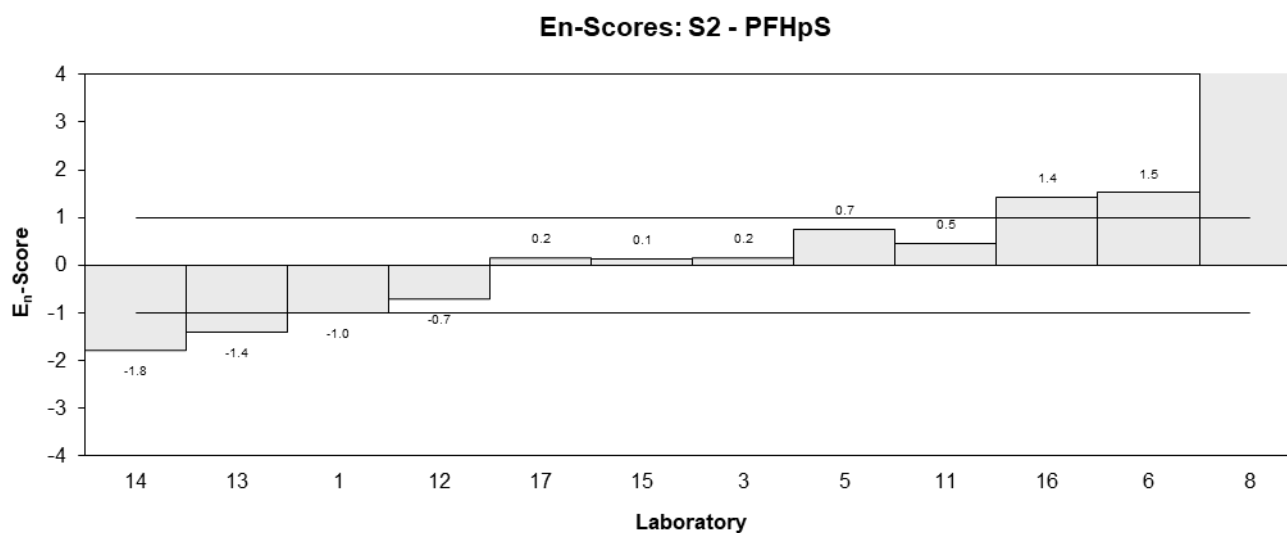
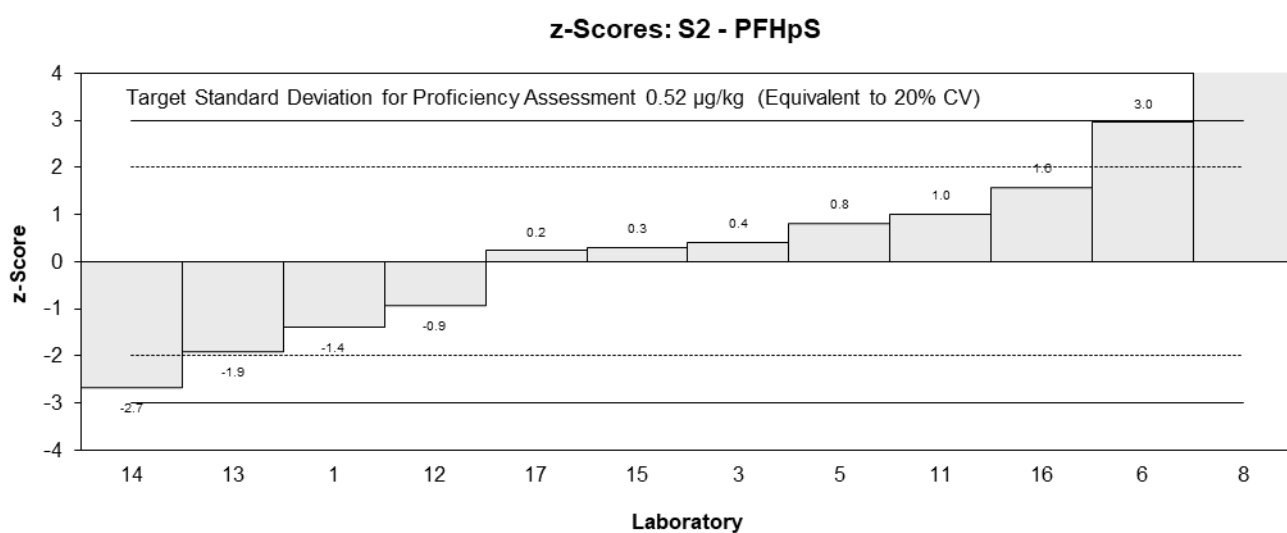
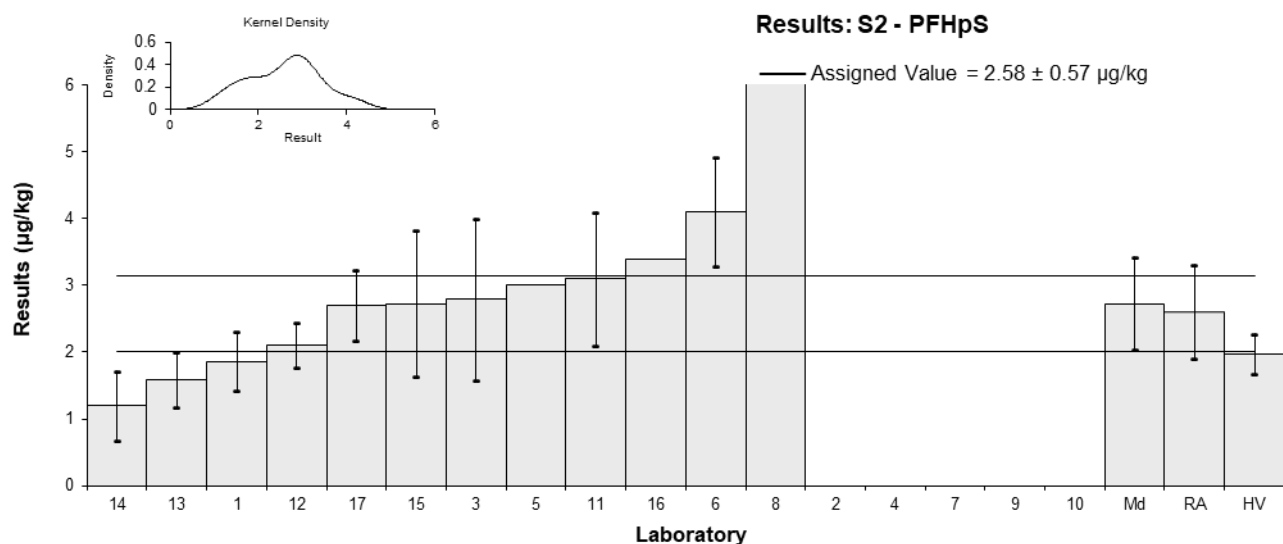


Figure 13

Table 17

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	PFOS
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	13.3	4.58	73.7	-0.87	-0.54
2	NS	NS	NS		
3	13.1	0.333	32	-0.93	-1.19
4	NR	NR	NR		
5	20	10	110	1.21	0.38
6	14	2.8	NR	-0.65	-0.56
7	20	18	117	1.21	0.21
8**	12801.22	NR	NR	3,970.53	5,114.05
9	17	4	NR	0.28	0.19
10	NS	NS	NS		
11	21	7	89	1.52	0.66
12	13	2.0	139	-0.96	-0.97
13	11.9	2.856	91.1	-1.30	-1.11
14	14.6	6.57	NR	-0.47	-0.21
15	15.6	4.67	81	-0.16	-0.09
16	16.76	NR	37	0.20	0.26
17	19.3	NR	NR	0.99	1.28

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	16.1	2.5
Spike Value	14.3	0.7
Homogeneity Value	12.4	1.9
Robust Average	16.1	2.5
Median	15.6	2.6
Mean	16.1	
N	13	
Max	21	
Min	11.9	
Robust SD	3.5	
Robust CV	22%	

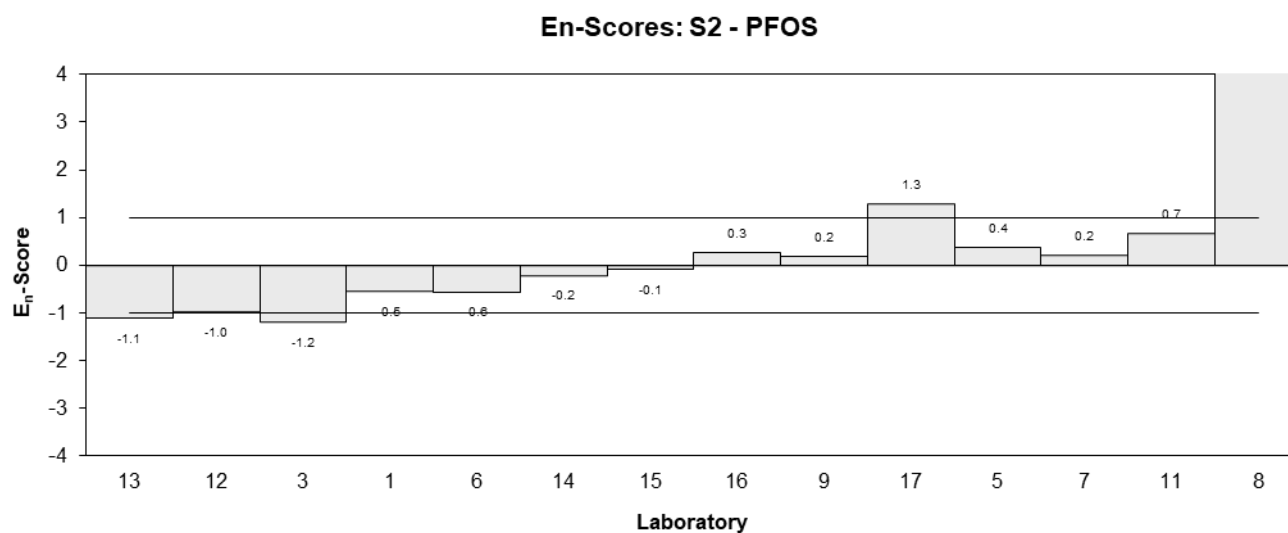
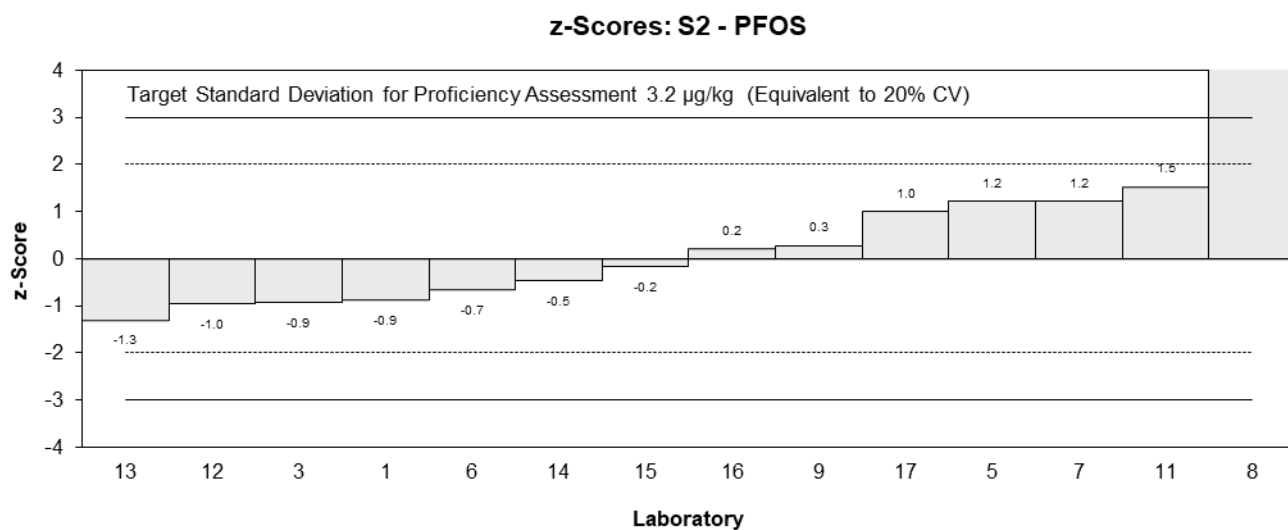
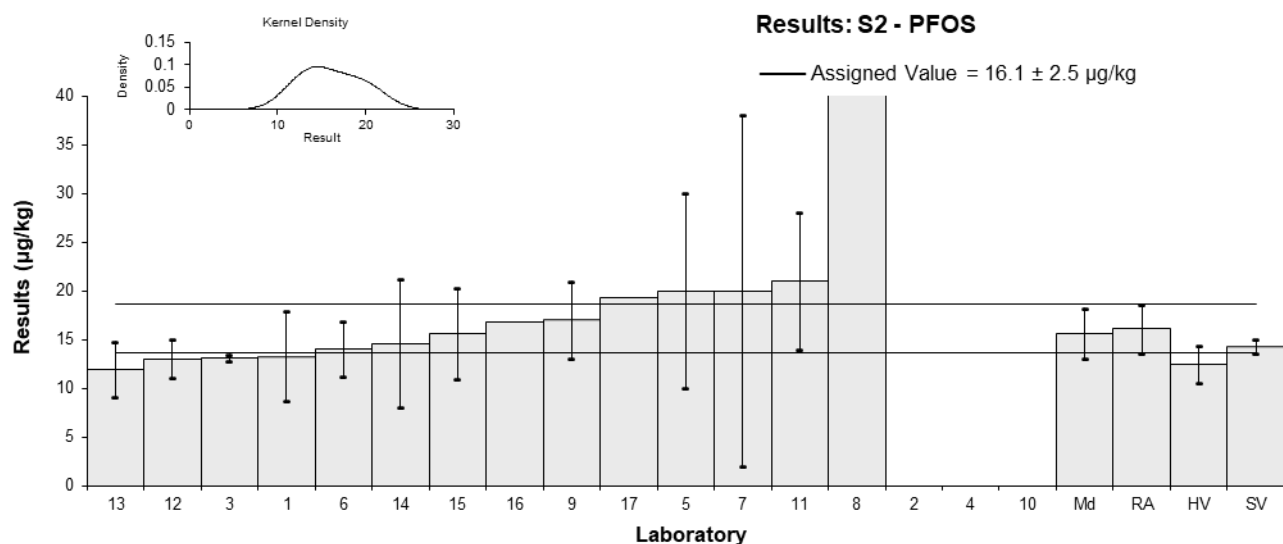


Figure 14

Table 18

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	PFOS_L
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	7.87	2.68	73.7	-1.03	-0.60
2	NS	NS	NS		
3	8.62	1.22	32	-0.65	-0.53
4	NR	NR	NR		
5	14	7	110	2.07	0.56
6	7.3	1.5	NR	-1.31	-1.01
7*	20	NR	NR	5.10	4.81
8**	9279.38	NR	NR	4,681.56	4,414.04
9	NR	NR	NR		
10	NS	NS	NS		
11	12	4	89	1.06	0.46
12	8.9	1.3	NR	-0.51	-0.40
13	NT	NT	NT		
14	8.789	4.3	NR	-0.56	-0.23
15	8.71	2.61	81	-0.60	-0.36
16	9.523	NR	37	-0.19	-0.18
17	13.3	3.93	75	1.72	0.76

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	9.9	2.1
Spike Value	9.66	0.47
Homogeneity Value	8.8	1.3
Robust Average	10.4	2.3
Median	8.9	1.2
Mean	10.8	
N	11	
Max	20	
Min	7.3	
Robust SD	3.0	
Robust CV	29%	

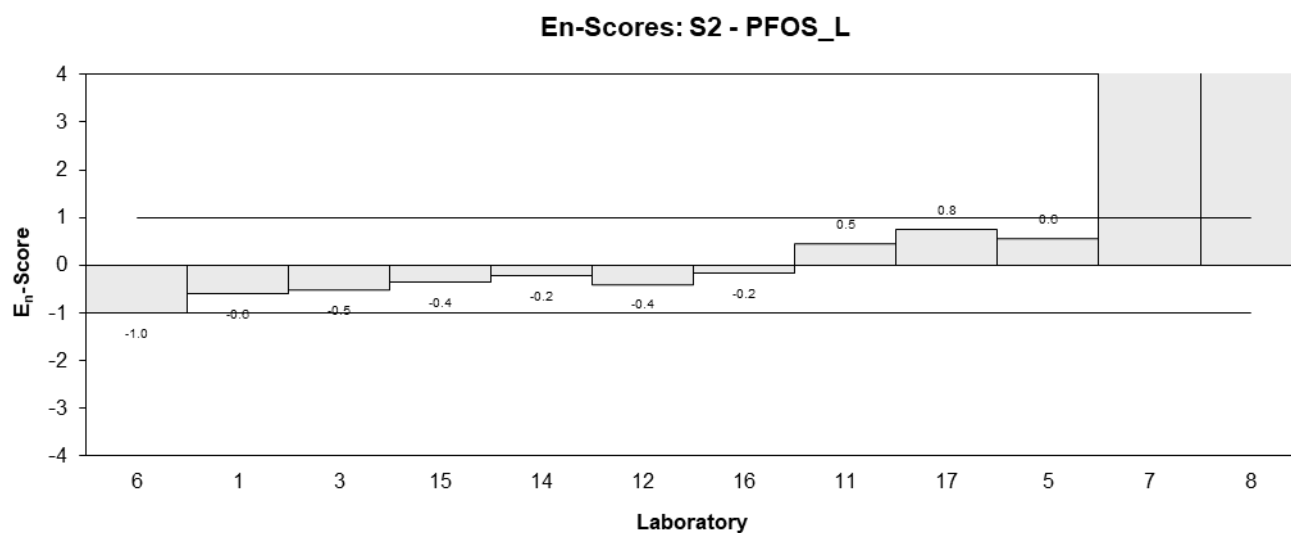
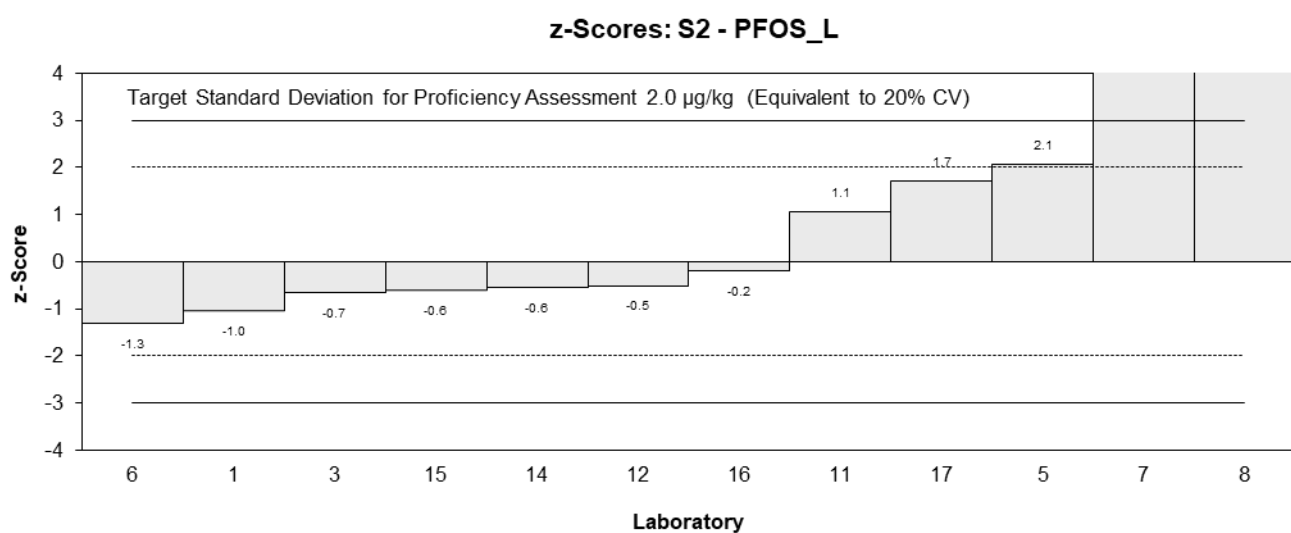
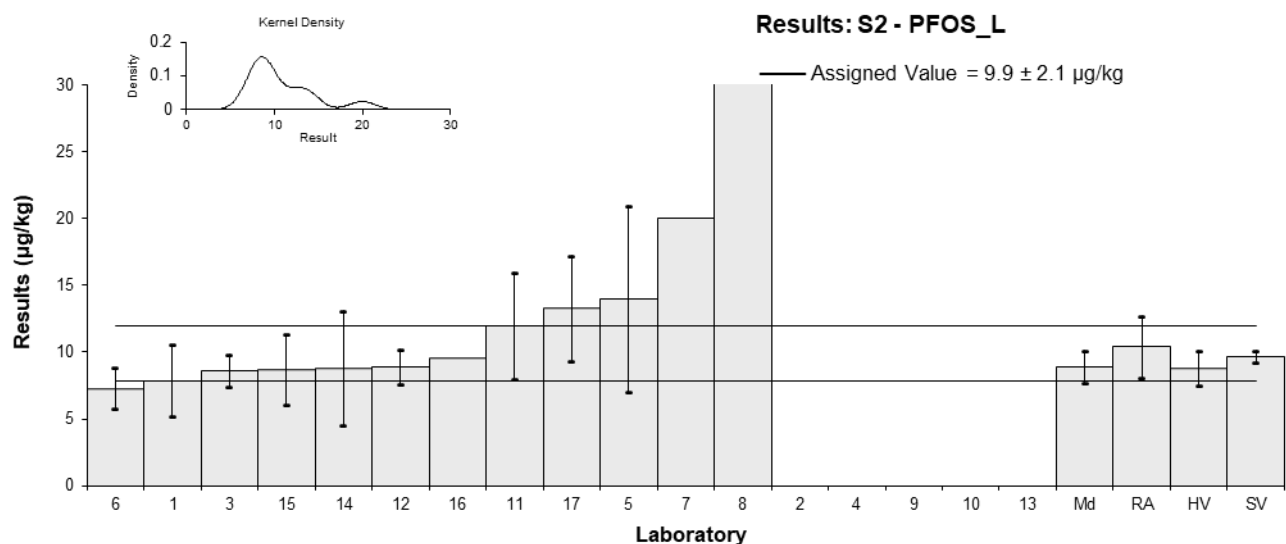


Figure 15

Table 19

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	6:2FTS
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	34.6	26.1	64.4	-1.26	-0.43
2	NS	NS	NS		
3	44.1	4.5	84	-0.24	-0.22
4	NR	NR	NR		
5	51	NR	109	0.51	0.54
6	39	7.8	NR	-0.79	-0.62
7*	200	16	110	16.60	8.44
8**	31029.83	NR	NR	3,345.95	3,561.33
9	NR	NR	NR		
10	NS	NS	NS		
11	63	20	123	1.80	0.77
12	47	11	1504	0.08	0.05
13	32.8	7.872	80.3	-1.46	-1.15
14	73.3	32.98	NR	2.92	0.79
15	43.1	21.6	125	-0.35	-0.14
16	40.208	NR	302	-0.66	-0.70
17	50.5	10.5	130	0.45	0.31

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	46.3	8.7
Spike Value	50.1	2.5
Homogeneity Value	48.3	7.2
Robust Average	49	11
Median	45.6	6.4
Mean	60	
N	12	
Max	200	
Min	32.8	
Robust SD	15	
Robust CV	30%	

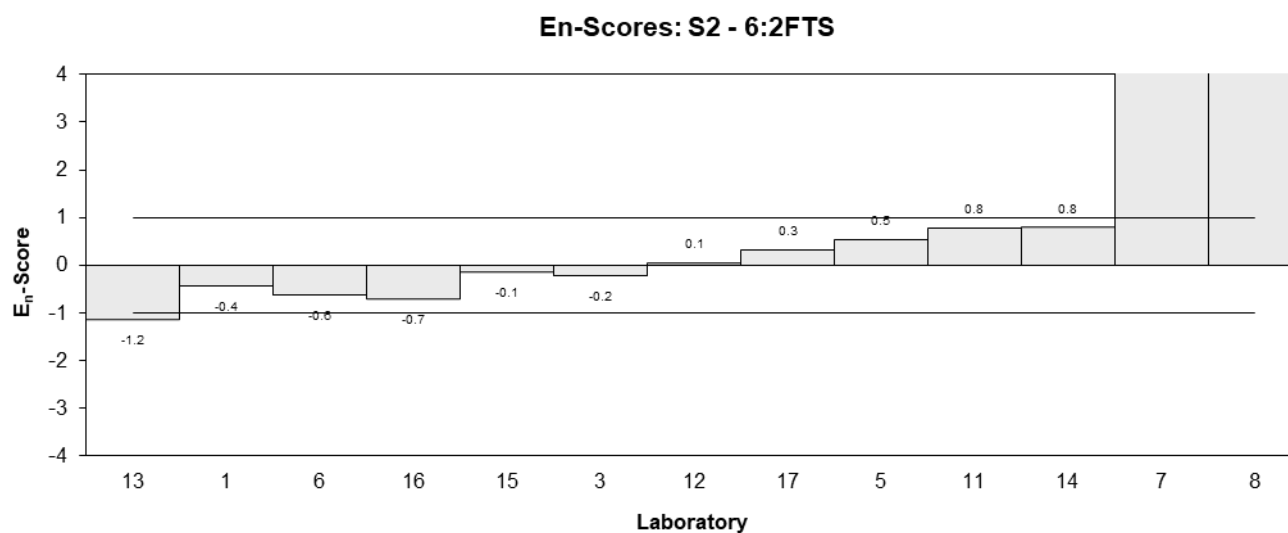
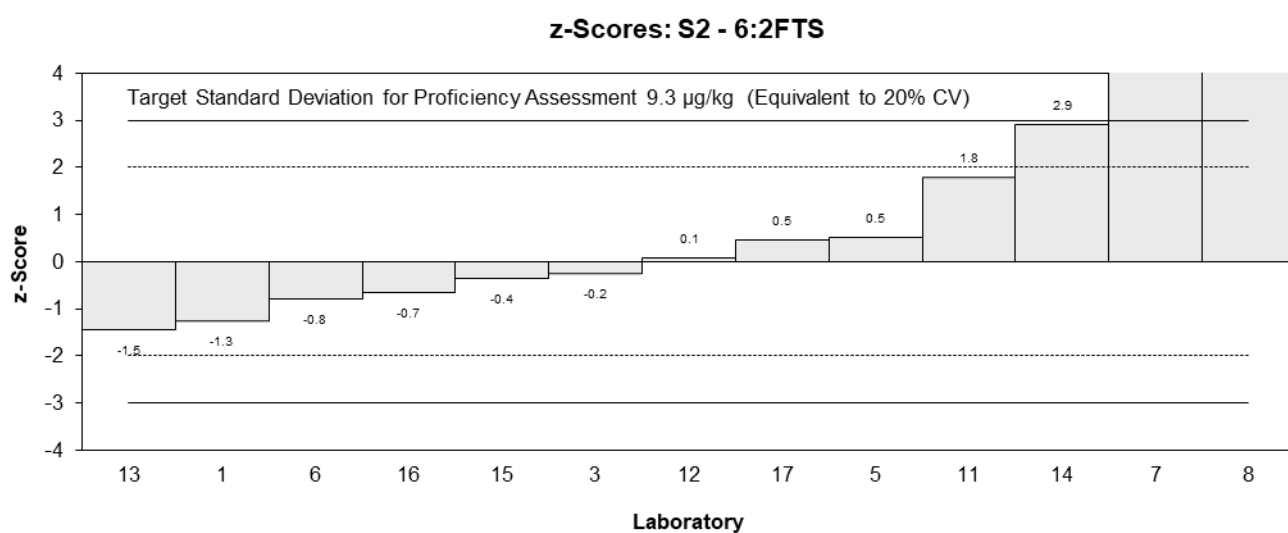
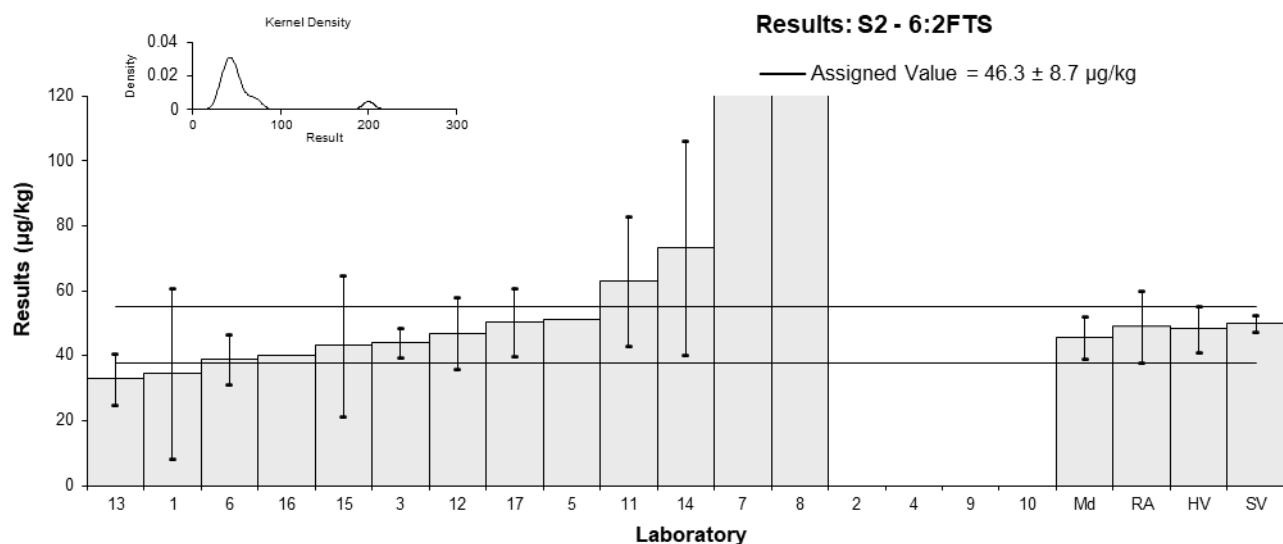


Figure 16

Table 20

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	6:2diPAP
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E _n
1	NT	NT	NT		
2	NS	NS	NS		
3	0.58	0.026	14	-0.92	-0.68
4	NR	NR	NR		
5	0.98	NR	137	1.90	1.42
6	NT	NT	NT		
7	<25	NR	NR		
8**	616.56	NR	NR	4,336.97	3,241.32
9	NR	NR	NR		
10	NS	NS	NS		
11	NT	NT	NT		
12	0.59	0.15	NR	-0.85	-0.50
13	NT	NT	NT		
14	0.7	0.315	NR	-0.07	-0.03
15	NT	NT	NT		
16	0.569	NR	18	-0.99	-0.74
17	0.823	0.203	71	0.80	0.41

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	0.71	0.19
Spike Value	Not Spiked	
Robust Average	0.71	0.19
Median	0.65	0.11
Mean	0.71	
N	6	
Max	0.98	
Min	0.569	
Robust SD	0.19	
Robust CV	27%	

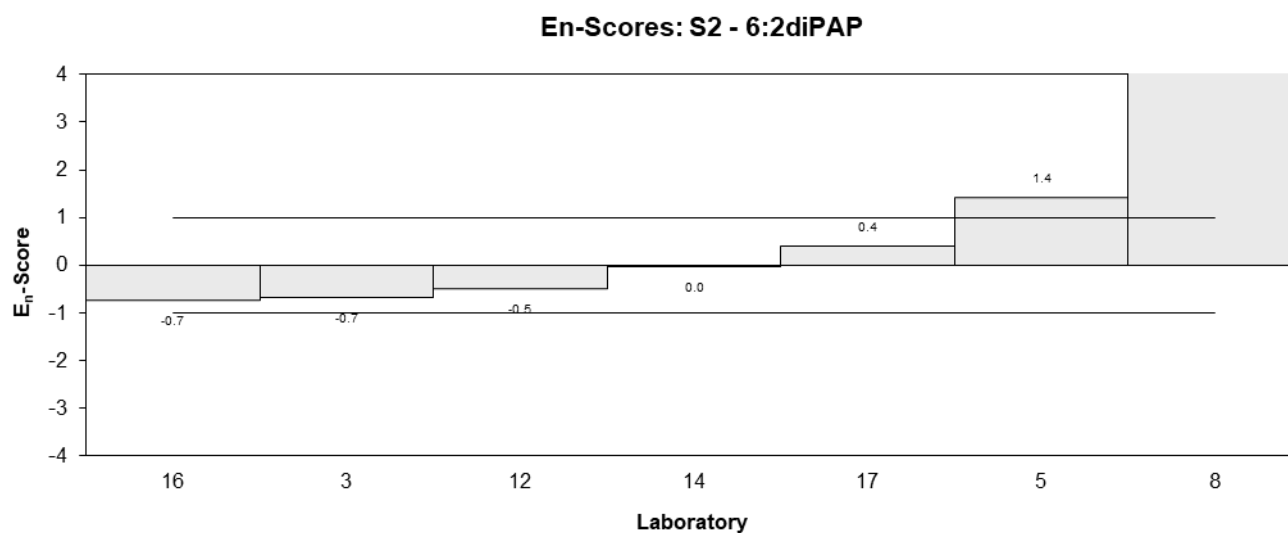
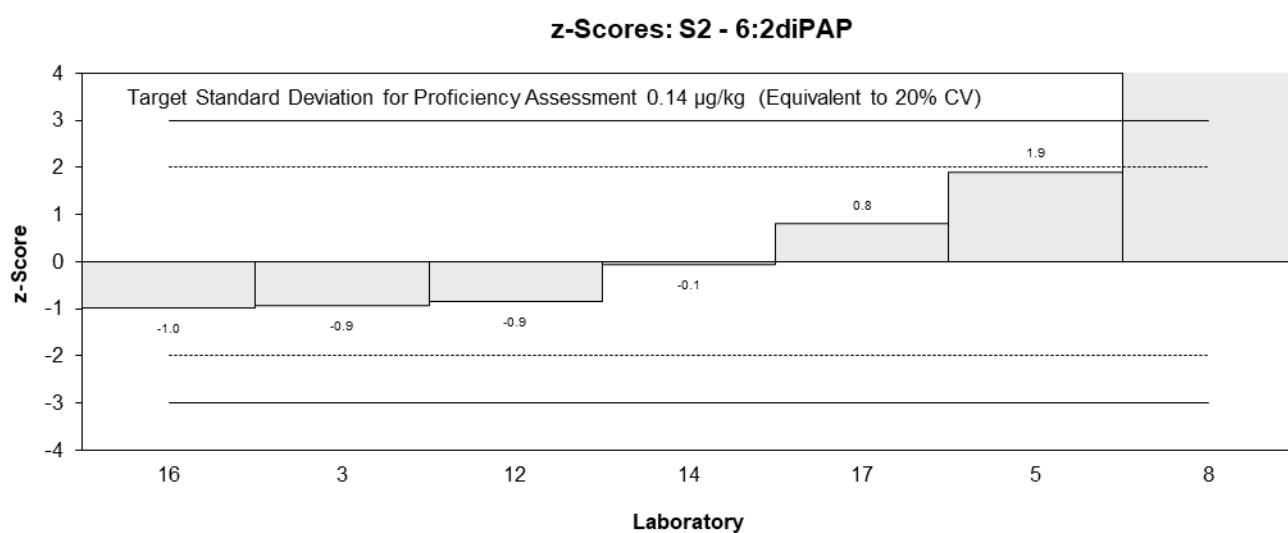
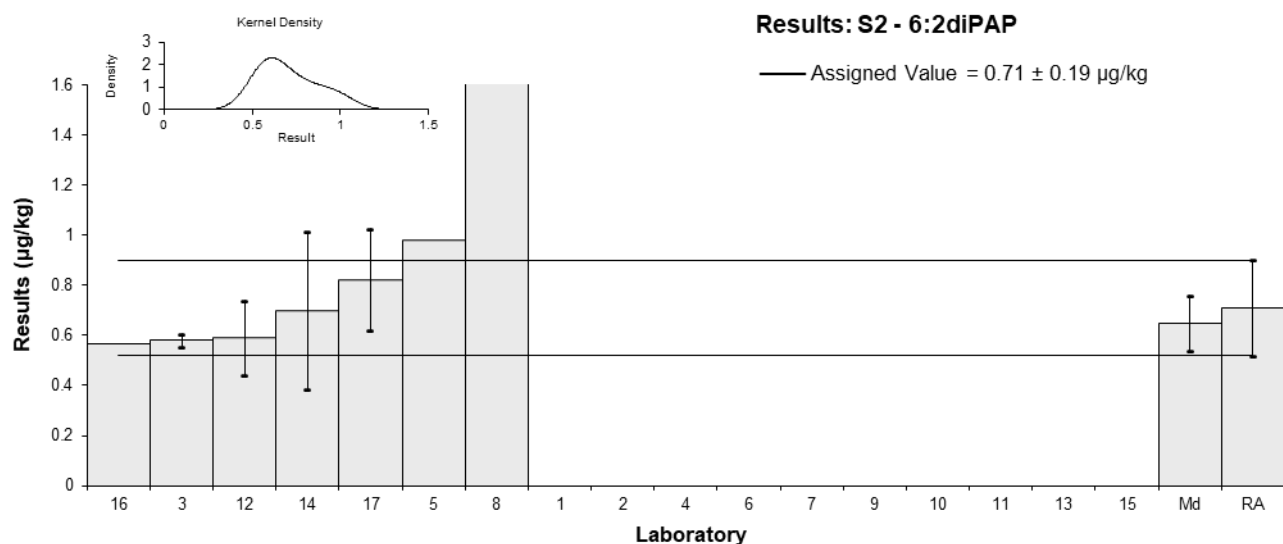


Figure 17

Table 21

Sample Details

Sample No.	S2
Matrix	Cardboard
Analyte	8:2diPAP
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	NT	NT	NT
2	NS	NS	NS
3	0.447	0.11	15
4	NR	NR	NR
5	<0.50	NR	130
6	NT	NT	NT
7	<25	NR	NR
8	NT	NT	NT
9	NR	NR	NR
10	NS	NS	NS
11	NT	NT	NT
12	0.33	0.050	265
13	NT	NT	NT
14	0.43	0.2	NR
15	NT	NT	NT
16	0.535	NR	3
17	0.929	0.280	62

Statistics

Assigned Value	Not Set	0.15
Spike Value	Not Spiked	
Robust Average	NA (N<6)	
Median	0.45	
Mean	0.53	
N	5	
Max	0.929	
Min	0.33	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

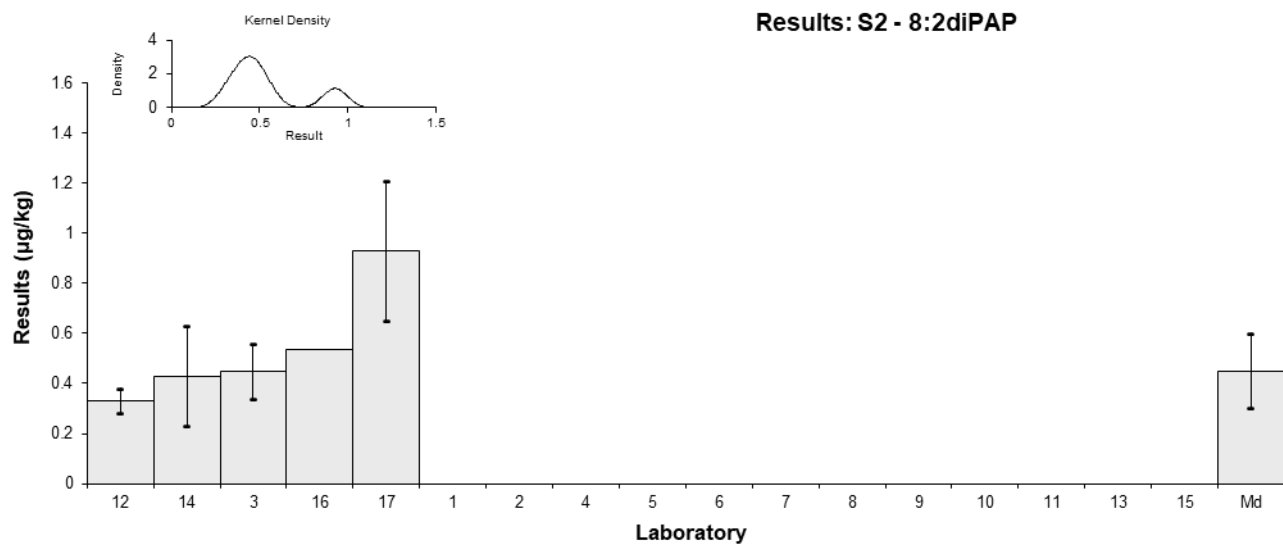


Figure 18

Table 22

Sample Details

Sample No.	S3
Matrix	Plastic
Analyte	Total Fluorine
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty
1	NR	NR
2	<0.2	NR
3	NS	NS
4	9	NR
5	NS	NS
6	NS	NS
7	< 10	2
8	< 5	NR
9	NS	NS
10	<10	2
11	2	1
12	NS	NS
13	<0.140	0.01848
14	1.73	0.692
15	<5.00	NR
16	NS	NS
17	NT	NT

Statistics

Assigned Value	Not Set	NR
Spike Value	Not Spiked	
Homogeneity Value	<10	
Robust Average	NA (N<6)	
Median	2.00	
Mean	4.2	
N	3	
Max	9	
Min	1.73	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

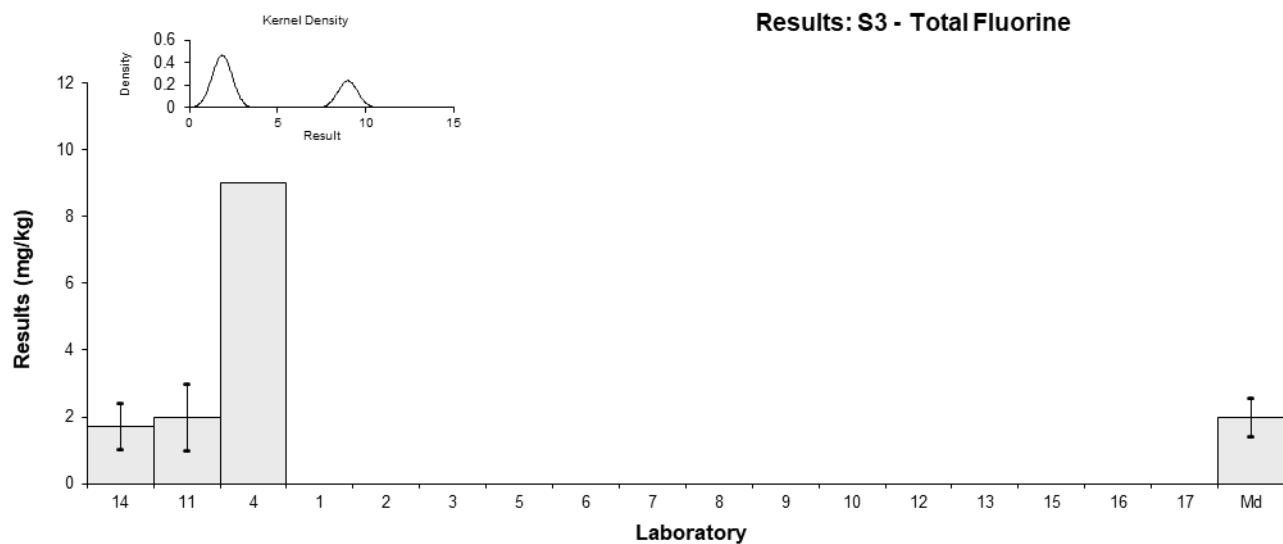


Figure 19

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. Results less than 50% or more than 150% of the robust average were removed before calculation of the assigned value.^{7,9} Appendix 2 sets out the calculation for the expanded uncertainty of the robust average of PFOS in Sample S2.

Laboratory 8 results were consistently higher than the assigned value by a factor of 1000. This discrepancy may be due to an error in the dilution factor calculation, an incorrect calibration curve, or the use of wrong reporting units. These results were excluded from robust average calculations and summary statistics as extreme outlier.

No assigned values were set for PFNA and 8:2diPAP in Sample S2 because there were too few results reported. No assigned value was set for TF in Sample S3 because this was a pilot sample.

A comparison of the assigned value versus spiked value for all fortified analytes in Sample S2 is presented in Table 23. Where applicable the spike value includes the incurred value.

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 23 Comparison of Assigned Value and Spiked Value

Sample	Matrix	Analyte	Spiked Value (µg/kg)	Assigned Value (µg/kg)	Assigned / Spike (%)
S2	Cardboard	PFBA	25.3	22.7	90%
S2	Cardboard	PFPeA	5.98	6.35	106%
S2	Cardboard	PFHxA	264	233	88%
S2	Cardboard	PFOA	20.1	19.2	96%
S2	Cardboard	PFDA	1.50	1.06	71%
S2	Cardboard	PFDoA	15.0	8.2	55%
S2	Cardboard	PFBS	4.12	3.69	90%
S2	Cardboard	PFHxS	60.1	60	100%
S2	Cardboard	PFHxS_L	60.1	61	101%
S2	Cardboard	PFOS	14.3	16.1	113%
S2	Cardboard	PFOS_L	9.66	9.9	102%
S2	Cardboard	6:2FTS	50.1	46.3	92%

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an evaluation of the expanded measurement uncertainty associated with their results. It is a requirement of ISO/IEC 17025 that laboratories have procedures to evaluate the uncertainty of chemical measurements and to report this in specific circumstances, including when the client's instruction so requires.¹¹

Of 189 numerical results, 146 (77%) were reported with an expanded measurement uncertainty. The magnitude of the reported expanded uncertainties was within the range 1.2% to 90% of the reported value. The participants used a wide variety of procedures to evaluate their expanded measurement uncertainties. These are presented in Tables 2 and 3.

Participation in PT programs allows participants to check that their evaluations of measurement uncertainty is reasonable. Results and the expanded MU are presented in the bar charts for each analyte in this study (Figures 2 to 19).

Laboratories 2, 3 and 7 should review their procedure for evaluating measurement uncertainty as some of the relative uncertainties reported by them were lower than 10%, which the study coordinator believes is unrealistically small for a routine PFAS measurement.

Laboratories 1, 5, 7, 11, and 15 who reported relative uncertainties greater than 50% should also review their procedure as it might not be fit-for-purpose.

Laboratories 7, 10, and 13 attached an evaluation of the expanded measurement uncertainty to a result reported as being less than their limit of reporting. An evaluation of uncertainty expressed as a numerical value cannot be attached to a result expressed as a range.¹²

Laboratory 8 reported some results and/or uncertainties with a large number of significant figures. Although all significant figures were used for results assessment (z-score and E_n -score calculation), the last digits were omitted from the tables in Chapter 5, due to lack of space.

In some cases 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 a result of “ $12.808 \pm 2.818 \mu\text{g/kg}$ ”, should instead be expressed as “ $12.8 \pm 2.8 \mu\text{g/kg}$ ”).¹²

6.3 z-Score

The z-score compares the participant’s deviation from the assigned value with the standard deviation for proficiency assessment (SDPA).

A SDPA equivalent to 20% performance coefficient of variation (PCV) was used to calculate z-scores. Unlike the standard deviation based on between-laboratory CV, setting the SDPA as a realistic set value enables z-scores to be used as fixed reference value points for assessment of laboratory performance, independent of group performance.

The between-laboratory coefficient of variation predicted by the modified Horwitz equation⁶ and the between-laboratory CV are presented for comparison in Table 24.

To account for possible bias in the consensus values due to laboratories using inefficient analytical/extraction techniques, z-scores were adjusted for PFHxA, PFDA, and PFDoA in Sample S2.

Where the assigned value is less than 88% of the spiked value, a maximum acceptable result is set to two standard deviations for proficiency assessment more than the spiked level and z-scores greater than 2.0 are adjusted to a value of 2.0. E_n -scores could not be calculated. When the results are higher than the maximum acceptable result, z-scores were not adjusted. This approach ensures that laboratories reporting results close to the spiked value were not penalised. z-Scores of less than 2.0 were left unaltered.

The dispersal of participants’ z-scores is graphically presented by laboratory in Figures 20 and 21 and by analyte in Figure 22.

Of the 175 results for which z-scores were calculated, 144 (82%) returned an acceptable z-score of $|z| \leq 2.0$ and 11 (6%) were questionable with a z-score of $2.0 < |z| < 3.0$. Participants with multiple z-scores larger than 2.0 or smaller than -2.0 should check for method or laboratory bias.

Table 24 Standard Deviation for Proficiency Assessment, Thompson/Horwitz and Between-Laboratory CV

Sample	Analyte	Assigned value (µg/kg)	SPDA (as PCV, %)	Thompson Horwitz CV (%)	Between- laboratory CV* (%)
S1	Total Fluorine	72 (mg/kg)	20	8.4	16
S2	PFBA	22.7	20	22	20
S2	PFPeA	6.35	20	22	18
S2	PFHxA	233	20	20	27
S2	PFOA	19.2	20	22	19
S2	PFNA**	0.161	Not Set	22	15
S2	PFDA	1.06	20	22	36
S2	PFDoA	8.2	20	22	29
S2	PFBS	3.69	20	22	23
S2	PFHxS	60	20	22	23
S2	PFHxS_L	61	20	22	22
S2	PFHpS	2.58	20	22	26
S2	PFOS	16.1	20	22	22
S2	PFOS_L	9.9	20	22	26
S2	6:2FTS	46.3	20	22	25
S2	6:2diPAP	0.71	20	22	27
S2	8:2diPAP**	0.45	Not Set	22	49
S3	Total Fluorine**	2.00 (mg/kg)	Not Set	14	110

*Robust between-laboratory CV with outliers removed; **Median Value (Assigned Value not set).

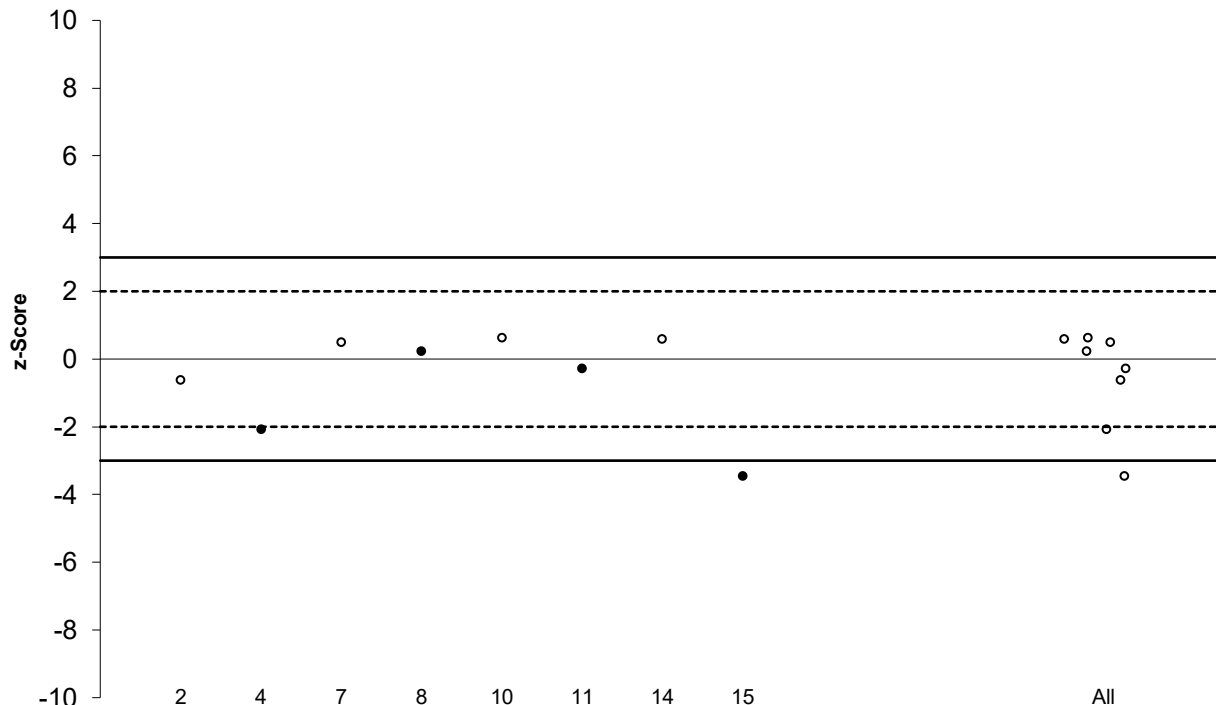
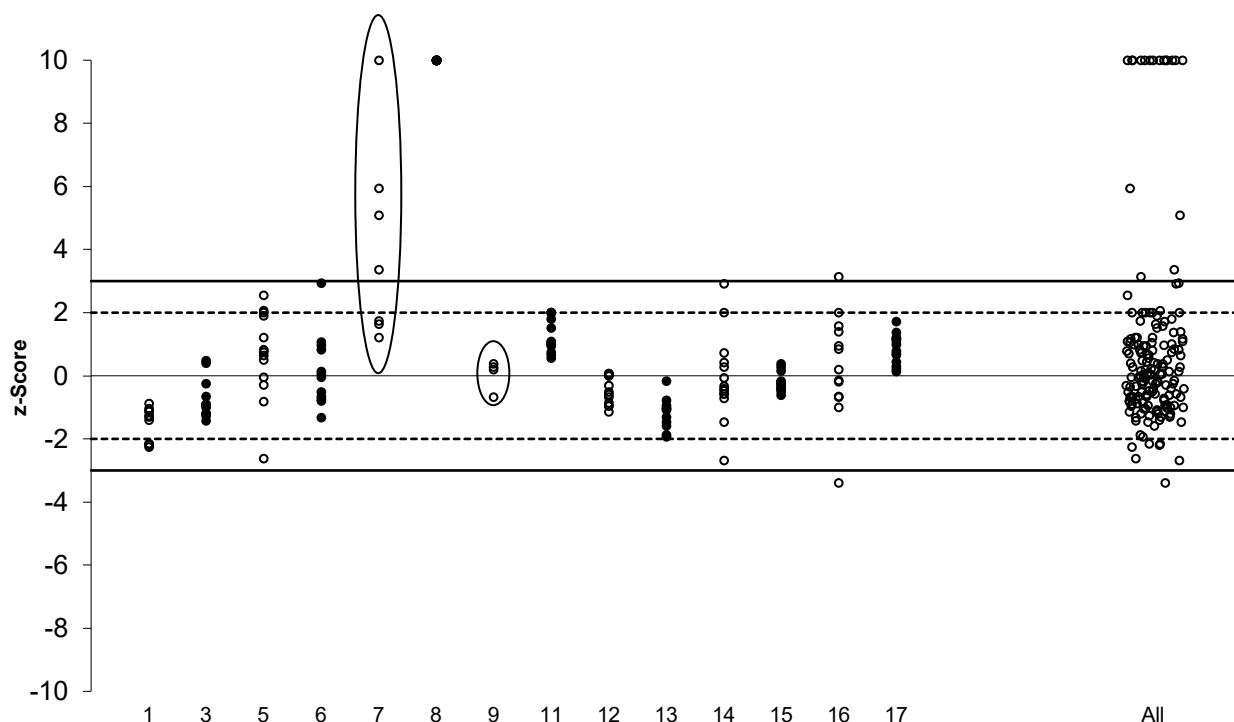
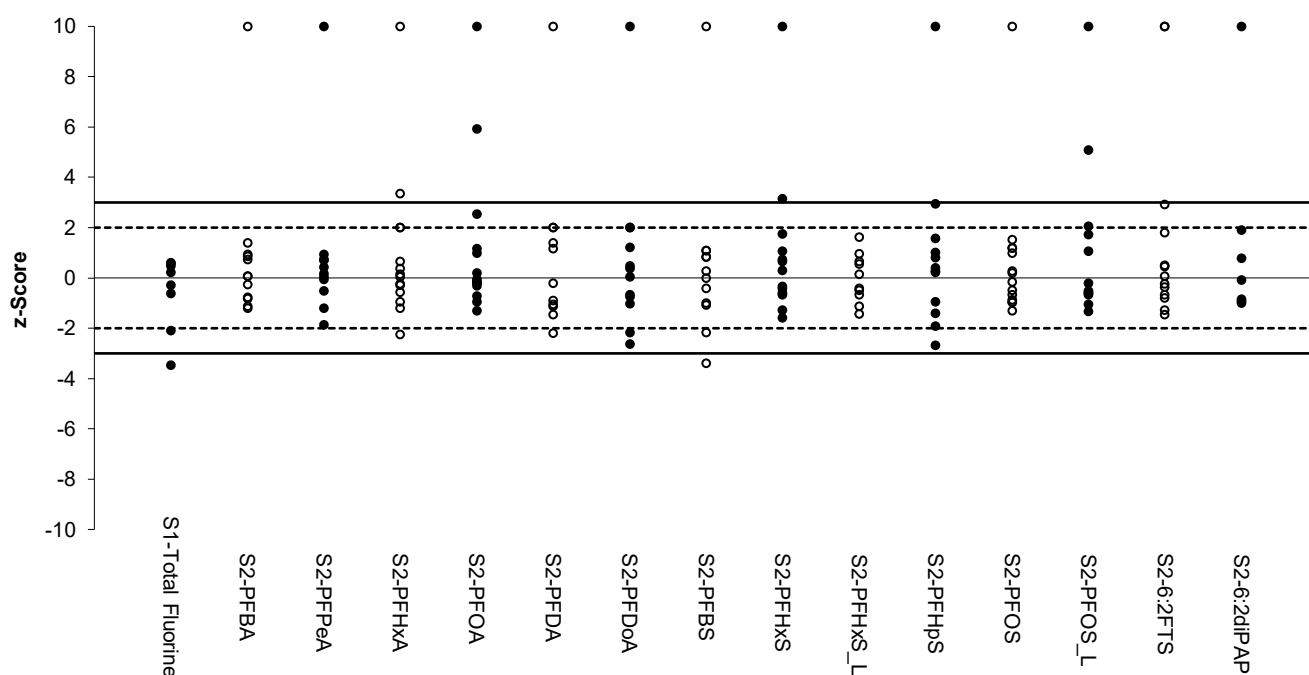


Figure 20 S1-TF z-Score Dispersal by Laboratory



Scores greater than 10 have been plotted as 10. See comments in Subchapter 6.5 for the circled laboratories.

Figure 21 S2-PFAS z-Scores Dispersal by Laboratory



Scores greater than 10 have been plotted as 10.

Figure 22 S2-PFAS z-Scores Dispersal by Analyte

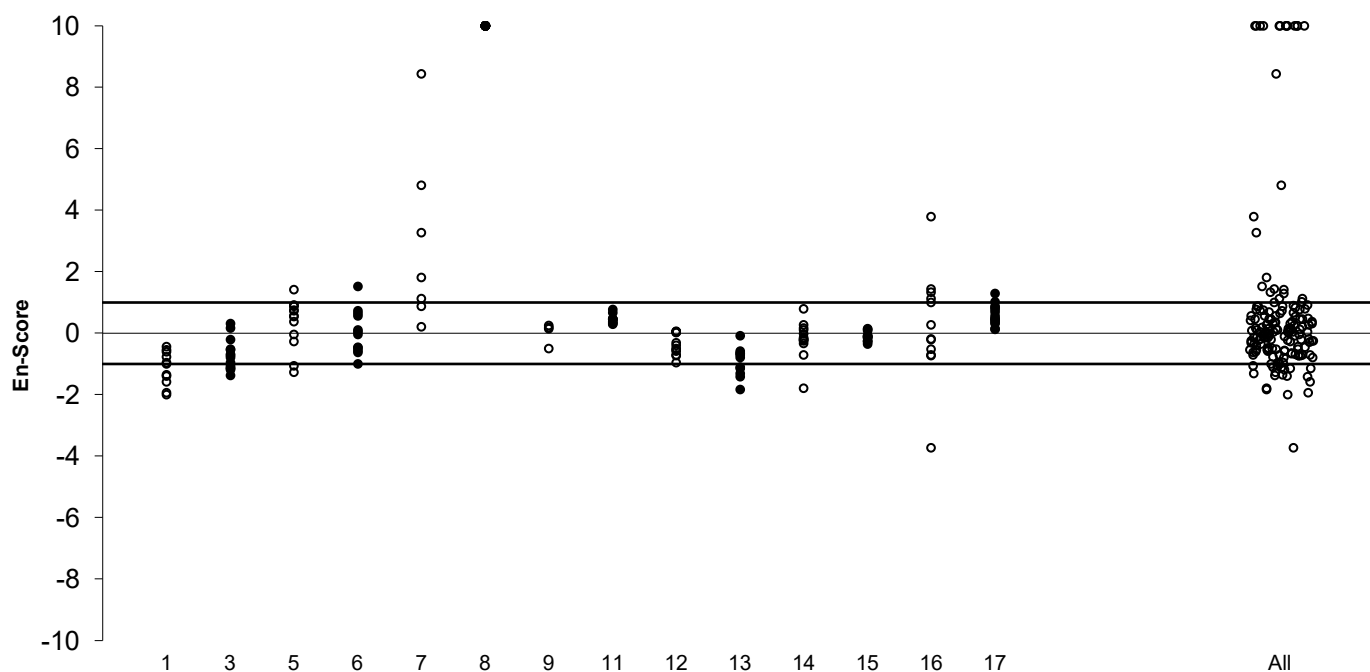
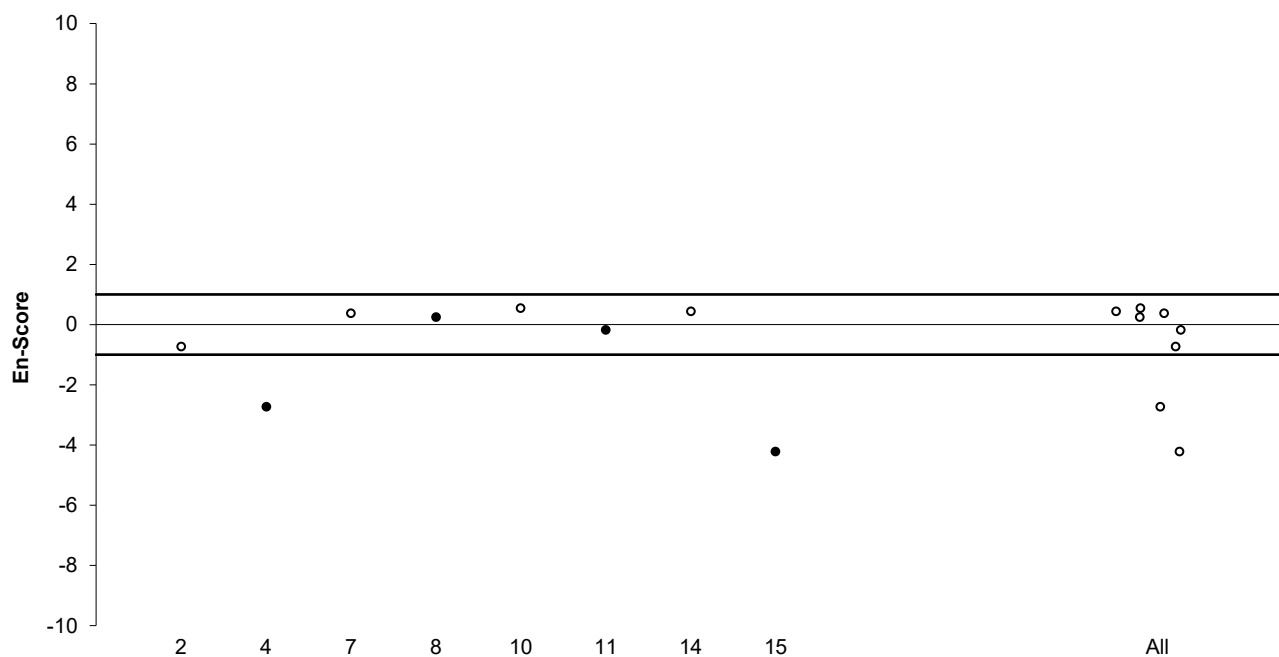
6.4 E_n -Score

E_n -score can be interpreted 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 unacceptable E_n score can either be caused by an inappropriate measurement, an inappropriate evaluation of measurement uncertainty, or both.

The dispersal of participants' E_n -scores is graphically presented in Figures 23 and 24. 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.

For results whose z-scores were adjusted, no E_n -score has been calculated.

Of 169 results for which E_n -scores were calculated, 119 (70%) returned an acceptable score of $|E_n| < 1.0$ indicating agreement of the participants' results with the assigned values within their respective expanded measurement uncertainties.



Scores greater than 10 have been plotted as 10.

Figure 24 S2 PFAS E_n -Scores Dispersal by Laboratory

Table 25 Summary of Participants' Results and Performance for Samples S1, S2, and S3*

Lab. Code	S1-Total Fluorine (mg/kg)	S3-Total Fluorine (mg/kg)	S2-PFBA (µg/kg)	S2-PFPeA (µg/kg)	S2-PFHxA (µg/kg)	S2-PFOA (µg/kg)	S2-PFNA (µg/kg)	S2-PFDA (µg/kg)	S2-PFDoA (µg/kg)
AV	72	Not Set	22.7	6.35	233	19.2	Not Set	1.06	8.2
SV	Not Spiked	Not Spiked	25.3	5.98	264	20.1	Not Spiked	1.50	15.0
HV	87	<10	22.9	5.73	253	18.1	NA	NA	7.3
1	NR	NR	17.656	<0.584	128	14.2	0.139	0.596	4.66
2	63	<0.2	NS	NS	NS	NS	NS	NS	NS
3	NS	NS	17.3	4.84	177.4	15.6	0.161	0.872	8.99
4	42	9	NR	NR	NR	NR	NR	NR	NR
5	NS	NS	19	6.3	220	29	0.19	1.5	3.9
6	NS	NS	23	5.7	240	19	<0.5	<1	7
7	79	< 10	<10	<10	390	42	<10	<10	<10
8	75.5	< 5	15248.61	5694.58	117353.89	14677.70	163.49	855.11	7195.12
9	NS	NS	NR	NR	251	20	NR	NR	NR
10	81	<10	NS	NS	NS	NS	NS	NS	NS
11	68	2	27	7.3	330	23	< 0.5	1.5	12
12	NS	NS	23	6.4	235	18	0.14	0.82	8.3
13	<0.140	<0.140	19.2	3.99	189	18.6	0.162	0.84	6.55
14	80.44	1.73	26	6.9	207	16.5	<0.1	0.75	16.5
15	22.2	<5.00	21.5	6.57	222	18.4	<1	1.02	8.84
16	NS	NS	26.626	7.561	331.12	18.621	<0.2	1.359	7.13
17	NT	NT	29.0	7.27	264	23.7	<0.332	1.31	10.2

* AV = Assigned Value, SV = Spiked Value, NS = Not Sent, NT = Not Tested, NR = Not Reported. Shaded cells are results which returned a questionable or unacceptable z-score.

Table 25 Summary of Participants' Results and Performance for Samples S1, S2, and S3 (continued)*

Lab. Code	S2-PFBS (µg/kg)	S2-PFHxS (µg/kg)	S2-PFHxS_L (µg/kg)	S2-PFHpS (µg/kg)	S2-PFOS (µg/kg)	S2-PFOS_L (µg/kg)	S2-6:2FTS (µg/kg)	S2-6:2diPAP (µg/kg)	S2-8:2diPAP (µg/kg)
AV	3.69	60	61	2.58	16.1	9.9	46.3	0.71	Not Set
SV	4.12	60.1	60.1	Not Spiked	14.3	9.66	50.1	Not Spiked	Not Spiked
HV	3.52	51.0	51.0	1.97	12.4	8.8	48.3	NA	NA
1	2.1	NT	47.4	1.86	13.3	7.87	34.6	NT	NT
2	NS	NS	NS	NS	NS	NS	NS	NS	NS
3	2.96	44.9	43.7	2.79	13.1	8.62	44.1	0.58	0.447
4	NR	NR	NR	NR	NR	NR	NR	NR	NR
5	4.3	69	69	3.0	20	14	51	0.98	<0.50
6	4.3	73	73	4.1	14	7.3	39	NT	NT
7	<10	81	81	<10	20	20	200	<25	<25
8	4146.26	37365.19	NT	1081.61	12801.22	9279.38	31029.83	616.56	NT
9	NR	52	NR	NR	17	NR	NR	NR	NR
10	NS	NS	NS	NS	NS	NS	NS	NS	NS
11	4.5	68	68	3.1	21	12	63	NT	NT
12	3.7	53	53	2.1	13	8.9	47	0.59	0.33
13	2.89	41.1	NT	1.59	11.9	NT	32.8	NT	NT
14	3.9	56	56	1.2	14.6	8.789	73.3	0.7	0.43
15	3.39	55.1	55.1	2.73	15.6	8.71	43.1	NT	NT
16	1.19	97.845	NT	3.393	16.76	9.523	40.208	0.569	0.535
17	4.50	63.6	62.8	2.70	19.3	13.3	50.5	0.823	0.929

* AV = Assigned Value, SV = Spiked Value, NS = Not Sent, NT = Not Tested, NR = Not Reported. Shaded cells are results which returned a questionable or unacceptable z-score.

Summary of Participants' Performance in AQA 25-18 Samples S1 and S2

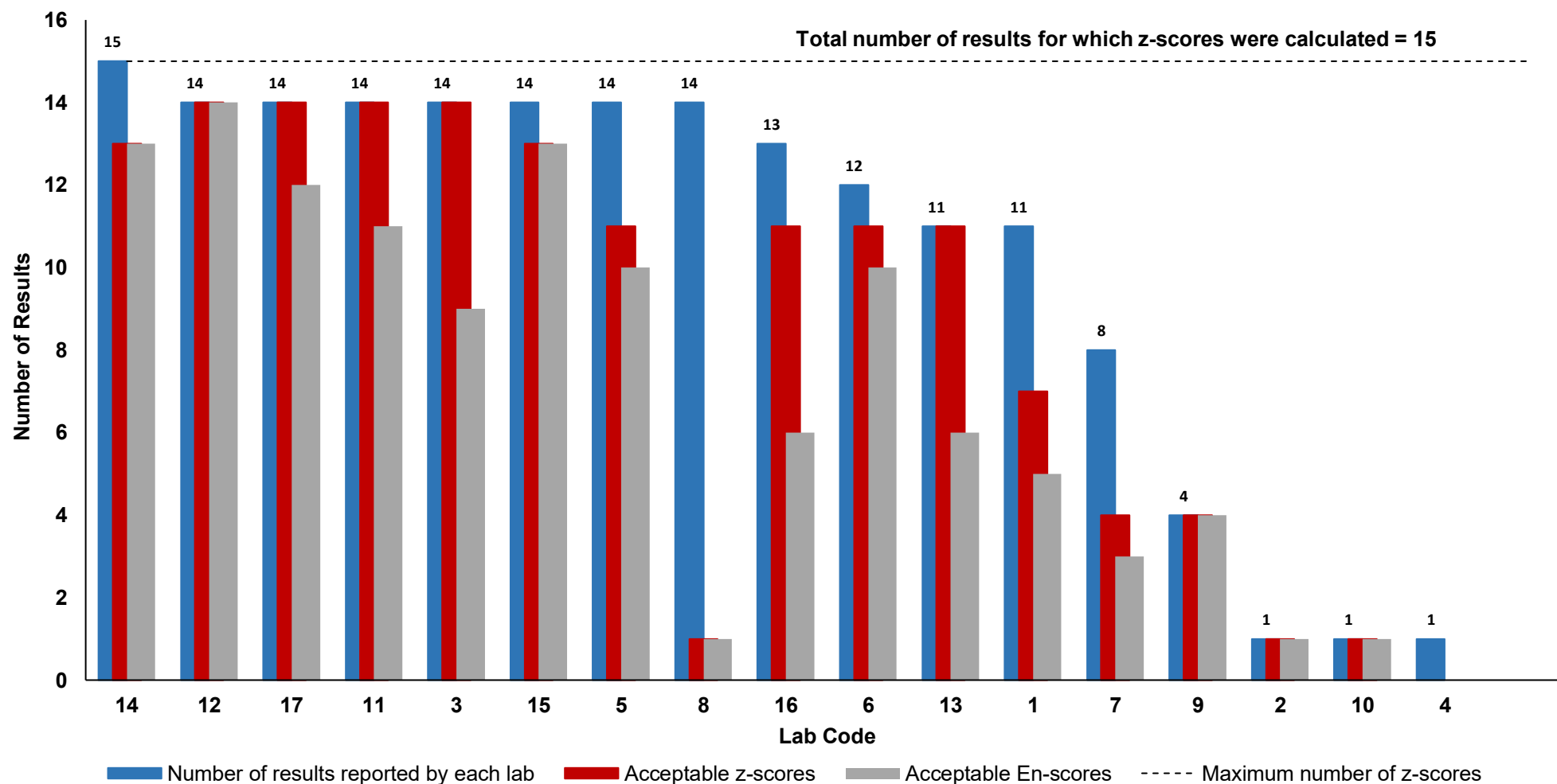


Figure 25 Summary of Participants' Performance in Packaging Material Samples S1 and S2

6.5 Summary of Participants' Results and Performances

Summaries of participants' results and performance for scored analytes in this PT study are presented in Table 25 and Figures 20 to 25.

No laboratory returned acceptable z-scores and E_n -scores for all 15 analytes for which scores were calculated. Laboratory 12 returned acceptable z-scores and E_n -scores for all 14 results they reported.

Eight laboratories reported at least one PFAS analyte that was not spiked into test sample S2. These results are presented in Appendix 3.

All 14 results reported by Laboratories **3**, **11**, and **17**, returned acceptable z-scores (Figure 25).

Laboratory 8 may have accidentally reported the results for Sample S2 using incorrect units. Their results were not included in the analyses of extraction methods and of instrumental techniques employed by participants.

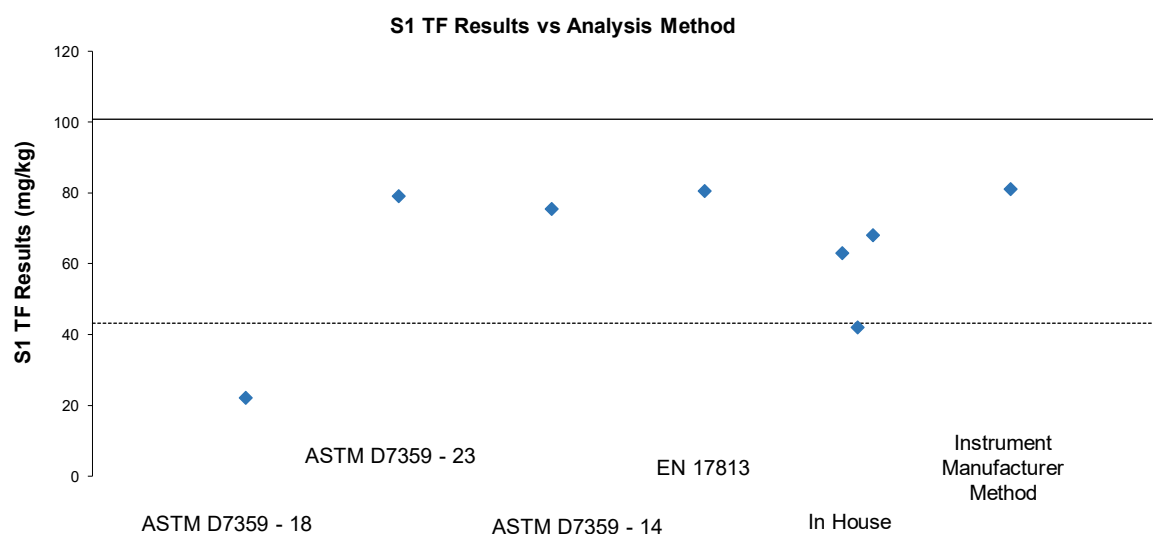
6.6 Participants' Results and Analytical Methods for Total Fluorine in Cardboard and Plastic Packaging Materials

Sample S1 was a cardboard packaging material and Sample S3 was a plastic packaging material. Participants were requested to analyse the samples using their normal test method and to report a single TF result as they would normally report to a client. The method descriptions provided by participants for TF measurements in the two samples are presented in Appendix 5.

Nine laboratories measured TF in Samples S1 and S3. Participants performed well in Sample S1; the between laboratory CV was 16%.

The assigned value for TF in Sample S1 was 72 ± 11 mg/kg. Laboratory 13 should review their method as they reported a result of <0.140 mg/kg.

Three laboratories reported using the combustion ion chromatographic (CIC) method based on various editions of the ASTM D7359, one participant used the CIC method based on the method published by European Committee for Standardization, EN 17813, and 5 used in house developed methods or instrument manufacturer method (Figure 26).



Horizontal lines correspond with a z-score of 2 and -2.

Figure 26 S1 TF Participants Results vs. Method

Sample S3 was a pilot sample aimed at helping laboratories to assess their method detection level. The results reported for this sample varied from <0.140 mg/kg to 9 mg/kg.

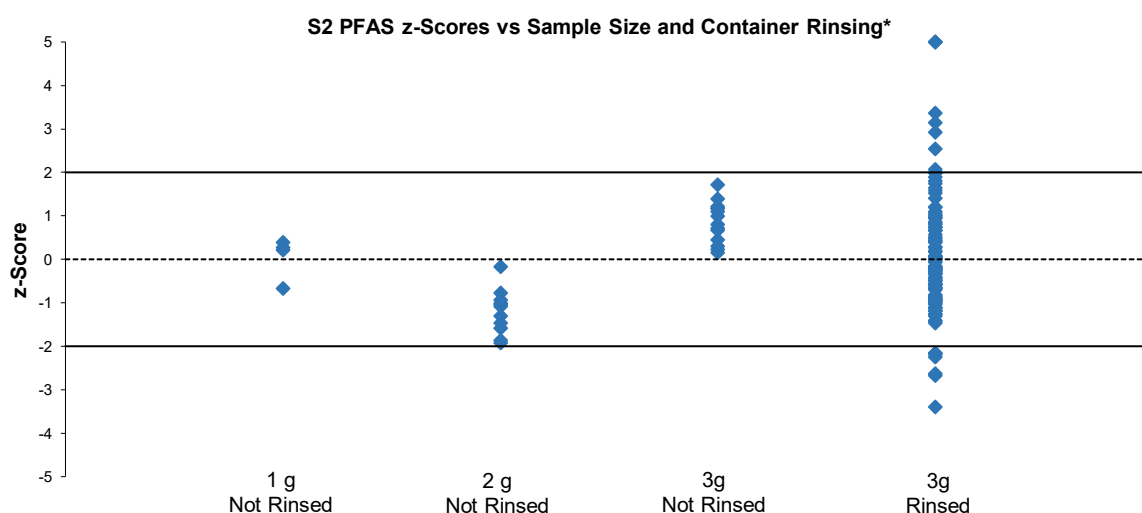
The TF homogeneity value in Sample S3 was <10 mg/kg and of 9 results reported, 6 were less than 10 mg/kg, 5 mg/kg, 0.2 mg/kg or 0.140 mg/kg.

6.7 Participants' Results and Analytical Methods for PFAS in Cardboard Packaging Material

The method descriptions provided by participants for PFAS measurements in the cardboard packaging material sample S2 are presented in Appendix 6.

Sample S2 was a cardboard packaging material fortified with 12 individual PFAS compounds. Participants were requested to analyse the samples using their normal test method but “to use the entire sample and to rinse the sample container with the reagent used for extraction”. Two identical containers with the test sample were sent to participating laboratories for repeat analysis if required.

Of 13 participants who reported results for PFAS in S2 (laboratory 8 excluded), 2 did not use the entire container and 3 did not rinse the container. Plots of participants' performance versus the amount of sample used for analysis are presented in Figure 27. No trends with the amount of sample taken for analysis were evident



. Scores greater than 5 or smaller than -5 have been plotted as 5 and -5 respectively.

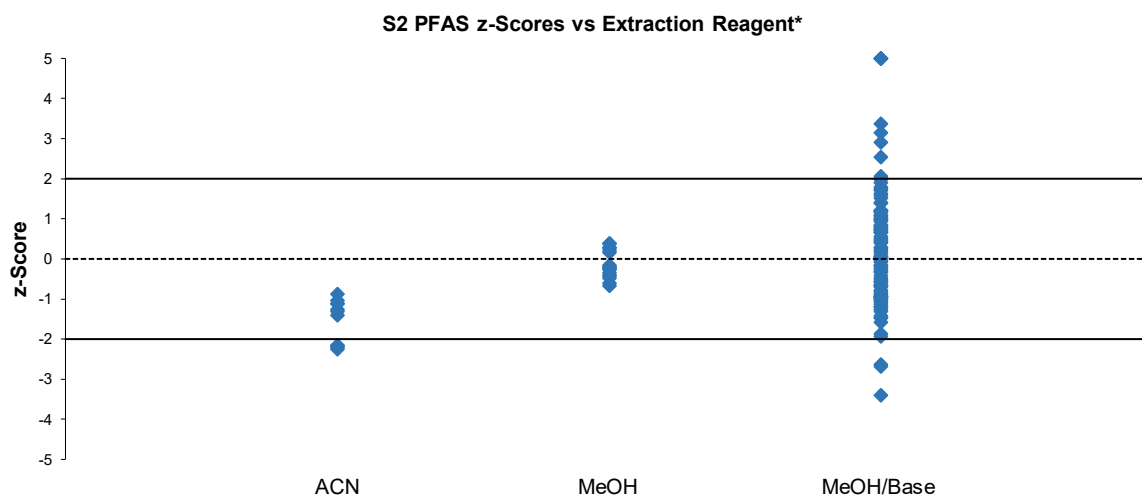
Figure 27 S2 PFAS z-Scores vs Sample Size

Of 13 participants who reported results for PFAS in Sample S2, 9 reported adding isotopically labelled internal standards before extraction and 6 of them left the sample to equilibrate for 5 to 30 min.

Laboratories 7 and 9 did not add isotopically labelled internal standards prior to extraction to account for potential errors introduced during sample preparation.¹³ Their overall performance is highlighted (circled) in (Figure 21).

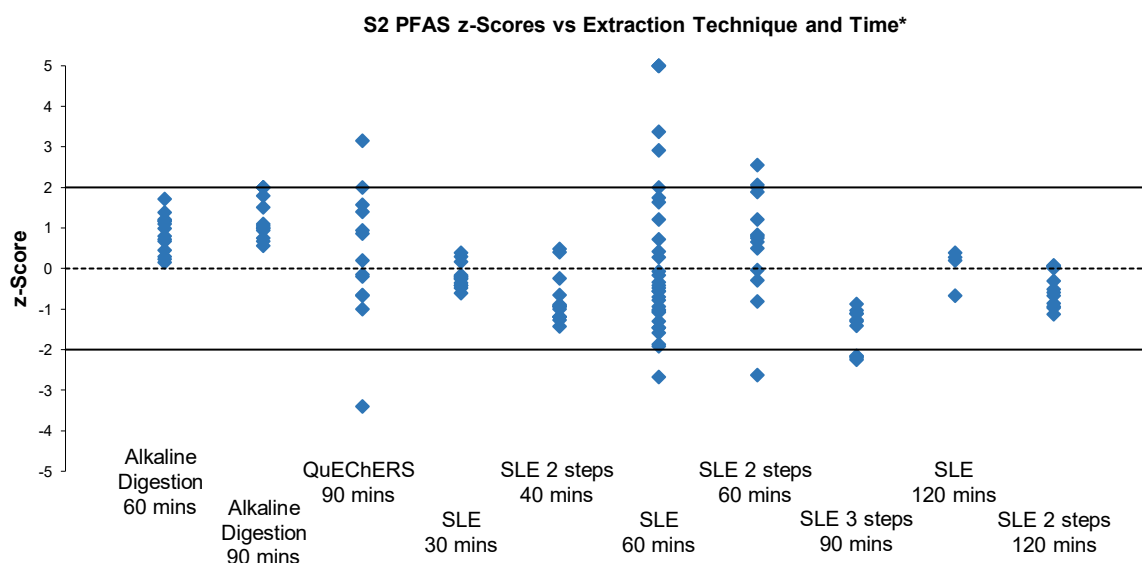
Base modified methanol was the preferred extraction reagent. Although PFAS results were generally compatible with each other, discrepancies were noticed between the results produced by methods which involved methanol or base-modified methanol as extraction solvent and those produced by acetonitrile (ACN) extraction (Figure 28). When ACN is used in LC-MS the chromatographic peaks for the short chain carboxylic acids might appear wide

and partially split, and a decrease in sensitivity for these analytes' responses is sometimes noted.¹⁴



*Scores greater than 5 or smaller than -5 have been plotted as 5 and -5 respectively.

Figure 28 S2 PFAS z-Scores vs Extraction Reagent



*Scores greater than 5 or smaller than -5 have been plotted as 5 and -5 respectively.

Figure 29 S2 PFAS z-Scores vs Extraction Technique and Time

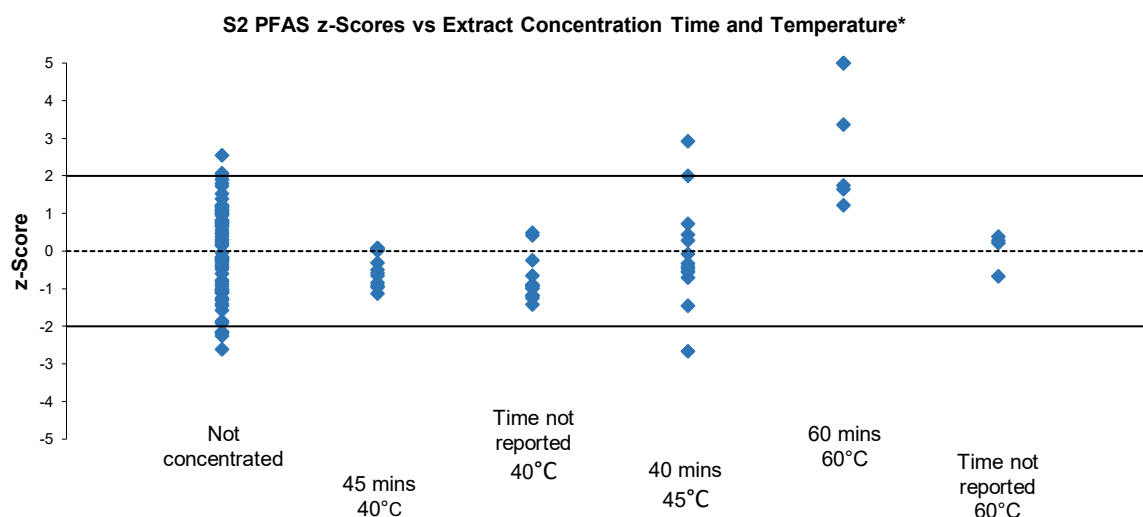
Most participants used the Solid Liquid Extraction (SLE) technique, one used QuEChERS and two Alkaline Digestion. Four participants reported using a staggered extraction: 3 conducted SLE extraction over 2 steps and one laboratory used a staggered SLE extraction over 3 steps. Plots of participants performance versus the extraction technique and extraction time are presented in Figure 29. Results from Alkaline Digestions were higher than some results from SLE and QuEChERS technique.

Two laboratories added loose carbon to the sample extract to facilitate better adsorption of interferent organics.¹³

Five laboratories concentrated their sample extracts at temperatures between 40°C and 60°C, with one laboratory concentrating the extract for 60 minutes at 60°C (Figure 30). According to the USEPA Method 1633, if all methanol is evaporated then the extract can be too

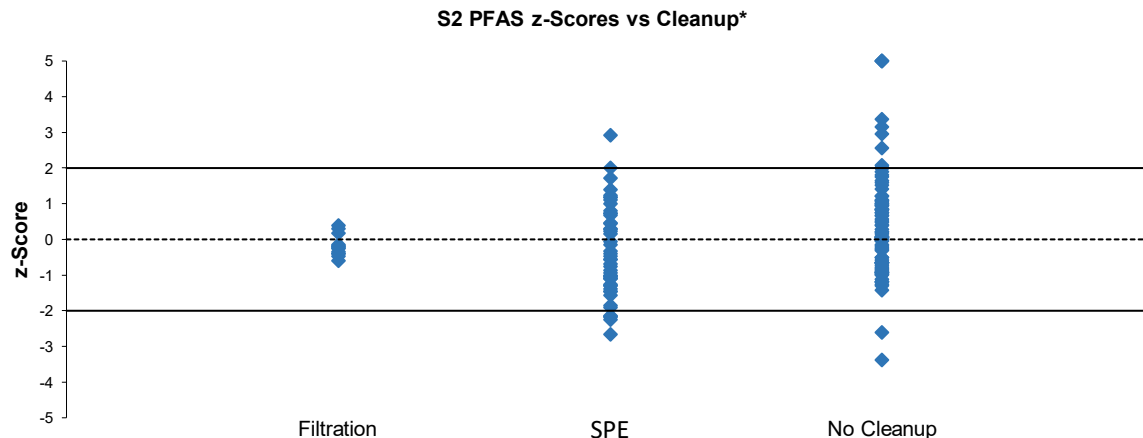
concentrated and/or losses of neutral compounds can occur (FOSEs and FOSAs). Alternatively, if excess methanol is present during SPE cleanup then long chain carboxylic acids and sulfonates are likely to have poor recovery.¹³

Cleanup of the crude extracts is an important step in the removal of matrix constituents that may interfere in instrumental determination. Almost half of the laboratories who reported results for PFAS in Sample S2 have not conducted a cleanup step after extraction. (Figure 31).



* Scores greater than 5 or smaller than -5 have been plotted as 5 and -5 respectively.

Figure 30 S2 PFAS z-Scores vs Extract Concentration Temperature and Time



* Scores greater than 5 or smaller than -5 have been plotted as 5 and -5 respectively.

Figure 31 S2 PFAS z-Scores vs Extraction Cleanup Procedure

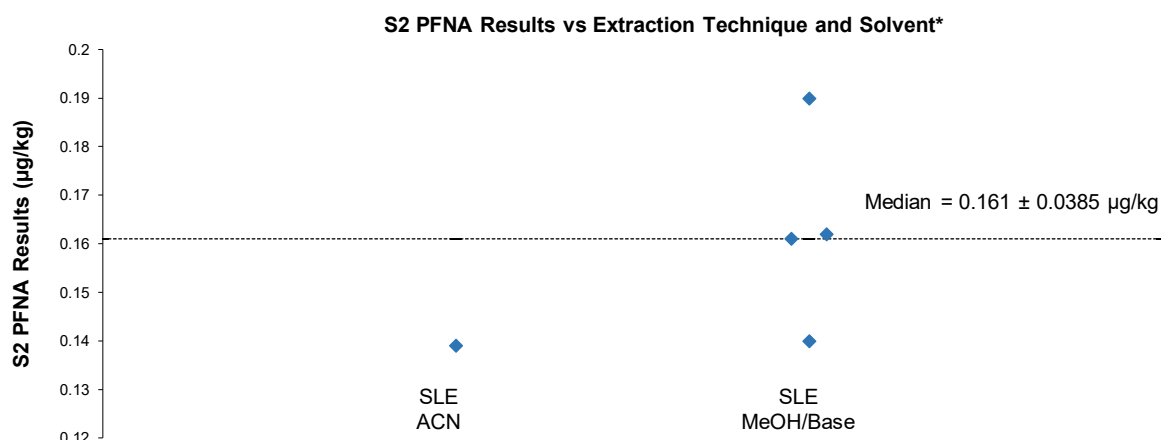
The most popular sample preparation method used for PFAS in cardboard packaging materials was a SLE extraction based on the method that involved using the entire sample (3 g) as instructed, base modified methanol as extraction solvent and no extract concentration and cleanup.

LC-MS/MS (triple quadrupole, QQQ) was the instrumental technique of choice for all participants.

6.7.1 Individual PFCA Analytes in Packaging Material

The between-laboratory CVs for PFCA analytes in Sample S2 varied from 15% to 36%.

PFNA was present in the cardboard packaging material as incurred PFAS compound. Of 14 laboratories who measured PFAS in Sample S2, only 6 identified and quantified this analyte (laboratory 8 included). All reported results were in good agreement with each other, except for one (Figure 32).



*The results reported by Laboratory 8 was not included.

Figure 32 S2 PFNA Results vs Method

PFDA and PFDoA were identified from literature as well as previous experience as being analytes which are at risk of being adsorbed onto the wall of the container during sample preparation and/or during analysis.¹⁴⁻²⁰ All analytes including these long chain PFCAs were spiked directly into each container with the aim of minimising loss during preparation. The participants were advised “to use the entire sample and to rinse the sample container with the reagent used for extraction”. Plots of participants’ z-scores versus sample size are presented in Figures 33 and 34. No trends were evident between the results from participants who did not use the entire sample container and/or did not rinse it, and those who did.

All unsatisfactory PFDA and PFDoA results were lower than the assigned value by 50% indicating potential issues with sample or standards preparation and dilution procedures. Laboratory 1 should check for method or laboratory bias as most of their unsatisfactory results were lower than assigned value by 50%

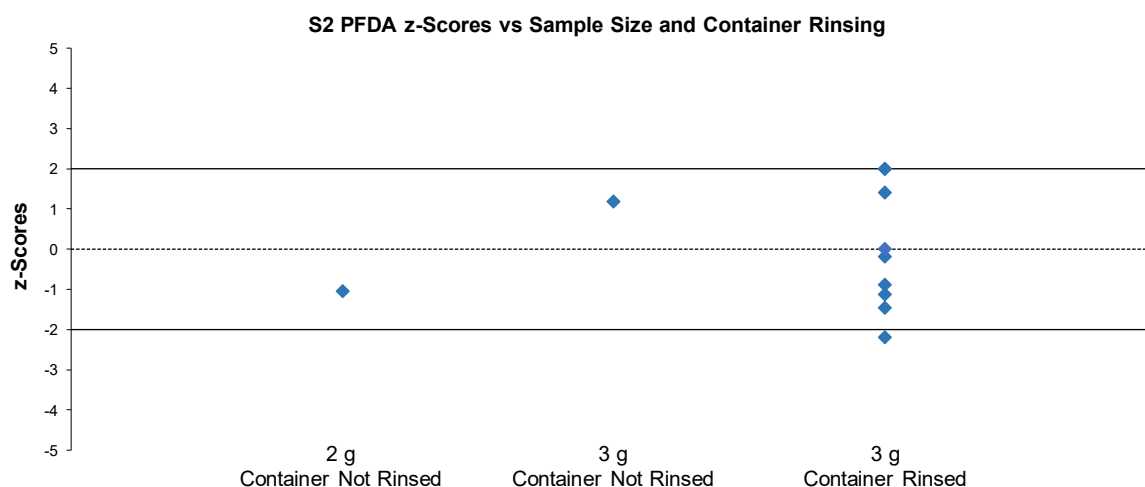


Figure 33 S2 PFDA z-Scores vs Sample Size

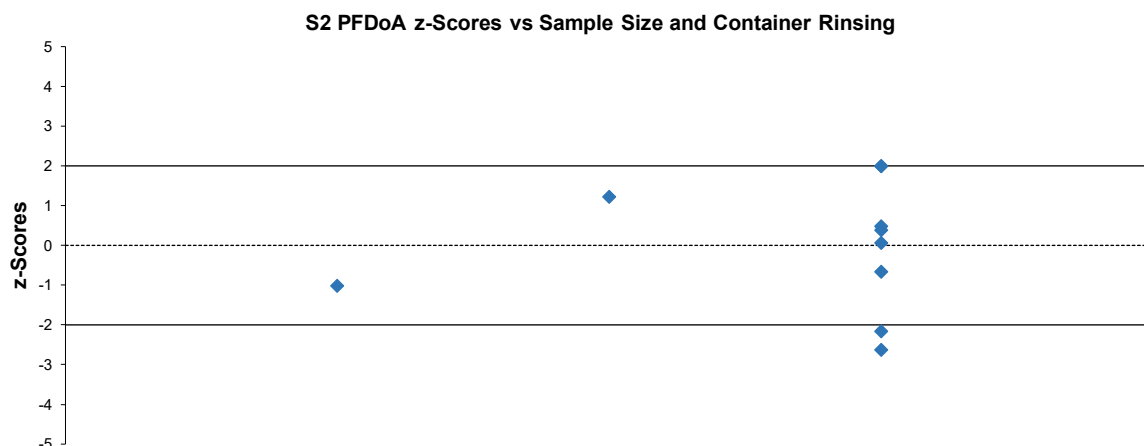
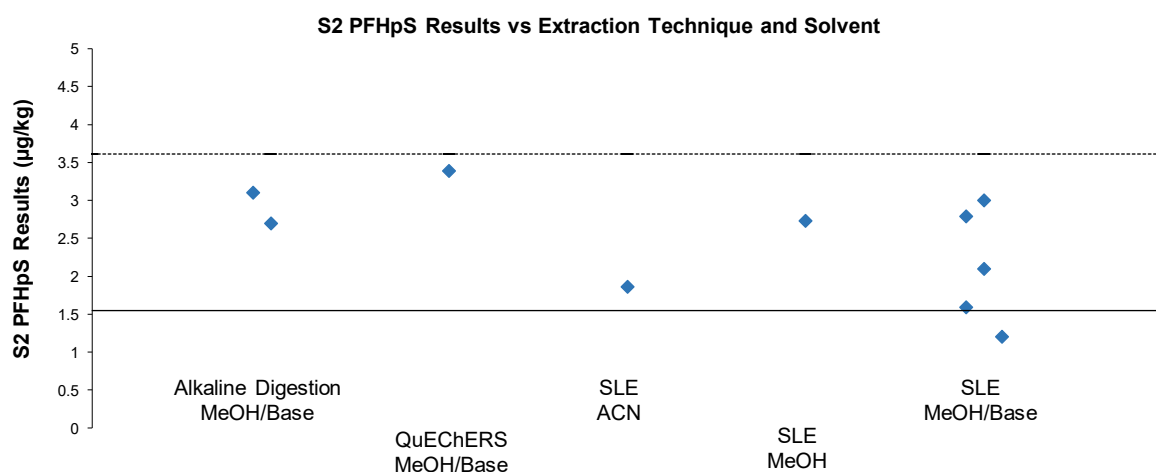


Figure 34 S2 PFD0A z-Scores vs Sample Size

6.7.2 Individual PFSA Analytes in Packaging Material

PFHpS was incurred with a concentration of 2.58 µg/kg, which may have challenged participants' analytical techniques. The between-laboratory CV was 26%, participants' performance versus analytical techniques used are presented in Figure 35. Due to limited data and the variety of extraction techniques used, no significant trends in extraction and sample preparation procedures were identified.



Horizontal lines correspond with a z-score of 2 and -2.

Figure 35 S2 PFHpS Results vs Method

PFOS in Sample S2 was spiked with a standard containing both linear and branched isomers. The spiked value for total PFOS was 14.3 µg/kg while linear PFOS was 9.66 µg/kg. While the consensus result for linear PFOS (9.9 µg/kg) was in good agreement with the spiked value, the assigned value for total PFOS was 113% higher than the spiked value.

An investigation conducted by NMI using LC-QToF, identified possible interferences under some extraction and chromatographic conditions (Figure 36). These can contribute to the branched PFOS response and may cause a positive bias in participants' results

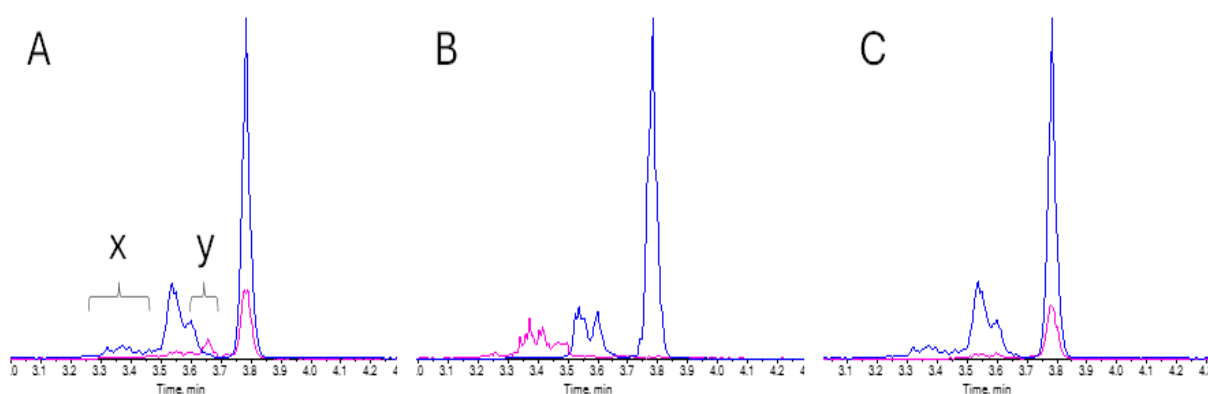


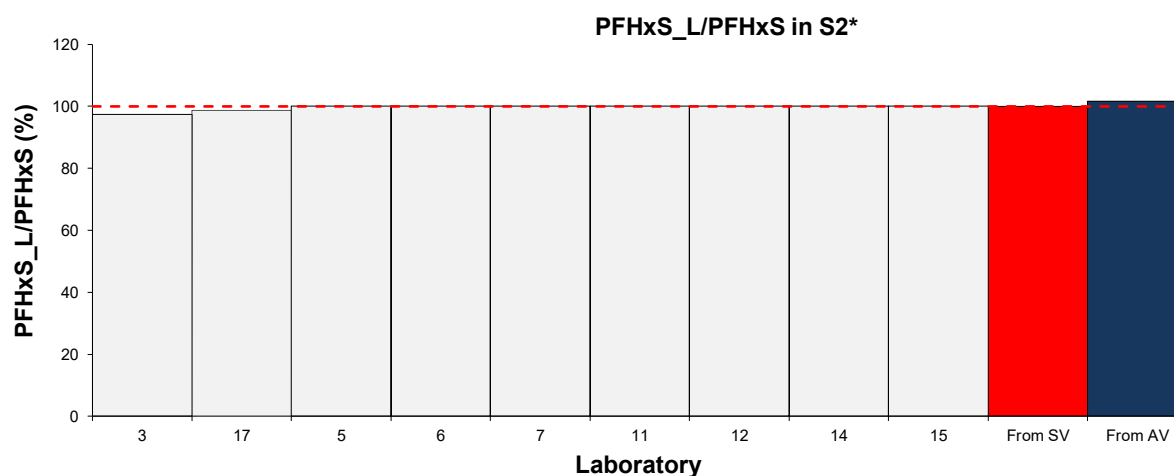
Figure 36 Sample S2 PFOS Chromatograms

In Figure 36, Chromatogram A demonstrates interference observable for unit mass resolution for branched PFOS in the 499>80 (blue, region x) and 499>99 (purple, region y) transitions. Chromatogram B shows interference on the precursor ion (498.93 m/z, blue) at 499.17 m/z (purple), which cannot be resolved on unit mass resolution mass spectrometers. Chromatogram C shows high resolution transitions 499>79.65 (blue) and 499>98.95 (purple). Using high resolution mass spectrometry and knowledge of precursors ions, the interference still at 499>79.65 can be avoided during peak integration, and the interference from 499>99.05 is no longer present.

Laboratories should be aware of these types of interferences as a triple quad cannot differentiate between branched PFOS and an interference for the precursor mass as the interference gives the same product mass.

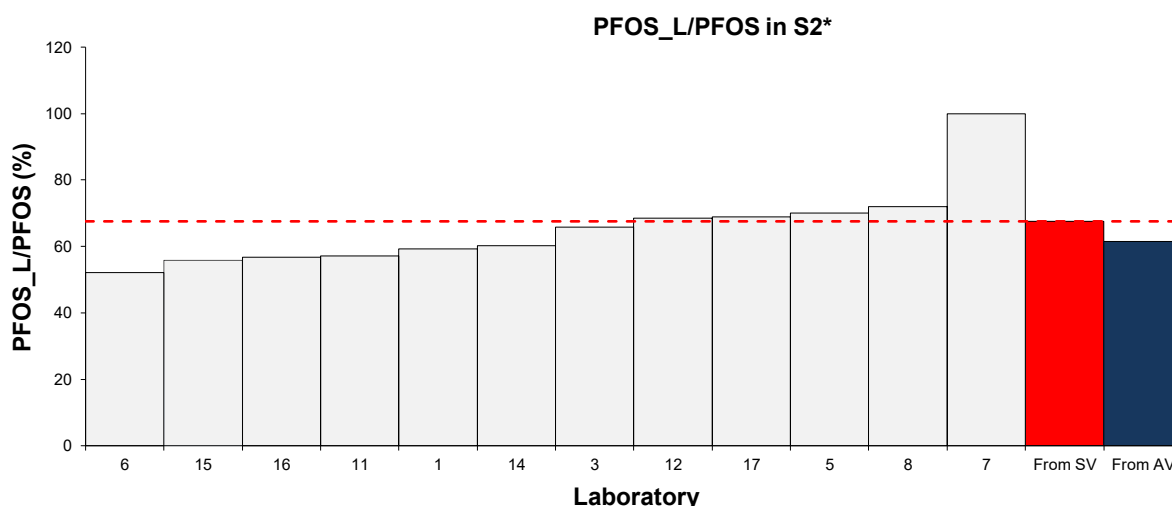
PFOS/PFOS_L and PFHxS/PFHxS_L For PFAS that contain linear and branched isomers, participants were asked to report total results (the sum of linear and branched) whereas for PFOS and PFHxS they were asked to report both total (the sum of linear and branched isomers) and linear (the linear isomer only) results.

Of 13 participants who reported results in cardboard packaging material sample S2, 9 reported results for both total and linear PFHxS. The ratios of linear PFHxS versus total PFHxS in S2 were between 97% and 100% (Figure 37). The cardboard material sample S2 was spiked with linear PFHxS; the ratio of linear PFHxS to total was expected to be 100% for this sample.



*The ratio from the AV is calculated based on the results reported by all participants including those who reported results for only one analyte.

Figure 37 Bar Charts of PFHxS_L/PFHxS_T in Sample S2



*The ratio from the AV is calculated based on the results reported by all participants including those who reported results for only one analyte.

Figure 38 Bar Charts of PFOS_L/PFOS_T in Sample S2

Figure 38 presents bar charts with ratios of linear PFOS results versus total PFOS results in Sample S2. Twelve participants reported results for both PFOS isomers in Sample S2.

The reported ratios of linear PFOS versus total PFOS in S2 were between 52% and 100% while the expected value ratio between the two isomers was 67%.

When a laboratory is using a combined branched/linear standard and integrate branched/linear together for totals, the result could be different to a linear only result due to response factor differences between the isomers.¹³

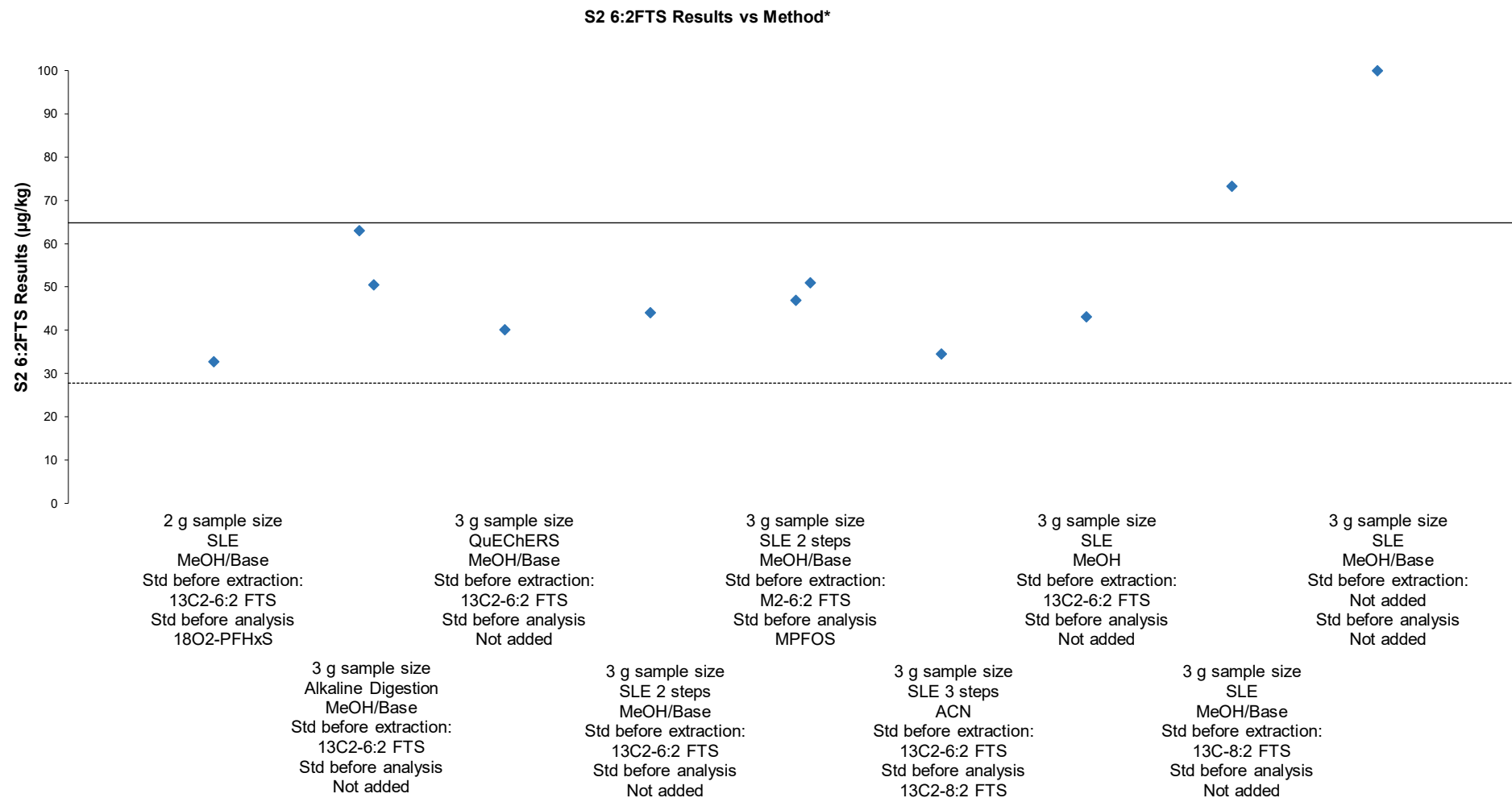
6.7.3 Individual PFAA Precursors or Related Compounds

6:2FTS Twelve participants reported results for 6:2FTS in S2, all (laboratory 8 excluded) had an acceptable performance except Laboratory 7. Laboratory 7 should check their sample/standard preparation procedure as most of the results reported by them were higher than the assigned value by a factor of 2 or 4.

Plots of participants results versus the method used are presented in Figure 39.

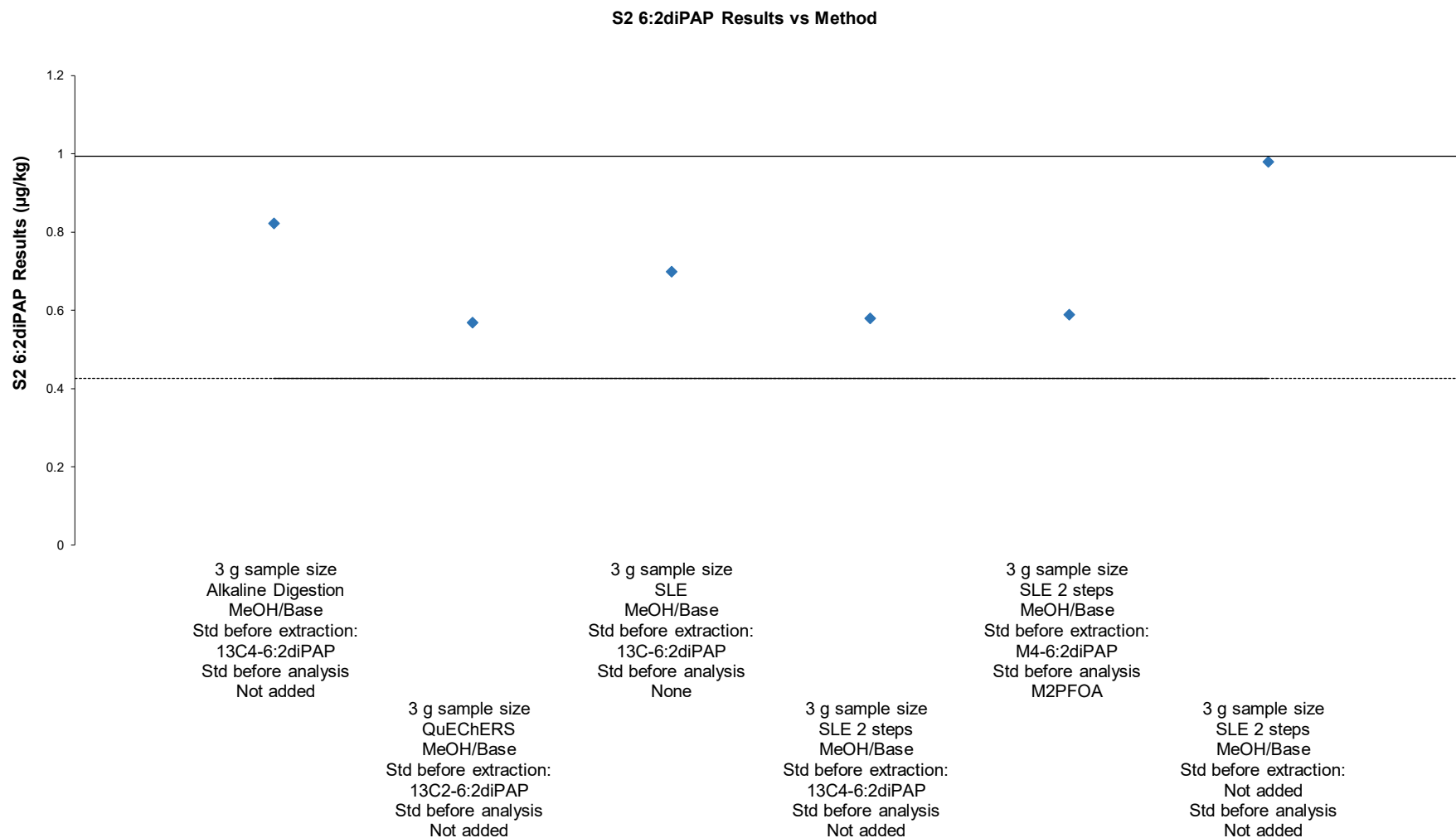
6:2diPAP and 8:2diPAP are primarily used for their surfactant properties in the paper industry, and were not spiked in the study sample, but were present as incurred compounds. Six laboratories detected and quantified 6:2diPAP, and all performed acceptably (laboratory 8 excluded). Laboratory 12 used M2PFOA as internal standard after extraction, no other laboratory added a labelled internal standard before instrumental analysis (Figure 40).

Five laboratories detected and quantified 8:2diPAP. The results reported for this test were in good agreement with each other. However, no assigned value could be set due to the limited number of reported results (Figure 41).



* The result of 200 µg/kg has been plotted as 100 µg/kg. Horizontal lines correspond with a z-score of 2 and -2.

Figure 39 S2 6:2FTS Results vs Method



* Horizontal lines correspond with a z-score of 2 and -2.

Figure 40 S2 6:2diPAP Results vs Method

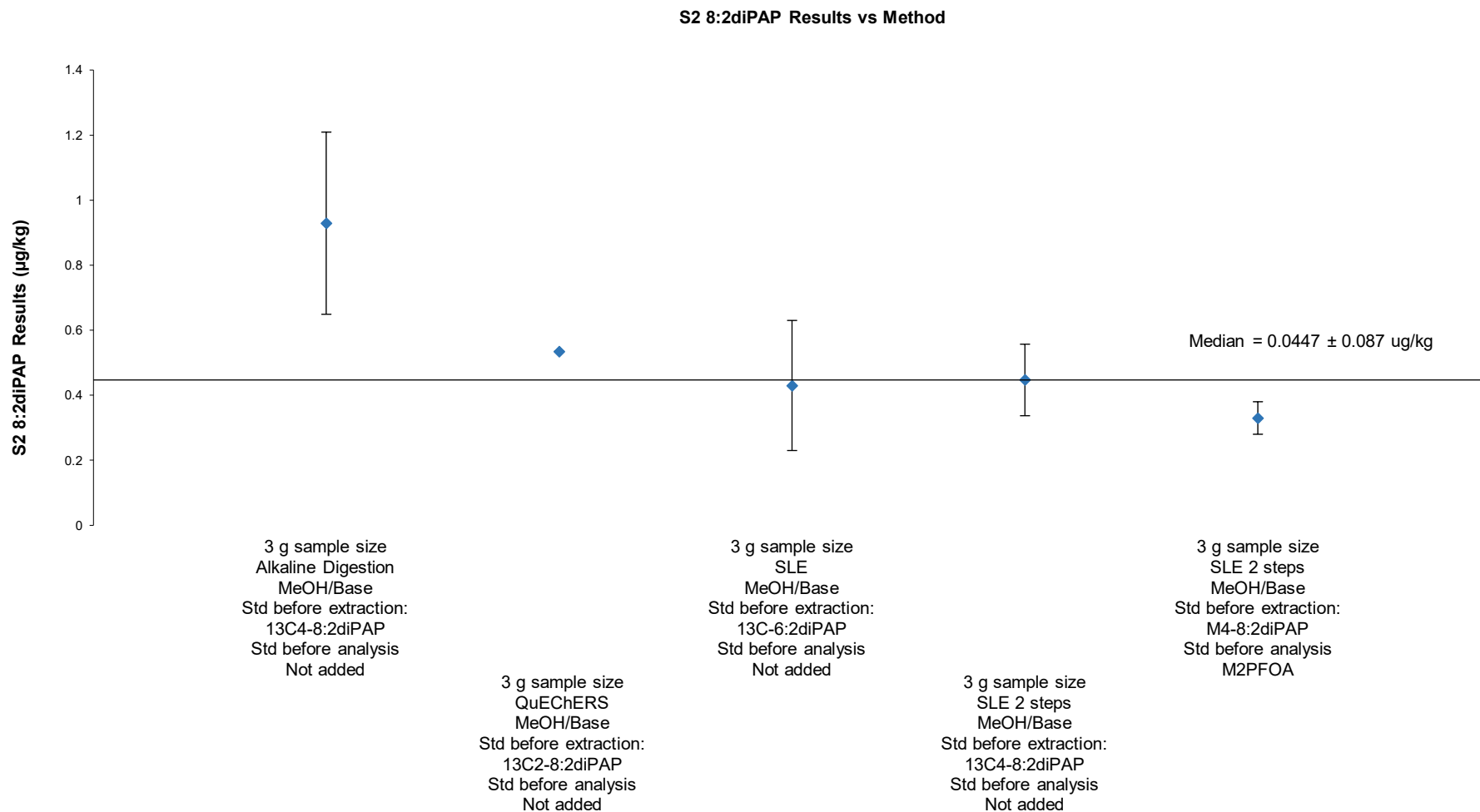


Figure 41 S2 8:2diPAP Results vs Method

6.8 False Negatives

Appendix 4 presents false negative results. These are analytes present in the samples which a participant tested for, but did not report a result; for example, when participants reported a 'less-than' result ($< x$) when the assigned value was higher than their limit of reporting (LOR), or did not report anything (NR). However, results reported as NR may or may not be false negatives as this is depending on the participant's actual LOR.

7 REFERENCES

Note: For all undated references, the latest edition of the referenced document (including any amendments) applies.

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APPENDIX 1 - SAMPLE PREPARATION, SAMPLE ANALYSIS and HOMOGENEITY and STABILITY ASSESSMENT

A1.1 Sample Preparation

Sample S1, was a cardboard packaging material, cut into approximately equal-sized pieces and weighed into 3 g portions.

Sample S2 was a cardboard packaging material cut into approximately equal- sized pieces, divided into accurately weighed 3 g portions and fortified for 12 PFAS analytes.

Sample S3 was a plastic food packaging material cut into approximately equal- sized pieces, and weighed into portions of 1 g each.

Samples S1 and S3 were stored at room temperature prior to dispatch. Sample S2 was stored at 4°C.

A1.2 Sample Analysis and Homogeneity Assessment for TF in S1 and S3

Sample Analysis

The analysis for TF homogeneity in S1 and S3 were conducted by the Inorganics section of NMI as per method NWS20.²¹ The cardboard and plastic material samples were placed onto quartz boats and introduced into a combustion tube heated to 1050 °C. The samples underwent pyrolytic hydrolysis in the presence of water and oxygen. The resulting vapours were carried by argon gas into a sample tube containing water for absorption. The collected analytes were then transferred to the ion chromatograph for analysis. NMI holds ISO/IEC 17825 accreditation for this method.

Homogeneity Assessment

A full homogeneity test was conducted for Samples S1 and S3. Homogeneity testing for these samples was based on that described by Thompson and Fearn,²² which is also the procedure described in the International Harmonised Protocol for Proficiency Testing.⁸ Seven containers from each of Samples S1 and S3 were selected at random. Duplicate test-portions were taken from each container and the concentration of TF measured. Measurements were made under repeatability conditions in random order. Results for homogeneity testing for both samples are presented in Tables 26 and 27. The cardboard material sample S1 was found homogeneous for TF. The TF level in S3 was below the level of reporting of 10 mg/kg

Table 26 Sample S1 TF Homogeneity Testing .

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
1	84.01	86.11
7	87.02	93.85
16	81.50	92.57
37	84.64	85.07
43	87.51	88.37
55	76.25	87.80
67	84.91	93.43
Mean	86.65	
CV	4.7%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.35	0.73	Pass
s_{an}/σ	0.48	0.5	Pass
s^2_{sam}	0.000	61.10	Pass

Table 27 Sample S3 Total Fluorine Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
1	< 10	< 10
9	< 10	< 10
12	< 10	< 10
13	< 10	< 10
17	< 10	< 10
21	< 10	< 10
22	< 10	< 10
Mean	< 10	

A1.3 Sample Analysis and Homogeneity and Stability Assessment for PFAS in Sample S2

Sample Analysis

Measurements for PFAS in Sample S2 were conducted by the Organics section of NMI as per method NR70.²³ In brief, the method involved SLE extraction over 120 minutes, using a 3 g sample and KOH in MeOH as the extraction solvent. The extract was concentrated at 40 °C, and analysed by LC-MS/MS.

Although NMI holds ISO/IEC 17025 accreditation for this method, packaging material is outside the scope of this accreditation.

Homogeneity Assessment

Homogeneity testing was conducted for PFAS in cardboard sample with the exception of PFNA, PFDA, 6:2diPAP and 8:2diPAP (Tables 28 to 39).

Homogeneity testing was based on that described in the International Protocol. Seven samples (each consisting of 3 g cardboard material) were analysed in random order. The average of the results was reported as the homogeneity value.^{7, 22}

Since the entire sample was used in each analysis, it was not possible to apply analysis of variance (ANOVA) to determine if samples were sufficiently homogeneous. When it is not possible to conduct replicate measurements, the standard deviation of the results (sd) will be compared with the standard deviation for proficiency assessment- SDPA (σ) calculated as described in Section 4.5. The proficiency test samples may be considered sufficiently homogeneous if: $sd \leq 0.3 \sigma$.⁹

The cardboard material sample S2 was found to be sufficiently homogeneous for all PFAS analytes of interest.

Table 28 S2 PFBA Homogeneity Data

Sample number	Result (µg/kg)
S2 - 72	24
S2 - 2	23
S2 - 42	21
S2 - 8	23
S2 - 18	22
S2 - 17	23
S2 - 73	23
S2 - 50	25
S2 - 39	21
S2 - 51	24
Overall Average	22.9
CV	5.6%

	Value	Critical (<30% of SDPA)	Result
CV	5.6%	6%	Pass

Table 30 S2 PFHxA Homogeneity Data

Sample number	Result (µg/kg)
S2 - 72	250
S2 - 2	240
S2 - 42	250
S2 - 8	240
S2 - 18	230
S2 - 17	250
S2 - 73	260
S2 - 50	280
S2 - 39	260
S2 - 51	270
Overall Average	253
CV	5.9%

	Value	Critical (<30% of SDPA)	Result
CV	5.9%	6%	Pass

Table 29 S2 PFPeA Homogeneity Data

Sample number	Result (µg/kg)
S2 - 72	5.6
S2 - 2	5.7
S2 - 42	5.7
S2 - 8	5.8
S2 - 18	5.6
S2 - 17	5.5
S2 - 73	5.9
S2 - 50	5.7
S2 - 39	6.1
S2 - 51	5.7
Overall Average	5.73
CV	3.0%

	Value	Critical (<30% of SDPA)	Result
CV	3%	6%	Pass

Table 31 S2 PFOA Homogeneity Data

Sample number	Result (µg/kg)
S2 - 72	18
S2 - 2	20
S2 - 42	16
S2 - 8	18
S2 - 18	18
S2 - 17	18
S2 - 73	18
S2 - 50	19
S2 - 39	18
S2 - 51	18
Overall Average	18.1
CV	5.5%

	Value	Critical (<30% of SDPA)	Result
CV	5.5%	6%	Pass

Table 32 S2 PFDoA Homogeneity Data

Sample number	Result (µg/kg)
S2 - 72	7.3
S2 - 2	7.1
S2 - 42	7.5
S2 - 8	7.3
S2 - 18	7.2
S2 - 17	7.3
S2 - 73	7.6
S2 - 50	6.8
S2 - 39	7.3
S2 - 51	7.2
Overall Average	7.26
CV	3%

	Value	Critical (<30% of SDPA)	Result
CV	3%	6%	Pass

Table 34 S2 PFHxS Homogeneity Data

Sample number	Result (µg/kg)
S2 - 72	52
S2 - 2	53
S2 - 42	48
S2 - 8	49
S2 - 18	49
S2 - 17	51
S2 - 73	52
S2 - 50	52
S2 - 39	52
S2 - 51	52
Overall Average	51
CV	3.3%

	Value	Critical (<30% of SDPA)	Result
CV	3.3%	6%	Pass

Table 33 S2 PFBS Homogeneity Data

Sample number	Result (µg/kg)
S2 - 72	3.5
S2 - 2	3.4
S2 - 42	3.6
S2 - 8	3.7
S2 - 18	3.5
S2 - 17	3.4
S2 - 73	3.6
S2 - 50	3.4
S2 - 39	3.8
S2 - 51	3.3
Overall Average	3.52
CV	4.4%

	Value	Critical (<30% of SDPA)	Result
CV	4.4%	6%	Pass

Table 35 S2 PFHxS (Linear) Homogeneity Data

Sample number	Result (µg/kg)
S2 - 72	52
S2 - 2	53
S2 - 42	48
S2 - 8	49
S2 - 18	49
S2 - 17	51
S2 - 73	52
S2 - 50	52
S2 - 39	52
S2 - 51	52
Overall Average	51
CV	3%

	Value	Critical (<30% of SDPA)	Result
CV	3%	6%	Pass

Table 36 S2 PFHpS Homogeneity Data

Sample number	Result (µg/kg)
S2 - 72	2
S2 - 2	2
S2 - 42	1.9
S2 - 8	1.9
S2 - 18	1.9
S2 - 17	2.1
S2 - 73	2.2
S2 - 50	1.9
S2 - 39	2
S2 - 51	1.8
Overall Average	1.97
CV	5.9%

	Value	Critical (<30% of SDPA)	Result
CV	5.9%	6%	Pass

Table 38 S2 PFOS (Linear) Homogeneity Data

Sample number	Result (µg/kg)
S2 - 72	9.1
S2 - 2	9.5
S2 - 42	7.9
S2 - 8	8.5
S2 - 18	8.3
S2 - 17	9
S2 - 73	9
S2 - 50	9
S2 - 39	8.9
S2 - 51	8.5
Overall Average	8.77
CV	5.0%

	Value	Critical (<30% of SDPA)	Result
CV	5%	6%	Pass

Table 37 S2 PFOS Homogeneity Data

Sample number	Result (µg/kg)
S2 - 72	13
S2 - 42	11
S2 - 8	12
S2 - 18	12
S2 - 17	13
S2 - 73	13
S2 - 50	13
S2 - 39	13
S2 - 51	12
Overall Average	12.4
CV	5.8%

	Value	Critical (<30% of SDPA)	Result
CV	5.8%	6%	Pass

Table 39 S2 6:2FTS Homogeneity Data

Sample number	Result (µg/kg)
S2 - 72	52
S2 - 2	46
S2 - 42	46
S2 - 8	50
S2 - 18	50
S2 - 17	49
S2 - 73	47
S2 - 50	48
S2 - 39	48
S2 - 51	47
Overall Average	48.3
CV	4.0%

	Value	Critical (<30% of SDPA)	Result
CV	4%	6%	Pass

A1.3 Stability Assessment for TF in Sample S1 and PFAS in Sample S2

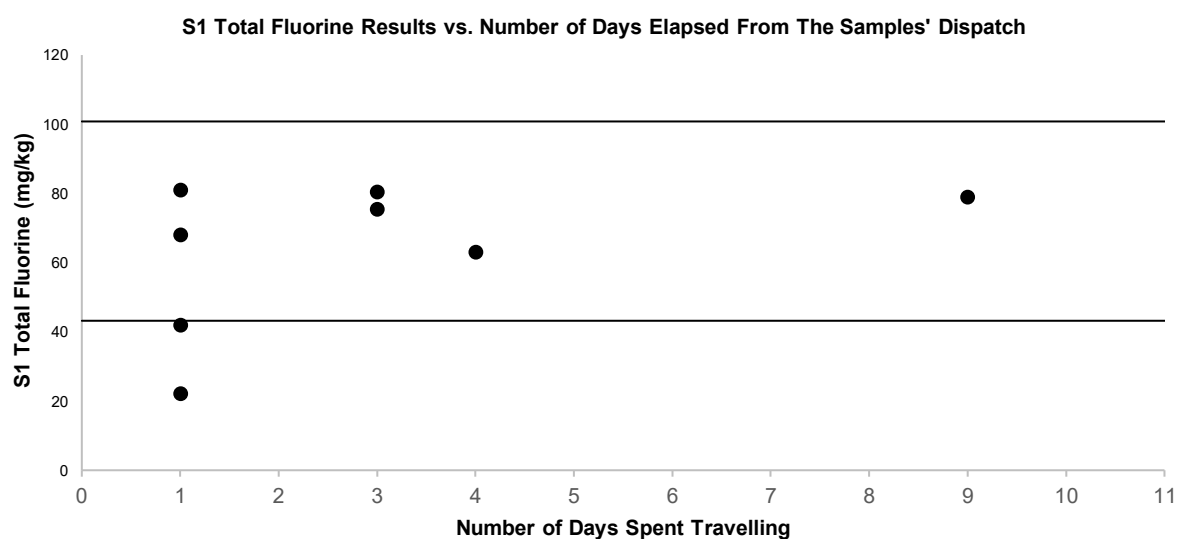
Stability Assessment

The tests samples were dispatched on 02/06/2025. The date when the samples were received by each participant are presented in Table 40.

Table 40 Received Date by Laboratories

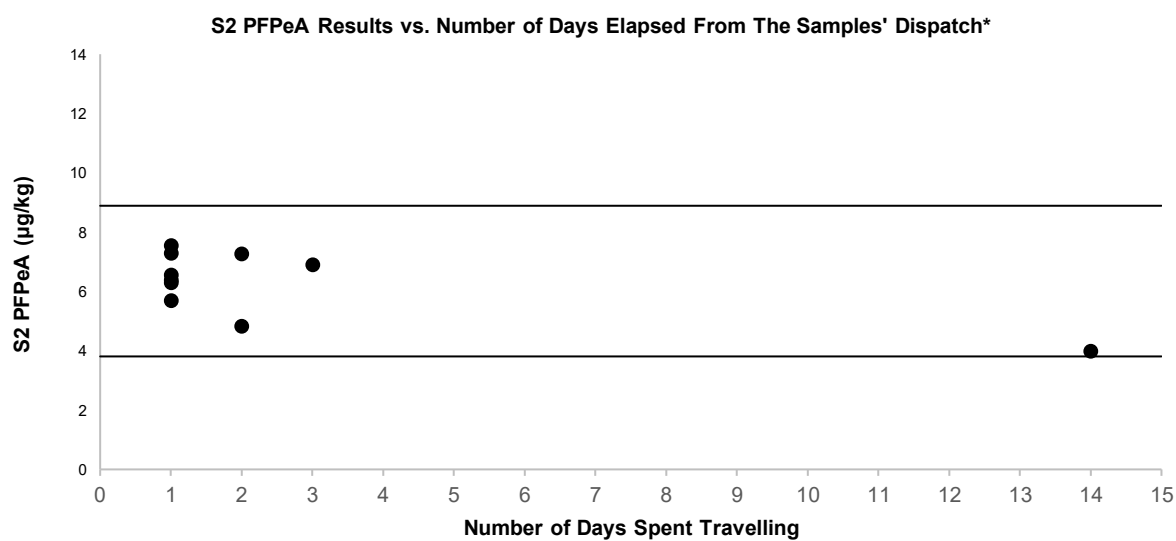
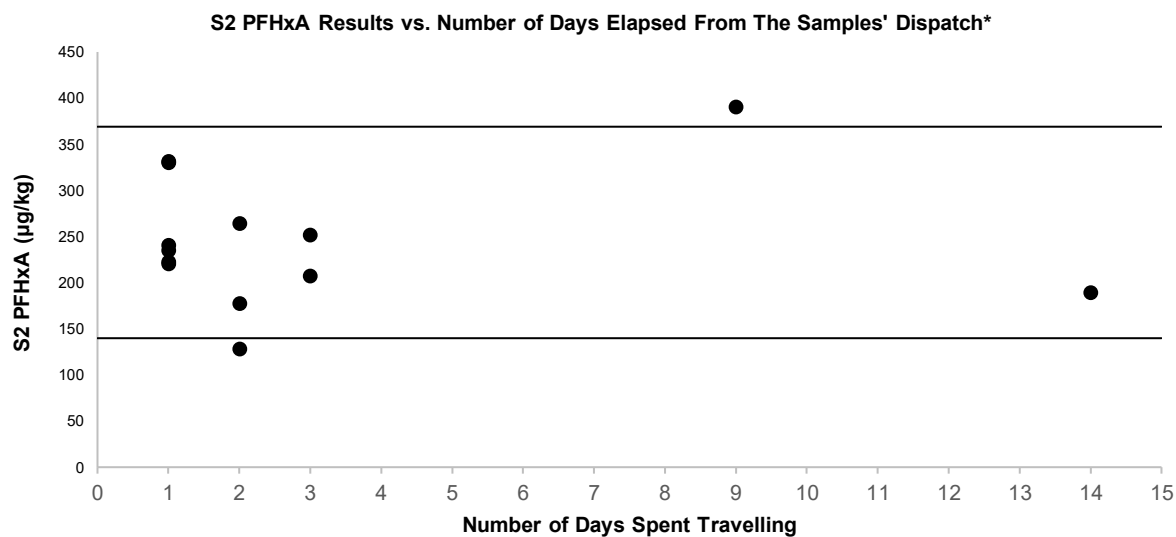
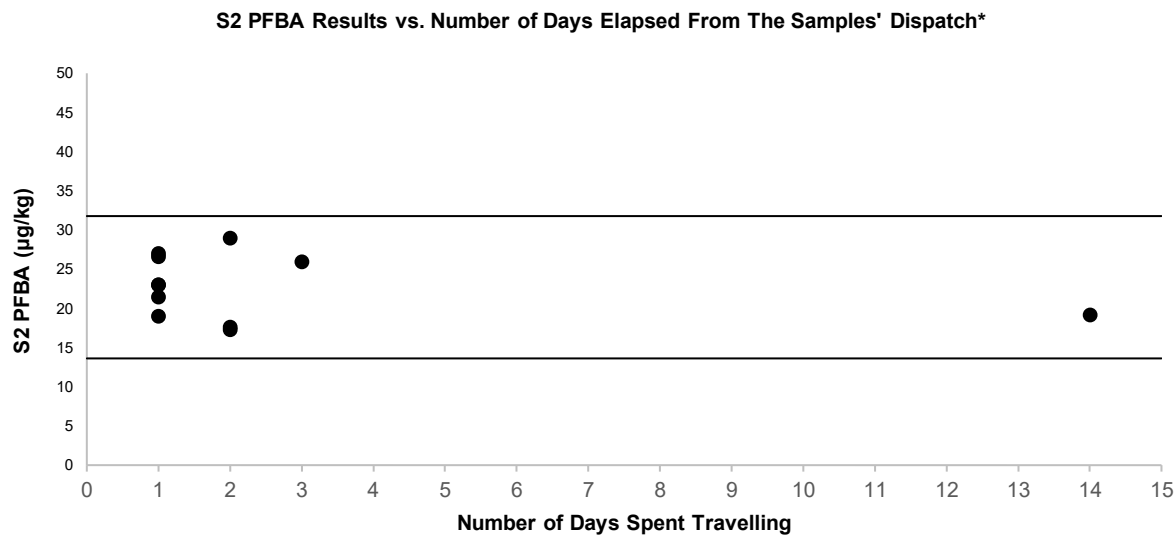
Lab. Code	Received Date
1	4/06/2025
2	6/06/2025
3	4/06/2025
4	3/06/2025
5	3/06/2025
6	3/06/2025
7	11/06/2025
8	5/06/2025
9	5/06/2025
10	3/06/2025
11	3/06/2025
12	3/06/2025
13	16/06/2025
14	5/06/2025
15	3/06/2025
16	3/06/2025
17	4/06/2025

No relationship was evident between the results reported for TF in Sample S1 and for PFAS in Sample S2 and the date when the sample was received (Figures 42 and 43).



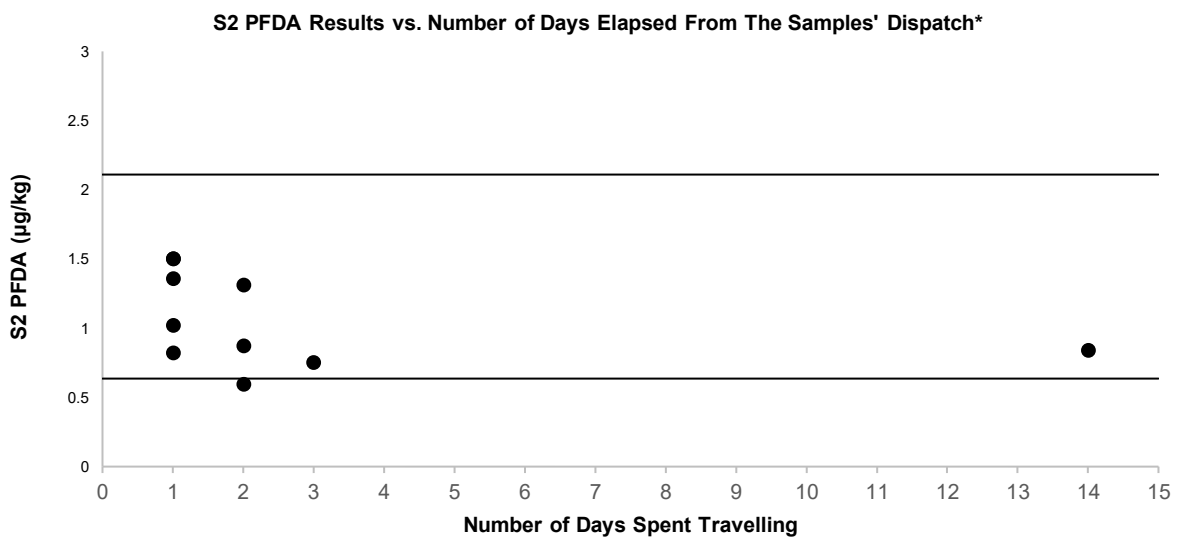
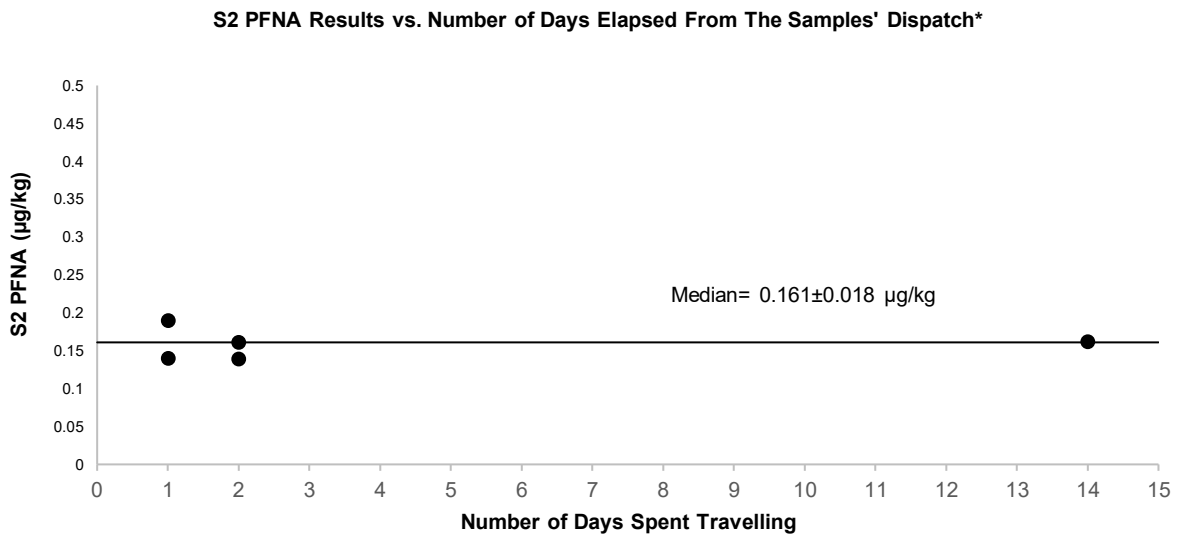
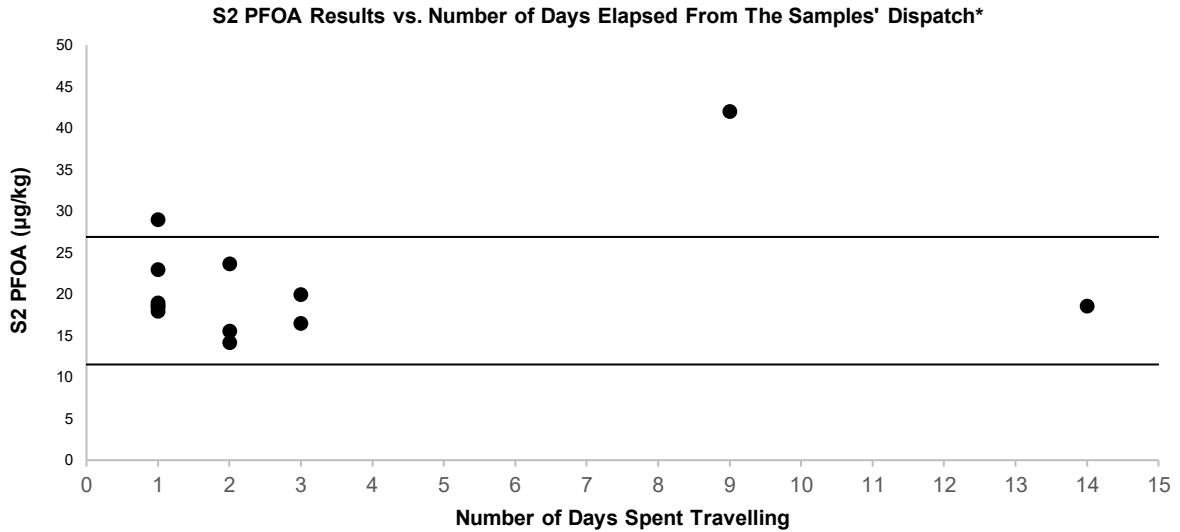
Horizontal lines on charts are the results corresponding to z-scores of 2 and -2.

Figure 42 Results vs Days Spent in Transit



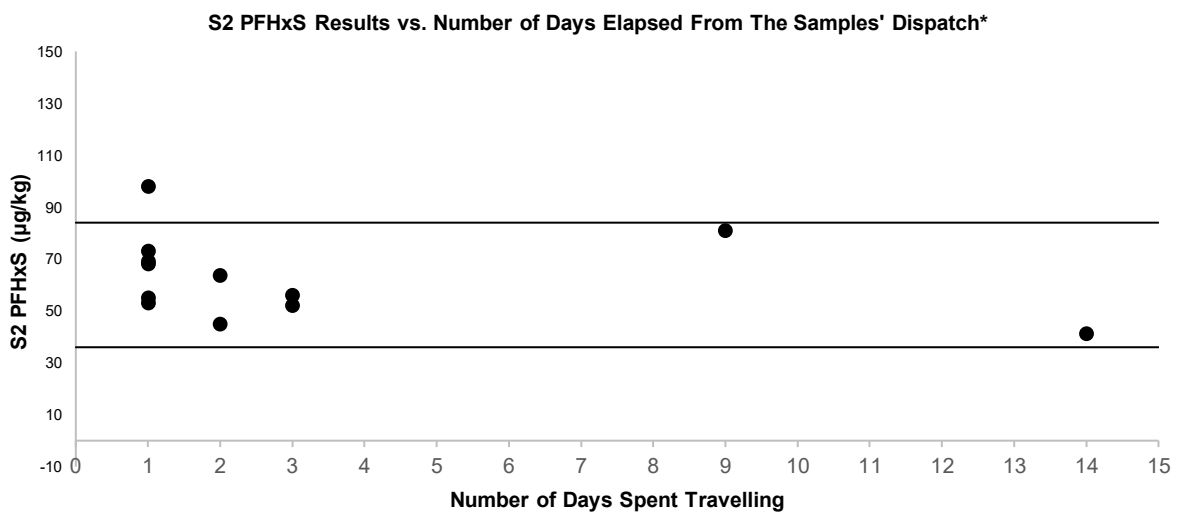
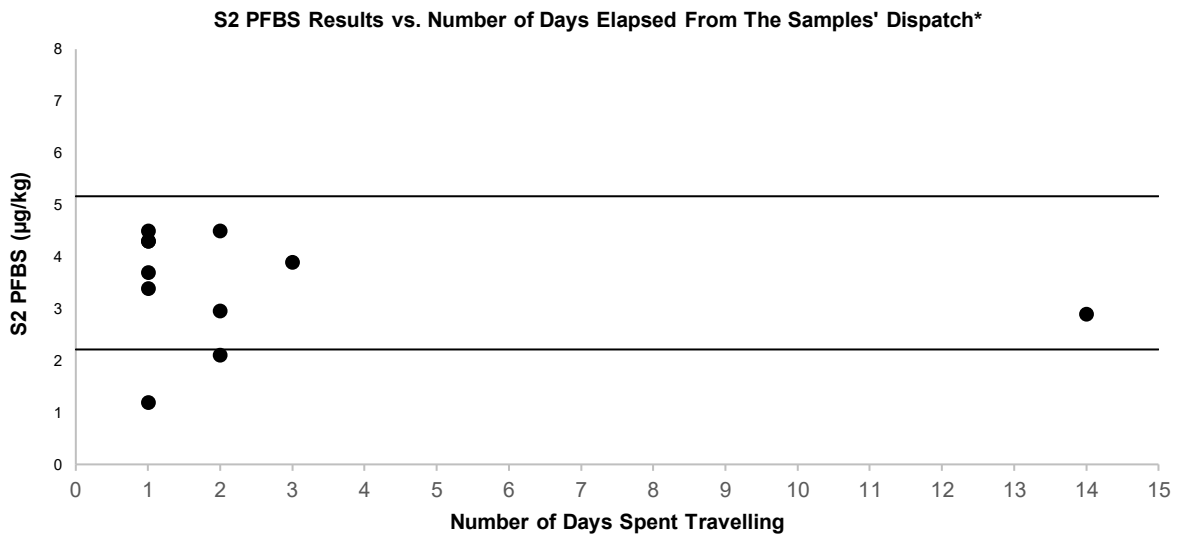
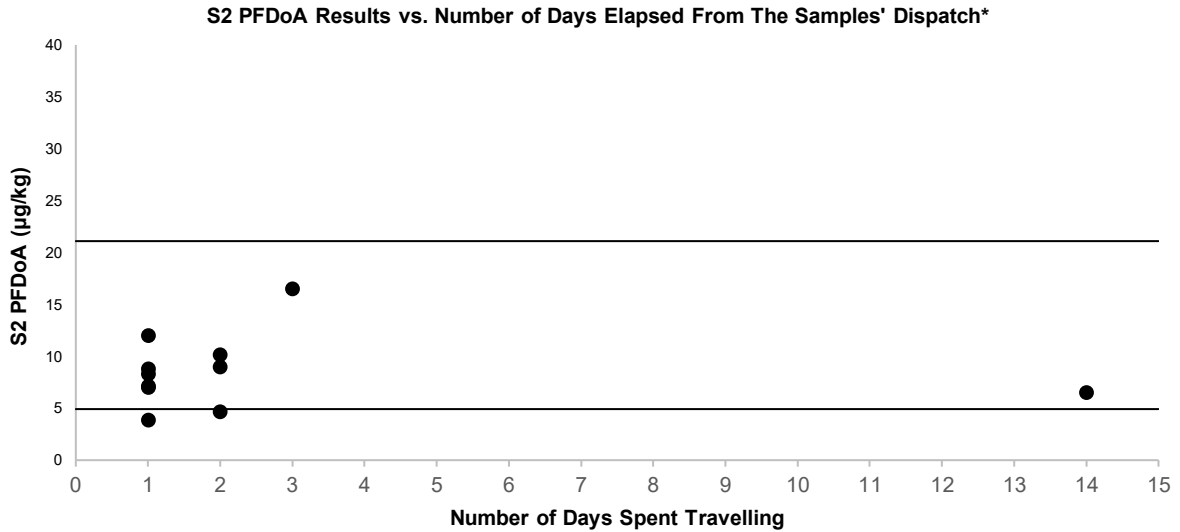
*Laboratory 8 excluded. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2.

Figure 42 Results vs Days Spent in Transit (continued)



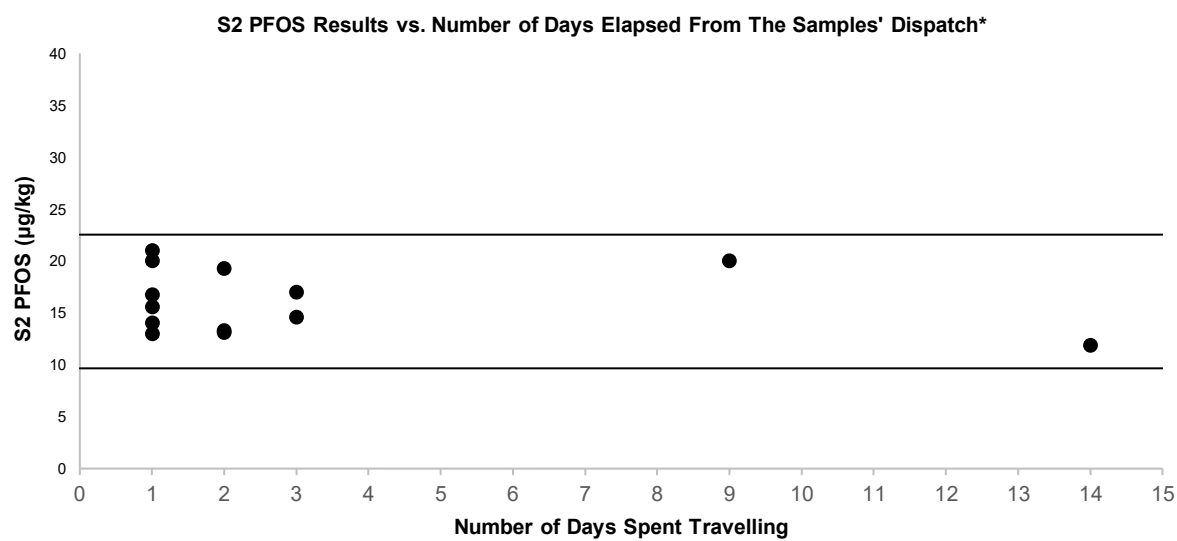
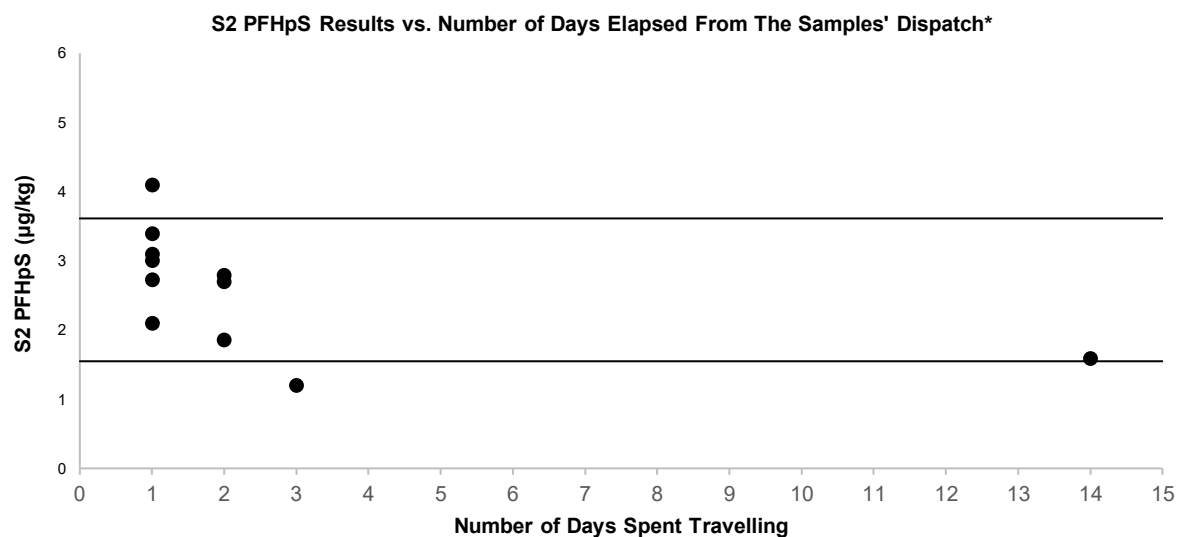
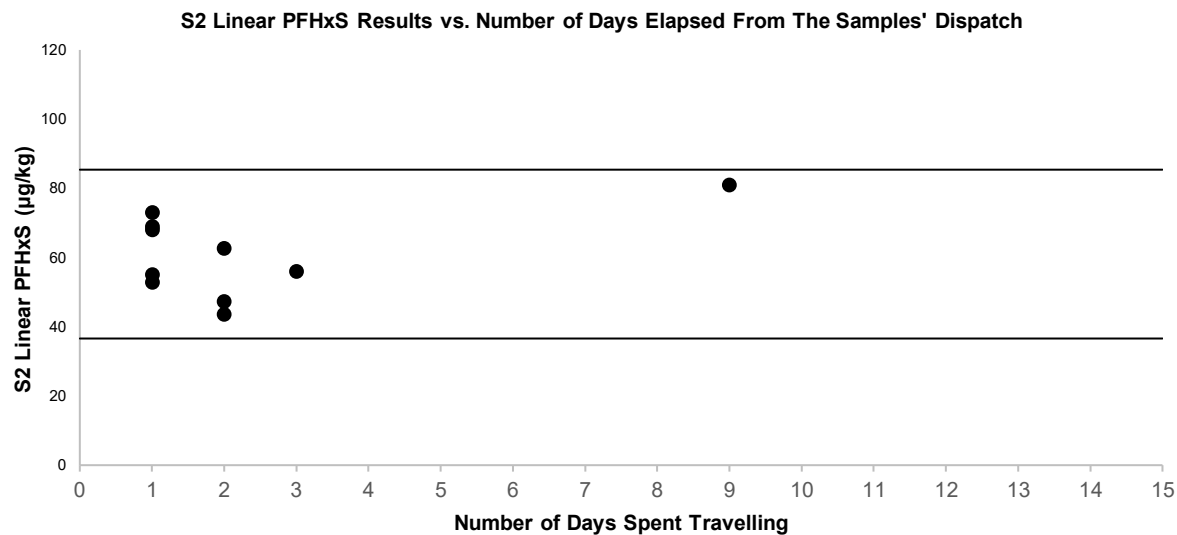
*Laboratory 8 excluded. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2.

Figure 42 Results vs Days Spent in Transit (continued)



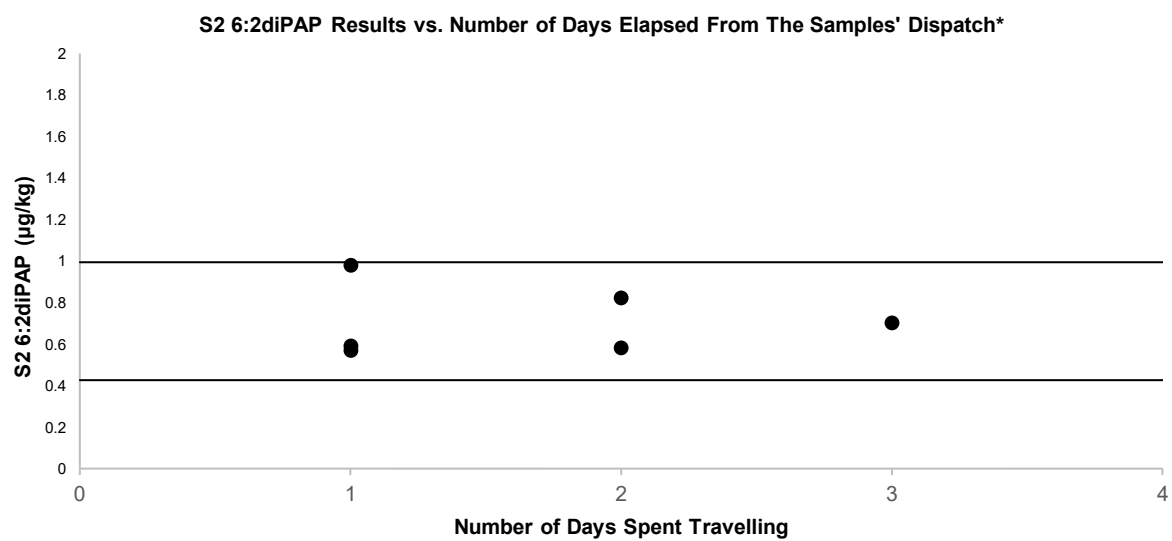
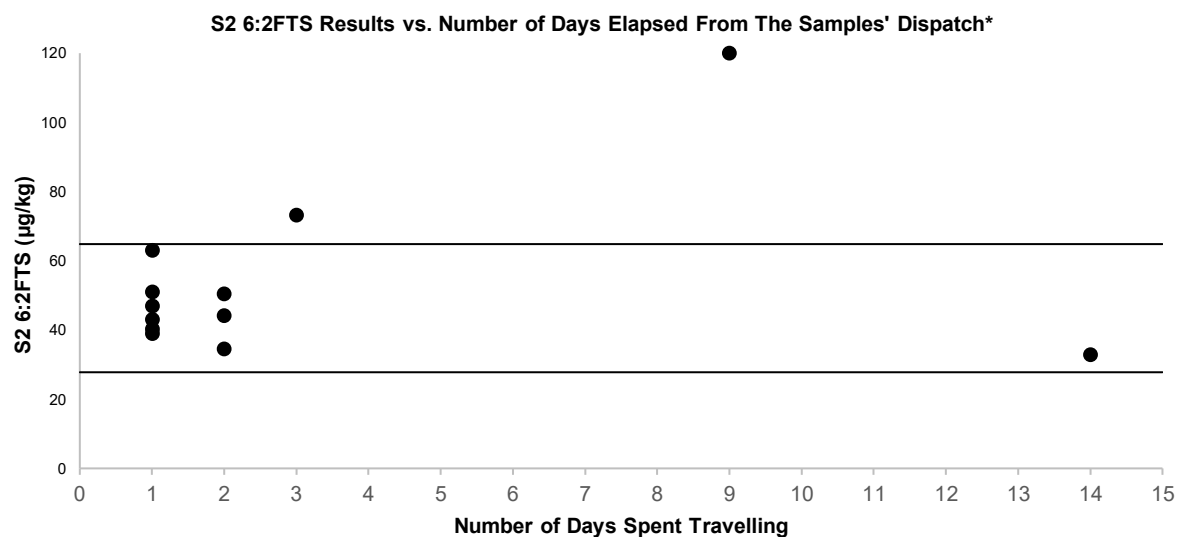
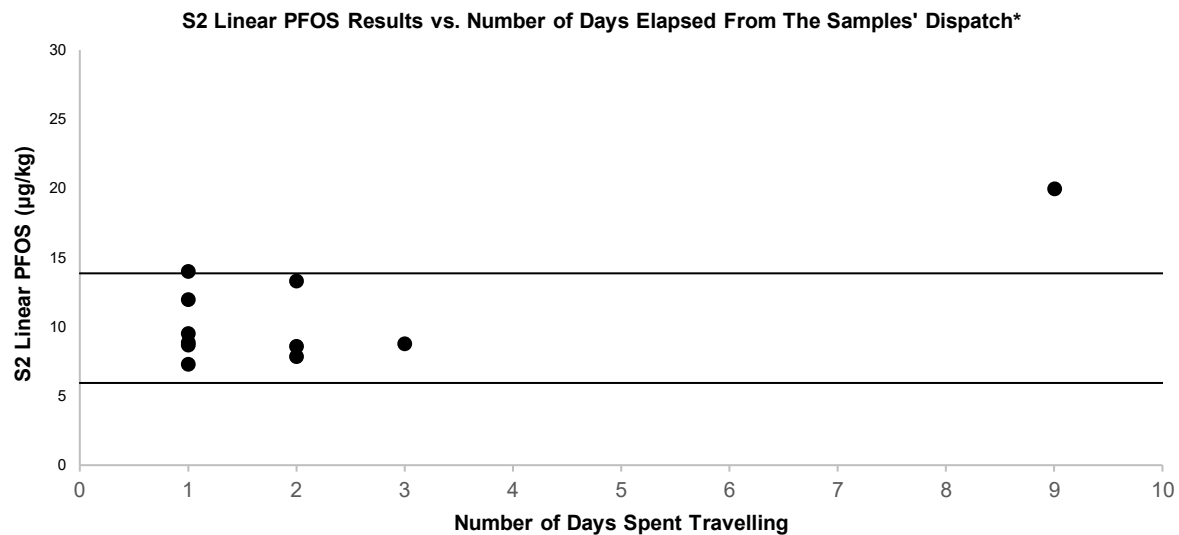
*Laboratory 8 excluded. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2.

Figure 42 Results vs Days Spent in Transit (continued)



*Laboratory 8 excluded. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2.

Figure 42 Results vs Days Spent in Transit (continued)



*Laboratory 8 excluded. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2.

Figure 42 Results vs Days Spent in Transit (continued)

Results from samples analysed before the samples' dispatch (homogeneity value-HV), were in good agreement with the results from samples analysed at the end of the study and the assigned value within their stated uncertainties (Figure 43).

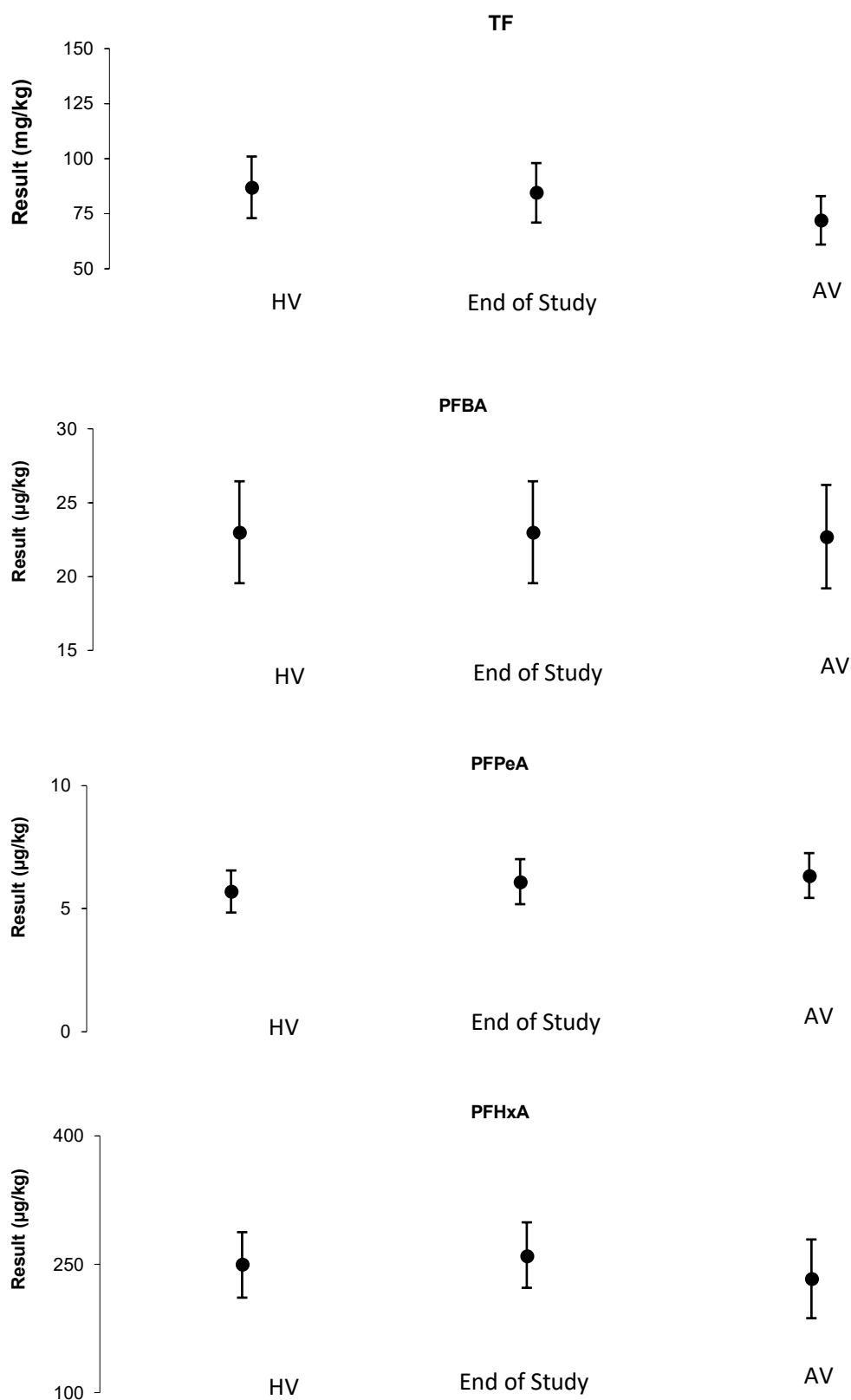


Figure 43 Stability Study Results

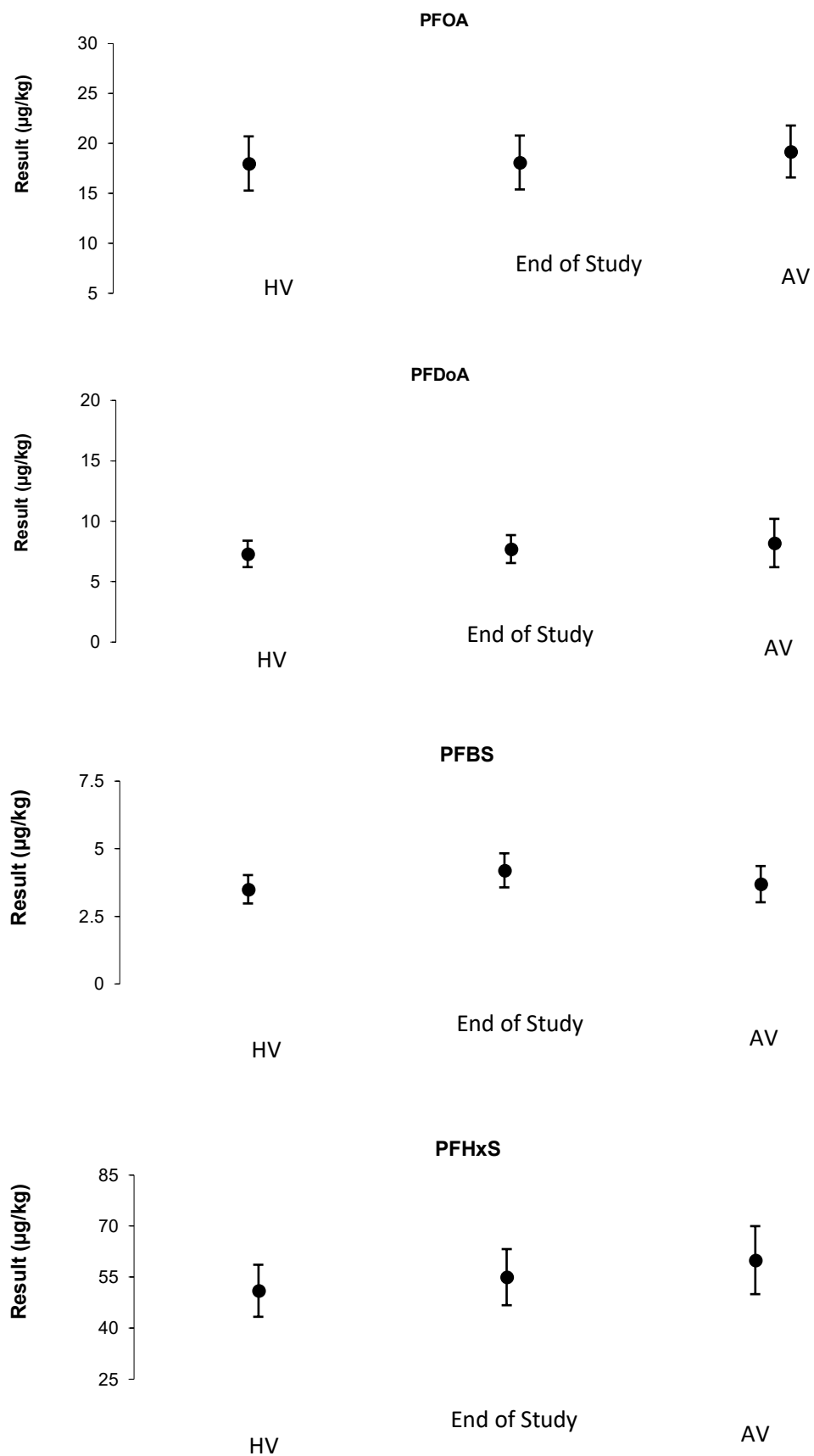


Figure 43 Stability Study Results (continued)

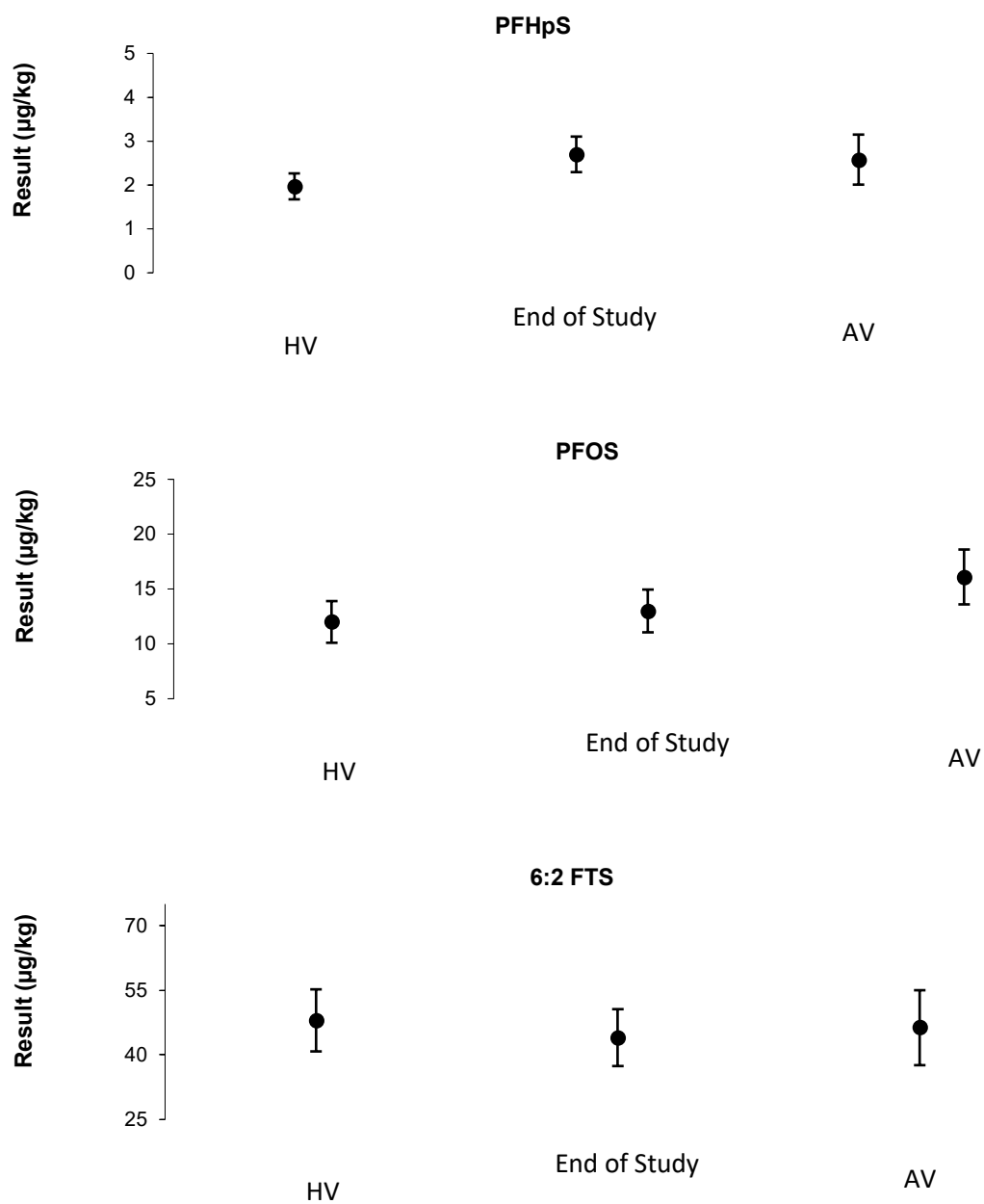


Figure 43 Stability Study Results (continued)

APPENDIX 2- ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, Z-SCORE AND E_N-SCORE CALCULATIONS

A2.1 Robust Average and Associated Uncertainty

The robust average was calculated using the procedure described in ISO 13528:2015 Annex C.⁵ The uncertainty was evaluated as:

$$u_{\text{rob average}} = 1.25 \times S_{\text{rob average}} / \sqrt{p} \quad \text{Equation 4}$$

where:

$u_{\text{rob average}}$ is the standard uncertainty of the robust average

$S_{\text{rob average}}$ is the standard deviation of the robust average

p is the number of results

The expanded uncertainty ($U_{\text{rob average}}$) 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 41.

Table 41 Uncertainty Evaluation for PFOS in Sample S2

No. results (p)*	13
Robust Average	16.1 µg/kg
$S_{\text{rob av}}$	3.5 µg/kg
$u_{\text{rob av}}$	1.2 µg/kg
k	2
$U_{\text{rob av}}$	2.5 µg/kg

*Outliers excluded

Therefore, the robust average for PFOS in Sample S2 is **16.1 ± 2.5 µg/kg**.

A2.2 z-Score and E_N-Score Calculations

For each participant's result, a z-score and E_N-score are calculated according to Equations 2 and 3 respectively (see page 12).

A worked example is set out below in Table 42.

Table 42 z-Score and E_N-Score for Sample S2 PFOS Result Reported by Laboratory 15

Participant Result (µg/kg)	Assigned Value (µg/kg)	Standard Deviation for Proficiency Assessment	z-Score	E _N -Score
15.6 ± 4.67	16.1 ± 2.5	20% as PCV, or: 0.2 × 16.1 = 3.22 µg/kg	$z\text{-Score} = \frac{15.6 - 16.1}{3.22} = -0.16$	$E_N\text{-Score} = \frac{15.6 - 16.1}{\sqrt{4.67^2 + 2.5^2}} = -0.09$

APPENDIX 3 - ADDITIONAL ANALYTES

Table 43 Additional Analytes

Lab. Code	Sample	Analyte	Result (µg/kg)	Uncertainty (µg/kg)	Recovery (%)
1	S2	PFHpA	0.069	0.018	47.6
		PFUdA	0.037	0.013	25
		PFTTrDA	0.028	0.01	50.8
		PFDS	0.01	0.005	73.7
		EtFOSAA	0.023	0.001	49.5
		8:2FTS	0.037	0.005	64.4
3	S2	PFHpA	0.07	0.0045	31
		PFUdA	0.086	0.0055	23
		8:2FTS	0.038	0.001	25
5	S2	PFUdA	0.13	NR	101
8	S2	PFHpA	71.06	NR	NR
		PFUdA	83.49	NR	NR
		PFTTrDA	95.5	NR	NR
		PFTeDA	62.61	NR	NR
		PFHxDA	68.32	NR	NR
		PFDS	68.95	NR	NR
		PFUdS	72.12	NR	NR
		MeFOSE	107.30	NR	NR
		EtFOSE	139.97	NR	NR
		8:2FTS	66.22	NR	NR
		10:2FTS	51.24	NR	NR
		5:3FTCA	72.76	NR	NR

Lab. Code	Sample	Analyte	Result (µg/kg)	Uncertainty (µg/kg)	Recovery (%)
8	S2	7:3FTCA	87.76	NR	NR
		GenX	379.60	NR	NR
		PFMPA	70.28	NR	NR
		PFEESA	4508.58	NR	NR
		PFECHS	361.96	NR	NR
12	S2	NFDHA	1.4	0.25	NR
13	S2	PFHpA	0.0909	0.020907	114
14	S2	PFODA	0.11	0.05	NR
		FOUEA	0.11	0.05	NR
		PFEESA	4.4	1.98	NR
17	S2	MeFOSE	0.368	0.069	57
		EtFOSE	0.484	0.117	39

APPENDIX 4 - FALSE NEGATIVES

Table 44 False Negatives

Lab. Code	Sample	Analyte	Units	Assigned Value	Spiked Value	Reported Result*
1	S1	Total Fluorine	mg/kg	72	Not Spiked	NR
	S2	PFPeA	µg/kg	6.35	5.98	<0.584
4	S2	PFBA	µg/kg	22.7	25.3	NR
		PFPeA	µg/kg	6.35	5.98	NR
		PFHxA	µg/kg	233	264	NR
		PFOA	µg/kg	19.2	20.1	NR
		PFDA	µg/kg	1.06	1.50	NR
		PFDoA	µg/kg	8.2	15.0	NR
		PFBS	µg/kg	3.69	4.12	NR
		PFHxS	µg/kg	60	60.1	NR
		PFHxS_L	µg/kg	61	60.1	NR
		PFHpS	µg/kg	2.58	Not Spiked	NR
		PFOS	µg/kg	16.1	14.3	NR
		PFOS_L	µg/kg	9.9	9.66	NR
		6:2FTS	µg/kg	46.3	50.1	NR
		6:2diPAP	µg/kg	0.71	Not Spiked	NR
6	S2	PFDA	µg/kg	1.06	1.50	<1
7	S2	PFBA	µg/kg	22.7	25.3	<10
9	S2	PFBA	µg/kg	22.7	25.3	NR
		PFPeA	µg/kg	6.35	5.98	NR
		PFDA	µg/kg	1.06	1.50	NR
		PFDoA	µg/kg	8.2	15.0	NR
		PFBS	µg/kg	3.69	4.12	NR

Lab. Code	Sample	Analyte	Units	Assigned Value	Spiked Value	Reported Result*
9	S2	PFHxS_L	µg/kg	61	60.1	NR
		PFHpS	µg/kg	2.58	Not Spiked	NR
		PFOS_L	µg/kg	9.9	9.66	NR
		6:2FTS	µg/kg	46.3	50.1	NR
		6:2diPAP	µg/kg	0.71	Not Spiked	NR
13	S1	Total Fluorine	mg/kg	72	Not Spiked	<0.140

*Results reported as NR may or may not be false negatives, depending on the participant's actual LOR.

APPENDIX 5 – PARTICIPANTS’ TEST METHODS FOR TOTAL FLUORINE SAMPLES

Participants’ methods for analysis of Total Fluorine in Samples S1 and S3 are presented in Tables 45 to 46.

Table 45 Participants’ Total Fluorine Methodology

Lab. Code	Method	Method Reference
2*	Combustion Ion Chromatography	In house combustion IC method
4	Combustion Ion Chromatography	In-House
7*	Combustion Ion Chromatography	ASTM D7359 - 23 Ref BS EN 14582-2016
8	Combustion Ion Chromatography	Internal Method Based on ASTM method D7359-14a
10*	Combustion Ion Chromatography	
11	Combustion Ion Chromatography	No. In-house
13*	Combustion Ion Chromatography	Lab SOP
14*	Combustion Ion Chromatography	EN 17813
15	Combustion Ion Chromatography	ASTM D7359-18 Standard Test Method for Total Fluorine, Chlorine and Sulfur in Aromatic Hydrocarbons and their Mixtures by Oxidative Pyrohydrolytic combustion followed by Ion Chromatography Detection (Combustion Ion Chromatography-CIC)

*Additional Information in Table 46.

Table 46 Participants’ Total Fluorine Methodology – Additional Information

Lab. Code	Total Fluorine Additional Information
2	The samples are weighed (in threefold) in alumina boats. The alumina boats with samples are introduced automatically in the oven of the system. The samples are burned in a gas mixture of argon/oxygen and water vapour. The released gases are trapped in an absorption solution. A little part of the absorption solution is introduced into a mobile phase via an injection loop. Subsequently the sample is pumped with the eluent through an analytical ion-exchange column. Due to differences in affinity of the sample ions towards the mobile phase and ion-exchange material the ions will travel with different velocities through the analytical column. As a result, the various ions are separated in time and detected one by one with a conductivity detector. Quantification is performed by comparison of the measured peak areas with those produced by standard solutions. The carbon samples are combusted a second time because after the first combustion still some F is presented in the boats. The reported F is the sum of F detected after the 2 combustions
7	LOQ 10 mg/kg
10	Run according to manufacturer method
13	S1 & S3: Analyte was not detected above the Laboratory MDL
14	Standard for halogen (including Fluor eg Total Fluor) in environmental matrices by CIC

APPENDIX 6 – PARTICIPANTS’ TEST METHODS FOR PFAS SAMPLE

Participants’ methods for Sample S2 cardboard are presented in Tables 47 to 68.

Table 47 Participants’ PFAS Methodology – Extraction

Lab. Code	Entire Container Used?	Container Rinsed?	If Not Entire Container, Sample Amount Used (g)	Labelled Standard Added Before Extraction?	Labelled Standard Directly into Container?	Equilibration Time (min)	Extraction Solvent	Extraction Technique	If Staggered Extraction, Number of Steps	Extraction Time (min)	Carbon Cleanup?	Extract Concentration Temperature (°C)	If Staggered Concentration, Number of Steps	Extract Concentration Time (min)	Concentrator/Evaporator Tube Rinsed?	Cleanup	Elution Solvent	Final pH Adjustment	Labelled Standard Added Before Instrument Analysis?
1	Yes	Yes		Yes	No	30 min	ACN	Solid-Liquid Extraction (vortexed/sonicated/shook and centrifuged)	3	90 min	No	Room Temperature				Solid-Phase Extraction	NaOH/MeOH	No	Yes
3	Yes	Yes		Yes	No	5	NH ₄ OH/MeOH	Solid-Liquid Extraction (vortexed/sonicated/shook and centrifuged)	2	40	Yes	40			No	None		No	Yes
5	Yes	Yes	NA	Yes	No	NA	DI, 3% AcOH; MeOH, 0.3% NH ₃	Solid-Liquid Extraction (vortexed/sonicated/shook and centrifuged)	2	60	No	NA	NA	NA	NA	NA	NA	NA	NA
7	Yes	Yes	NA	No	No	NA	NaOH/MeOH	Solid-Liquid Extraction (vortexed/sonicated/shook and centrifuged)	NA	60	NA	60	NA	60	NA	None	NA	Yes	Yes

Lab. Code	Entire Container Used?	Container Rinsed?	If Not Entire Container, Sample Amount Used (g)	Labelled Standard Added Before Extraction?	Labelled Standard Directly into Container?	Equilibration Time (min)	Extraction Solvent	Extraction Technique	If Staggered Extraction, Number of Steps	Extraction Time (min)	Carbon Cleanup?	Extract Concentration Temperature (°C)	If Staggered Concentration, Number of Steps	Extract Concentration Time (min)	Concentrator/Evaporator Tube Rinsed?	Cleanup	Elution Solvent	Final pH Adjustment	Labelled Standard Added Before Instrument Analysis?
8	No	No	1g				MeOH, 0.3% NH ₃	Solid-Liquid Extraction (vortexed/sonicated/shook and centrifuged)		60	No							NA	
9	No	No	1g	No	No		MeOH	Solid-Liquid Extraction (vortexed/sonicated/shook and centrifuged)		120 min	No	60°C			No	None			Yes
11	Yes	Yes	NA	Yes	Yes	15	Basic MeOH	Alkaline Digestion	NA	90	No	NA	NA	NA	NA	None	NA	Yes	No
12	Yes	Yes		Yes	No		KOH/MeOH	Solid-Liquid Extraction (vortexed/sonicated/shook and centrifuged)	2	120	No	40		45	NA	None	NA	No	Yes
13	no	no	2g	yes	no	30 min	0.4% (w/v) methanolic potassium hydroxide	shake sonic bath		60 min	Yes	NA	NA	NA	NA	Solid-Phase Extraction	1% NH ₄ OH :MeOH	Yes	yes

Lab. Code	Entire Container Used?	Container Rinsed?	If Not Entire Container, Sample Amount Used (g)	Labelled Standard Added Before Extraction?	Labelled Standard Directly into Container?	Equilibration Time (min)	Extraction Solvent	Extraction Technique	If Staggered Extraction, Number of Steps	Extraction Time (min)	Carbon Cleanup?	Extract Concentration Temperature (°C)	If Staggered Concentration, Number of Steps	Extract Concentration Time (min)	Concentrator/Evaporator Tube Rinsed?	Cleanup	Elution Solvent	Final pH Adjustment	Labelled Standard Added Before Instrument Analysis?
14*	Yes	Yes		Yes	Yes	30 min	KOH/ MeOH	Solid-Liquid Extraction (vortexed/ sonicated/ shook and centrifuged)	1	60	No	45	1	40	No	Solid-Phase Extraction	NH ₄ OH /MeOH	No	No
15*	Yes	Yes		Yes	Yes	30	80% MeOH	Solid-Liquid Extraction (vortexed/ sonicated/ shook and centrifuged)	1	30	No			0	No	Filtration	NA	No	No
16	Yes	Yes		Yes	Yes		ACN/ MeOH in 0.1% NH ₄ OH	QuEChERS		90 min	No				NA		ACN/ MeOH in 0.1% NH ₄ OH	No	No
17	No	No	3.01				KOH/ MeOH	Alkaline Digestion		60		Room Temperature				Solid-Phase Extraction			

*Additional Information in Table 48

Table 48 Participants' PFAS Methodology – Extraction Additional Information

Lab. Code	Extraction Additional Information
14	Dilution during the extraction process due to matrix difficulties
15	Due to the matrix of the sample, Internal standard, Spike and solvent amount had to be doubled

Table 49 Participants' PFAS Methodology – Instrumental Technique and Analysis

Lab. Code	Instrument	Dilution Factor	Blank Correction?
1	LC-MS/MS		Yes
3	LC-MSMS or LC-QQQ	No	No
5*	LC-MSMS or LC-QQQ	1:10	Yes
7	LC-MSMS or LC-QQQ	No	Yes
8	LC-MSMS or LC-QQQ	1in10	No
9	LC-MSMS or LC-QQQ	2	Yes
11	LC-MSMS or LC-QQQ	5	No
12	LC-MSMS or LC-QQQ		No
13*	LC-MSMS or LC-QQQ	DF1/DF10	no
14	LC-MSMS or LC-QQQ	No	No
15	LC-MSMS or LC-QQQ	4.16667	No
16	LC-MSMS or LC-QQQ	No	No
17	LC-MSMS or LC-QQQ	No	No

*Additional Information in Table 50.

Table 50 Participants' PFAS Methodology – Instrumental Technique Additional Information

Lab. Code	Instrumental Technique Additional Information
5	A 1:10 dilution was applied in the case of any analytes where the concentration was greater than the calibration range, or where recovery of the labelled std was outside of acceptance criteria.
13	Isotope dilution calibration is used.

Table 51 Participants' PFAS Methodology – Labelled Standards

Lab. Code	Labelled Standard Source	Secondary Source Used to Check Standard?	Recovery Correction?	Standard Method?	Labelled Standards Additional Information
1	Wellington		Yes	No	
3	Wellington	No	Yes	Isotopic dilution	
5	Wellington Laboratories	No	No		
7	Wellington	LGC	No	ISO 17681-1: 2025	
8	Wellington		No		
9	Wellington	Yes	Yes	EN17681-1:2022	
11	Wellington	Yes	Yes	No. In-house	
12	Wellington		Yes	USEPA 537	
13	Wellington	yes	yes	1633A	
14	YES	YES	YES	NO	
15	Wellington Labs	Yes. ICC standard	No	In-House	
16	Wellington Laboratories				
17	Wellington (Cambridge Isotope Labs)	Yes	Yes		

Table 52 Labelled Standards for PFBA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFBA	13C8-PFOA
2	NS	NS
3	13C4-PFBA	13C3-PFBA
4		
5	PFBA-13C4; PFBA-13C3	
6		
7		
8		
9		
10	NS	NS
11	13C4-PFBA	NA
12	MPFBA	M3PFBA
13	13C4-PFBA	13C3-PFBA
14	13C-PFBA	None
15	13C4-PFBA	
16	13C4-PFBA	
17	13C4 PFBA	

Table 53 Labelled Standards for PFPeA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C5-PFPeA	13C8-PFOA
2	NS	NS
3	13C4-PFPeA	13C5-PFPeA
4		
5	PFPeA-13C5	
6		
7		
8		
9		
10	NS	NS
11	13C3-PFPeA	NA
12	M5PFPeA	M3PFBA
13	13C5-PFPeA	13C2-PFHxA
14	13C-PFPeA	None
15	13C5-PFPeA	
16	13C5-PFPeA	
17	13C5 PFPeA	13C2 PFOA

Table 54 Labelled Standards for PFHxA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C5-PFHxA	13C8-PFOA
2	NS	NS
3	13C2-PFHxA	13C5-PFPeA
4		
5	PFHxA-13C5	
6		
7		
8		
9		13C4PFOA
10	NS	NS
11	13C2-PFHxA	NA
12	M5PFHxA	M3PFBA
13	13C5-PFHxA	13C2-PFHxA
14	13C-PFHxA	None
15	13C2-PFHxA	
16	13C5-PFHxA	
17	13C2 PFHxA	

Table 55 Labelled Standards for PFOA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	13C8-PFOA
2	NS	NS
3	13C4-PFOA	13C8-PFOA
4		
5	PFOA-13C4; PFOA-13C2	
6		
7		
8		
9		13C4PFOA
10	NS	NS
11	13C4-PFOA	NA
12	M8PFOA	M2PFOA
13	13C8-PFOA	13C4-PFOA
14	13C-PFOA	None
15	13C8-PFOA	
16	13C8-PFOA	
17	13C4 PFOA	

Table 56 Labelled Standards for PFNA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C9-PFNA	13C5-PFNA
2	NS	NS
3	13C5-PFNA	13C8-PFOA
4		
5	PFNA-13C9; PFNA-13C5	
6		
7		
8		
9		
10	NS	NS
11	13C5-PFNA	NA
12	M9PFNA	M2PFOA
13	13C9-PFNA	13C5-PFNA
14	13C-PFNA	None
15	13C5-PFNA	
16	13C5-PFNA	
17	13C5 PFNA	

Table 57 Labelled Standards for PFDA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C2-PFDA	13C5-PFNA
2	NS	NS
3	13C2-PFDA	13C8-PFOA
4		
5	PFDA-13C6; PFDA-13C2	
6		
7		
8		
9		
10	NS	NS
11	13C2-PFDA	NA
12	M6PFDA	MPFDA
13	13C6-PFDA	13C2-PFDA
14	13C-PFDA	None
15	13C6-PFDA	
16	13C6-PFDA	
17	13C2 PFDA	

Table 58 Labelled Standards for PFDoA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C2-PFDoA	13C5-PFNA
2	NS	NS
3	13C2-PFDoA	13C8-PFOA
4		
5	PFDoA-13C2	
6		
7		
8		
9		
10	NS	NS
11	13C2-PFDoDA	NA
12	MPFDoA	MPFDA
13	13C2-PFDoA	13C2-PFDA
14	13C-PFDoDA	None
15	13C2-PFDoA	
16	13C2-PFDoDA	
17	13C2 PFDoA	

Table 59 Labelled Standards for PFBS

Lab. Code	Before Extraction	Before Instrument Analysis
1	18O2-PFHxS	18O2-PFOS
2	NS	NS
3	13C3-PFBS	13C3-PFHxS
4		
5	PFBS-13C3	
6		
7		
8		
9		
10	NS	NS
11	13C3-PFBS	NA
12	M3PFBS	MPFDA
13	13C3-PFBS	18O2-PFHxS
14	13C-PFBS	None
15	13C3-PFBS	
16	13C3-PFBS	
17	13C3 PFBS	

Table 60 Labelled Standards for PFHxS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	NS	NS
3	18O2-PFHxS	13C3-PFHxS
4		
5	PFHxS-13C3; PFHxS-18O2	
6		
7		
8		
9		13C4PFOS
10	NS	NS
11	18O2-PFHxS	NA
12	M3PFHxS	MPFDA
13	13C3-PFHxS	18O2-PFHxS
14	13C-PFHxS	None
15	18O2-PFHxS	
16	16O2-PFHxS	
17	18O2 PFHxS	

Table 61 Labelled Standards for PFHxS_L

Lab. Code	Before Extraction	Before Instrument Analysis
1	18O2-PFHxS	18O2-PFOS
2	NS	NS
3	18O2-PFHxS	13C3-PFHxS
4		
5	PFHxS-13C3; PFHxS-18O2	
6		
7		
8		
9		
10	NS	NS
11	18O2-PFHxS	NA
12	M3PFHxS	MPFDA
13		
14	13C-PFHxS	None
15	18O2-PFHxS	
16	Not applicable	
17	18O2 PFHxS	

Table 62 Labelled Standards for PFHpS

Lab. Code	Before Extraction	Before Instrument Analysis
1	18O2-PFHxS	18O2-PFOS
2	NS	NS
3	18O2-PFHxS	13C3-PFHxS
4		
5		
6		
7		
8		
9		
10	NS	NS
11	13C4-PFOS	NA
12	M3PFHxS	MPFDA
13	13C8-PFOS	13C4-PFOS
14	13C-PFOS	None
15	18O2-PFHxS	
16	13C8-PFOS	
17	13C4 PFOS	

Table 63 Labelled Standards for PFOS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOS	18O2-PFOS
2	NS	NS
3	13C4-PFOS	13C8-PFOS
4		
5	PFOS-13C8; PFOS-13C4	
6		
7		
8		
9		13C4PFOS
10	NS	NS
11	13C4-PFOS	NA
12	M8PFOS	MPFOS
13	13C8-PFOS	13C4-PFOS
14	13C-PFOS	None
15	13C8-PFOS	
16	13C4-PFOS	
17	13C4 PFOS	

Table 64 Labelled Standards for PFOS_L

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOS	18O2-PFOS
2	NS	NS
3	13C4-PFOS	13C8-PFOS
4		
5	PFOS-13C8; PFOS-13C4	
6		
7		
8		
9		
10	NS	NS
11	13C4-PFOS	NA
12	M8PFOS	MPFOS
13		
14	13C-PFOS	None
15	13C8-PFOS	
16	13C8-PFOS	
17	13C4 PFOS	

Table 65 Labelled Standards for 6:2FTS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C2-6:2 FTS	13C2-8:2 FTS
2	NS	NS
3	13C2-6:2 FTS	
4		
5	6:2FTS-13C2	
6		
7		
8		
9		
10	NS	NS
11	13C2,12C6 6:2-FTS	NA
12	M2-6:2 FTS	MPFOS
13	13C2-6:2-FTS	18O2-PFHxS
14	13C-8:2 FTS	None
15	13C2-6-2 FTS	
16	13C2-6:2 FTS	
17	13C2 6:2 FTS	

Table 66 Labelled Standards for 6:2diPAP

Lab. Code	Before Extraction	Before Instrument Analysis
1	NT	NT
2	NS	NS
3	13C4-6:2 DiPAP	
4		
5		
6		
7		
8		
9		
10	NS	NS
11	NT	NT
12	M4-6:2diPAP	M2PFOA
13		
14	13C-6:2 DiPAP	None
15		
16	13C2-6:2diPAP	
17	13C4-6:2 Fluorotelomer phosphate diester	

Table 67 Labelled Standards for 8:2diPAP

Lab. Code	Before Extraction	Before Instrument Analysis
1	NT	NT
2	NS	NS
3	13C4-8:2 diPAP	
4		
5		
6		
7		
8		
9		
10	NS	NS
11	NT	NT
12	M4-8:2diPAP	M2PFOA
13		
14	13C-6:2 DiPAP	None
15		
16	13C2-8:2diPAP	
17	13C4-8:2 Fluorotelomer phosphate diester	

Table 68 Participant Methodology for PFAS Samples– Additional Information

Lab. Code	Additional Information
13	Linear only data was not quantified by the laboratory

APPENDIX 7 – ACRONYMS AND ABBREVIATIONS

6:2FTS	1H, 1H, 2H, 2H-perfluorooctane sulfonate
8:2diPAP	Bisperfluorooctyl phosphate
ACN	Acetonitrile
AQA	Analytical and Quality Assurance
AV	Assigned Value
CITAC	Co-Operation on International Traceability in Analytical Chemistry
CV	Coefficient of Variation
EPA	Environment Protection Authority
GUM	Guide for Uncertainty Measurement
HV	Homogeneity Value
ISO	International Standards Organisation
LC-MSMS	Liquid Chromatography with Tandem Mass Spectrometry
LOR	Limit of Reporting
Max	Maximum value in a set of results
Md	Median
MeOH	Methanol
MeOH/Base	Base modified methanol
Min	Minimum value in a set of results
MU	Measurement Uncertainty
NATA	National Association of Testing Authorities, Australia
NMI	National Measurement Institute (of Australia)
NR	Not Reported
NS	Not Sent
NT	Not Tested
PFAS	Per- and polyfluoroalkyl substances
PFBA	Perfluoro-n-butanoic acid
PFBS	Potassium perfluoro-1-butanesulfonate
PFCA	Perfluorinated carboxylic acids
PFDA	Perfluoro-n-decanoic acid
PFDoA	Perfluorododecanoic acid
PFHpS	Perfluoroheptane sulfonate
PFHxA	Perfluoro-n-hexanoic acid
PFHxS	Potassium perfluorohexanesulfonate
PFHxS_L	Potassium perfluorohexanesulfonate linear
PFNA	Perfluoro-n-nonanoic acid
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctane sulfonate
PFOS_L	Perfluorooctane sulfonate linear

PFPeA	Perfluoro-n-pentanoic acid
PT	Proficiency Test
QC	Quality Control
QQQ	Triple Quadrupole (mass spectrometry)
QuEChERS	Quick, Easy, Cheap, Effective, Rugged and Safe extraction method
RA	Robust Average
RM	Reference Material
Robust CV	Robust Coefficient of Variation
Robust SD	Robust Standard Deviation
RT	Room Temperature
SD	Standard Deviation
SLE	Solid-Liquid Extraction
SPE	Solid Phase Extraction
SPLP	Synthetic Precipitation Leaching Procedure
SDPA	Standard Deviation for Proficiency Assessment
SS	Spiked Samples
SV	Spiked or formulated concentration of a PT sample (Spike Value)
Target SD	Standard deviation for proficiency assessment
USEPA	United States Environmental Protection Agency

END OF REPORT