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National Measurement Institute

Proficiency Test Final Report
AQA 21-08
PFAS in Food

January 2022

ACKNOWLEDGMENTS

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

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

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SUMMARY

AQA 21-08 PFAS in Food commenced in July 2021. Eighteen laboratories registered to participate, and seventeen participants submitted results.

The sample set consisted of one spiked beef meat sample (Sample S1) and one spiked celery sample (Sample S2). The per- and polyfluoroalkyl substances (PFAS) analytes assessed in this study were: PFBS, PFPeS, PFHxS, PFHpS, PFOS, PFDS, PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUdA, PFOSA, MeFOSE, 8:2 FTS, 10:2 FTS and GenX.

Of 544 results, 437 numeric results (80%) were submitted. Sixty-six results were a 'less than' value (< *x*) or Not Reported (NR), and 41 results were Not Tested (NT).

The assigned values for all scored analytes were the robust averages of participants' results, and associated uncertainties were estimated from the robust standard deviations.

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.

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

- *Assess the ability of participants to correctly identify PFAS in food matrices.*

Fifteen participants analysed both matrices, and two participants analysed Sample S2 Celery only. Laboratories **7** and **16** reported numeric results for all scored analytes in this study.

Seven participants did not report results for analytes that they tested for and were spiked into the samples (total of 31 results), while eight participants reported analytes that were not spiked into the samples (total of 24 results).

- *Compare the performances of participants and assess their accuracy in the measurement of PFAS in food matrices.*

Of 422 z-scores, 383 (91%) returned $|z| \leq 2.0$, indicating a satisfactory performance.

Of 422 E_n-scores, 331 (78%) returned $|E_n| \leq 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Laboratory **7** returned satisfactory z-scores for all scored analytes (32). Laboratory **3** analysed S2 celery only and returned satisfactory z-scores for all scored analytes in this matrix (16).

- *Evaluate the participants' test methods for PFAS in food analysis.*

Participants used a variety of methods for extraction and analysis. No significant bias due to methodology was evident. Participants should take care to report results on the correct basis.

- *Develop the practical application of traceability and measurement uncertainty.*

Of 437 numeric results for analytes of interest in this study, 436 were reported with an associated expanded measurement uncertainty, with a variety of procedures used to estimate uncertainty. The magnitude of the reported measurement uncertainties for spiked analytes in this study was within the range 0.05% to 100% of the reported value.

- *Compare the performance of participants with their past performance.*

The proportion of total possible results being reported by participants as numeric results has remained fairly consistent, even with the increased number of PFAS analytes over the last few studies, indicating that participants have the capacity to analyse a wide range of PFAS. Proportions of satisfactory z-scores and E_n-scores have remained relatively high this year, indicating good consensus of reported results.

- *Produce materials that can be used in method validation and as control samples*.

The test samples of this proficiency study are homogeneous and are well characterised. Surplus samples are available for purchase from NMI and can be used for quality control and method validation purposes.

1 INTRODUCTION

1.1 NMI Proficiency Testing Program

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

Proficiency testing (PT) is 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:

- pesticide residues in fruit and vegetables, soil and water;
- petroleum hydrocarbons in soil and water;
- PFAS in soil, water, biota and food;
- inorganic analytes in soil, water, filters, food and pharmaceuticals;
- controlled drug assay, drugs in wipes and clandestine laboratory; and
- allergens in food.

1.2 Study Background

Per- and polyfluoroalkyl substances (PFAS) are chemicals found in industrial products such as fire-fighting foams and non-stick coatings. Their resistance to degradation and potential toxicity makes them a growing global environmental concern. These complex contaminants can be challenging to measure at the concentrations of interest and also near and/or at current guideline levels.

1.3 Study Aims

The aims of the study were to:

- assess the ability of participants to correctly identify PFAS in food matrices.
- compare the performances of participants and assess their accuracy in the measurement of PFAS in food matrices;
- evaluate the participants' test methods for PFAS in food analysis;
- develop the practical application of traceability and measurement uncertainty;
- compare the performance of participants with their past performance; and
- produce materials that can be used in method validation and as control samples.

The choice of test method was left to the participating laboratories.

1.4 Study Conduct

The conduct of NMI proficiency tests is described in the NMI Study Protocol for Proficiency Testing.[2] The statistical methods used are described in the NMI Chemical Proficiency Testing Statistical Manual.[3] These documents have been prepared with reference to ISO/IEC 17043:2010,[1] and The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.[4]

NMI is accredited by the National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043 as a provider of proficiency testing schemes. This study falls within the scope of NMI's accreditation.

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of the study was:

Invitation issued: 5 July 2021

Samples dispatched: 3 August 2021

Results due: 8 October 2021

Interim report issued: 19 October 2021

2.2 Participation and Laboratory Code

Eighteen laboratories registered to participate, and all participants were assigned a confidential laboratory code number for this study. Seventeen participants submitted results by the due date.

2.3 Selection of PFAS Analytes

A list of potential PFAS analytes spiked into the samples is presented in Table 1.

Table 1 Potential Spiked PFAS Analytes

PFBS	PFTrDS	PFDoA	N-EtFOSE
PFPeS	PFBA	PFTrDA	4:2 FTS
PFHxS	PFPeA	PFTeDA	6:2 FTS
PFHpS	PFHxA	PFOSA	8:2 FTS
PFOS	PFHpA	N-MeFOSA	10:2 FTS
PFNS	PFOA	N-EtFOSA	GenX
PFDS	PFNA	N-MeFOSAA	ADONA
PFUdS	PFDA	N-EtFOSAA	9Cl-PF3ONS
PFDoS	PFUdA	N-MeFOSE	11Cl-PF3OUdS

2.4 Test Material Preparation

Two samples were prepared in July 2021. Care was taken to avoid any PFAS contamination during sample preparation. The prepared samples were:

- Sample S1: Beef meat (5 g portions) spiked with 15 different PFAS analytes.
- Sample S2: Celery (40 g portions) spiked with 15 different PFAS analytes.

Details of spiked analytes and values are presented in Table 2. Participants were requested to report both the linear isomers and total values of PFHxS and PFOS.

Table 2 Spiked Values of Test Samples

Analyte	Sample S1 Beef Meat (µg/kg)	Sample S2 Celery (µg/kg)
PFBS	2.90	1.50
PFPeS	Not Spiked	11.3
PFHxS*	3.68	9.45
PFHxS (linear)	3.68	9.45
PFHpS	1.92	1.10
PFOS*	37.2	1.43

Analyte	Sample S1 Beef Meat (µg/kg)	Sample S2 Celery (µg/kg)
PFOS (linear)	37.2	1.43
PFDS	23.1	4.79
PFBA	17.5	2.48
PFPeA	0.970	0.746
PFHxA	0.677	15.0
PFHpA	1.95	0.795
PFOA	38.6	1.81
PFNA	4.36	0.990
PFDA	4.35	Not Spiked
PFUdA	0.387	Not Spiked
PFOSA	Not Spiked	3.53
MeFOSE	Not Spiked	4.00
8:2 FTS	9.26	Not Spiked
10:2 FTS	Not Spiked	3.39
GenX	7.28	Not Spiked

* Only linear standards were used for spiking.

Further sample preparation details can be found in Appendix 1.

2.5 Homogeneity and Stability of Test Materials

Beef meat was introduced as a matrix for the first time in this PFAS PT study. The homogeneity and stability testing for this sample are presented in Appendix 2. It was demonstrated to be sufficiently homogeneous and stable for the evaluation of participants' performance in this study.

No homogeneity or stability testing was conducted on the celery sample. This sample was prepared, packaged and stored using a process that has been demonstrated to produce homogeneous and stable samples for similar matrices in previous NMI PFAS PT studies.

Participants' robust averages for scored analytes were within 70% to 101% and 68% to 111% of the spiked values for Samples S1 and S2 respectively, which were similar to values observed in previous PFAS in food PT studies and provides support for the stability of these analytes.

2.6 Test Material Storage and Dispatch

After preparation, the test material were dispensed into sample tubes, labelled and shrink-wrapped. Prior to sample dispatch, the beef meat and celery samples were stored frozen at -80 °C and -20 °C respectively.

Samples were packed into insulated polystyrene foam boxes with cooler bricks and sent by courier on 3 August 2021.

The following items were packaged with the samples:

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

An Excel spreadsheet for the electronic reporting of results was emailed to all participants.

2.7 Instructions to Participants

Participants were instructed as follows:

- Quantitatively analyse the samples for PFAS, using your routine test method and report results in units of µg/kg on an as received basis.
    - For PFAS analytes that contain linear and branched isomers, report total (the sum of linear and branched isomers).
    - For PFOS and PFHxS you are asked to report total (the sum of linear and branched isomers) and linear (the linear isomers only).
- 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 µg/kg (e.g. 0.50 ± 0.02 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 estimates as required by the results sheet.
- If determined, report your internal standard percentage recovery. This will be presented in the report for information only.
- Return the completed results sheet by email (proficiency@measurement.gov.au) by 10 September 2021.

Due to delivery delays to participants, the results due date was extended to 8 October 2021 for all participants.

2.8 Interim Report

An interim report was emailed to all participants on 19 October 2021. The interim report was delayed due to extended delivery delays to a small number of participants.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Participants' Test Methods

Participants were requested to provide information about their methodology. Responses are presented in Appendix 3.

3.2 Basis of Participants' Measurement Uncertainty Estimates

Participants were requested to provide information about their basis of measurement uncertainty (MU). Responses are presented in Tables 3 and 4. Responses may be modified so that the participant cannot be identified.

Table 3 Basis of Participants' Uncertainty Estimate

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
1	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)	Duplicate analysis Instrument calibration	Instrument calibration Standard purity	ISO/GUM
3	Professional judgment	Control samples - SS Duplicate analysis Instrument calibration	Instrument calibration Laboratory bias from PT studies Recoveries of SS	Professional judgment
4	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM Duplicate analysis Instrument calibration	Recoveries of SS	NATA GAG Estimating and Reporting MU
6	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS Duplicate analysis Instrument calibration	CRM Instrument calibration Recoveries of SS Standard purity	NATA GAG Estimating and Reporting MU
7	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis Instrument calibration	Laboratory bias from PT studies Recoveries of SS	NATA GAG Estimating and Reporting MU
8				
10	Top Down - precision and estimates of the method and laboratory bias	Standard deviation from PT studies only		NMI Uncertainty Course
		Control samples - CRM Duplicate analysis Instrument calibration	CRM Laboratory bias from PT studies Recoveries of SS	
11	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS	CRM Recoveries of SS Standard purity	NATA GAG Estimating and Reporting MU
12		Standard deviation from PT studies only		
		Control samples - SS Duplicate analysis		
13	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM Duplicate analysis Instrument calibration	Recoveries of SS	Eurachem/CITAC Guide
14	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM Duplicate analysis	CRM Laboratory bias from PT studies Recoveries of SS	Nordtest Report TR537

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
15	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS	Recoveries of SS	Statistics and Chemometrics for Analytical Chemistry, Miller and Miller, 5th Edition
16	Standard deviation of replicate analyses multiplied by 2 or 3	Standard deviation from PT studies only		Eurachem/CITAC Guide
		Control samples - SS Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS	
17	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples Duplicate analysis Instrument calibration	Recoveries of SS	NATA GAG Estimating and Reporting MU
18	Standard deviation of replicate analyses multiplied by 2 or 3	Duplicate analysis	Recoveries of SS	NMI Uncertainty Course
19	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS	Recoveries of SS	NATA - Estimating and reporting MU of chemical test results.
20	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS	Recoveries of SS	NATA - Estimating and reporting MU of chemical test results.

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

Table 4 Uncertainty Estimate Additional Comments

Lab. Code	Uncertainty Estimate Comments
6	Recovery and uncertainty data given for analytes at method limit of reporting.
8	The measurement of uncertainty for all validated analytes in fish is 35%, so in lieu of accurate data for meat and celery this has been applied in this case.
12	standard deviation of triplicate measurements
15	Measurement Uncertainty (U) estimated from the standard deviation (u) of replicate recovery samples using the expression U = 2 x u. Procedure as set out in Statistics and Chemometrics for Analytical Chemistry, Miller and Miller, 5th Edition
16	Uncertainty calculated as 3xSD of replicate analysis.

3.3 Participants' Comments

Participants were invited to make comments on the samples, this PT study, or suggestions for future studies. Such feedback may be useful in improving future studies. Participants' comments are presented in Table 5, along with the study coordinator's response where applicable. Responses may be modified so that the participant cannot be identified.

Table 5 Participants' Comments

Lab. Code	Sample	Participant's Comments	Study Coordinator's Response
3	S1	due to custom problems it was not possible to receive the meat (S1)	There were some difficulties with the dispatch of the beef meat sample to international participants as some participants were not aware of their local authority's importing requirements. We will further clarify in future studies that participants should confirm any importing requirements with their local authority (e.g. import permits) at time of enrolment.
6	S2	Extra compounds detected < LOR PFDS at 2.8 ug/kg; N-MeFOSE at 3.0 ug/kg; 10:2FTS at 1.4 ug/kg	
8	S1	PFBA, L-PFOS and L-PFDS are all reading higher than the top of the curve. PFUda failed on ion ratio and so would not be reported to client.	
	S2	PFHxA is reading higher than top of curve	
	All	This method applied to fish is waiting on ISO/IEC 17025 accreditation. For this study we have applied the same method however it has not been validated in these matrices.	
10	S2	The sample had separated into liquid and solid phases after thawing. The sample was centrifuged and each phase extracted separately (with the liquid phase undergoing SPE via Water HLB Oasis), then the extracts recombined for analysis. This separation is not normally seen for vegetable samples as they are blended and subsampled onsite so do not separate before analysis.	
12	All	We use a technical mixture for PFOS as an analytical standard. It appears there is only linear PFOS in this sample, this may result in some bias compared to using just a linear isomer standard	
15	S1	NT = not tested	
	S2	10:2 FTS is not reported (NR) because of a poor recovery of our QC sample NT = not tested	

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are presented in Tables 6 to 39 with the summary statistics: robust average, median, mean, number of numeric results (N), maximum (Max.), minimum (Min.), robust standard deviation (Robust SD) and robust coefficient of variation (Robust CV). Bar charts of results and performance scores are presented in Figures 2 to 35. An example chart with interpretation guide is shown in Figure 1.

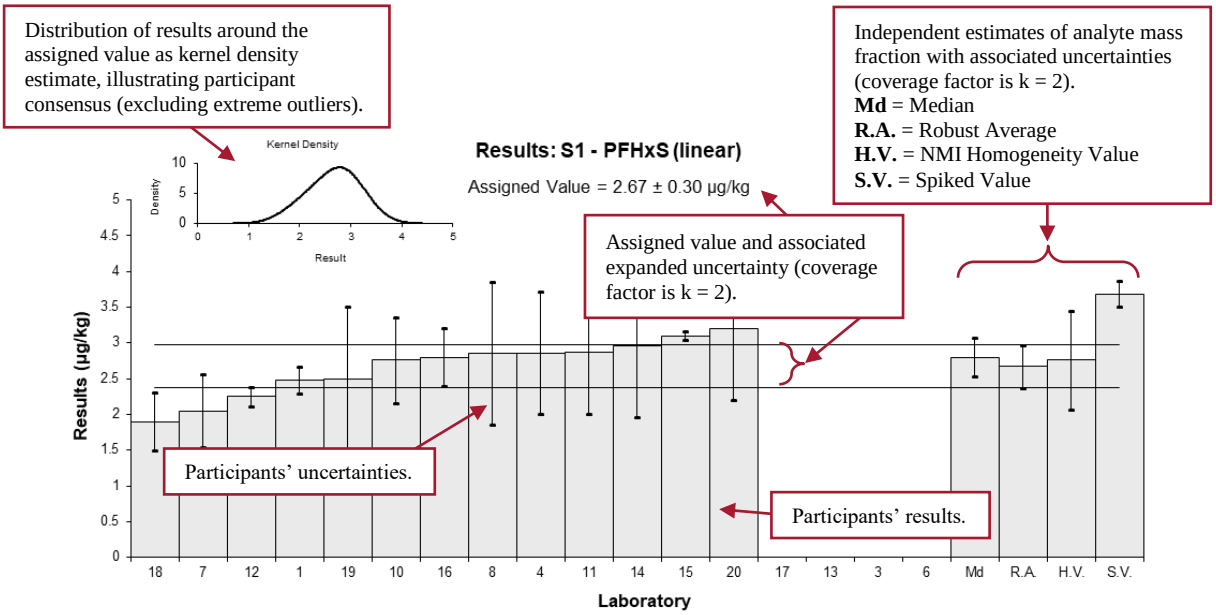


Figure 1 Guide to Presentation of Results

4.2 Outliers and Extreme Outliers

Outliers were results less than 50% and greater than 150% of the robust average, and these were removed before the calculation of the assigned value.[3,4] Extreme outliers were obvious blunders and gross errors, e.g. results reported with incorrect units or basis, or for a different analyte or sample, and such results were removed for the calculation of all summary statistics.[3,4]

4.3 Assigned Value

The assigned value is defined as the 'value attributed to a particular property of a proficiency test item'.[1] In this PT study, the property is the mass fraction of analytes in the samples. Assigned values in this study were the robust averages of participants' results and the expanded uncertainties were estimated from the associated robust SDs (Appendix 4).

4.4 Robust Average and Robust Between-Laboratory Coefficient of Variation

The robust averages and associated expanded MUs, and robust CVs (a measure of the variability of participants' results) were calculated using the procedure described in ISO 13528:2015.[5]

4.5 Performance Coefficient of Variation (PCV)

The performance coefficient of variation (PCV) is a fixed measure of the between-laboratory variation that in the judgement of the study coordinator would be expected from participants given the levels of analytes present. The PCV is not the CV of participants' results; it is set by the study coordinator and is based on the mass fraction of the analytes and experience from previous studies, and is supported by mathematical models such as the Thompson-Horwitz equation.[6] By setting a fixed and realistic value for the PCV, a participant's performance does not depend on the performance of other participants and can be compared from study to study.

4.6 Target Standard Deviation

The target standard deviation (σ) is the product of the assigned value (X) and the PCV, as presented in Equation 1.

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

4.7 z-Score

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

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

where:

z is z-score

χ is a participant's result

X is the assigned value

σ is the target standard deviation from Equation 1

For the absolute value of a z-score:

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

To account for potential low bias in consensus values due to inefficient methodologies, scores may be adjusted for a 'maximum acceptable concentration'. Additional information is given in Section 6.3.

4.8 E_n-Score

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

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

where:

E_n is E_n-score

χ is a participant's result

X is the assigned value

U_χ is the expanded uncertainty of the participant's result

U_X is the expanded uncertainty of the assigned value

For the absolute value of an E_n-score:

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

4.9 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC 17025 must establish and demonstrate the traceability and measurement uncertainty associated with their test results.[7]

Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.[8]

5 TABLES AND FIGURES

Table 6

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	PFBS
Units	µg/kg

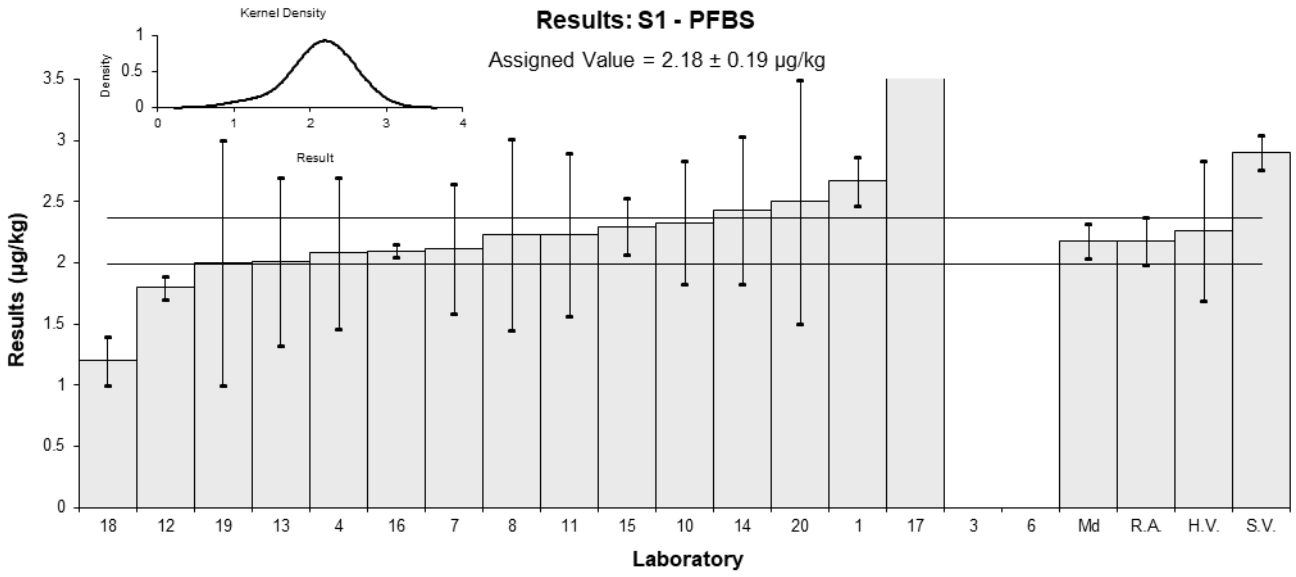
Participant Results

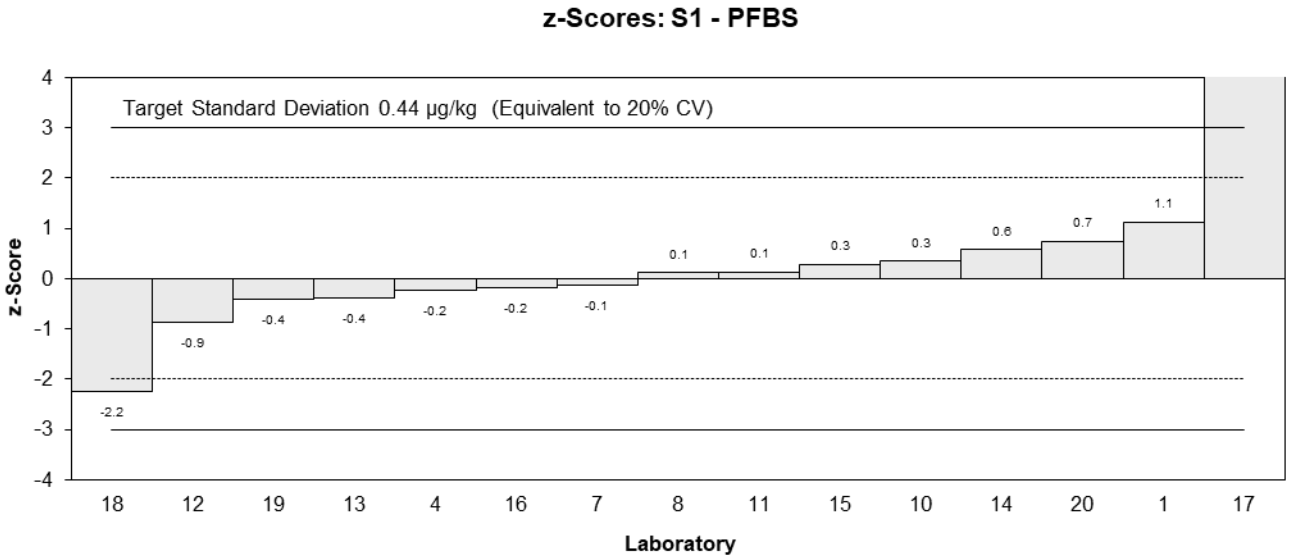
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	2.67	0.2	NR	1.12	1.78
3	NT	NT	NT		
4	2.08	0.62	137	-0.23	-0.15
6	NT	NT	NT		
7	2.12	0.53	80	-0.14	-0.11
8	2.23	0.780653035	144	0.11	0.06
10	2.33	0.5	61	0.34	0.28
11	2.233	0.67	85	0.12	0.08
12	1.8	0.093	78	-0.87	-1.80
13	2.01	0.69	135	-0.39	-0.24
14	2.43	0.600	95.2	0.57	0.40
15	2.3	0.23	106	0.28	0.40
16	2.1	0.05	88	-0.18	-0.41
17	10.84	0.27	88	19.86	26.23
18	1.2	0.2	NR	-2.25	-3.55
19	2	1	89	-0.41	-0.18
20	2.5	1	93	0.73	0.31

Statistics*

Assigned Value	2.18	0.19
Spike	2.90	0.14
Homogeneity Value	2.26	0.57
Robust Average	2.18	0.19
Median	2.18	0.14
Mean	2.14	
N	14	
Max.	2.67	
Min.	1.2	
Robust SD	0.28	
Robust CV	13%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Results: S1 - PFBS
Assigned Value = 2.18 ± 0.19 µg/kg
Kernel Density
Density
Result
Results (µg/kg)
Laboratory
18 12 19 13 4 16 7 8 11 15 10 14 20 1 17 3 6 Md R.A. H.V. S.V.


z-Scores: S1 - PFBS
Target Standard Deviation 0.44 µg/kg (Equivalent to 20% CV)
z-Score
Laboratory
18 12 19 13 4 16 7 8 11 15 10 14 20 1 17
-2.2 -0.9 -0.4 -0.4 -0.2 -0.2 -0.1 0.1 0.1 0.3 0.3 0.6 0.7 1.1

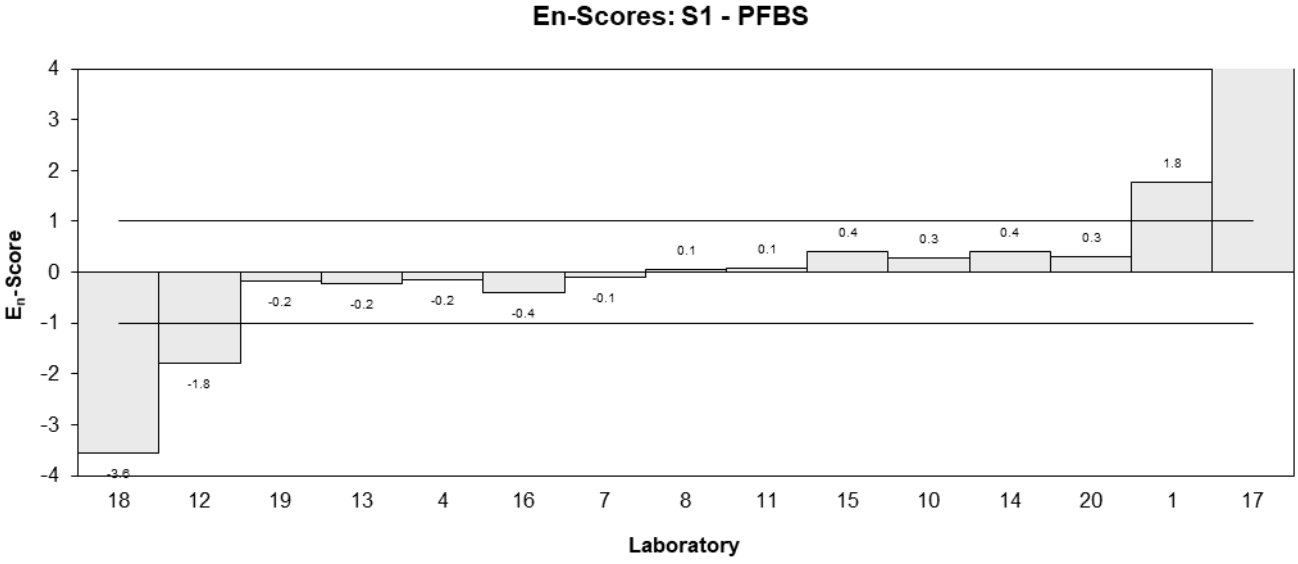

En-Scores: S1 - PFBS
En-Score
Laboratory
18 12 19 13 4 16 7 8 11 15 10 14 20 1 17
-3.6 -1.8 -0.2 -0.2 -0.2 -0.4 -0.1 0.1 0.1 0.4 0.3 0.4 0.3 1.8

Figure 2

Table 7

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	PFHxS
Units	µg/kg

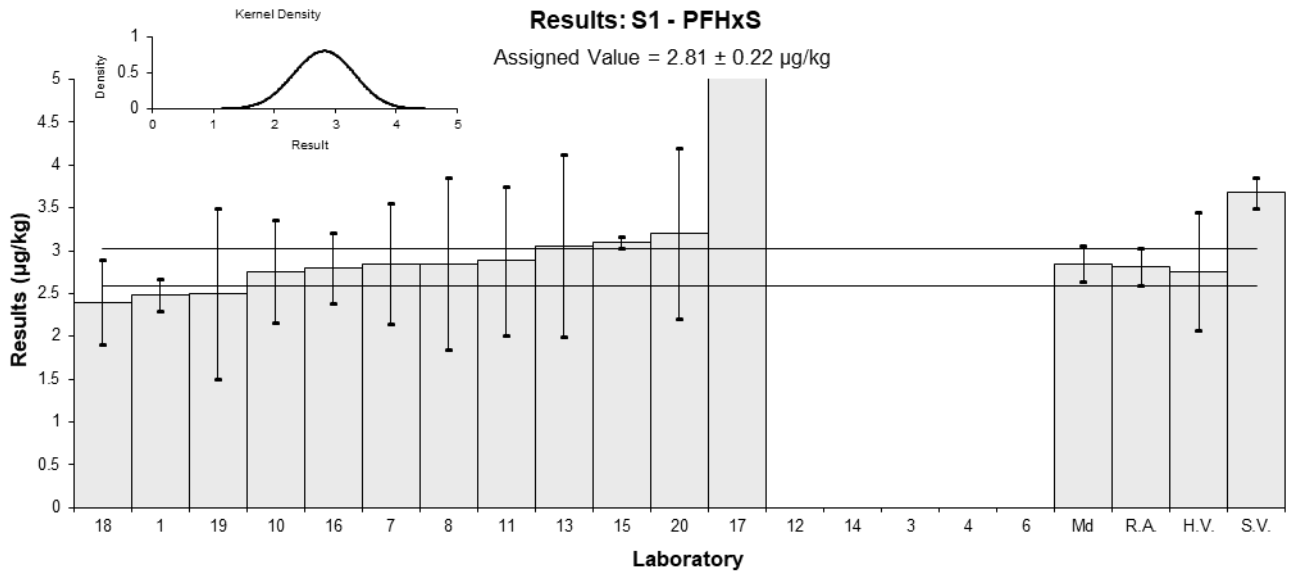
Participant Results

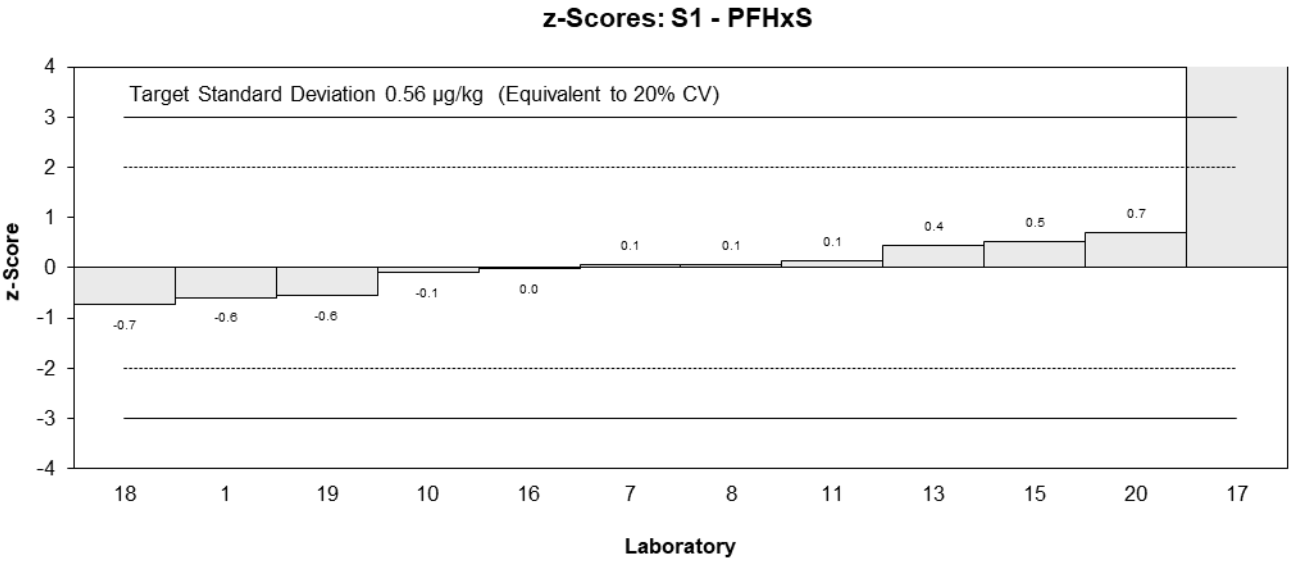
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	2.48	0.19	NR	-0.59	-1.14
3	NT	NT	NT		
4	NT	NT	NT		
6	NT	NT	NT		
7	2.85	0.71	85	0.07	0.05
8	2.85	1.00	150	0.07	0.04
10	2.76	0.6	NR	-0.09	-0.08
11	2.885	0.866	85	0.13	0.08
12	NR	NR	NR		
13	3.06	1.07	135	0.44	0.23
14	NT	NT	NT		
15	3.1	0.064	102	0.52	1.27
16	2.8	0.41	92	-0.02	-0.02
17	12.64	1.07	79	17.49	9.00
18	2.4	0.5	NR	-0.73	-0.75
19	2.5	1	89	-0.55	-0.30
20	3.2	1	98	0.69	0.38

Statistics*

Assigned Value	2.81	0.22
Spike	3.68	0.18
Homogeneity Value	2.76	0.69
Robust Average	2.81	0.22
Median	2.85	0.21
Mean	2.81	
N	11	
Max.	3.2	
Min.	2.4	
Robust SD	0.30	
Robust CV	11%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S1 - PFHxS
Assigned Value = 2.81 ± 0.22 µg/kg
Results (µg/kg)
18 1 19 10 16 7 8 11 13 15 20 17 12 14 3 4 6 Md R.A. H.V. S.V.
Laboratory


z-Scores: S1 - PFHxS
Target Standard Deviation 0.56 µg/kg (Equivalent to 20% CV)
z-Score
-0.7 -0.6 -0.6 -0.1 0.0 0.1 0.1 0.1 0.4 0.5 0.7
18 1 19 10 16 7 8 11 13 15 20 17
Laboratory

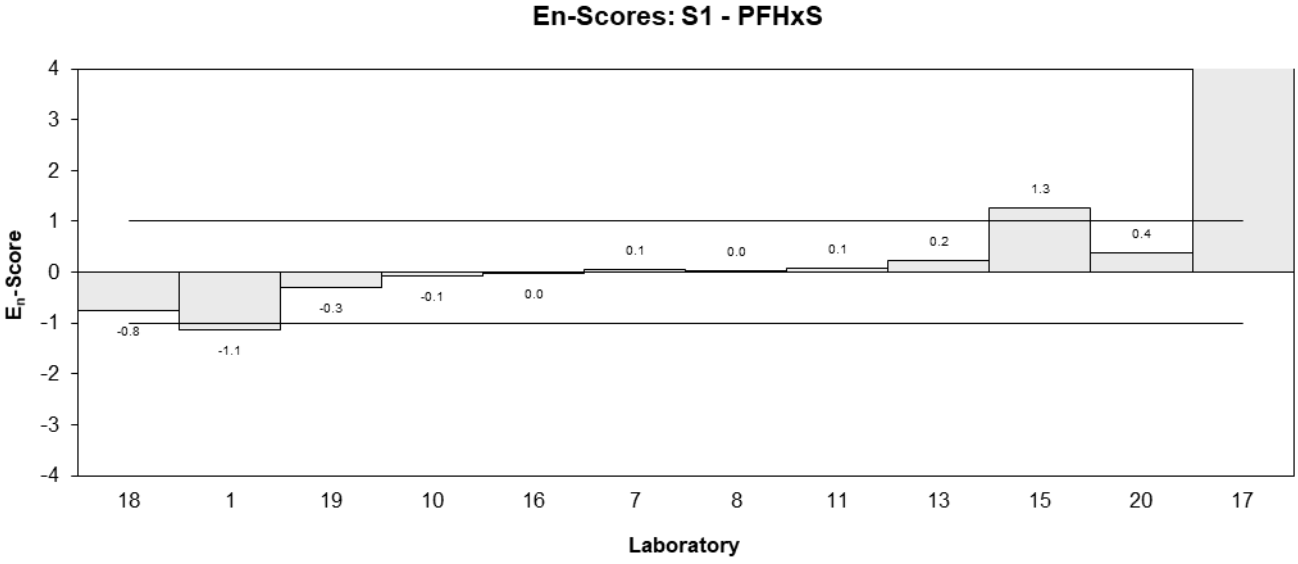

En-Scores: S1 - PFHxS
En-Score
-0.8 -1.1 -0.3 -0.1 0.0 0.1 0.0 0.1 0.2 1.3 0.4
18 1 19 10 16 7 8 11 13 15 20 17
Laboratory

Figure 3

Table 8

Sample Details

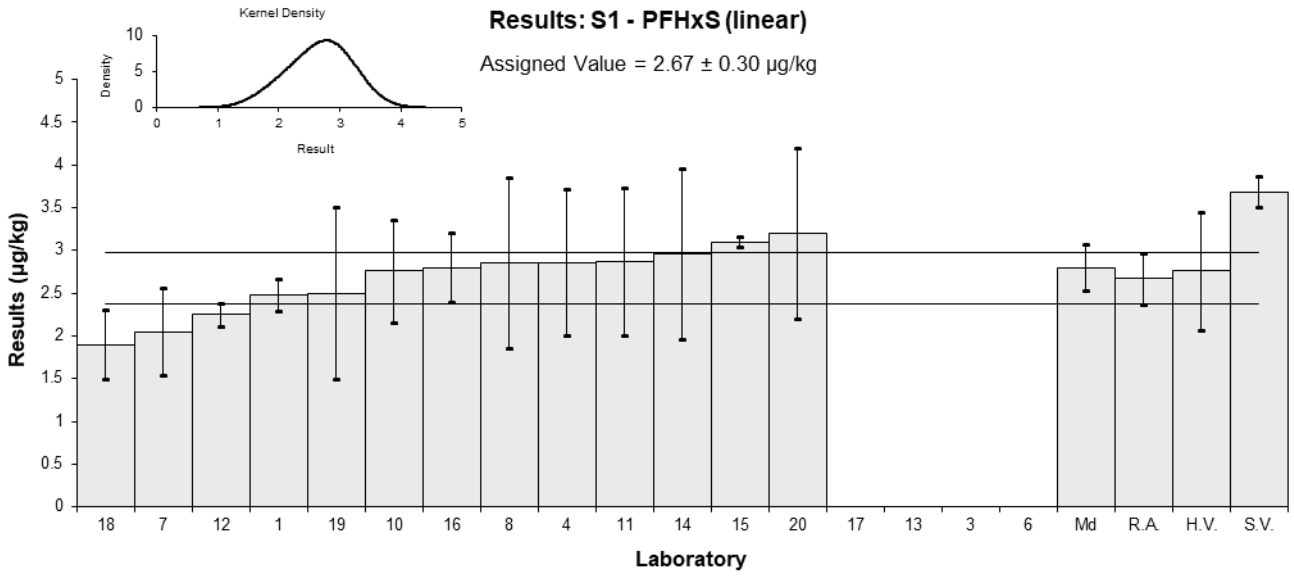
Sample No.	S1
Matrix	Meat
Analyte	PFHxS (linear)
Units	µg/kg

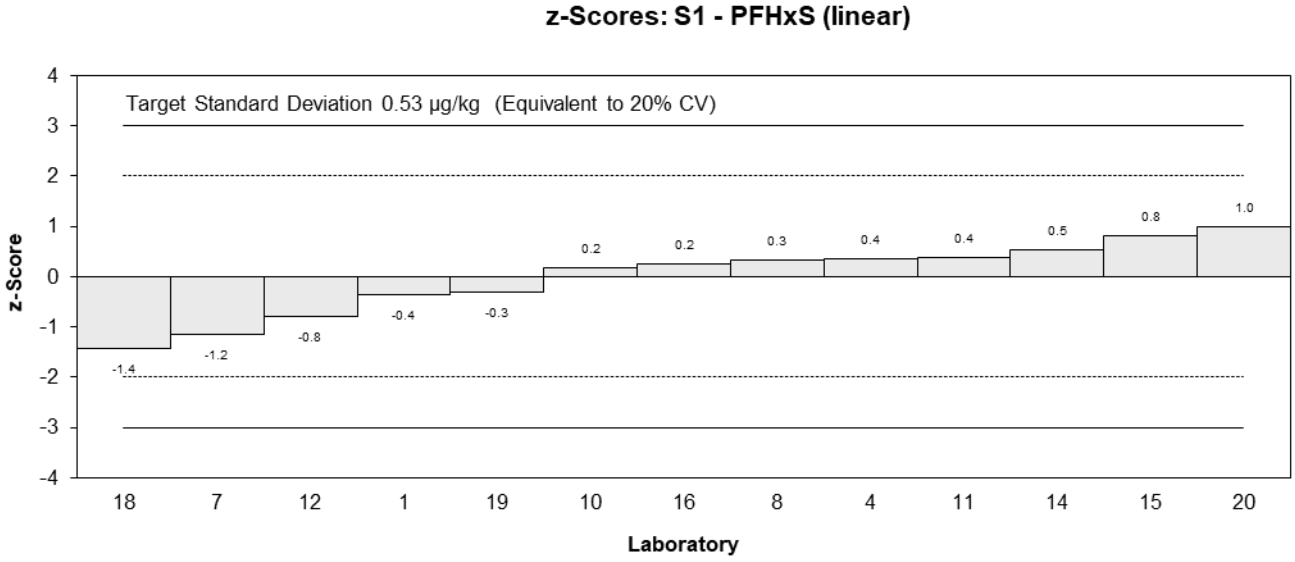
Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	2.48	0.19	NR	-0.36	-0.54
3	NT	NT	NT		
4	2.86	0.86	127	0.36	0.21
6	NT	NT	NT		
7	2.05	0.51	84	-1.16	-1.05
8	2.85	1.00	150	0.34	0.17
10	2.76	0.6	58	0.17	0.13
11	2.874	0.862	85	0.38	0.22
12	2.248	0.138	89	-0.79	-1.28
13	NT	NT	NT		
14	2.96	0.993	95.2	0.54	0.28
15	3.1	0.064	102	0.81	1.40
16	2.8	0.41	92	0.24	0.26
17	NR	NR	NR		
18	1.9	0.4	NR	-1.44	-1.54
19	2.5	1	89	-0.32	-0.16
20	3.2	1	98	0.99	0.51

Statistics

Assigned Value	2.67	0.30
Spike	3.68	0.18
Homogeneity Value	2.76	0.69
Robust Average	2.67	0.30
Median	2.80	0.27
Mean	2.66	
N	13	
Max.	3.2	
Min.	1.9	
Robust SD	0.43	
Robust CV	16%	


Kernel Density
Density
Result
Results: S1 - PFHxS (linear)
Assigned Value = 2.67 ± 0.30 µg/kg
Results (µg/kg)
Laboratory
18 7 12 1 19 10 16 8 4 11 14 15 20 17 13 3 6 Md R.A. H.V. S.V.


z-Scores: S1 - PFHxS (linear)
Target Standard Deviation 0.53 µg/kg (Equivalent to 20% CV)
z-Score
Laboratory
18 7 12 1 19 10 16 8 4 11 14 15 20
-1.4 -1.2 -0.8 -0.4 -0.3 0.2 0.2 0.3 0.4 0.4 0.5 0.8 1.0

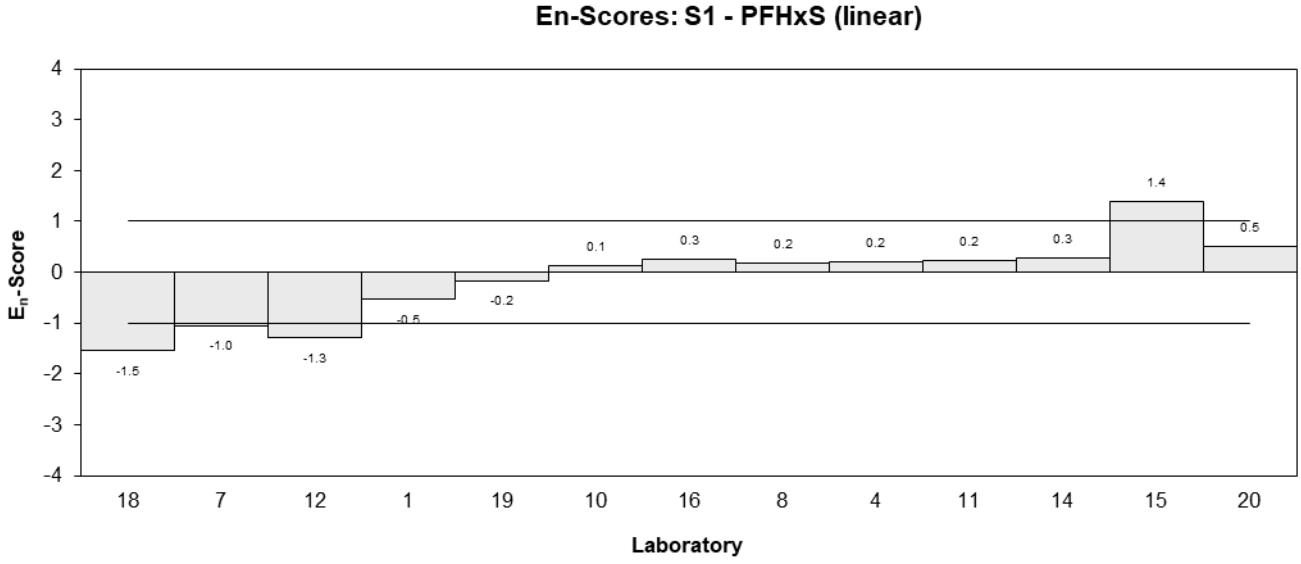

En-Scores: S1 - PFHxS (linear)
En-Score
Laboratory
18 7 12 1 19 10 16 8 4 11 14 15 20
-1.5 -1.0 -1.3 -0.5 -0.2 0.1 0.3 0.2 0.2 0.2 0.3 1.4 0.5

Figure 4

Table 9

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	PFHpS
Units	µg/kg

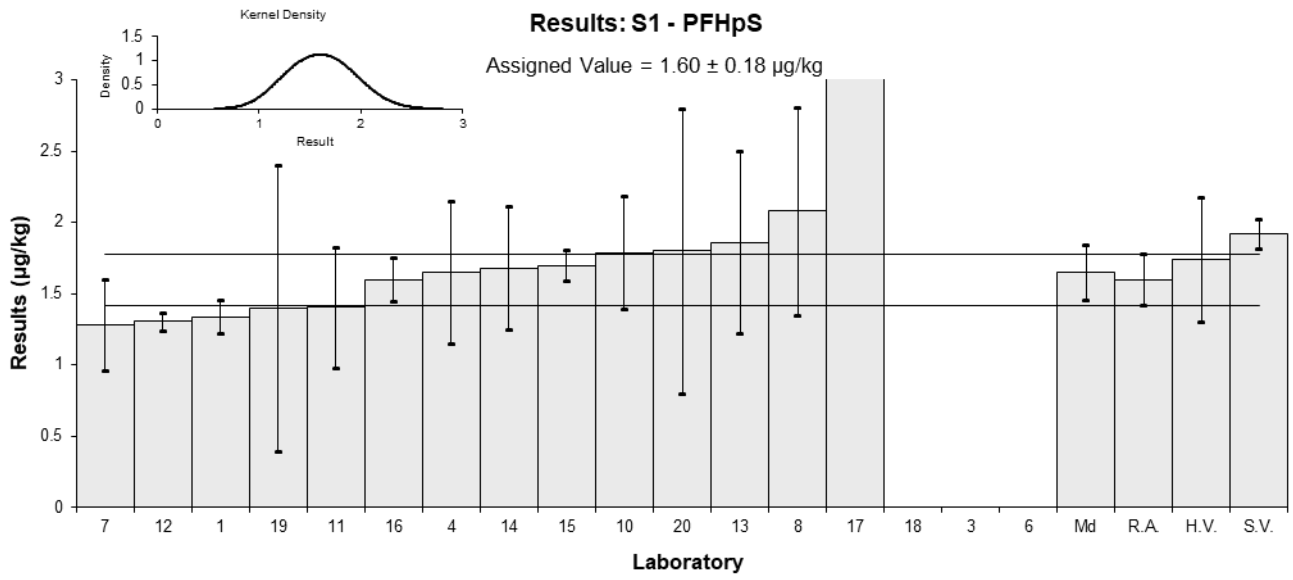
Participant Results

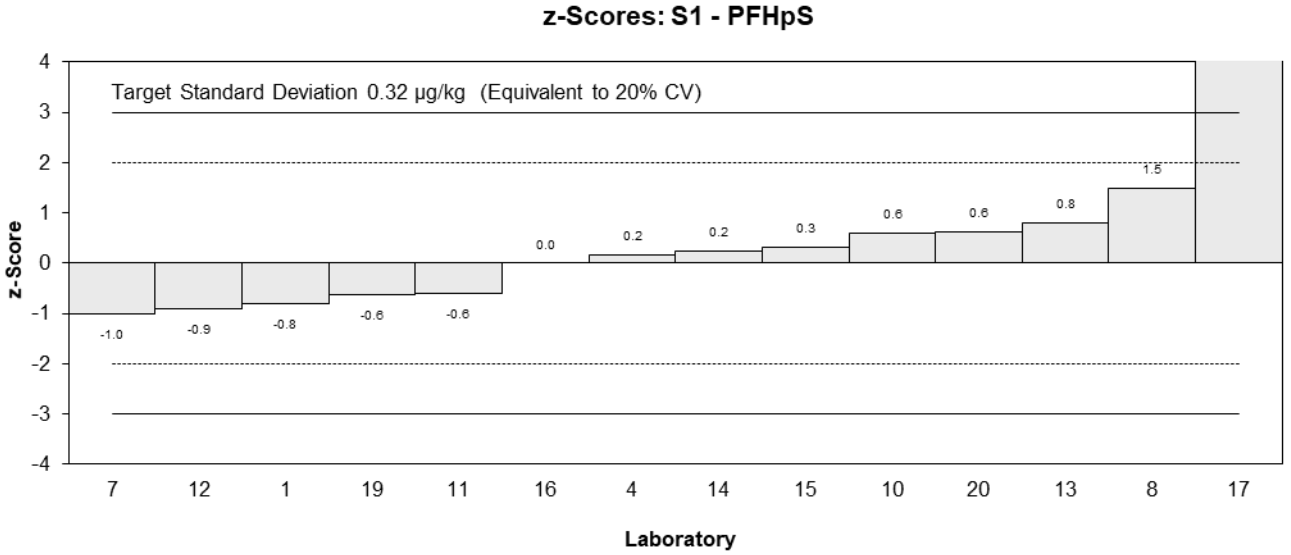
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	1.34	0.12	NR	-0.81	-1.20
3	NT	NT	NT		
4	1.65	0.50	NR	0.16	0.09
6	NT	NT	NT		
7	1.28	0.32	84	-1.00	-0.87
8	2.08	0.73	104	1.50	0.64
10	1.79	0.4	NR	0.59	0.43
11	1.404	0.421	85	-0.61	-0.43
12	1.306	0.064	89	-0.92	-1.54
13	1.86	0.64	151	0.81	0.39
14	1.68	0.431	95.2	0.25	0.17
15	1.7	0.11	102	0.31	0.47
16	1.6	0.15	92	0.00	0.00
17	6.32	0.51	NR	14.75	8.73
18	NT	NT	NT		
19	1.4	1	89	-0.63	-0.20
20	1.8	1	95	0.62	0.20

Statistics*

Assigned Value	1.60	0.18
Spike	1.92	0.10
Homogeneity Value	1.74	0.44
Robust Average	1.60	0.18
Median	1.65	0.19
Mean	1.61	
N	13	
Max.	2.08	
Min.	1.28	
Robust SD	0.27	
Robust CV	17%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S1 - PFHpS
Assigned Value = 1.60 ± 0.18 µg/kg
Results (µg/kg)
Laboratory
7 12 1 19 11 16 4 14 15 10 20 13 8 17 18 3 6 Md R.A. H.V. S.V.


z-Scores: S1 - PFHpS
Target Standard Deviation 0.32 µg/kg (Equivalent to 20% CV)
z-Score
-1.0 -0.9 -0.8 -0.6 -0.6 0.0 0.2 0.2 0.3 0.6 0.6 0.8 1.5
Laboratory
7 12 1 19 11 16 4 14 15 10 20 13 8 17

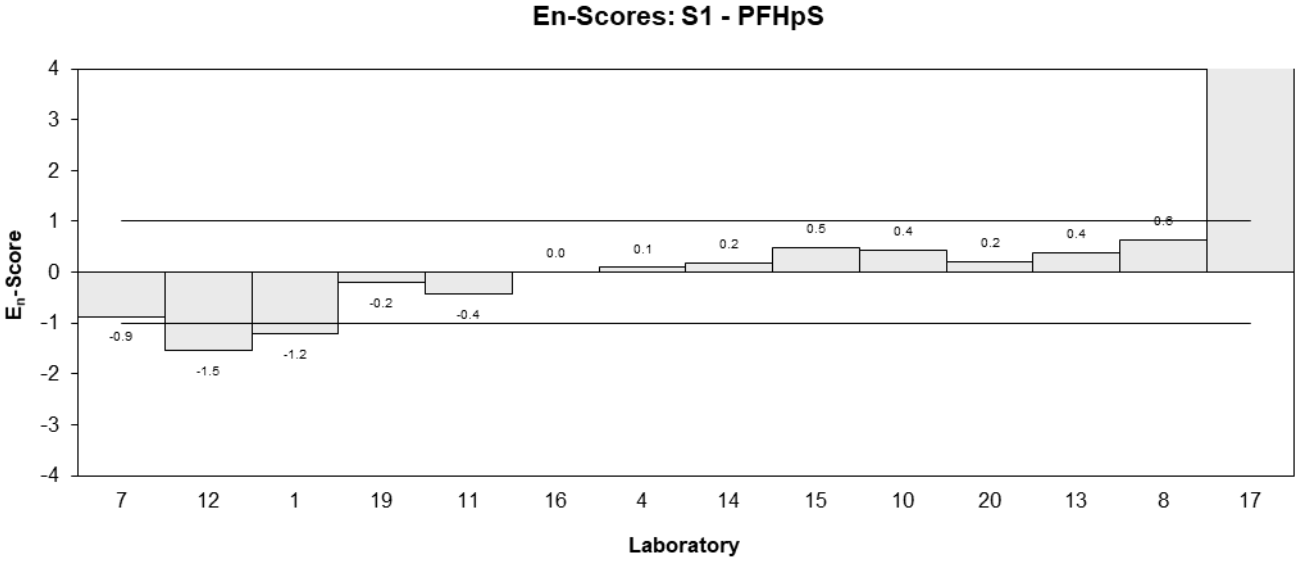

En-Scores: S1 - PFHpS
En-Score
-0.9 -1.5 -1.2 -0.2 -0.4 0.0 0.1 0.2 0.5 0.4 0.2 0.4 0.6
Laboratory
7 12 1 19 11 16 4 14 15 10 20 13 8 17

Figure 5

Table 10

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	PFOS
Units	µg/kg

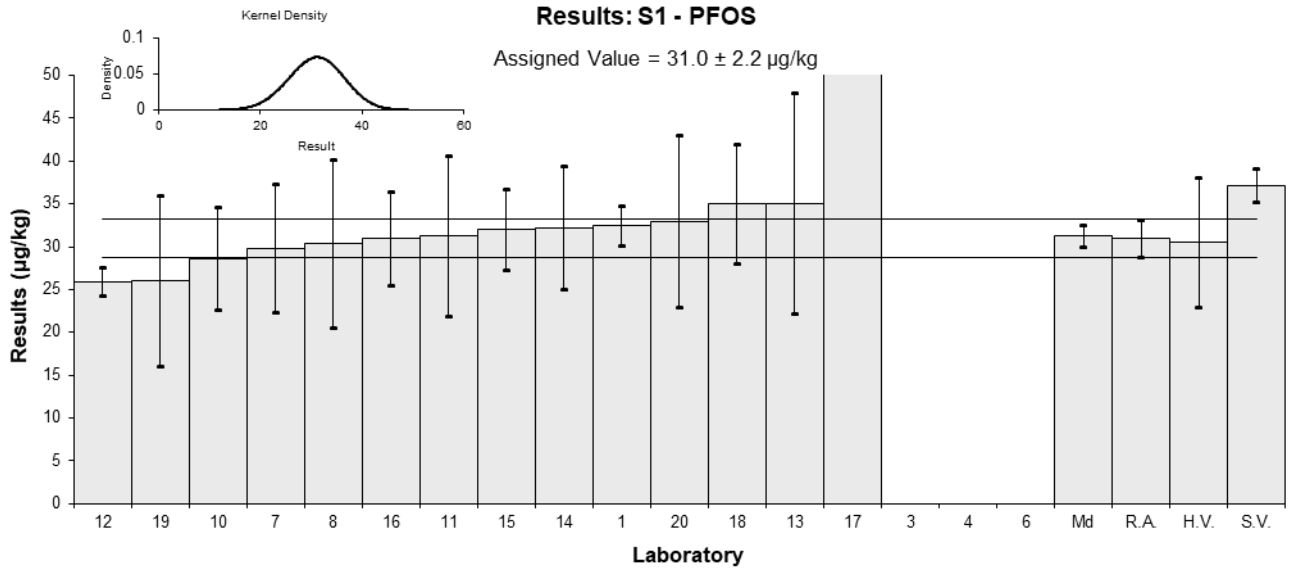
Participant Results

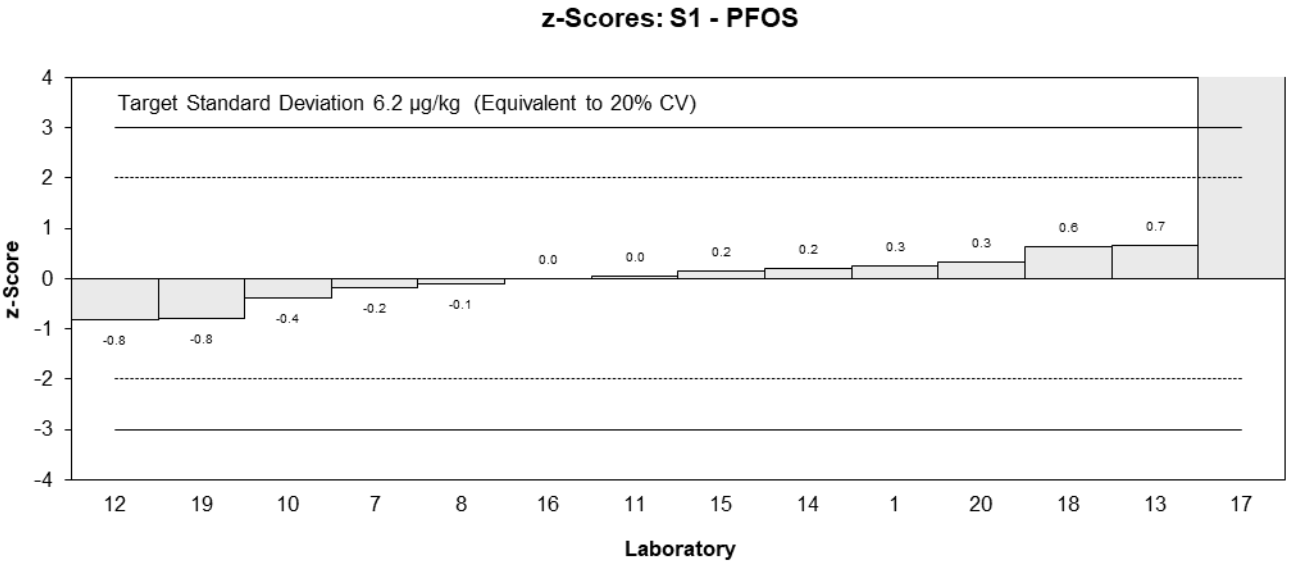
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	32.55	2.32	NR	0.25	0.48
3	NT	NT	NT		
4	NT	NT	NT		
6	NT	NT	NT		
7	29.8	7.5	90	-0.19	-0.15
8	30.33	9.83	104	-0.11	-0.07
10	28.6	6	NR	-0.39	-0.38
11	31.266	9.380	86	0.04	0.03
12	25.952	1.679	88	-0.81	-1.82
13	35.1	12.92	151	0.66	0.31
14	32.2	7.18	89.1	0.19	0.16
15	32	4.7	99	0.16	0.19
16	31	5.49	92	0.00	0.00
17	139.42	14.61	80	17.49	7.34
18	35	7.0	NR	0.65	0.55
19	26	10	89	-0.81	-0.49
20	33	10	95	0.32	0.20

Statistics*

Assigned Value	31.0	2.2
Spike	37.2	1.9
Homogeneity Value	30.5	7.6
Robust Average	31.0	2.2
Median	31.3	1.3
Mean	31.0	
N	13	
Max.	35.1	
Min.	25.952	
Robust SD	3.2	
Robust CV	10%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Results: S1 - PFOS
Kernel Density
Density
Result
Assigned Value = 31.0 ± 2.2 µg/kg
Results (µg/kg)
Laboratory
12 19 10 7 8 16 11 15 14 1 20 18 13 17 3 4 6 Md R.A. H.V. S.V.


z-Scores: S1 - PFOS
Target Standard Deviation 6.2 µg/kg (Equivalent to 20% CV)
z-Score
-0.8 -0.8 -0.4 -0.2 -0.1 0.0 0.0 0.2 0.2 0.3 0.3 0.6 0.7
Laboratory
12 19 10 7 8 16 11 15 14 1 20 18 13 17

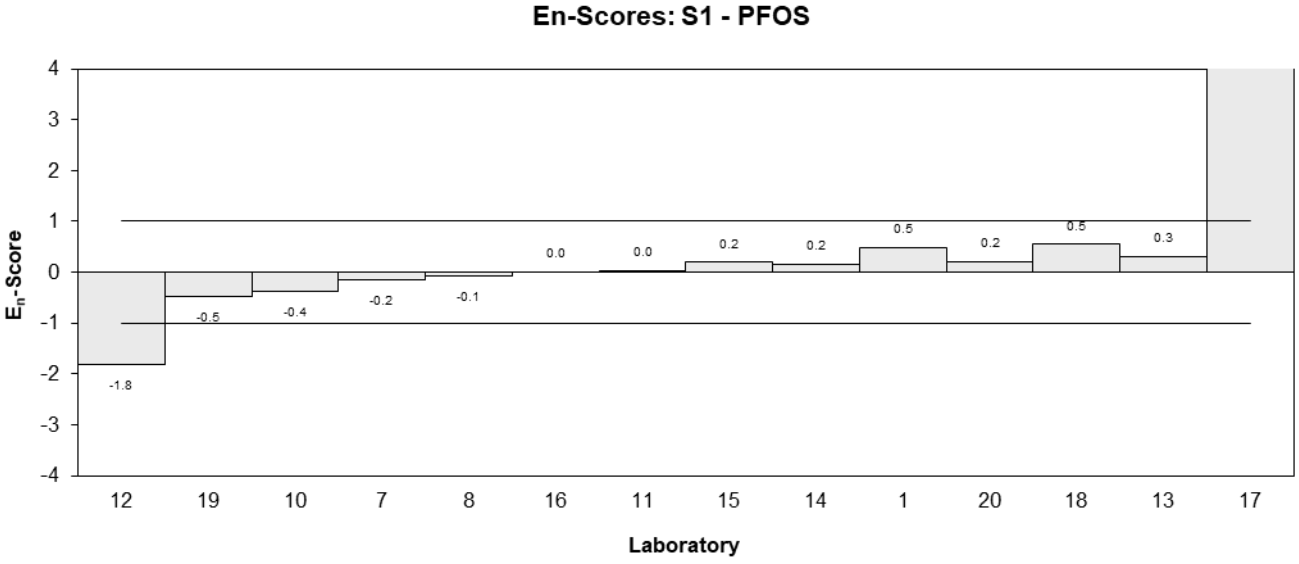

En-Scores: S1 - PFOS
En-Score
-1.8 -0.5 -0.4 -0.2 -0.1 0.0 0.0 0.2 0.2 0.5 0.2 0.5 0.3
Laboratory
12 19 10 7 8 16 11 15 14 1 20 18 13 17

Figure 6

Table 11

Sample Details

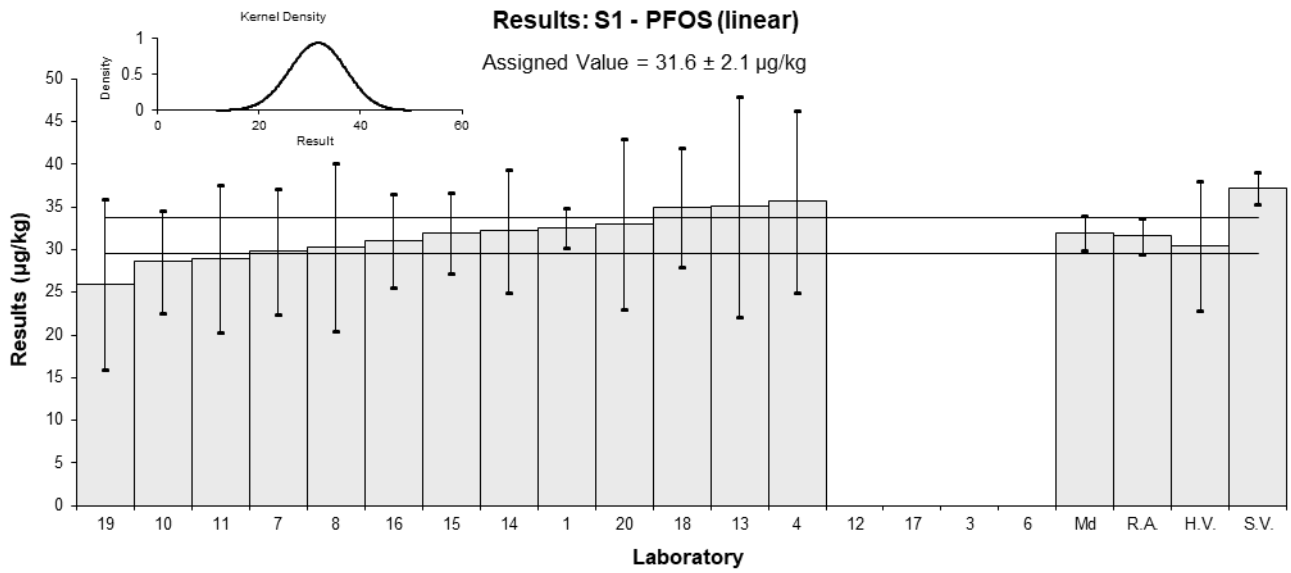
Sample No.	S1
Matrix	Meat
Analyte	PFOS (linear)
Units	µg/kg

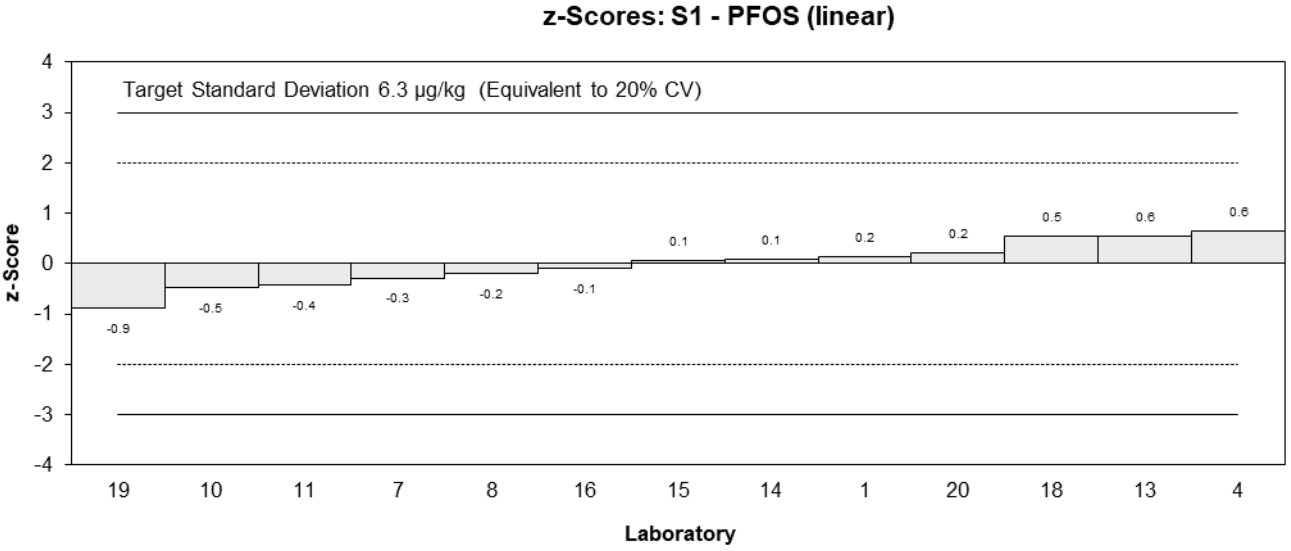
Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	32.55	2.32	NR	0.15	0.30
3	NT	NT	NT		
4	35.68	10.70	92	0.65	0.37
6	NT	NT	NT		
7	29.8	7.4	90	-0.28	-0.23
8	30.33	9.83	104	-0.20	-0.13
10	28.6	6	68	-0.47	-0.47
11	28.93	8.679	86	-0.42	-0.30
12	NR	NR	NR		
13	35.1	12.92	151	0.55	0.27
14	32.2	7.18	89.1	0.09	0.08
15	32	4.7	99	0.06	0.08
16	31	5.49	92	-0.09	-0.10
17	NR	NR	NR		
18	35	7.0	NR	0.54	0.47
19	26	10	89	-0.89	-0.55
20	33	10	95	0.22	0.14

Statistics

Assigned Value	31.6	2.1
Spike	37.2	1.9
Homogeneity Value	30.5	7.6
Robust Average	31.6	2.1
Median	32.0	2.0
Mean	31.6	
N	13	
Max.	35.68	
Min.	26	
Robust SD	3.0	
Robust CV	9.6%	


Results: S1 - PFOS (linear)
Kernel Density
Density
Result
Assigned Value = 31.6 ± 2.1 µg/kg
Results (µg/kg)
19 10 11 7 8 16 15 14 1 20 18 13 4 12 17 3 6 Md R.A. H.V. S.V.
Laboratory


z-Scores: S1 - PFOS (linear)
Target Standard Deviation 6.3 µg/kg (Equivalent to 20% CV)
z-Score
-0.9 -0.5 -0.4 -0.3 -0.2 -0.1 0.1 0.1 0.2 0.2 0.5 0.6 0.6
19 10 11 7 8 16 15 14 1 20 18 13 4
Laboratory

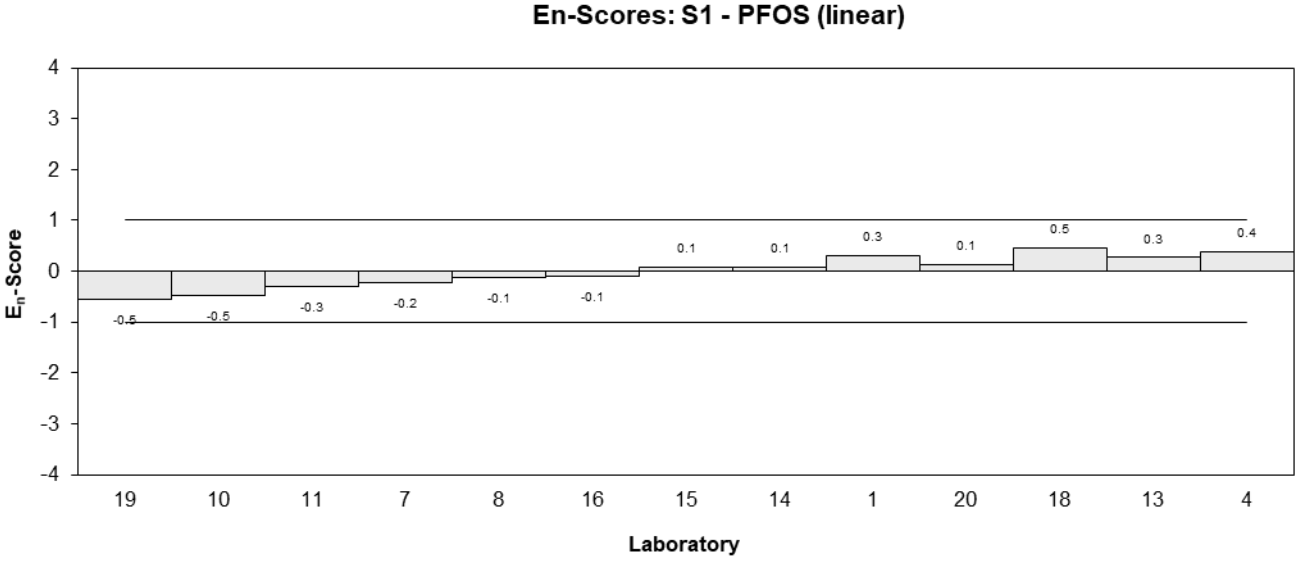

En-Scores: S1 - PFOS (linear)
En-Score
-0.5 -0.5 -0.3 -0.2 -0.1 -0.1 0.1 0.1 0.3 0.1 0.5 0.3 0.4
19 10 11 7 8 16 15 14 1 20 18 13 4
Laboratory

Figure 7

Table 12

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	PFDS
Units	µg/kg

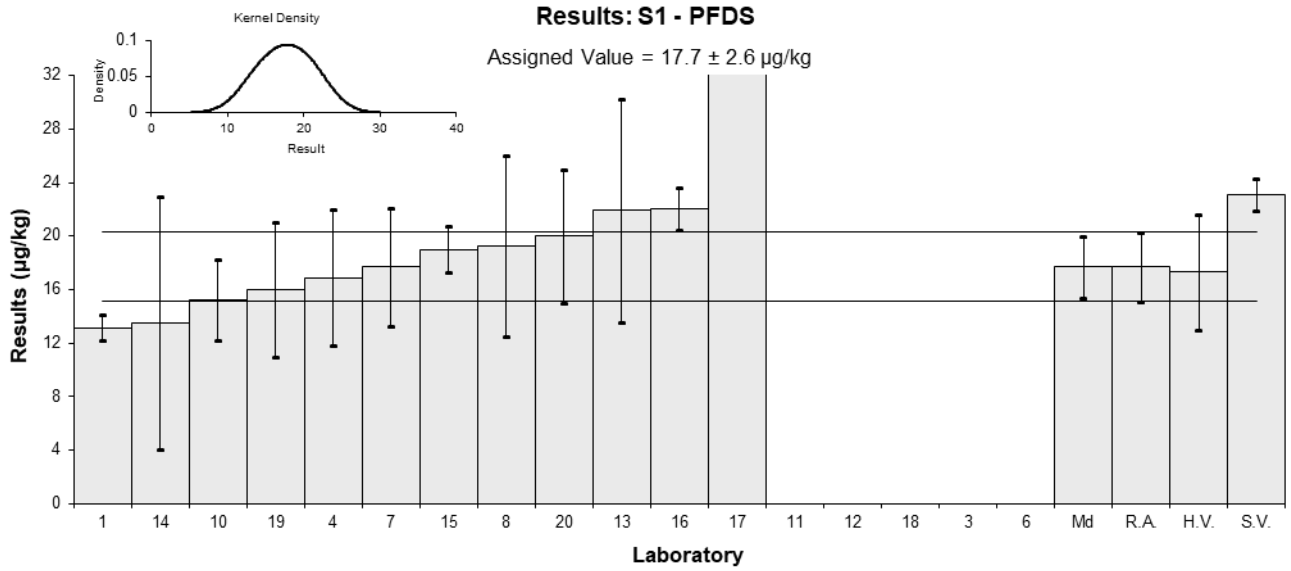
Participant Results

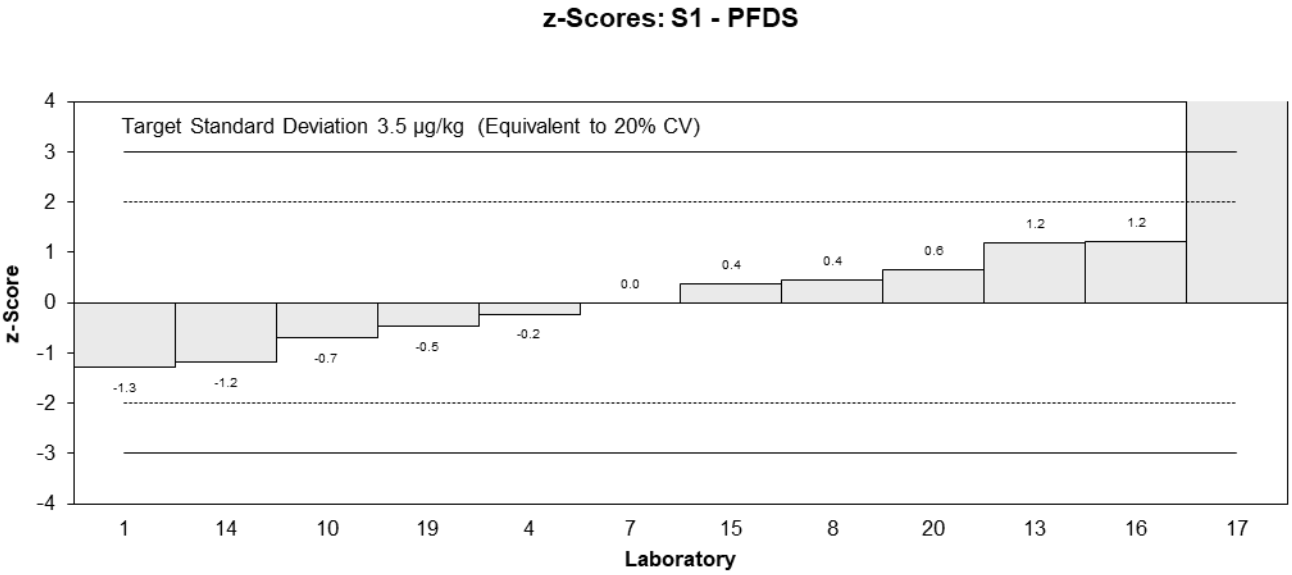
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	13.16	0.94	NR	-1.28	-1.64
3	NT	NT	NT		
4	16.89	5.07	NR	-0.23	-0.14
6	NT	NT	NT		
7	17.7	4.4	73	0.00	0.00
8	19.27	6.7451895	104	0.44	0.22
10	15.2	3	NR	-0.71	-0.63
11	<0.5	NR	NR		
12	NT	NT	NT		
13	21.9	8.37	151	1.19	0.48
14	13.5	9.43	89.1	-1.19	-0.43
15	19	1.7	99	0.37	0.42
16	22	1.58	92	1.21	1.41
17	55.25	3.1	NR	10.61	9.28
18	NT	NT	NT		
19	16	5	91	-0.48	-0.30
20	20	5	95	0.65	0.41

Statistics*

Assigned Value	17.7	2.6
Spike	23.1	1.2
Homogeneity Value	17.3	4.3
Robust Average	17.7	2.6
Median	17.7	2.3
Mean	17.7	
N	11	
Max.	22	
Min.	13.16	
Robust SD	3.5	
Robust CV	20%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Results: S1 - PFDS
Kernel Density
Density
Result
Assigned Value = 17.7 ± 2.6 µg/kg
Results (µg/kg)
Laboratory
1 14 10 19 4 7 15 8 20 13 16 17 11 12 18 3 6 Md R.A. H.V. S.V.


z-Scores: S1 - PFDS
Target Standard Deviation 3.5 µg/kg (Equivalent to 20% CV)
z-Score
Laboratory
1 14 10 19 4 7 15 8 20 13 16 17
-1.3 -1.2 -0.7 -0.5 -0.2 0.0 0.4 0.4 0.6 1.2 1.2

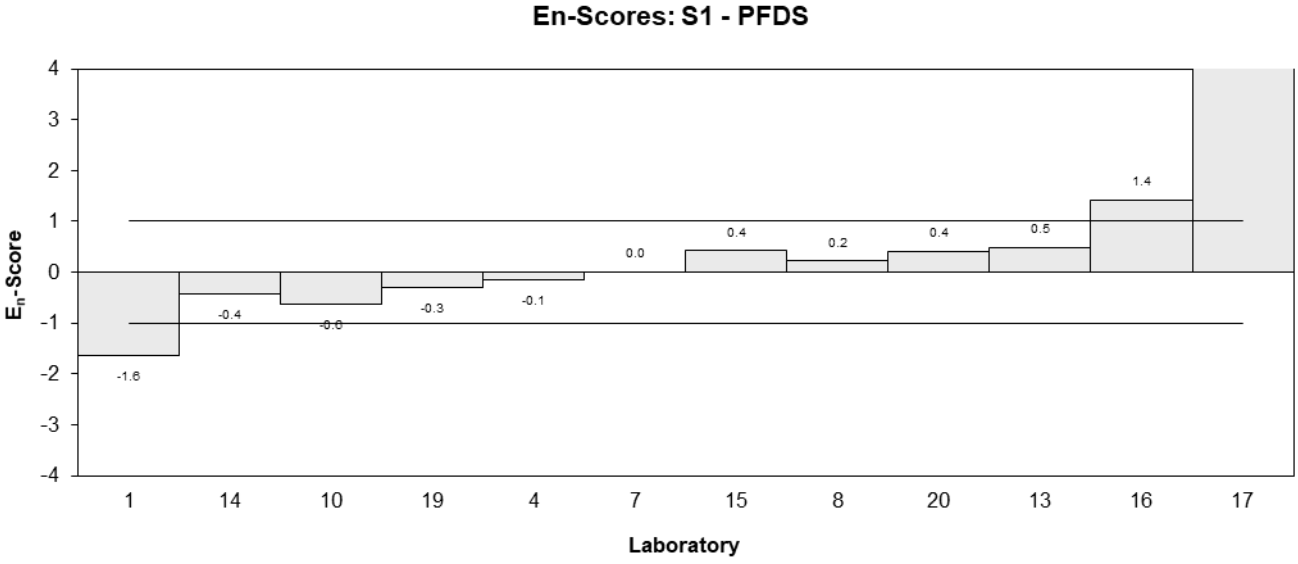

En-Scores: S1 - PFDS
En-Score
Laboratory
1 14 10 19 4 7 15 8 20 13 16 17
-1.6 -0.4 -0.6 -0.3 -0.1 0.0 0.4 0.2 0.4 0.5 1.4

Figure 8

Table 13

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	PFBA
Units	µg/kg

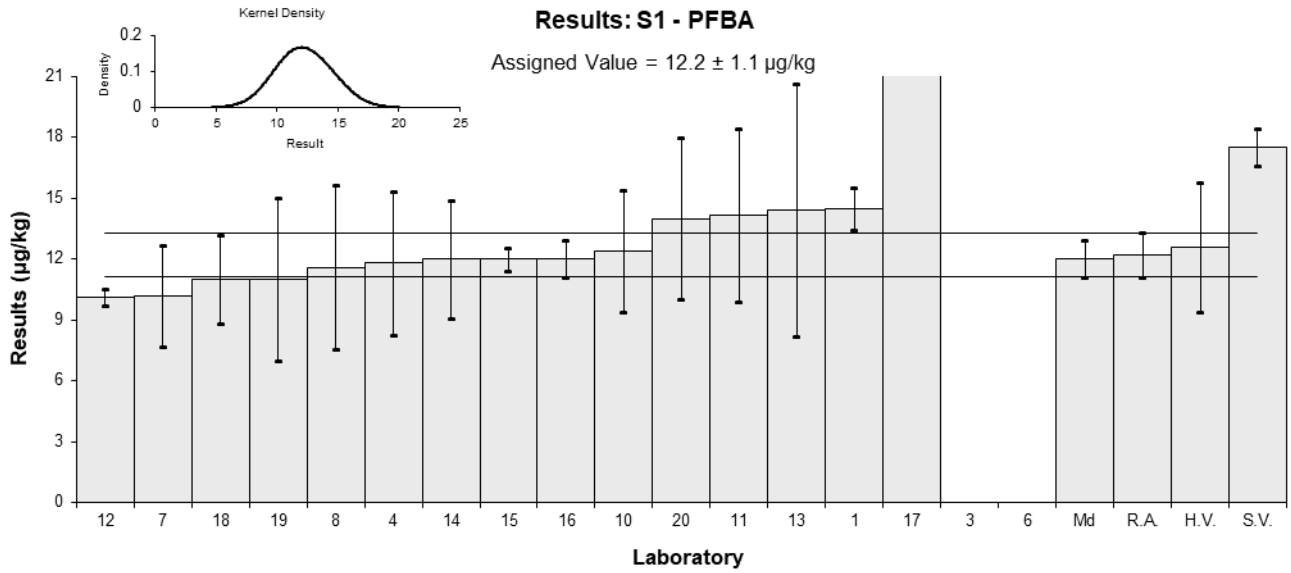
Participant Results

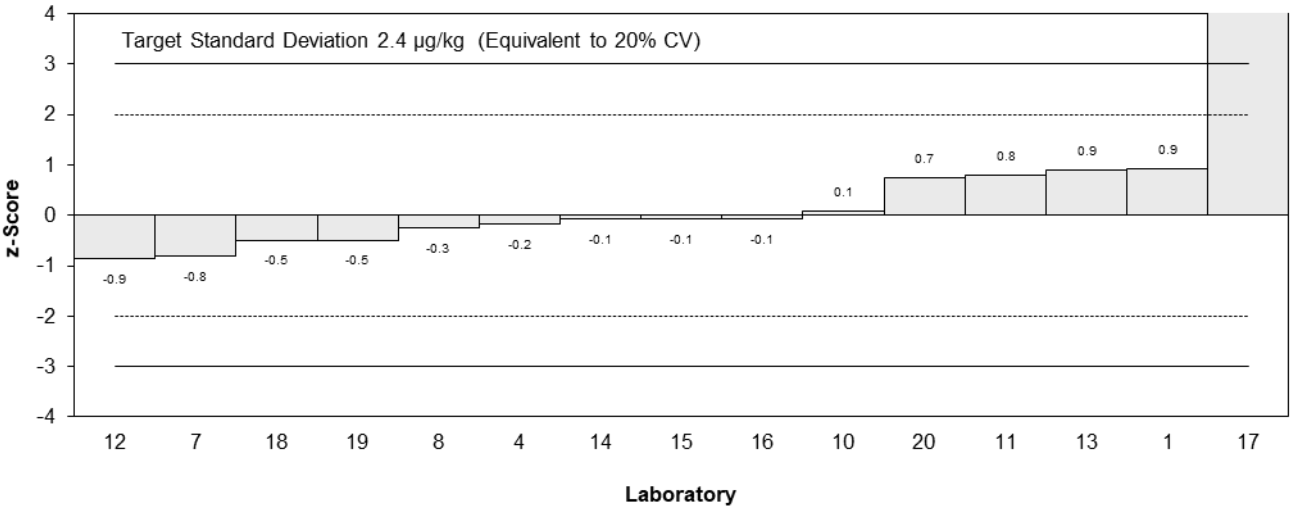
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	14.47	1.03	NR	0.93	1.51
3	NT	NT	NT		
4	11.80	3.54	92	-0.16	-0.11
6	NT	NT	NT		
7	10.2	2.5	77	-0.82	-0.73
8	11.58	4.05	88	-0.25	-0.15
10	12.4	3	55	0.08	0.06
11	14.166	4.250	83	0.81	0.45
12	10.125	0.393	65	-0.85	-1.78
13	14.4	6.22	128	0.90	0.35
14	12.0	2.91	89.4	-0.08	-0.06
15	12	0.57	127	-0.08	-0.16
16	12	0.92	87	-0.08	-0.14
17	50.96	4.13	77	15.89	9.07
18	11	2.2	NR	-0.49	-0.49
19	11	4	101	-0.49	-0.29
20	14	4	91	0.74	0.43

Statistics*

Assigned Value	12.2	1.1
Spike	17.5	0.9
Homogeneity Value	12.6	3.2
Robust Average	12.2	1.1
Median	12.0	0.9
Mean	12.2	
N	14	
Max.	14.47	
Min.	10.125	
Robust SD	1.7	
Robust CV	14%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S1 - PFBA
Assigned Value = 12.2 ± 1.1 µg/kg
Results (µg/kg)
12 7 18 19 8 4 14 15 16 10 20 11 13 1 17 3 6 Md R.A. H.V. S.V.
Laboratory


z-Scores: S1 - PFBA
Target Standard Deviation 2.4 µg/kg (Equivalent to 20% CV)
z-Score
-0.9 -0.8 -0.5 -0.5 -0.3 -0.2 -0.1 -0.1 -0.1 0.1 0.7 0.8 0.9 0.9
12 7 18 19 8 4 14 15 16 10 20 11 13 1 17
Laboratory

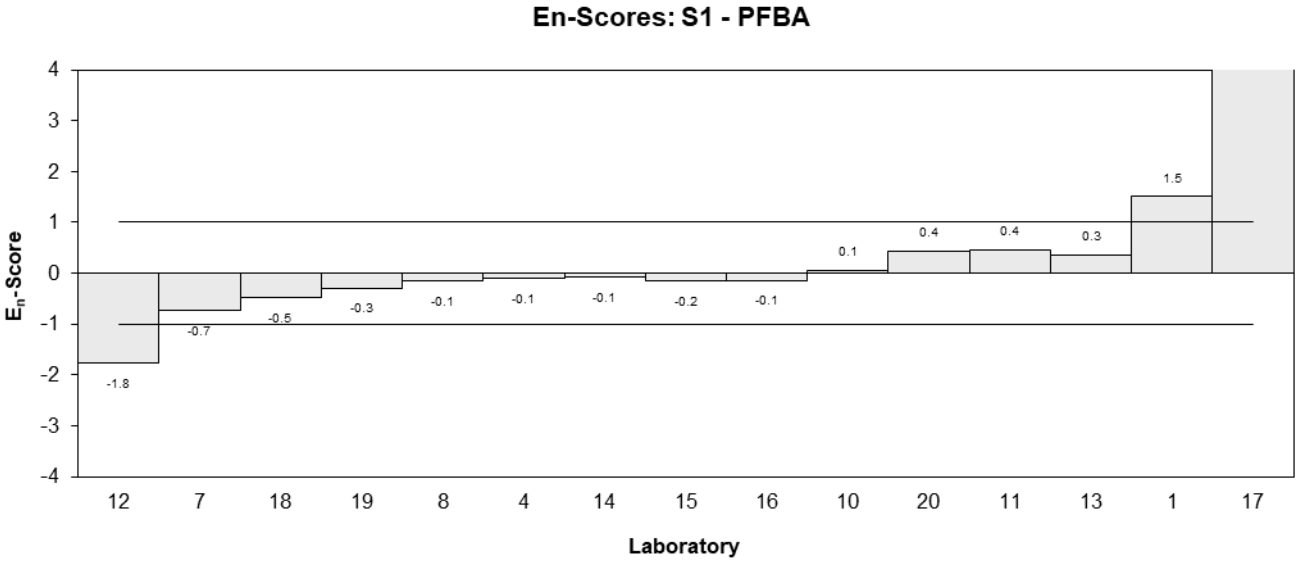

En-Scores: S1 - PFBA
En-Score
-1.8 -0.7 -0.5 -0.3 -0.1 -0.1 -0.1 -0.2 -0.1 0.1 0.4 0.4 0.3 1.5
12 7 18 19 8 4 14 15 16 10 20 11 13 1 17
Laboratory

Figure 9

Table 14

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	PFPeA
Units	µg/kg

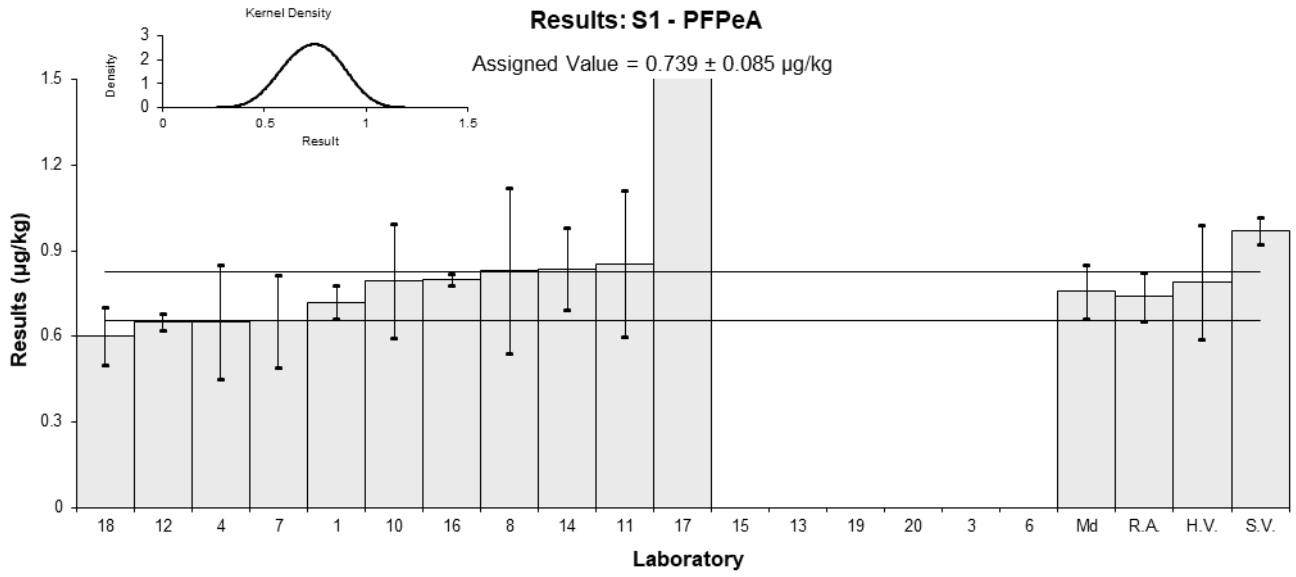
Participant Results

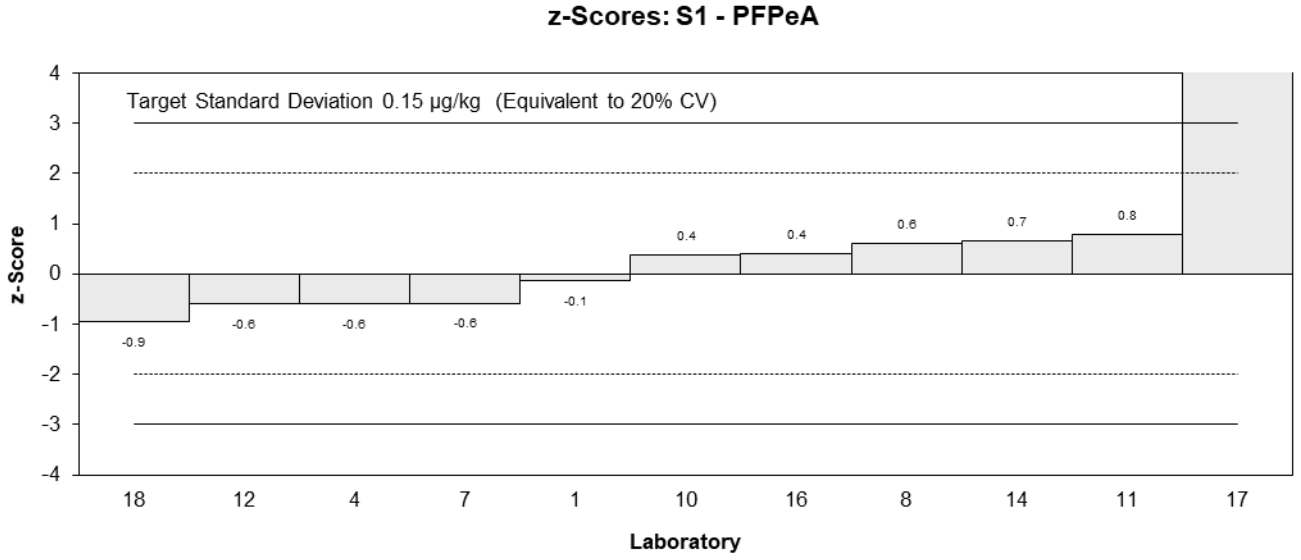
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.72	0.06	NR	-0.13	-0.18
3	NT	NT	NT		
4	0.65	0.20	115	-0.60	-0.41
6	NT	NT	NT		
7	0.653	0.16	76	-0.58	-0.47
8	0.83	0.29	77	0.62	0.30
10	0.794	0.2	58	0.37	0.25
11	0.855	0.257	92	0.78	0.43
12	0.65	0.028	70	-0.60	-0.99
13	<2	NR	138		
14	0.837	0.143	77.6	0.66	0.59
15	< 1.0	NR	125		
16	0.8	0.02	88	0.41	0.70
17	4.02	0.25	93	22.20	12.43
18	0.6	0.1	NR	-0.94	-1.06
19	<2	NR	98		
20	<2	NR	92		

Statistics*

Assigned Value	0.739	0.085
Spike	0.970	0.048
Homogeneity Value	0.79	0.20
Robust Average	0.739	0.085
Median	0.757	0.094
Mean	0.739	
N	10	
Max.	0.855	
Min.	0.6	
Robust SD	0.11	
Robust CV	15%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S1 - PFPeA
Assigned Value = 0.739 ± 0.085 µg/kg
Results (µg/kg)
Laboratory
18 12 4 7 1 10 16 8 14 11 17 15 13 19 20 3 6 Md R.A. H.V. S.V.


z-Scores: S1 - PFPeA
Target Standard Deviation 0.15 µg/kg (Equivalent to 20% CV)
z-Score
-0.9 -0.6 -0.6 -0.6 -0.1 0.4 0.4 0.6 0.7 0.8
Laboratory
18 12 4 7 1 10 16 8 14 11 17

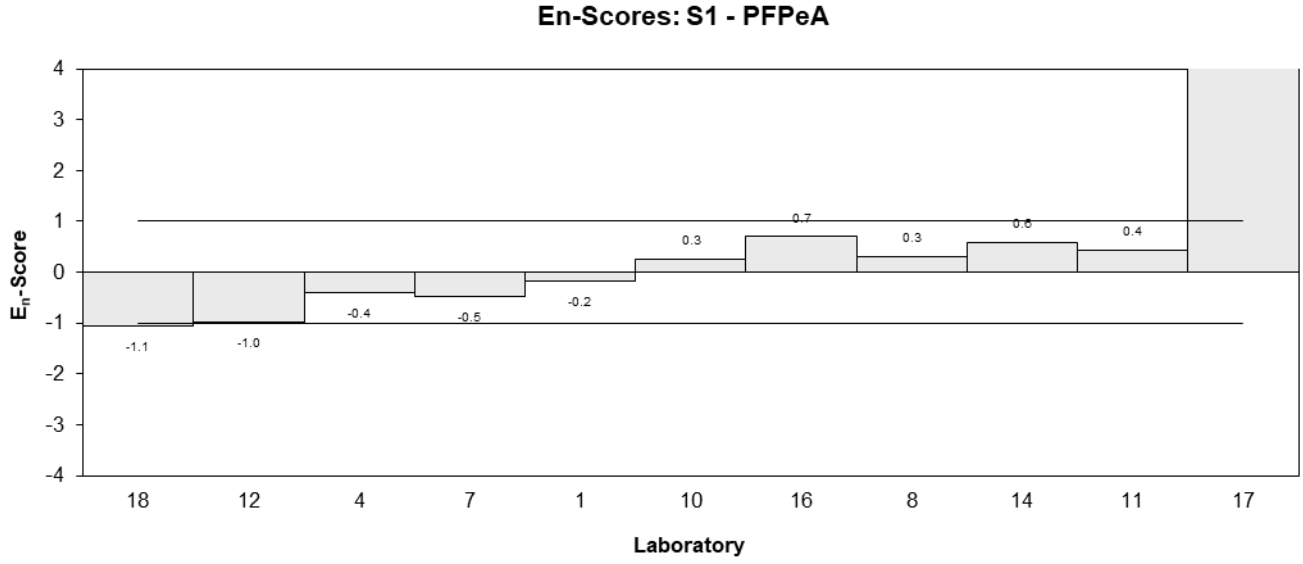

En-Scores: S1 - PFPeA
En-Score
-1.1 -1.0 -0.4 -0.5 -0.2 0.3 0.7 0.3 0.6 0.4
Laboratory
18 12 4 7 1 10 16 8 14 11 17

Figure 10

Table 15

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	PFHxA
Units	µg/kg

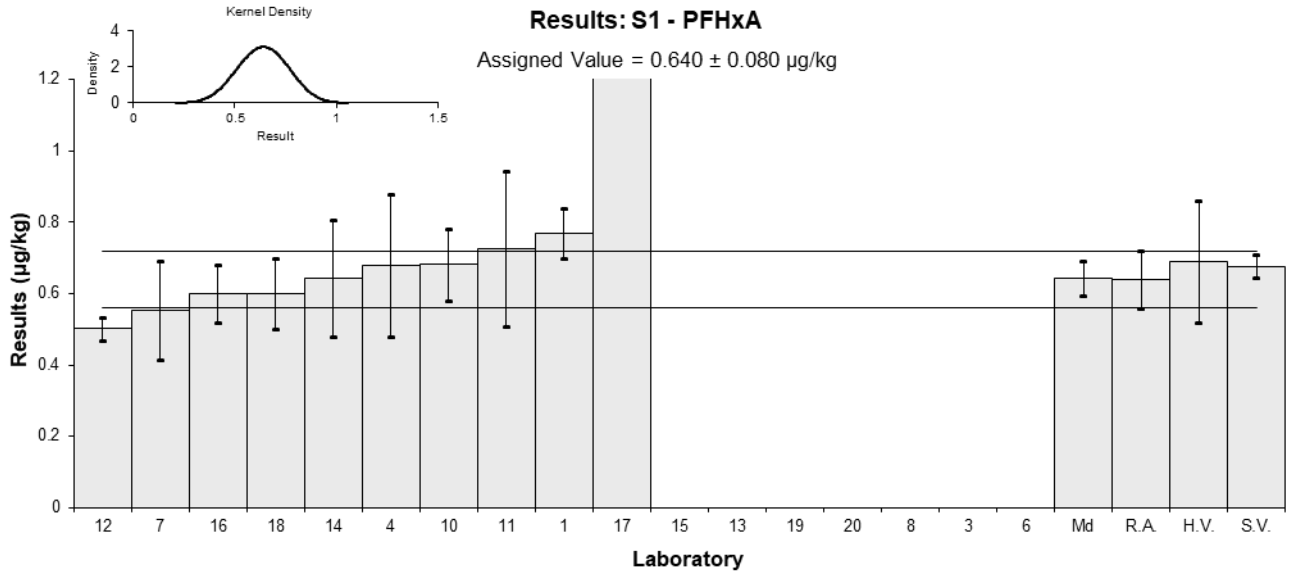
Participant Results

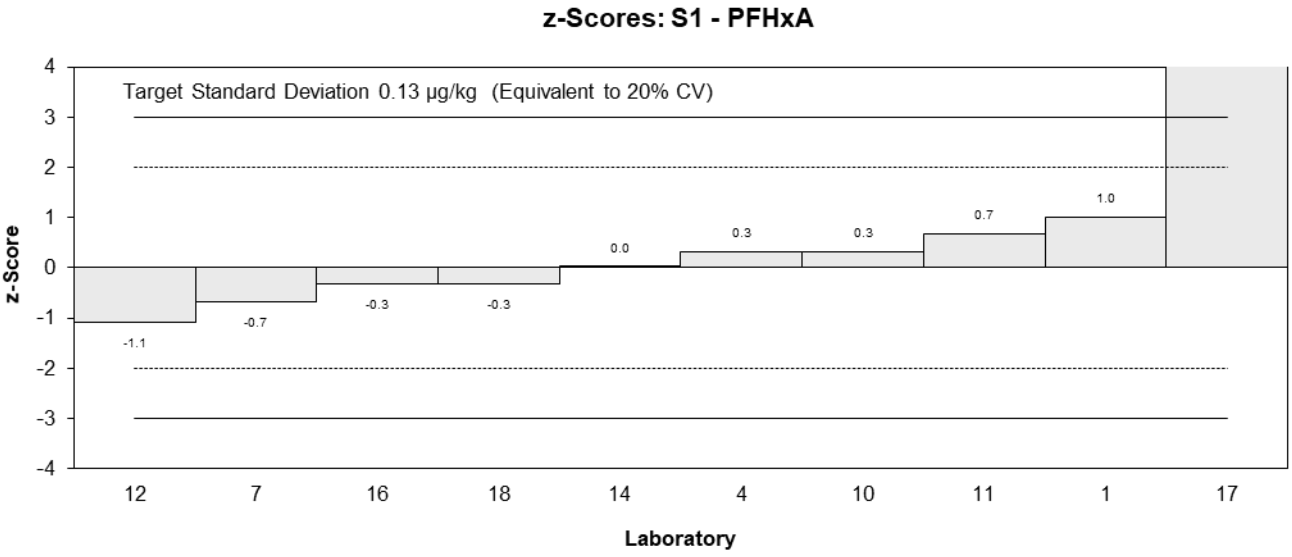
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.77	0.07	NR	1.02	1.22
3	NT	NT	NT		
4	0.68	0.20	123	0.31	0.19
6	NT	NT	NT		
7	0.553	0.14	80	-0.68	-0.54
8	NR	NR	62		
10	0.681	0.1	61	0.32	0.32
11	0.727	0.218	89	0.68	0.37
12	0.502	0.032	74	-1.08	-1.60
13	<1	NR	124		
14	0.643	0.163	88.6	0.02	0.02
15	< 1.0	NR	121		
16	0.6	0.08	88	-0.31	-0.35
17	2.54	0.18	77	14.84	9.65
18	0.6	0.1	NR	-0.31	-0.31
19	<1	NR	95		
20	<1	NR	99		

Statistics*

Assigned Value	0.640	0.080
Spike	0.677	0.034
Homogeneity Value	0.69	0.17
Robust Average	0.640	0.080
Median	0.643	0.049
Mean	0.640	
N	9	
Max.	0.77	
Min.	0.502	
Robust SD	0.096	
Robust CV	15%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S1 - PFHxA
Assigned Value = 0.640 ± 0.080 µg/kg
Results (µg/kg)
Laboratory
12 7 16 18 14 4 10 11 1 17 15 13 19 20 8 3 6 Md R.A. H.V. S.V.


z-Scores: S1 - PFHxA
Target Standard Deviation 0.13 µg/kg (Equivalent to 20% CV)
z-Score
-1.1 -0.7 -0.3 -0.3 0.0 0.3 0.3 0.7 1.0
Laboratory
12 7 16 18 14 4 10 11 1 17

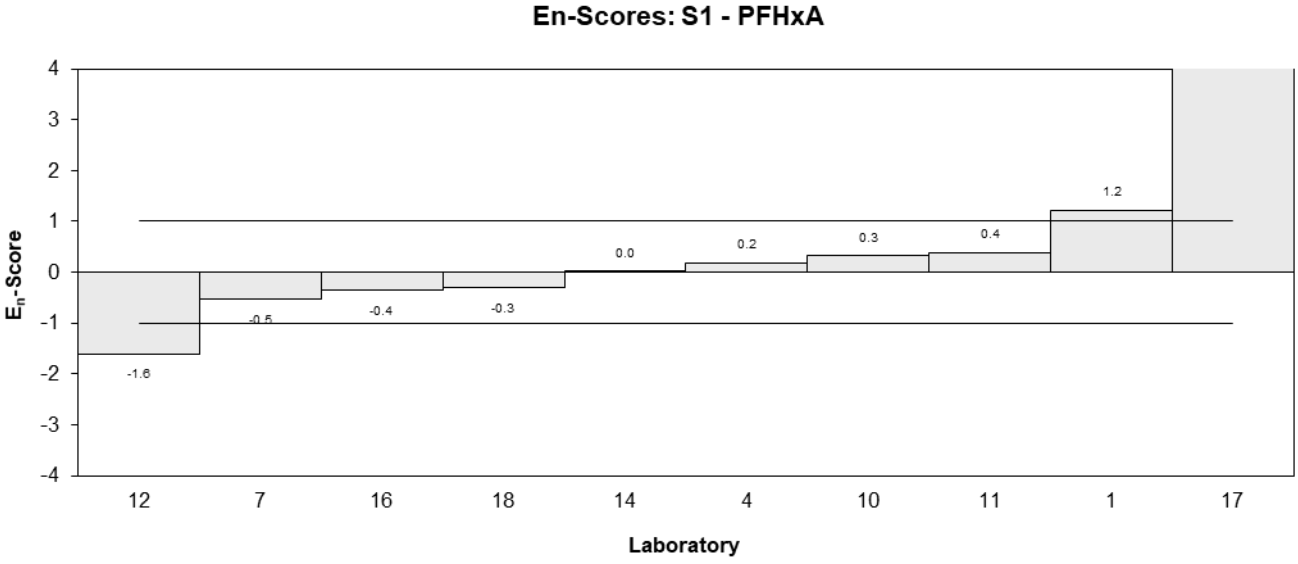

En-Scores: S1 - PFHxA
En-Score
-1.6 -0.5 -0.4 -0.3 0.0 0.2 0.3 0.4 1.2
Laboratory
12 7 16 18 14 4 10 11 1 17

Figure 11

Table 16

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	PFHpA
Units	µg/kg

Participant Results

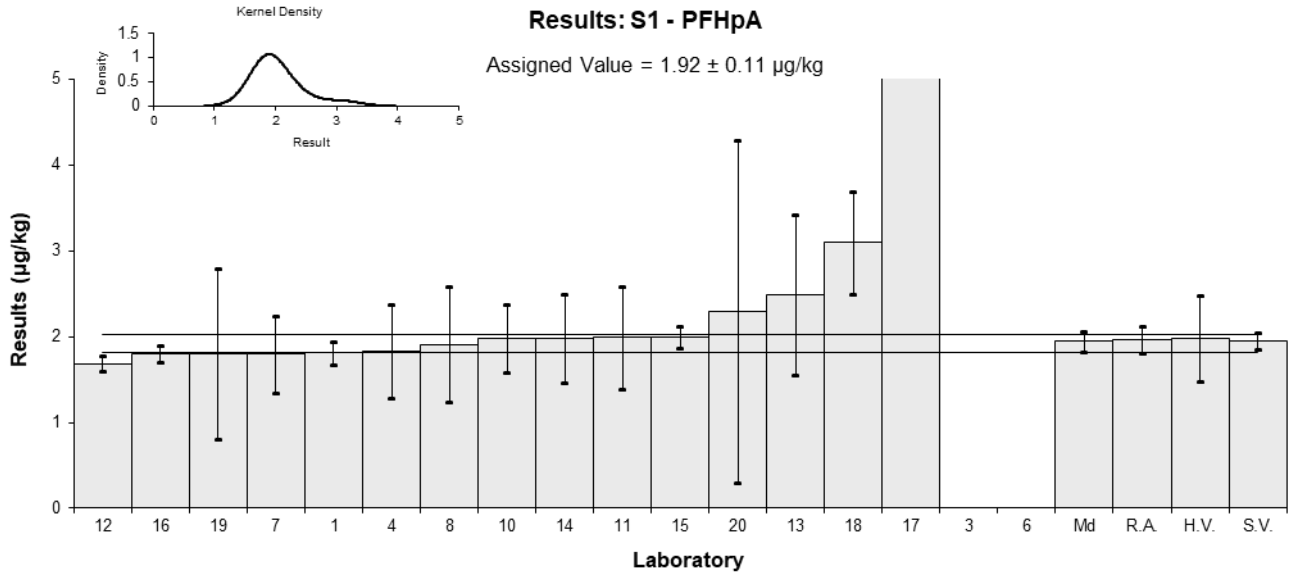
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	1.81	0.13	NR	-0.29	-0.65
3	NT	NT	NT		
4	1.83	0.55	127	-0.23	-0.16
6	NT	NT	NT		
7	1.8	0.45	88	-0.31	-0.26
8	1.91	0.67	74	-0.03	-0.01
10	1.98	0.4	62	0.16	0.14
11	1.994	0.598	111	0.19	0.12
12	1.689	0.086	74	-0.60	-1.65
13	2.49	0.93	122	1.48	0.61
14	1.98	0.521	87.5	0.16	0.11
15	2.0	0.13	119	0.21	0.47
16	1.8	0.10	91	-0.31	-0.81
17	7.71	0.33	80	15.08	16.65
18	3.1	0.6	NR	3.07	1.93
19	1.8	1	87	-0.31	-0.12
20	2.3	2	93	0.99	0.19

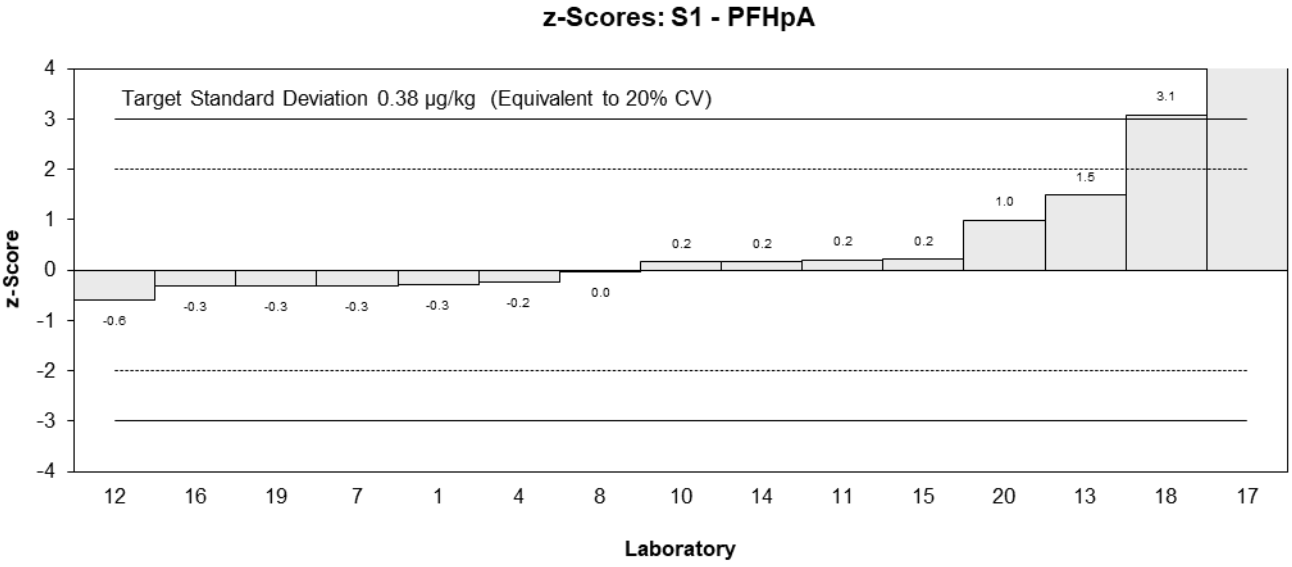
Statistics*

Assigned Value**	1.92	0.11
Spike	1.95	0.10
Homogeneity Value	1.98	0.50
Robust Average	1.97	0.16
Median	1.95	0.12
Mean	2.03	
N	14	
Max.	3.1	
Min.	1.689	
Robust SD	0.24	
Robust CV	12%	

* Laboratory 17 was omitted from all statistical calculations (gross error).

** Robust average excluding Laboratory 18.


Kernel Density
Density
Result
Results: S1 - PFHpA
Assigned Value = 1.92 ± 0.11 µg/kg
Results (µg/kg)
12 16 19 7 1 4 8 10 14 11 15 20 13 18 17 3 6 Md R.A. H.V. S.V.
Laboratory


z-Scores: S1 - PFHpA
Target Standard Deviation 0.38 µg/kg (Equivalent to 20% CV)
z-Score
-0.6 -0.3 -0.3 -0.3 -0.3 -0.2 0.0 0.2 0.2 0.2 0.2 1.0 1.5 3.1
12 16 19 7 1 4 8 10 14 11 15 20 13 18 17
Laboratory

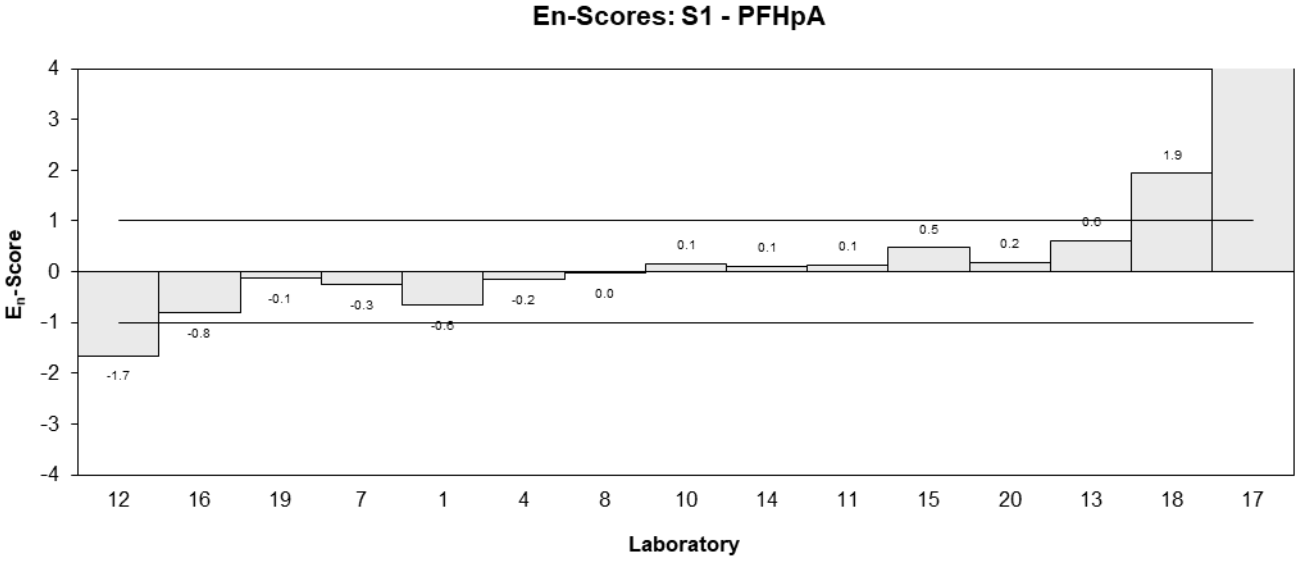

En-Scores: S1 - PFHpA
En-Score
-1.7 -0.8 -0.1 -0.3 -0.6 -0.2 0.0 0.1 0.1 0.1 0.5 0.2 0.6 1.9
12 16 19 7 1 4 8 10 14 11 15 20 13 18 17
Laboratory

Figure 12

Table 17

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	PFOA
Units	µg/kg

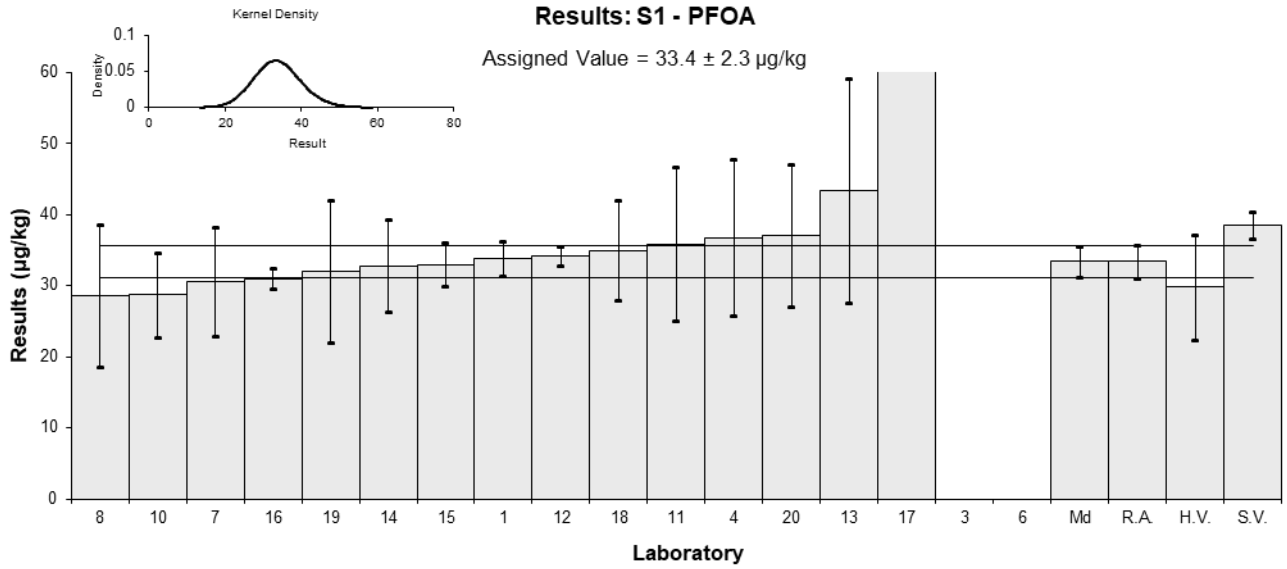
Participant Results

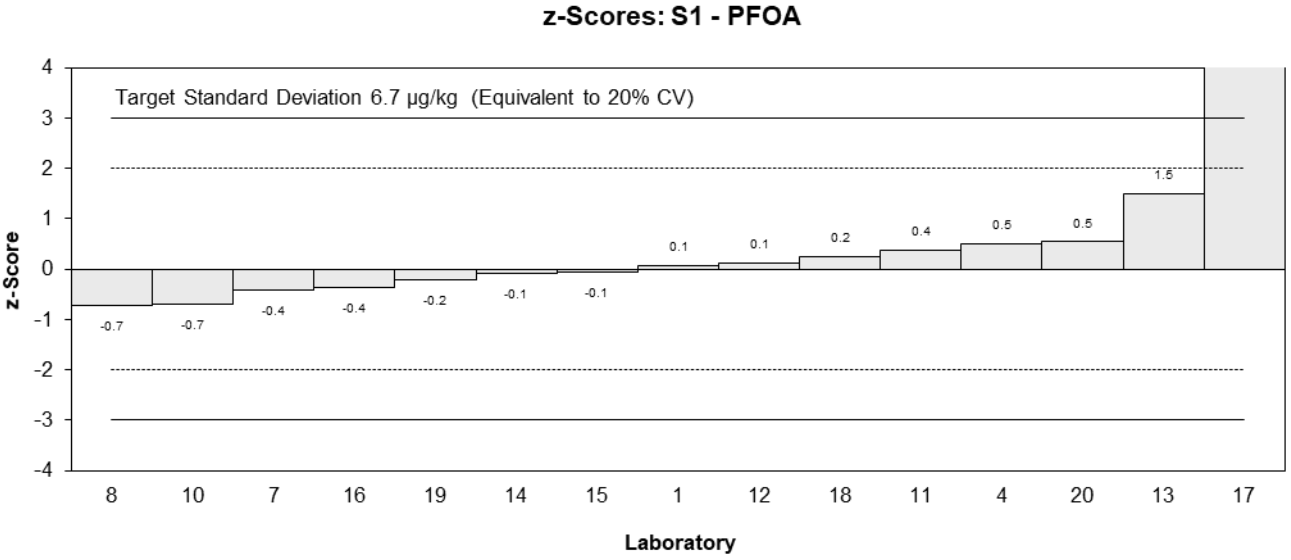
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	33.85	2.41	NR	0.07	0.14
3	NT	NT	NT		
4	36.78	11.03	76	0.51	0.30
6	NT	NT	NT		
7	30.6	7.6	87	-0.42	-0.35
8	28.53	9.99	58	-0.73	-0.48
10	28.7	6	66	-0.70	-0.73
11	35.891	10.767	104	0.37	0.23
12	34.215	1.415	77	0.12	0.30
13	43.4	15.8	136	1.50	0.63
14	32.8	6.50	87.8	-0.09	-0.09
15	33	3.0	116	-0.06	-0.11
16	31	1.45	81	-0.36	-0.88
17	122.23	12.69	77	13.30	6.89
18	35	7.0	NR	0.24	0.22
19	32	10	81	-0.21	-0.14
20	37	10	96	0.54	0.35

Statistics*

Assigned Value	33.4	2.3
Spike	38.6	1.9
Homogeneity Value	29.8	7.4
Robust Average	33.4	2.3
Median	33.4	2.1
Mean	33.8	
N	14	
Max.	43.4	
Min.	28.53	
Robust SD	3.5	
Robust CV	10%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Results: S1 - PFOA
Assigned Value = 33.4 ± 2.3 µg/kg
Kernel Density
Density
Result
Results (µg/kg)
Laboratory
8 10 7 16 19 14 15 1 12 18 11 4 20 13 17 3 6 Md R.A. H.V. S.V.


z-Scores: S1 - PFOA
Target Standard Deviation 6.7 µg/kg (Equivalent to 20% CV)
z-Score
Laboratory
8 10 7 16 19 14 15 1 12 18 11 4 20 13 17
-0.7 -0.7 -0.4 -0.4 -0.2 -0.1 -0.1 0.1 0.1 0.2 0.4 0.5 0.5 1.5

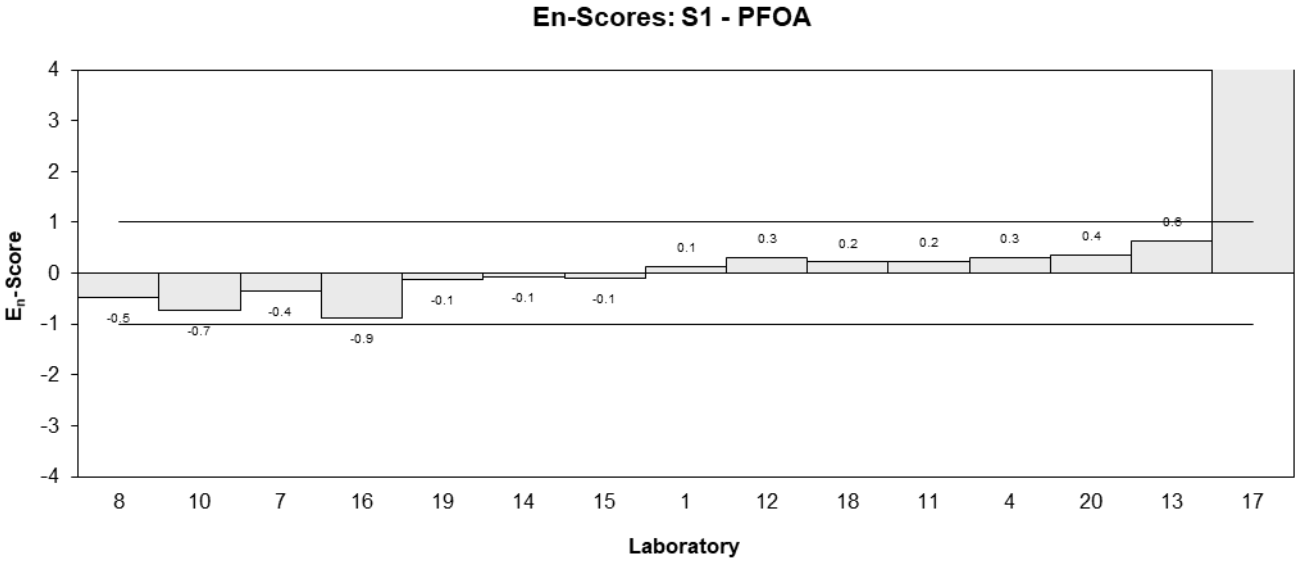

En-Scores: S1 - PFOA
En-Score
Laboratory
8 10 7 16 19 14 15 1 12 18 11 4 20 13 17
-0.5 -0.7 -0.4 -0.9 -0.1 -0.1 -0.1 0.1 0.3 0.2 0.2 0.3 0.4 0.6

Figure 13

Table 18

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	PFNA
Units	µg/kg

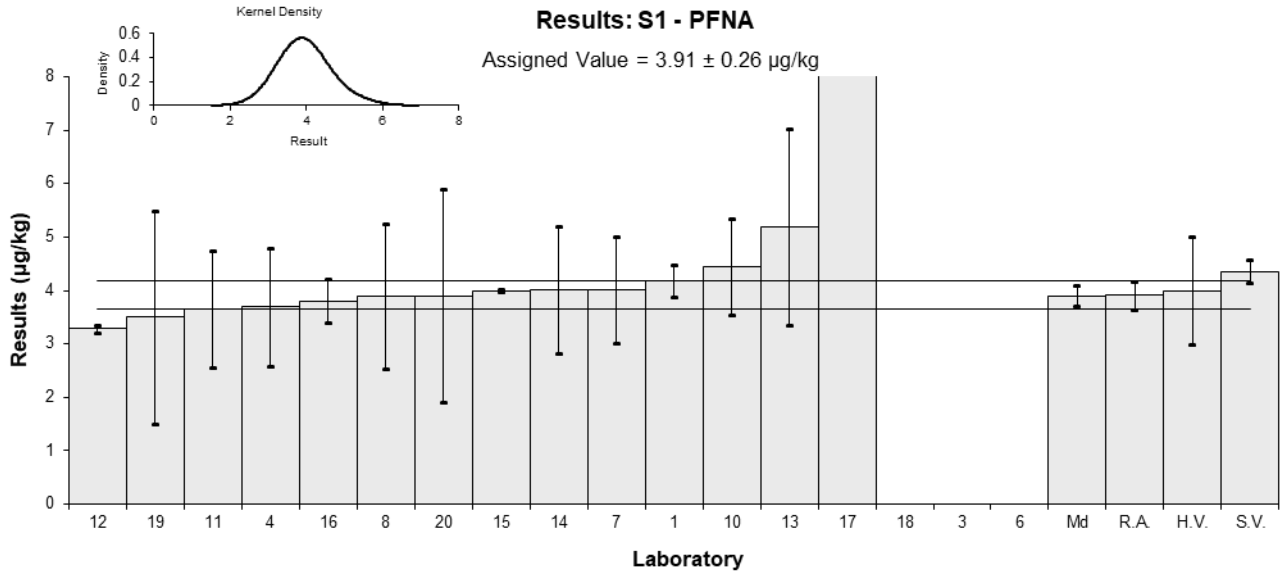
Participant Results

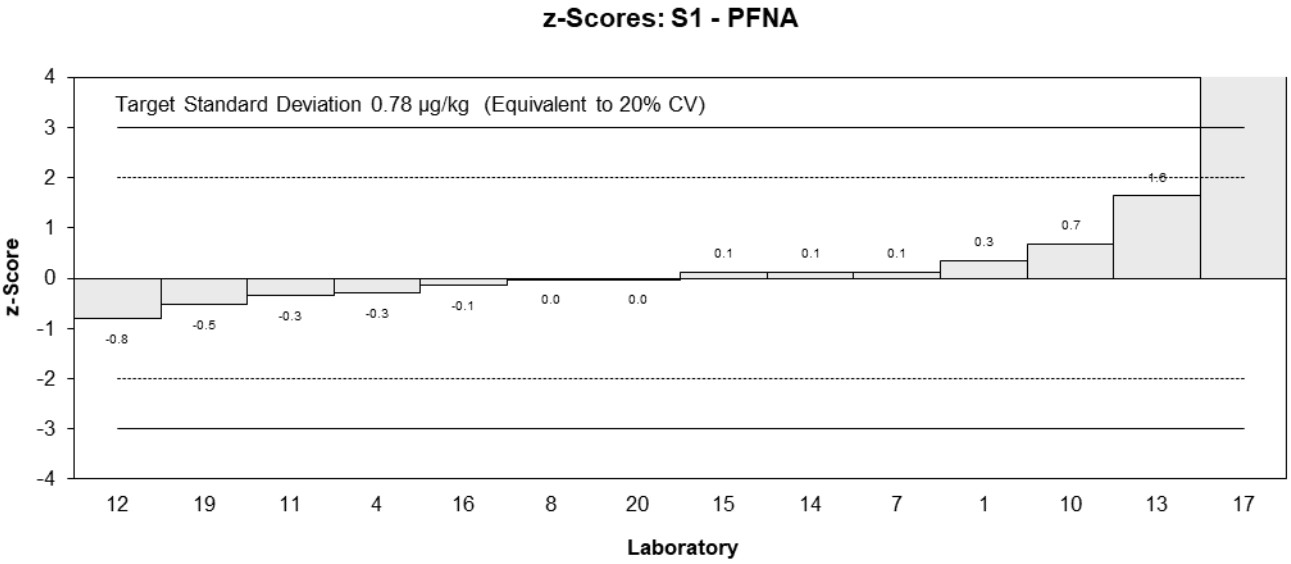
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	4.18	0.3	NR	0.35	0.68
3	NT	NT	NT		
4	3.69	1.11	120	-0.28	-0.19
6	NT	NT	NT		
7	4.01	1	77	0.13	0.10
8	3.89	1.36	71	-0.03	-0.01
10	4.44	0.9	55	0.68	0.57
11	3.641	1.092	96	-0.34	-0.24
12	3.284	0.078	77	-0.80	-2.31
13	5.19	1.83	149	1.64	0.69
14	4.01	1.19	87.1	0.13	0.08
15	4.0	0.034	116	0.12	0.34
16	3.8	0.41	82	-0.14	-0.23
17	17.53	1.48	77	17.42	9.06
18	NT	NT	NT		
19	3.5	2	82	-0.52	-0.20
20	3.9	2	95	-0.01	0.00

Statistics*

Assigned Value	3.91	0.26
Spike	4.36	0.22
Homogeneity Value	4.0	1.0
Robust Average	3.91	0.26
Median	3.90	0.19
Mean	3.96	
N	13	
Max.	5.19	
Min.	3.284	
Robust SD	0.38	
Robust CV	9.6%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S1 - PFNA
Assigned Value = 3.91 ± 0.26 µg/kg
Results (µg/kg)
12 19 11 4 16 8 20 15 14 7 1 10 13 17 18 3 6 Md R.A. H.V. S.V.
Laboratory


z-Scores: S1 - PFNA
Target Standard Deviation 0.78 µg/kg (Equivalent to 20% CV)
z-Score
-0.8 -0.5 -0.3 -0.3 -0.1 0.0 0.0 0.1 0.1 0.1 0.3 0.7 1.6
12 19 11 4 16 8 20 15 14 7 1 10 13 17
Laboratory

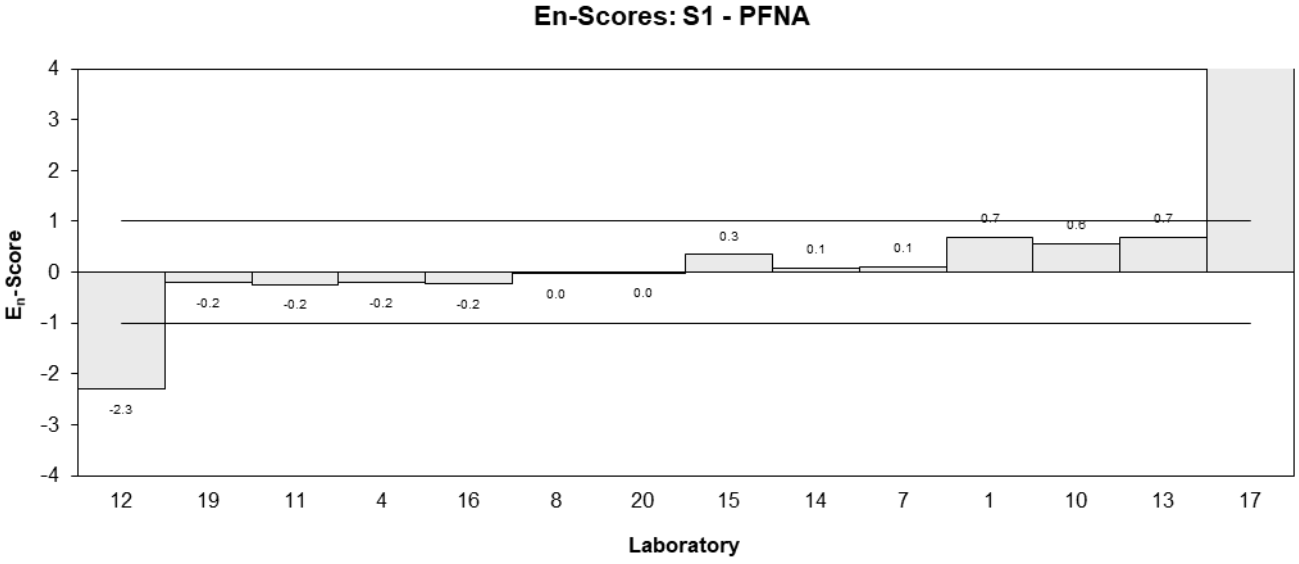

En-Scores: S1 - PFNA
En-Score
-2.3 -0.2 -0.2 -0.2 -0.2 0.0 0.0 0.3 0.1 0.1 0.7 0.6 0.7
12 19 11 4 16 8 20 15 14 7 1 10 13 17
Laboratory

Figure 14

Table 19

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	PFDA
Units	µg/kg

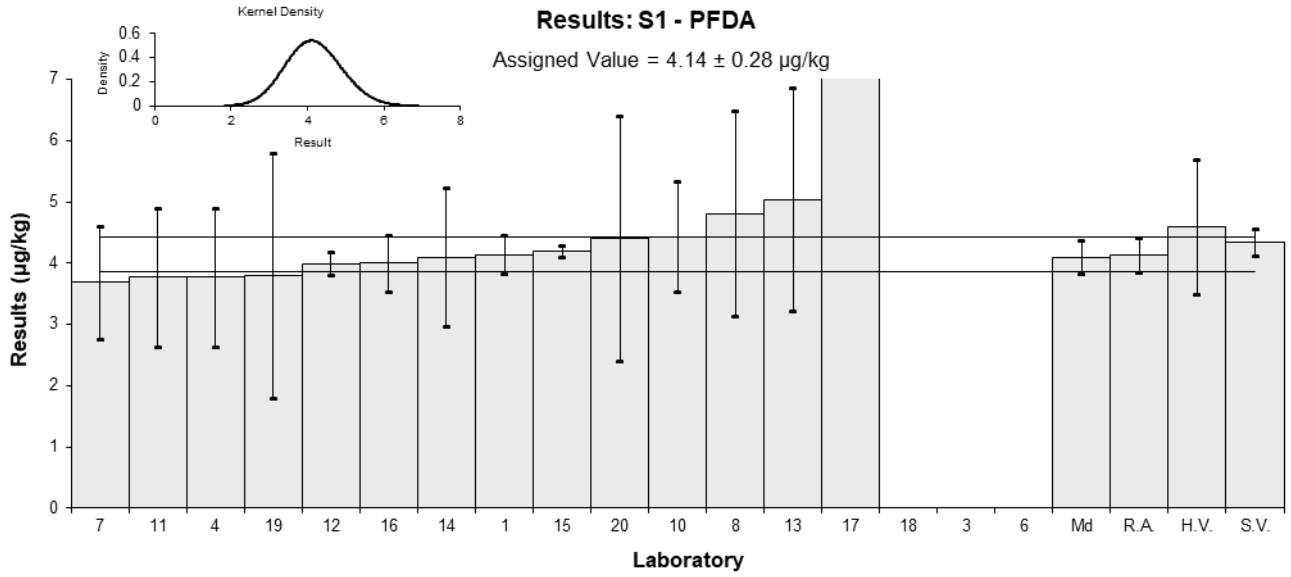
Participant Results

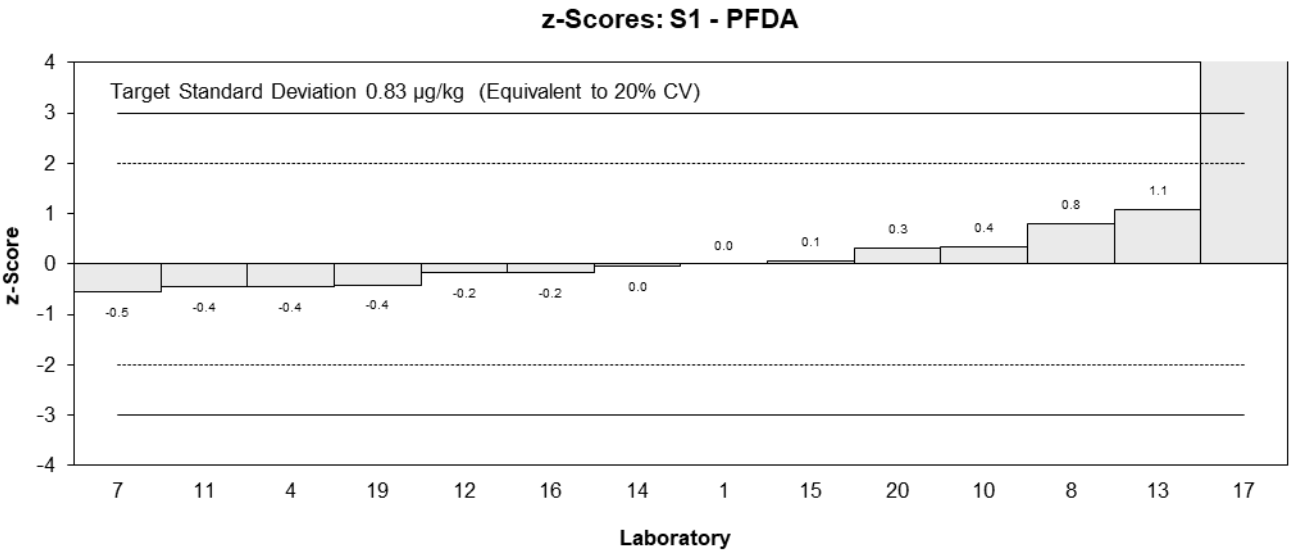
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	4.14	0.31	NR	0.00	0.00
3	NT	NT	NT		
4	3.77	1.13	132	-0.45	-0.32
6	NT	NT	NT		
7	3.69	0.92	90	-0.54	-0.47
8	4.81	1.68	86	0.81	0.39
10	4.43	0.9	71	0.35	0.31
11	3.770	1.131	99	-0.45	-0.32
12	3.999	0.196	77	-0.17	-0.41
13	5.04	1.82	168	1.09	0.49
14	4.10	1.13	67.9	-0.05	-0.03
15	4.2	0.086	120	0.07	0.20
16	4.0	0.46	99	-0.17	-0.26
17	15.96	1.35	77	14.28	8.57
18	NT	NT	NT		
19	3.8	2	83	-0.41	-0.17
20	4.4	2	102	0.31	0.13

Statistics*

Assigned Value	4.14	0.28
Spike	4.35	0.22
Homogeneity Value	4.6	1.1
Robust Average	4.14	0.28
Median	4.10	0.27
Mean	4.17	
N	13	
Max.	5.04	
Min.	3.69	
Robust SD	0.40	
Robust CV	9.7%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Results: S1 - PFDA
Assigned Value = 4.14 ± 0.28 µg/kg
Kernel Density
Density
Result
Results (µg/kg)
Laboratory
7 11 4 19 12 16 14 1 15 20 10 8 13 17 18 3 6 Md R.A. H.V. S.V.


z-Scores: S1 - PFDA
Target Standard Deviation 0.83 µg/kg (Equivalent to 20% CV)
z-Score
-0.5 -0.4 -0.4 -0.4 -0.2 -0.2 0.0 0.0 0.1 0.3 0.4 0.8 1.1
Laboratory
7 11 4 19 12 16 14 1 15 20 10 8 13 17

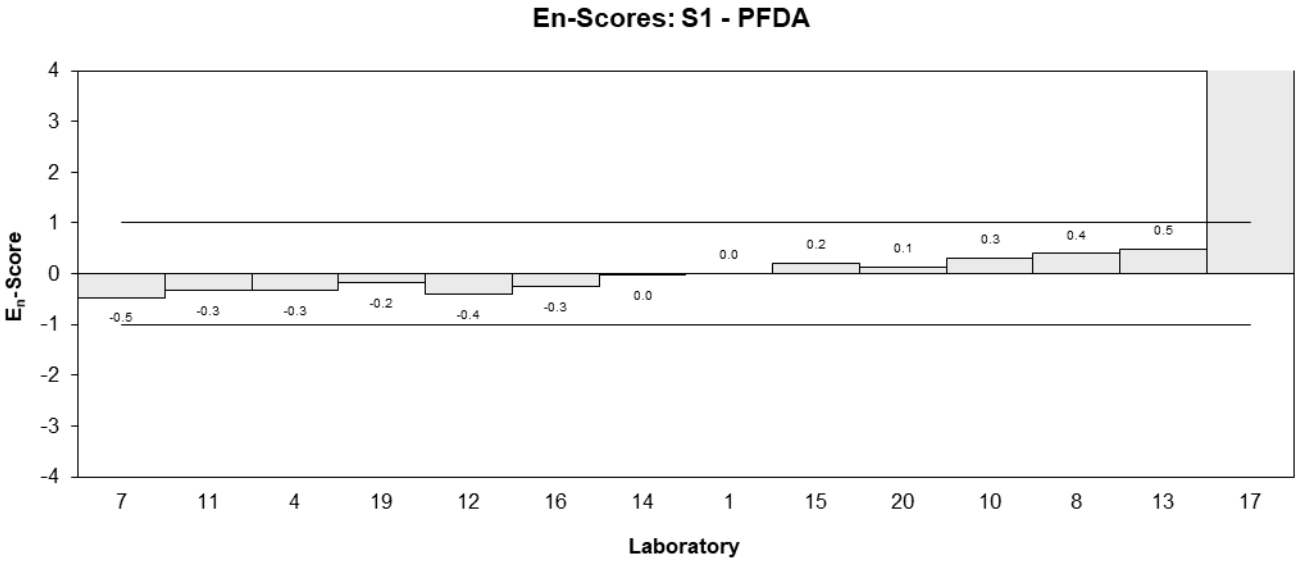

En-Scores: S1 - PFDA
En-Score
-0.5 -0.3 -0.3 -0.2 -0.4 -0.3 0.0 0.0 0.2 0.1 0.3 0.4 0.5
Laboratory
7 11 4 19 12 16 14 1 15 20 10 8 13 17

Figure 15

Table 20

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	PFUdA
Units	µg/kg

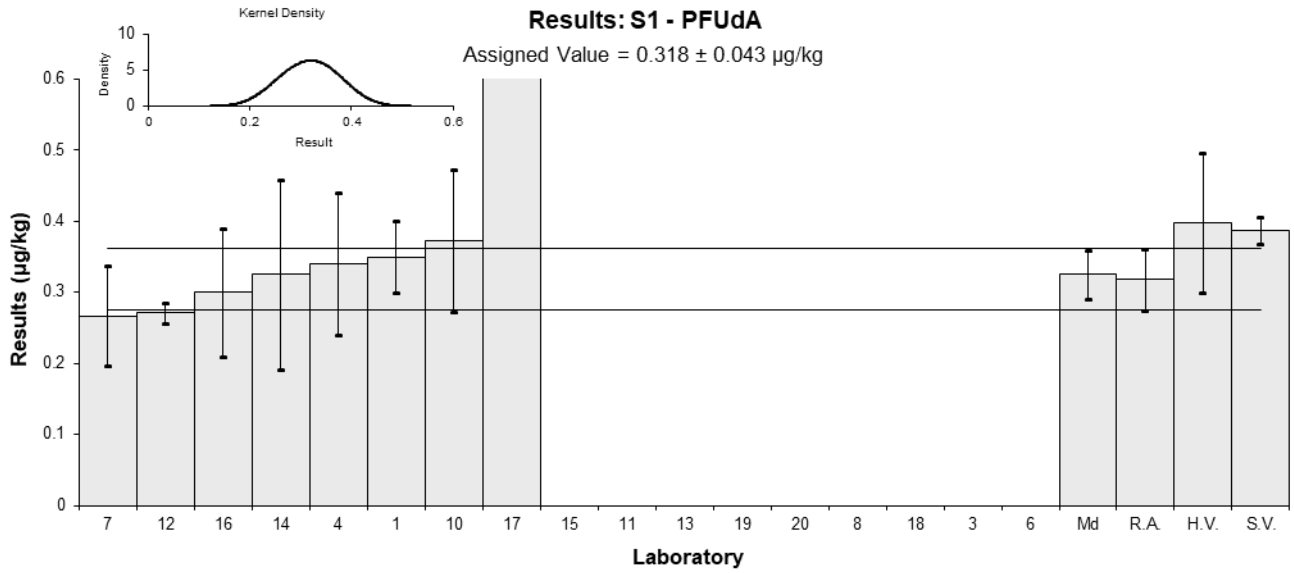
Participant Results

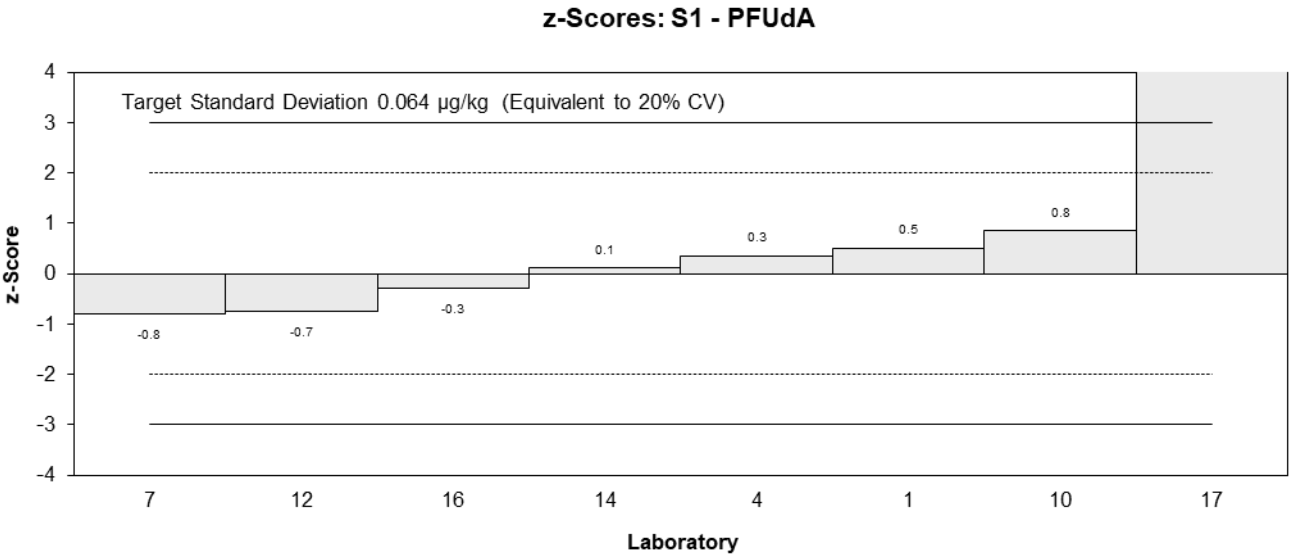
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.35	0.05	NR	0.50	0.49
3	NT	NT	NT		
4	0.34	0.10	114	0.35	0.20
6	NT	NT	NT		
7	0.267	0.07	90	-0.80	-0.62
8	NR	NR	NR		
10	0.372	0.1	70	0.85	0.50
11	<0.5	NR	NR		
12	0.271	0.0144	94	-0.74	-1.04
13	<1	NR	159		
14	0.325	0.134	70.6	0.11	0.05
15	< 1.0	NR	130		
16	0.3	0.09	97	-0.28	-0.18
17	1.21	0.06	78	14.03	12.08
18	NT	NT	NT		
19	<2	NR	91		
20	<2	NR	101		

Statistics*

Assigned Value	0.318	0.043
Spike	0.387	0.019
Homogeneity Value	0.398	0.099
Robust Average	0.318	0.043
Median	0.325	0.034
Mean	0.318	
N	7	
Max.	0.372	
Min.	0.267	
Robust SD	0.045	
Robust CV	14%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S1 - PFUdA
Assigned Value = 0.318 ± 0.043 µg/kg
Results (µg/kg)
Laboratory
7 12 16 14 4 1 10 17 15 11 13 19 20 8 18 3 6 Md R.A. H.V. S.V.


z-Scores: S1 - PFUdA
Target Standard Deviation 0.064 µg/kg (Equivalent to 20% CV)
z-Score
-0.8
-0.7
-0.3
0.1
0.3
0.5
0.8
Laboratory
7 12 16 14 4 1 10 17

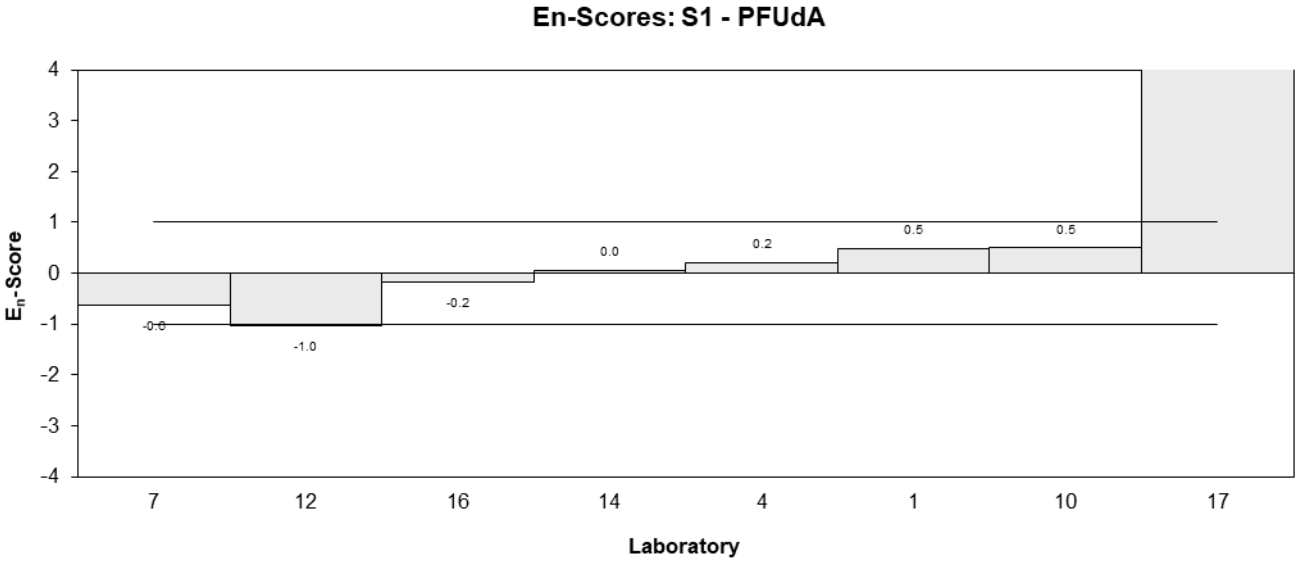

En-Scores: S1 - PFUdA
En-Score
-0.6
-1.0
-0.2
0.0
0.2
0.5
0.5
Laboratory
7 12 16 14 4 1 10 17

Figure 16

Table 21

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	8:2 FTS
Units	µg/kg

Participant Results

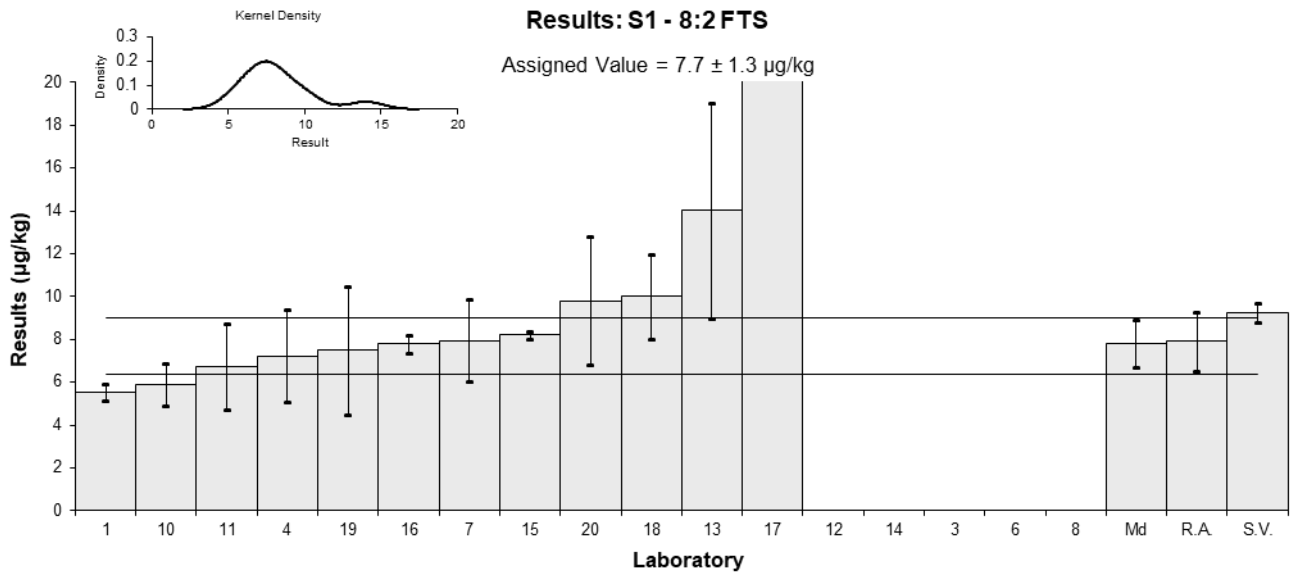
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	5.53	0.4	NR	-1.41	-1.60
3	NT	NT	NT		
4	7.23	2.17	96	-0.31	-0.19
6	NT	NT	NT		
7	7.95	1.9	112	0.16	0.11
8	NT	NT	NT		
10	5.91	1	94	-1.16	-1.09
11	6.738	2.022	220	-0.62	-0.40
12	NT	NT	NT		
13	14.01	5.02	286	4.10	1.22
14	NT	NT	NT		
15	8.2	0.16	104	0.32	0.38
16	7.8	0.41	129	0.06	0.07
17	36.17	2.29	59	18.49	10.81
18	10	2.0	NR	1.49	0.96
19	7.5	3	104	-0.13	-0.06
20	9.8	3	99	1.36	0.64

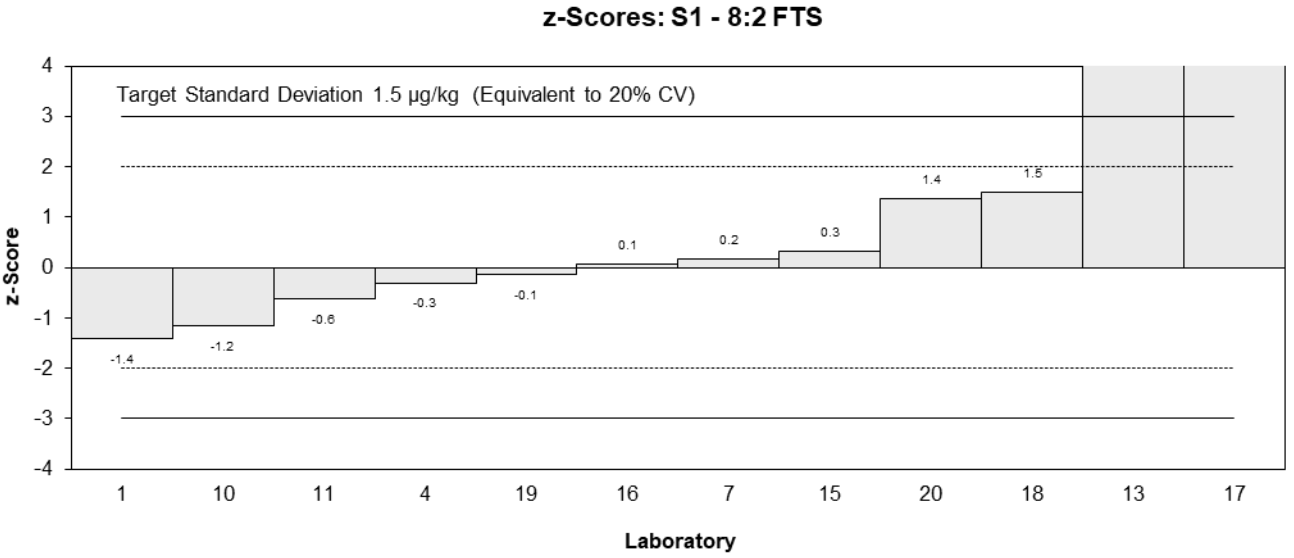
Statistics*

Assigned Value**	7.7	1.3
Spike	9.26	0.46
Robust Average	7.9	1.4
Median	7.8	1.1
Mean	8.2	
N	11	
Max.	14.01	
Min.	5.53	
Robust SD	1.9	
Robust CV	24%	

* Laboratory 17 was omitted from all statistical calculations (gross error).

** Robust average excluding Laboratory 13.


Kernel Density
Density
Result
Results: S1 - 8:2 FTS
Assigned Value = 7.7 ± 1.3 µg/kg
Results (µg/kg)
1 10 11 4 19 16 7 15 20 18 13 17 12 14 3 6 8 Md R.A. S.V.
Laboratory


z-Scores: S1 - 8:2 FTS
Target Standard Deviation 1.5 µg/kg (Equivalent to 20% CV)
z-Score
-1.4 -1.2 -0.6 -0.3 -0.1 0.1 0.2 0.3 1.4 1.5
1 10 11 4 19 16 7 15 20 18 13 17
Laboratory

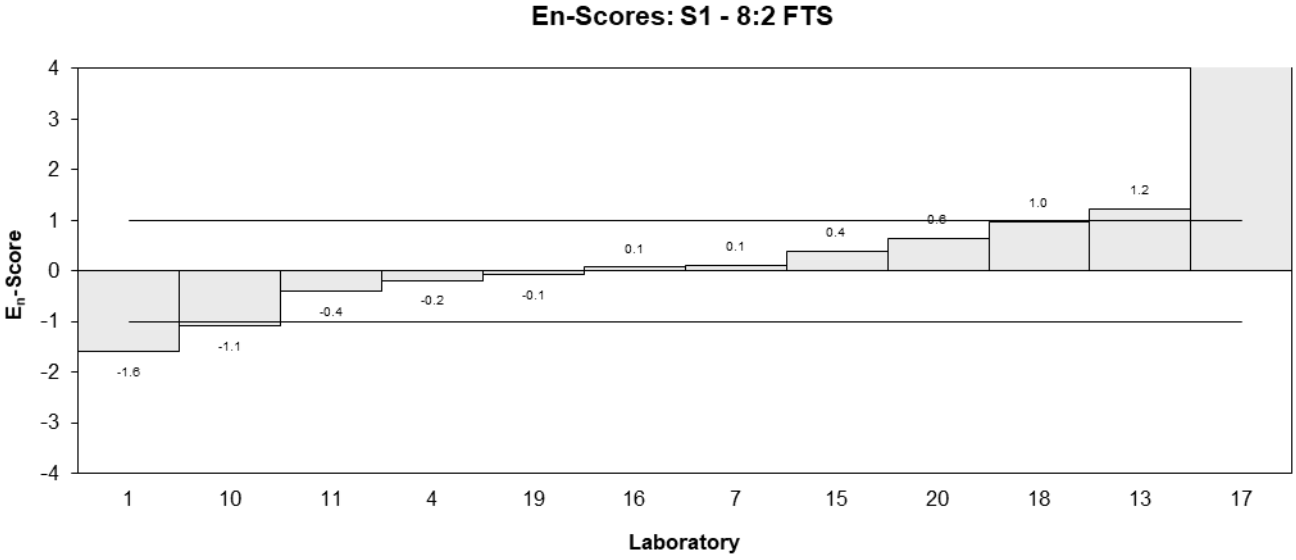

En-Scores: S1 - 8:2 FTS
En-Score
-1.6 -1.1 -0.4 -0.2 -0.1 0.1 0.1 0.4 0.6 1.0 1.2
1 10 11 4 19 16 7 15 20 18 13 17
Laboratory

Figure 17

Table 22

Sample Details

Sample No.	S1
Matrix	Meat
Analyte	GenX
Units	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Recovery
1	NT	NT	NT
3	NT	NT	NT
4	NT	NT	NT
6	NT	NT	NT
7	8.49	2.1	70
8	NT	NT	NT
10	NT	NT	NT
11	6.393	1.918	80
12	5.128	0.118	72
13	NT	NT	NT
14	NT	NT	NT
15	7.5	1.9	143
16	5.2	0.69	81
17	<0.01	NR	NR
18	NT	NT	NT
19	NT	NT	NT
20	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spike	7.28	0.36
Robust Average	6.5	1.9
Median	6.4	2.2
Mean	6.5	
N	5	
Max.	8.49	
Min.	5.128	
Robust SD	1.7	
Robust CV	25%	

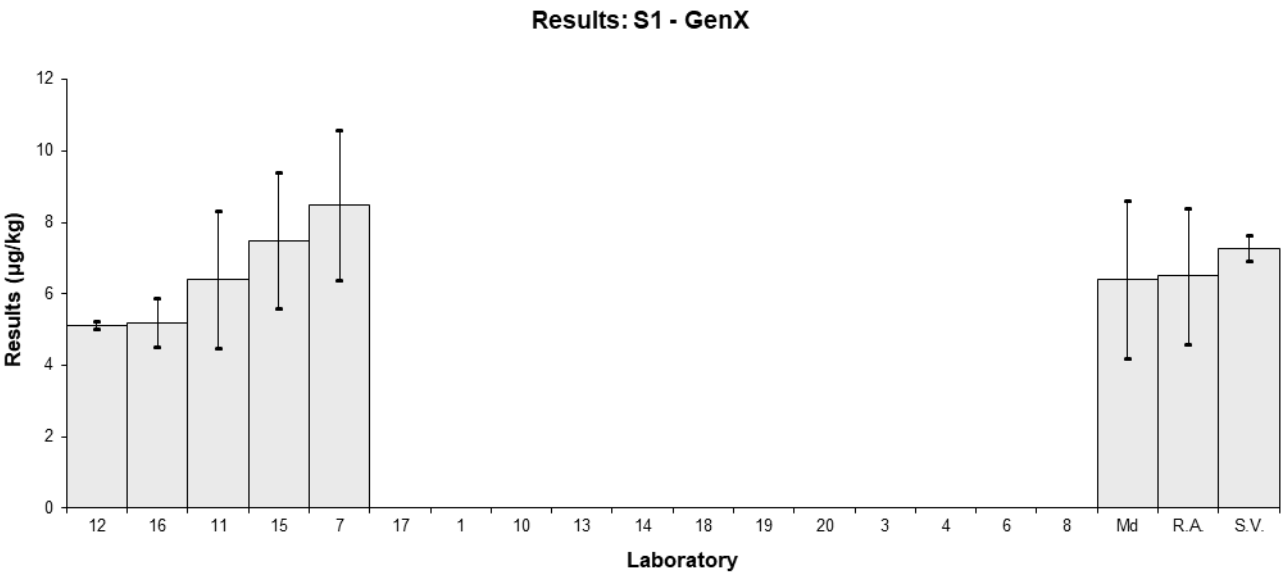

Results: S1 - GenX
Results (µg/kg)
0
2
4
6
8
10
12
12
16
11
15
7
17
1
10
13
14
18
19
20
3
4
6
8
Md
R.A.
S.V.
Laboratory

Figure 18

Table 23

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFBS
Units	µg/kg

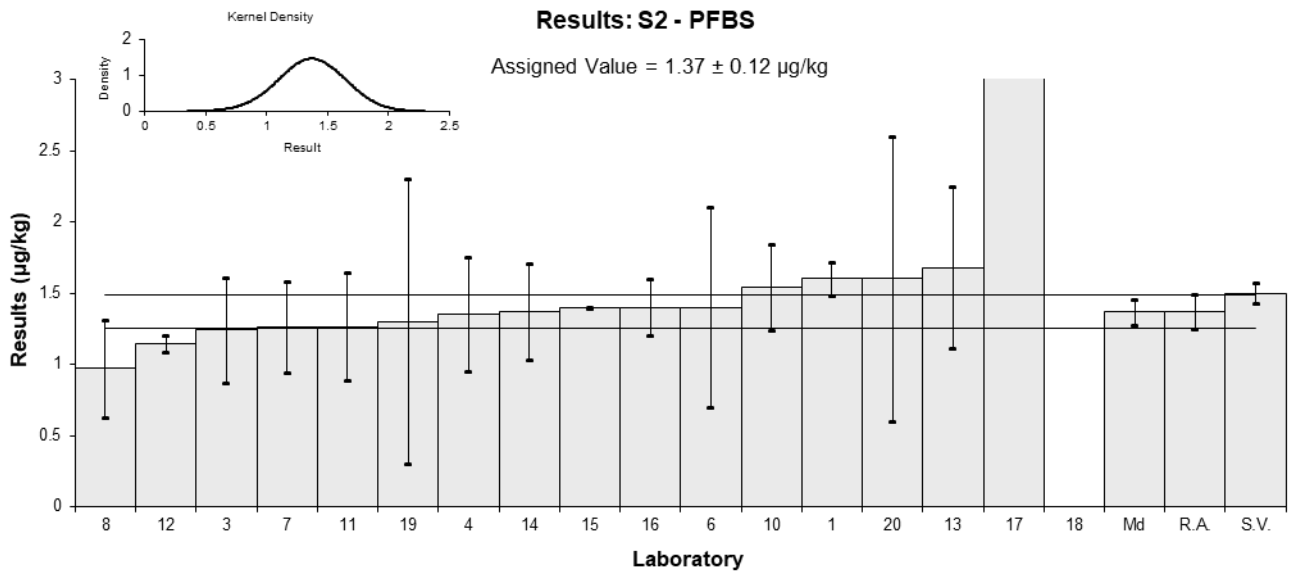
Participant Results

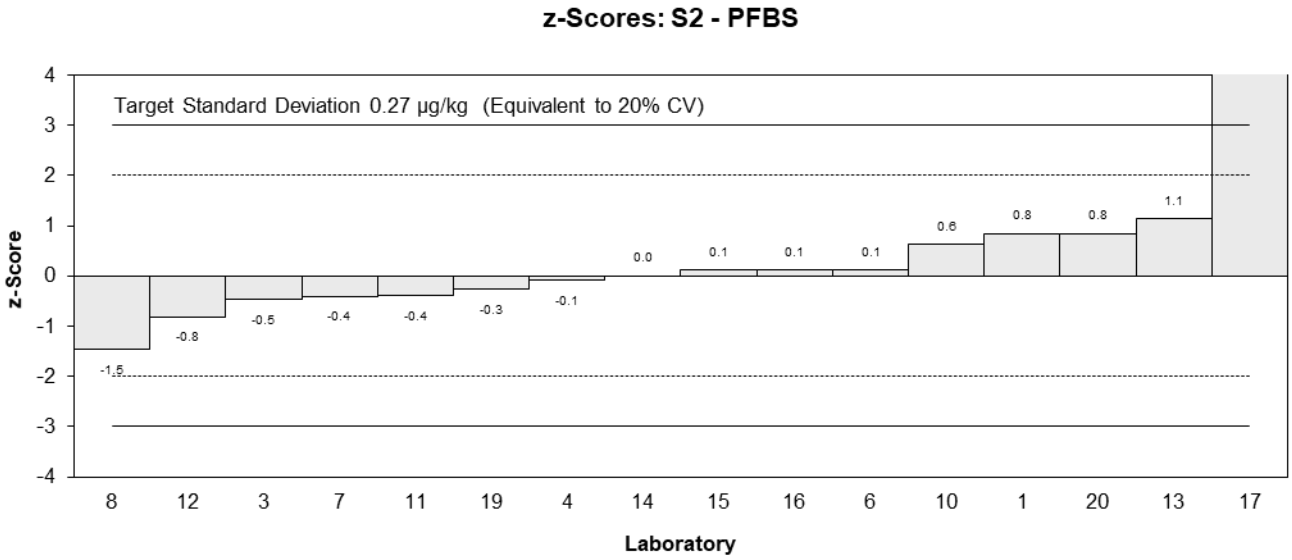
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	1.6	0.12	NR	0.84	1.36
3	1.24	0.372	70.3	-0.47	-0.33
4	1.35	0.40	115	-0.07	-0.05
6	1.4	0.70	96	0.11	0.04
7	1.26	0.32	84	-0.40	-0.32
8	0.97	0.34	134	-1.46	-1.11
10	1.54	0.3	96	0.62	0.53
11	1.263	0.379	88	-0.39	-0.27
12	1.148	0.057	89	-0.81	-1.67
13	1.68	0.57	128	1.13	0.53
14	1.37	0.337	92.7	0.00	0.00
15	1.4	0.0042	101	0.11	0.25
16	1.4	0.2	90	0.11	0.13
17	33.52	0.26	65.3	117.34	112.27
18	NR	NR	NR		
19	1.3	1	80	-0.26	-0.07
20	1.6	1	85	0.84	0.23

Statistics*

Assigned Value	1.37	0.12
Spike	1.50	0.07
Robust Average	1.37	0.12
Median	1.37	0.09
Mean	1.37	
N	15	
Max.	1.68	
Min.	0.97	
Robust SD	0.19	
Robust CV	14%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Results: S2 - PFBS
Kernel Density
Density
Result
Assigned Value = 1.37 ± 0.12 µg/kg
Results (µg/kg)
8 12 3 7 11 19 4 14 15 16 6 10 1 20 13 17 18 Md R.A. S.V.
Laboratory


z-Scores: S2 - PFBS
Target Standard Deviation 0.27 µg/kg (Equivalent to 20% CV)
z-Score
-1.5 -0.8 -0.5 -0.4 -0.4 -0.3 -0.1 0.0 0.1 0.1 0.1 0.6 0.8 0.8 1.1
8 12 3 7 11 19 4 14 15 16 6 10 1 20 13 17
Laboratory

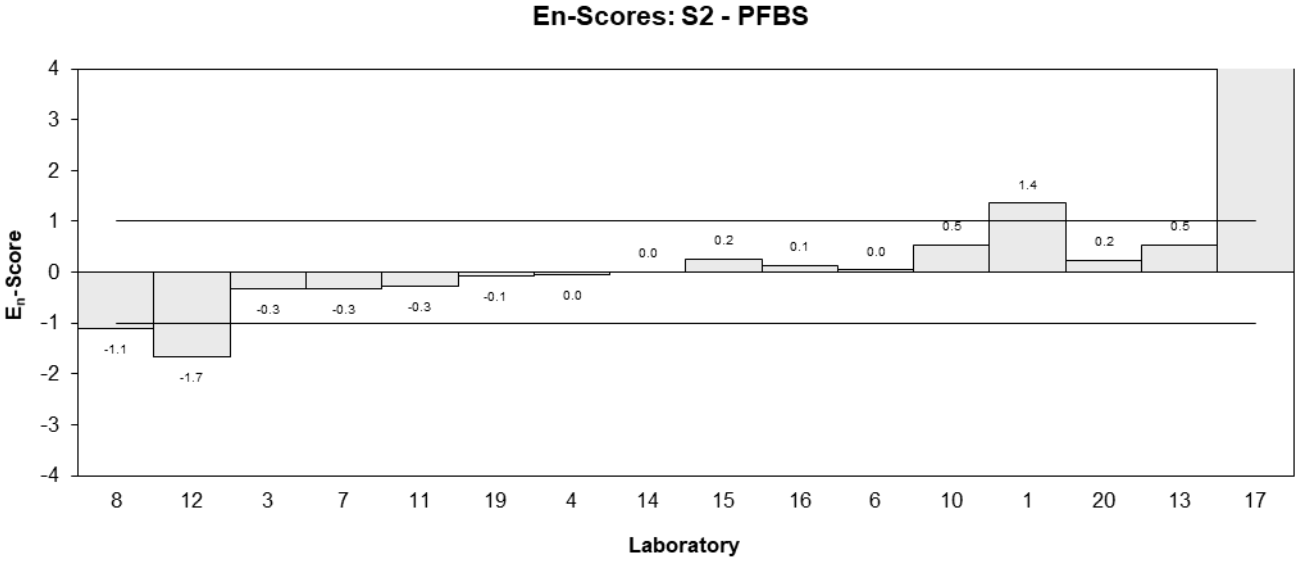

En-Scores: S2 - PFBS
En-Score
-1.1 -1.7 -0.3 -0.3 -0.3 -0.1 0.0 0.0 0.2 0.1 0.0 0.5 1.4 0.2 0.5
8 12 3 7 11 19 4 14 15 16 6 10 1 20 13 17
Laboratory

Figure 19

Table 24

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFPeS
Units	µg/kg

Participant Results

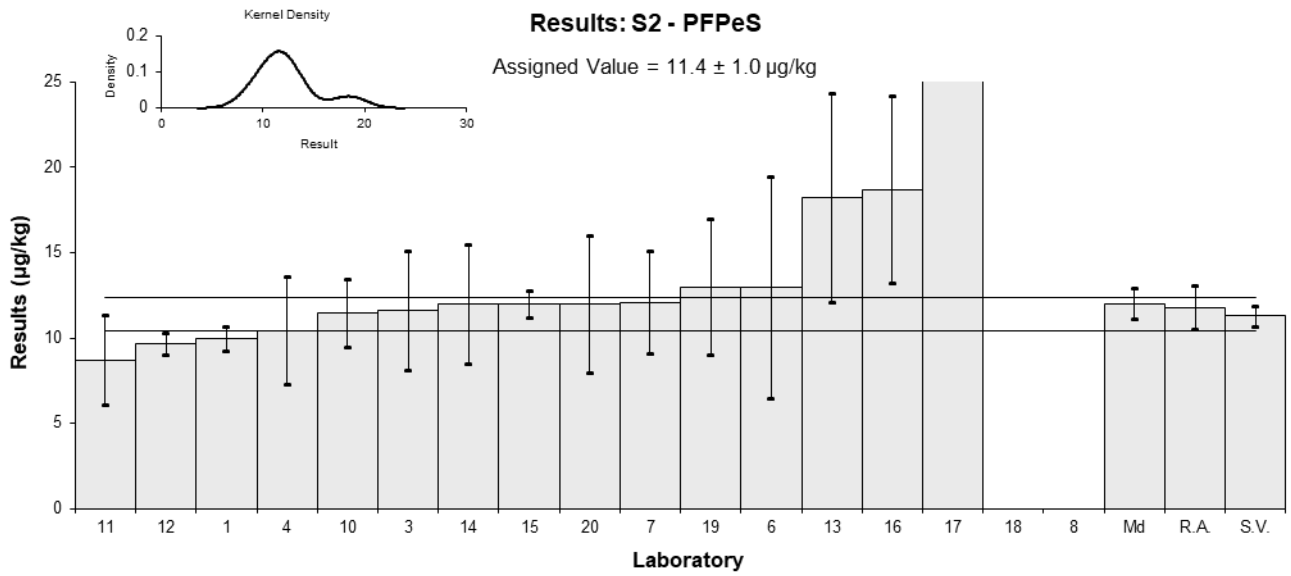
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	10	0.71	NR	-0.61	-1.14
3	11.6	3.48	70.3	0.09	0.06
4	10.45	3.13	NR	-0.42	-0.29
6	13	6.5	NR	0.70	0.24
7	12.1	3	79	0.31	0.22
8	NT	NT	NT		
10	11.5	2	NR	0.04	0.04
11	8.713	2.614	94	-1.18	-0.96
12	9.682	0.628	94	-0.75	-1.45
13	18.21	6.12	128	2.99	1.10
14	12.0	3.51	92.7	0.26	0.16
15	12	0.76	129	0.26	0.48
16	18.7	5.5	107	3.20	1.31
17	105.29	2.6	65	41.18	33.70
18	NR	NR	NR		
19	13	4	85	0.70	0.39
20	12	4	90	0.26	0.15

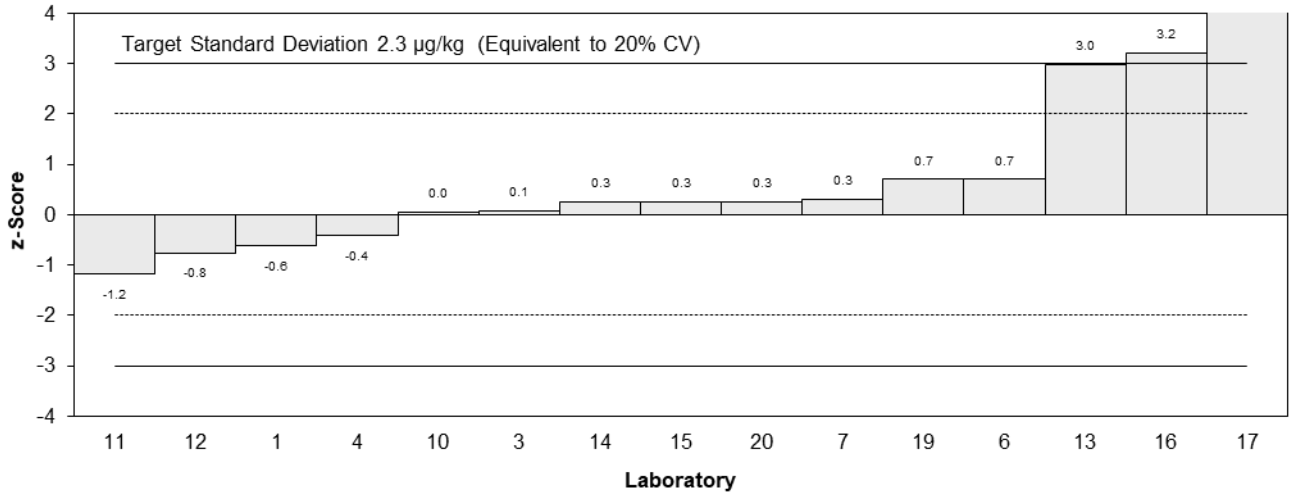
Statistics*

Assigned Value**	11.4	1.0
Spike	11.3	0.6
Robust Average	11.8	1.3
Median	12.0	0.9
Mean	12.4	
N	14	
Max.	18.7	
Min.	8.713	
Robust SD	2.0	
Robust CV	17%	

* Laboratory 17 was omitted from all statistical calculations (gross error).

** Robust average excluding Laboratories 13 and 16.


Kernel Density
Density
Result
Results: S2 - PFPeS
Assigned Value = 11.4 ± 1.0 µg/kg
Results (µg/kg)
Laboratory
11 12 1 4 10 3 14 15 20 7 19 6 13 16 17 18 8 Md R.A. S.V.


z-Scores: S2 - PFPeS
Target Standard Deviation 2.3 µg/kg (Equivalent to 20% CV)
z-Score
Laboratory
11 12 1 4 10 3 14 15 20 7 19 6 13 16 17
-1.2 -0.8 -0.6 -0.4 0.0 0.1 0.3 0.3 0.3 0.3 0.7 0.7 3.0 3.2

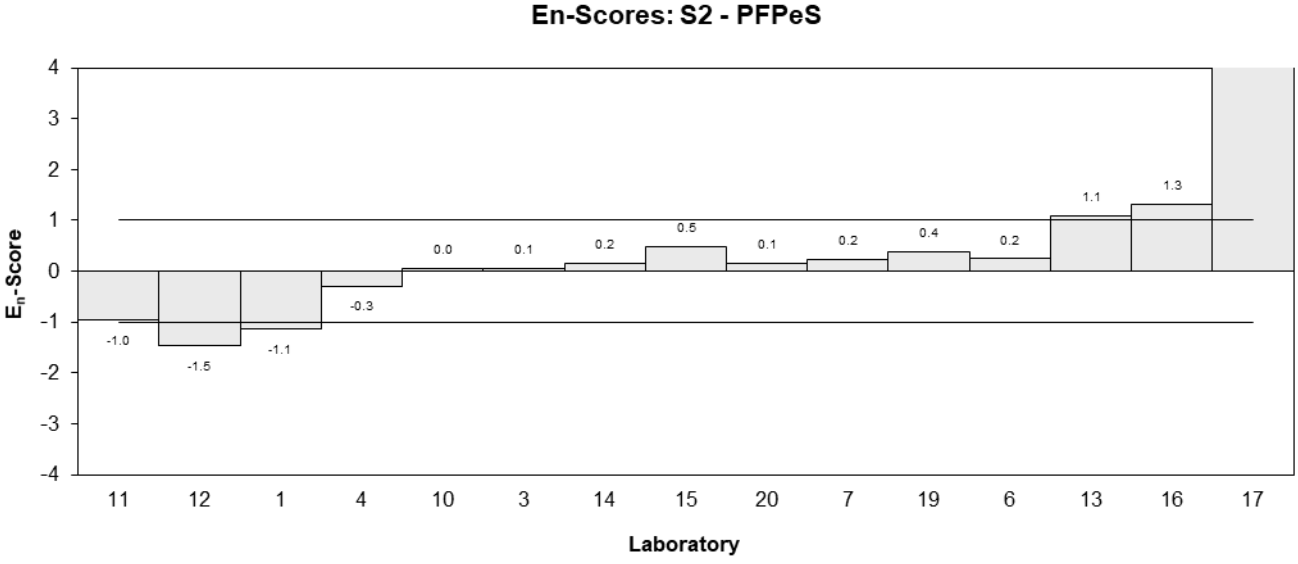

En-Scores: S2 - PFPeS
En-Score
Laboratory
11 12 1 4 10 3 14 15 20 7 19 6 13 16 17
-1.0 -1.5 -1.1 -0.3 0.0 0.1 0.2 0.5 0.1 0.2 0.4 0.2 1.1 1.3

Figure 20

Table 25

Sample Details

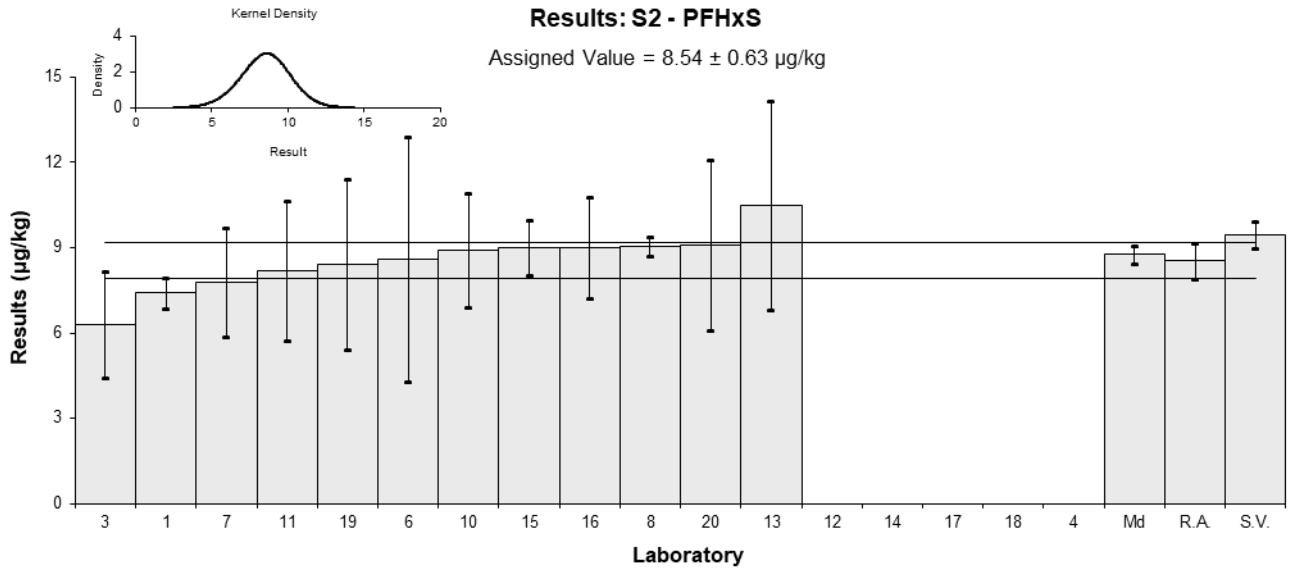
Sample No.	S2
Matrix	Celery
Analyte	PFHxS
Units	µg/kg

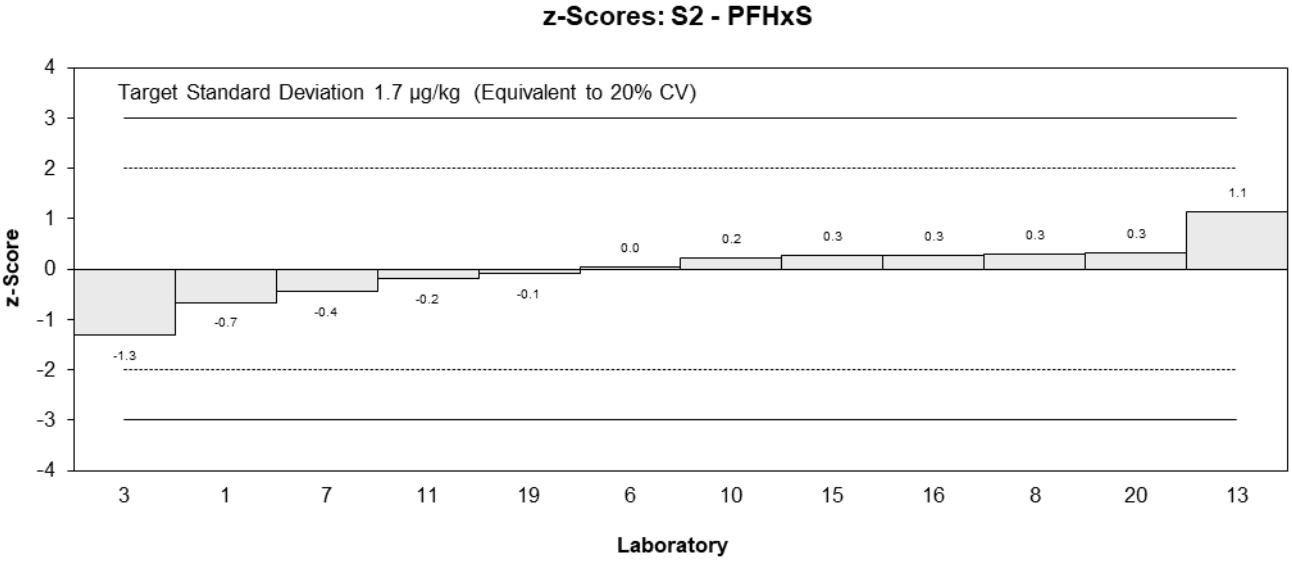
Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	7.41	0.53	NR	-0.66	-1.37
3	6.3	1.89	84.5	-1.31	-1.12
4	NT	NT	NT		
6	8.6	4.3	87	0.04	0.01
7	7.78	1.9	89	-0.44	-0.38
8	9.04	0.33	92	0.29	0.70
10	8.92	2	NR	0.22	0.18
11	8.199	2.460	94	-0.20	-0.13
12	NR	NR	NR		
13	10.5	3.68	136	1.15	0.52
14	NR	NR	NR		
15	9.0	0.96	101	0.27	0.40
16	9	1.8	91	0.27	0.24
17	NR	NR	NR		
18	NR	NR	NR		
19	8.4	3	85	-0.08	-0.05
20	9.1	3	90	0.33	0.18

Statistics

Assigned Value	8.54	0.63
Spike	9.45	0.47
Robust Average	8.54	0.63
Median	8.76	0.33
Mean	8.52	
N	12	
Max.	10.5	
Min.	6.3	
Robust SD	0.88	
Robust CV	10%	


Results: S2 - PFHxS
Assigned Value = 8.54 ± 0.63 µg/kg
Kernel Density
Density
Result
Results (µg/kg)
Laboratory
3 1 7 11 19 6 10 15 16 8 20 13 12 14 17 18 4 Md R.A. S.V.


z-Scores: S2 - PFHxS
Target Standard Deviation 1.7 µg/kg (Equivalent to 20% CV)
z-Score
Laboratory
-1.3 -0.7 -0.4 -0.2 -0.1 0.0 0.2 0.3 0.3 0.3 0.3 1.1
3 1 7 11 19 6 10 15 16 8 20 13

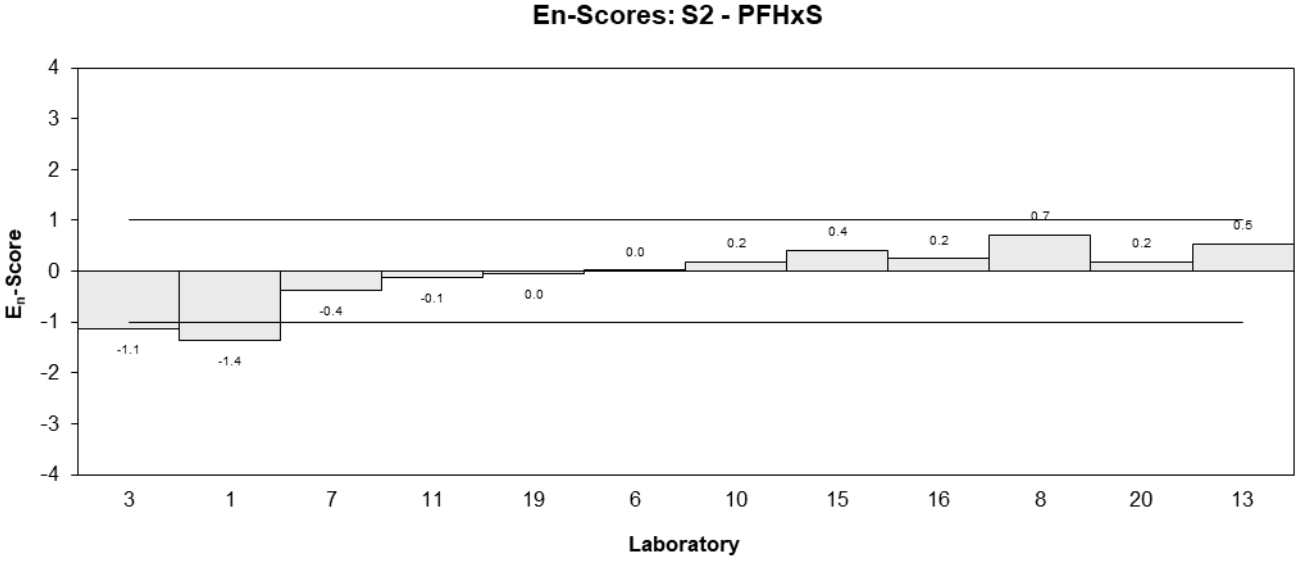

En-Scores: S2 - PFHxS
En-Score
Laboratory
-1.1 -1.4 -0.4 -0.1 0.0 0.0 0.2 0.4 0.2 0.7 0.2 0.5
3 1 7 11 19 6 10 15 16 8 20 13

Figure 21

Table 26

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFHxS (linear)
Units	µg/kg

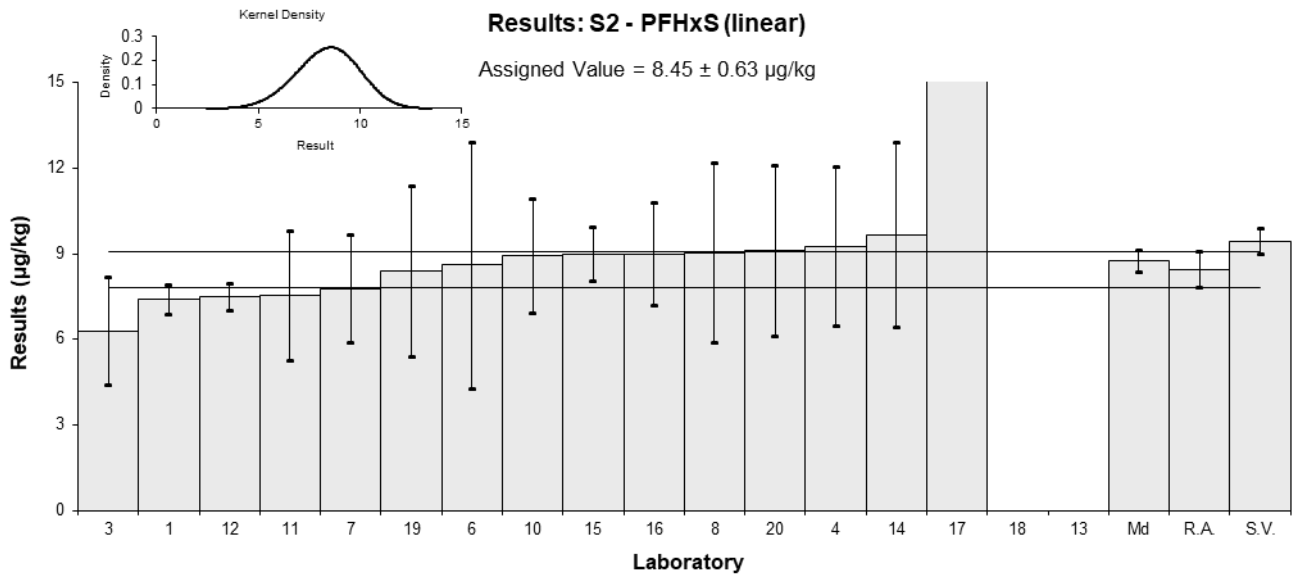
Participant Results

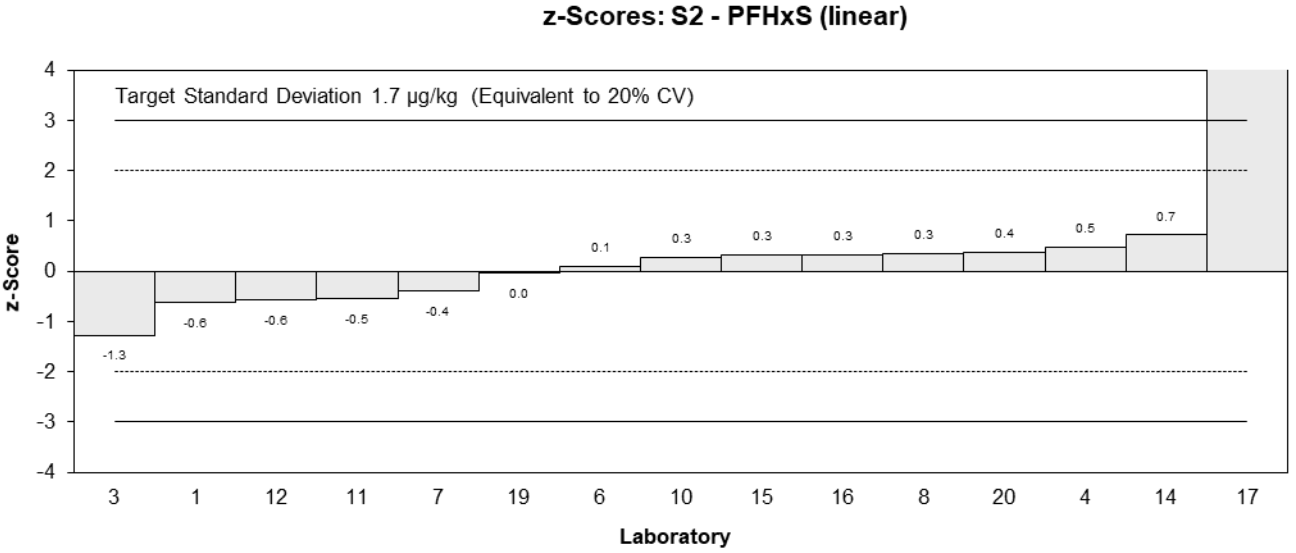
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	7.41	0.53	NR	-0.62	-1.26
3	6.3	1.89	84.5	-1.27	-1.08
4	9.26	2.78	118	0.48	0.28
6	8.6	4.3	NR	0.09	0.03
7	7.78	1.9	88	-0.40	-0.33
8	9.04	3.16	92	0.35	0.18
10	8.92	2	85	0.28	0.22
11	7.548	2.264	94	-0.53	-0.38
12	7.503	0.479	94	-0.56	-1.20
13	NT	NT	NT		
14	9.67	3.25	92.7	0.72	0.37
15	9.0	0.96	101	0.33	0.48
16	9	1.8	91	0.33	0.29
17	158.7	3.5	45.8	88.91	42.25
18	NR	NR	NR		
19	8.4	3	85	-0.03	-0.02
20	9.1	3	90	0.38	0.21

Statistics*

Assigned Value	8.45	0.63
Spike	9.45	0.47
Robust Average	8.45	0.63
Median	8.76	0.37
Mean	8.40	
N	14	
Max.	9.67	
Min.	6.3	
Robust SD	0.94	
Robust CV	11%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S2 - PFHxS (linear)
Assigned Value = 8.45 ± 0.63 µg/kg
Results (µg/kg)
Laboratory
3 1 12 11 7 19 6 10 15 16 8 20 4 14 17 18 13 Md R.A. S.V.


z-Scores: S2 - PFHxS (linear)
Target Standard Deviation 1.7 µg/kg (Equivalent to 20% CV)
z-Score
Laboratory
3 1 12 11 7 19 6 10 15 16 8 20 4 14 17
-1.3 -0.6 -0.6 -0.5 -0.4 0.0 0.1 0.3 0.3 0.3 0.3 0.4 0.5 0.7

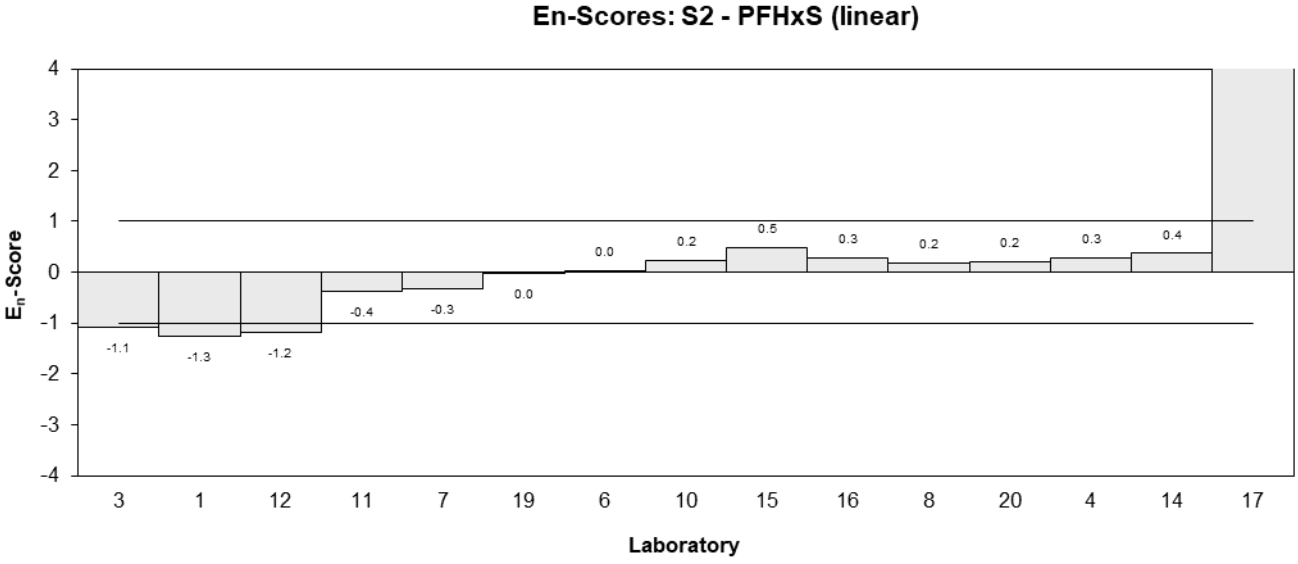

En-Scores: S2 - PFHxS (linear)
En-Score
Laboratory
3 1 12 11 7 19 6 10 15 16 8 20 4 14 17
-1.1 -1.3 -1.2 -0.4 -0.3 0.0 0.0 0.2 0.5 0.3 0.2 0.2 0.3 0.4

Figure 22

Table 27

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFHpS
Units	µg/kg

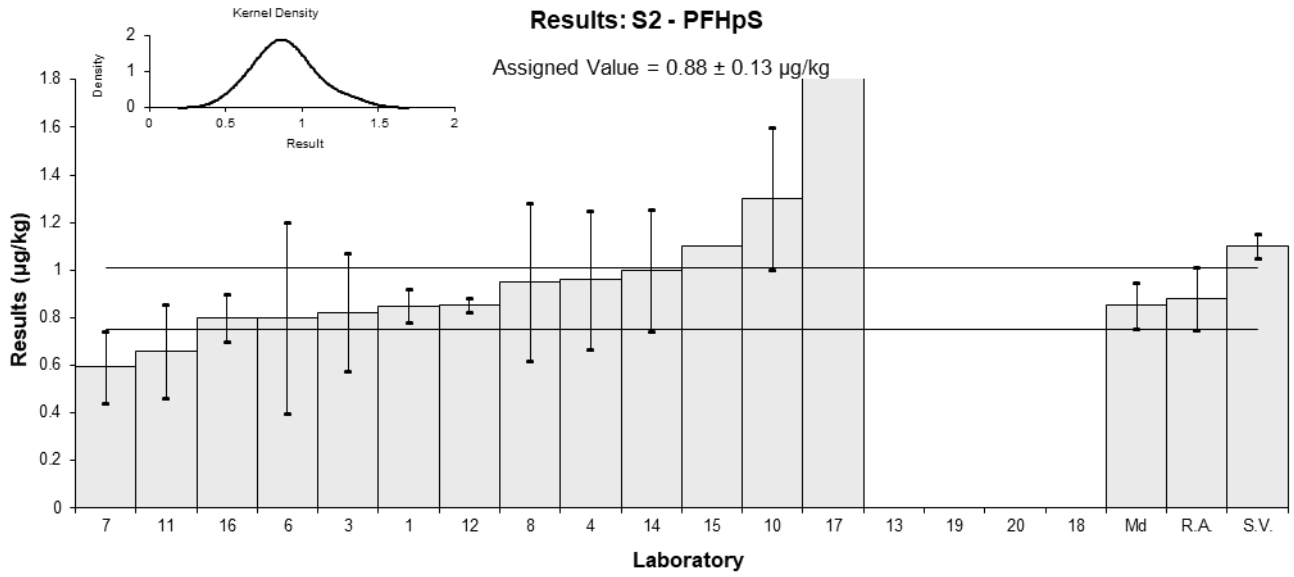
Participant Results

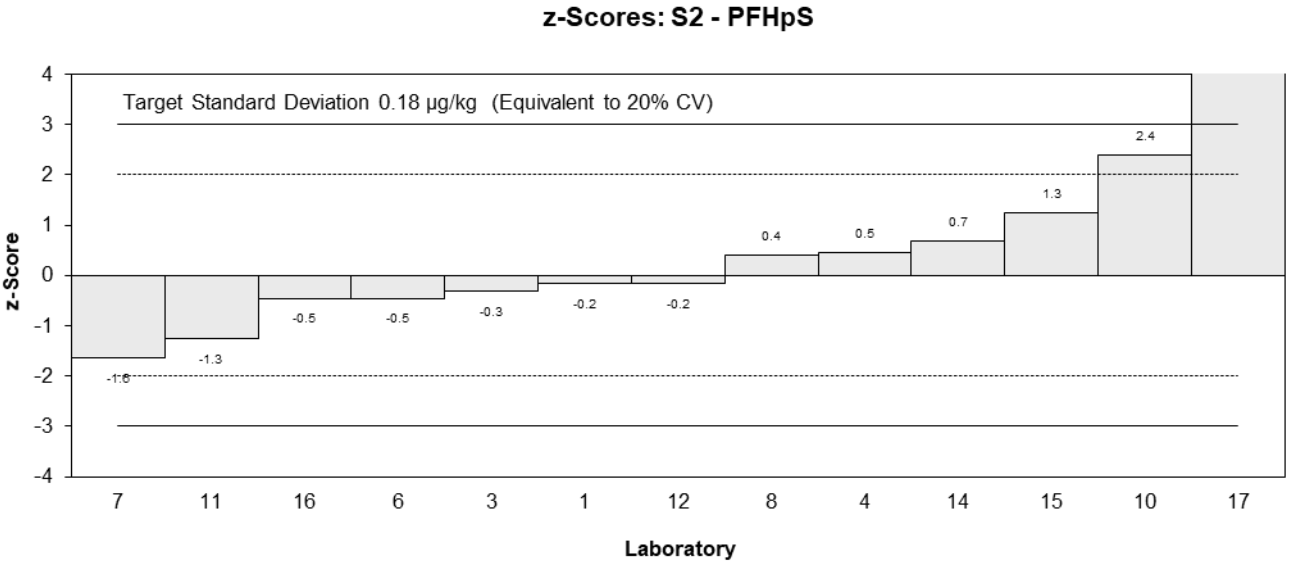
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.85	0.07	NR	-0.17	-0.20
3	0.823	0.247	84.5	-0.32	-0.20
4	0.96	0.29	NR	0.45	0.25
6	0.8	0.40	NR	-0.45	-0.19
7	0.592	0.15	88	-1.64	-1.45
8	0.95	0.33	101	0.40	0.20
10	1.3	0.3	NR	2.39	1.28
11	0.658	0.197	94	-1.26	-0.94
12	0.853	0.031	94	-0.15	-0.20
13	<1	NR	137		
14	0.999	0.256	92.7	0.68	0.41
15	1.1	NR	101	1.25	1.69
16	0.8	0.1	91	-0.45	-0.49
17	6.32	0.28	NR	30.91	17.62
18	NR	NR	NR		
19	<1	NR	85		
20	<1	NR	90		

Statistics*

Assigned Value	0.88	0.13
Spike	1.10	0.05
Robust Average	0.88	0.13
Median	0.852	0.097
Mean	0.890	
N	12	
Max.	1.3	
Min.	0.592	
Robust SD	0.19	
Robust CV	21%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S2 - PFHpS
Assigned Value = 0.88 ± 0.13 µg/kg
Results (µg/kg)
7 11 16 6 3 1 12 8 4 14 15 10 17 13 19 20 18 Md R.A. S.V.
Laboratory


z-Scores: S2 - PFHpS
Target Standard Deviation 0.18 µg/kg (Equivalent to 20% CV)
z-Score
-1.6 -1.3 -0.5 -0.5 -0.3 -0.2 -0.2 0.4 0.5 0.7 1.3 2.4
7 11 16 6 3 1 12 8 4 14 15 10 17
Laboratory

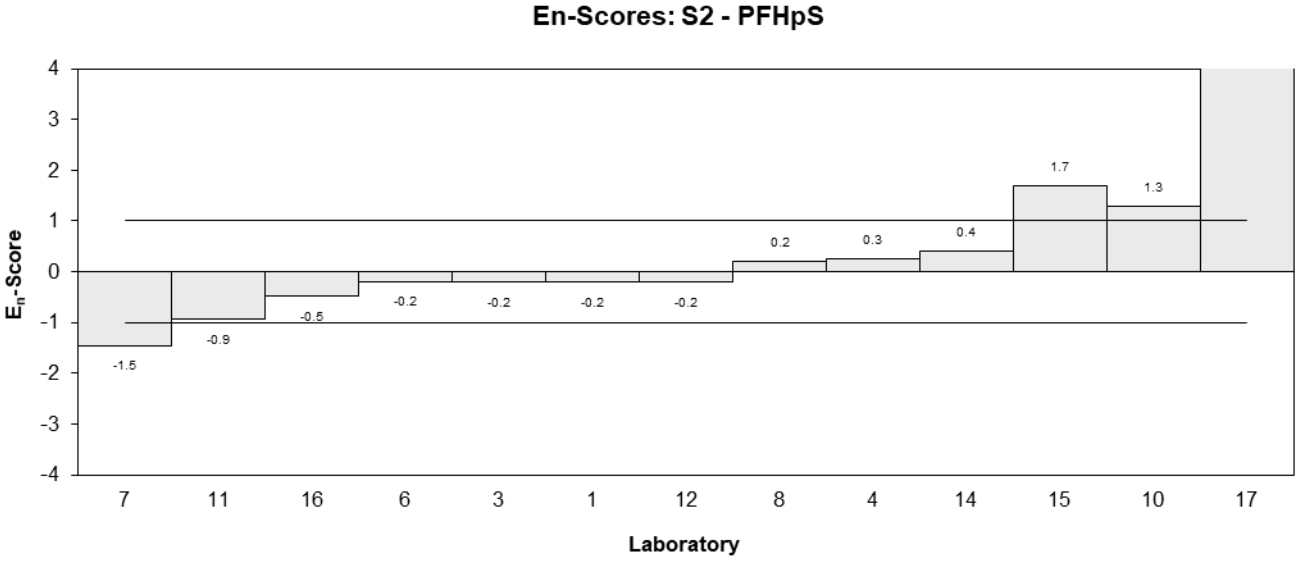

En-Scores: S2 - PFHpS
En-Score
-1.5 -0.9 -0.5 -0.2 -0.2 -0.2 -0.2 0.2 0.3 0.4 1.7 1.3
7 11 16 6 3 1 12 8 4 14 15 10 17
Laboratory

Figure 23

Table 28

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFOS
Units	µg/kg

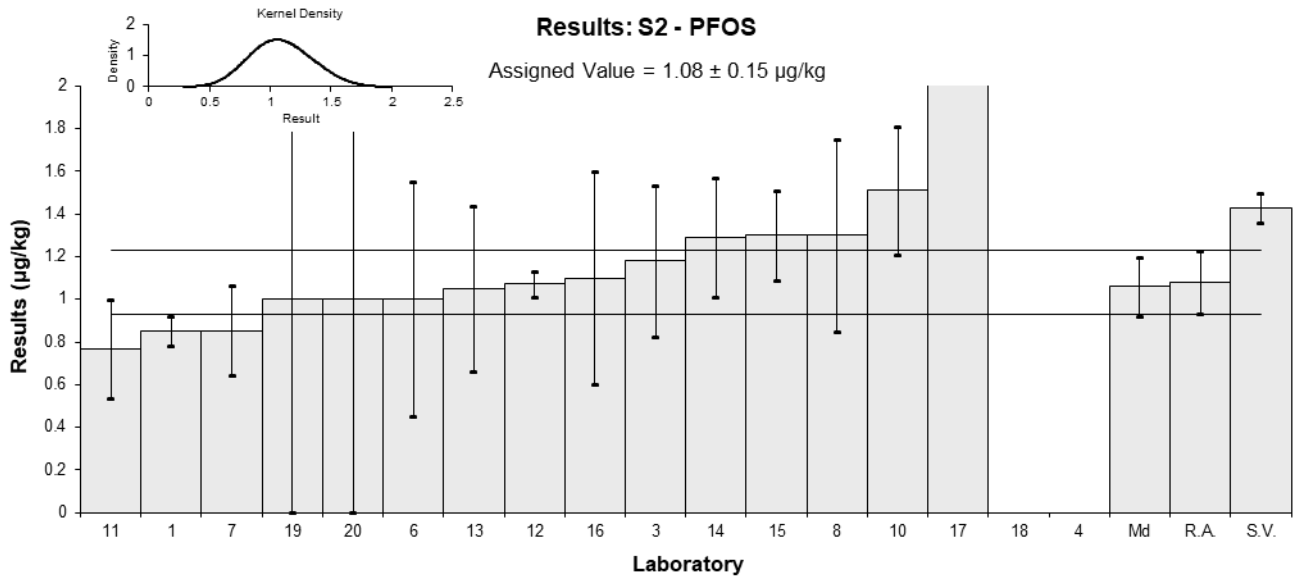
Participant Results

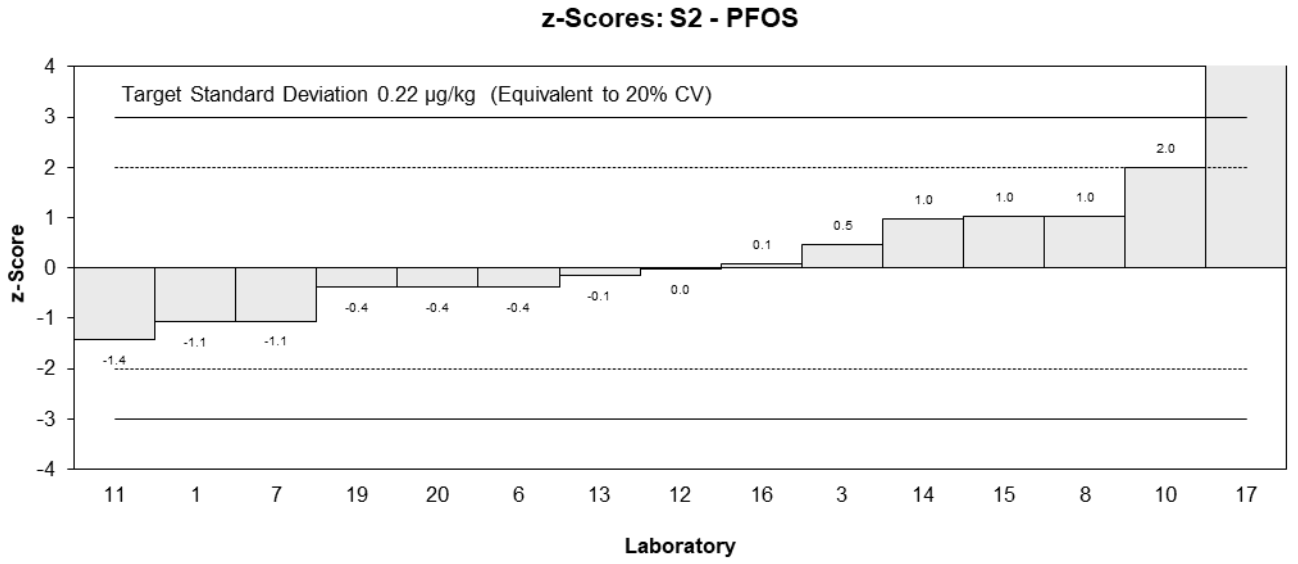
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.85	0.07	NR	-1.06	-1.39
3	1.18	0.354	104	0.46	0.26
4	NT	NT	NT		
6	1.0	0.55	91	-0.37	-0.14
7	0.852	0.21	94	-1.06	-0.88
8	1.3	0.45	101	1.02	0.46
10	1.51	0.3	NR	1.99	1.28
11	0.770	0.231	99	-1.44	-1.13
12	1.073	0.06	96	-0.03	-0.04
13	1.05	0.39	137	-0.14	-0.07
14	1.29	0.280	84.0	0.97	0.66
15	1.3	0.21	102	1.02	0.85
16	1.1	0.5	107	0.09	0.04
17	81.24	0.34	NR	371.11	215.71
18	NR	NR	NR		
19	1	1	83	-0.37	-0.08
20	1.0	1	90	-0.37	-0.08

Statistics*

Assigned Value	1.08	0.15
Spike	1.43	0.07
Robust Average	1.08	0.15
Median	1.06	0.14
Mean	1.09	
N	14	
Max.	1.51	
Min.	0.77	
Robust SD	0.22	
Robust CV	20%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S2 - PFOS
Assigned Value = 1.08 ± 0.15 µg/kg
Results (µg/kg)
11 1 7 19 20 6 13 12 16 3 14 15 8 10 17 18 4 Md R.A. S.V.
Laboratory


z-Scores: S2 - PFOS
Target Standard Deviation 0.22 µg/kg (Equivalent to 20% CV)
z-Score
-1.4 -1.1 -1.1 -0.4 -0.4 -0.4 -0.1 0.0 0.1 0.5 1.0 1.0 1.0 2.0
11 1 7 19 20 6 13 12 16 3 14 15 8 10 17
Laboratory

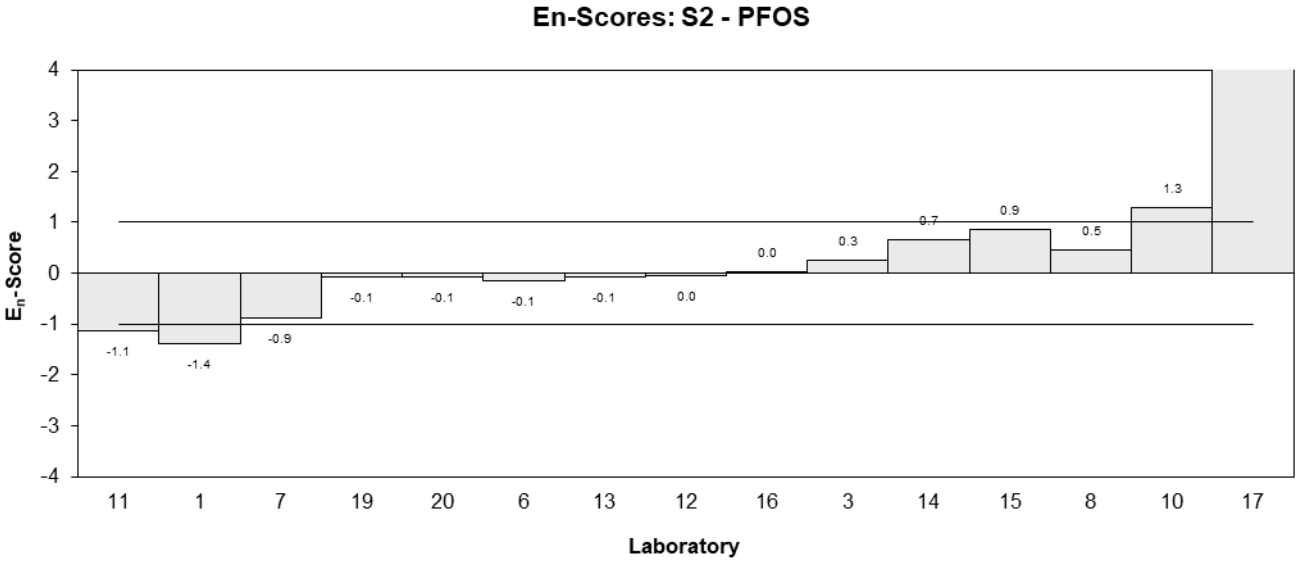

En-Scores: S2 - PFOS
En-Score
-1.1 -1.4 -0.9 -0.1 -0.1 -0.1 -0.1 0.0 0.0 0.3 0.7 0.9 0.5 1.3
11 1 7 19 20 6 13 12 16 3 14 15 8 10 17
Laboratory

Figure 24

Table 29

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFOS (linear)
Units	µg/kg

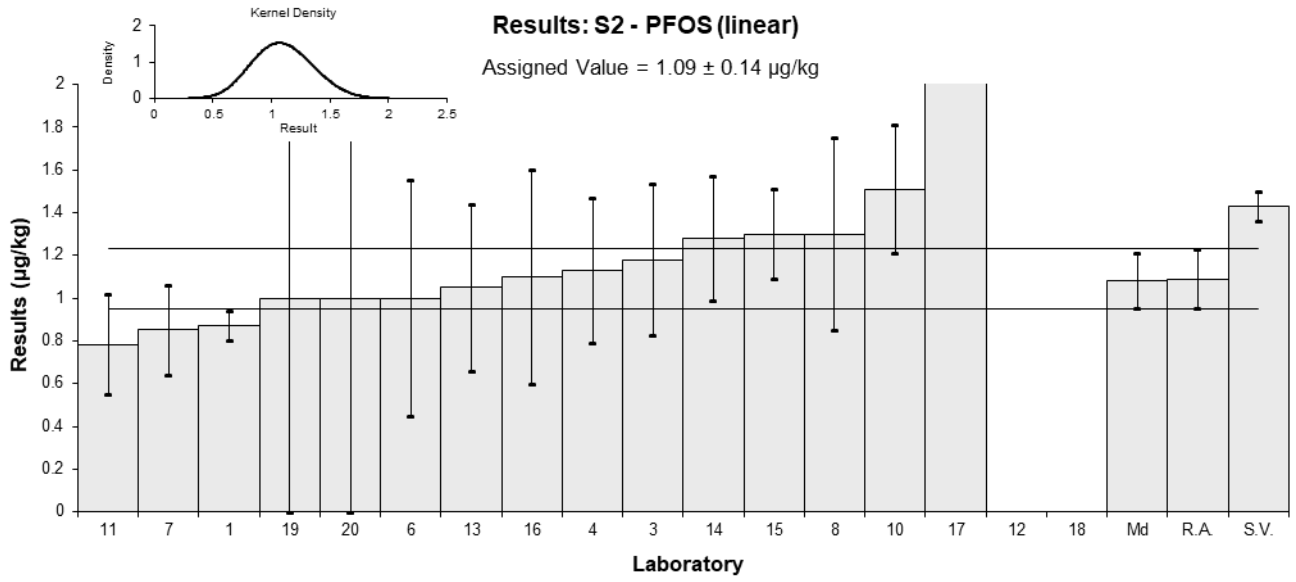
Participant Results

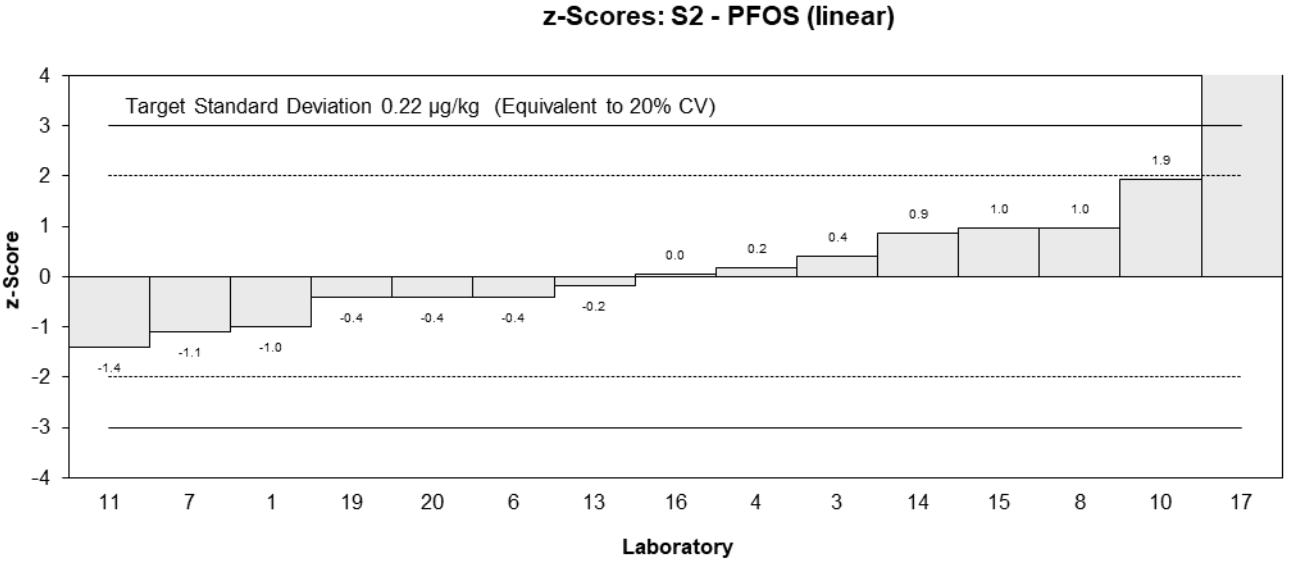
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.87	0.07	NR	-1.01	-1.41
3	1.18	0.354	104	0.41	0.24
4	1.13	0.34	113	0.18	0.11
6	1.0	0.55	NR	-0.41	-0.16
7	0.852	0.21	94	-1.09	-0.94
8	1.3	0.45	101	0.96	0.45
10	1.51	0.3	71	1.93	1.27
11	0.783	0.235	99	-1.41	-1.12
12	NR	NR	NR		
13	1.05	0.39	137	-0.18	-0.10
14	1.28	0.290	84.0	0.87	0.59
15	1.3	0.21	102	0.96	0.83
16	1.1	0.5	107	0.05	0.02
17	63.71	0.24	44	287.25	225.37
18	NR	NR	NR		
19	1	1	83	-0.41	-0.09
20	1.0	1	90	-0.41	-0.09

Statistics*

Assigned Value	1.09	0.14
Spike	1.43	0.07
Robust Average	1.09	0.14
Median	1.08	0.13
Mean	1.10	
N	14	
Max.	1.51	
Min.	0.783	
Robust SD	0.21	
Robust CV	20%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S2 - PFOS (linear)
Assigned Value = 1.09 ± 0.14 µg/kg
Results (µg/kg)
Laboratory
11 7 1 19 20 6 13 16 4 3 14 15 8 10 17 12 18 Md R.A. S.V.


z-Scores: S2 - PFOS (linear)
Target Standard Deviation 0.22 µg/kg (Equivalent to 20% CV)
z-Score
Laboratory
11 7 1 19 20 6 13 16 4 3 14 15 8 10 17
-1.4 -1.1 -1.0 -0.4 -0.4 -0.4 -0.2 0.0 0.2 0.4 0.9 1.0 1.0 1.9

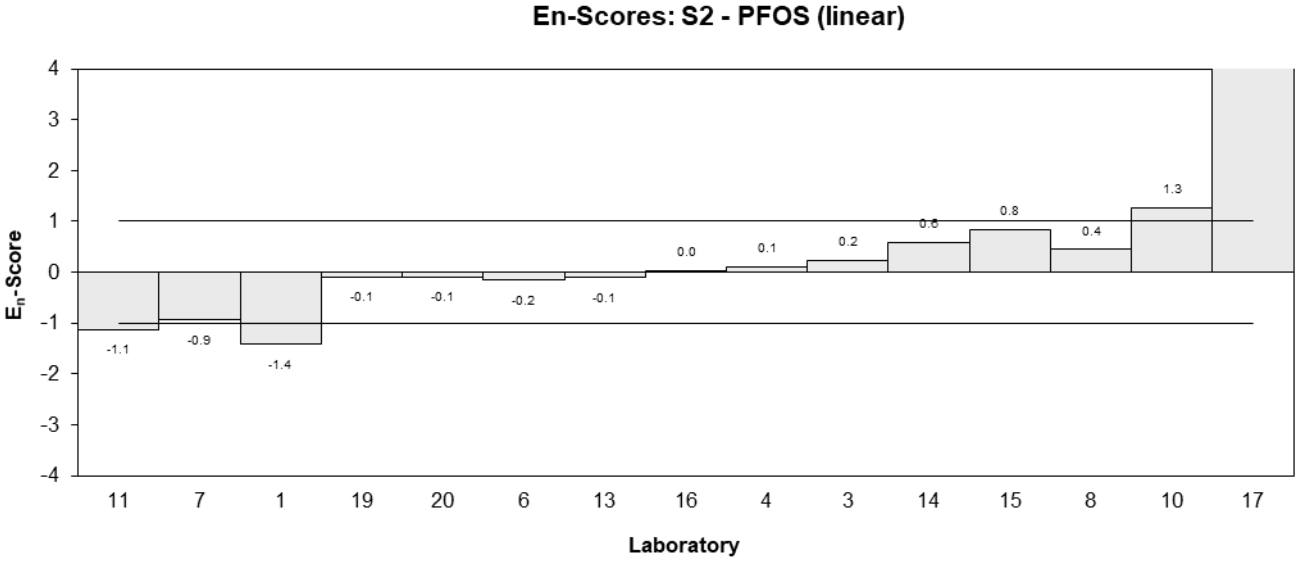

En-Scores: S2 - PFOS (linear)
En-Score
Laboratory
11 7 1 19 20 6 13 16 4 3 14 15 8 10 17
-1.1 -0.9 -1.4 -0.1 -0.1 -0.2 -0.1 0.0 0.1 0.2 0.6 0.8 0.4 1.3

Figure 25

Table 30

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFDS
Units	µg/kg

Participant Results

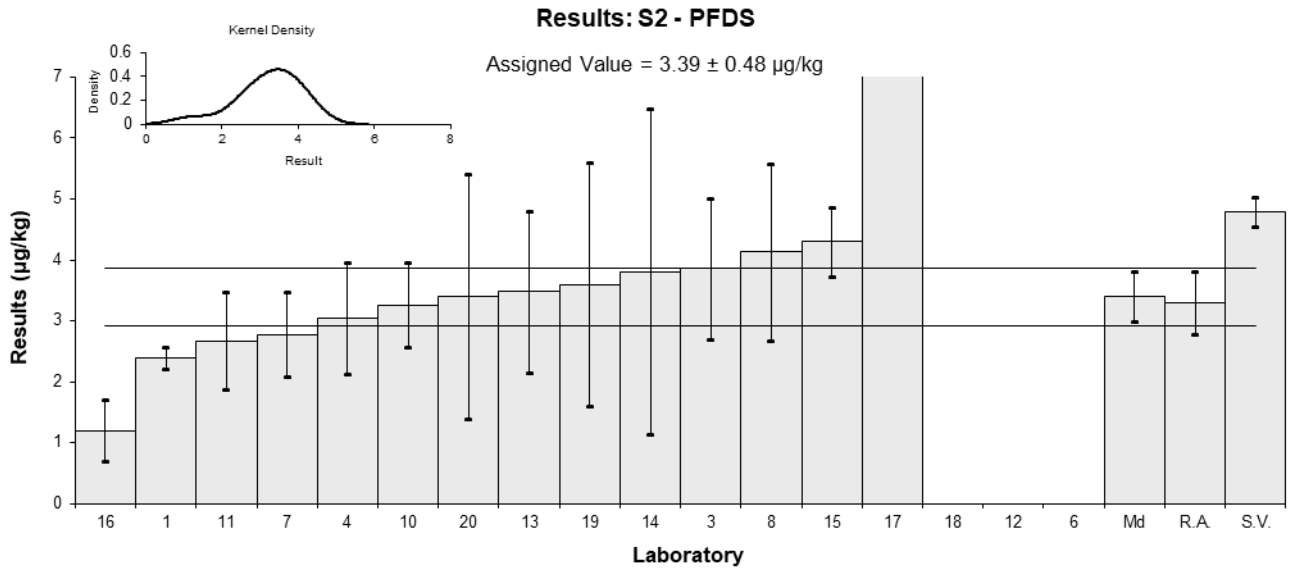
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	2.39	0.17	NR	-1.47	-1.96
3	3.86	1.16	100	0.69	0.37
4	3.04	0.91	NR	-0.52	-0.34
6	NT	NT	NT		
7	2.78	0.69	77	-0.90	-0.73
8	4.13	1.45	101	1.09	0.48
10	3.26	0.7	NR	-0.19	-0.15
11	2.666	0.8	99	-1.07	-0.78
12	NT	NT	NT		
13	3.48	1.33	128	0.13	0.06
14	3.81	2.67	84.0	0.62	0.15
15	4.3	0.57	102	1.34	1.22
16	1.2	0.5	107	-3.23	-3.16
17	16.79	0.46	NR	19.76	20.16
18	NR	NR	NR		
19	3.6	2	84	0.31	0.10
20	3.4	2	90	0.01	0.00

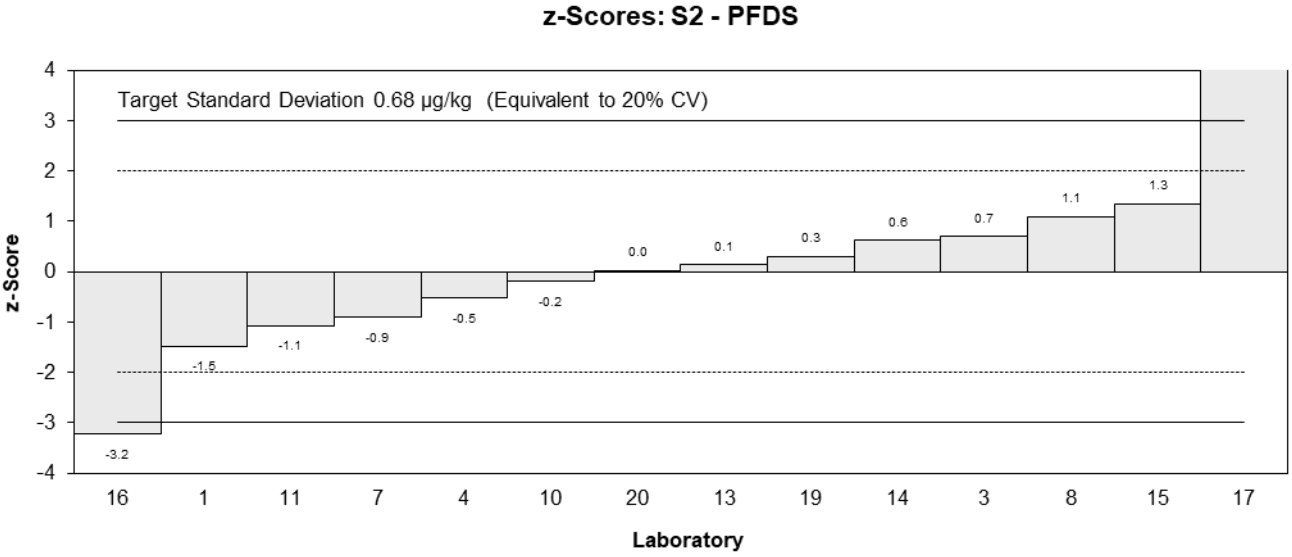
Statistics*

Assigned Value**	3.39	0.48
Spike	4.79	0.24
Robust Average	3.30	0.52
Median	3.40	0.41
Mean	3.22	
N	13	
Max.	4.3	
Min.	1.2	
Robust SD	0.75	
Robust CV	23%	

* Laboratory 17 was omitted from all statistical calculations (gross error).

** Robust average excluding Laboratory 16.


Results: S2 - PFDS
Kernel Density
Density
Result
Assigned Value = 3.39 ± 0.48 µg/kg
Results (µg/kg)
16 1 11 7 4 10 20 13 19 14 3 8 15 17 18 12 6 Md R.A. S.V.
Laboratory


z-Scores: S2 - PFDS
Target Standard Deviation 0.68 µg/kg (Equivalent to 20% CV)
z-Score
-3.2 -1.5 -1.1 -0.9 -0.5 -0.2 0.0 0.1 0.3 0.6 0.7 1.1 1.3
16 1 11 7 4 10 20 13 19 14 3 8 15 17
Laboratory

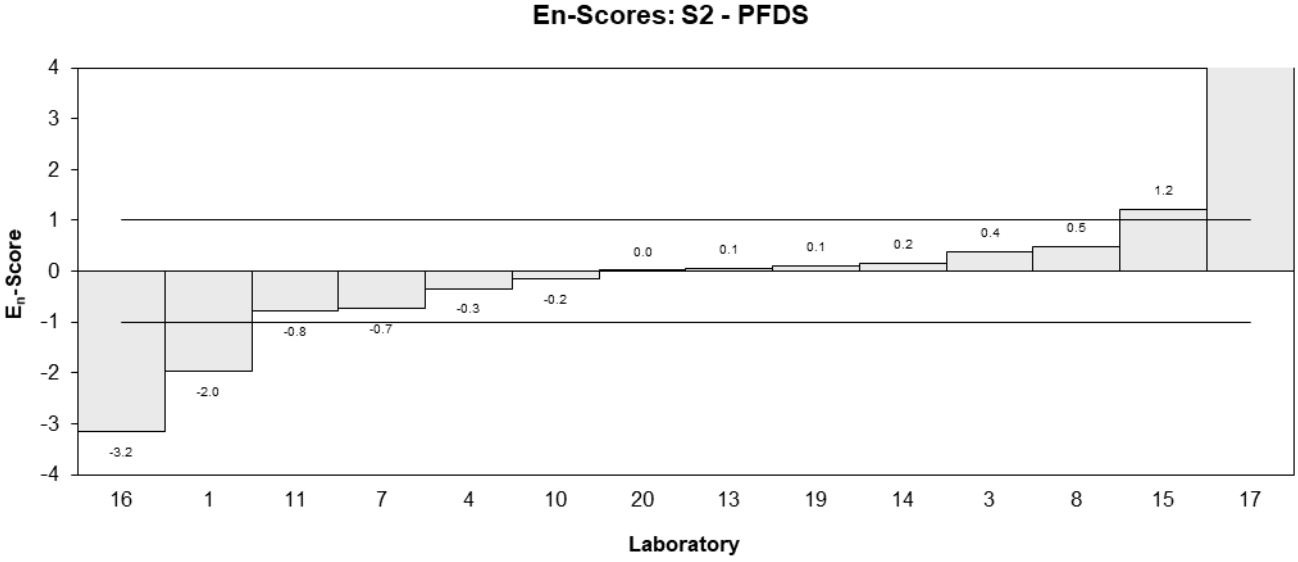

En-Scores: S2 - PFDS
En-Score
-3.2 -2.0 -0.8 -0.7 -0.3 -0.2 0.0 0.1 0.1 0.2 0.4 0.5 1.2
16 1 11 7 4 10 20 13 19 14 3 8 15 17
Laboratory

Figure 26

Table 31

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFBA
Units	µg/kg

Participant Results

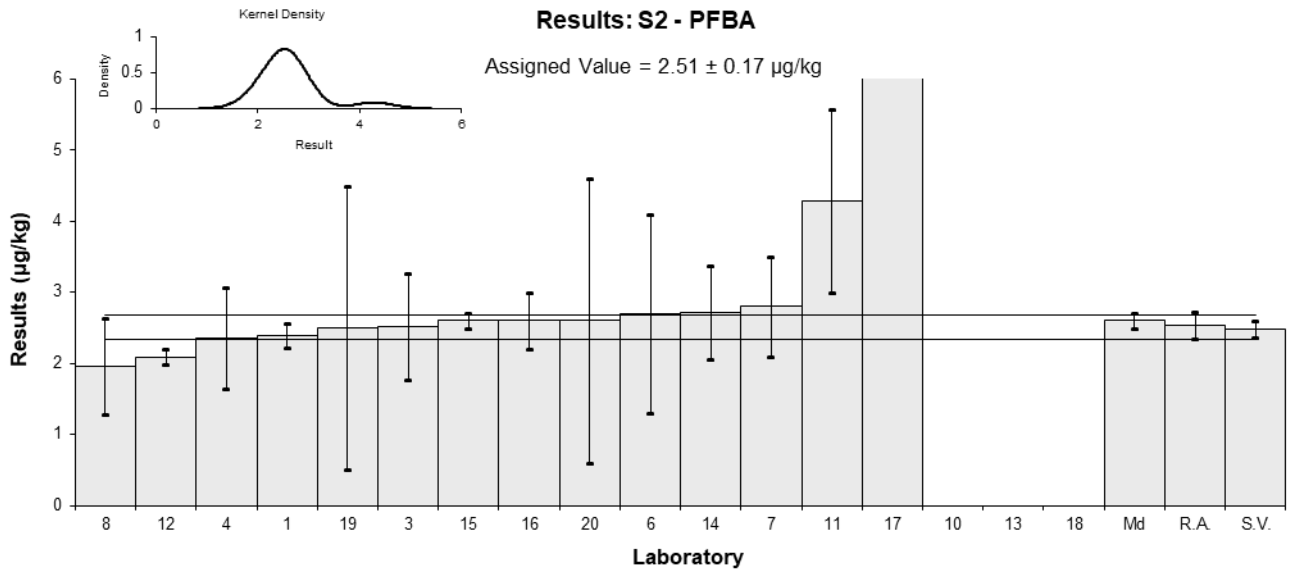
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	2.39	0.17	NR	-0.24	-0.50
3	2.52	0.756	53.9	0.02	0.01
4	2.36	0.71	92	-0.30	-0.21
6	2.7	1.4	95	0.38	0.13
7	2.8	0.7	81	0.58	0.40
8	1.96	0.68	47	-1.10	-0.78
10	<5	1	75		
11	4.283	1.285	54	3.53	1.37
12	2.095	0.103	72	-0.83	-2.09
13	<5	NR	91		
14	2.72	0.662	63.2	0.42	0.31
15	2.6	0.11	110	0.18	0.44
16	2.6	0.4	86	0.18	0.21
17	40.55	1.37	59	75.78	27.56
18	NR	NR	NR		
19	2.5	2	91	-0.02	0.00
20	2.6	2	80	0.18	0.04

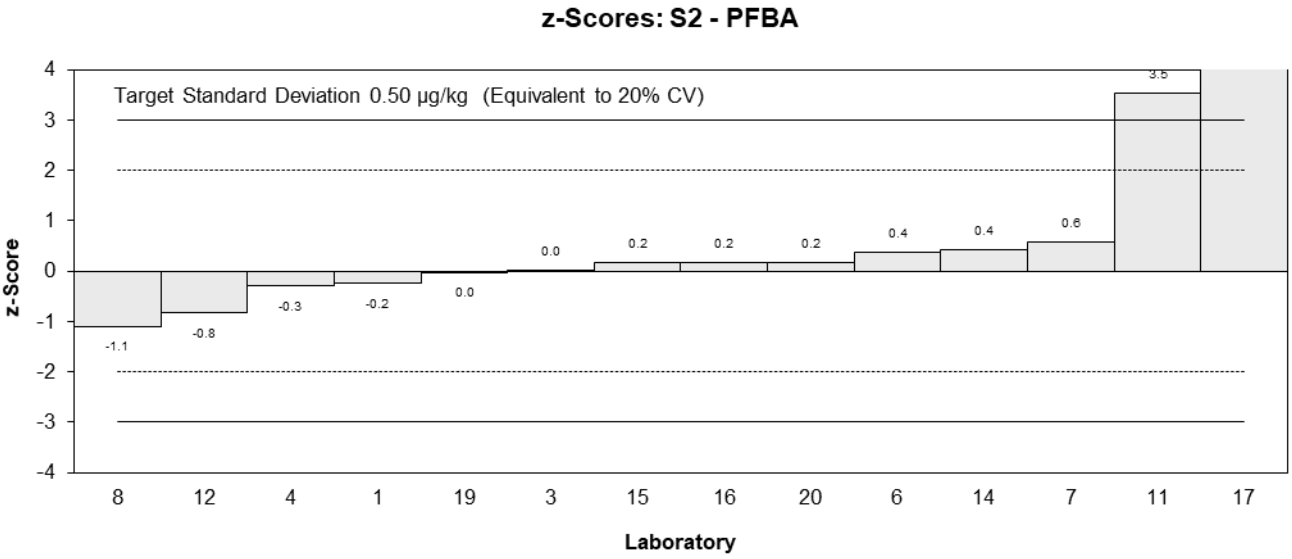
Statistics*

Assigned Value**	2.51	0.17
Spike	2.48	0.12
Robust Average	2.54	0.19
Median	2.60	0.11
Mean	2.63	
N	13	
Max.	4.283	
Min.	1.96	
Robust SD	0.27	
Robust CV	11%	

* Laboratory 17 was omitted from all statistical calculations (gross error).

** Robust average excluding Laboratory 11.


Results: S2 - PFBA
Kernel Density
Density
Result
Assigned Value = 2.51 ± 0.17 µg/kg
Results (µg/kg)
Laboratory
8 12 4 1 19 3 15 16 20 6 14 7 11 17 10 13 18 Md R.A. S.V.


z-Scores: S2 - PFBA
Target Standard Deviation 0.50 µg/kg (Equivalent to 20% CV)
z-Score
Laboratory
8 12 4 1 19 3 15 16 20 6 14 7 11 17
-1.1 -0.8 -0.3 -0.2 0.0 0.0 0.2 0.2 0.2 0.4 0.4 0.6 3.5

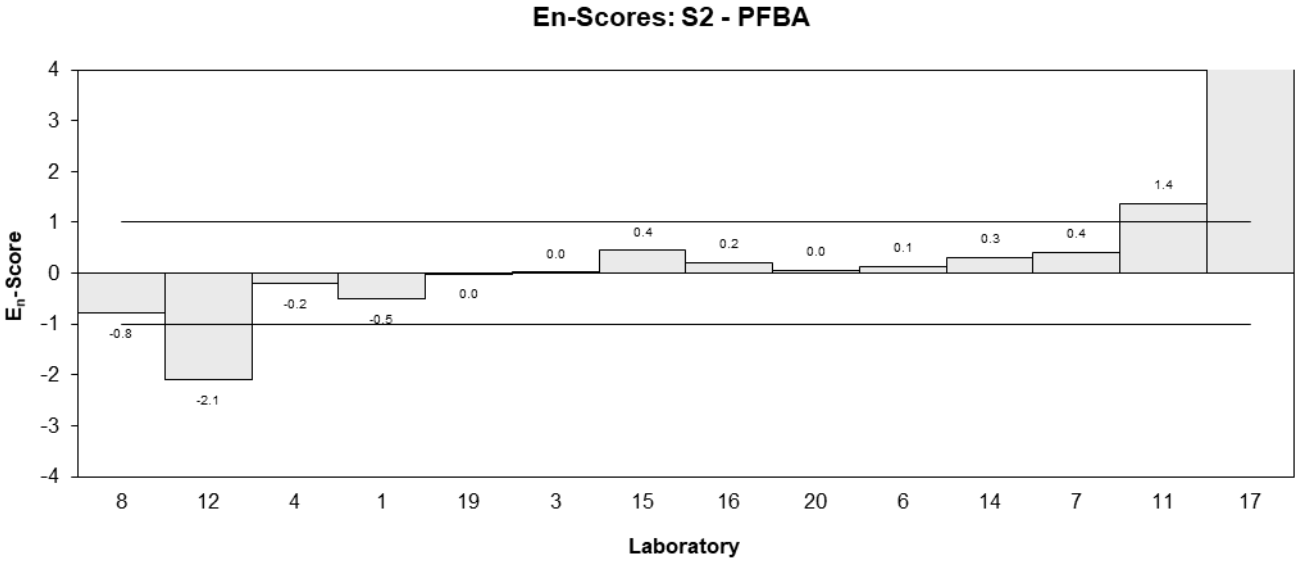

En-Scores: S2 - PFBA
En-Score
Laboratory
8 12 4 1 19 3 15 16 20 6 14 7 11 17
-0.8 -2.1 -0.2 -0.5 0.0 0.0 0.4 0.2 0.0 0.1 0.3 0.4 1.4

Figure 27

Table 32

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFPeA
Units	µg/kg

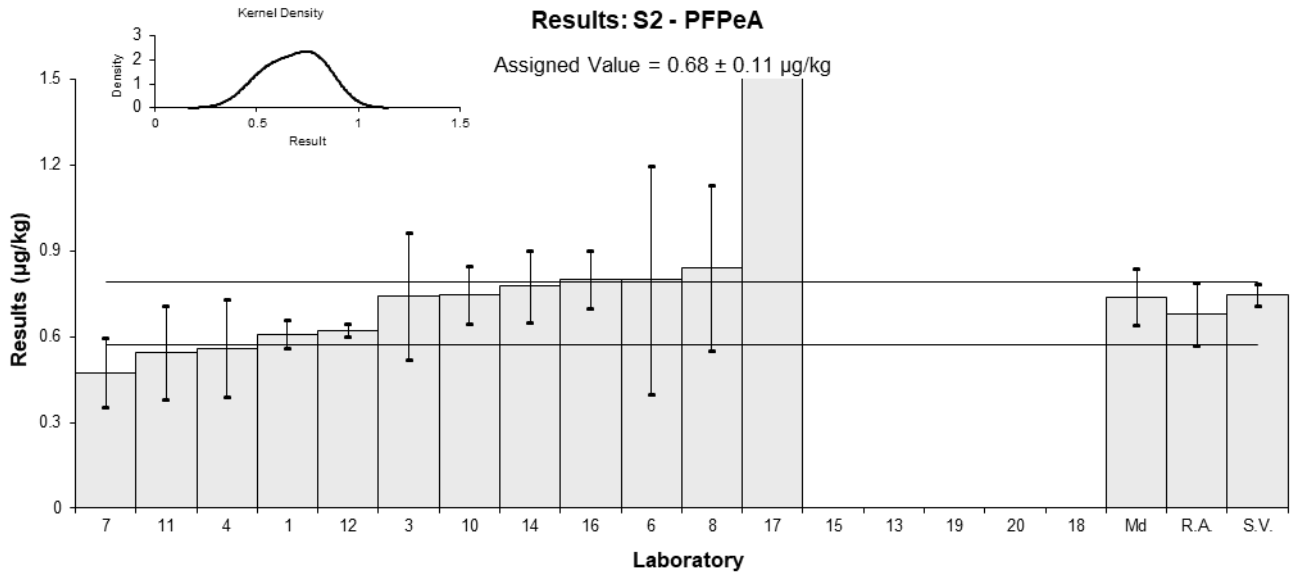
Participant Results

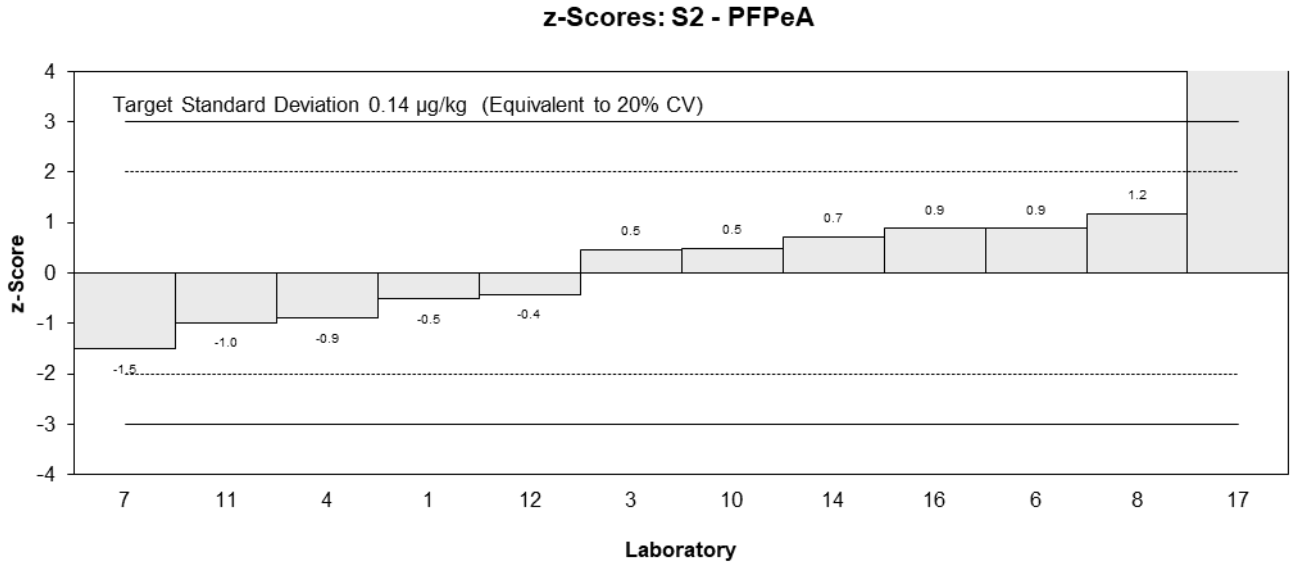
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.61	0.05	NR	-0.51	-0.58
3	0.743	0.223	64.1	0.46	0.25
4	0.56	0.17	99	-0.88	-0.59
6	0.80	0.40	93	0.88	0.29
7	0.475	0.12	80	-1.51	-1.26
8	0.84	0.29	42	1.18	0.52
10	0.746	0.1	94	0.49	0.44
11	0.547	0.164	76	-0.98	-0.67
12	0.623	0.022	77	-0.42	-0.51
13	<2	NR	133		
14	0.777	0.127	56.9	0.71	0.58
15	< 1.0	NR	104		
16	0.8	0.1	87	0.88	0.81
17	17.15	0.14	63	121.10	92.50
18	NR	NR	NR		
19	<2	NR	92		
20	<2	NR	91		

Statistics*

Assigned Value	0.68	0.11
Spike	0.746	0.037
Robust Average	0.68	0.11
Median	0.740	0.097
Mean	0.684	
N	11	
Max.	0.84	
Min.	0.475	
Robust SD	0.14	
Robust CV	21%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S2 - PFPeA
Assigned Value = 0.68 ± 0.11 µg/kg
Results (µg/kg)
Laboratory
7 11 4 1 12 3 10 14 16 6 8 17 15 13 19 20 18 Md R.A. S.V.


z-Scores: S2 - PFPeA
Target Standard Deviation 0.14 µg/kg (Equivalent to 20% CV)
z-Score
-1.5 -1.0 -0.9 -0.5 -0.4 0.5 0.5 0.7 0.9 0.9 1.2
Laboratory
7 11 4 1 12 3 10 14 16 6 8 17

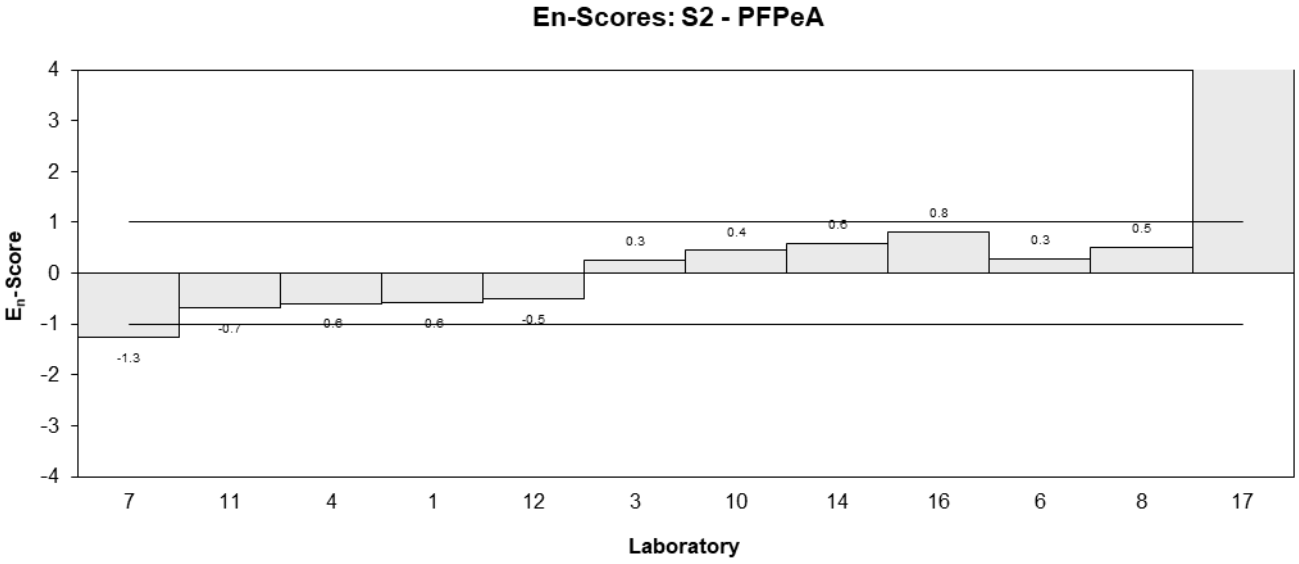

En-Scores: S2 - PFPeA
En-Score
-1.3 -0.7 -0.6 -0.6 -0.5 0.3 0.4 0.6 0.8 0.3 0.5
Laboratory
7 11 4 1 12 3 10 14 16 6 8 17

Figure 28

Table 33

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFHxA
Units	µg/kg

Participant Results

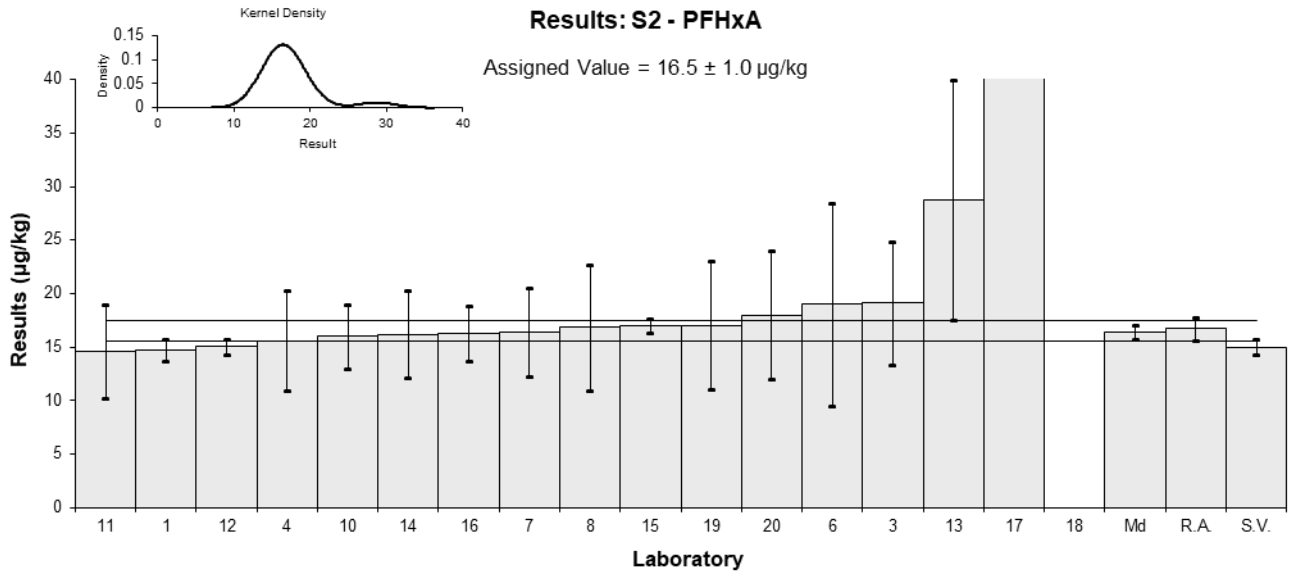
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	14.74	1.05	NR	-0.53	-1.21
3	19.1	5.73	73.1	0.79	0.45
4	15.58	4.67	109	-0.28	-0.19
6	19	9.5	101	0.76	0.26
7	16.4	4.1	84	-0.03	-0.02
8	16.83	5.89	45	0.10	0.06
10	16	3	91	-0.15	-0.16
11	14.632	4.390	84	-0.57	-0.41
12	15.033	0.723	80	-0.44	-1.19
13	28.74	11.21	122	3.71	1.09
14	16.2	4.11	81.0	-0.09	-0.07
15	17	0.68	129	0.15	0.41
16	16.3	2.6	92	-0.06	-0.07
17	346.53	0.16	51	100.01	325.89
18	NR	NR	NR		
19	17	6	89	0.15	0.08
20	18	6	101	0.45	0.25

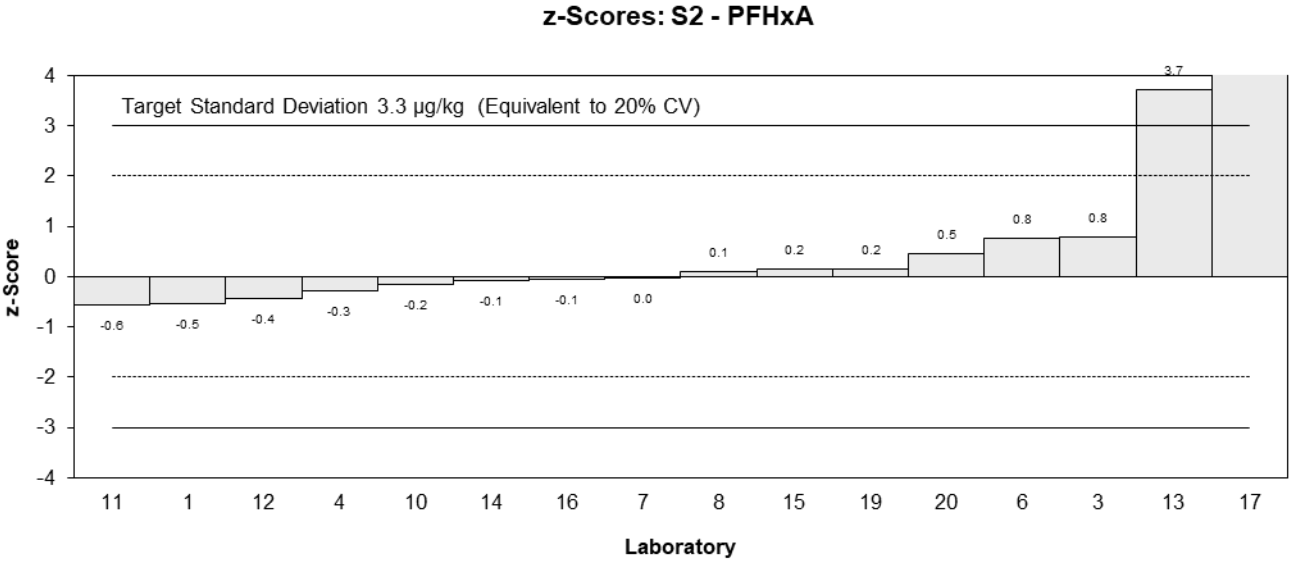
Statistics*

Assigned Value**	16.5	1.0
Spike	15.0	0.7
Robust Average	16.7	1.1
Median	16.4	0.7
Mean	17.4	
N	15	
Max.	28.74	
Min.	14.632	
Robust SD	1.7	
Robust CV	10%	

* Laboratory 17 was omitted from all statistical calculations (gross error).

** Robust average excluding Laboratory 13.


Kernel Density
Density
Result
Results: S2 - PFHxA
Assigned Value = 16.5 ± 1.0 µg/kg
Results (µg/kg)
Laboratory
11 1 12 4 10 14 16 7 8 15 19 20 6 3 13 17 18 Md R.A. S.V.


z-Scores: S2 - PFHxA
Target Standard Deviation 3.3 µg/kg (Equivalent to 20% CV)
z-Score
Laboratory
11 1 12 4 10 14 16 7 8 15 19 20 6 3 13 17
-0.6 -0.5 -0.4 -0.3 -0.2 -0.1 -0.1 0.0 0.1 0.2 0.2 0.5 0.8 0.8 3.7

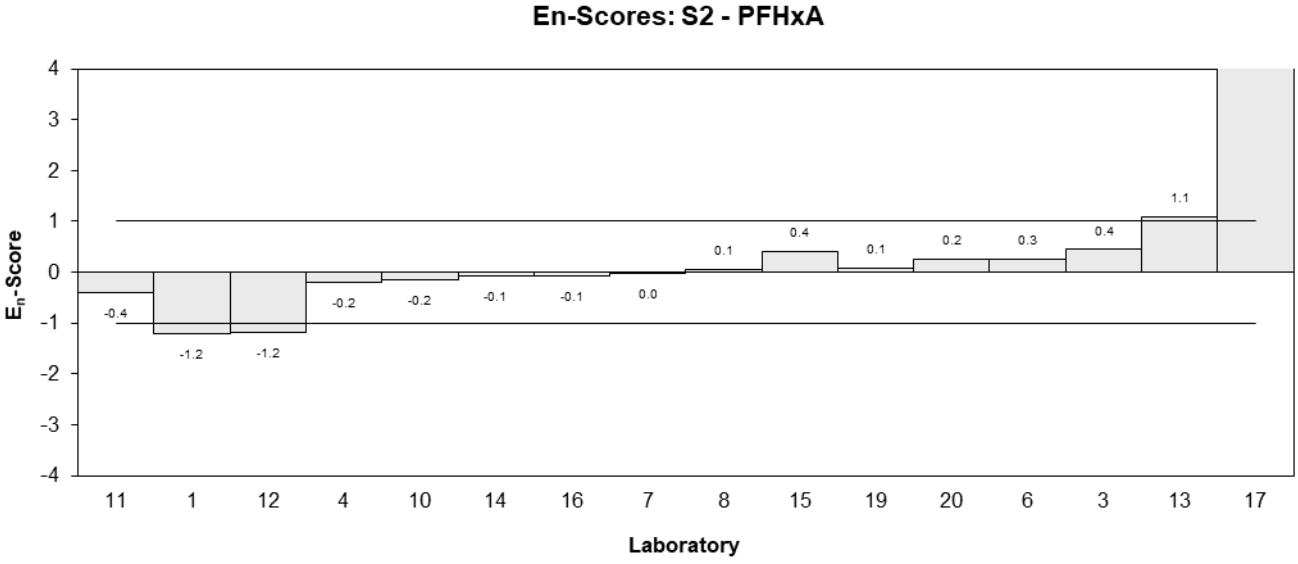

En-Scores: S2 - PFHxA
En-Score
Laboratory
11 1 12 4 10 14 16 7 8 15 19 20 6 3 13 17
-0.4 -1.2 -1.2 -0.2 -0.2 -0.1 -0.1 0.0 0.1 0.4 0.1 0.2 0.3 0.4 1.1

Figure 29

Table 34

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFHpA
Units	µg/kg

Participant Results

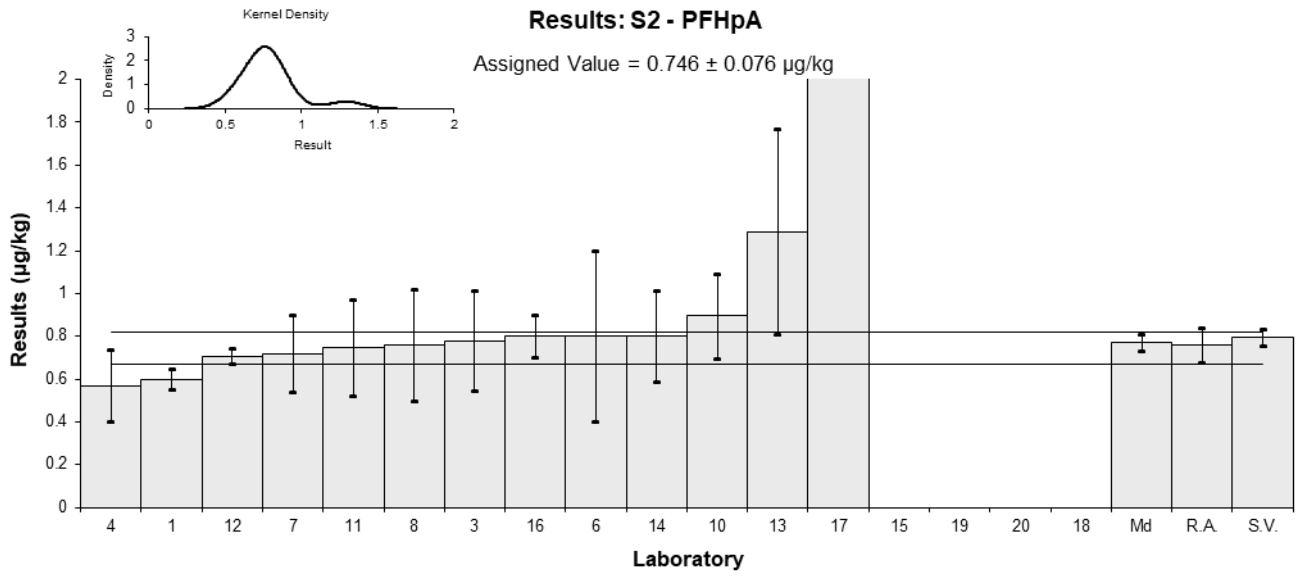
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.6	0.05	NR	-0.98	-1.60
3	0.779	0.234	88.1	0.22	0.13
4	0.57	0.17	123	-1.18	-0.95
6	0.80	0.40	89	0.36	0.13
7	0.719	0.18	92	-0.18	-0.14
8	0.76	0.26	65	0.09	0.05
10	0.895	0.2	88	1.00	0.70
11	0.750	0.225	90	0.03	0.02
12	0.708	0.036	80	-0.25	-0.45
13	1.29	0.48	125	3.65	1.12
14	0.801	0.211	87.8	0.37	0.25
15	< 1.0	NR	101		
16	0.8	0.1	87	0.36	0.43
17	12.87	0.3	45	81.26	39.18
18	NR	NR	NR		
19	<1	NR	84		
20	<1	NR	93		

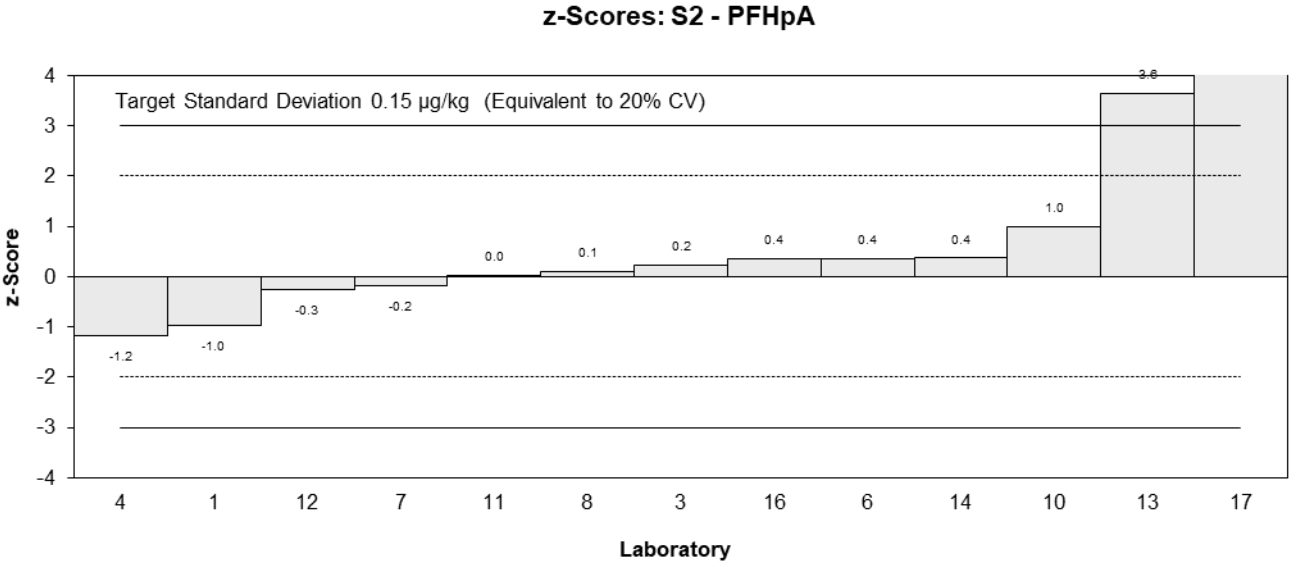
Statistics*

Assigned Value**	0.746	0.076
Spike	0.795	0.040
Robust Average	0.760	0.083
Median	0.770	0.039
Mean	0.789	
N	12	
Max.	1.29	
Min.	0.57	
Robust SD	0.11	
Robust CV	15%	

* Laboratory 17 was omitted from all statistical calculations (gross error).

** Robust average excluding Laboratory 13.


Kernel Density
Density
Result
Results: S2 - PFHpA
Assigned Value = 0.746 ± 0.076 µg/kg
Results (µg/kg)
Laboratory
4 1 12 7 11 8 3 16 6 14 10 13 17 15 19 20 18 Md R.A. S.V.


z-Scores: S2 - PFHpA
Target Standard Deviation 0.15 µg/kg (Equivalent to 20% CV)
z-Score
-1.2 -1.0 -0.3 -0.2 0.0 0.1 0.2 0.4 0.4 0.4 1.0 3.6
Laboratory
4 1 12 7 11 8 3 16 6 14 10 13 17

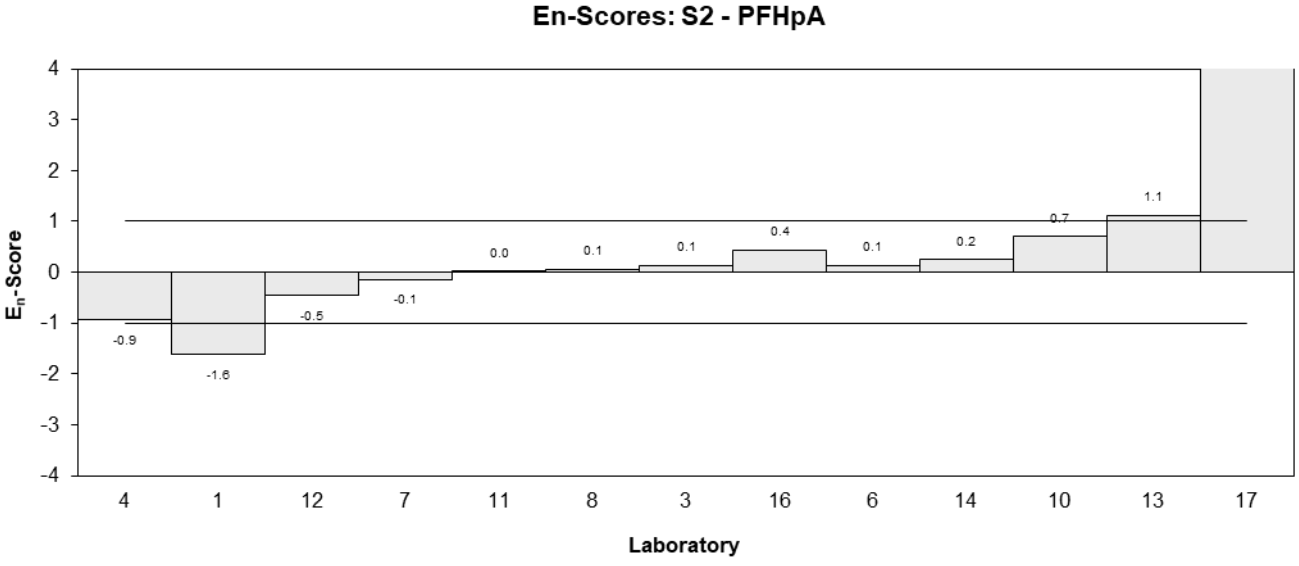

En-Scores: S2 - PFHpA
En-Score
-0.9 -1.6 -0.5 -0.1 0.0 0.1 0.1 0.4 0.1 0.2 0.7 1.1
Laboratory
4 1 12 7 11 8 3 16 6 14 10 13 17

Figure 30

Table 35

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFOA
Units	µg/kg

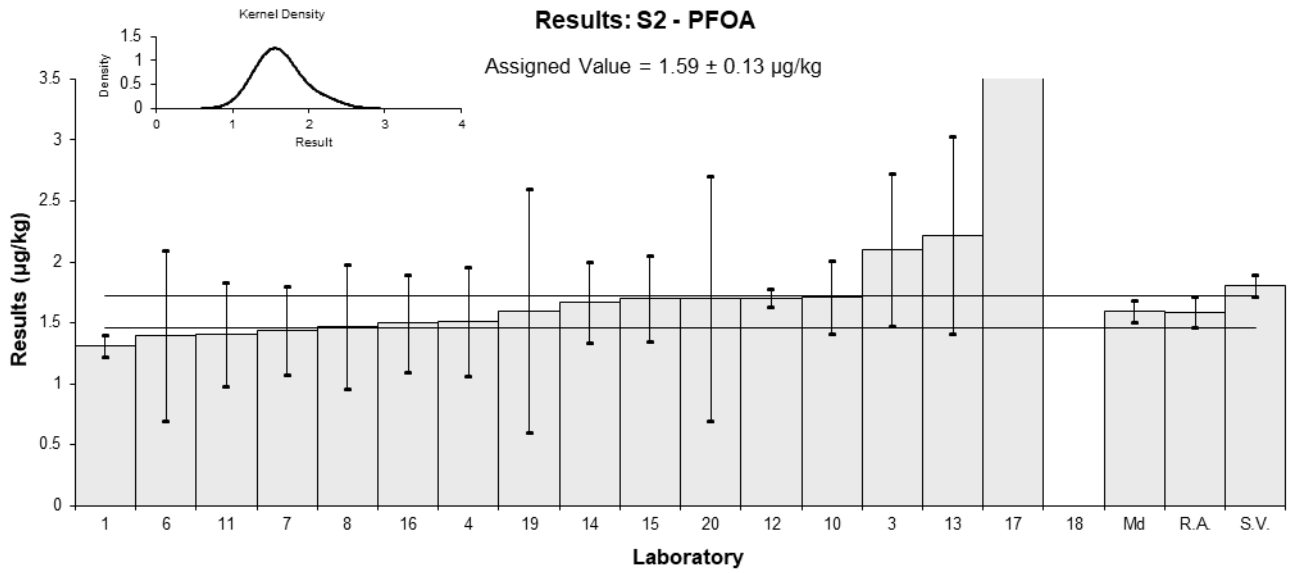
Participant Results

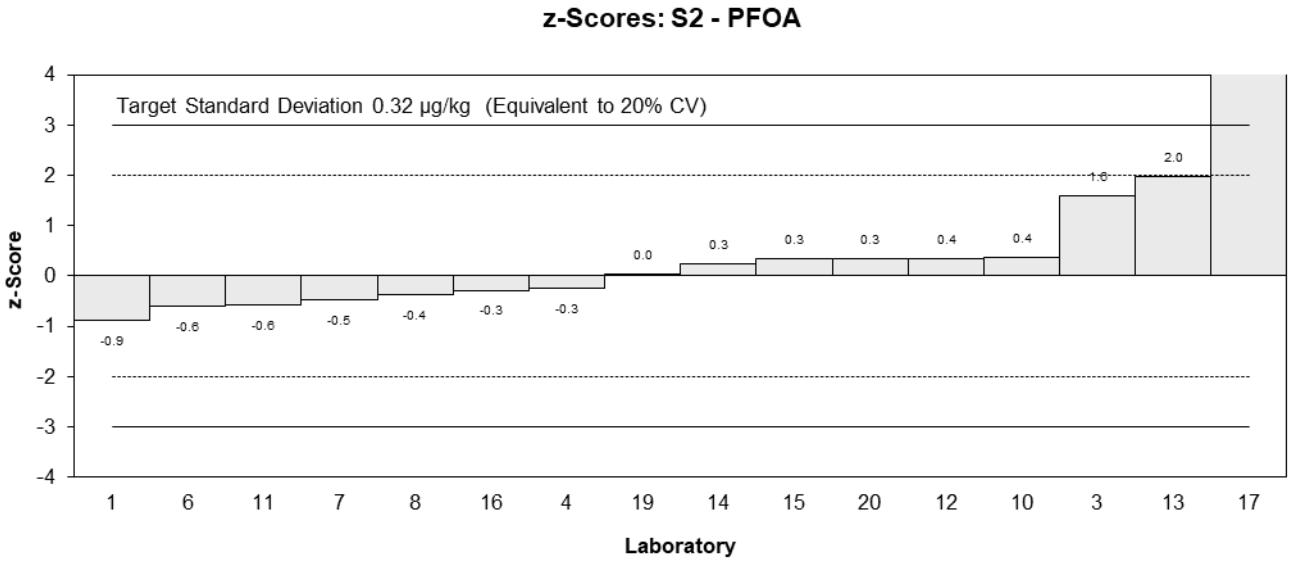
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	1.31	0.09	NR	-0.88	-1.77
3	2.1	0.63	104	1.60	0.79
4	1.51	0.45	129	-0.25	-0.17
6	1.4	0.70	80	-0.60	-0.27
7	1.44	0.36	91	-0.47	-0.39
8	1.47	0.51	83	-0.38	-0.23
10	1.71	0.3	88	0.38	0.37
11	1.406	0.422	92	-0.58	-0.42
12	1.703	0.072	83	0.36	0.76
13	2.22	0.81	156	1.98	0.77
14	1.67	0.331	81.9	0.25	0.22
15	1.7	0.35	99	0.35	0.29
16	1.5	0.4	89	-0.28	-0.21
17	24.38	0.26	44	71.67	78.40
18	NR	NR	NR		
19	1.6	1	86	0.03	0.01
20	1.7	1	91	0.35	0.11

Statistics*

Assigned Value	1.59	0.13
Spike	1.81	0.09
Robust Average	1.59	0.13
Median	1.60	0.09
Mean	1.63	
N	15	
Max.	2.22	
Min.	1.31	
Robust SD	0.20	
Robust CV	13%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Results: S2 - PFOA
Kernel Density
Density
Result
Assigned Value = 1.59 ± 0.13 µg/kg
Results (µg/kg)
1 6 11 7 8 16 4 19 14 15 20 12 10 3 13 17 18 Md R.A. S.V.
Laboratory


z-Scores: S2 - PFOA
Target Standard Deviation 0.32 µg/kg (Equivalent to 20% CV)
z-Score
-0.9 -0.6 -0.6 -0.5 -0.4 -0.3 -0.3 0.0 0.3 0.3 0.3 0.4 0.4 1.6 2.0
1 6 11 7 8 16 4 19 14 15 20 12 10 3 13 17
Laboratory

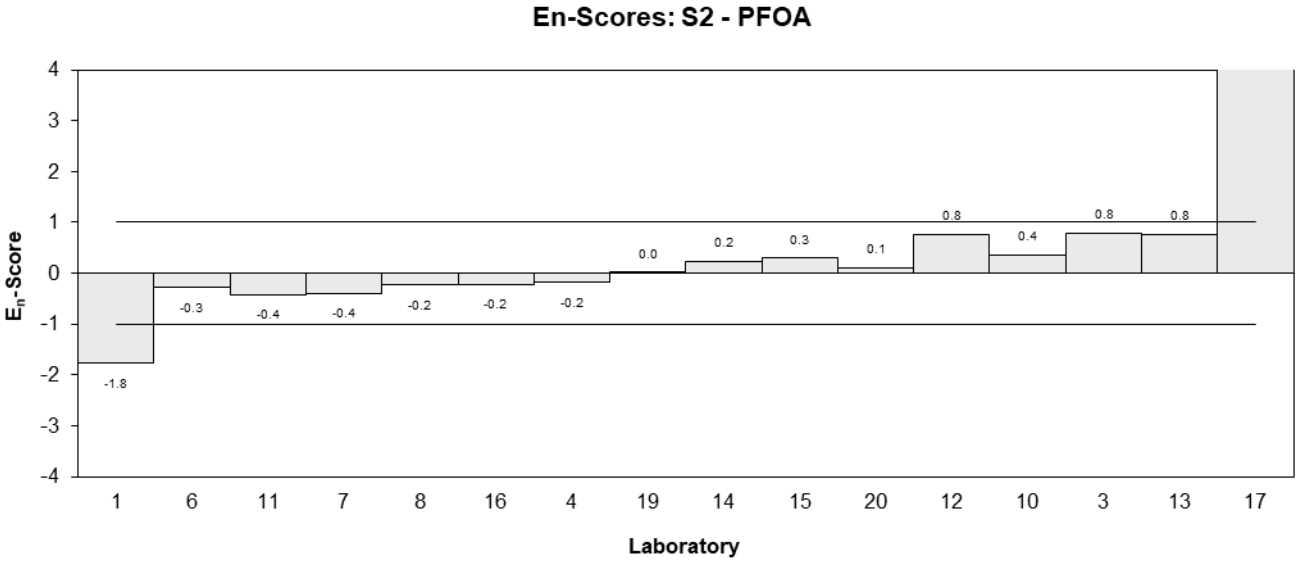

En-Scores: S2 - PFOA
En-Score
-1.8 -0.3 -0.4 -0.4 -0.2 -0.2 -0.2 0.0 0.2 0.3 0.1 0.8 0.4 0.8 0.8
1 6 11 7 8 16 4 19 14 15 20 12 10 3 13 17
Laboratory

Figure 31

Table 36

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFNA
Units	µg/kg

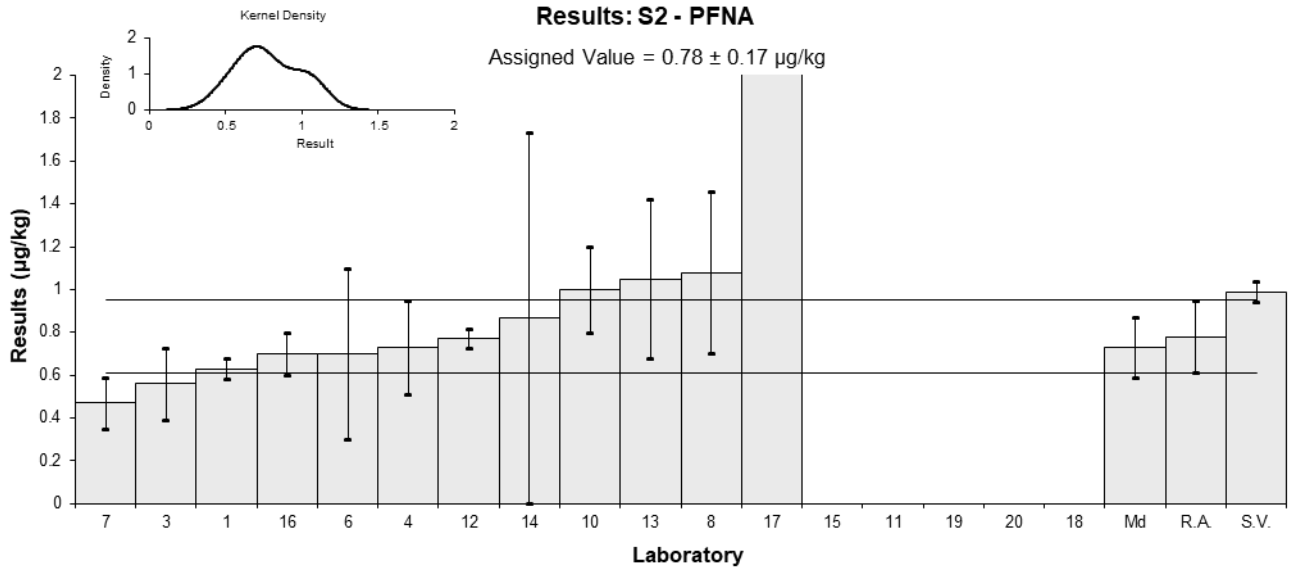
Participant Results

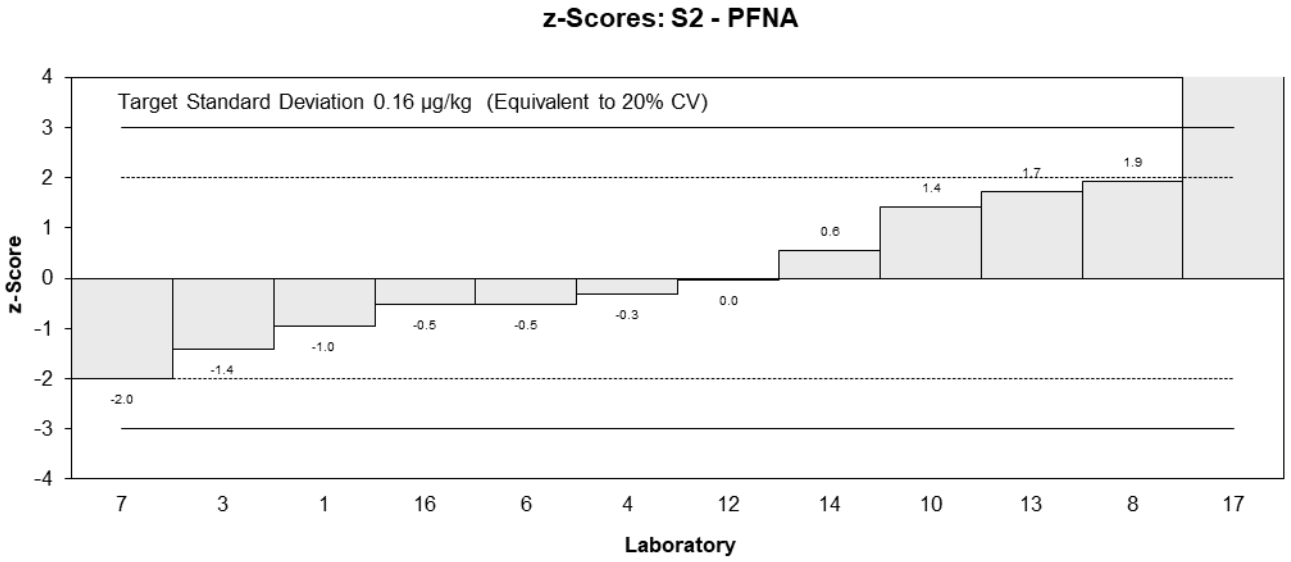
Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	0.63	0.05	NR	-0.96	-0.85
3	0.561	0.168	134	-1.40	-0.92
4	0.73	0.22	114	-0.32	-0.18
6	0.70	0.40	91	-0.51	-0.18
7	0.469	0.12	81	-1.99	-1.49
8	1.08	0.38	80	1.92	0.72
10	1	0.2	78	1.41	0.84
11	<0.2	NR	NR		
12	0.773	0.045	83	-0.04	-0.04
13	1.05	0.37	145	1.73	0.66
14	0.867	0.867	81.5	0.56	0.10
15	< 1.0	NR	102		
16	0.7	0.1	65	-0.51	-0.41
17	14.19	0.3	36	85.96	38.89
18	NR	NR	NR		
19	<1	NR	87		
20	<1	NR	88		

Statistics*

Assigned Value	0.78	0.17
Spike	0.990	0.050
Robust Average	0.78	0.17
Median	0.73	0.14
Mean	0.78	
N	11	
Max.	1.08	
Min.	0.469	
Robust SD	0.23	
Robust CV	29%	

* Laboratory 17 was omitted from all statistical calculations (gross error).


Results: S2 - PFNA
Assigned Value = 0.78 ± 0.17 µg/kg
Kernel Density
Density
Result
Results (µg/kg)
Laboratory
7 3 1 16 6 4 12 14 10 13 8 17 15 11 19 20 18 Md R.A. S.V.


z-Scores: S2 - PFNA
Target Standard Deviation 0.16 µg/kg (Equivalent to 20% CV)
z-Score
-2.0 -1.4 -1.0 -0.5 -0.5 -0.3 0.0 0.6 1.4 1.7 1.9
Laboratory
7 3 1 16 6 4 12 14 10 13 8 17

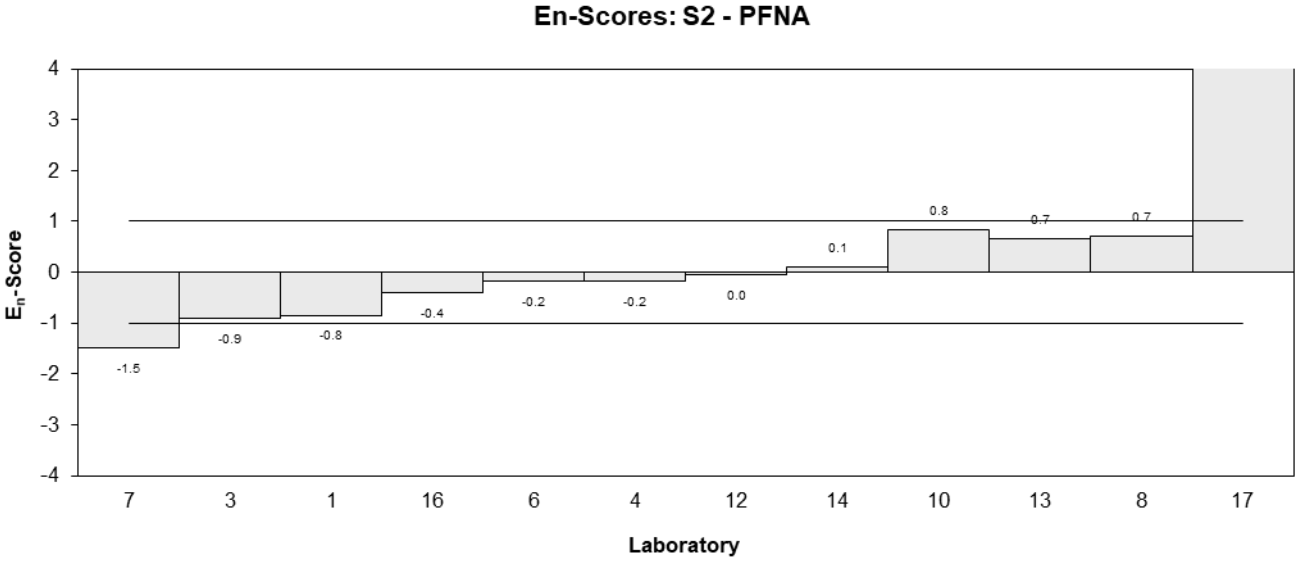

En-Scores: S2 - PFNA
En-Score
-1.5 -0.9 -0.8 -0.4 -0.2 -0.2 0.0 0.1 0.8 0.7 0.7
Laboratory
7 3 1 16 6 4 12 14 10 13 8 17

Figure 32

Table 37

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	PFOSA
Units	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	1.07	0.08	NR	-2.76	-2.38
3	3.24	0.972	38.1	1.78	0.76
4	2.50	0.75	100	0.23	0.12
6	2.9	1.4	54	1.07	0.34
7	1.83	0.46	92	-1.17	-0.78
8	NT	NT	NT		
10* **	3.79	0.8	60	2.00	1.00
11	1.644	0.493	69	-1.56	-1.01
12	NT	NT	NT		
13	<5	NR	121		
14	2.21	0.352	25.0	-0.38	-0.28
15	2.7	0.63	111	0.65	0.37
16	2.1	0.7	17	-0.61	-0.33
17	11.21	2.3	87	18.45	3.73
18	NR	NR	NR		
19	<5	NR	79		
20	<5	NR	87		

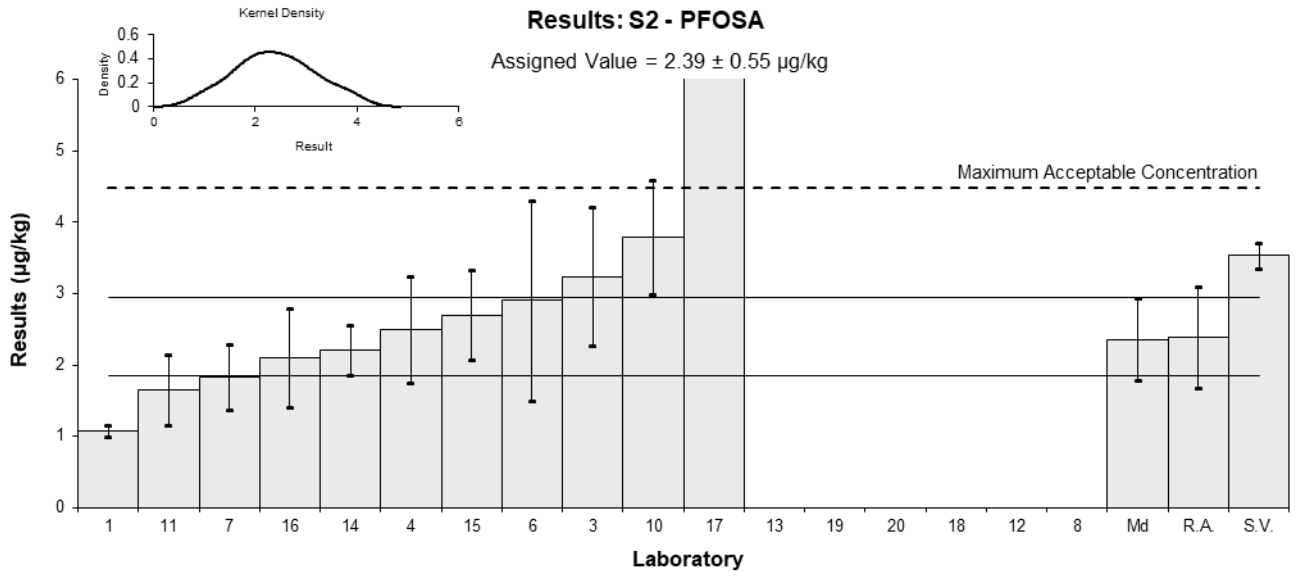
Statistics*

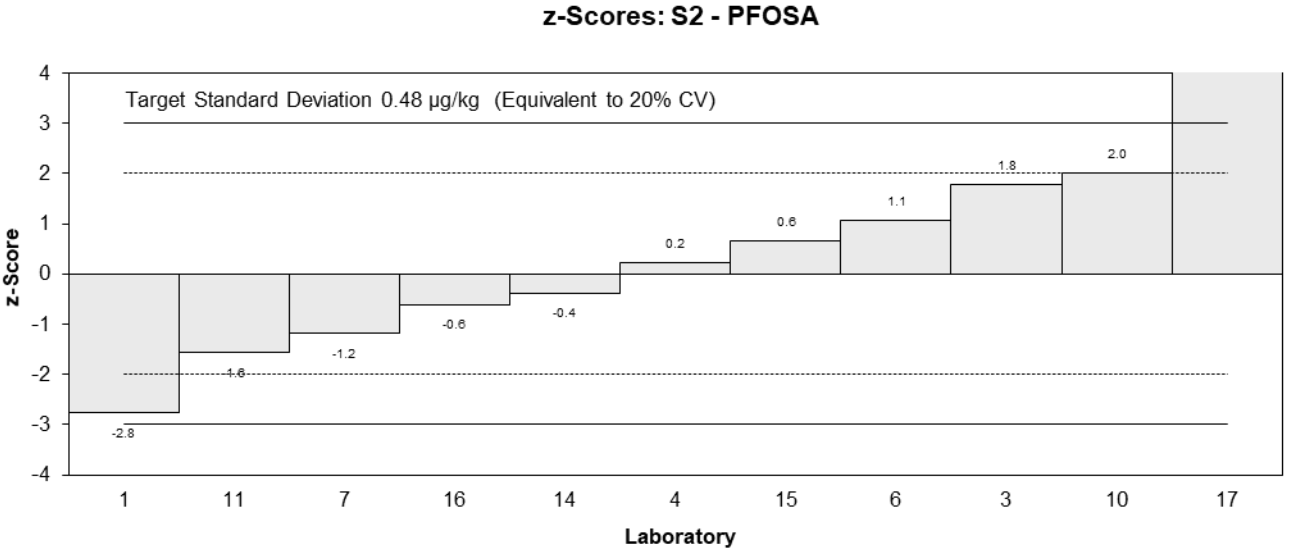
Assigned Value**	2.39	0.55
Spike	3.53	0.18
Max. Acceptable Concentration***	4.49	
Robust Average	2.39	0.71
Median	2.36	0.57
Mean	2.40	
N	10	
Max.	3.79	
Min.	1.07	
Robust SD	0.90	
Robust CV	38%	

* Laboratory 17 was omitted from all statistical calculations (gross error).

** Robust average excluding Laboratories 1 and 10.

*** z-Score adjusted to 2.00 (see Section 6.3).


Kernel Density
Density
Result
Results: S2 - PFOSA
Assigned Value = 2.39 ± 0.55 µg/kg
Maximum Acceptable Concentration
Results (µg/kg)
Laboratory
1 11 7 16 14 4 15 6 3 10 17 13 19 20 18 12 8 Md R.A. S.V.


z-Scores: S2 - PFOSA
Target Standard Deviation 0.48 µg/kg (Equivalent to 20% CV)
z-Score
Laboratory
1 11 7 16 14 4 15 6 3 10 17
-2.8 -1.6 -1.2 -0.6 -0.4 0.2 0.6 1.1 1.8 2.0

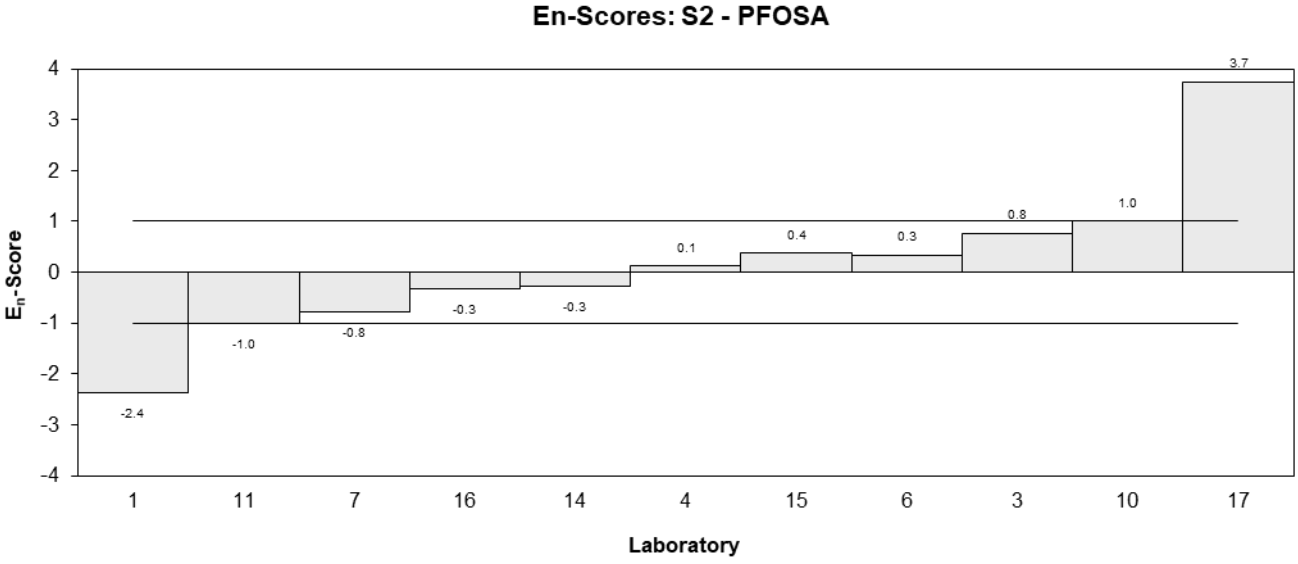

En-Scores: S2 - PFOSA
En-Score
Laboratory
1 11 7 16 14 4 15 6 3 10 17
-2.4 -1.0 -0.8 -0.3 -0.3 0.1 0.4 0.3 0.8 1.0 3.7

Figure 33

Table 38

Sample Details

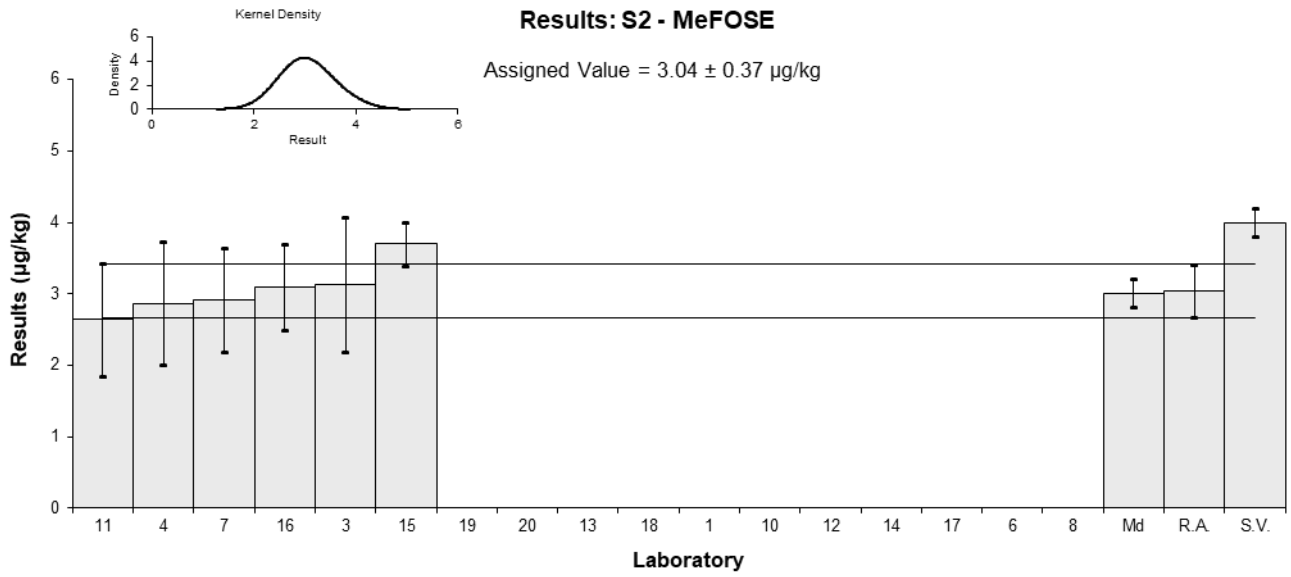
Sample No.	S2
Matrix	Celery
Analyte	MeFOSE
Units	µg/kg

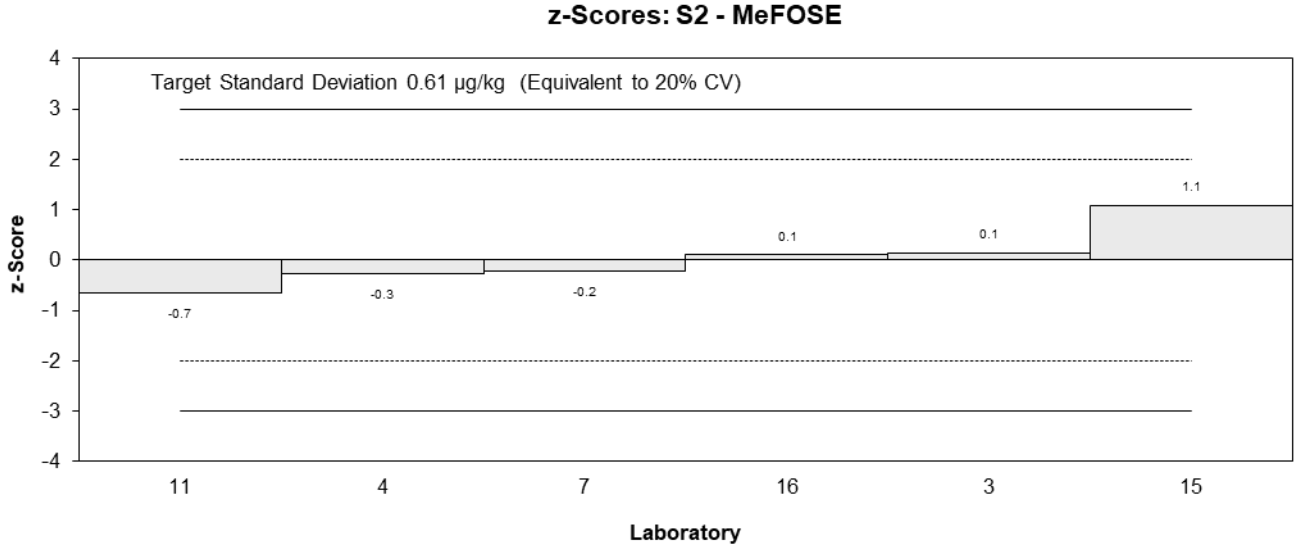
Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	NT	NT	NT		
3	3.13	0.939	41	0.15	0.09
4	2.87	0.86	74	-0.28	-0.18
6	NT	NT	NT		
7	2.91	0.73	81	-0.21	-0.16
8	NT	NT	NT		
10	NT	NT	NT		
11	2.64	0.792	70	-0.66	-0.46
12	NT	NT	NT		
13	<5	NR	146		
14	NT	NT	NT		
15	3.7	0.30	109	1.09	1.39
16	3.1	0.6	9.9	0.10	0.09
17	NT	NT	NT		
18	NR	NR	NR		
19	<10	NR	69		
20	<10	NR	81		

Statistics

Assigned Value	3.04	0.37
Spike	4.00	0.20
Robust Average	3.04	0.37
Median	3.01	0.20
Mean	3.06	
N	6	
Max.	3.7	
Min.	2.64	
Robust SD	0.36	
Robust CV	12%	


Kernel Density
Density
Result
Results: S2 - MeFOSE
Assigned Value = 3.04 ± 0.37 µg/kg
Results (µg/kg)
Laboratory
11 4 7 16 3 15 19 20 13 18 1 10 12 14 17 6 8 Md R.A. S.V.


z-Scores: S2 - MeFOSE
Target Standard Deviation 0.61 µg/kg (Equivalent to 20% CV)
z-Score
-0.7
-0.3
-0.2
0.1
0.1
1.1
Laboratory
11 4 7 16 3 15

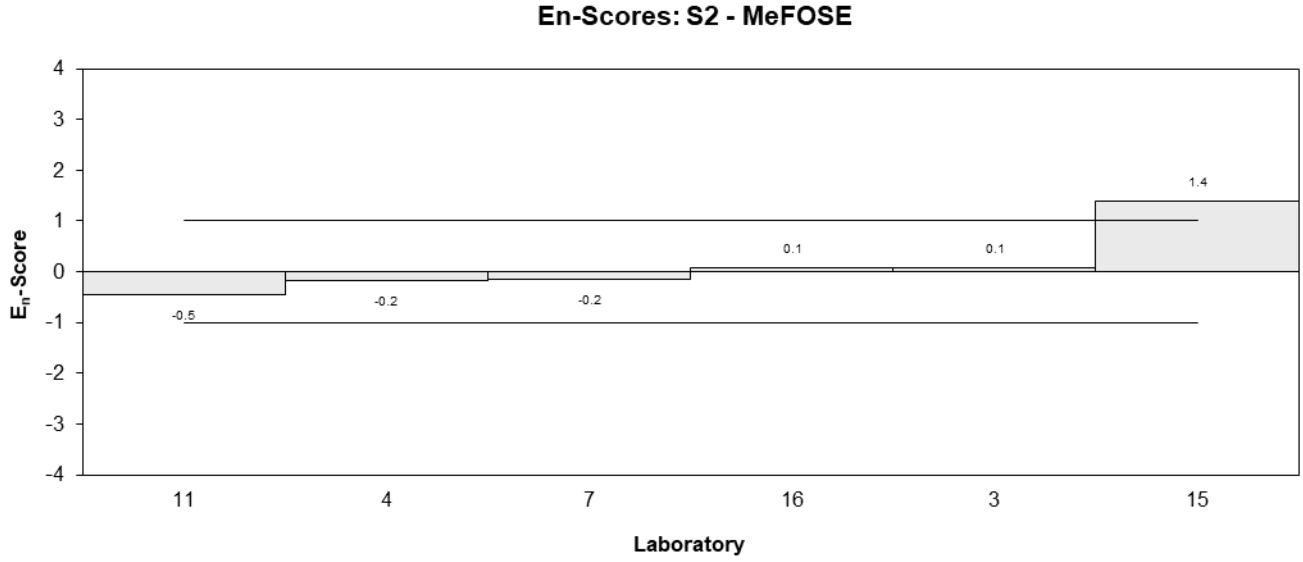

En-Scores: S2 - MeFOSE
En-Score
-0.5
-0.2
-0.2
0.1
0.1
1.4
Laboratory
11 4 7 16 3 15

Figure 34

Table 39

Sample Details

Sample No.	S2
Matrix	Celery
Analyte	10:2 FTS
Units	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Recovery
1	1.12	0.02	NR
3	2.89	0.867	152
4	1.76	0.53	92
6	NT	NT	NT
7	0.937	0.23	115
8	NT	NT	NT
10	3.92	0.8	NR
11	1.723	0.517	110
12	NT	NT	NT
13	<2	NR	501
14	NT	NT	NT
15	NR	NR	NR
16	1.1	0.5	29
17	5.04	0.61	NR
18	NR	NR	NR
19	2.6	2	90
20	2.8	2	83

Statistics*

Assigned Value	Not Set	
Spike	3.39	0.17
Robust Average	2.10	0.92
Median	1.76	0.94
Mean	2.09	
N	9	
Max.	3.92	
Min.	0.937	
Robust SD	1.1	
Robust CV	53%	

* Laboratory 17 was omitted from all statistical calculations (gross error).

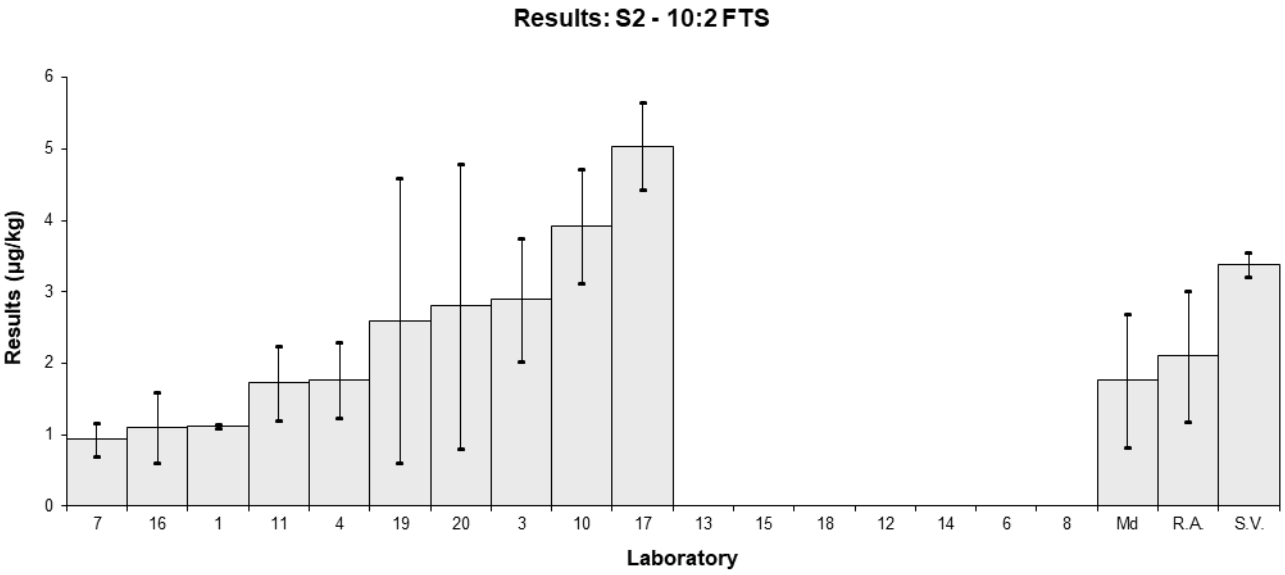

Results: S2 - 10:2 FTS
Results (µg/kg)
0
1
2
3
4
5
6
7
16
1
11
4
19
20
3
10
17
13
15
18
12
14
6
8
Md
R.A.
S.V.
Laboratory

Figure 35

6 DISCUSSION OF RESULTS

6.1 Assigned Value

The robust average of participants' results was used as the assigned value for each scored analyte. The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528:2015.[5] Results less than 50% and greater than 150% of the robust average were removed before the calculation of the assigned value.[3,4] The calculation of the expanded uncertainty for the robust average is presented in Appendix 4, using PFHxS (linear) in Sample S1 as an example.

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.

No assigned value was set for Sample S1 GenX as there were too few numeric results reported. No assigned value was set for Sample S2 10:2 FTS as reported numeric results were too variable; the variability may have been due to difficulties in the analysis of this analyte caused by the matrix, analyte mass fraction level, properties of the analyte itself, or a combination of these factors.

A comparison of the assigned value (or robust average if the analyte was not scored) and spiked value of each analyte is presented in Table 40. For this study, the assigned values for scored analytes were within 70% to 98% and 68% to 110% of the spiked values for Samples S1 and S2 respectively. These are similar to what has been observed in previous PFAS in Food PT studies, and provides good support for the assigned values and analyte stability.

Table 40 Comparison of Assigned Values (or Robust Averages) and Spiked Values

Sample	Analyte	Assigned Value *(Robust Average)* (µg/kg)	Spiked Value (µg/kg)	Assigned Value *(Robust Average)* / Spiked Value (%)
S1 (Beef Meat)	PFBS	2.18	2.90	75
	PFHxS	2.81	3.68	76
	PFHxS (linear)	2.67	3.68	73
	PFHpS	1.60	1.92	83
	PFOS	31.0	37.2	83
	PFOS (linear)	31.6	37.2	85
	PFDS	17.7	23.1	77
	PFBA	12.2	17.5	70
	PFPeA	0.739	0.970	76
	PFHxA	0.640	0.677	95
	PFHpA	1.92	1.95	98
	PFOA	33.4	38.6	87
	PFNA	3.91	4.36	90
	PFDA	4.14	4.35	95
	PFUdA	0.318	0.387	82
	8:2 FTS	7.7	9.26	83
	GenX	*(6.5)*	7.28	*(89)*

Sample	Analyte	Assigned Value *(Robust Average)* (µg/kg)	Spiked Value (µg/kg)	Assigned Value *(Robust Average)* / Spiked Value (%)
S2 (Celery)	PFBS	1.37	1.50	91
	PFPeS	11.4	11.3	101
	PFHxS	8.54	9.45	90
	PFHxS (linear)	8.45	9.45	89
	PFHpS	0.88	1.10	80
	PFOS	1.08	1.43	76
	PFOS (linear)	1.09	1.43	76
	PFDS	3.39	4.79	71
	PFBA	2.51	2.48	101
	PFPeA	0.68	0.746	91
	PFHxA	16.5	15.0	110
	PFHpA	0.746	0.795	94
	PFOA	1.59	1.81	88
	PFNA	0.78	0.990	79
	PFOSA	2.39	3.53	68
	MeFOSE	3.04	4.00	76
	10:2 FTS	*(2.10)*	3.39	*(62)*

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded MU associated with their results and the basis of this uncertainty estimate. It is a requirement of ISO/IEC 17025:2017 that laboratories have procedures to estimate the uncertainty of chemical measurements and to report this in specific circumstances, including when the client's instruction so requires.[7]

Of 437 numeric results reported for analytes of interest in this study, 436 were reported with an uncertainty. Laboratory **15** did not provide an uncertainty for one of their reported analytes; this participant was not accredited. Laboratory **10** attached an estimate of MU to a non-numeric result reported; an uncertainty expressed as a value should not be attached to a non-value result.[8]

Participants' procedures for estimating their uncertainty are presented in Table 3. A number of participants reported using the NATA GAG Estimating and Reporting MU as their guide; NATA no longer publishes this document.[9]

The magnitude of the MUs for analytes in this study was within the range 0.05% to 100% of the reported value. In general, an expanded uncertainty of less than 10% relative is likely to be unrealistically small for the routine analysis of PFAS, while over 50% is likely too large. Of the 436 MUs, 104 were less than 10% relative and 28 were greater than 50% relative.

Uncertainties associated with results returning a satisfactory z-score but an unsatisfactory E_n-score may have been underestimated.

In some cases, results and/or uncertainties were reported with an inappropriate number of significant figures. Including too many significant figures may inaccurately reflect the precision of measurements. The recommended format is to write the uncertainty to no more than two significant figures and then to write the result with the corresponding number of decimal places. For example, instead of 31.266 ± 9.380 µg/kg, it is better to report this as 31.3 ± 9.4 µg/kg.[8]

6.3 z-Score

Target SDs equivalent to 20% PCV were used to calculate z-scores. CVs predicted by the Thompson-Horwitz equation,[6] target SDs (as PCVs), and the between-laboratory CVs obtained in this study for scored analytes are presented for comparison in Table 41.

Table 41 Comparison of Thompson-Horwitz CVs, Target SDs and Between-Laboratory CVs

Sample	Analyte	Assigned Value (µg/kg)	Thompson-Horwitz CV (%)	Target SD (as PCV) (%)	Between-Laboratory CV* (%)
S1 (Beef Meat)	PFBS	2.18	22	20	13
	PFHxS	2.81	22	20	11
	PFHxS (linear)	2.67	22	20	16
	PFHpS	1.60	22	20	17
	PFOS	31.0	22	20	10
	PFOS (linear)	31.6	22	20	9.6
	PFDS	17.7	22	20	20
	PFBA	12.2	22	20	14
	PFPeA	0.739	22	20	15
	PFHxA	0.640	22	20	15
	PFHpA	1.92	22	20	8.6
	PFOA	33.4	22	20	10
	PFNA	3.91	22	20	9.6
	PFDA	4.14	22	20	9.7
	PFUdA	0.318	22	20	14
	8:2 FTS	7.7	22	20	22
S2 (Celery)	PFBS	1.37	22	20	14
	PFPeS	11.4	22	20	12
	PFHxS	8.54	22	20	10
	PFHxS (linear)	8.45	22	20	11
	PFHpS	0.88	22	20	21
	PFOS	1.08	22	20	20
	PFOS (linear)	1.09	22	20	20
	PFDS	3.39	22	20	20
	PFBA	2.51	22	20	9.4
	PFPeA	0.68	22	20	21
	PFHxA	16.5	22	20	9.2
	PFHpA	0.746	22	20	13
	PFOA	1.59	22	20	13
	PFNA	0.78	22	20	29
	PFOSA	2.39	22	20	26
	MeFOSE	3.04	22	20	12

* Robust between-laboratory CV with outliers removed, if applicable. Shaded cells are between-laboratory CVs which were higher than both the target SD and the Thompson-Horwitz CV.

To account for possible low bias in the consensus value due to laboratories using inefficient analytical or extraction techniques, one z-score was adjusted for Sample S2 PFOSA. A maximum acceptable concentration was set to two target SDs more than the spiked value, and results lower than the maximum acceptable concentration but with a z-score greater than 2.0 had their z-score adjusted to 2.0. This ensured that laboratories reporting results close to the spiked value were not penalised. z-Scores for results higher than the maximum acceptable concentration were not adjusted, and z-scores less than 2.0 were left unaltered.

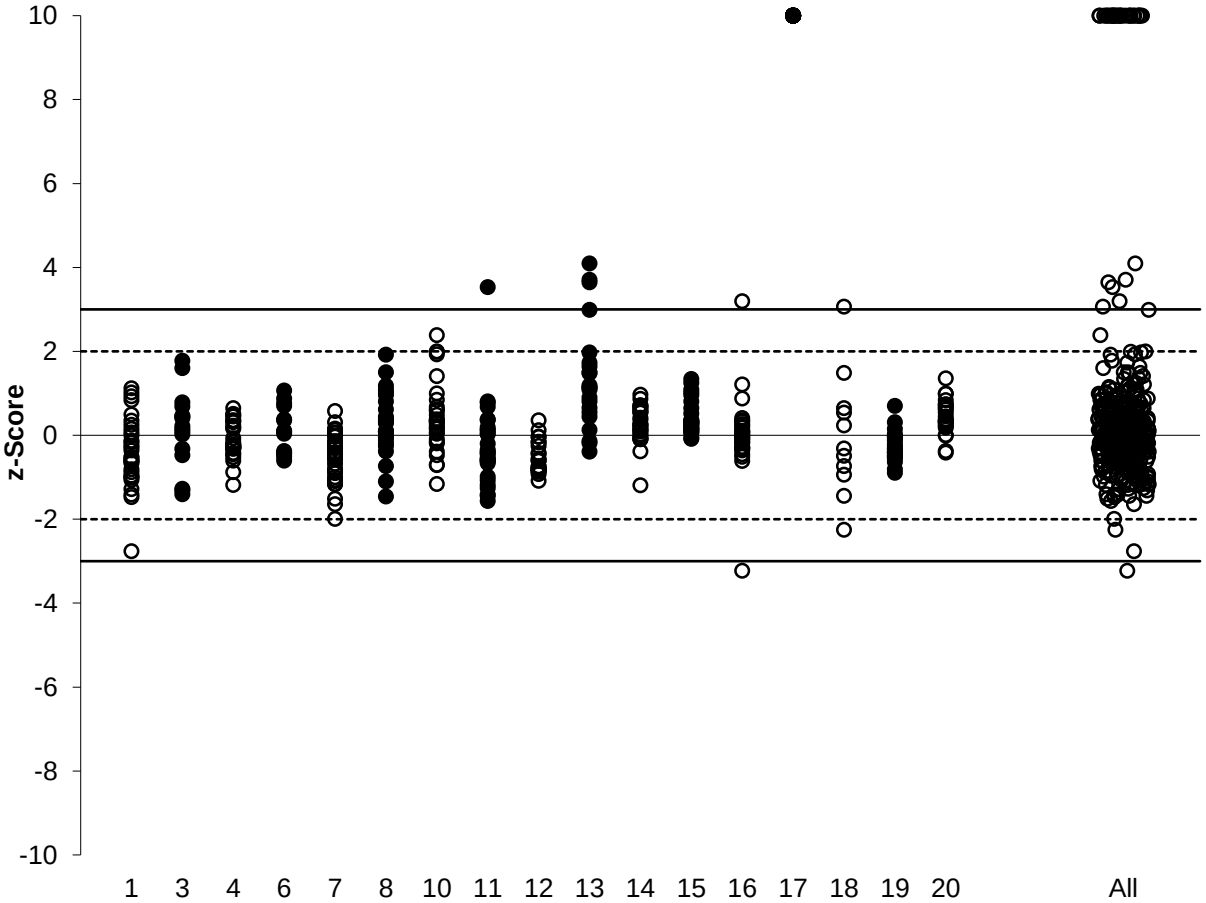
Of 422 results for which z-scores were calculated, 383 (91%) returned $|z| \leq 2.0$, indicating a satisfactory performance.

Fifteen participants analysed both samples, with Laboratories **7** and **16** reporting numeric results for all 32 scored analytes in these samples. Laboratory **7** returned satisfactory z-scores for all analytes. Laboratories **4** (28), **14** (28), **8** (26), **15** (26), **12** (23), **19** (23) and **20** (23) returned satisfactory z-scores for all reported results.

Laboratory **17** returned unsatisfactory z-scores for all reported results (28), with all numeric results significantly higher than the assigned value (z-scores ranged from 10.61 to 22.20 for Sample S1, and from 18.45 to 371.11 for Sample S2).

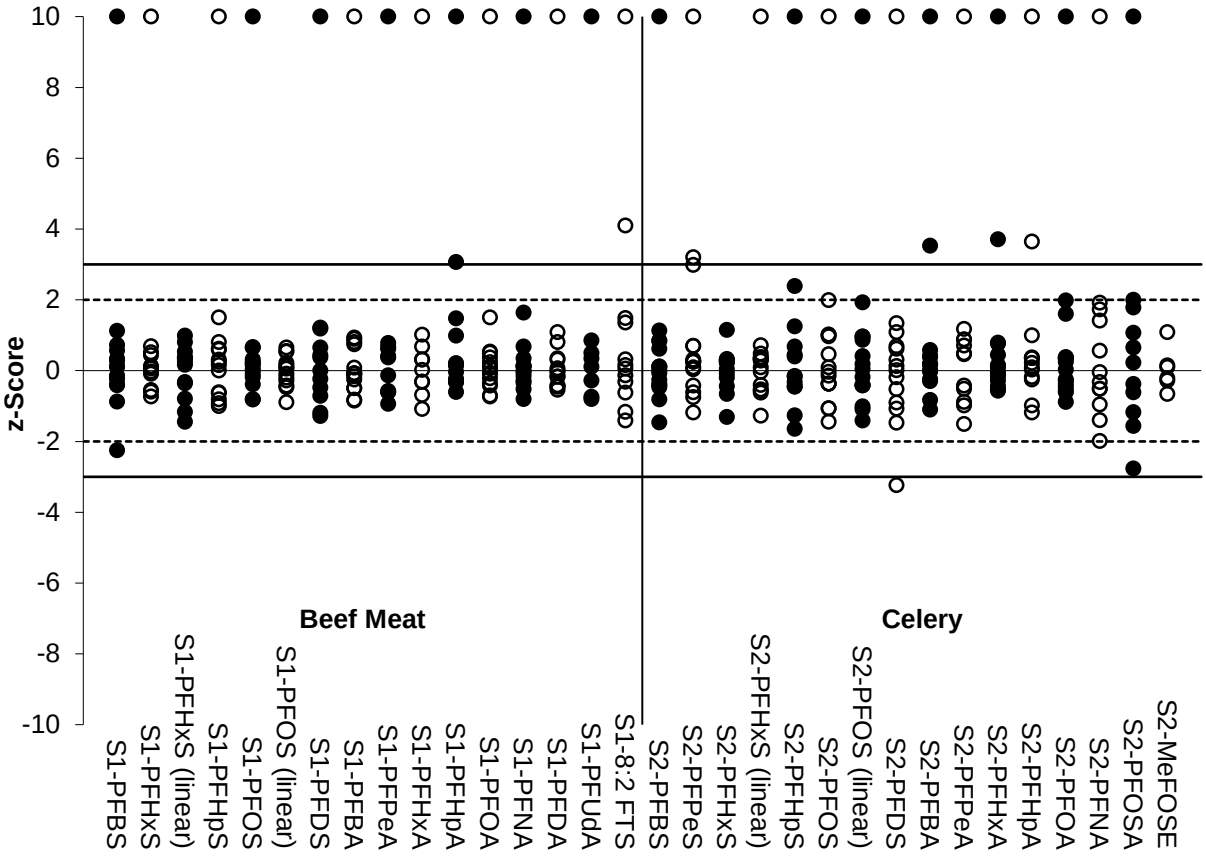
Two participants analysed Sample S2 (celery) only. Laboratory **3** returned satisfactory z-scores for all scored analytes in this sample (16). Laboratory **6** returned satisfactory z-scores for all reported results (14).

The dispersal of participants' z-scores is presented graphically by laboratory in Figure 36 and by analyte in Figure 37.



z-Scores greater than 10 have been plotted as 10.

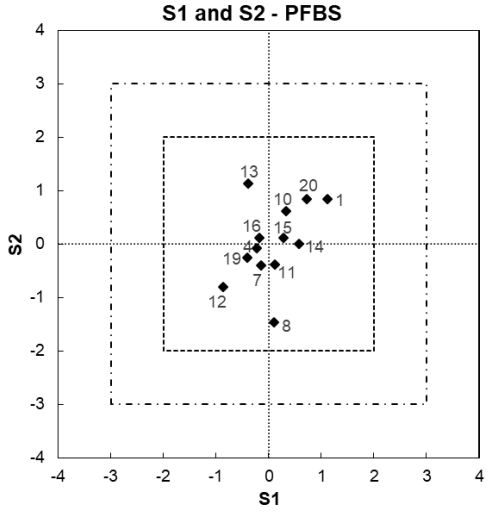
Figure 36 z-Score Dispersal by Laboratory



z-Scores greater than 10 have been plotted as 10.

Figure 37 z-Score Dispersal by Analyte

Scatter plots of z-scores for analytes present in both Samples S1 (Beef Meat) and S2 (Celery) are presented in Figures 38 to 50. Scores are predominantly in the upper right and lower left quadrants, indicating that laboratory bias is the major contributor to the variability of results. Points close to the diagonal axis demonstrate excellent repeatability, while points close to the zero demonstrate excellent repeatability and accuracy.



Laboratory 17 is off-scale.

Figure 38 z-Score Scatter Plot – PFBS

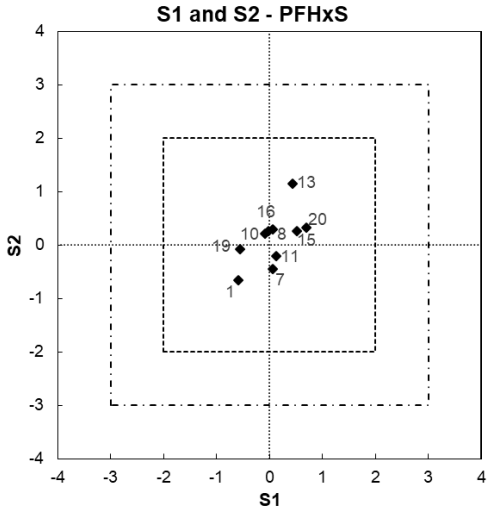


Figure 39 z-Score Scatter Plot – PFHxS

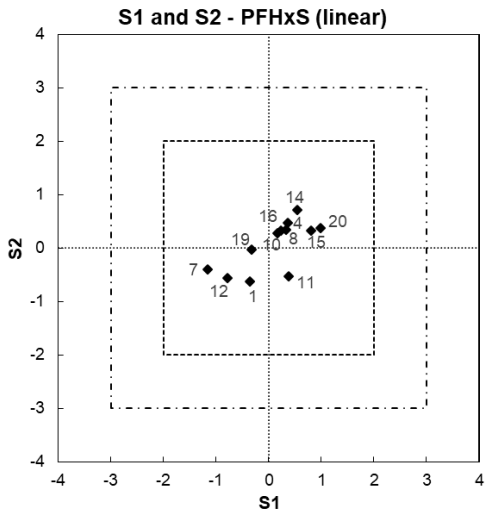
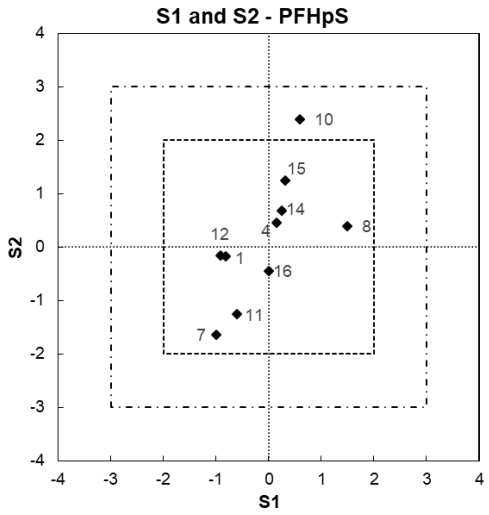
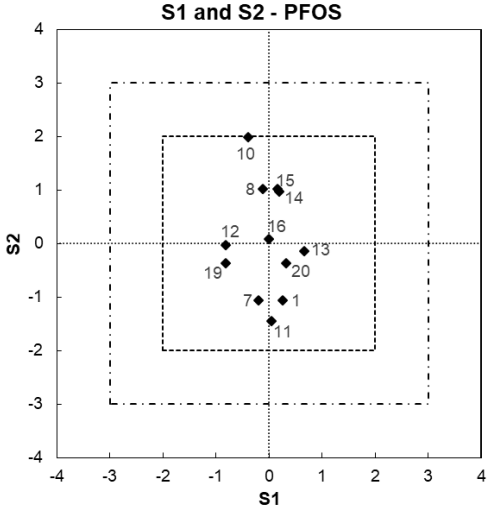


Figure 40 z-Score Scatter Plot – PFHxS (linear)



Laboratory 17 is off-scale.

Figure 41 z-Score Scatter Plot – PFHpS



Laboratory 17 is off-scale.

Figure 42 z-Score Scatter Plot – PFOS

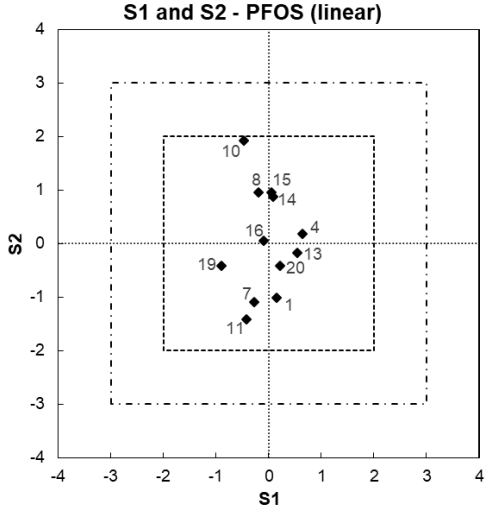
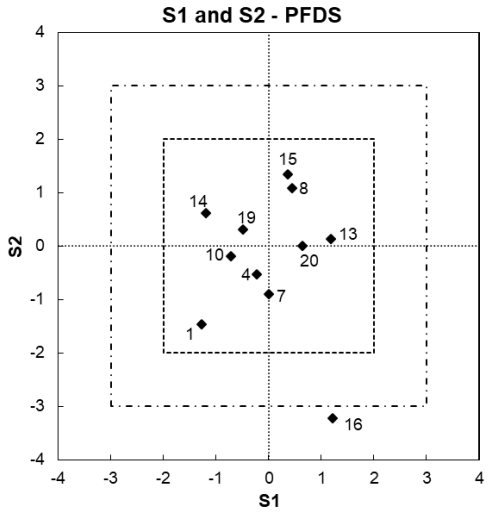
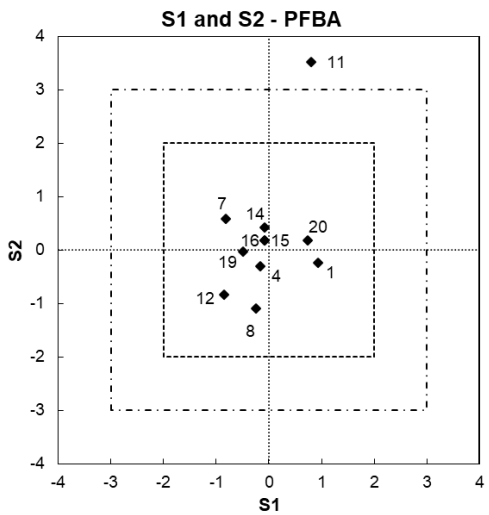


Figure 43 z-Score Scatter Plot – PFOS (linear)



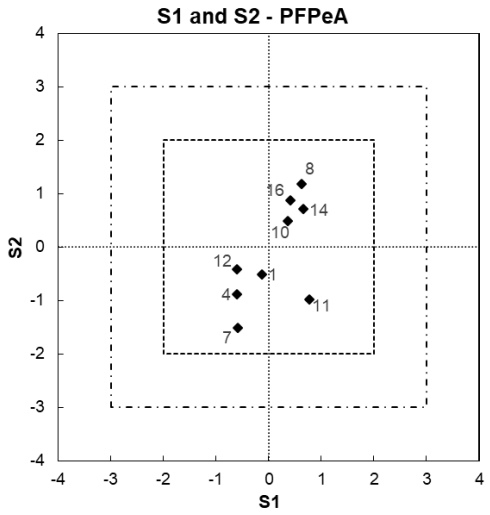
Laboratory 17 is off-scale.

Figure 44 z-Score Scatter Plot – PFDS



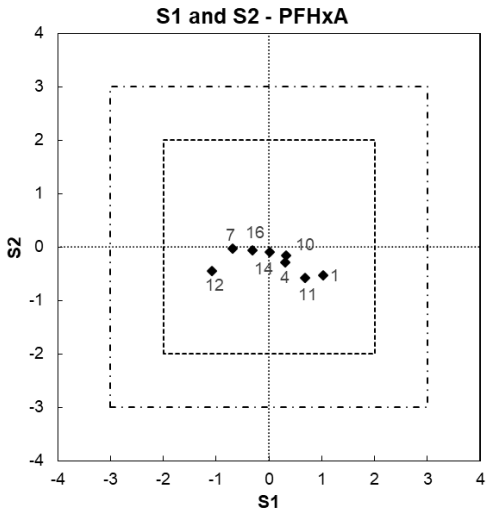
Laboratory 17 is off-scale.

Figure 45 z-Score Scatter Plot – PFBA



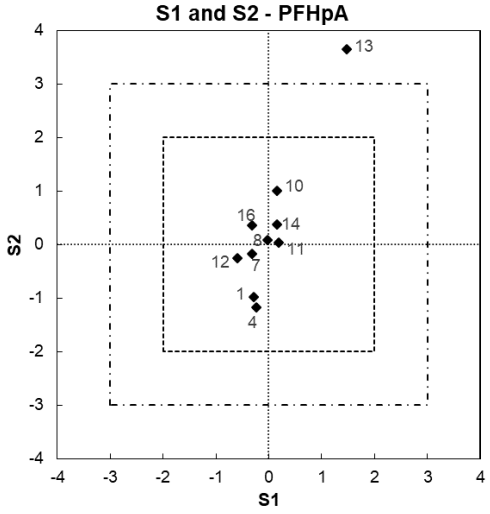
Laboratory 17 is off-scale.

Figure 46 z-Score Scatter Plot – PFPeA



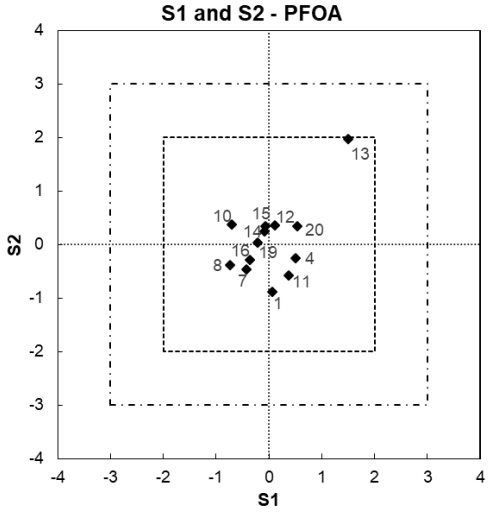
Laboratory 17 is off-scale.

Figure 47 z-Score Scatter Plot – PFHxA



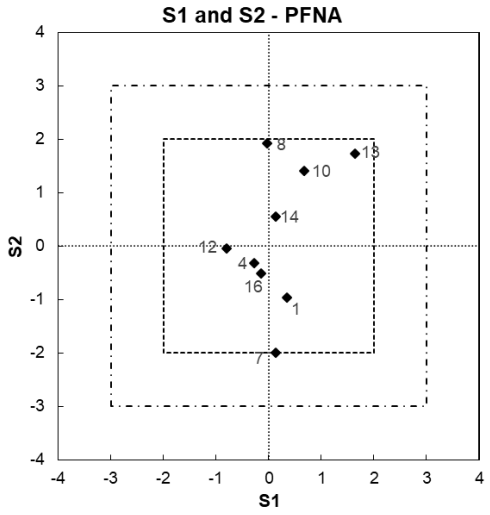
Laboratory 17 is off-scale.

Figure 48 z-Score Scatter Plot – PFHpA



Laboratory 17 is off-scale.

Figure 49 z-Score Scatter Plot – PFOA



Laboratory 17 is off-scale.

Figure 50 z-Score Scatter Plot – PFNA

6.4 E_n-Score

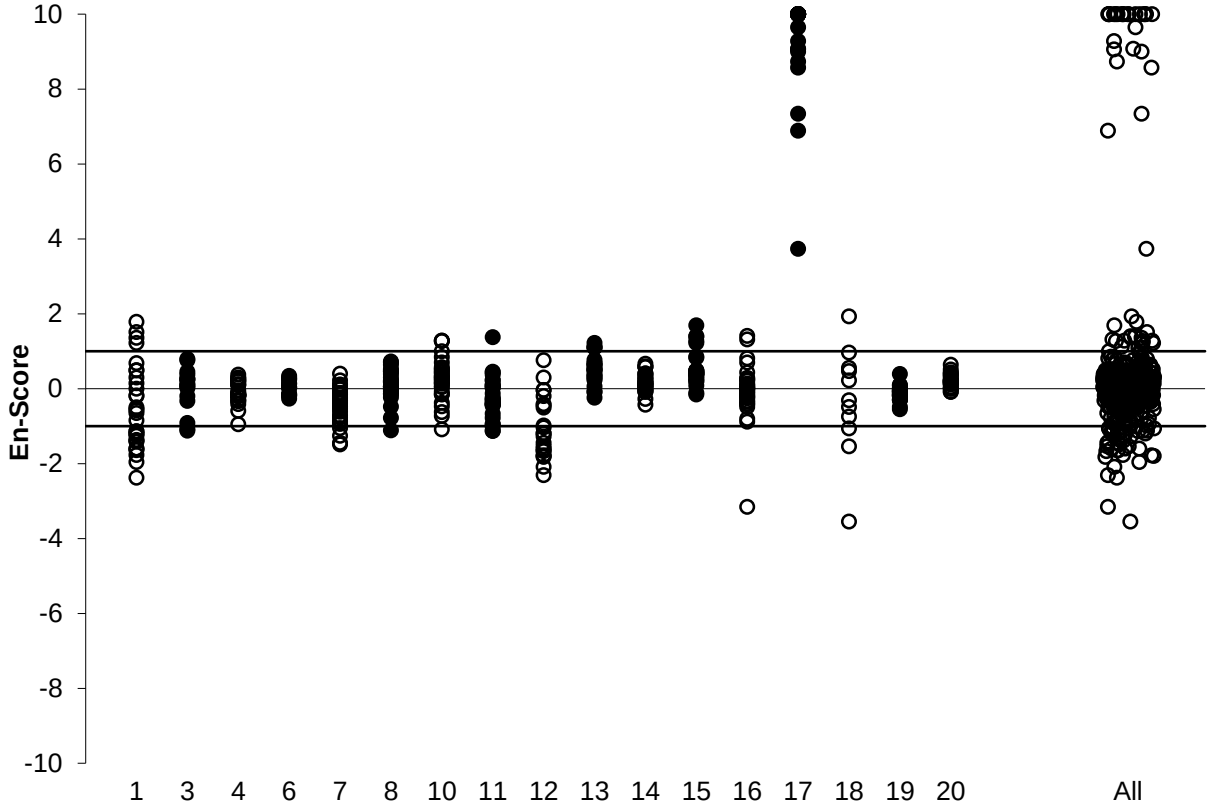
E_n-scores should be interpreted in conjunction with z-scores; an unsatisfactory E_n-score can either be caused by an inappropriate measurement, or uncertainty, or both. If a participant did not report any uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the E_n-score. E_n-scores greater than 1.0 were set to 1.0 for results with z-scores that were adjusted as discussed in Section 6.3 z-Scores.

Of 422 results for which E_n-scores were calculated, 331 (78%) returned $|E_n| \leq 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

No participant returned satisfactory E_n-scores for all analytes of interest in this study. Of the participants analysing both matrices, Laboratories **4** (28), **14** (28), **19** (23) and **20** (23) returned satisfactory E_n-scores for all reported results. Of the participants analysing Sample S2 (celery) only, Laboratory **6** (14) returned satisfactory E_n-scores for all reported results.

Laboratory **17** returned unsatisfactory E_n-scores for all reported results (28).

The dispersal of participants' E_n-scores is presented graphically in Figure 51.



E_n-Scores greater than 10 have been plotted as 10.

Figure 51 E_n-Score Dispersal by Laboratory

6.5 False Negatives

Table 42 presents false negative results. These are analytes present in the samples which a participant tested for, but did not report a result (for example, participants reporting a 'less-than' result (<*x*) when the assigned and spiked values were higher than their limit of reporting (LOR), or laboratories that did not report any value). For analytes where no assigned value was set, results have only been considered to be false negatives where the robust average and spiked value were significantly higher than the participants' LOR, or if no value was reported.

Table 42 False Negatives

Lab. Code	Sample	Analyte	Assigned Value *(Robust Average)* (µg/kg)	Spiked Value (µg/kg)	Result* (µg/kg)
8	S1	PFUdA	0.318	0.387	NR
		PFHxA	0.640	0.677	NR
11	S1	PFDS	17.7	23.1	<0.5
	S2	PFNA	0.78	0.99	<0.2
12	S1	PFHxS	2.81	3.68	NR
		PFOS (linear)	31.6	37.2	NR
	S2	PFHxS	8.54	9.45	NR
		PFOS (linear)	1.09	1.43	NR
14	S2	PFHxS	8.54	9.45	NR
15	S2	10:2 FTS	*(2.10)*	3.39	NR
17	S1	PFHxS (linear)	2.67	3.68	NR
		PFOS (linear)	31.6	37.2	NR
		GenX	*(6.5)*	7.28	<0.01
	S2	PFHxS	8.54	9.45	NR
18	S2	PFBS	1.37	1.50	NR
		PFPeS	11.4	11.3	NR
		PFHxS	8.54	9.45	NR
		PFHxS (linear)	8.45	9.45	NR
		PFHpS	0.88	1.10	NR
		PFOS	1.08	1.43	NR
		PFOS (linear)	1.09	1.43	NR
		PFDS	3.39	4.79	NR
		PFBA	2.51	2.48	NR
		PFPeA	0.68	0.746	NR
		PFHxA	16.5	15.0	NR
		PFHpA	0.746	0.795	NR
		PFOA	1.59	1.81	NR
		PFNA	0.78	0.990	NR
		PFOSA	2.39	3.53	NR
		MeFOSE	3.04	4.00	NR
		10:2 FTS	*(2.10)*	3.39	NR

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

6.6 Reporting of Additional Analytes

Eight laboratories reported at least one PFAS analyte that was not spiked into the test samples by the study coordinator. These results are presented in Table 43. Participants should take care to avoid any potential cross-contamination when analysing their samples.

Table 43 Non-Spiked Analytes Reported by Participants

Lab. Code	Sample	Analyte	Result (µg/kg)	Uncertainty (µg/kg)	Recovery (%)
1	S1	PFOSA	0.06	0.02	NR
		4:2 FTS	0.11	0.03	NR
		6:2 FTS	0.01	0.05	NR
		ADONA	0.02	0.04	NR
	S2	PFDA	0.04	0.02	NR
		4:2 FTS	0.1	0.03	NR
		6:2 FTS	0.005	0.05	NR
7	S2	PFDA	0.00448	0.0011	94
10	S2	PFUdA	0.15	0.05	59
		PFDoA	0.2	0.05	38
		PFTrDA	21.5	4	NR
11	S2	PFDA	0.564	0.169	87
12	S2	PFDA	0.045	0.007	83
14	S1	PFDoS	0.104	0.055	67.4
		EtFOSA	1.84	0.258	16.3
	S2	PFDoS	0.045	0.024	83.7
16	S2	EtFOSAA	0.8	NR	17
		EtFOSE	0.6	NR	11
17	S1	ADONA	10.64	0.96	NR
	S2	PFDA	0.48	0.3	41
		PFTrDA	0.026	0.34	NR
		PFTeDA	0.36	0.51	NR
		6:2 FTS	34.55	0.2	52
		ADONA	31.09	0.16	NR

6.7 Range of PFAS Analysed by Participants

Participants were provided with a list of potential analytes that could have been spiked into the test samples (Table 1). Of these, 19 different PFAS analytes were spiked for this study, with 15 analytes spiked into each sample. For PFHxS and PFOS, both samples were spiked with linear only isomers, and participants were requested to reported both linear isomers only and total value. Participants were not required to test for all potential PFAS analytes, and were requested to report “NT” (for “Not Tested”) for any PFAS they did not analyse the samples for.

A summary of participants’ testing of the spiked PFAS is presented in Table 44. Where information is only applicable to one sample, for each cell the top left corresponds to Sample

S1 beef meat and the bottom right corresponds to Sample S2 celery. Black indicates that the analyte was not spiked into the sample and grey indicates that the participant did not analyse that sample.

Of the participants who analysed both samples, Laboratories **7**, **11**, **15** and **16** reported that they tested for all spiked analytes. Of the participants who only analysed Sample S2 celery, Laboratory **3** reported that they tested for all analytes spiked into this sample. All participants tested for at least one spiked analyte, with the proportion of analytes being tested for by each participant ranging from 71% to 100%. Laboratories **14** and **18** reported testing for some analytes in one sample but not the other.

Out of the spiked analytes in this study, PFBS, PFOS (linear), PFBA, PFPeA, PFHxA, PFHpA and PFOA were tested for by the highest proportion of participants (100% for all). In general, perfluoroalkyl acids were very well represented by participants, with the overall proportion of analysis by participants being 96%. A lower proportion of participants analysed the perfluoroalkane sulfonamide (PFOSA) and fluorotelomer (8:2 FTS and 10:2 FTS) analytes, being 88% and 78% respectively. Significantly fewer participants analysed for the perfluoroalkane sulfonamido (MeFOSE) and PFAS replacement (GenX) compounds, at 59% and 40% respectively.

Table 44 Summary of PFAS Analysed by Participants

Analyte \ Lab. Code	1	3	4	6	7	8	10	11	12	13	14	15	16	17	18	19	20	Proportion of Participants (%)
PFBS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	100
PFPeS	✓	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	94
PFHxS	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	NT / ✓	✓	✓	✓	✓	✓	✓	91
PFHxS (linear)	✓	✓	✓	✓	✓	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	94
PFHpS	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	NT / ✓	✓	✓	97
PFOS	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	94
PFOS (linear)	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	100
PFDS	✓	✓	✓	NT	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	NT / ✓	✓	✓	85
PFBA	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	100
PFPeA	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	100
PFHxA	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	100
PFHpA	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	100
PFOA	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	100
PFNA	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	NT / ✓	✓	✓	97
PFDA	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	NT	✓	✓	93
PFUdA	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	NT	✓	✓	93
PFOSA	✓	✓	✓	✓	✓	NT	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	88
MeFOSE	NT	✓	✓	NT	✓	NT	NT	✓	NT	✓	NT	✓	✓	NT	✓	✓	✓	59
8:2 FTS	✓	✓	✓	✓	✓	NT	✓	✓	NT	✓	NT	✓	✓	✓	✓	✓	✓	80
10:2 FTS	✓	✓	✓	NT	✓	NT	✓	✓	NT	✓	NT	✓	✓	✓	✓	✓	✓	76
GenX	NT	NT	NT	✓	NT	NT	✓	✓	NT	NT	✓	✓	✓	NT	NT	NT	NT	40
Proportion of Analytes (%)	90	100	86	82	100	71	90	100	76	90	79	100	100	95	79	95	95	

6.8 PFAS in Food Trigger Points

There are currently no maximum regulatory limits in Australia for PFAS contaminants in food. However, Food Standards Australia New Zealand (FSANZ) has proposed non-regulatory 'trigger points' in a variety of food products for 3 common PFAS compounds, namely PFHxS, PFOS and PFOA, based on food consumption rates and set tolerable daily intakes for these analytes.[10] Where an analyte is found to be exceeding the corresponding trigger point, this may indicate that further investigation is required.

The assigned values and relevant FSANZ trigger points for these analytes in this study are given in Table 45. Sample S1 PFHxS and Sample S2 PFOA are below the trigger points, while Sample S1 PFOS and PFOA, and Sample S2 PFHxS are above the trigger points. The assigned value for Sample S2 PFOS is just below the trigger point, with the uncertainty spanning the trigger point.

Table 45 Assigned Values and FSANZ Trigger Points for PFHxS, PFOS and PFOA

Sample	Classification	PFHxS (µg/kg)		PFOS (µg/kg)		PFOA (µg/kg)	
		Assigned Value	Trigger Point	Assigned Value	Trigger Point	Assigned Value	Trigger Point
S1 (Beef Meat)	Meat mammalian	2.81 ± 0.22	3.5	31.0 ± 2.2	3.5	33.4 ± 2.3	28
S2 (Celery)	Vegetables	8.54 ± 0.63	1.1	1.08 ± 0.15	1.1	1.59 ± 0.13	8.8

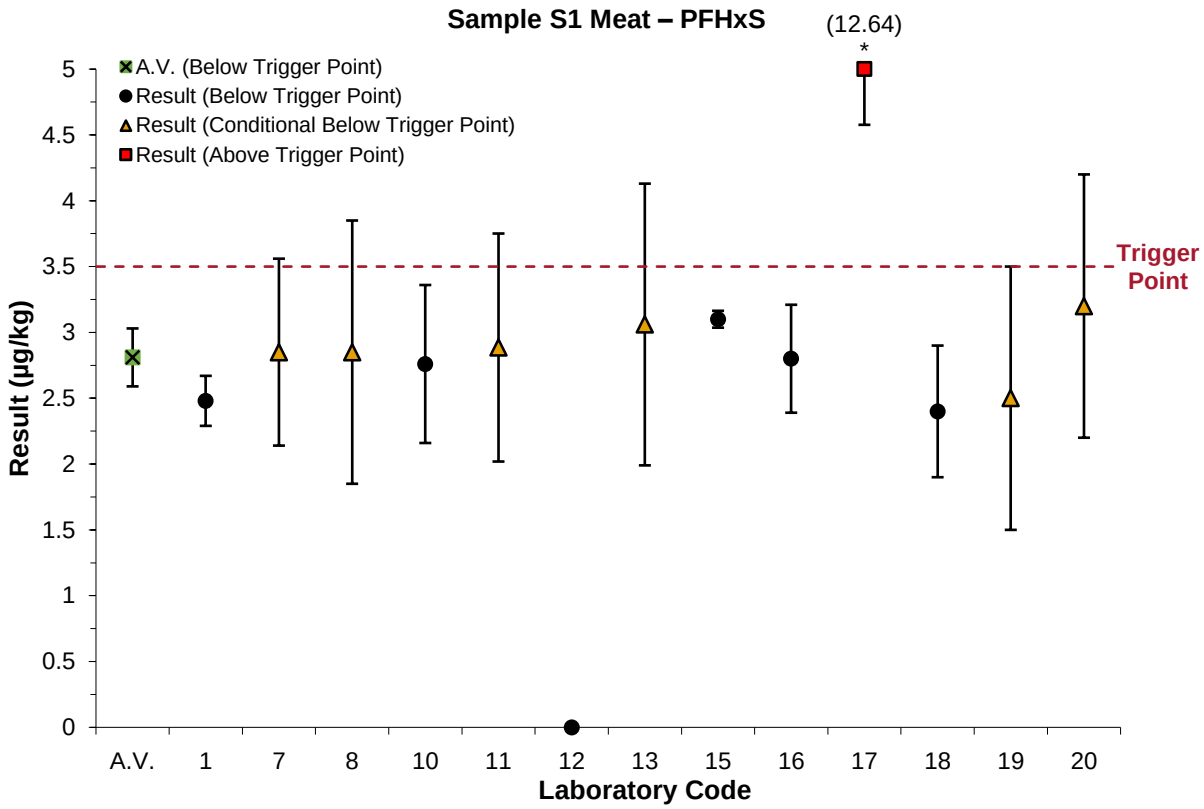
Figures 52 to 57 show comparisons of the assigned values (A.V.), participants' results, and FSANZ trigger points for Samples S1 and S2 PFHxS, PFOS and PFOA (total where relevant). Where no numeric result or LOR was reported, and the participant did not report that the analyte was not tested for, these results have been plotted as zero (0).

The majority of participants' results matched the assigned values with respect to being above or below the FSANZ trigger points. Of 75 results assessed, 54 (72%) were correctly above or below the trigger point inclusive of uncertainty, and a further 15 (20%) were correctly above or below the trigger point with uncertainty spanning the trigger point. Laboratories **1** (5), **15** (5), **16** (5), **3** (2) and **6** (2) correctly identified whether the analyte mass fractions (inclusive of uncertainties) were above or below the trigger points for all reported analytes assessed.

Laboratory **17** has likely reported results on an incorrect basis (dried instead of as received, which was requested for this study) and therefore the majority of their numeric results were significantly higher than the assigned value. For the analytes in this study which were below the trigger point, Sample S1 PFHxS and Sample S2 PFOA, this participant's results would have incorrectly indicated the need for further investigation.

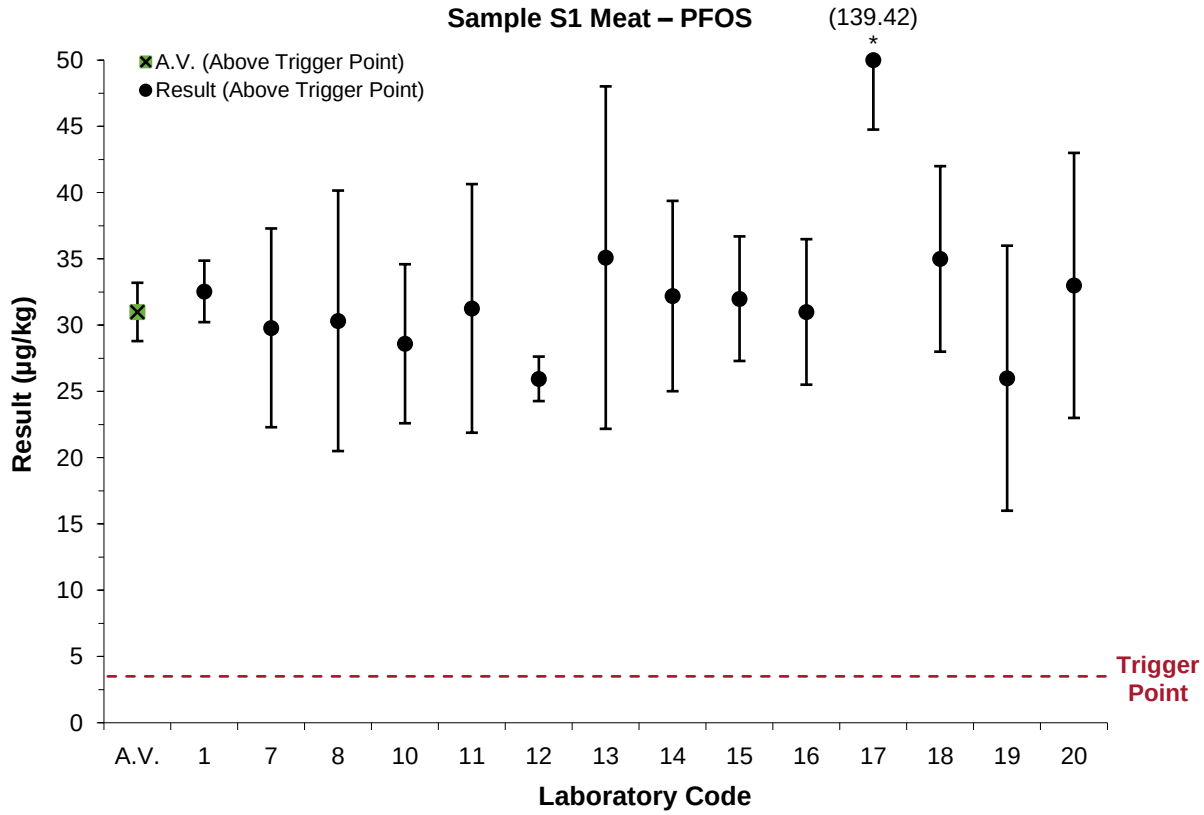
For Sample S2 PFHxS the assigned value was higher than the trigger point, however Laboratories **12**, **14**, **17** and **18** did not report values for this analyte and therefore would have incorrectly indicated no need for further investigation.

The assigned value for Sample S2 PFOS was just below the trigger point, with the uncertainty spanning across the trigger point. The majority of participants' results also spanned or were close to the trigger point. Laboratory **17** reported a value significantly higher than the trigger point, while Laboratory **18** did not report any result for this analyte.



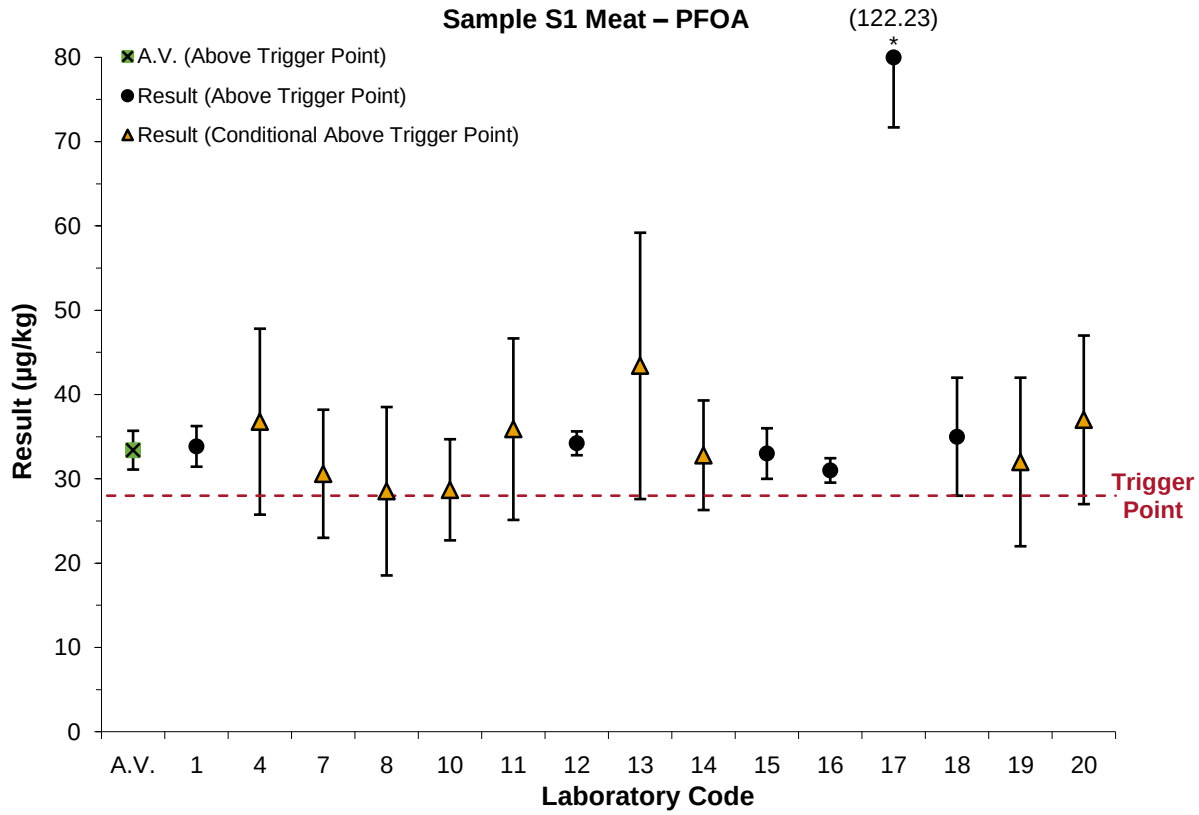
* Result from Laboratory 17 has been scaled to fit on the chart; original result in parentheses.

Figure 52 Sample S1 Meat PFHxS Assigned Value, Participant Results and Trigger Point



* Result from Laboratory 17 has been scaled to fit on the chart; original result in parentheses.

Figure 53 Sample S1 Meat PFOS Assigned Value, Participant Results and Trigger Point



* Result from Laboratory 17 has been scaled to fit on the chart; original result in parentheses.

Figure 54 Sample S1 Meat PFOA Assigned Value, Participant Results and Trigger Point

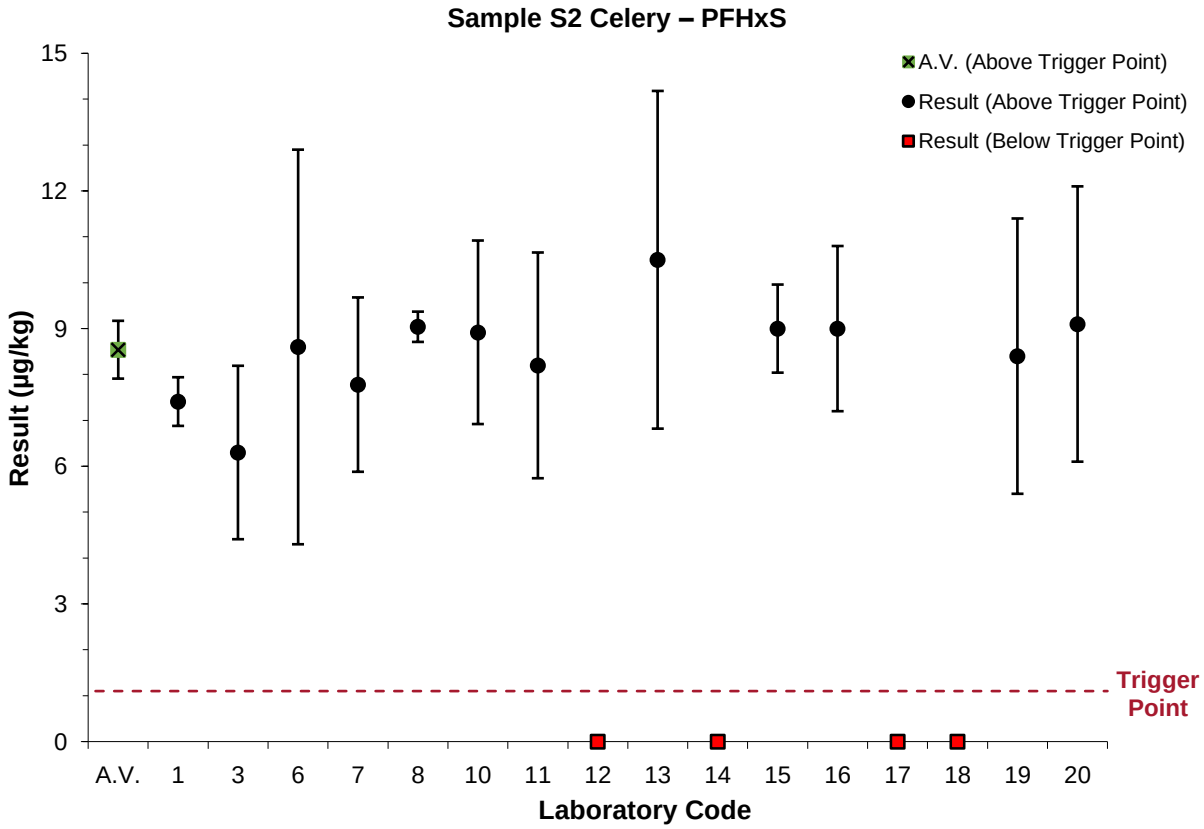
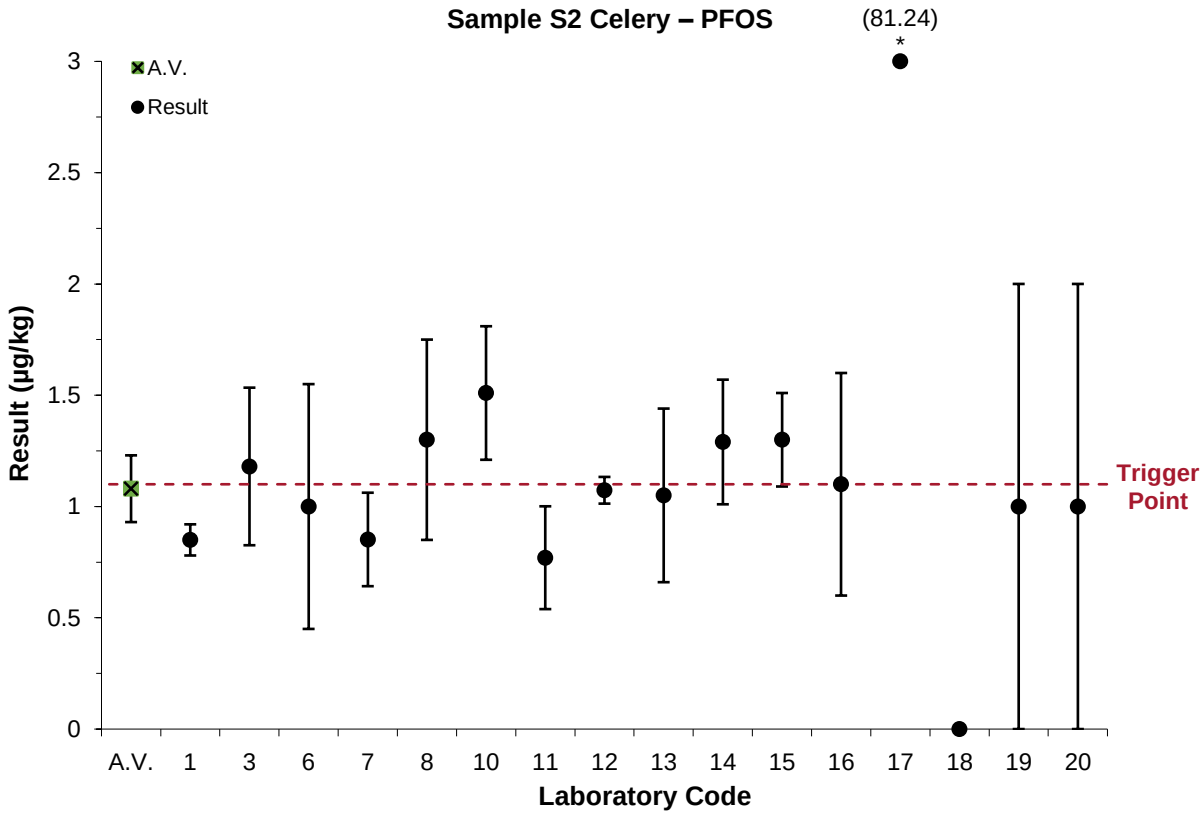
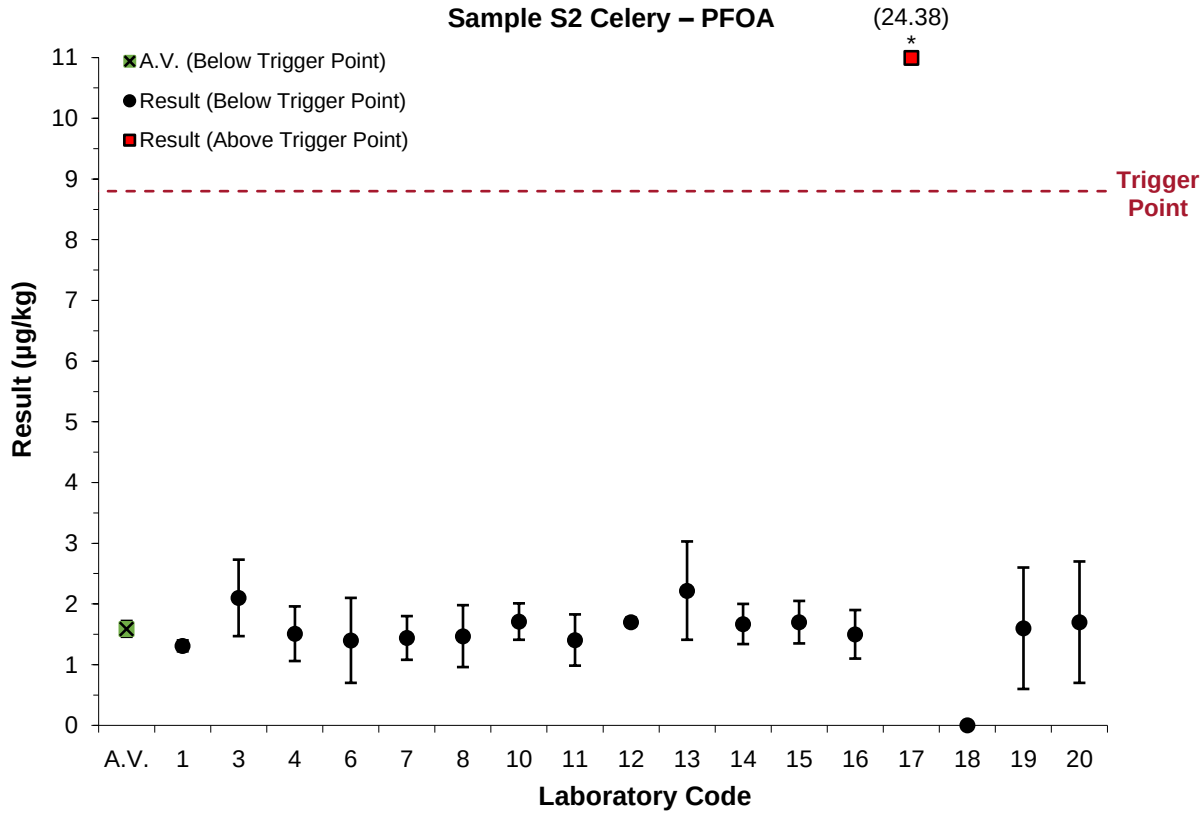


Figure 55 Sample S2 Celery PFHxS Assigned Value, Participant Results and Trigger Point



* Result from Laboratory 17 has been scaled to fit on the chart; original result in parentheses.

Figure 56 Sample S2 Celery PFOS Assigned Value, Participant Results and Trigger Point



* Result from Laboratory 17 has been scaled to fit on the chart; original result in parentheses.

Figure 57 Sample S2 Celery PFOA Assigned Value, Participant Results and Trigger Point

6.9 Participants' Methods

Participants were requested to analyse the samples using their normal test method and to report a single result as they would normally report to a client. Method descriptions provided by participants are presented in Appendix 3. A summary is presented below as technique (number):

- Pre-treatment
  - Sample S1: homogenisation (9), freeze-drying (2), pH adjustment (1), no pre-treatment (2)
  - Sample S2: homogenisation (10), freeze-drying (2), pH adjustment (1), no pre-treatment (2)
- Extraction Technique
  - Sample S1: alkaline digestion (6), QuEChERS (5), SLE (3), shaking / sonication (2)
  - Sample S2: alkaline digestion (4), QuEChERS (7), SLE (2), shaking (1), SPE (2)
- Extraction Solvent
  - Sample S1: acetonitrile (8), methanol/base (4), acetonitrile/acid(/water) (2)
  - Sample S2: acetonitrile (8), methanol/base (5), acetonitrile/acid(/water) (2)
- Extraction Temperature
  - Sample S1: room temperature (13)
  - Sample S2: room temperature (10), chilled then room temperature (1), heated and room temperature (1), heated (1)
- Extraction Time (total)
  - Sample S1: 1 min (1), 8 min (1), 20 min (1), 30 min (2), 1 h (5), 8 h (1), 16 h (1)
  - Sample S2: 8 min (1), 20 min (1), 30 min (3), 1 h (6), 8 h (1), 16 h (1)
- Clean-up
  - Sample S1: SPE / dSPE (carbon: 8, other / not specified: 9), centrifugation (2), LLE (1)
  - Sample S2: SPE / dSPE (carbon: 9, other / not specified: 9), centrifugation (1)
- Instrument
  - Sample S1: LC-MS/MS or LC-QQQ (13), LC-Orbitrap (1)
  - Sample S2: LC-MS/MS or LC-QQQ (12), LC-Orbitrap (2)
- Dilution
  - Sample S1: Yes (4), No (9)
  - Sample S2: Yes (4), No (7)
- Guard Column
  - Sample S1: Yes (9), No (3)
  - Sample S2: Yes (12), No (3)
- Delay Column
  - Sample S1: Yes (14)
  - Sample S2: Yes (15)
- Blank Correction
  - Sample S1: Yes (2), No (11)
  - Sample S2: Yes (3), No (11)
- Labelled Standard Source
  - Sample S1: Wellington Laboratories (13)
  - Sample S2: Wellington Laboratories (13)
- Recovery Correction
  - Sample S1: Yes (12), No (1)
  - Sample S2: Yes (12), No (2)

Laboratory **17** reported significantly higher results for the majority of analytes (except for GenX, where they reported a false negative). Numeric results reported for Sample S1 were greater than the assigned value by factors of 3.1 to 5.4, while the numeric results reported for Sample S2 were extremely varied, being greater than the assigned value by factors of 4.7 to 75. This participant reported using freeze-drying as a pre-treatment, and their results are likely based on the dry sample instead of on as received basis as requested for this study; another participant who also reported using freeze-drying returned mostly satisfactory z-scores. All results from Laboratory **17** were excluded from statistical calculations and subsequent methodology analysis.

Comparisons of z-scores with various extraction and analysis parameters are given in Figures 58 to 63. In general, no significant bias was identified for when more than one participant used a particular technique, and participants' results were compatible with each other. The most popular methodology for this study was homogenisation as pre-treatment, followed by QuEChERS extraction using acetonitrile and SPE clean-up, and then analysis on LC-MS/MS.

Sample S2 celery results from Laboratory **1** were generally satisfactory though biased low – this was the only participant who reported using freeze-drying as a pre-treatment and also used a 16 hour extraction time. This participant may need to review if their methodology introduced bias to their measurements.

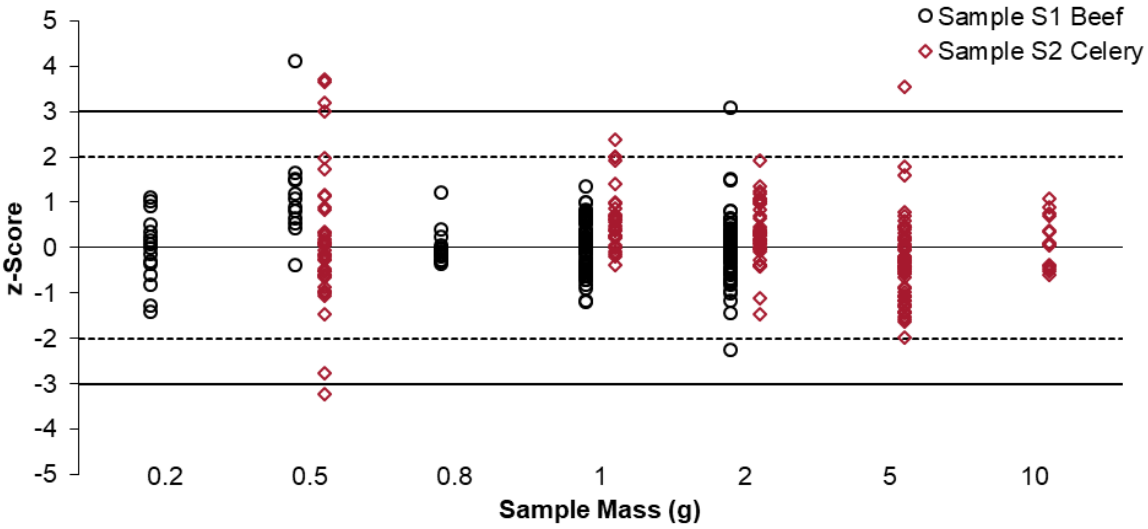


Figure 58 z-Score vs Sample Mass Used for Analysis

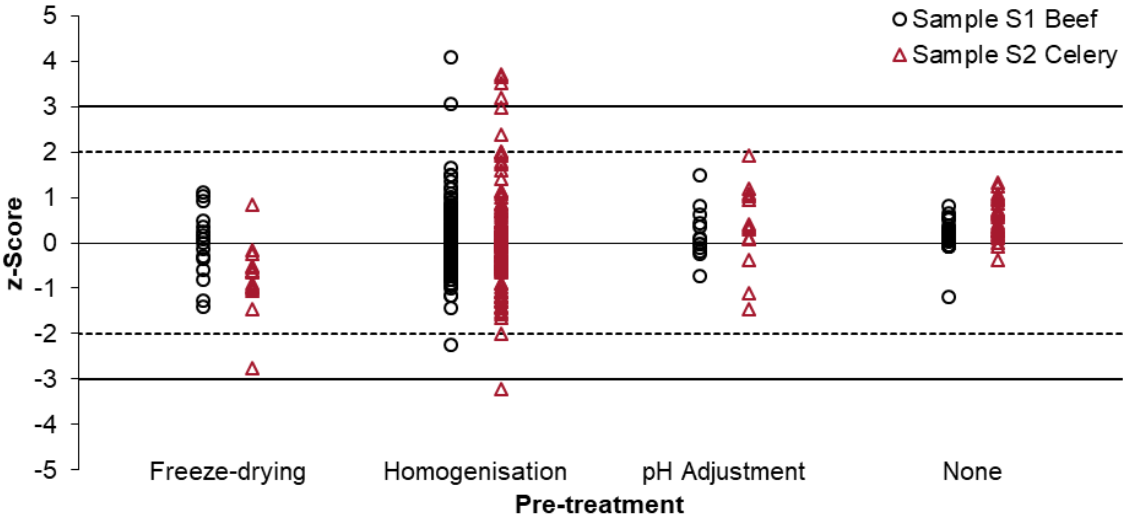


Figure 59 z-Score vs Pre-Treatment

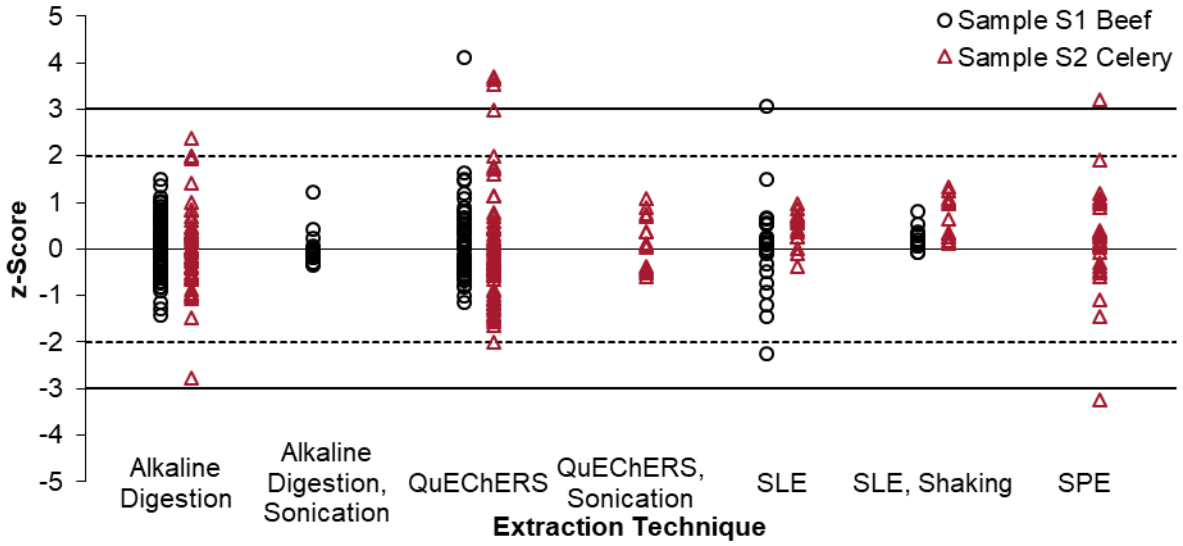


Figure 60 z-Score vs Extraction Technique

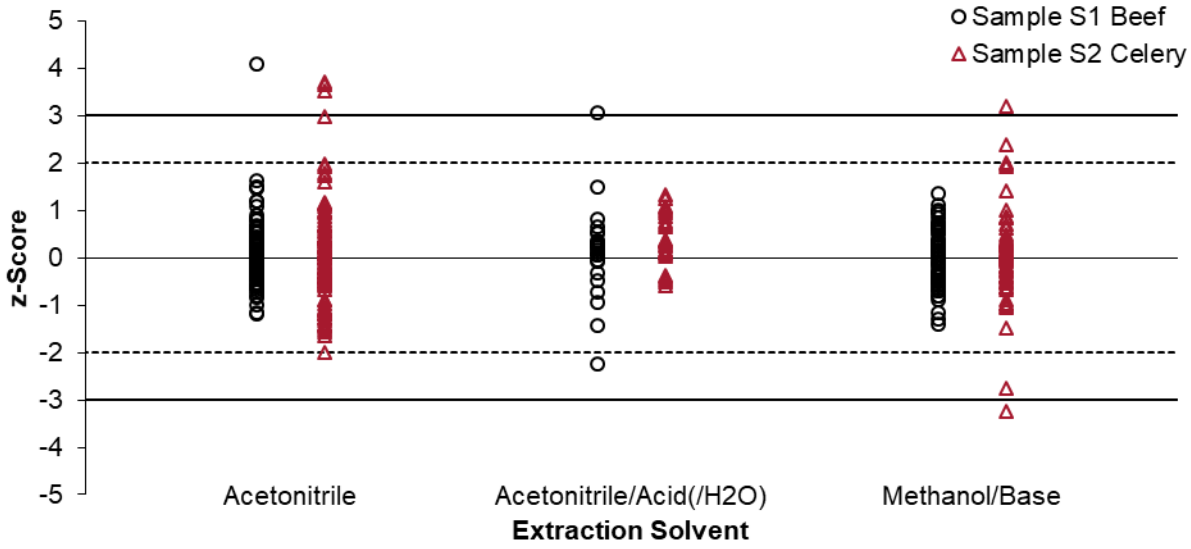


Figure 61 z-Score vs Extraction Solvent

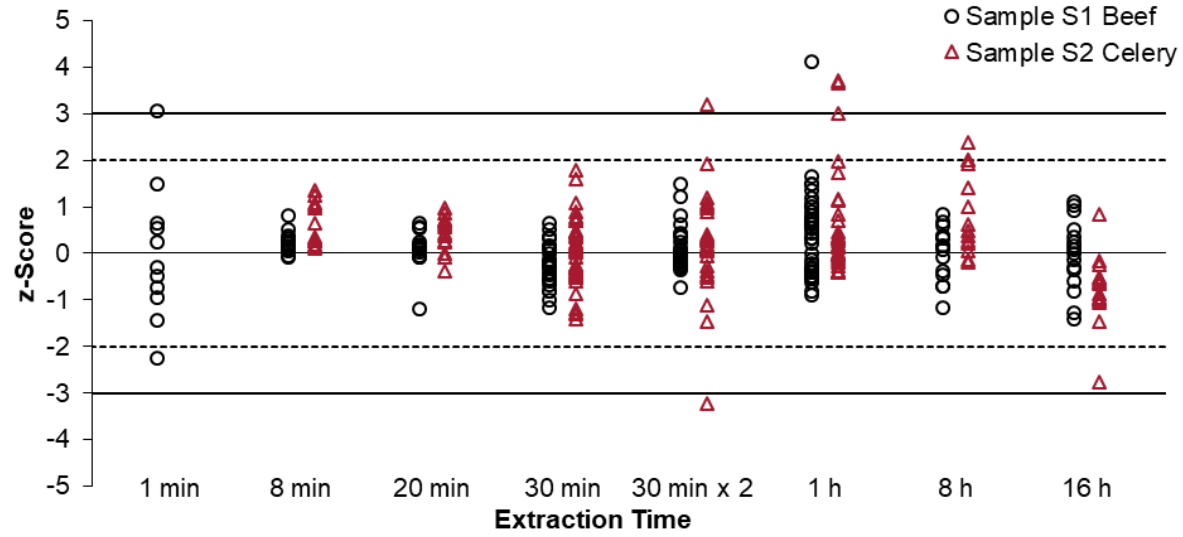


Figure 62 z-Score vs Extraction Time

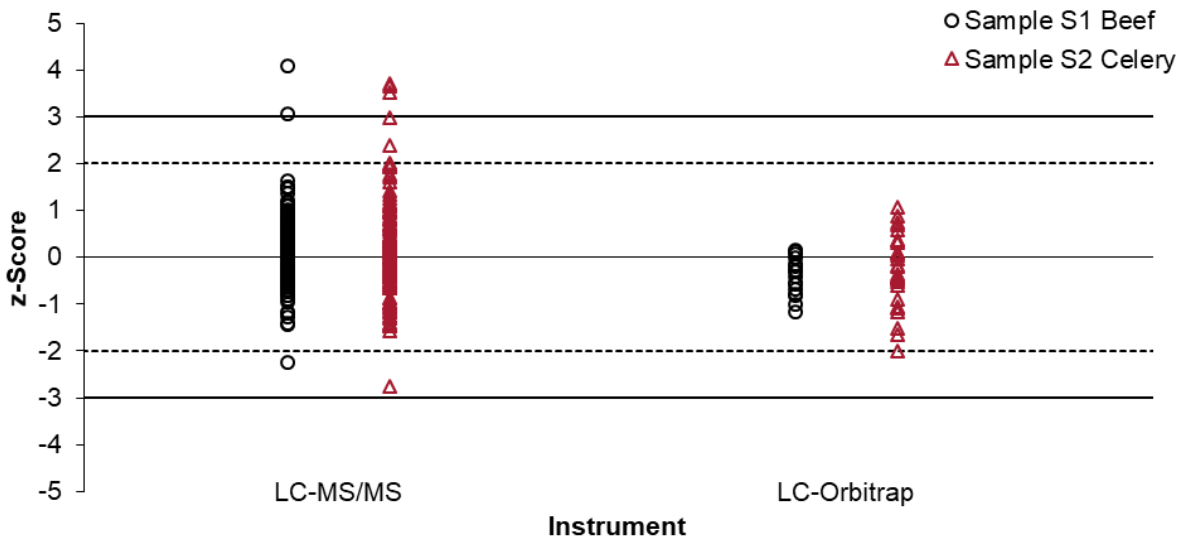


Figure 63 z-Score vs Measurement Instrument

6.10 Linear and Branched Isomers – PFHxS and PFOS

Participants were requested to report both the linear isomers only and the total (sum of linear and branched isomers) for PFHxS and PFOS. A summary of results reported by participants is presented in Table 46.

Table 46 Number of Participants Reporting Numeric PFHxS and PFOS Results

Sample	PFHxS			PFOS		
	Linear and Total	Linear Only	Total Only	Linear and Total	Linear Only	Total Only
S1	10	3	2	12	1	2
S2	11	4	1	14	1	1

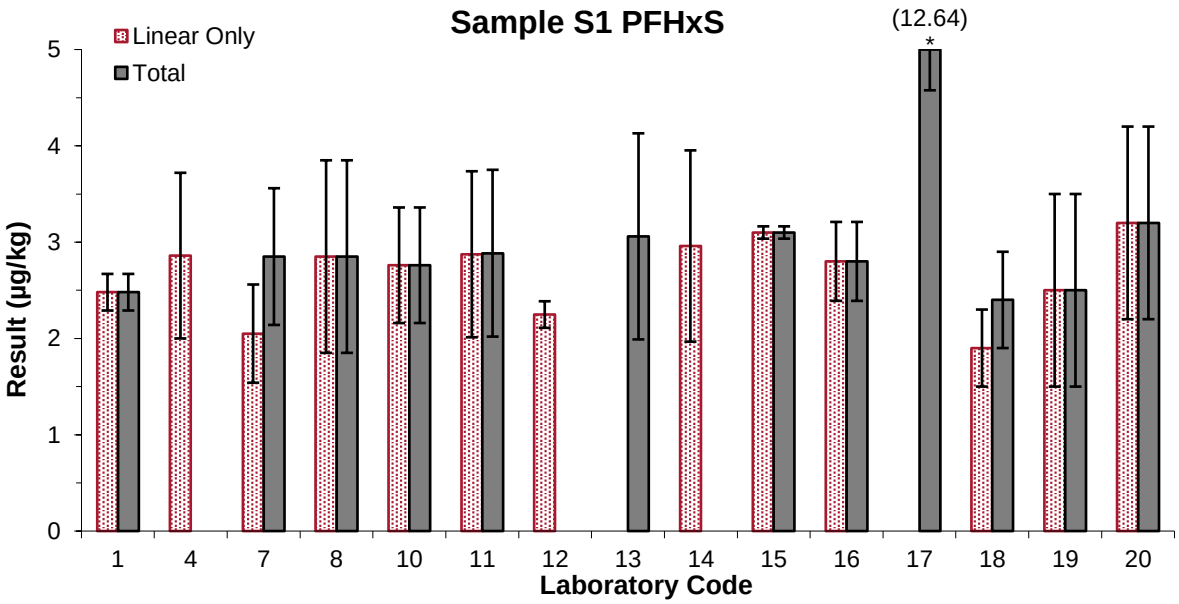
Most participants either reported results for linear only, total only, or both consistently across both samples. However, Laboratory **17** reported varied results; for PFHxS they reported only total in Sample S1 but only linear in Sample S2, while for PFOS they reported only total in Sample S1 but both linear and total in Sample S2.

For this study, both samples were only spiked with linear PFHxS and linear PFOS standards, and therefore the linear to total ratio was expected to be 100% for both samples.

PFHxS

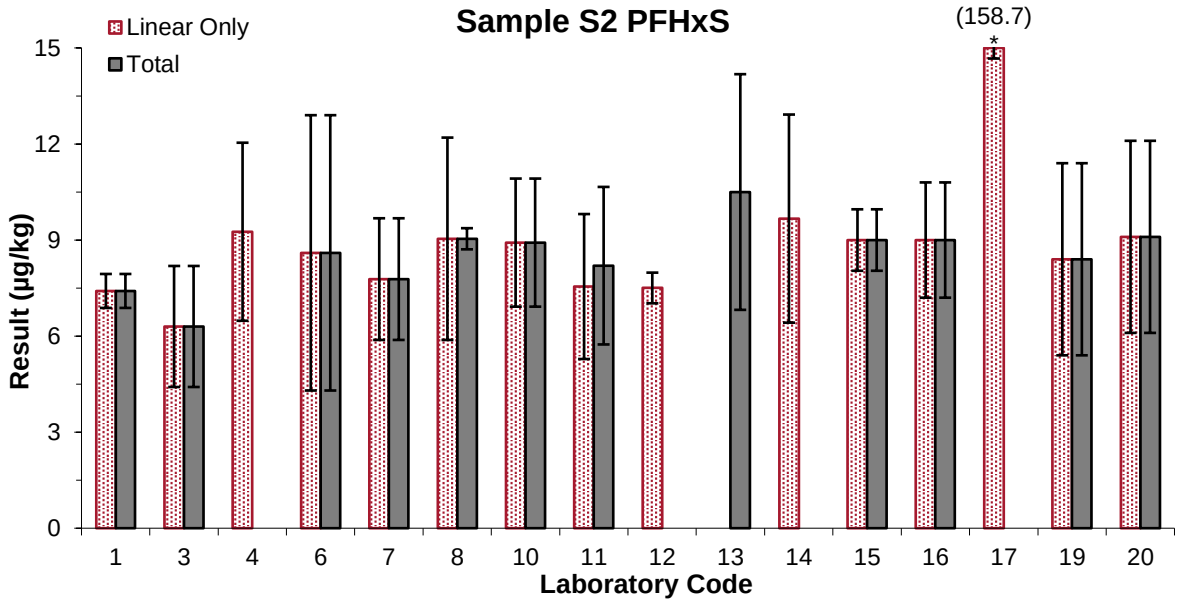
Summaries of participants' results for linear and total PFHxS in Samples S1 and S2 are presented in Figures 64 and 65.

For PFHxS, Laboratories **1**, **3** (analysed S2 only), **6** (analysed S2 only), **7**, **8**, **10**, **11**, **15**, **16**, **18** (numeric results for S1 only), **19** and **20** reported both linear and total values. Of these, all sets of results were in agreement with each other within their respective uncertainties, and the majority also correctly reported the same result for linear and total. Laboratory **8** reported the same values for linear and total in both samples, though in Sample S2 their uncertainty for PFHxS total was significantly smaller than for linear only. Laboratory **11** reported very slightly different values for linear and total in Sample S1, and slightly lower linear values for Sample S2 (92% of total). The Sample S1 linear results from Laboratories **7** and **18** were relatively low (72% and 79% of total respectively).



* Laboratory 17 result and uncertainty have been scaled to fit on the chart; original result in parentheses.

Figure 64 Participant Results for Sample S1 PFHxS (linear and total)



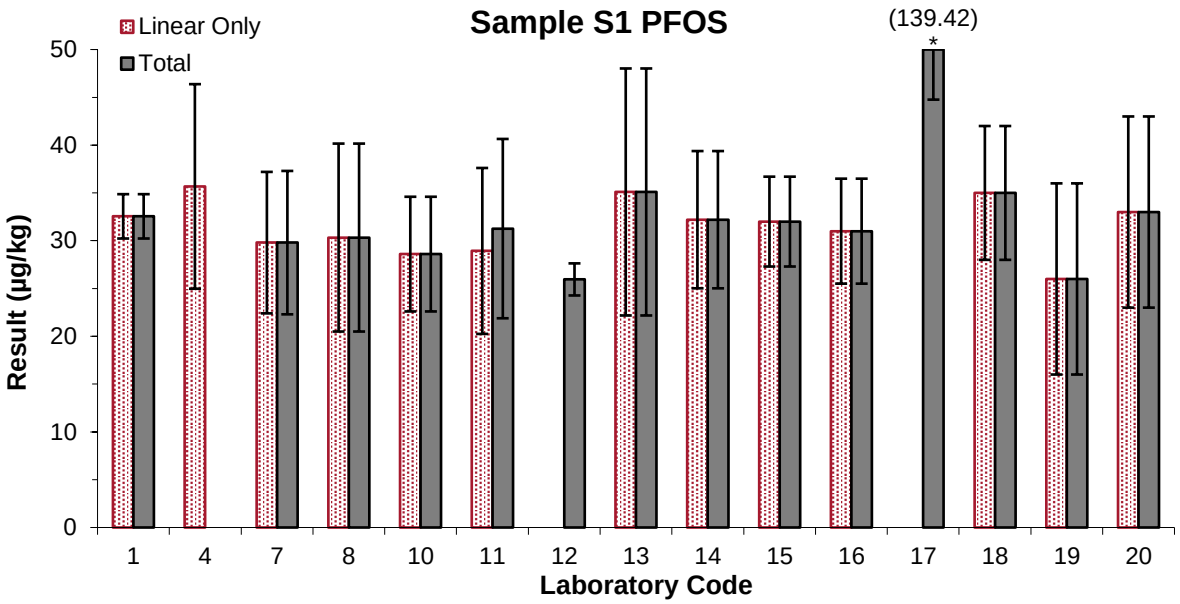
* Laboratory 17 result and uncertainty have been scaled to fit on the chart; original result in parentheses.

Figure 65 Participant Results for Sample S2 PFHxS (linear and total)

PFOS

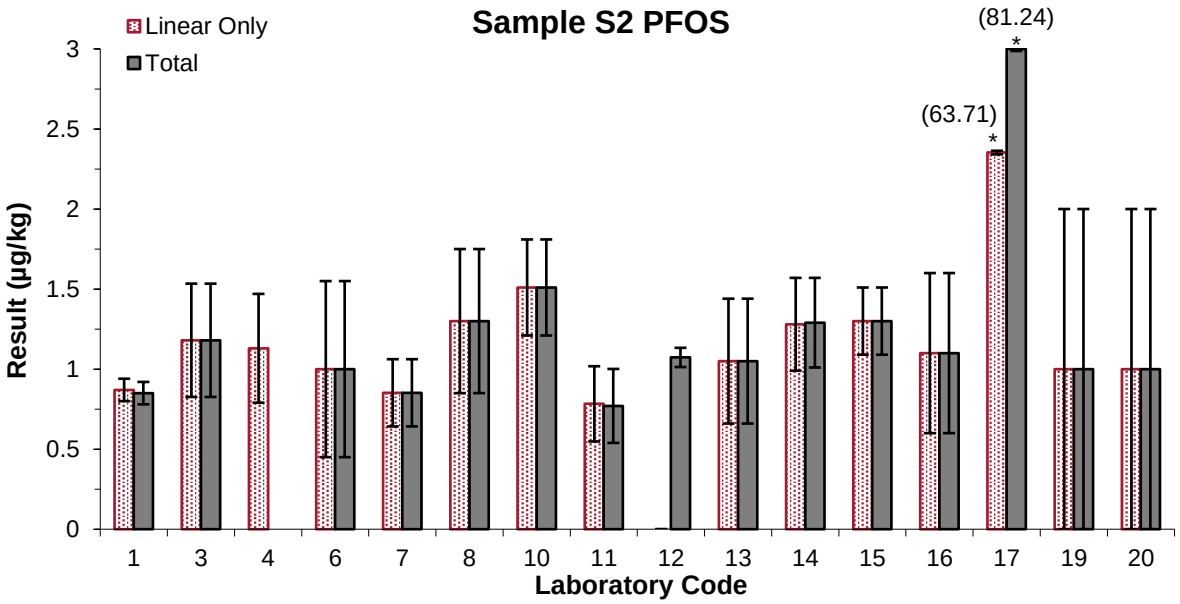
Summaries of participants' results for linear and total PFOS in Samples S1 and S2 are presented in Figures 66 and 67.

For PFOS, Laboratories **1**, **3** (analysed S2 only), **6** (analysed S2 only), **7**, **8**, **10**, **11**, **13**, **14**, **15**, **16**, **17** (for S2 only), **18** (numeric results for S1 only), **19** and **20** reported both linear and total values. Of these, all except one set of results were in agreement with each other within their respective uncertainties, and the majority also correctly reported the same result for linear isomers and total. Laboratory **17** Sample S2 results were not in agreement as their linear to total ratio was 78% and their uncertainties reported were extremely small (less than 0.5% relative uncertainty for both). Laboratories **11** and **14** reported slightly lower linear values in Sample S1 (93% of total) and Sample S2 (99% of total) respectively. Laboratories **1** and **11** reported linear values slightly higher than their total values in Sample S2 (both 102% of total).



* Laboratory 17 result and uncertainty have been scaled to fit on the chart; original result in parentheses.

Figure 66 Participant Results for Sample S1 PFOS (linear and total)



* Laboratory 17 results and uncertainties have been scaled to fit on the chart; original results in parentheses.

Figure 67 Participant Results for Sample S2 PFOS (linear and total)

6.11 Effects of Sample Matrix

The samples in this study were beef meat (Sample S1) and celery (Sample S2). A summary of the results reported and z-scores obtained by matrix is presented in Table 47.

Participants overall performed better with the beef meat matrix, with a higher proportion of numeric results reported and a higher proportion of satisfactory z-scores.

Table 47 Result Comparison by Matrix

Sample	Matrix	Expected Number of Results	Numeric Results Reported	z-Scores Calculated	Satisfactory z-Scores
S1	Beef Meat (spiked)	255	212 (83%)	207	190 (92%)
S2	Celery (spiked)	289	225 (78%)	215	193 (90%)

6.12 Summary of Participants' Results and Performances

Summaries of participants' results and performances for scored analytes in this PT study are presented in Tables 48 and 49, and Figure 68.

Table 48 Summary of Participants' Sample S1 Results*

Lab. Code	PFBS	PFHxS	PFHxS (linear)	PFHpS	PFOS	PFOS (linear)	PFDS	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	8:2 FTS
A.V.	2.18	2.81	2.67	1.60	31.0	31.6	17.7	12.2	0.739	0.64	1.92	33.4	3.91	4.14	0.318	7.7
H.V.	2.26	2.76	2.76	1.74	30.5	30.5	17.3	12.6	0.79	0.69	1.98	29.8	4.0	4.6	0.398	N/A
S.V.	2.90	3.68	3.68	1.92	37.2	37.2	23.1	17.5	0.970	0.677	1.95	38.6	4.36	4.35	0.387	9.26
1	2.67	2.48	2.48	1.34	32.55	32.55	13.16	14.47	0.72	0.77	1.81	33.85	4.18	4.14	0.35	5.53
3	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
4	2.08	NT	2.86	1.65	NT	35.68	16.89	11.80	0.65	0.68	1.83	36.78	3.69	3.77	0.34	7.23
6	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
7	2.12	2.85	2.05	1.28	29.8	29.8	17.7	10.2	0.653	0.553	1.8	30.6	4.01	3.69	0.267	7.95
8	2.23	2.85	2.85	2.08	30.33	30.33	19.27	11.58	0.83	NR	1.91	28.53	3.89	4.81	NR	NT
10	2.33	2.76	2.76	1.79	28.6	28.6	15.2	12.4	0.794	0.681	1.98	28.7	4.44	4.43	0.372	5.91
11	2.233	2.885	2.874	1.404	31.266	28.93	<0.5	14.166	0.855	0.727	1.994	35.891	3.641	3.770	<0.5	6.738
12	1.8	NR	2.248	1.306	25.952	NR	NT	10.125	0.65	0.502	1.689	34.215	3.284	3.999	0.271	NT
13	2.01	3.06	NT	1.86	35.1	35.1	21.9	14.4	<2	<1	2.49	43.4	5.19	5.04	<1	14.01
14	2.43	NT	2.96	1.68	32.2	32.2	13.5	12.0	0.837	0.643	1.98	32.8	4.01	4.10	0.325	NT
15	2.3	3.1	3.1	1.7	32	32	19	12	< 1.0	< 1.0	2.0	33	4.0	4.2	< 1.0	8.2
16	2.1	2.8	2.8	1.6	31	31	22	12	0.8	0.6	1.8	31	3.8	4.0	0.3	7.8
17	10.84	12.64	NR	6.32	139.42	NR	55.25	50.96	4.02	2.54	7.71	122.23	17.53	15.96	1.21	36.17
18	1.2	2.4	1.9	NT	35	35	NT	11	0.6	0.6	3.1	35	NT	NT	NT	10
19	2	2.5	2.5	1.4	26	26	16	11	<2	<1	1.8	32	3.5	3.8	<2	7.5
20	2.5	3.2	3.2	1.8	33	33	20	14	<2	<1	2.3	37	3.9	4.4	<2	9.8

* A.V. = Assigned Value; H.V. = Homogeneity Value; S.V. = Spiked Value. All values are in µg/kg. Shaded cells are results which returned a questionable or unsatisfactory z-score.

Table 49 Summary of Participants' Sample S2 Results*

Lab. Code	PFBS	PFPeS	PFHxS	PFHxS (linear)	PFHpS	PFOS	PFOS (linear)	PFDS	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFOSA	MeFOSE
A.V.	1.37	11.4	8.54	8.45	0.88	1.08	1.09	3.39	2.51	0.68	16.5	0.746	1.59	0.78	2.39	3.04
S.V.	1.50	11.3	9.45	9.45	1.10	1.43	1.43	4.79	2.48	0.746	15.0	0.795	1.81	0.990	3.53	4.00
1	1.6	10	7.41	7.41	0.85	0.85	0.87	2.39	2.39	0.61	14.74	0.6	1.31	0.63	1.07	NT
3	1.24	11.6	6.3	6.3	0.823	1.18	1.18	3.86	2.52	0.743	19.1	0.779	2.1	0.561	3.24	3.13
4	1.35	10.45	NT	9.26	0.96	NT	1.13	3.04	2.36	0.56	15.58	0.57	1.51	0.73	2.50	2.87
6	1.4	13	8.6	8.6	0.8	1.0	1.0	NT	2.7	0.80	19	0.80	1.4	0.70	2.9	NT
7	1.26	12.1	7.78	7.78	0.592	0.852	0.852	2.78	2.8	0.475	16.4	0.719	1.44	0.469	1.83	2.91
8	0.97	NT	9.04	9.04	0.95	1.3	1.3	4.13	1.96	0.84	16.83	0.76	1.47	1.08	NT	NT
10	1.54	11.5	8.92	8.92	1.3	1.51	1.51	3.26	<5	0.746	16	0.895	1.71	1	3.79	NT
11	1.263	8.713	8.199	7.548	0.658	0.770	0.783	2.666	4.283	0.547	14.632	0.750	1.406	<0.2	1.644	2.64
12	1.148	9.682	NR	7.503	0.853	1.073	NR	NT	2.095	0.623	15.033	0.708	1.703	0.773	NT	NT
13	1.68	18.21	10.5	NT	<1	1.05	1.05	3.48	<5	<2	28.74	1.29	2.22	1.05	<5	<5
14	1.37	12.0	NR	9.67	0.999	1.29	1.28	3.81	2.72	0.777	16.2	0.801	1.67	0.867	2.21	NT
15	1.4	12	9.0	9.0	1.1	1.3	1.3	4.3	2.6	< 1.0	17	< 1.0	1.7	< 1.0	2.7	3.7
16	1.4	18.7	9	9	0.8	1.1	1.1	1.2	2.6	0.8	16.3	0.8	1.5	0.7	2.1	3.1
17	33.52	105.29	NR	158.7	6.32	81.24	63.71	16.79	40.55	17.15	346.53	12.87	24.38	14.19	11.21	NT
18	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
19	1.3	13	8.4	8.4	<1	1	1	3.6	2.5	<2	17	<1	1.6	<1	<5	<10
20	1.6	12	9.1	9.1	<1	1.0	1.0	3.4	2.6	<2	18	<1	1.7	<1	<5	<10

* A.V. = Assigned Value; S.V. = Spiked Value. All values are in µg/kg. Shaded cells are results which returned a questionable or unsatisfactory z-score.

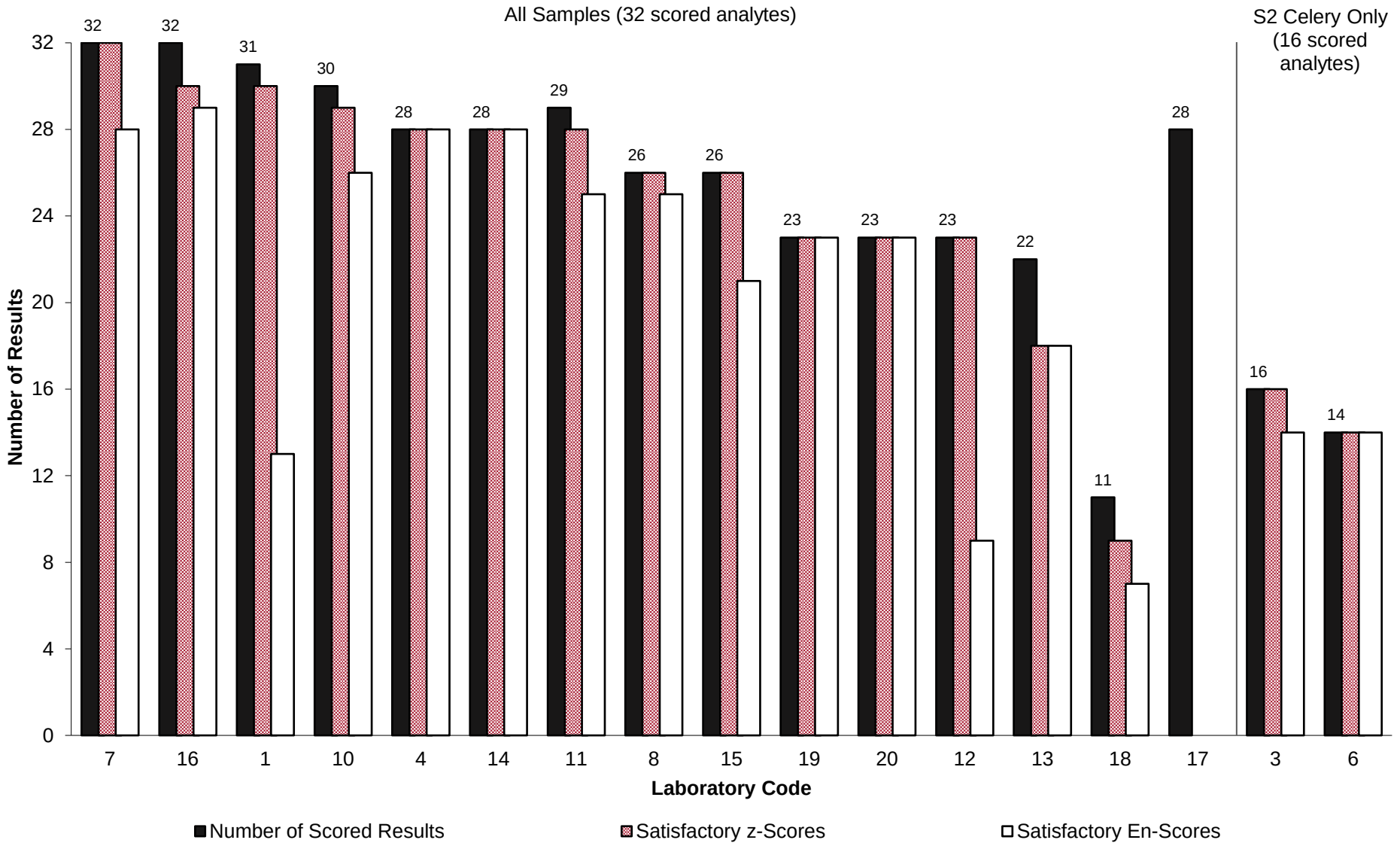


Figure 68 Summary of Participants' Performance

6.13 Comparison with Previous PFAS in Food Studies

NMI has coordinated PFAS in Food PT studies since 2016. A summary of participation and reported results rates over the last 6 studies (2016 to 2021) is presented in Figure 69. Proportions of PFAS analysed and numeric results reported have remained relatively high over this period, despite the increased number of spiked analytes as compared to the original studies.

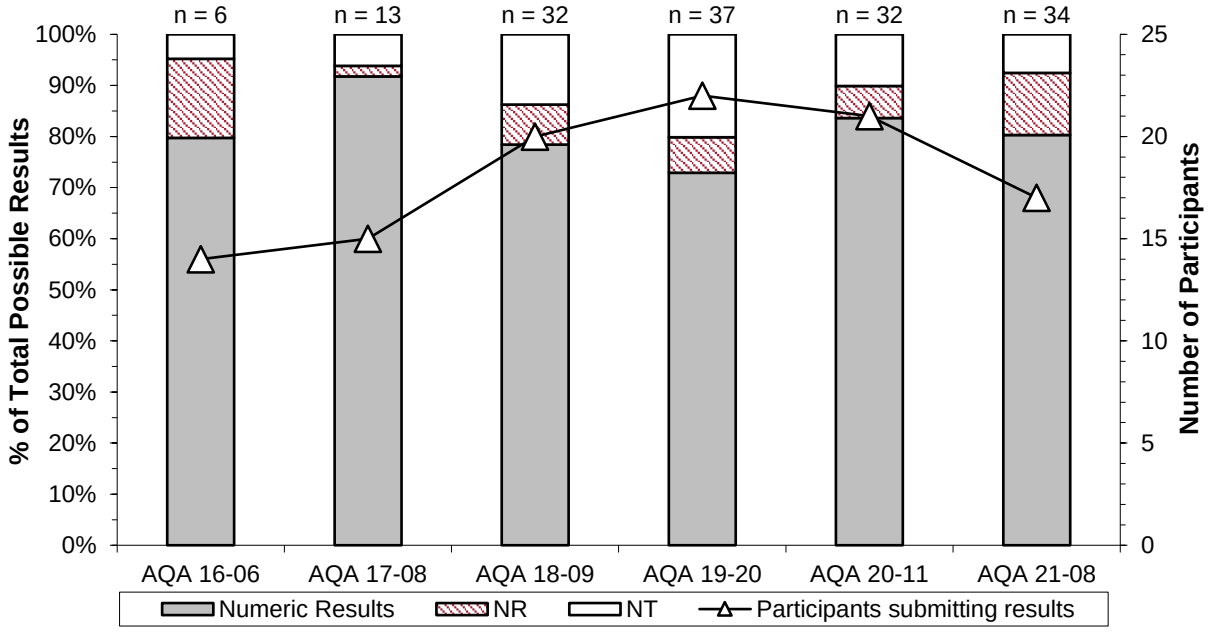


Figure 69 Summary of Participation and Reported Results in PFAS in Food PT Studies (n = number of spiked analytes).

A summary of the satisfactory performance (presented as a percentage of the total number of scores for each study) in PFAS in Food PT studies over the last 6 studies (2016 to 2021) is presented in Figure 70. The target SD used to calculate z-scores has been kept constant at 20% PCV which enables comparison between different studies. Proportions of satisfactory scores has remained relatively high, with the average proportion of satisfactory scores over this period being 89% for z-scores and 77% for E_n-scores.

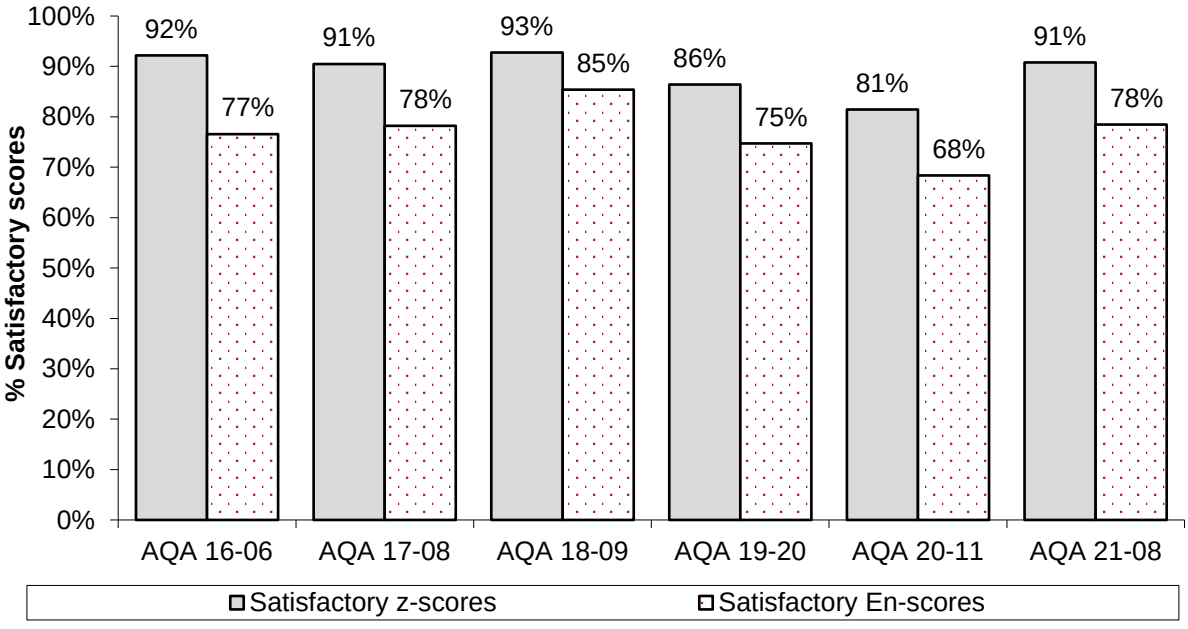


Figure 70 Summary of Participants' Performance for PFAS in Food PT Studies

The number of analytes assessed in each study has increased significantly as compared to the initial PFAS in Food study, and the studies have increased in size and complexity. As a point of comparison, PFOS and PFOA have been assessed in every study, and a summary of the proportion of satisfactory scores for these analytes over the last 6 studies is presented in Figure 71.

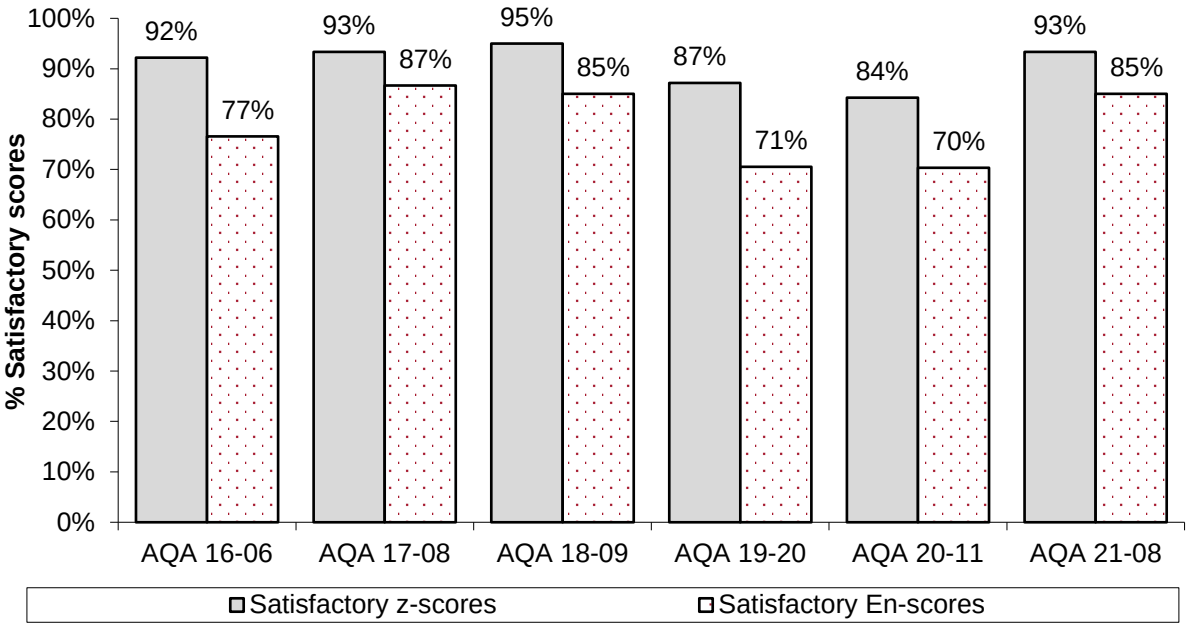


Figure 71 Summary of Participants' Performance for PFOS and PFOA in Food PT Studies

Individual performance history reports are emailed to participants at the end of each PT study; the consideration of z-scores over time provides much more useful information than a single z-score. Over time, laboratories should expect at least 95% of their z-scores to lie within the range $|z| \leq 2.0$. Scores in the range $2.0 < |z| < 3.0$ can occasionally occur, however these should be interpreted in conjunction with the other scores obtained by that laboratory. For example, a trend of z-scores on one side of the zero line is an indication of method or laboratory bias.

7 REFERENCES

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APPENDIX 1 – SAMPLE PREPARATION

PFAS standards used for spiking samples were bought from Toronto Research Chemicals, HPC Standards GmbH and Wellington Laboratories Canada.

Sample S1: Three 500 g packs of Extra Lean Mince were bought from a local supermarket. The mince was blended to yield a puree. The pureed mince was placed in a tray and sprayed with a spiking solution containing PFAS analytes in methanol. The mince was thoroughly mixed, before being divided into patties of no more than 6 cm in diameter, placed on a tray, covered, and placed into the freezer overnight at -80 °C. The frozen patties were then ground using a Retsch SM2000 Knife Mill which was kept cold using liquid nitrogen and dry ice. The dry ice was then allowed to sublime off, before 5 g portions of the spiked mince were packed into sample tubes. The tubes were labelled, shrink-wrapped, and then stored at -80 °C prior to dispatch.

Sample S2: Organic celery was bought from a Sydney organic fruit and vegetable wholesaler. The celery were rinsed, cut, blended, and then passed through an 850 µm sieve. The celery was spiked with PFAS analytes and then stirred for at least 2 hours, before 40 mL portions were dispensed into sample tubes. The tubes were labelled, shrink-wrapped, and then stored at -20 °C prior to dispatch.

APPENDIX 2 – HOMOGENEITY AND STABILITY OF TEST MATERIALS

A2.1 Homogeneity and Stability Testing

No homogeneity or stability testing was conducted on Sample S2 celery, which was prepared and packaged using a process previously demonstrated to produce suitable samples.

As beef meat was a new matrix for PFAS analytes introduced in this study, homogeneity and stability testing was performed on Sample S1. Samples were analysed at NMI North Ryde. Samples were prepared in duplicate by accurately weighing 1 g of the sample then spiking with 25 µL of labelled internal standard in methanol. The samples were extracted by overnight tumbling in alkaline methanol (0.01 N potassium hydroxide), then centrifuged and a portion was purified by passing through activated carbon (SUPLCLEAN ENVI-CARB, 500 mg, 120-400 Mesh) eluted using methanol. After evaporation under nitrogen, the extract was reconstituted to 600 µL in mobile phase and spiked with 20 µL labelled recovery standard in methanol. All chemicals were analytical reagents or LCMS grade solvents. Instrument analysis was performed using an Ultra Performance Liquid Chromatography (UPLC) coupled with a Liquid Chromatography Qtrap Mass Spectrometer (ABSciex 6500+), operating in multiple reaction monitoring mode. 2 µL of extract was injected onto a Waters Acquity BEH C18 column (2.1 mm x 100 mm x 1.7 µm, 130 Å) with a mobile phase gradient consisting of water:methanol (2 mM ammonium acetate). Two mass transitions were monitored for each target analyte and labelled internal standard, and abundance ratios checked. The instrument mass accuracy was calibrated annually during preventative maintenance, and the six point calibration curve established for each analytical batch. A solvent batch blank was extracted and analysed with each batch, and sample results were reported if results were at least three times the level of any analyte detected in the batch blank. Quantification was based on the use of the labelled internal standards using relative retention factors from the multipoint calibration, and was corrected for internal standard recoveries. The analysis was based on USEPA Method 537 and used calibration, internal and recovery standards supplied by Wellington Laboratories.

Homogeneity checks were based on that described by Thompson and Fearn,[11] which is also the procedure as described in the International Harmonized Protocol.[4] Measurements were made under repeatability conditions in random order. The mean result of each analyte was used as the NMI homogeneity value. Results of the Sample S1 homogeneity testing are presented in Tables 50 to 64. Samples were found to be sufficiently homogeneous for use in this PT study with a PCV of 20%.

Table 50 Sample S1 PFBS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	2.20	2.31
12	2.27	2.31
21	2.18	2.15
36	2.35	2.33
41	2.32	2.33
46*	2.38	1.99
47	2.30	2.23
Mean	2.26	
CV	4.6%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.62	0.78	**Pass**
S_{an}/σ	0.09	0.5	**Pass**
s^2_{sam}	0.003	0.04	**Pass**

* Results from container 46 were not included in the test for homogeneity, being identified as Cochran outliers due to the difference between replicates.[11]

Table 51 Sample S1 PFHxS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	2.83	3.01
12	2.77	2.69
21	2.61	2.60
36	2.74	2.87
41	2.81	2.58
46	2.81	2.66
47	2.93	2.77
Mean	2.76	
CV	4.6%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.35	0.73	**Pass**
S_{an}/σ	0.19	0.5	**Pass**
s^2_{sam}	0.005	0.07	**Pass**

Table 52 Sample S1 PFHxS (linear) Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	2.83	3.01
12	2.77	2.69
21	2.61	2.60
36	2.74	2.87
41	2.81	2.58
46	2.81	2.66
47	2.93	2.77
Mean	2.76	
CV	4.6%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.35	0.73	**Pass**
S_{an}/σ	0.19	0.5	**Pass**
s^2_{sam}	0.005	0.07	**Pass**

Table 53 Sample S1 PFHpS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	1.91	1.85
12	1.78	1.77
21	1.94	1.60
36	1.63	1.81
41	1.72	1.93
46	1.83	1.52
47	1.68	1.40
Mean	1.74	
CV	9.1%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.31	0.73	**Pass**
S_{an}/σ	0.46	0.5	**Pass**
s^2_{sam}	0.000	0.06	**Pass**

Table 54 Sample S1 PFOS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	32.0	33.3
12	29.5	28.5
21	31.9	30.6
36	29.7	34.4
41	28.4	27.7
46	29.2	32.3
47	31.9	27.3
Mean	30.5	
CV	7.2%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.38	0.73	**Pass**
S_{an}/σ	0.33	0.5	**Pass**
s^2_{sam}	0.707	12.97	**Pass**

Table 55 Sample S1 PFOS (linear) Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	32.0	33.3
12	29.5	28.5
21	31.9	30.6
36	29.7	34.4
41	28.4	27.7
46	29.2	32.3
47	31.9	27.3
Mean	30.5	
CV	7.2%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.38	0.73	**Pass**
S_{an}/σ	0.33	0.5	**Pass**
s^2_{sam}	0.707	12.97	**Pass**

Table 56 Sample S1 PFDS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	18.8	17.9
12	16.9	20.1
21	17.4	17.8
36	16.2	17.5
41	14.5	18.3
46	15.8	18.2
47	17.5	15.1
Mean	17.3	
CV	8.6%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.36	0.73	**Pass**
S_{an}/σ	0.48	0.5	**Pass**
s^2_{sam}	0.000	6.23	**Pass**

Table 57 Sample S1 PFBA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	12.1	13.2
12	11.9	12.6
21	12.7	12.5
36	12.8	12.2
41	12.3	13.6
46	13.1	13.6
47	12.2	12.3
Mean	12.6	
CV	4.3%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.40	0.73	**Pass**
S_{an}/σ	0.21	0.5	**Pass**
s^2_{sam}	0.024	1.61	**Pass**

Table 58 Sample S1 PFPeA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	0.78	0.75
12	0.79	0.84
21	0.80	0.76
36	0.84	0.78
41	0.80	0.82
46	0.81	0.78
47	0.77	0.81
Mean	0.79	
CV	3.3%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.30	0.73	**Pass**
S_{an}/σ	0.17	0.5	**Pass**
s^2_{sam}	0.000	0.01	**Pass**

Table 59 Sample S1 PFHxA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	0.65	0.69
12	0.67	0.74
21	0.76	0.65
36	0.75	0.72
41	0.68	0.72
46	0.65	0.63
47	0.71	0.69
Mean	0.69	
CV	5.9%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.54	0.73	**Pass**
S_{an}/σ	0.28	0.5	**Pass**
s^2_{sam}	0.000	0.01	**Pass**

Table 60 Sample S1 PFHpA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	2.02	1.82
12	1.85	2.20
21	2.05	1.81
36	1.95	1.97
41	2.25	1.93
46	1.95	1.91
47	2.00	2.05
Mean	1.98	
CV	6.4%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.37	0.73	**Pass**
S_{an}/σ	0.38	0.5	**Pass**
s^2_{sam}	0.000	0.06	**Pass**

Table 61 Sample S1 PFOA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	28.8	29.4
12	30.7	29.6
21	27.9	28.4
36	31.1	29.5
41	31.2	30.3
46	30.8	29.4
47	30.2	29.8
Mean	29.8	
CV	3.4%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.37	0.73	**Pass**
S_{an}/σ	0.12	0.5	**Pass**
s^2_{sam}	0.513	7.46	**Pass**

Table 62 Sample S1 PFNA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	3.9	3.7
12	4.3	4.2
21	3.9	3.6
36	3.9	3.9
41	4.0	4.4
46	4.1	3.8
47	4.1	4.1
Mean	4.0	
CV	5.4%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.47	0.73	**Pass**
S_{an}/σ	0.23	0.5	**Pass**
s^2_{sam}	0.013	0.17	**Pass**

Table 63 Sample S1 PFDA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	4.7	4.3
12	5.1	4.6
21	4.7	4.2
36	4.5	4.5
41	4.6	4.6
46	4.6	4.3
47	4.9	4.4
Mean	4.6	
CV	5.7%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.28	0.73	**Pass**
S_{an}/σ	0.32	0.5	**Pass**
s^2_{sam}	0.000	0.28	**Pass**

Table 64 Sample S1 PFUdA Homogeneity Testing

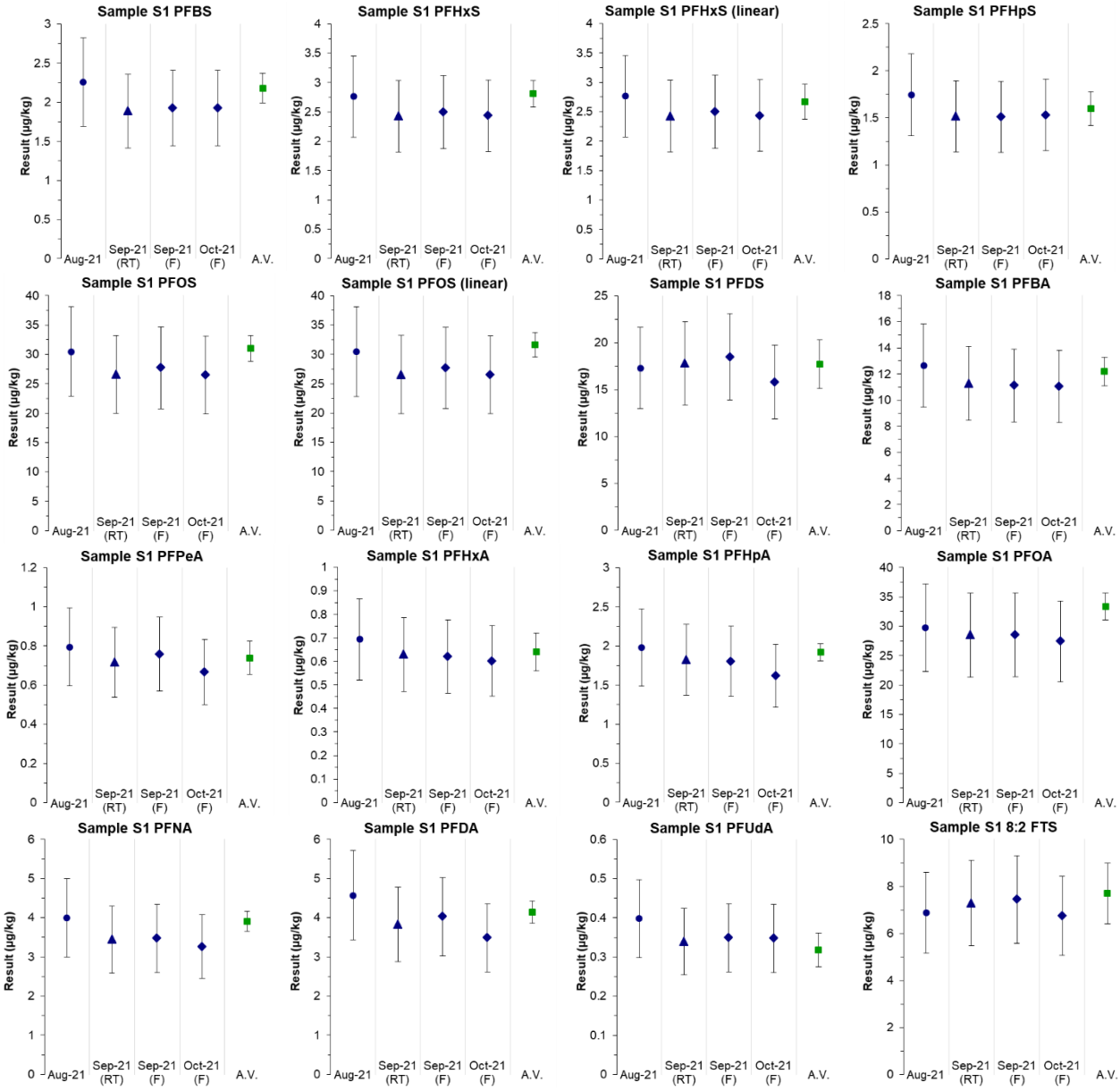
Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	0.359	0.438
12	0.386	0.428
21	0.371	0.368
36	0.473	0.426
41	0.359	0.401
46	0.417	0.388
47	0.424	0.331
Mean	0.398	
CV	9.7%	

Thompson and Fearn Homogeneity Tests[11]

Test	Value	Critical	Result
Cochran	0.40	0.73	**Pass**
S_{an}/σ	0.49	0.5	**Pass**
s^2_{sam}	0.000	0.00	**Pass**

The beef meat samples were analysed at an initial time point in August 2021 (approximately the sample dispatch date). Samples were then analysed after being stored at both ambient room temperature (around 1 month, to reflect transportation stability) and freezer temperature (around 1 and 2.5 months, to reflect storage stability at a participant's laboratory).

Results were in good agreement with each other and the assigned value within their respective uncertainties (Figure 72). The samples were also shown to be adequately stable when assessed against the criteria specified in ISO 13528:2015.[5]



RT = Room Temperature; F = Freezer Temperature

Figure 72 Stability Results and Assigned Value (A.V.) for Sample S1

A2.2 Comparison of Results and Bottle Numbers

Comparisons of z-scores obtained to the bottle number analysed for all scored analytes in both samples are presented for information in Figure 73 (only results with known bottle numbers have been included as some participants were sent multiple samples and bottle number used was not requested; gross errors have been removed).

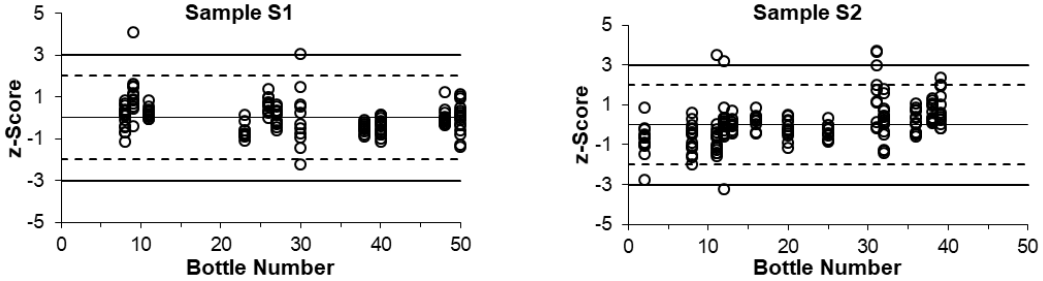
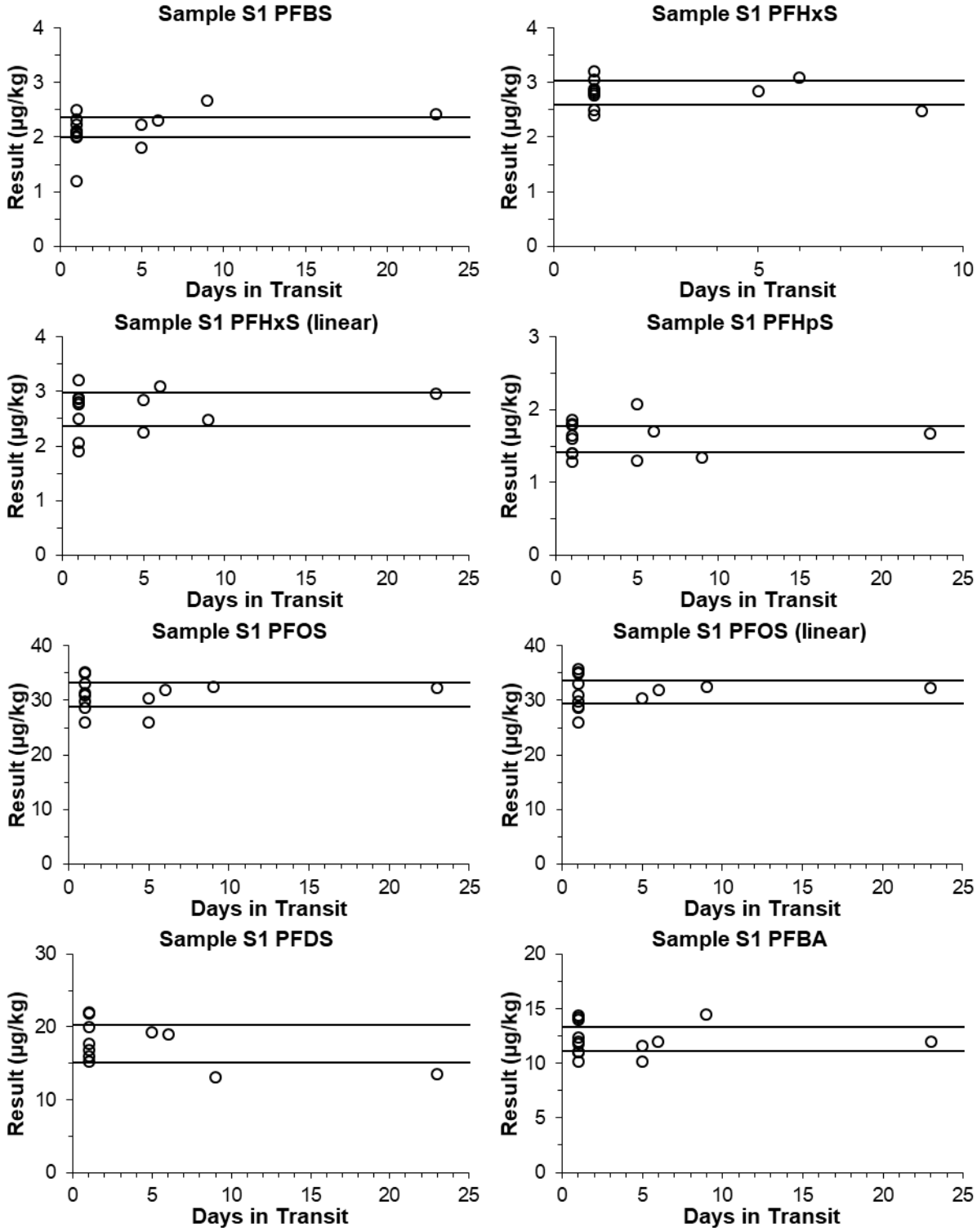


Figure 73 z-Scores vs Bottle Number

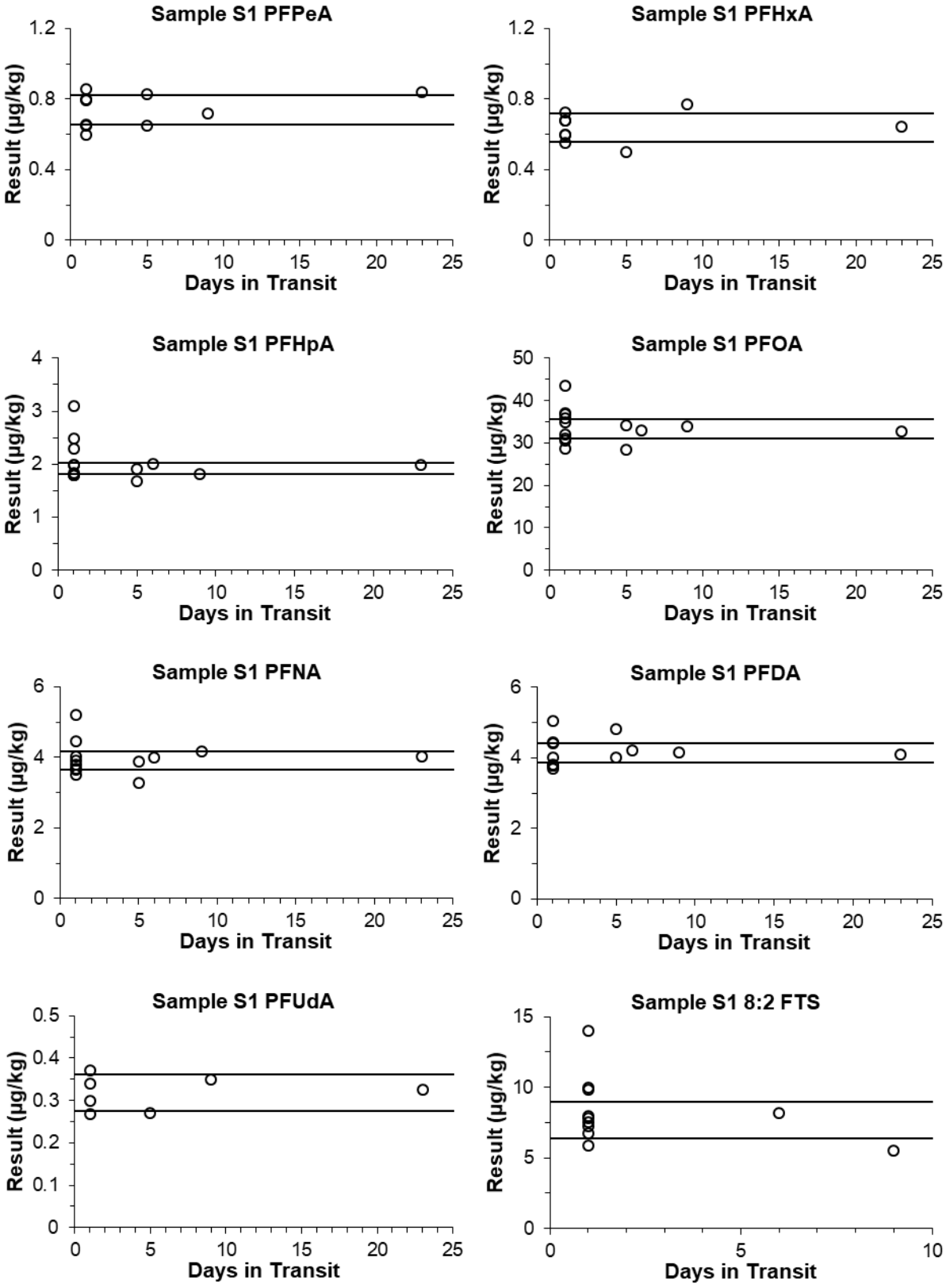
A2.3 Comparison of Results and Days in Transit

The samples were stored in freezers at approximately -80 °C and -20 °C for Samples S1 and S2 respectively after preparation and prior to dispatch. Samples were dispatched in insulated polystyrene foam boxes with cooler bricks. Comparisons of results reported to the number of days the samples spent in transit for all scored analytes are presented for information in Figures 74 and 75 (gross errors have been removed). No evidence of analyte degradation with respect to the amount of time spent in transit was evident.



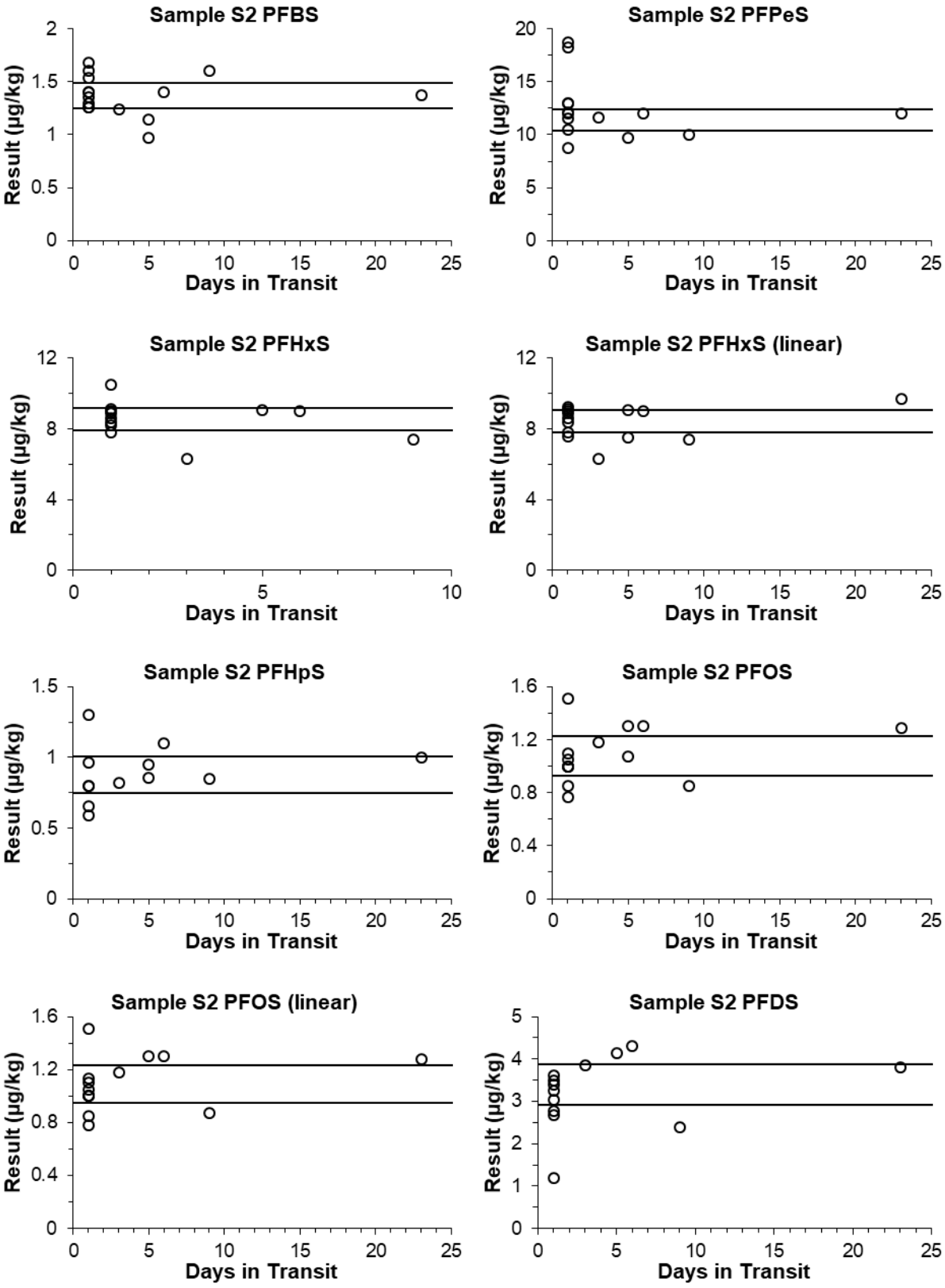
Solid lines correspond to the assigned value ± U for each analyte.

Figure 74 Result vs Days in Transit for Sample S1 Analytes



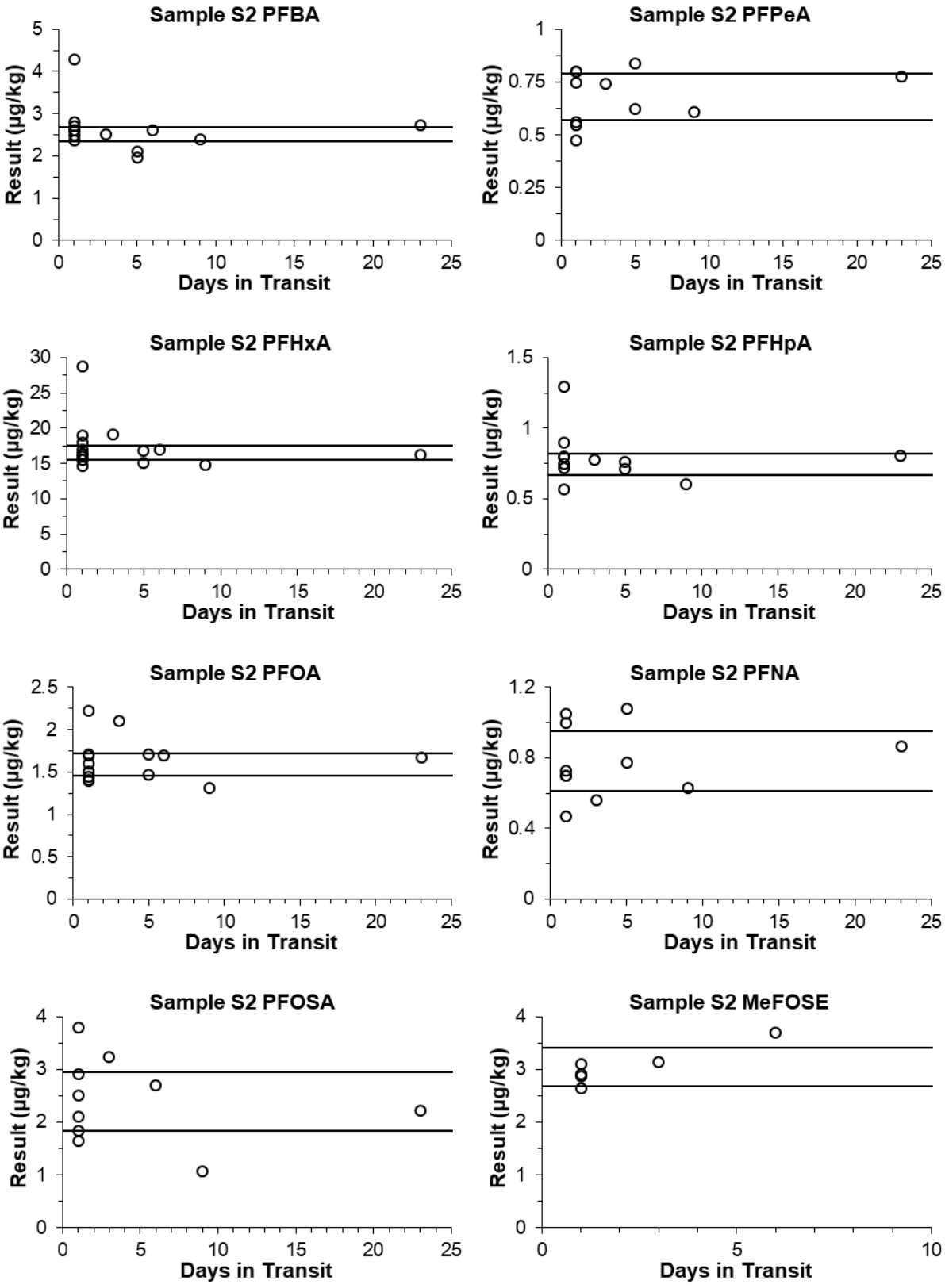
Solid lines correspond to the assigned value ± U for each analyte.

Figure 74 (continued) Result vs Days in Transit for Sample S1 Analytes



Solid lines correspond to the assigned value ± U for each analyte.

Figure 75 Result vs Days in Transit for Sample S2 Analytes



Solid lines correspond to the assigned value ± U for each analyte.

Figure 75 (continued) Result vs Days in Transit for Sample S2 Analytes

APPENDIX 3 – PARTICIPANTS' TEST METHODS

Participants' responses to the methodology questionnaire are presented in Tables 65 to 104. Some responses have been modified so that the participant cannot be identified.

Table 65 Participant Methodology – Sample S1 Beef Meat Extraction

Lab. Code	Sample Weight (g)	Sample Pretreatment	Extraction Technique	Extraction Solvent	Extraction Temperature	Extraction Time	Clean-Up
1	0.2	Freeze-drying	Alkaline Digestion	NaOH-methanol	RT	16h	Solid-Phase Extraction
3	NT						
4	2	Homogenisation	QuEChERS	Acetonitrile	Ambient	30 mins	Solid-Phase Extraction
6	NT						
7	2	Homogenisation	QuEChERS	ACN	Room	30 minutes	Solid-Phase Extraction
8	2.0064	pH Adjustment	Alkaline Digestion	Acetonitrile	Room temp	30mins x 2	Solid-Phase Extraction 2D-SPE (Waters Oasis WAX SPE + Strata GCB cartridge)
10	1	Homogenisation	Alkaline Digestion	KOH-methanol	Room temp	8 hrs	Active carbon SPE
11	1	Homogenisation	QuEChERS	Acetonitrile	Room temperature		Solid-Phase Extraction
12							
13	0.5	Homogenisation	QuEChERS	Acetonitrile	Room	60 minutes	C18 & Activated Carbon
14	1	No	Solid-Liquid Extraction	Acetonitrile	Room temperature	20 min	SPE-WAX, ultracentrifugation
15	2.012	NA	Solid-Liquid Extraction (SLE) Merris-Minimix shaker	2% formic acid in acetonitrile	Room temperature	8 min	dSPE (C18, Envicarb, MgSO4)
16	0.8	Homogenisation	Alkaline Digestion followed by addition of Acetonitrile and Sonication	Acetonitrile	Room Temperature	30 min x2	Centrifuging, Liquid-liquid extraction of lipids using n-Hexane and finally clean up by pushing samples through carbon cartridges
17	0.1	Freeze-drying	QuEChERS	Acetonitrile			Graphitized carbon

Lab. Code	Sample Weight (g)	Sample Pretreatment	Extraction Technique	Extraction Solvent	Extraction Temperature	Extraction Time	Clean-Up
18	2	Homogenisation	Solid-Liquid Extraction	10 mL acetonitrile/H2O (4:1) with 0.2% formic acid	ambient	1 min	Solid-Phase Extraction hexane wash
19	1	Homogenisation	Alkaline Digestion	Basified MeOH	Room	60 mins	Envicarb
20	1	Homogenisation	Alkaline Digestion	Basified MeOH	Room	60 mins	Envicarb

Table 66 Participant Methodology – Sample S1 Beef Meat Instrumental Technique and Analysis

Lab. Code	Instrument	Guard Column	Instrument Column	Dilution Factor	Delay Column?	Blank Correction?	Standard Method?
1	LC-MSMS or LC-QQQ	PFP 5mm×2.1mm×1.8µm	PFP 150mm×2.1mm×1.8µm	No	Yes	Yes	
3	NT						
4	LC-MSMS or LC-QQQ	UHPLC guard column; AU; InfinityLabPoroshell 120; EC-C18; 4.6 mm; 4 um	LC column; AU; Poroshell 120 HPH C18; 2.1x50 mm; 2.7 um; narrow bore	0.5	Yes	No	Isotope dilutions
6	NT						
7	Orbitrap	C18	C18		Yes		
8	LC-MSMS or LC-QQQ	Acquity Column in-line filter 0.2um	Waters Acquity UPLC CSH Phenyl-Hexyl 1.7um, 2.1 x 100mm	No	Yes	No	No - method developed in house
10	LC-MSMS or LC-QQQ	No	C18, 50 mm	No	Yes	No	No
11	LC-MSMS or LC-QQQ	C18	C18 3mm	20	Yes	No	
12							
13	LC-MSMS or LC-QQQ	nil	C18 1.6µm, 2.0mm x 50mm	No	Yes	No	QuEChERS
14	LC-MSMS or LC-QQQ	Gemini NX-C18; 4 mm x 2.0 mm ID	NX-C18; 15cm x 2 mm x 3 µm	No	Yes	Yes	No
15	LC-MSMS or LC-QQQ	NA	Zorbax XDB-C18, 100 mm x 2.1 mm, 1.8µm	NA	Yes	No	No
16	LC-MSMS or LC-QQQ	Evo C18 2 x 2.1mm	Evo C18 2.6 u 100x2.1 mm	No	Yes	No	Isotopic Dilution
17	LCMSMS		C18 100*2.1	no	Yes	No	
18	LC-MSMS or LC-QQQ		C18 1.6 x 50	No	Yes	No	
19	LC-MSMS or LC-QQQ	Pre-column Filter 0.2µm	C18 50mm x 2.1mm x 1.8µm	50	Yes	No	No. In-house
20	LC-MSMS or LC-QQQ	Pre-column Filter 0.2µm	C18 50mm x 2.1mm x 1.8µm	50	Yes	No	No. In-house

Table 67 Participant Methodology – Sample S1 Beef Meat Labelled Standards

Lab. Code	Labelled Standard Source	Recovery Correction?	Labelled Standards Additional Information
1	Wellington	No	
3	NT		
4	Wellington Laboratories	Yes	
6	NT		
7	Wellington	Yes	Results corrected by ISTD added before instrumentation
8	Wellington (Greyhound)	Yes	N/A
10	Wellington	Yes	
11	Wellington	Yes	
12			
13	Wellington Laboratories	Yes	
14	Wellington	Yes	
15	Wellington Laboratory	Yes	NA
16	Wellington	Yes	
17			
18	Wellington	Yes	
19	Wellington	Yes	
20	Wellington	Yes	

Table 68 Labelled Standards for S1 PFBS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C3-PFBS	
3		
4	13C3-PFBS	
6	Not applicable	Not applicable
7	PFOS-C8	PFBS-13C3
8	13C-PFBS	MPFOS
10	13C3 PFBS	
11	PFBS-13C3	
12		
13	13C3-PFBS	
14	18O2-PFHxS	18O2-PFOS
15	M3PFBS	NA
16	13C3-PFBS	13C3-PFHxS
17	yes	
18		18O2 PFHxS
19	13C3-PFBS	N/A
20	13C3-PFBS	N/A

Table 69 Labelled Standards for S1 PFHxS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C3-PFHxS	
3		
4	18O2-PFHxS	
6	Not applicable	Not applicable
7	PFOS-C8	PFHxS-18O2
8	13C-PFHxS	MPFOS
10	13C3 PFHxS	
11	PFHxS-18O2	
12		
13	16O2-PFHxS	
14		
15	M3PFHxS	NA
16	18O2-PFHxS	13C3-PFHxS
17		
18		18O2 PFHxS
19	18O2-PFHxS	N/A
20	18O2-PFHxS	N/A

Table 70 Labelled Standards for S1 PFHxS (linear)

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C3-PFHxS	
3		
4	18O2-PFHxS	
6	Not applicable	Not applicable
7	PFOS-C8	PFHxS-18O2
8	13C-PFHxS	MPFOS
10	13C3 PFHxS	
11	PFHxS-18O2	
12		
13	NT	
14	18O2-PFHxS	18O2-PFOS
15	M3PFHxS	NA
16	18O2-PFHxS	13C3-PFHxS
17	yes	
18		18O2 PFHxS
19	18O2-PFHxS	N/A
20	18O2-PFHxS	N/A

Table 71 Labelled Standards for S1 PFHpS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C3-PFHxS	
3		
4		
6	Not applicable	Not applicable
7	PFOS-C8	PFOS-C4
8	13C-PFOS	MPFOS
10	13C3 PFHxS	
11	PFHxS-18O2	
12		
13	13C8-PFOS	
14	18O2-PFHxS	18O2-PFOS
15	M3PFHxS	NA
16	18O2-PFHxS	13C3-PFHxS
17		
18		
19	13C4-PFOS	N/A
20	13C4-PFOS	N/A

Table 72 Labelled Standards for S1 PFOS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C8-PFOS	
3		
4	13C8-PFOS	
6	Not applicable	Not applicable
7	PFOS-C8	PFOS-C4
8	13C-PFOS	MPFOS
10	13C8 PFOS	13C4 PFOS
11	PFOS-13O4	
12		
13	13C8-PFOS	
14	13C4-PFOS	18O2-PFOS
15	M8PFOS	NA
16	13C4-PFOS	13C8-PFOS
17		
18		13C4 PFOS
19	13C4-PFOS	N/A
20	13C4-PFOS	N/A

Table 73 Labelled Standards for S1 PFOS (linear)

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C8-PFOS	
3		
4	13C8-PFOS	
6	Not applicable	Not applicable
7	PFOS-C8	PFOS-C4
8	13C-PFOS	MPFOS
10	13C8 PFOS	
11	PFOS-13O4	
12		
13	13C8-PFOS	
14	13C4-PFOS	18O2-PFOS
15	M8PFOS	NA
16	13C4-PFOS	13C8-PFOS
17	yes	
18		13C4 PFOS
19	13C4-PFOS	N/A
20	13C4-PFOS	N/A

Table 74 Labelled Standards for S1 PFDS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C8-PFOS	
3		
4		
6	Not applicable	Not applicable
7	PFOS-C8	PFBA-13C4
8	13C-PFOS	MPFOS
10	13C8 PFOS	
11	PFOS-13O4	
12		
13	13C8-PFOS	
14	13C4-PFOS	18O2-PFOS
15	M8PFOS	NA
16	13C4-PFOS	13C8-PFOS
17		
18		13C4 PFOS
19	13C4-PFOS	N/A
20	13C4-PFOS	N/A

Table 75 Labelled Standards for S1 PFBA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFBA	
3		
4	13C4-PFBA	
6	Not applicable	Not applicable
7	PFOS-C8	PFBA-13C4
8	13C-PFBA	M3PFBA
10	13C4 PFBA	13C3 PFBA
11	PFBA-13C3	
12		
13	13C4-PFBA	
14	13C4-PFBA	13C8-PFOA
15	M4PFBA	NA
16	13C4-PFBA	13C3-PFBA
17	yes	
18		13C4 PFBA
19	13C4-PFBA	N/A
20	13C4-PFBA	N/A

Table 76 Labelled Standards for S1 PFPeA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C5-PFPeA	
3		
4	13C5-PFPeA	
6	Not applicable	Not applicable
7	PFOS-C8	PFPeA-13C3
8	13C-PFPeA	M3PFBA
10	13C5 PFPeA	
11	PFPeA-13C3	
12		
13	13C5-PFPeA	
14	13C5-PFPeA	13C8-PFOA
15	M5PFPeA	NA
16	13C4-PFPeA	13C5 -PFPeA
17	yes	
18		13C4 PFBA
19	13C3-PFPeA	N/A
20	13C3-PFPeA	N/A

Table 77 Labelled Standards for S1 PFHxA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C5-PFHxA	
3		
4	13C2-PFHXA	
6	Not applicable	Not applicable
7	PFOS-C8	PFHxA-13C2
8	13C-PFHxA	M2PFOA
10	13C5 PFHxA	
11	PFHxA-13C2	
12		
13	13C5-PFHxA	
14	13C5-PFHxA	13C8-PFOA
15	M5PFHxA	NA
16	13C2-PFHxA	13C8-PFOA
17	yes	
18		13C2 PFHxA
19	13C2-PFHxA	N/A
20	13C2-PFHxA	N/A

Table 78 Labelled Standards for S1 PFHpA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFHpA	
3		
4	13C4-PFHpA	
6	Not applicable	Not applicable
7	PFOS-C8	PFHpA-13C4
8	13C-PFHpA	M2PFOA
10	13C4 PFHpA	
11	PFHpA-13C4	
12		
13	13C4-PFHpA	
14	13C4-PFHpA	13C8-PFOA
15	MPFHpA	NA
16	13C3-PFHpA	13C8-PFOA
17	yes	
18		13C2 PFHxA
19	13C4-PFHpA	N/A
20	13C4-PFHpA	N/A

Table 79 Labelled Standards for S1 PFOA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C8-PFOA	
3		
4	13C8-PFOA	
6	Not applicable	Not applicable
7	PFOS-C8	PFOA-13C4
8	13C-PFOA	M2PFOA
10	13C8 PFOA	13C2 PFOA
11	PFOA-13C4	
12		
13	13C4-PFOA	
14	13C4-PFOA	13C8-PFOA
15	M8PFOA	NA
16	13C4-PFOA	13C8-PFOA
17	yes	
18		13C4 PFOA
19	13C4-PFOA	N/A
20	13C4-PFOA	N/A

Table 80 Labelled Standards for S1 PFNA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C9-PFNA	
3		
4	13C5-PFNA	
6	Not applicable	Not applicable
7	PFOS-C8	PFNA-13C5
8	13C-PFNA	M2PFOA
10	13C9 PFNA	
11	PFNA-13C5	
12		
13	13C5-PFNA	
14	13C9-PFNA	13C5-PFNA
15	M9PFNA	NA
16	13C5-PFNA	13C8-PFOA
17	yes	
18		
19	13C5-PFNA	N/A
20	13C5-PFNA	N/A

Table 81 Labelled Standards for S1 PFDA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C6-PFDA	
3		
4	13C6-PFDA	
6	Not applicable	Not applicable
7	PFOS-C8	PFDA-13C2
8	13C-PFDA	MPFDA
10	13C6 PFDA	13C2 PFDA
11	PFDA-13C2	
12		
13	13C6-PFDA	
14	13C2-PFDA	13C5-PFNA
15	M6PFDA	NA
16	13C2-PFDA	13C8-PFOA
17	yes	
18		
19	13C2-PFDA	N/A
20	13C2-PFDA	N/A

Table 82 Labelled Standards for S1 PFUdA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C7-PFUnA	
3		
4	13C2-PFUnA	
6	Not applicable	Not applicable
7	PFOS-C8	PFUNDA-13C2
8	13C-PFUdA	MPFDA
10	13C7 PFUnA	
11	PFUdA-13C2	
12		
13	13C2-PFUnDA	
14	13C2-PFUdA	13C5-PFNA
15	M7PFUnDA	NA
16	13C2-PFUdA	13C8-PFOA
17	yes	
18		
19	13C2-PFUdA	N/A
20	13C2-PFUdA	N/A

Table 83 Labelled Standards for S1 8:2 FTS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C2-8:2FTS	
3		
4	13C2-82FTS	
6	Not applicable	Not applicable
7	PFOS-C8	8:2 FTS-13C2
8		
10	13C2 8:2 FTS	
11	8:2 FTS-13C2	
12		
13	13C2-8:2 FTS	
14	NT	NT
15	M8:2 FTS	NA
16	13C2-8:2 FTS	
17	yes	
18		13C2 6:2 FTS
19	13C2 8:2-FTS	N/A
20	13C2 8:2-FTS	N/A

Table 84 Labelled Standards for S1 GenX

Lab. Code	Before Extraction	Before Instrument Analysis
1		
3		
4		
6	Not applicable	Not applicable
7	PFOS-C8	PFPeA-13C3
8		
10		
11	HFPO-DA-13C3	
12		
13	NT	
14	NT	NT
15	M3HFPO-DA	NA
16	13C4-PFOA	
17		
18		
19	13C312C3HF11O3	N/A
20	13C312C3HF11O3	N/A

Table 85 Participant Methodology – Sample S2 Celery Extraction

Lab. Code	Sample Weight (g)	Sample Pretreatment	Extraction Technique	Extraction Solvent	Elution Solvent	Extraction Temperature	Extraction Time	Clean-Up
1	0.5g	Freeze-drying	Alkaline Digestion	NaOH-methanol		RT	16h	Solid-Phase Extraction
3	5 / 1	Homogenisation	QuEChERS	ACN	Methanol + Ammonium-ACN and Acetone	40 °C	30 min	Solid-Phase Extraction
4	5	Homogenisation	QuEChERS	Acetonitrile		Ambient	30 mins	Solid-Phase Extraction
6	10	Homogenisation	QuEChERS	Acetonitrile with 1% Acetic Acid		Room	Sonicate 30 min at 30-35 degrees	envicarb
7	5	Homogenisation	QuEChERS	ACN				Solid-Phase Extraction
8	2.0206	pH Adjustment	SPE: Oasis WAX	Acetonitrile	2% ammonium hydroxide in methanol	Room temp	30min x 2	Solid-Phase Extraction 2D-SPE (Waters Oasis WAX SPE + Strata GCB cartridge)
10	1	Homogenisation	Alkaline Digestion	KOH-methanol	Methanol	Room temp	8 hrs	Active carbon SPE
11	5	Homogenisation	QuEChERS	Acetonitrile		Room temperature		Solid-Phase Extraction
12								
13	0.5	Homogenisation	QuEChERS	Acetonitrile		Room	60 minutes	C18 & Activated Carbon
14	1	No	Solid-Liquid Extraction	Acetonitrile		Room temperature	20 min	SPE-WAX, ultracentrifugation
15	2.004	NA	Solid-Liquid Extraction (SLE) Merris-Minimix shaker	2% formic acid in acetonitrile	NA	Room temperature	8 min	dSPE (C18, Envicarb, MgSO4)

Lab. Code	Sample Weight (g)	Sample Pretreatment	Extraction Technique	Extraction Solvent	Elution Solvent	Extraction Temperature	Extraction Time	Clean-Up
16	0.5	Homogenisation	Bond Elut carbon cartridge	Sodium hydroxide in Methanol		Fridge overnight, room temperature	2x 30 mins	Bond Elut carbon cartridge
17	0.2	Freeze-drying	QuEChERS	ACN			60 minute	Carbon
18								
19	2	Homogenisation	Alkaline Digestion	Basified MeOH	N/A	Room	60mins	Envicarb
20	2	Homogenisation	Alkaline Digestion	Basified MeOH	N/A	Room	60mins	Envicarb

Table 86 Participant Methodology – Sample S2 Celery Instrumental Technique and Analysis

Lab. Code	Instrument	Guard Column	Instrument Column	Dilution Factor	Delay Column?	Blank Correction?	Standard Method?
1	LC-MSMS or LC-QQQ	PFP 5mm×2.1mm×1.8µm	PFP 150mm×2.1mm×1.8µm	No	Yes	Yes	
3	LC-MSMS or LC-QQQ	UltraShield UHPLC 0.2 µm Restek	Raptor C18 1.8 µm 50 x 2.1 mm Restek		yes	no	
4	LC-MSMS or LC-QQQ	UHPLC guard column; AU; InfinityLabPoroshell 120; EC-C18; 4.6 mm; 4 um	LC column; AU; Poroshell 120 HPH C18; 2.1x50 mm; 2.7 um; narrow bore	0.2	Yes	No	Isotope dilutions
6	Orbitrap	C18 3mm	Kinetex C18 100x3mm 2.6 um	x10	Yes	Yes	In house
7	Orbitrap	C18	C18		Yes		
8	LC-MSMS or LC-QQQ	Acquity Column in-line filter 0.2um	Waters Acquity UPLC CSH Phenyl-Hexyl 1.7um, 2.1 x 100mm	No	Yes	No	No - method developed in house
10	LC-MSMS or LC-QQQ	No	C18, 50 mm	No	Yes	No	No
11	LC-MSMS or LC-QQQ	C18	C18 3mm		Yes	No	
12							
13	LC-MSMS or LC-QQQ	nil	C18 1.6µm, 2.0mm x 50mm	No	Yes	No	QuEChERS
14	LC-MSMS or LC-QQQ	Gemini NX-C18; 4 mm x 2.0 mm ID	NX-C18; 15cm x 2 mm x 3 µm	No	Yes	Yes	No
15	LC-MSMS or LC-QQQ	NA	Zorbax XDB-C18, 100 mm x 2.1 mm, 1.8µm	NA	Yes	No	No
16		Evo C18 2 x 2.1mm	Evo C18 2.6 u 100x2.1 mm	No	Yes	No	Isotopic Dilution
17	LCMSMS	C18 20*3	C18, 2.1*100		Yes	No	
18							
19	LC-MSMS or LC-QQQ	Pre-column Filter 0.2µm	C18 50mm x 2.1mm x 1.8µm	25	Yes	No	No. In-house
20	LC-MSMS or LC-QQQ	Pre-column Filter 0.2µm	C18 50mm x 2.1mm x 1.8µm	25	Yes	No	No. In-house

Table 87 Participant Methodology – Sample S2 Celery Labelled Standards

Lab. Code	Labelled Standard Source	Recovery Correction?	Labelled Standards Additional Information
1	Wellington	No	
3		Yes	
4	Wellington Laboratories	Yes	
6	Wellington	No	
7	Wellington	Yes	Results corrected by ISTD added before instrumentation
8	Wellington (Greyhound)	Yes	N/A
10	Wellington	Yes	
11	Wellington	Yes	
12			
13	Wellington Laboratories	Yes	
14	Wellington	Yes	
15	Wellington Laboratory	Yes	NA
16	Wellington	Yes	
17			
18			
19	Wellington	Yes	
20	Wellington	Yes	

Table 88 Labelled Standards for S2 PFBS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C3-PFBS	
3	13C3-PFBS	13C4-PFOA
4	13C3-PFBS	
6	Sodium perfluoro-1-[2,3,4 13C3] butanesulfonate M3PFBS	
7	PFOS-C8	PFBS-13C3
8	13C-PFBS	MPFOS
10	13C3 PFBS	
11	PFBS-13C3	
12		
13	13C3-PFBS	
14	18O2-PFHxS	18O2-PFOS
15	M3PFBS	NA
16	13C3-PFBS	13C3-PFHxS
17	yes	
18		
19	13C3-PFBS	N/A
20	13C3-PFBS	N/A

Table 89 Labelled Standards for S2 PFPeS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C3-PFBS	
3	13C3-PFBS	13C4-PFOA
4		
6		
7	PFOS-C8	PFOS-C4
8		
10	13C3 PFBS	
11	PFHxS-18O2	
12		
13	16O2-PFHxS	
14	18O2-PFHxS	18O2-PFOS
15	M5PFHxA	NA
16	18O2-PFHxS	13C3-PFHxS
17		
18		
19	18O2-PFHxS	N/A
20	18O2-PFHxS	N/A

Table 90 Labelled Standards for S2 PFHxS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C3-PFHxS	
3	18O2-PFHxS	13C4-PFOA
4	18O2-PFHxS	
6	Sodium perfluoro-1-[1,2,3 13C3] hexanesulfonate M3PFHxS	
7	PFOS-C8	PFHxS-18O2
8	13C-PFHxS	MPFOS
10	13C3 PFHxS	
11	PFHxS-18O2	
12		
13	16O2-PFHxS	
14		
15	M3PFHxS	NA
16	18O2-PFHxS	13C3-PFHxS
17		
18		
19	18O2-PFHxS	N/A
20	18O2-PFHxS	N/A

Table 91 Labelled Standards for S2 PFHxS (linear)

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C3-PFHxS	
3	18O2-PFHxS	13C4-PFOA
4	18O2-PFHxS	
6		
7	PFOS-C8	PFHxS-18O2
8	13C-PFHxS	MPFOS
10	13C3 PFHxS	
11	PFHxS-18O2	
12		
13	NT	
14	18O2-PFHxS	18O2-PFOS
15	M3PFHxS	NA
16	18O2-PFHxS	13C3-PFHxS
17	yes	
18		
19	18O2-PFHxS	N/A
20	18O2-PFHxS	N/A

Table 92 Labelled Standards for S2 PFHpS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C3-PFHxS	
3	18O2-PFHxS	13C4-PFOA
4		
6		
7	PFOS-C8	PFOS-C4
8	13C-PFOS	MPFOS
10	13C3 PFHxS	
11	PFHxS-18O2	
12		
13	13C8-PFOS	
14	18O2-PFHxS	18O2-PFOS
15	M3PFHxS	NA
16	18O2-PFHxS	13C3-PFHxS
17		
18		
19	13C4-PFOS	N/A
20	13C4-PFOS	N/A

Table 93 Labelled Standards for S2 PFOS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C8-PFOS	
3	13C4-PFOS	13C4-PFOA
4	13C8-PFOS	
6	Sodium perfluoro-1-[13C8] ocatanesulfonate M8PFOS	
7	PFOS-C8	PFOS-C4
8	13C-PFOS	MPFOS
10	13C8 PFOS	13C4 PFOS
11	PFOS-13O4	
12		
13	13C8-PFOS	
14	13C4-PFOS	18O2-PFOS
15	M8PFOS	NA
16	13C4-PFOS	13C8-PFOS
17		
18		
19	13C4-PFOS	N/A
20	13C4-PFOS	N/A

Table 94 Labelled Standards for S2 PFOS (linear)

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C8-PFOS	
3	13C4-PFOS	13C4-PFOA
4	13C8-PFOS	
6		
7	PFOS-C8	PFOS-C4
8	13C-PFOS	MPFOS
10	13C8 PFOS	
11	PFOS-13O4	
12		
13	13C8-PFOS	
14	13C4-PFOS	18O2-PFOS
15	M8PFOS	NA
16	13C4-PFOS	13C8-PFOS
17	yes	
18		
19	13C4-PFOS	N/A
20	13C4-PFOS	N/A

Table 95 Labelled Standards for S2 PFDS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C8-PFOS	
3	13C2-PFUnA	13C4-PFOA
4		
6		
7	PFOS-C8	PFBA-13C4
8	13C-PFOS	MPFOS
10	13C8 PFOS	
11	PFOS-13O4	
12		
13	13C8-PFOS	
14	13C4-PFOS	18O2-PFOS
15	M8PFOS	NA
16	13C4-PFOS	13C8-PFOS
17		
18		
19	13C4-PFOS	N/A
20	13C4-PFOS	N/A

Table 96 Labelled Standards for S2 PFBA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFBA	
3	13C4-PFBA	13C4-PFOA
4	13C4-PFBA	
6	Perfluoro-n-[13C4]butanoic acid MPFBA	
7	PFOS-C8	PFBA-13C4
8	13C-PFBA	M3PFBA
10	13C4 PFBA	13C3 PFBA
11	PFBA-13C3	
12		
13	13C4-PFBA	
14	13C4-PFBA	13C8-PFOA
15	M4PFBA	NA
16	13C4-PFBA	13C3-PFBA
17	yes	
18		
19	13C4-PFBA	N/A
20	13C4-PFBA	N/A

Table 97 Labelled Standards for S2 PFPeA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C5-PFPeA	
3	13C5-PFPeA	13C4-PFOA
4	13C5-PFPeA	
6	Perfluoro-n-[13C5]pentanoic acid M5PFPeA	
7	PFOS-C8	PFPeA-13C3
8	13C-PFPeA	M3PFBA
10	13C5 PFPeA	
11	PFPeA-13C3	
12		
13	13C5-PFPeA	
14	13C5-PFPeA	13C8-PFOA
15	M5PFPeA	NA
16	13C4-PFPeA	13C5 -PFPeA
17	yes	
18		
19	13C3-PFPeA	N/A
20	13C3-PFPeA	N/A

Table 98 Labelled Standards for S2 PFHxA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C5-PFHxA	
3	13C2-PFHxA	13C4-PFOA
4	13C2-PFHXA	
6	Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid M5PFHxA	
7	PFOS-C8	PFHxA-13C2
8	13C-PFHxA	M2PFOA
10	13C5 PFHxA	
11	PFHxA-13C2	
12		
13	13C5-PFHxA	
14	13C5-PFHxA	13C8-PFOA
15	M5PFHxA	NA
16	13C2-PFHxA	13C8-PFOA
17	yes	
18		
19	13C2-PFHxA	N/A
20	13C2-PFHxA	N/A

Table 99 Labelled Standards for S2 PFHpA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFHpA	
3	13C4-PFHpA	13C4-PFOA
4	13C4-PFHpA	
6	Perfluoro-n-[1,2,3,4-13C4]heptanoic acid M4PFHpA	
7	PFOS-C8	PFHpA-13C4
8	13C-PFHpA	M2PFOA
10	13C4 PFHpA	
11	PFHpA-13C4	
12		
13	13C4-PFHpA	
14	13C4-PFHpA	13C8-PFOA
15	MPFHpA	NA
16	13C3-PFHpA	13C8-PFOA
17	yes	
18		
19	13C4-PFHpA	N/A
20	13C4-PFHpA	N/A

Table 100 Labelled Standards for S2 PFOA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C8-PFOA	
3	13C8-PFOA	13C4-PFOA
4	13C8-PFOA	
6	Perfluoro-n-[13C8]octanoic acid M8PFOA	
7	PFOS-C8	PFOA-13C4
8	13C-PFOA	M2PFOA
10	13C8 PFOA	13C2 PFOA
11	PFOA-13C4	
12		
13	13C4-PFOA	
14	13C4-PFOA	13C8-PFOA
15	M8PFOA	NA
16	13C4-PFOA	13C8-PFOA
17	yes	
18		
19	13C4-PFOA	N/A
20	13C4-PFOA	N/A

Table 101 Labelled Standards for S2 PFNA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C9-PFNA	
3	13C5-PFNA	13C4-PFOA
4	13C5-PFNA	
6	Perfluoro-n-[13C9]nonanoic acid M9PFNA	
7	PFOS-C8	PFNA-13C5
8	13C-PFNA	M2PFOA
10	13C9 PFNA	
11	PFNA-13C5	
12		
13	13C5-PFNA	
14	13C9-PFNA	13C5-PFNA
15	M9PFNA	NA
16	13C5-PFNA	13C8-PFOA
17	yes	
18		
19	13C5-PFNA	N/A
20	13C5-PFNA	N/A

Table 102 Labelled Standards for S2 PFOSA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C8-FOSA	
3	13C8-PFOSA	13C4-PFOA
4	13C8-FOSA	
6	Perfluoro-1-[13C8]otanesulfonamide	
7	PFOS-C8	FOSA-13C8
8		
10	13C8 PFOSA	
11	FOSA-13C8	
12		
13	13C8-FOSA	
14	13C8-PFOSA	13C2-PFTeDA
15	MPFOSA	NA
16	13C8-FOSA	
17		
18		
19	13C8-FOSA	N/A
20	13C8-FOSA	N/A

Table 103 Labelled Standards for S2 MeFOSE

Lab. Code	Before Extraction	Before Instrument Analysis
1		
3	d7-MeFOSE	13C4-PFOA
4	d7-MeFOSE	
6	d7-N-MeFOSE-M 2-(N-methyl-d3-perfluoro-1-octanesulfonamido) ethand4-ol	
7	PFOS-C8	MeFOSE-D3
8		
10	d7-N-MeFOSE	
11	MeFOSE-D7	
12		
13	d7-MeFOSE	
14	NT	NT
15	d7-NMeFOSE-M	NA
16	D7-N-Me FOSE	
17		
18		
19	D7-Me-FOSE	N/A
20	D7-Me-FOSE	N/A

Table 104 Labelled Standards for S2 10:2 FTS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C2-8:2FTS	
3	13C2-8:2 FTS	13C4-PFOA
4	13C2d4 10:2 FTS	
6		
7	PFOS-C8	10:2 FTS-13C2
8		
10	13C2 8:2 FTS	
11	10:2 FTS-13C2-D4	
12		
13	13C2-8:2 FTS	
14	NT	NT
15	MPFDoDA	NA
16	13C2-8:2 FTS	
17		
18		
19	13C2 8:2-FTS	N/A
20	13C2 8:2-FTS	N/A

APPENDIX 4 – ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, z-SCORE AND E_n-SCORE CALCULATIONS

A4.1 Robust Average and Associated Uncertainty

Robust averages were calculated using the procedure described in ISO 13528:2015.[5] The associated uncertainties were estimated as according to Equation 4.

$$u_{rob\ av} = \frac{1.25 \times S_{rob\ av}}{\sqrt{p}} \qquad \textit{Equation 4}$$

where:

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

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

p is the number of results

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

A worked example is set out below in Table 105.

Table 105 Uncertainty Estimate for Robust Average of PFHxS (linear) in Sample S1

Number of Results (p)	13
Robust Average	2.67 µg/kg
$S_{rob\ av}$	0.43 µg/kg
$u_{rob\ av}$	0.15 µg/kg
k	2
$U_{rob\ av}$	0.30 µg/kg

Therefore, the robust average for PFHxS (linear) in Sample S1 is 2.67 ± 0.30 µg/kg.

A4.2 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 (Section 4).

A worked example is set out below in Table 106.

Table 106 z-Score and E_n-Score for Sample S1 PFBS Result Reported by Laboratory 1

Participant Result (µg/kg)	Assigned Value (µg/kg)	Target Standard Deviation	z-Score	E_n-Score
2.67 ± 0.2	2.18 ± 0.19	20% as PCV, or: 0.2 × 2.18 = 0.436 µg/kg	z-Score = $\frac{2.67 - 2.18}{0.436}$ = 1.12	E_n-Score = $\frac{2.67 - 2.18}{\sqrt{0.2^2 + 0.19^2}}$ = 1.78

APPENDIX 5 – ACRONYMS AND ABBREVIATIONS

4:2 FTS	4:2 Fluorotelomer sulfonic acid
6:2 FTS	6:2 Fluorotelomer sulfonic acid
8:2 FTS	8:2 Fluorotelomer sulfonic acid
10:2 FTS	10:2 Fluorotelomer sulfonic acid
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonate
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonate
A.V.	Assigned Value
ADONA	Ammonium 4,8-dioxa-3H-perfluorononanoate
CITAC	Cooperation on International Traceability in Analytical Chemistry
CRM	Certified Reference Material
CV	Coefficient of Variation
dSPE	Dispersive SPE
EtFOSA	N-Ethyl perfluorooctane sulfonamide
EtFOSAA	N-Ethyl perfluorooctane sulfonamido acetic acid
EtFOSE	N-Ethyl perfluorooctane sulfonamido ethanol
FSANZ	Food Standards Australia New Zealand
GAG	(NATA) General Accreditation Guidance
GenX	Ammonium 2,3,3,3-tetrafluoro-2-(heptafluoropropoxy) propanoate
GUM	Guide to the Expression of Uncertainty in Measurement
H.V.	Homogeneity Value
IEC	International Electrotechnical Commission
ISO	International Organization for Standardization
LC	Liquid Chromatography
LLE	Liquid-Liquid Extraction
LOR	Limit of Reporting
Max.	Maximum value in a set of results
Md	Median
MeFOSA	N-Methyl perfluorooctane sulfonamide
MeFOSAA	N-Methyl perfluorooctane sulfonamido acetic acid
MeFOSE	N-Methyl perfluorooctane sulfonamido ethanol
Min.	Minimum value in a set of results
MS	Mass Spectrometry
MS/MS	Tandem Mass Spectrometry
MU	Measurement Uncertainty
N	Number of numeric results
NATA	National Association of Testing Authorities (Australia)
NMI	National Measurement Institute (Australia)
NR	Not Reported

NT	Not Tested
PCV	Performance Coefficient of Variation
PFAS	Per- and Polyfluoroalkyl Substances
PFBA	Perfluorobutanoic acid
PFBS	Perfluorobutane sulfonate
PFDA	Perfluorodecanoic acid
PFDS	Perfluorodecane sulfonate
PFDoA	Perfluorododecanoic acid
PFDoS	Perfluorododecane sulfonate
PFHpA	Perfluoroheptanoic acid
PFHpS	Perfluoroheptane sulfonate
PFHxA	Perfluorohexanoic acid
PFHxS	Perfluorohexane sulfonate
PFNA	Perfluorononanoic acid
PFNS	Perfluorononane sulfonate
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctane sulfonate
PFOSA	Perfluorooctane sulfonamide
PFPeA	Perfluoropentanoic acid
PFPeS	Perfluoropentane sulfonate
PFTeDA	Perfluorotetradecanoic acid
PFTrDA	Perfluorotridecanoic acid
PFTrDS	Perfluorotridecane sulfonate
PFUdA	Perfluoroundecanoic acid
PFUdS	Perfluoroundecane sulfonate
PT	Proficiency Test
QQQ	Triple Quadrupole
QuEChERS	Quick, Easy, Cheap, Effective, Rugged and Safe extraction method
R.A.	Robust Average
RM	Reference Material
S.V.	Spiked Value (Spiked or formulated concentration of a PT sample)
SD	Standard Deviation
SLE	Solid-Liquid Extraction
SPE	Solid Phase Extraction
SS	Spiked Samples
UPLC	Ultra Performance Liquid Chromatography
USEPA	United States Environmental Protection Agency

END OF REPORT