



Australian Government  
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National  
Measurement  
Institute

# Proficiency Test Final Report AQA 20-11 PFAS in Biota and Food

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## SUMMARY

AQA 20-11 PFAS in Biota and Food commenced in September 2020. Twenty-two laboratories registered to participate, and twenty-one participants submitted results.

The sample set consisted of one spiked prawn sample (Sample S1) and one spiked lettuce sample (Sample S2). The per- and polyfluorinated alkyl substances (PFAS) analytes considered in this study were: PFBS, PFHxS (total and linear isomers), PFHpS, PFOS (total and linear isomers), PFDS, PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUdA, PFOSA, ADONA and GenX.

Of a possible 593 results, 496 numeric results (84%) were submitted. Thirty-seven results were a 'less than' value ( $< x$ ) or Not Reported (NR), and sixty 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 biota and food matrices.*

Sixteen participants returned results for both matrices, three participants analysed biota only and two participants analysed food only. Laboratories **7**, **11**, **13** and **14** reported results for all analytes of interest in this study.

Eight participants did not report results for analytes that they tested for and were spiked into the samples (total of 15 results), while fourteen participants reported analytes that were not spiked into the samples (total of 32 results).

- *Compare the performances of participant laboratories and to assess their accuracy in the measurement of PFAS in biota and food matrices.*

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

Of 496 z-scores, 404 (81%) returned  $|z| \leq 2.0$ , indicating a satisfactory performance.

Of 496  $E_n$ -scores, 339 (68%) returned  $|E_n| \leq 1.0$ , indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Laboratories **7** and **14** returned satisfactory z-scores for all scored analytes (32). Laboratory **16** analysed biota only and returned satisfactory z-scores and  $E_n$ -scores for all scored analytes in this matrix (17).

- *Evaluate the laboratories' methods for PFAS in biota and food analysis.*

Participants used a variety of methods for extraction and analysis. No significant trend between methodology and results was evident, though participants may need to consider if using low sample masses for analysis or short extraction times could bias their results.

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

This study is NMI's 5<sup>th</sup> PFAS in Biota and Food PT study. Participants have been reporting fairly consistent proportions of numeric results even with the increased number of PFAS analytes in the last few studies, indicating that participants have the capacity to analyse a wide range of PFAS. However, in this study, the proportion of satisfactory z-scores and  $E_n$ -scores have decreased as compared to previous studies, indicating that the accuracy of reported results could be improved upon.

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

Of 496 numerical results for analytes of interest in this study, 476 (96%) were reported with an associated expanded MU, with a variety of procedures used to estimate uncertainty. The magnitude of the reported MUs for analytes in this study was within the range 0.03% to 10529% of the reported value.

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

The test samples of this PT study are homogeneous and are well characterised. Surplus of these 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.'<sup>1</sup> 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, food and pharmaceuticals;
- controlled drug assay and clandestine laboratory; and
- allergens in food.

### **1.2 Study Background**

Per- and polyfluorinated alkyl 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 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 biota and food matrices.
- compare the performances of participants and assess their accuracy in the measurement of PFAS in biota and food matrices;
- evaluate the participants' test methods for PFAS in biota and food analysis;
- compare the performance of participants with their past performance;
- develop the practical application of traceability and measurement uncertainty, and provide participants with information that will be useful in assessing their uncertainty estimates; and
- produce materials that can be used in method validation and as control samples.

### **1.4 Study Conduct**

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

NMI is accredited by the National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043 as a provider of proficiency testing schemes.<sup>1</sup> 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	4 September 2020
Samples dispatched	28 September 2020
Results due	23 November 2020
Interim report issued	1 December 2020

### 2.2 Participation

Twenty-two laboratories registered to participate in this study, and twenty-one participants submitted results.

### 2.3 Laboratory Code

All participants were assigned a confidential laboratory code number.

### 2.4 Test Material Preparation

Two test samples were prepared in September 2020 at NMI North Ryde. Care was taken to avoid any PFAS contamination during sample preparation.

- Sample S1: Prawn (5 g portions) spiked with 17 PFAS analytes.
- Sample S2: Lettuce (40 g portions) spiked with 15 PFAS analytes.

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

Details of spiked analytes and values are presented in Table 1. Participants were requested to report both the linear isomers and total of PFHxS and PFOS.

Table 1 Spiked Values of Test Samples

Analyte	S1 Prawn (Spiked) (µg/kg)	S2 Lettuce (Spiked) (µg/kg)
PFBS	0.201	3.05
PFHxS	6.53	6.08
PFHxS (linear)*	6.39	5.86
PFHpS	5.54	7.33
PFOS	12.3	37.2
PFOS (linear)*	8.54	25.8
PFDS	-	14.4
PFBA	0.983	19.3
PFPeA	1.01	30.1
PFHxA	1.19	33.9
PFHpA	1.92	-
PFOA	4.94	18.2
PFNA	0.976	4.53
PFDA	0.488	3.33
PFUdA	0.399	-

Analyte	S1 Prawn (Spiked) ( $\mu\text{g}/\text{kg}$ )	S2 Lettuce (Spiked) ( $\mu\text{g}/\text{kg}$ )
PFOSA	6.93	-
ADONA	9.35	27.4
GenX	12.4	20.8

\* Samples were spiked with a technical mixture; spiked values are best estimates based on analyses of mixtures.

Further sample preparation details can be found in Appendix 1.

## 2.5 Test Material Homogeneity and Stability Testing

Lettuce was introduced as a matrix for the first time in this PFAS PT study. The homogeneity and stability testing are presented in Appendix 1. The lettuce sample was demonstrated to be sufficiently homogeneous and stable for the evaluation of participants' performance.

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

Participants' robust averages for analytes in the two samples were within 73% to 105% of the spiked values, which provides support for the stability of these samples.

## 2.6 Sample Storage, Dispatch and Receipt

Prior to dispatch to participants, the prawn and lettuce samples were kept frozen. Prawn samples were kept at  $-80^{\circ}\text{C}$  and lettuce samples were kept at  $-20^{\circ}\text{C}$ .

Participants were sent 5 g prawn samples in Greiner tubes for Sample S1 and 40 g lettuce samples also in tubes for Sample S2. The samples were packed in foam boxes with cooler bricks and sent by courier on 28 September 2020.

The following items were packaged with the samples:

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

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

## 2.7 Instructions to Participants

Participants were instructed as follows:

- Quantitatively analyse the samples using your normal test method.
- Report results in units of  $\mu\text{g}/\text{kg}$  on as received basis for PFAS in Samples S1 and S2.
- For PFAS that contain linear and branched isomers, report TOTAL (the sum of linear and branched).
- For PFOS and PFHxS you are asked to report both TOTAL (the sum of linear and branched isomers) and LINEAR (the linear isomers only).
- 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 results sheet). This figure will be used in all statistical analysis in the study report.
- For each analyte report the associated expanded measurement uncertainty (e.g.  $0.50 \pm 0.02 \mu\text{g}/\text{kg}$ ).

- Report any analyte not tested as NT.
- 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.
- Please complete the method details as required in the Methodology sheet and report the basis of your uncertainty estimates (e.g. repeatability precision, long term result variability).
- If determined, report your internal standard percentage recovery. This will be presented in the report for information only.
- Return the completed results sheet by e-mail (proficiency@measurement.gov.au) by 26 October 2020.

Due to the exceptional international circumstances occurring over the course of this study, the results due date was extended to 23 November 2020 for all participants.

## **2.8 Interim Report**

An interim report was emailed to all participants on 1 December 2020.

### 3 PARTICIPANT LABORATORY INFORMATION

#### 3.1 Test Methods Reported by Participants

Participants were requested to provide methodology information. Responses are presented in Appendix 2. The study coordinator thanks participants for completing the questionnaire.

#### 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 2 and 3. Some responses have been modified so that the participant cannot be identified.

Table 2 Basis of Participants' Uncertainty Estimate

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
1	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	Recoveries of SS	
2	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS Duplicate analysis Instrument calibration	Instrument calibration Laboratory bias from PT studies Recoveries of SS Standard purity	NATA GAG Estimating and Reporting MU
3	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS Duplicate analysis	Instrument calibration Standard purity	NATA GAG Estimating and Reporting MU
4	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM Duplicate analysis Instrument calibration	CRM Laboratory bias from PT studies Recoveries of SS	NMI Uncertainty Course
5	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
6	Top Down - precision and estimates of the method and laboratory bias	Control samples	Laboratory bias from PT studies Recoveries of SS	Nordtest Report TR537
7	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS Duplicate analysis Instrument calibration	CRM Instrument calibration Laboratory bias from PT studies Recoveries of SS Standard purity	NATA GAG Estimating and Reporting MU
8	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
9	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS Standard purity	Eurachem/CITAC Guide
10	Top Down - precision and estimates of the method and laboratory bias	Control samples Duplicate analysis Instrument calibration	CRM Instrument calibration Recoveries of SS	ISO/GUM

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
11	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS	Recoveries of SS	USEPA SW-846
12	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS	ISO/GUM
13	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	
14	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS Standard purity	NATA GAG Estimating and Reporting MU
15	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)	Duplicate analysis Instrument calibration	Instrument calibration	ISO/GUM
16	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS		ISO/GUM
17	Top Down - precision and estimates of the method and laboratory bias	Control samples Duplicate analysis		Eurachem/CITAC Guide
18	Top Down - reproducibility (standard deviation) from PT studies used directly	Standard deviation from PT studies only		ISO/GUM
		Control samples Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS Standard purity	
19	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS	Recoveries of SS	ISO/GUM
20	Top Down - precision and estimates of the method and laboratory bias	Control samples Duplicate analysis	CRM Laboratory bias from PT studies Recoveries of SS	Nordtest Report TR537
21		Control samples - RM Duplicate analysis	Instrument calibration Laboratory bias from PT studies Recoveries of SS	

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

Table 3 Uncertainty Estimate Comments

Lab. Code	Approach to Estimating MU
8	Measurement Uncertainty (U) estimated from the standard deviation (u) of replicate recovery samples using the expression $U = 2 \times u$ . Procedure as set out in Statistics and Chemometrics for Analytical Chemistry, Miller and Miller, 5th Edition.
11	Standard practice for laboratories utilising US EPA's SW-846 document.
13	Uncertainty calculated as 3 x SD of replicate analysis
16	The expanded measurement uncertainty values were calculated using a coverage factor (K) value of 2.00 and at the 95% confidence limit.
19	This laboratory has an SOP that is based on ISO17025.

### 3.3 Participants' Comments

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

Table 4 Participants' Comments

Lab. Code	Sample	Participant's Comments	Study Coordinator's Response
13	S2	PFDS results were extremely variable with no clear reason for this	Participants' results for S2 PFDS gave no reason to question its homogeneity.
16	S1	The sample was received at a temperature of 19.3°C; which was above the laboratory method recommended sample storage temperature (less than or equal to 4°C).	Stability checks have been conducted for PFAS in prawn in previous studies. Samples have been confirmed stable at room temperature for at least 3 months.

## 4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

### 4.1 Results Summary

Participant results are presented in Tables 5 to 36 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 33. An example chart with interpretation guide is shown in Figure 1.

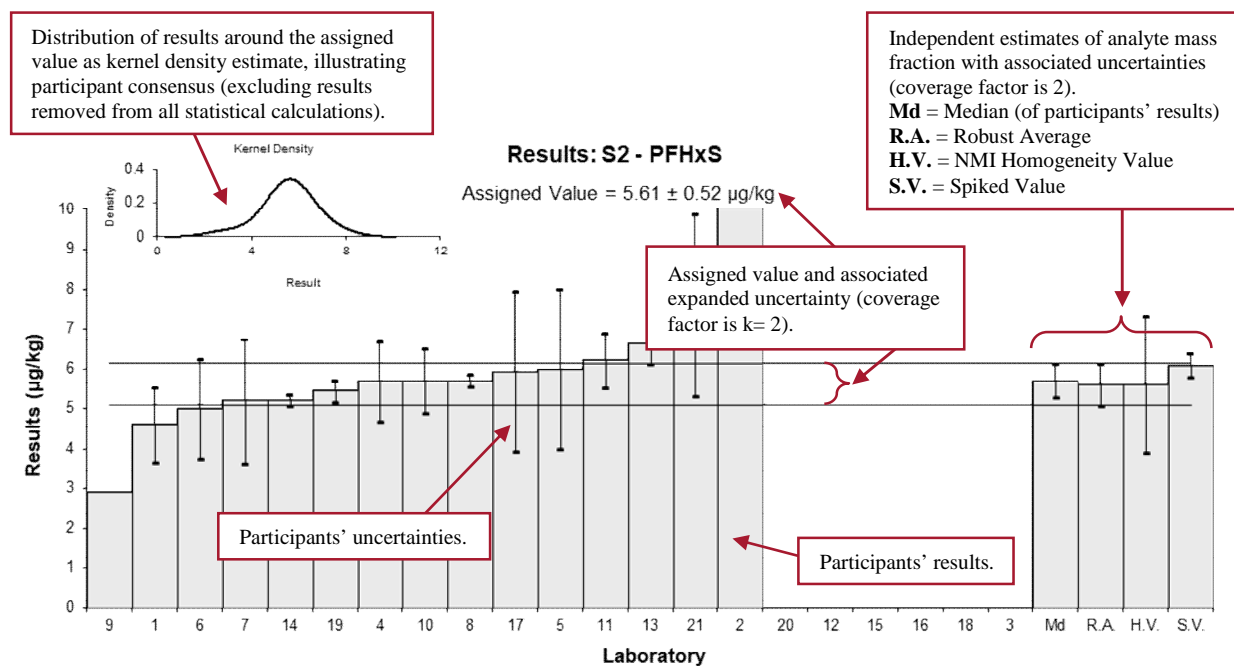


Figure 1 Guide to Presentation of Results

### 4.2 Assigned Value

The assigned value is defined as the: 'value attributed to a particular property of a proficiency test item'.<sup>1</sup> 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 (results less than 50% and greater than 150% of the robust average were removed before the calculation of the assigned value).<sup>3,4</sup> The expanded uncertainties were estimated from the associated robust SDs (Appendix 3).

### 4.3 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.<sup>5</sup>

### 4.4 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. It is important to note that this is a performance measure set by the study coordinator; it is not the CV of participant results. The PCV 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.<sup>6</sup> By setting a fixed and realistic value for the PCV, a participant's performance does not depend on the other participants' performance and can be compared from study to study.



#### 4.5 Target Standard Deviation

The target standard deviation ( $\sigma$ ) is the product of the assigned value ( $X$ ) and the PCV, as presented in Equation 1. This value is used for the calculation of z-scores.

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

#### 4.6 z-Score

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

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

where:

- $z$  is z-score
- $\chi$  is a participant's result
- $X$  is the assigned value
- $\sigma$  is the target standard deviation from Equation 1

For a z-score with absolute value ( $|z|$ ):

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

To account for potential low bias in the consensus value due to inefficient methodologies, a number of scores were adjusted for a 'maximum acceptable concentration', where results lower than the maximum acceptable concentration but with a z-score greater than 2.0 had their z-score adjusted to 2.0. Additional information is given in Section 6.3.

#### 4.7 E<sub>n</sub>-Score

The E<sub>n</sub>-score is complementary to the z-score in assessment of laboratory performance. E<sub>n</sub>-score includes measurement uncertainty and is calculated according to Equation 3.

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

where:

- $E_n$  is E<sub>n</sub>-score
- $\chi$  is a participant's result
- $X$  is the assigned value
- $U_\chi$  is the expanded uncertainty of the participant's result
- $U_X$  is the expanded uncertainty of the assigned value

For an E<sub>n</sub>-score with absolute value ( $|E_n|$ ):

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

#### 4.8 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC 17025:2017 must establish and demonstrate the traceability and measurement uncertainty associated with their test results.<sup>7</sup>

Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.<sup>8</sup>

## 5 TABLES AND FIGURES

Table 5

### Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFBS
<b>Units</b>	µg/kg

### Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	<0.5	NR	74.1		
2	0.55	0.02	NR	12.41	8.11
3	0.1	0.02	NR	-1.84	-1.20
4	0.169	0.04	69	0.35	0.18
5	<1	NR	82		
6	NR	NR	91		
7	0.0960	0.0960	95	-1.96	-0.59
8	< 1.0	NR	99		
9	NT	NT	NT		
10	<0.2	0.03	52		
11	0.152	0.0281	69	-0.19	-0.11
12	NT	NT	NT		
13***	0.227	0.040	85.0	2.00	1.00
14	0.113	0.028	95	-1.42	-0.86
15	0.196	0.082	NR	1.20	0.41
16	0.17	17.9	105	0.38	0.00
17	<1	NR	117		
18	0.529	0.109	67	11.74	3.16
19	NR	NR	158		
20	0.201	0.045	78	1.36	0.68
21	< 0.300	0.09	51.7		

### Statistics\*

<b>Assigned Value**</b>	0.158	0.044
<b>Spike</b>	0.201	0.010
<b>Max. Acceptable Conc.***</b>	0.264	
<b>Robust Average</b>	0.169	0.049
<b>Median</b>	0.170	0.047
<b>Mean</b>	0.195	
<b>N</b>	10	
<b>Max.</b>	0.529	
<b>Min.</b>	0.096	
<b>Robust SD</b>	0.063	
<b>Robust CV</b>	37%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratory 18.

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

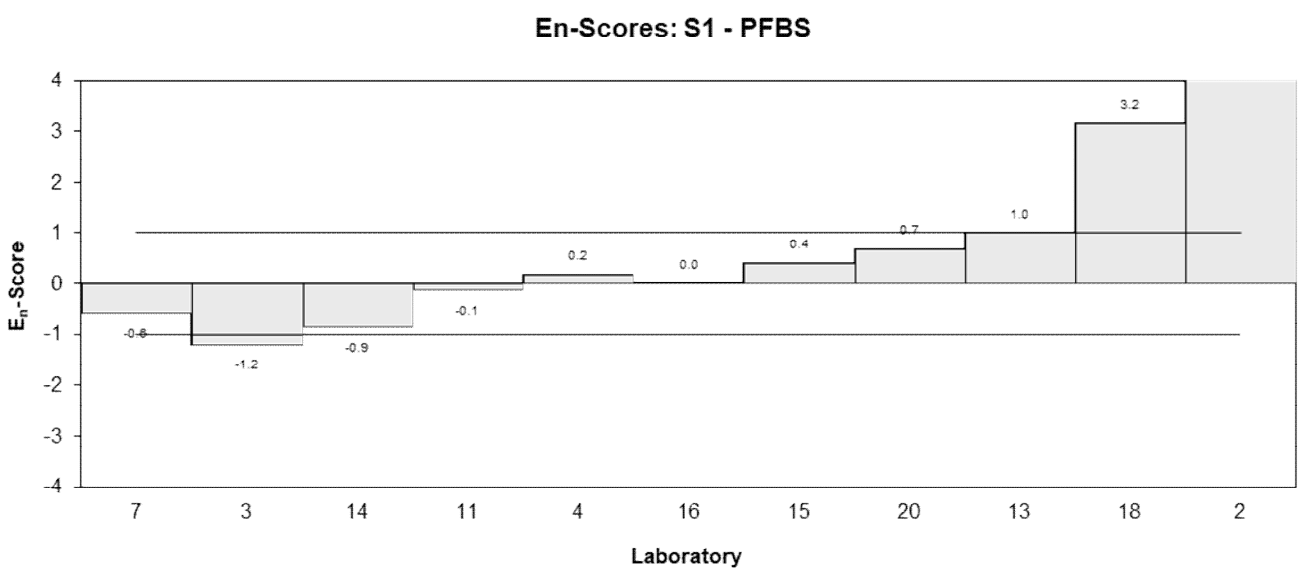
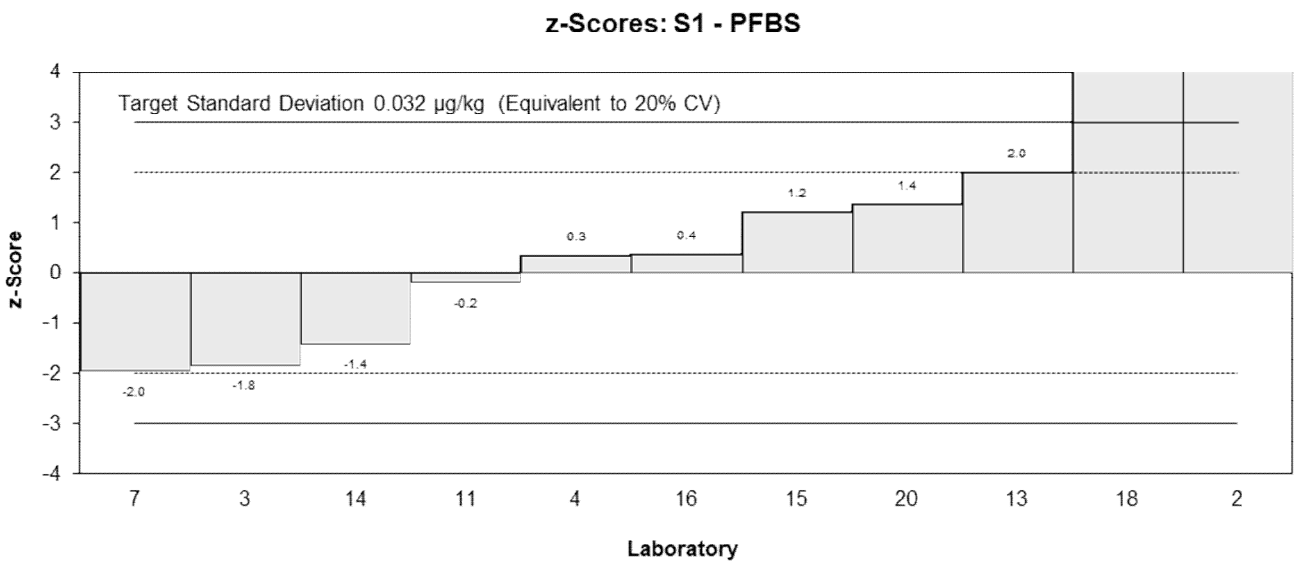
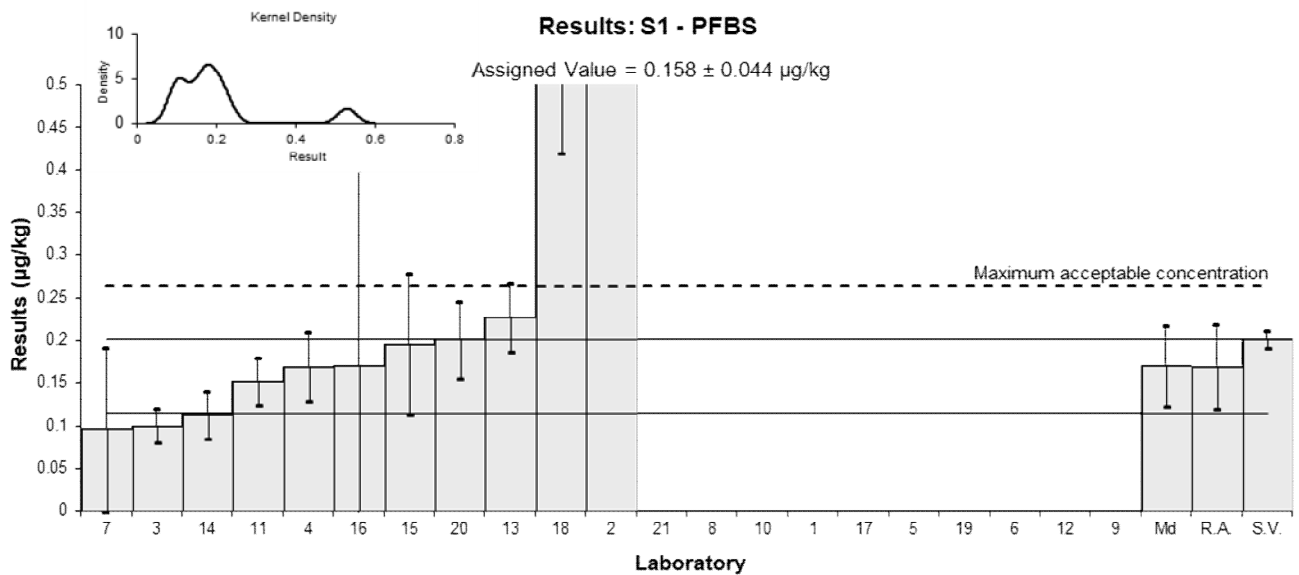


Figure 2

Table 6

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFHxS
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	5.8	1.16	74.1	0.42	0.34
2	19.24	0.43	NR	12.98	18.41
3	1.7	0.49	NR	-3.41	-4.62
4	5.71	2	NR	0.34	0.17
5	6	2	91	0.61	0.31
6	4.52	1.13	84	-0.78	-0.64
7	4.53	1.36	91	-0.77	-0.55
8	6.0	0.55	98	0.61	0.78
9	NT	NT	NT		
10	3.25	0.46	50	-1.96	-2.72
11	4.93	0.54	77	-0.39	-0.51
12	NT	NT	NT		
13	7.647	1.597	81.8	2.15	1.34
14	4.71	1.2	95	-0.60	-0.47
15	NT	NT	NT		
16	5.61	29.2	97.7	0.24	0.01
17	6.1	2.2	117	0.70	0.33
18	9.67	2.93	70	4.04	1.44
19	9.00	2.34	76	3.41	1.51
20	NR	NR	78		
21	4.95	1.49	55.7	-0.37	-0.25

## Statistics\*

<b>Assigned Value**</b>	5.35	0.62
<b>Spike</b>	6.53	0.33
<b>Robust Average</b>	5.6	1.1
<b>Median</b>	5.66	0.66
<b>Mean</b>	5.63	
<b>N</b>	16	
<b>Max.</b>	9.67	
<b>Min.</b>	1.7	
<b>Robust SD</b>	1.7	
<b>Robust CV</b>	30%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 3, 18 and 19.

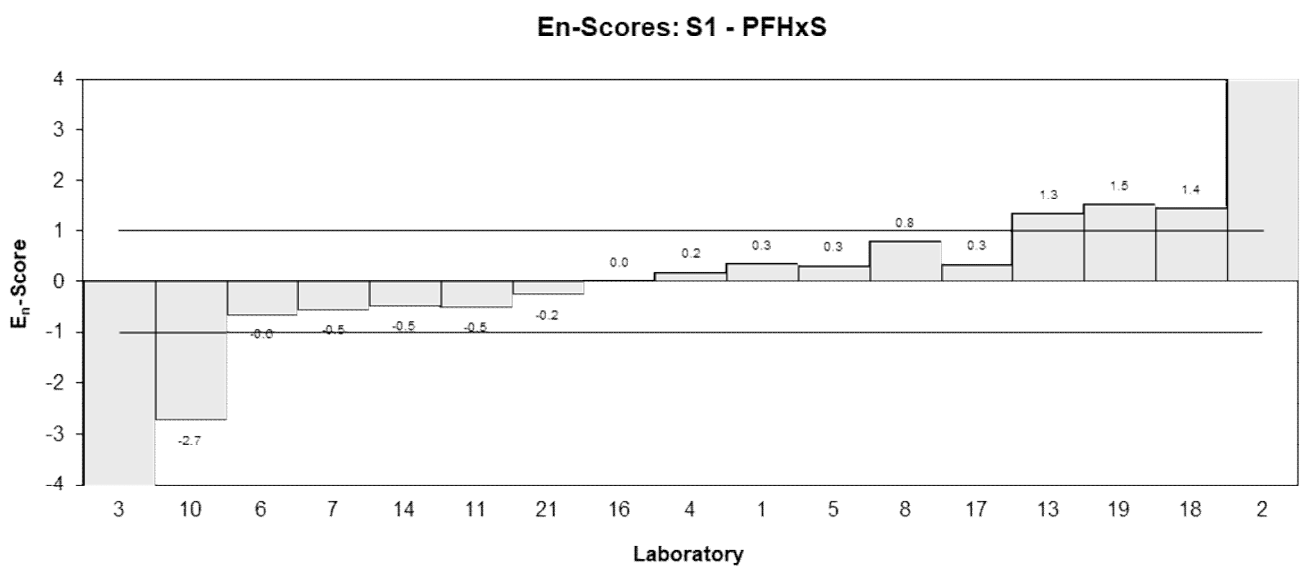
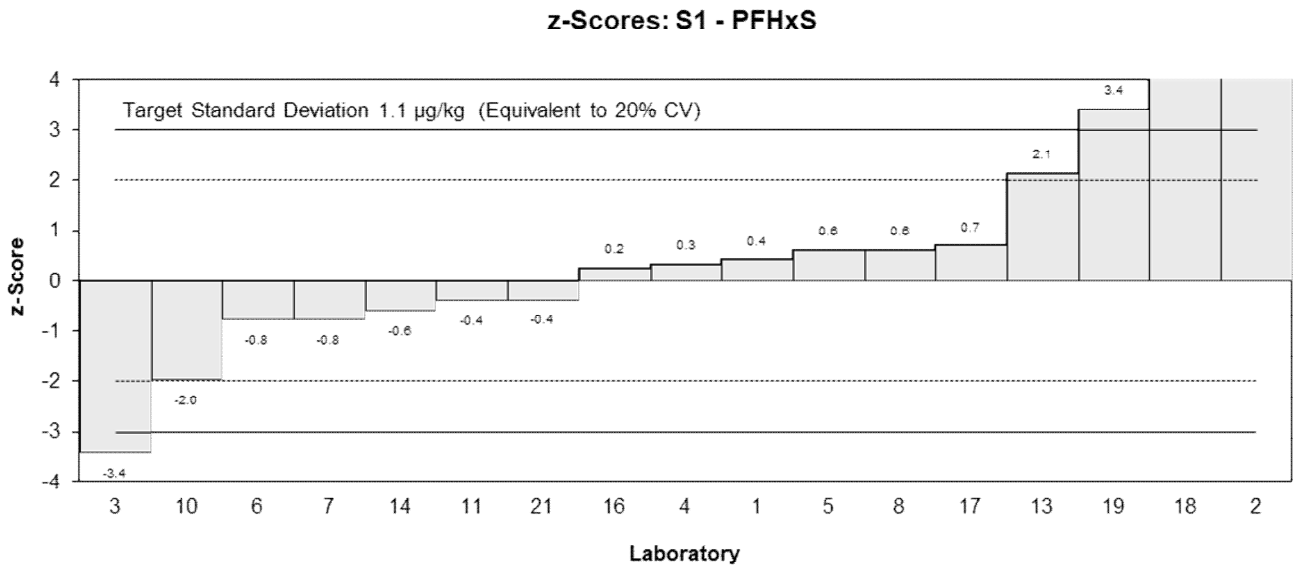
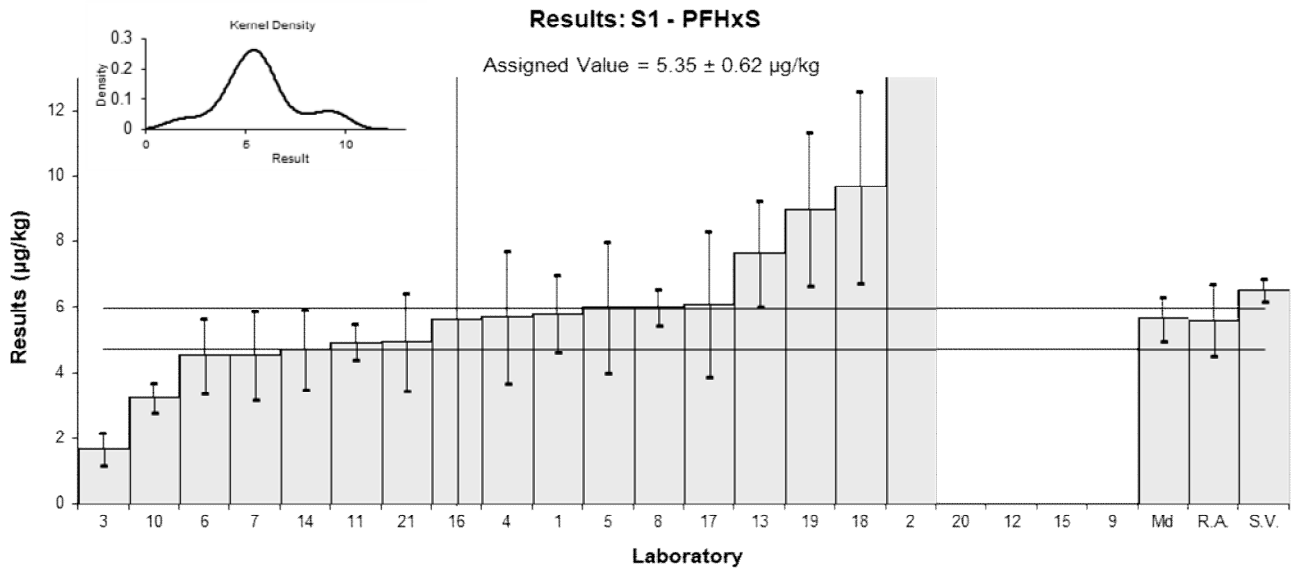


Figure 3

Table 7

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFHxS (linear)
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	NT	NT	NT		
2	19.03	0.39	69	12.99	15.76
3	NT	NT	NT		
4	5.37	2	77	0.08	0.04
5	6	2	91	0.67	0.33
6	4.52	1.13	NR	-0.73	-0.56
7	4.21	1.26	91	-1.02	-0.73
8	6.0	0.55	98	0.67	0.74
9	NT	NT	NT		
10	3.25	0.46	50	-1.93	-2.25
11	4.7	0.52	77	-0.56	-0.63
12	NT	NT	NT		
13	7.561	1.546	81.8	2.15	1.31
14	4.44	1.1	95	-0.80	-0.63
15	6.26	0.728	NR	0.92	0.91
16	5.53	29.2	97.7	0.23	0.01
17	NT	NT	NT		
18	9.21	2.67	70	3.71	1.41
19	8.87	2.31	76	3.38	1.47
20	6.3	2.2	78	0.95	0.43
21	4.91	1.47	55.7	-0.36	-0.23

## Statistics\*

<b>Assigned Value**</b>	5.29	0.78
<b>Spike</b>	6.39	0.32
<b>Robust Average</b>	5.7	1.1
<b>Median</b>	5.53	0.68
<b>Mean</b>	5.81	
<b>N</b>	15	
<b>Max.</b>	9.21	
<b>Min.</b>	3.25	
<b>Robust SD</b>	1.6	
<b>Robust CV</b>	29%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 18 and 19.

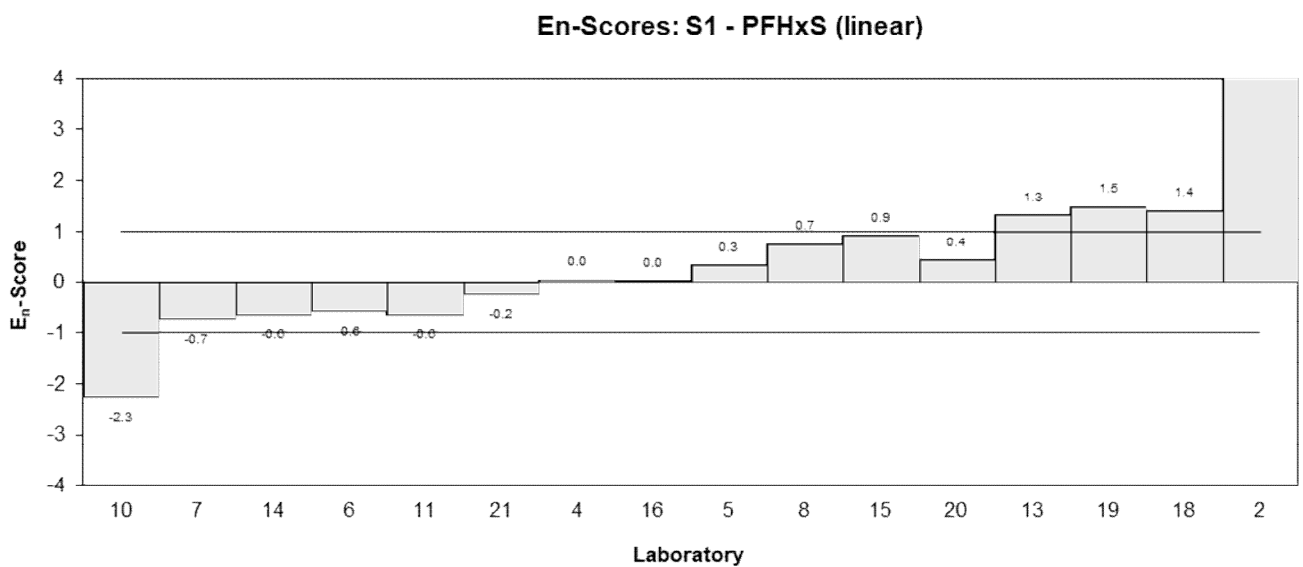
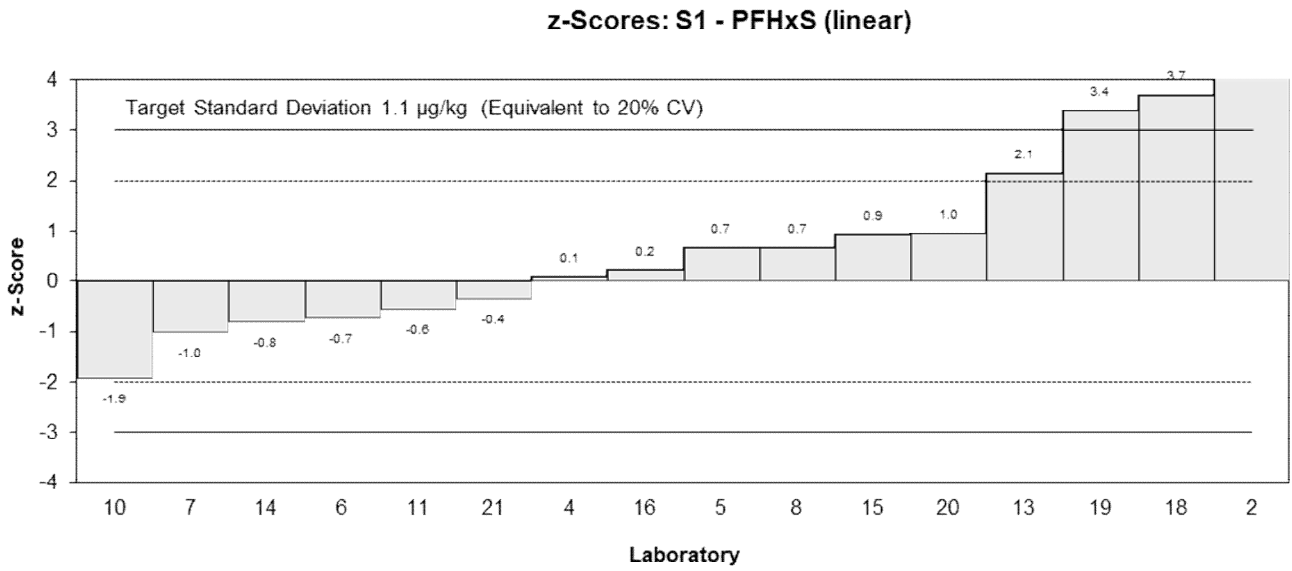
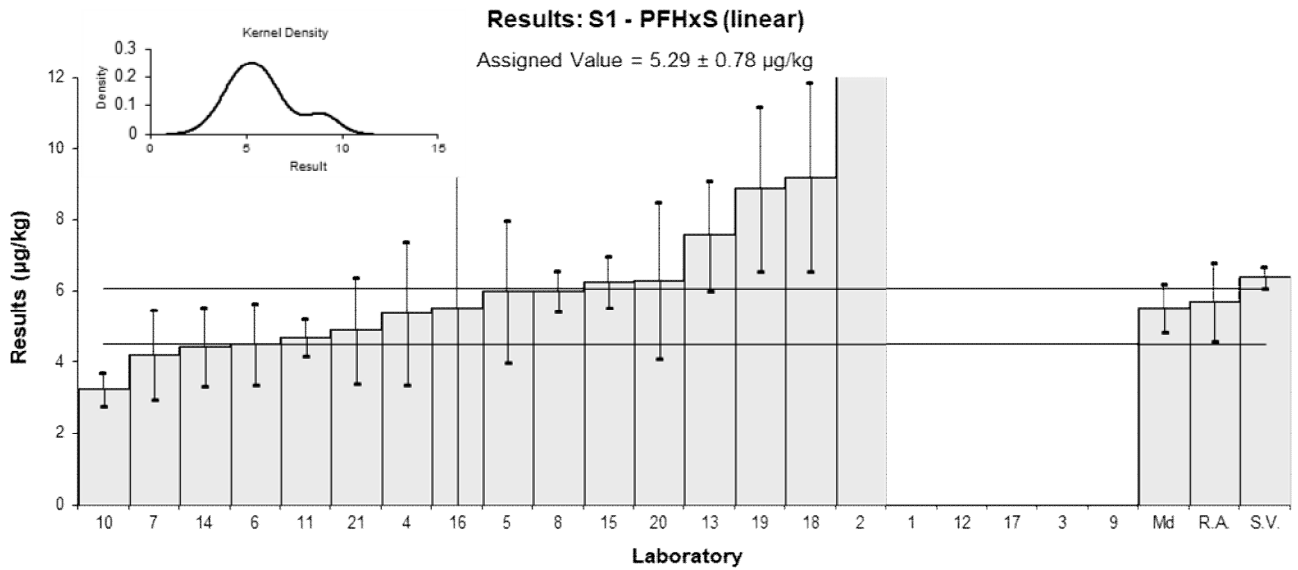


Figure 4

Table 8

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFHpS
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	4.5	NR	74.1	0.14	0.24
2	11.94	1.49	NR	8.63	4.81
3	3.7	0.52	NR	-0.78	-0.94
4	5.18	1	NR	0.91	0.72
5	5	2	97	0.71	0.30
6	2.52	0.63	NR	-2.12	-2.31
7	3.78	1.13	91	-0.68	-0.49
8	4.7	0.76	98	0.37	0.35
9	NT	NT	NT		
10	1.78	0.49	NR	-2.97	-3.71
11	4.48	0.97	77	0.11	0.09
12	NT	NT	NT		
13***	6.245	1.047	81.8	2.00	1.00
14	4.04	1	95	-0.39	-0.30
15	4.865	0.554	NR	0.55	0.65
16	4.2	28.8	105	-0.21	-0.01
17	5.0	1.7	131	0.71	0.35
18	3.90	0.26	NR	-0.55	-0.85
19	7.68	1.77	76	3.77	1.79
20	4.8	1.3	78	0.48	0.30
21	2.56	0.77	55.7	-2.08	-1.98

## Statistics\*

<b>Assigned Value**</b>	4.38	0.50
<b>Spike</b>	5.54	0.28
<b>Max. Acceptable Conc.***</b>	7.29	
<b>Robust Average</b>	4.34	0.72
<b>Median</b>	4.49	0.41
<b>Mean</b>	4.39	
<b>N</b>	18	
<b>Max.</b>	7.68	
<b>Min.</b>	1.78	
<b>Robust SD</b>	1.2	
<b>Robust CV</b>	28%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 10 and 19.

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



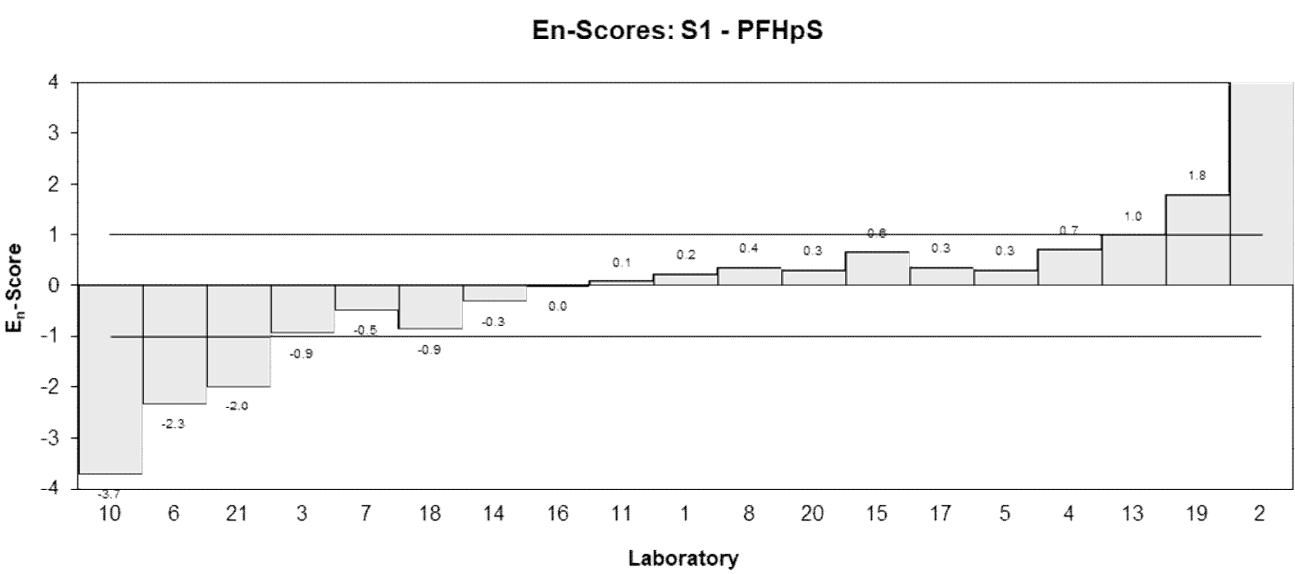
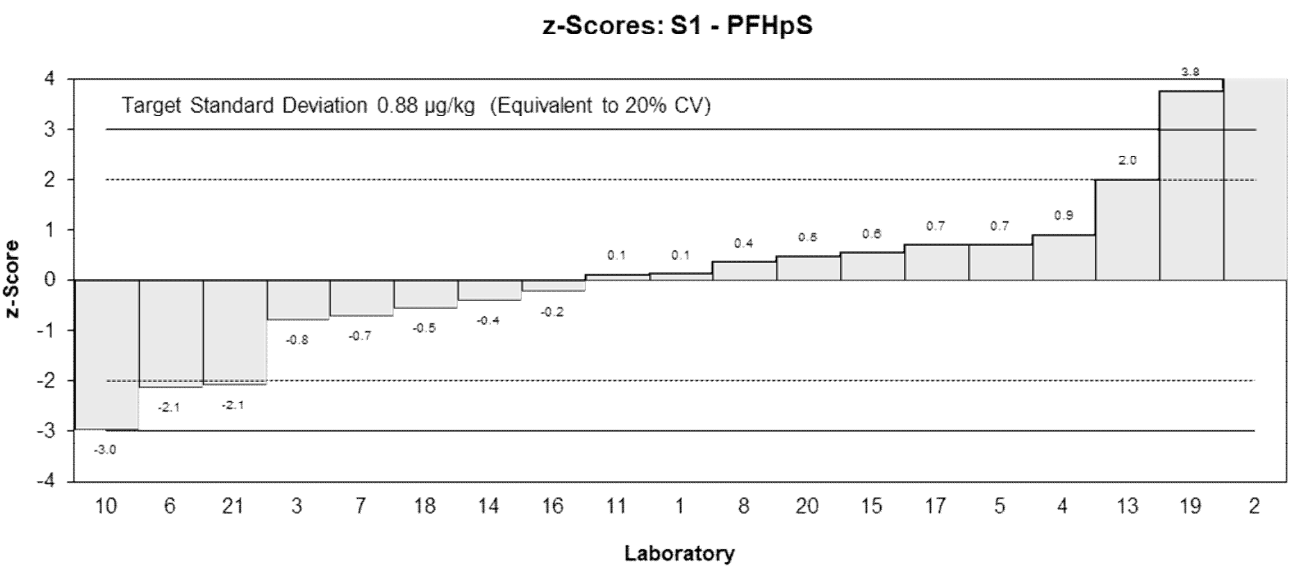
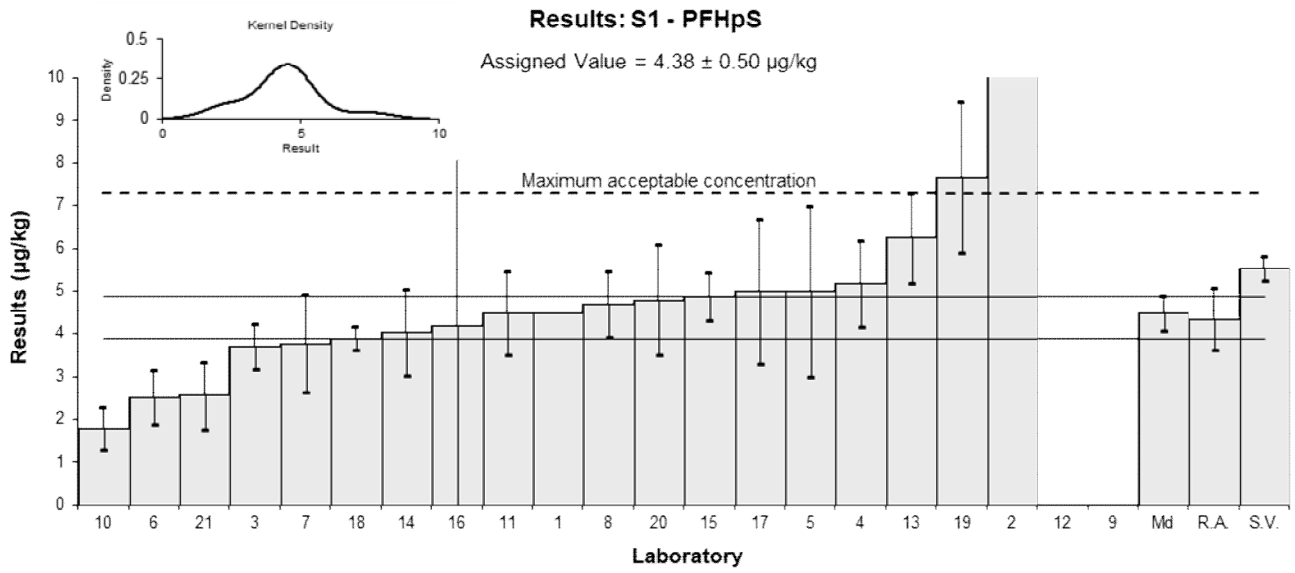


Figure 5

Table 9

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFOS
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	9.16	3.30676	74.1	0.26	0.13
2	33.1	1.39	NR	14.02	13.77
3	4.0	0.58	NR	-2.70	-3.78
4	8.3	2	NR	-0.23	-0.18
5	10	3	97	0.75	0.41
6	6.6	1.73	NR	-1.21	-1.02
7	6.68	2.00	86	-1.16	-0.88
8	9.2	0.31	94	0.29	0.44
9	NT	NT	NT		
10	6.95	0.92	63	-1.01	-1.22
11	10.1	5.5	71	0.80	0.25
12	NT	NT	NT		
13***	12.641	2.371	90.7	2.00	1.00
14	7.28	1.8	95	-0.82	-0.67
15	9.117	2.452	NR	0.24	0.16
16	9.03	23.4	105	0.19	0.01
17	9.8	4.2	131	0.63	0.25
18***	14.32	4.94	93	2.00	1.00
19***	14.6	4.2	84	2.00	1.00
20	9.9	2.6	76.4	0.69	0.43
21	6.89	2.07	69.8	-1.04	-0.77

## Statistics\*

<b>Assigned Value**</b>	8.7	1.1
<b>Spike</b>	12.3	0.6
<b>Max. Acceptable Conc.***</b>	15.8	
<b>Robust Average</b>	9.0	1.5
<b>Median</b>	9.1	1.0
<b>Mean</b>	9.1	
<b>N</b>	18	
<b>Max.</b>	14.6	
<b>Min.</b>	4	
<b>Robust SD</b>	2.5	
<b>Robust CV</b>	28%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 3, 18 and 19.

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

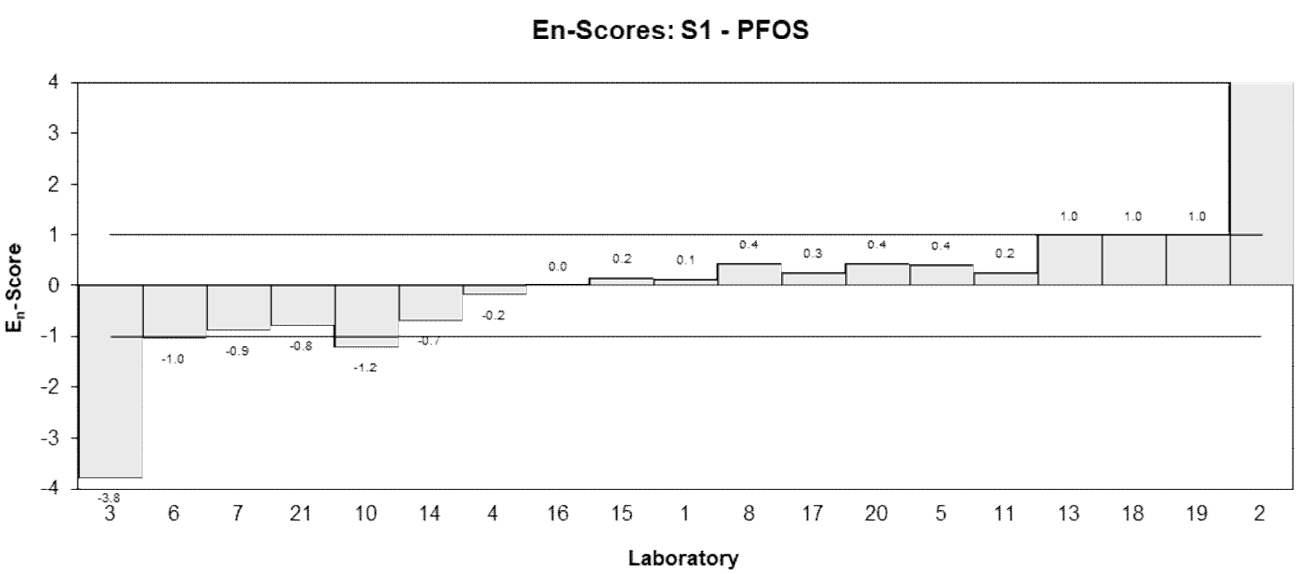
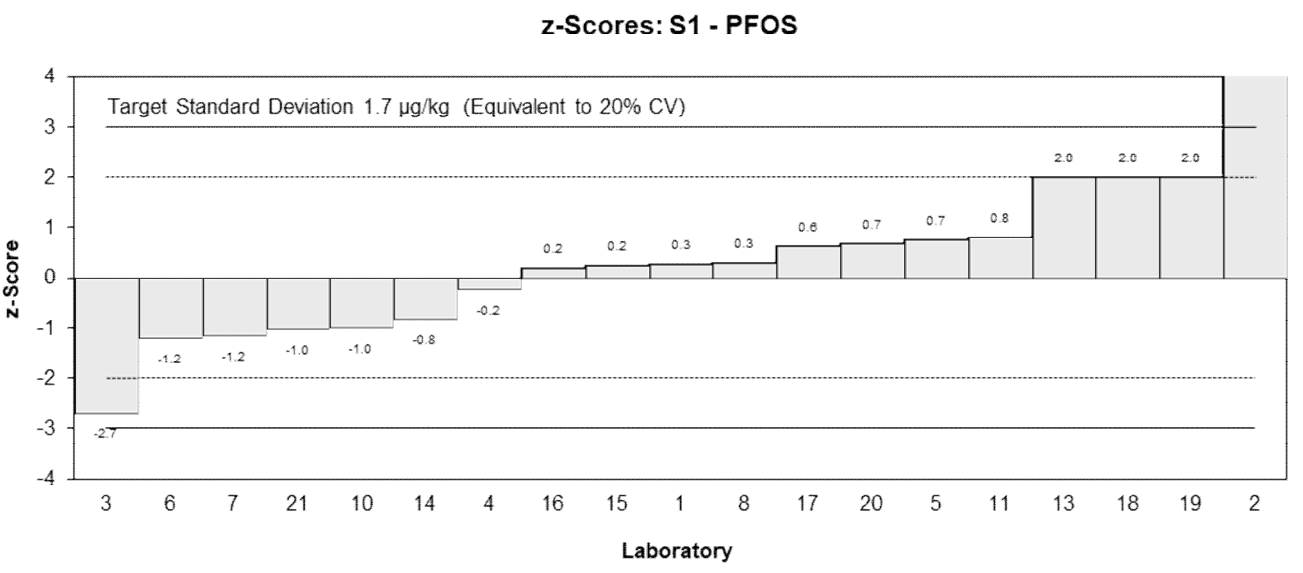
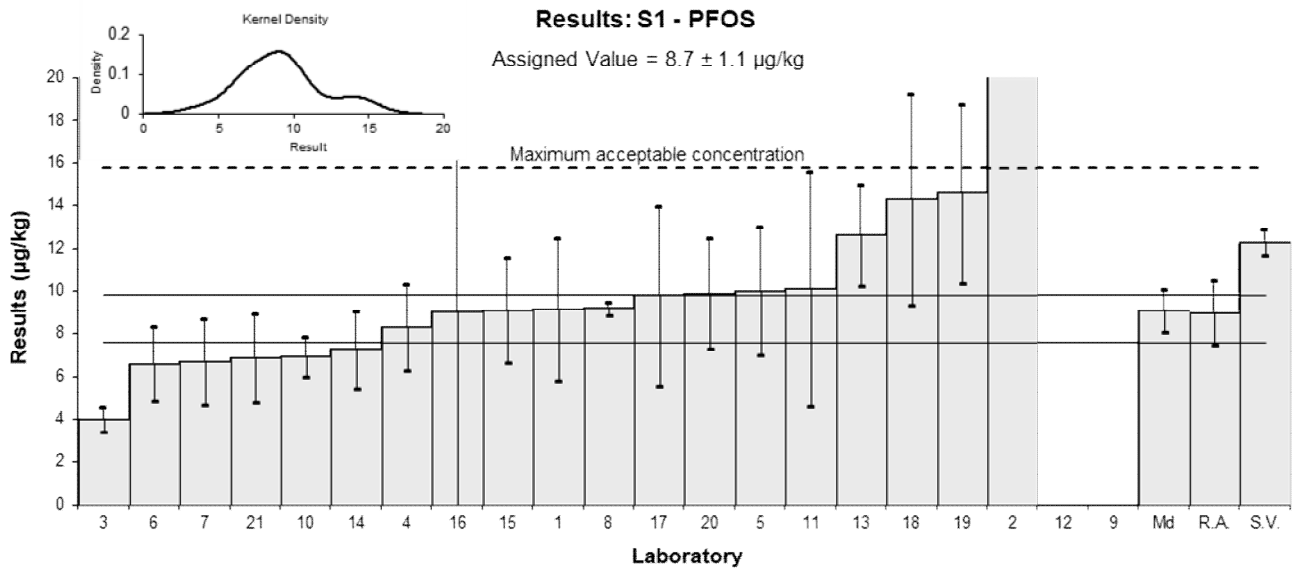


Figure 6

Table 10

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFOS (linear)
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	6.57	NR	NR	0.39	0.68
2	23.16	3.19	69	13.98	5.23
3	NT	NT	NT		
4	6.21	2	80	0.09	0.05
5	6	2	97	-0.08	-0.05
6	4.52	1.24	86	-1.30	-1.11
7	5.43	1.63	86	-0.55	-0.38
8	6.4	0.42	94	0.25	0.37
9	NT	NT	NT		
10	4.68	0.62	63	-1.16	-1.53
11	6.74	3.6	71	0.52	0.17
12	NT	NT	NT		
13	8.231	1.143	90.7	1.75	1.60
14	5.79	1.5	95	-0.25	-0.19
15	7.019	2.384	NR	0.75	0.37
16	6.34	23.4	105	0.20	0.01
17	6.2	2.6	131	0.08	0.04
18	11.61	4.55	93	4.52	1.20
19***	10.12	2.93	84	2.00	1.00
20	7.2	1.6	76.4	0.90	0.63
21	4.68	1.4	69.8	-1.16	-0.91

## Statistics\*

<b>Assigned Value**</b>	6.10	0.69
<b>Spike</b>	8.54	0.43
<b>Max. Acceptable Conc.***</b>	11.0	
<b>Robust Average</b>	6.41	0.85
<b>Median</b>	6.34	0.52
<b>Mean</b>	6.69	
<b>N</b>	17	
<b>Max.</b>	11.61	
<b>Min.</b>	4.52	
<b>Robust SD</b>	1.4	
<b>Robust CV</b>	22%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 18 and 19.

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

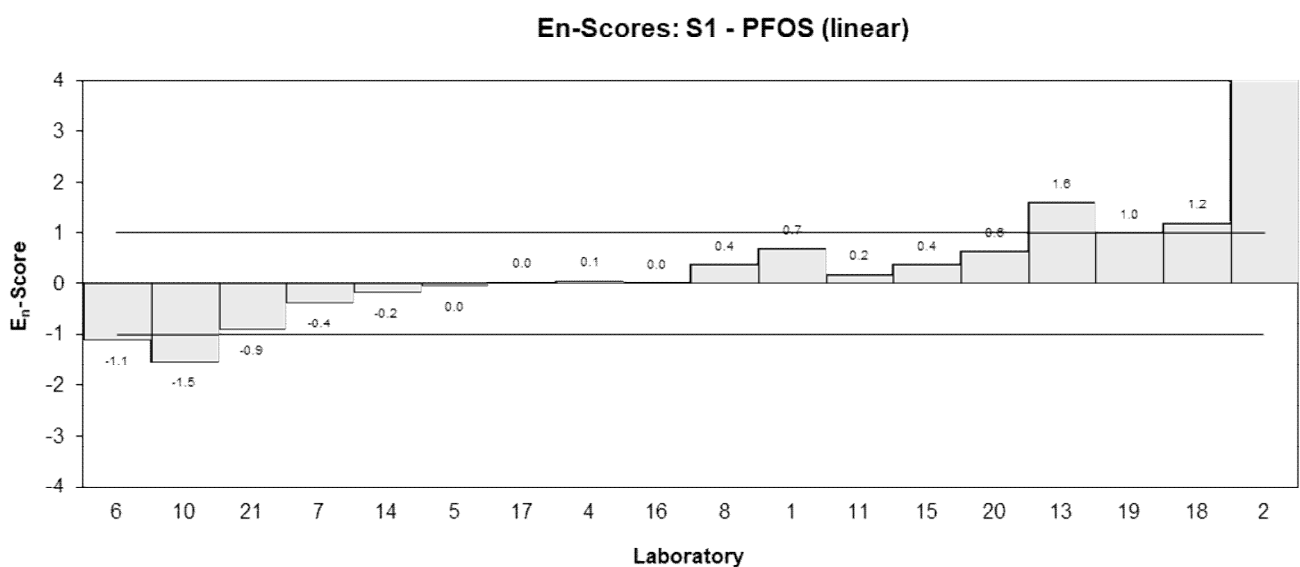
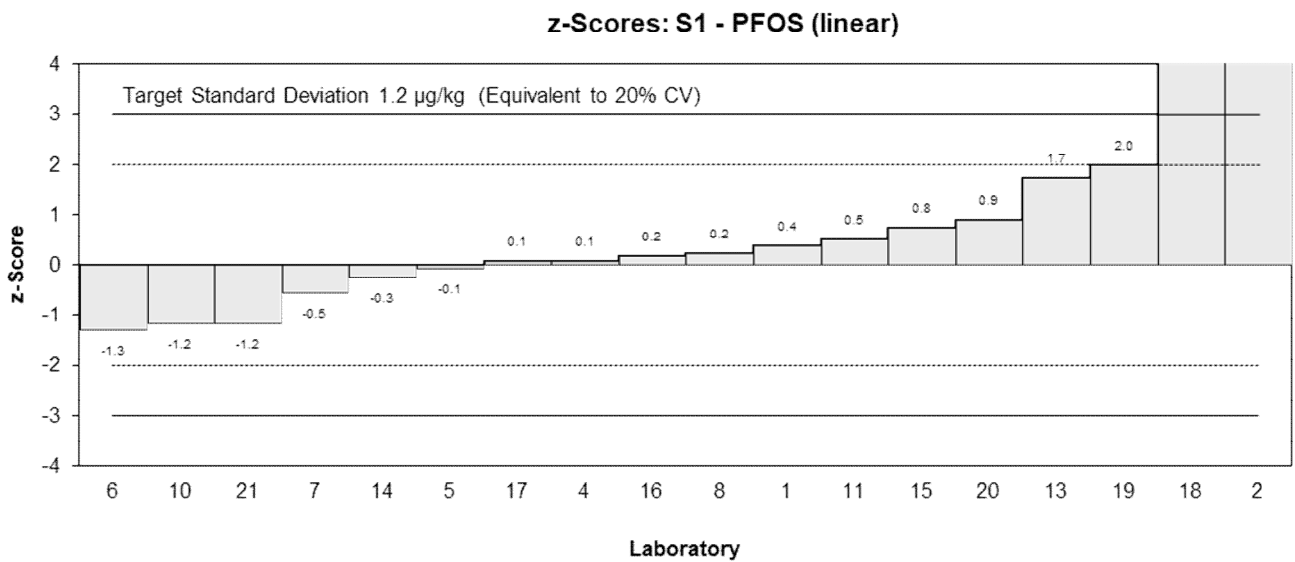
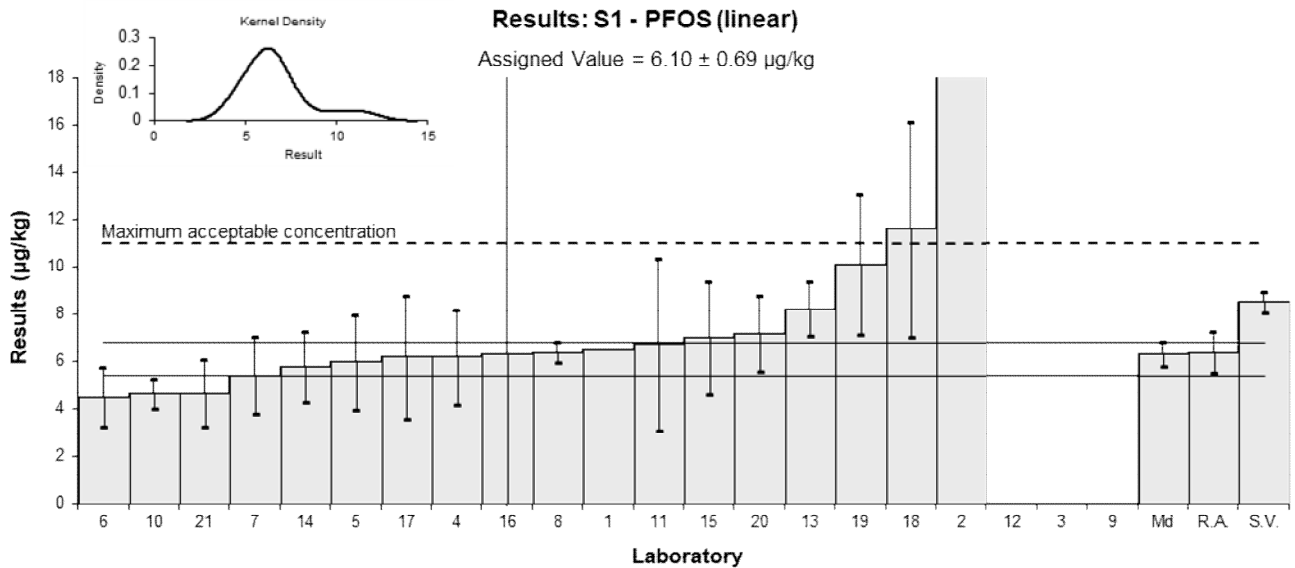


Figure 7

Table 11

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFBA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	1.8	0.1584	74.1	5.59	4.09
2	6.67	0.50	63	34.24	11.02
3	0.6	0.29	NR	-1.47	-0.74
4	1.01	0.3	79	0.94	0.46
5	<1	NR	96		
6	NR	NR	118		
7	0.749	0.224	75	-0.59	-0.36
8	< 1.0	NR	106		
9	NT	NT	NT		
10	0.81	0.11	62	-0.24	-0.20
11	1.38	0.32	56	3.12	1.46
12	NT	NT	NT		
13	0.900	0.048	45.1	0.29	0.28
14	0.737	0.18	95	-0.66	-0.46
15	5.583	1.332	NR	27.84	3.52
16	0.996	24.4	101	0.86	0.01
17	<5	NR	137		
18	0.594	0.073	27	-1.51	-1.38
19	1.57	0.71	86	4.24	0.99
20	<0.366	NR	94.5		
21	0.938	0.28	44.1	0.52	0.27

## Statistics\*

<b>Assigned Value**</b>	0.85	0.17
<b>Spike</b>	0.983	0.049
<b>Robust Average</b>	0.99	0.28
<b>Median</b>	0.92	0.17
<b>Mean</b>	1.0	
<b>N</b>	12	
<b>Max.</b>	1.8	
<b>Min.</b>	0.594	
<b>Robust SD</b>	0.39	
<b>Robust CV</b>	40%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received. Laboratory 15 was also omitted from all statistical calculations.

\*\* Robust average excluding Laboratories 1 and 19.

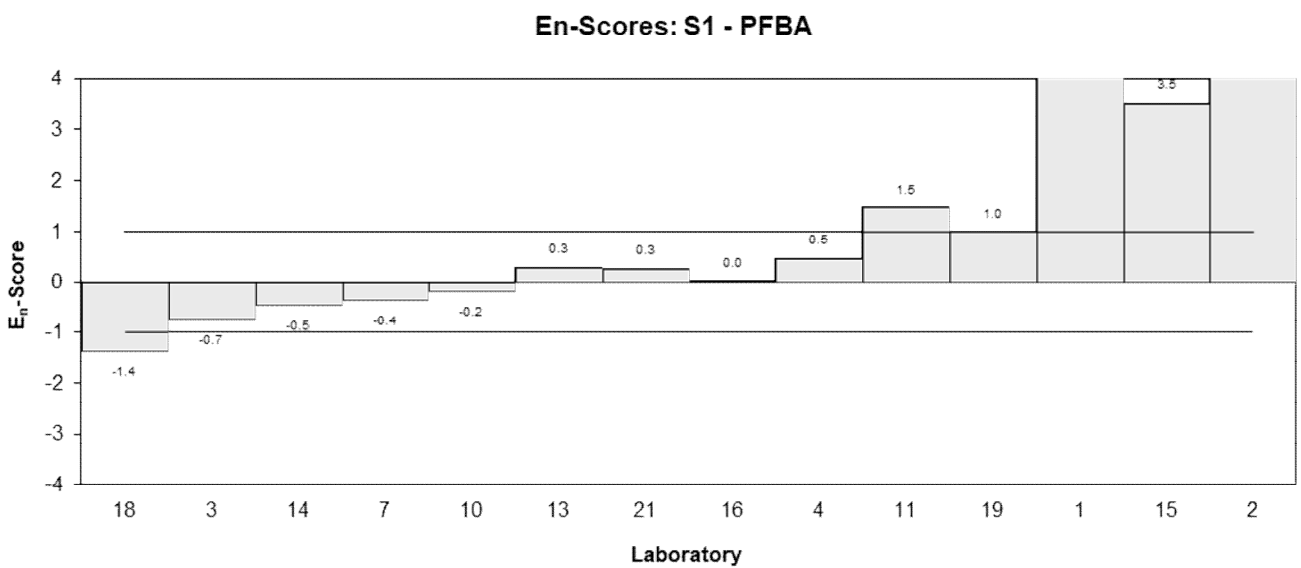
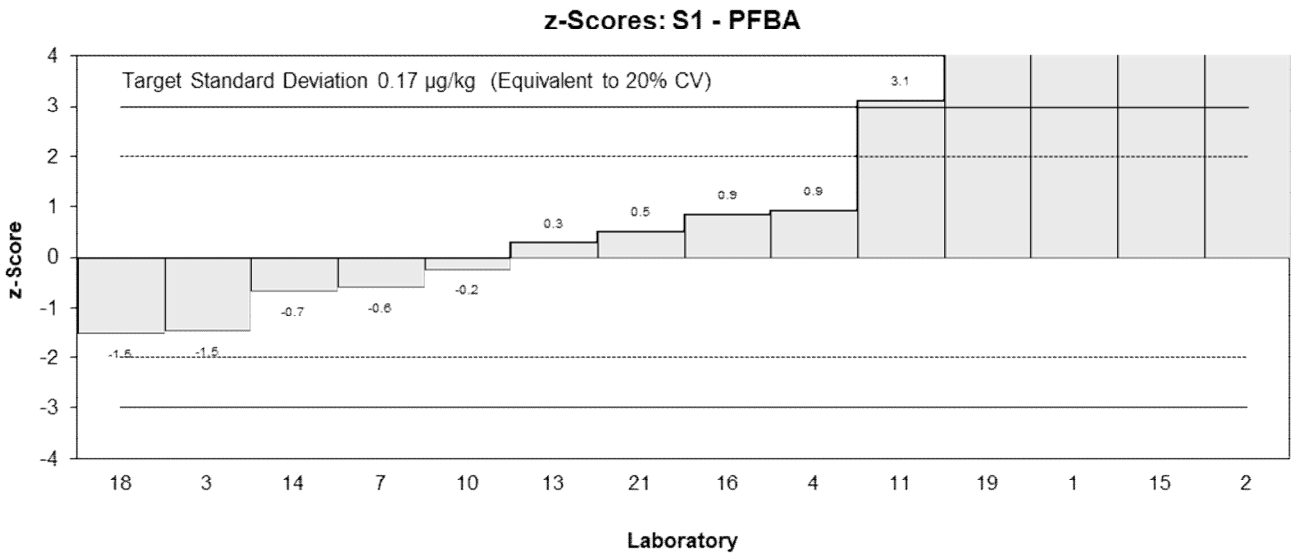
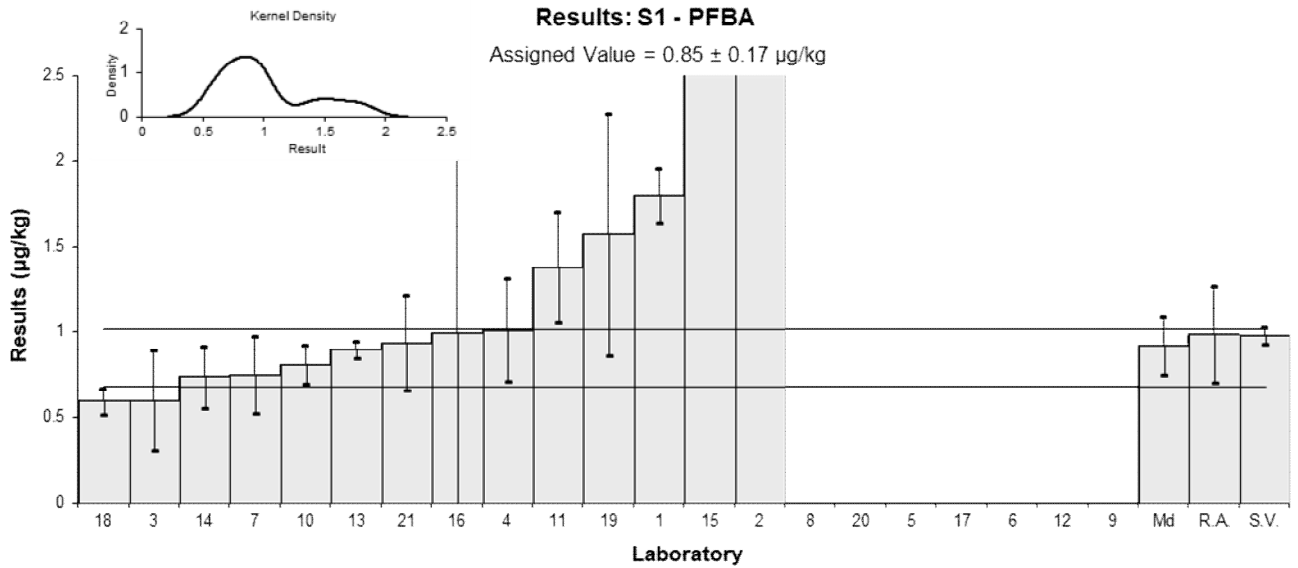


Figure 8

Table 12

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFPeA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	1	0.14	74.1	0.62	0.50
2	2.93	0.04	70	11.46	11.68
3	0.6	0.13	NR	-1.63	-1.36
4	1.01	0.3	73	0.67	0.35
5	<1	NR	77		
6	NR	NR	86		
7	0.693	0.208	79	-1.11	-0.73
8	< 1.0	NR	100		
9	NT	NT	NT		
10	0.82	0.11	53	-0.39	-0.35
11	0.895	0.08	58	0.03	0.03
12	NT	NT	NT		
13	1.141	0.110	49.4	1.41	1.24
14	0.722	0.18	95	-0.94	-0.68
15	1.356	0.253	NR	2.62	1.53
16	0.867	18	104	-0.13	0.00
17	<2	NR	123		
18	0.467	0.040	62	-2.38	-2.42
19	1.53	0.38	149	3.60	1.54
20	0.93	0.15	75.7	0.22	0.18
21	1.06	0.32	50.5	0.96	0.47

## Statistics\*

<b>Assigned Value**</b>	0.89	0.17
<b>Spike</b>	1.01	0.05
<b>Robust Average</b>	0.92	0.20
<b>Median</b>	0.91	0.14
<b>Mean</b>	0.94	
<b>N</b>	14	
<b>Max.</b>	1.53	
<b>Min.</b>	0.467	
<b>Robust SD</b>	0.29	
<b>Robust CV</b>	32%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratory 19.



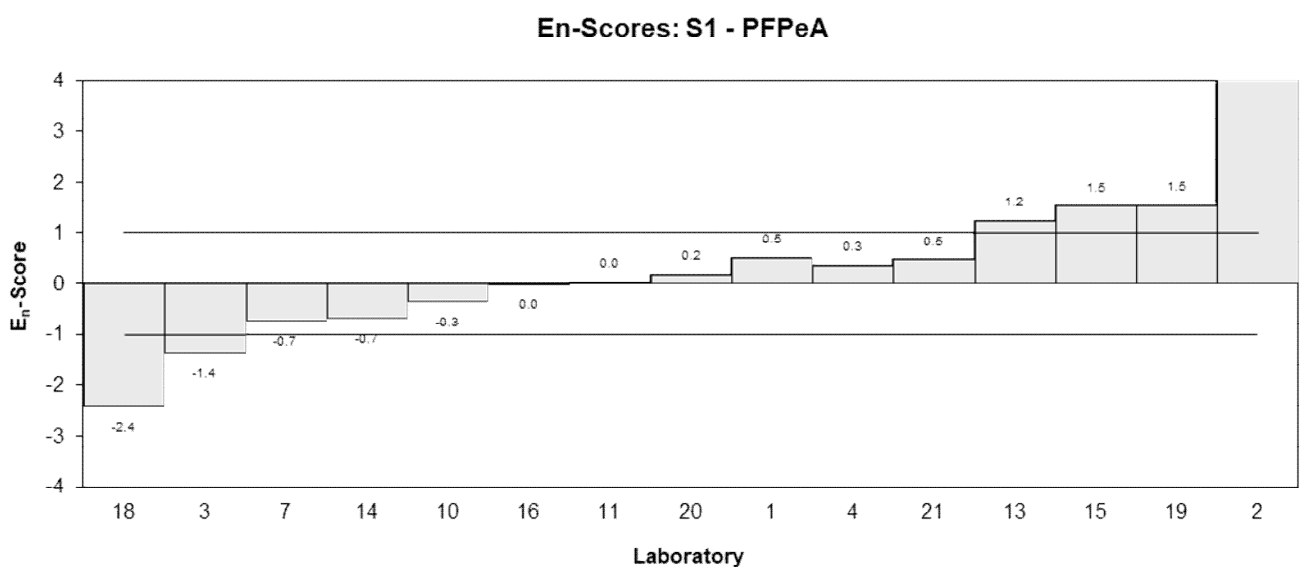
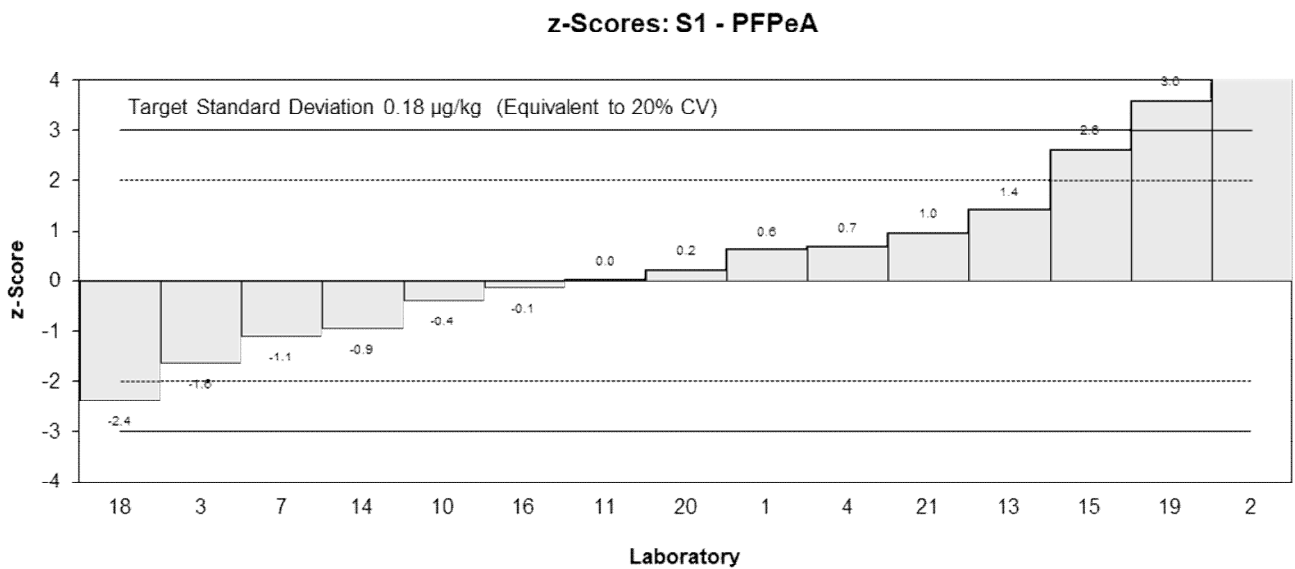
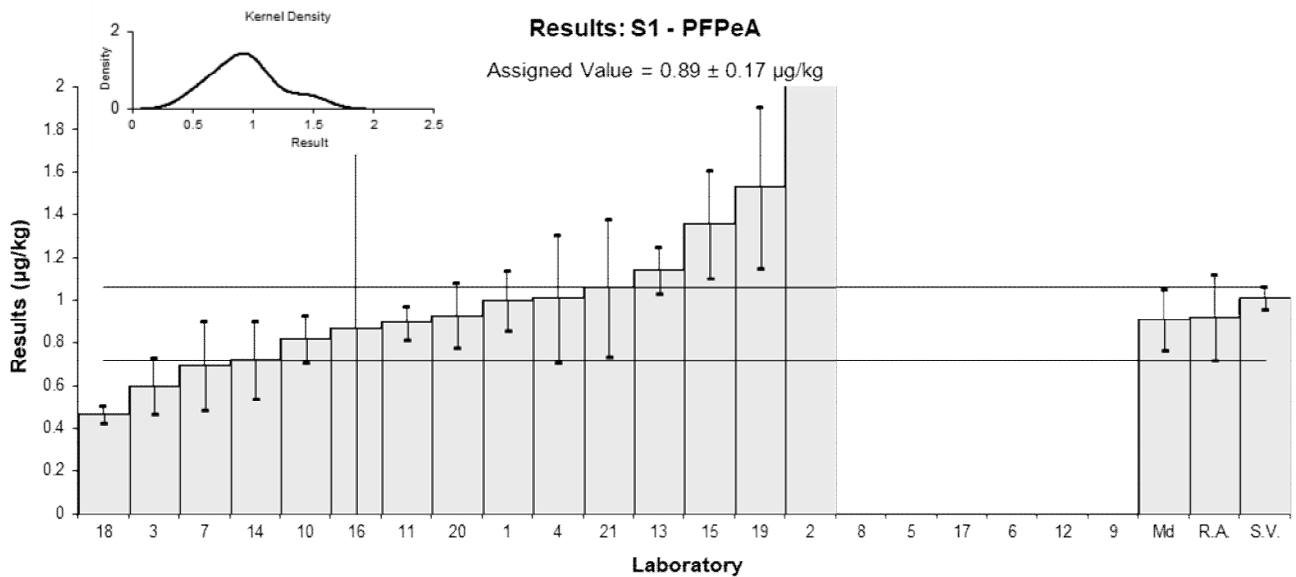


Figure 9

Table 13

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFHxA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	0.9	NR	74.1	-0.91	-1.33
2	3.75	0.24	61	12.05	9.36
3	0.6	0.15	NR	-2.27	-2.36
4	1.2	0.3	76	0.45	0.30
5	1	1	100	-0.45	-0.10
6	0.86	0.22	87	-1.09	-0.90
7	0.963	0.289	82	-0.62	-0.42
8	1.2	0.13	100	0.45	0.50
9	NT	NT	NT		
10	0.97	0.15	58	-0.59	-0.61
11	1.09	0.17	72	-0.05	-0.04
12	NT	NT	NT		
13	1.469	0.323	59.5	1.68	1.04
14	1.02	0.25	95	-0.36	-0.27
15	1.481	0.232	NR	1.73	1.38
16	1.14	37.5	101	0.18	0.00
17	1.4	0.5	130	1.36	0.57
18	0.831	0.016	60	-1.22	-1.78
19	2.08	0.60	81	4.45	1.58
20	1.27	0.35	77.1	0.77	0.45
21	1.24	0.37	67.1	0.64	0.35

## Statistics\*

<b>Assigned Value**</b>	1.10	0.15
<b>Spike</b>	1.19	0.06
<b>Robust Average</b>	1.13	0.16
<b>Median</b>	1.12	0.11
<b>Mean</b>	1.15	
<b>N</b>	18	
<b>Max.</b>	2.08	
<b>Min.</b>	0.6	
<b>Robust SD</b>	0.27	
<b>Robust CV</b>	24%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratory 19.

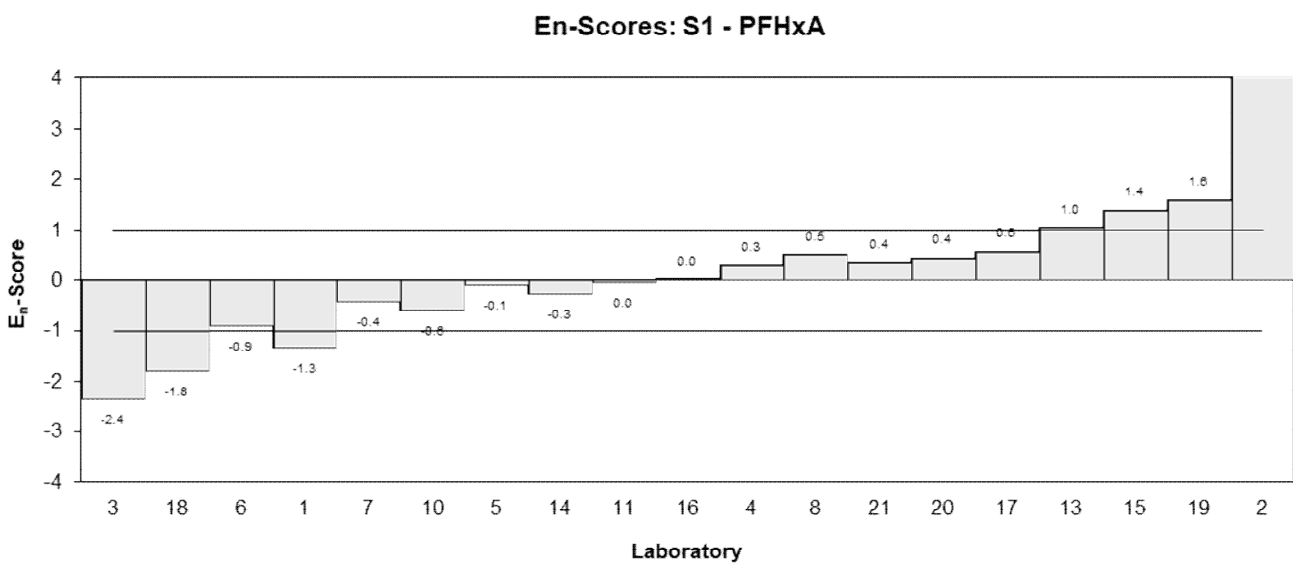
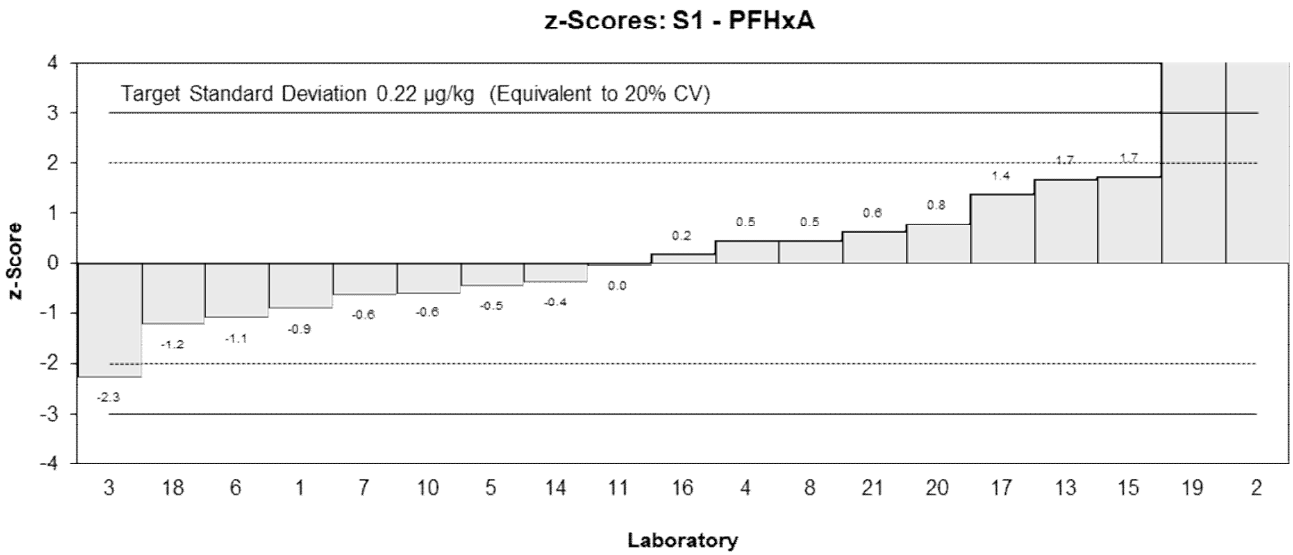
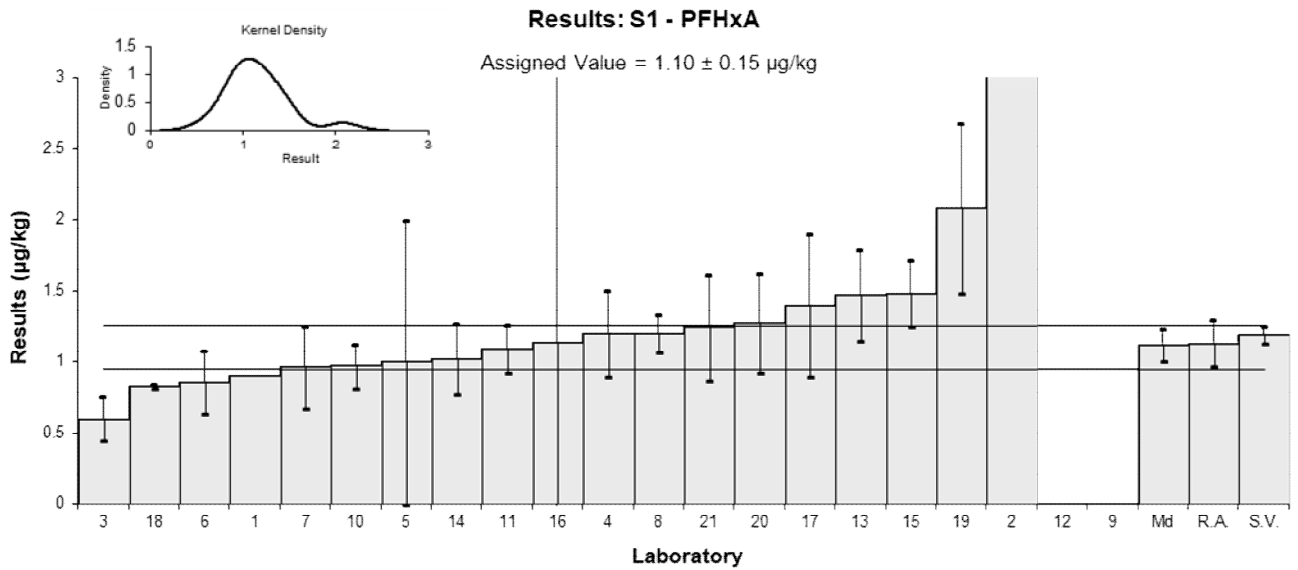


Figure 10

Table 14

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFHpA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	1.7	0.2584	74.1	0.28	0.27
2	5.93	0.002	59	13.42	19.64
3	1.3	0.10	NR	-0.96	-1.28
4	1.83	0.5	73	0.68	0.40
5	2	1	93	1.21	0.38
6	1.27	0.32	95	-1.06	-0.88
7	1.19	0.357	84	-1.30	-1.00
8	1.8	0.17	97	0.59	0.68
9	NT	NT	NT		
10	0.97	0.15	62	-1.99	-2.40
11	1.62	0.28	76	0.03	0.03
12	NT	NT	NT		
13	2.299	0.435	67.8	2.14	1.41
14	1.37	0.34	95	-0.75	-0.59
15	1.582	0.213	NR	-0.09	-0.09
16	1.69	32.5	110	0.25	0.00
17	1.9	0.7	125	0.90	0.40
18	1.20	0.005	77	-1.27	-1.86
19	3.37	1.01	81	5.47	1.70
20	1.89	0.53	79.3	0.87	0.49
21	1.75	0.53	64.6	0.43	0.24

## Statistics\*

<b>Assigned Value**</b>	1.61	0.22
<b>Spike</b>	1.92	0.10
<b>Robust Average</b>	1.65	0.24
<b>Median</b>	1.70	0.19
<b>Mean</b>	1.71	
<b>N</b>	18	
<b>Max.</b>	3.37	
<b>Min.</b>	0.97	
<b>Robust SD</b>	0.41	
<b>Robust CV</b>	25%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratory 19.

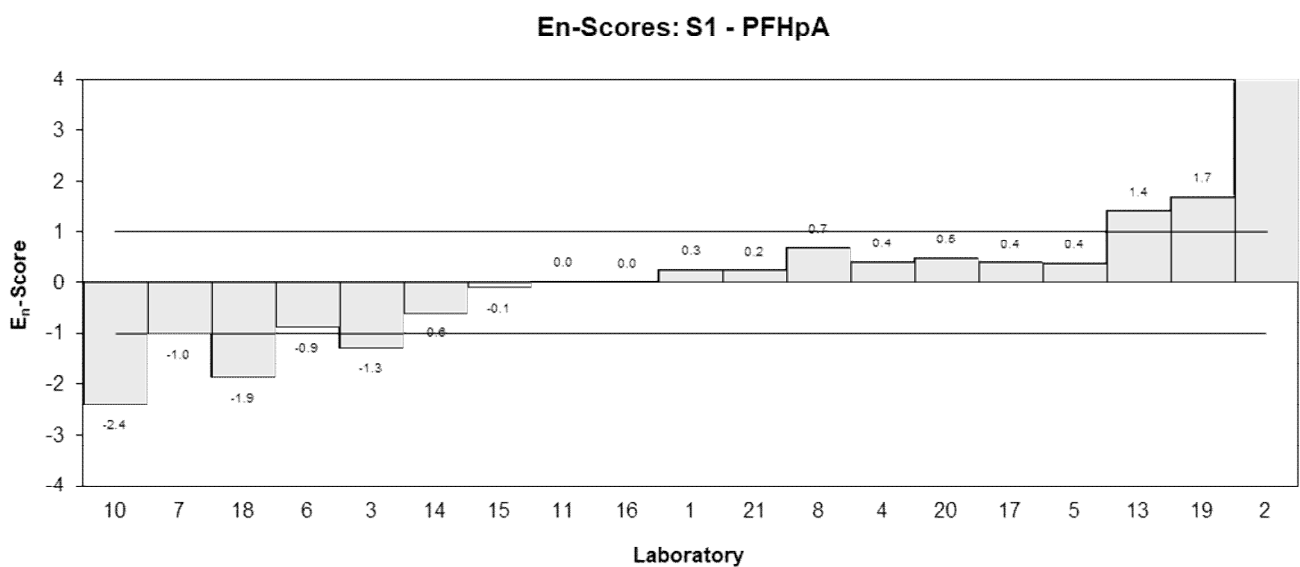
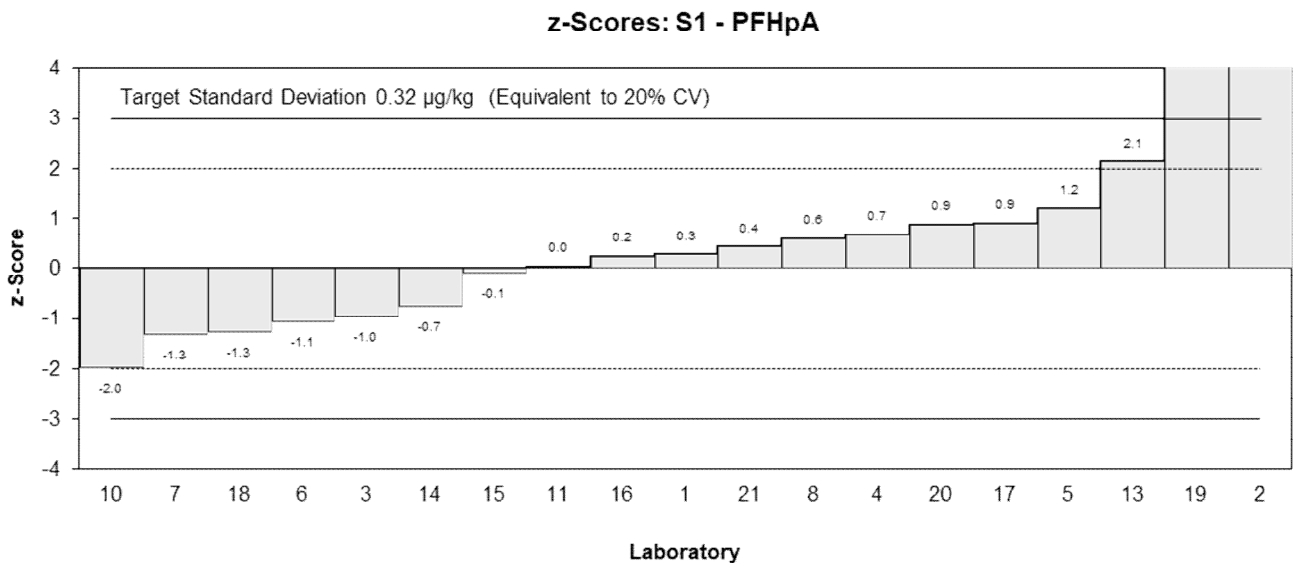
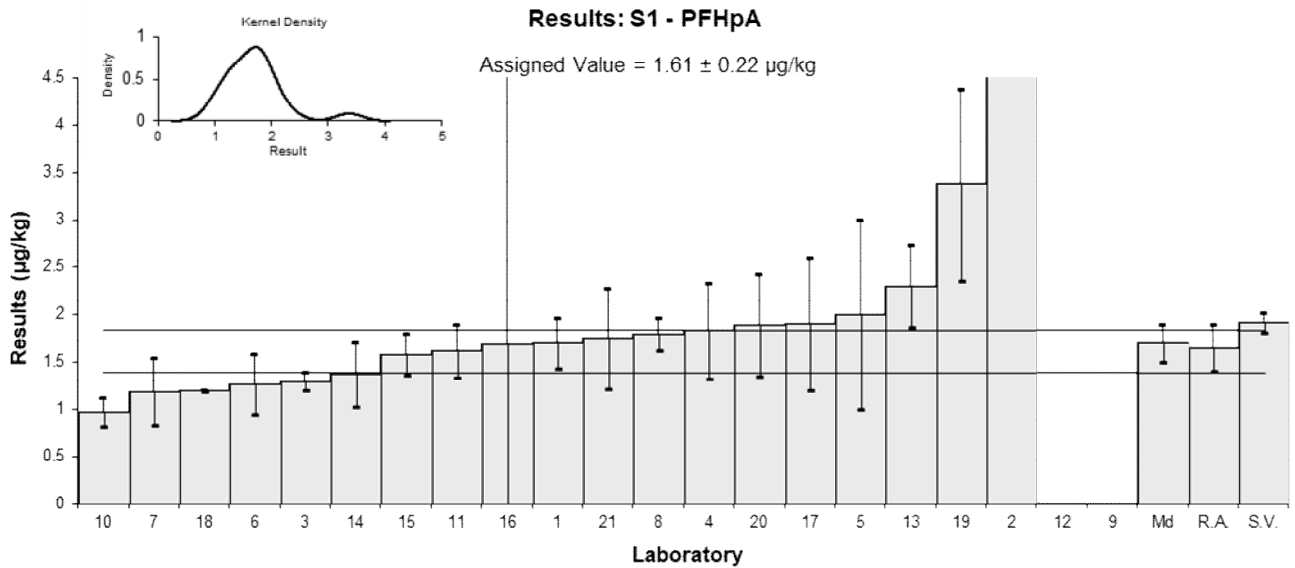


Figure 11

Table 15

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFOA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	3.5	0.7455	74.1	-0.48	-0.42
2	11.81	0.86	61	10.26	8.10
3	NT	NT	NT		
4	4.01	1	81	0.18	0.13
5	4	2	105	0.17	0.06
6	2.98	0.75	77	-1.15	-1.01
7	2.86	0.858	86	-1.30	-1.03
8	4.0	0.77	98	0.17	0.14
9	NT	NT	NT		
10	1.65	0.31	56	-2.87	-3.94
11	3.61	0.48	77	-0.34	-0.39
12	NT	NT	NT		
13	4.825	1.186	77.4	1.23	0.75
14	3.04	0.76	95	-1.07	-0.93
15	4.122	0.433	NR	0.33	0.39
16	3.79	29	106	-0.10	0.00
17	4.5	1.6	122	0.81	0.38
18	5.00	0.852	89	1.46	1.16
19	6.56	1.77	85	3.48	1.47
20	4.44	0.84	77.3	0.74	0.59
21	3.44	1.03	72	-0.56	-0.38

## Statistics\*

<b>Assigned Value**</b>	3.87	0.47
<b>Spike</b>	4.94	0.25
<b>Robust Average</b>	3.87	0.52
<b>Median</b>	4.00	0.38
<b>Mean</b>	3.90	
<b>N</b>	17	
<b>Max.</b>	6.56	
<b>Min.</b>	1.65	
<b>Robust SD</b>	0.86	
<b>Robust CV</b>	22%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 10 and 19.

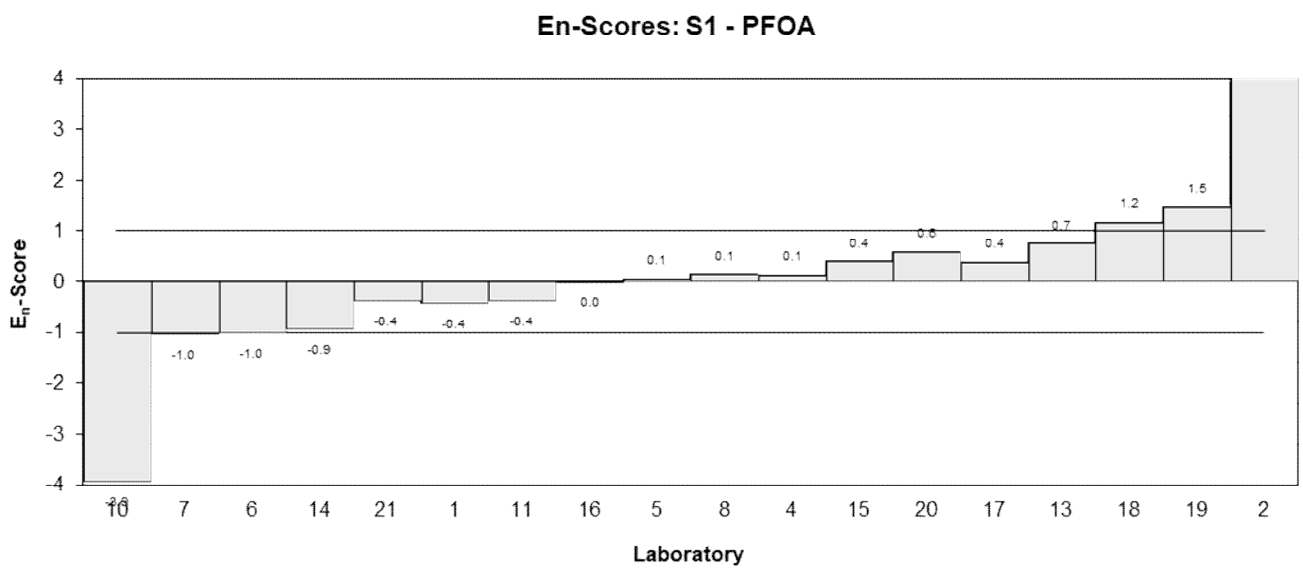
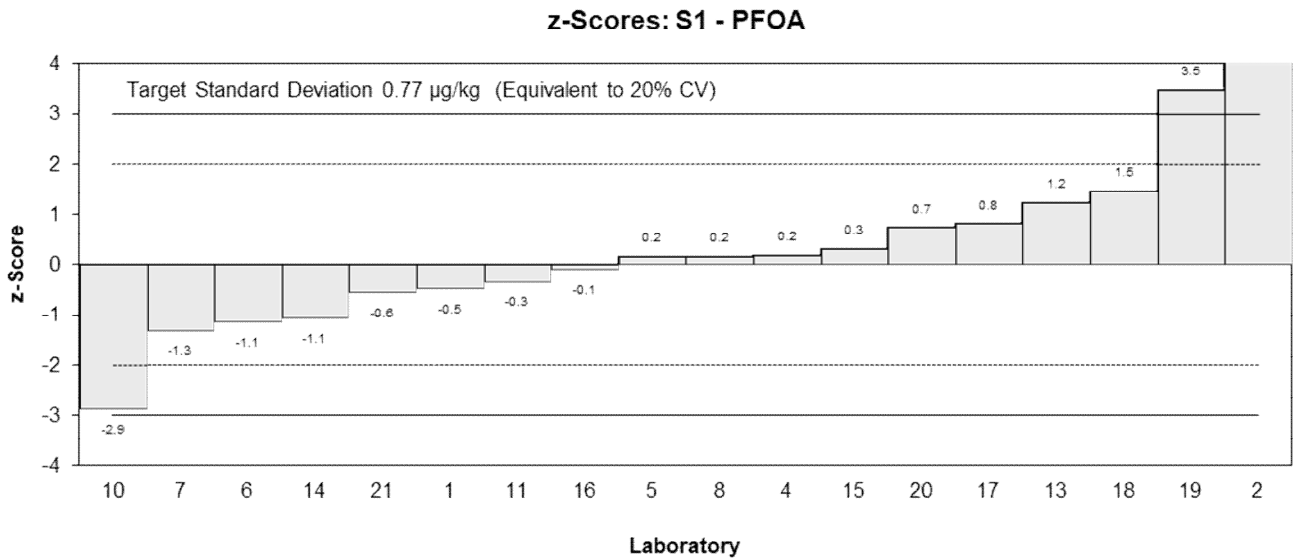
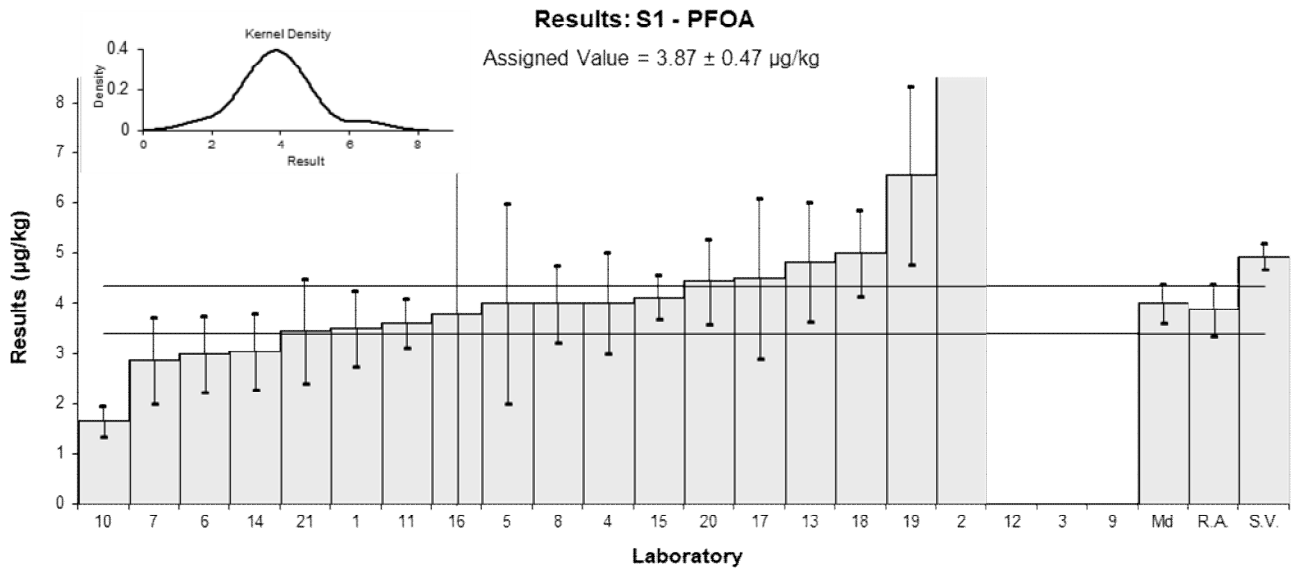


Figure 12

Table 16

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFNA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	1	0.15	74.1	1.41	1.04
2	2.26	0.02	52	9.49	9.78
3	0.5	0.04	NR	-1.79	-1.80
4	0.899	0.2	77	0.76	0.48
5	<1	NR	96		
6	0.57	0.14	83	-1.35	-1.02
7	0.588	0.176	88	-1.23	-0.83
8	< 1.0	NR	99		
9	NT	NT	NT		
10	0.32	0.05	69	-2.95	-2.91
11	0.801	0.163	82	0.13	0.09
12	NT	NT	NT		
13	1.045	0.278	73.5	1.70	0.84
14	0.629	0.16	95	-0.97	-0.69
15	0.868	0.206	NR	0.56	0.35
16	0.958	21.2	97.1	1.14	0.01
17	1.0	0.4	144	1.41	0.51
18	0.534	0.046	120	-1.58	-1.57
19	1.63	0.47	101	5.45	1.72
20	0.93	0.28	75.4	0.96	0.47
21	0.608	0.18	91.7	-1.10	-0.73

## Statistics\*

<b>Assigned Value**</b>	0.78	0.15
<b>Spike</b>	0.976	0.049
<b>Robust Average</b>	0.78	0.17
<b>Median</b>	0.83	0.16
<b>Mean</b>	0.81	
<b>N</b>	16	
<b>Max.</b>	1.63	
<b>Min.</b>	0.32	
<b>Robust SD</b>	0.27	
<b>Robust CV</b>	34%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 10 and 19.



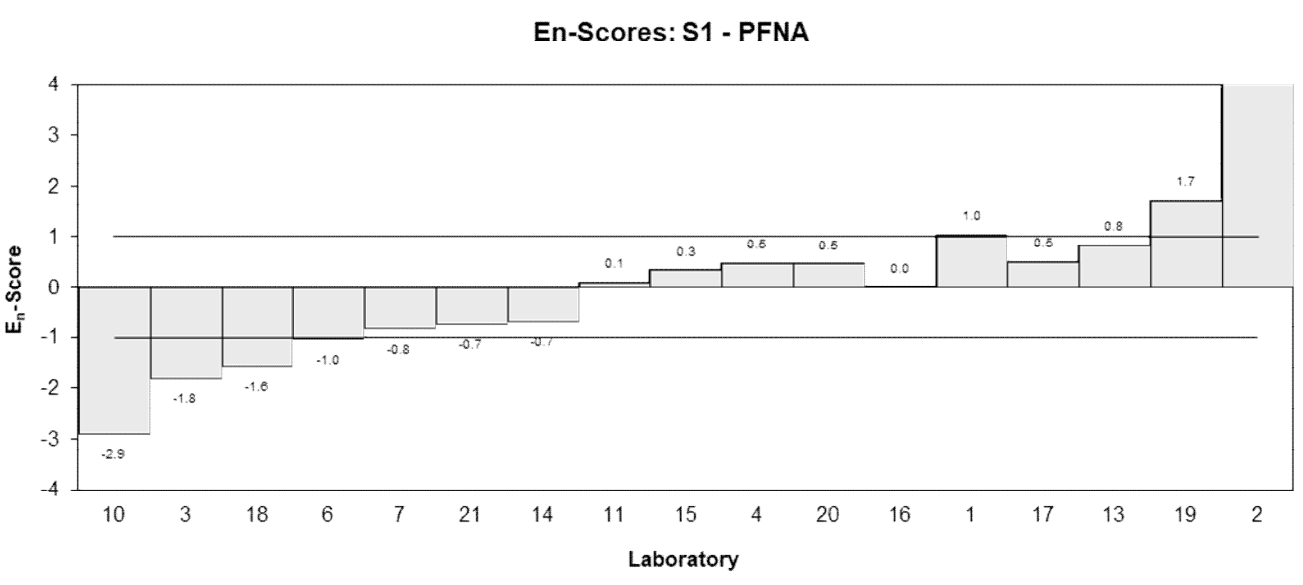
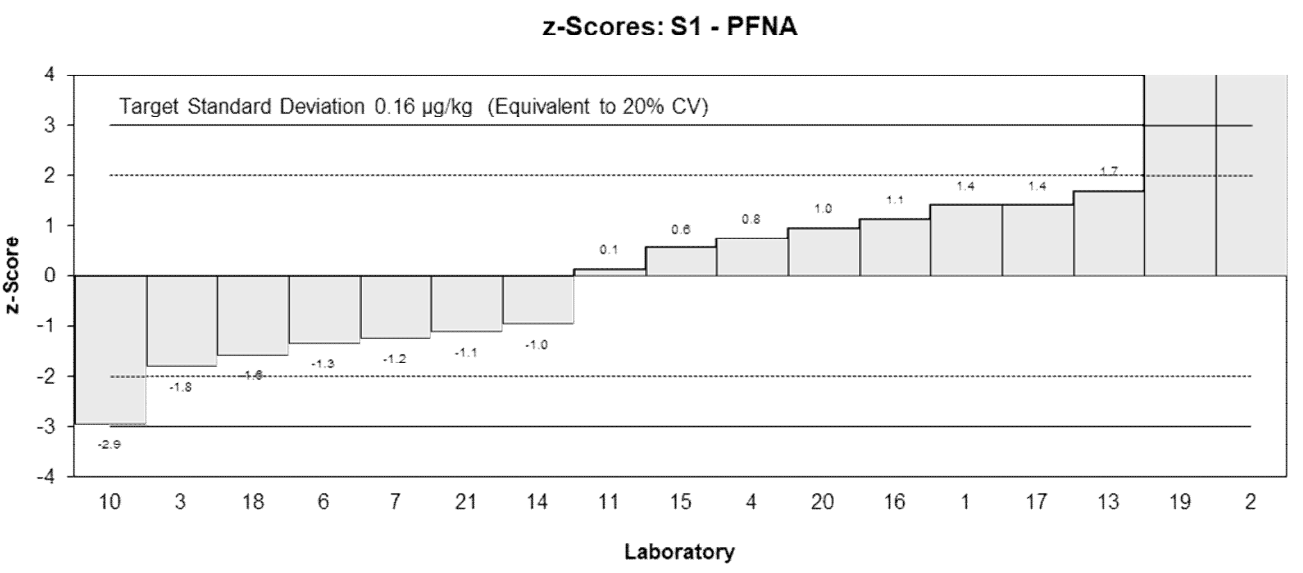
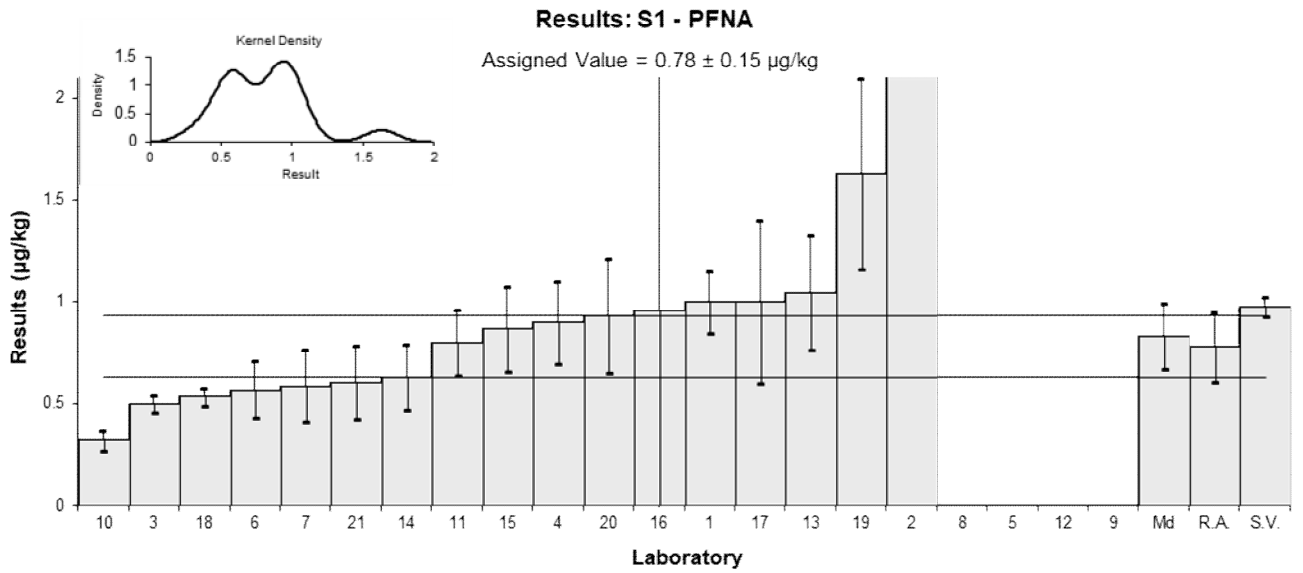


Figure 13

Table 17

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFDA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	<0.5	NR	74.1		
2	1.19	0.04	75	10.10	9.87
3	0.2	0.02	NR	-2.46	-2.66
4	0.47	0.1	73	0.96	0.62
5	<2	NR	100		
6	0.28	0.07	76	-1.45	-1.15
7	0.321	0.0970	83	-0.93	-0.61
8	< 1.0	NR	90		
9	NT	NT	NT		
10	<0.2	0.04	80		
11	0.390	0.076	77	-0.05	-0.04
12	NT	NT	NT		
13	0.653	0.264	77.7	3.29	0.95
14	0.315	0.081	95	-1.00	-0.74
15	0.447	0.319	NR	0.67	0.16
16	0.421	22.8	99.4	0.34	0.00
17	<1	NR	155		
18	NR	NR	130		
19	0.881	0.238	73	6.18	1.96
20	0.49	0.13	73.1	1.22	0.65
21	0.409	0.12	55.8	0.19	0.11

## Statistics\*

<b>Assigned Value**</b>	0.394	0.070
<b>Spike</b>	0.488	0.024
<b>Robust Average</b>	0.42	0.11
<b>Median</b>	0.415	0.080
<b>Mean</b>	0.440	
<b>N</b>	12	
<b>Max.</b>	0.881	
<b>Min.</b>	0.2	
<b>Robust SD</b>	0.16	
<b>Robust CV</b>	37%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 3, 13 and 19.

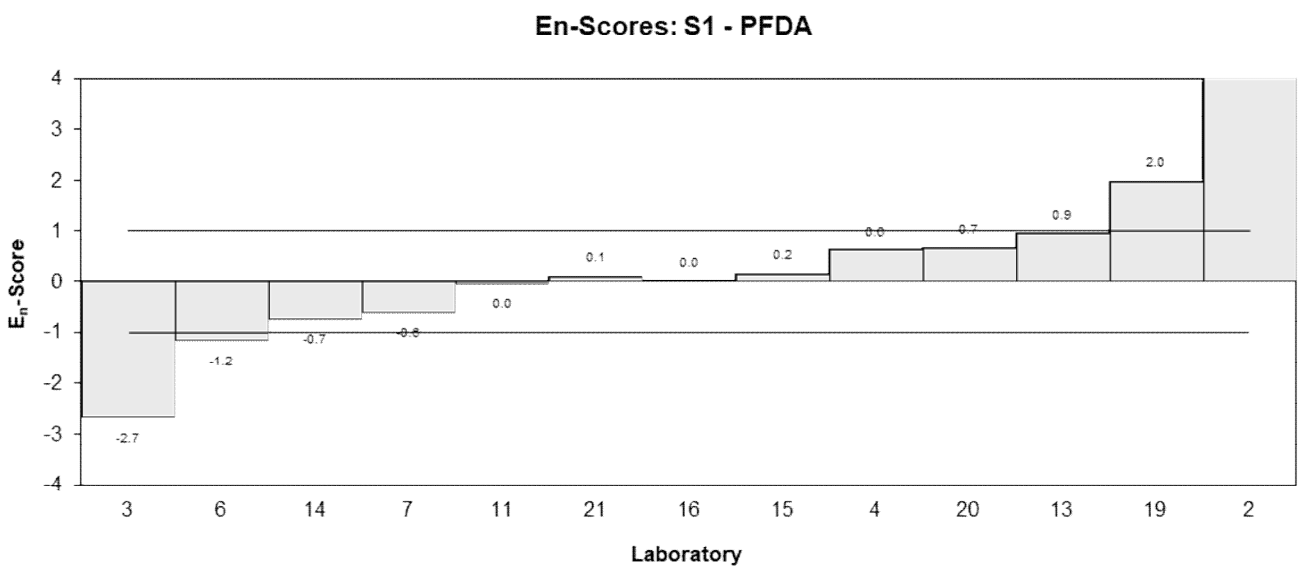
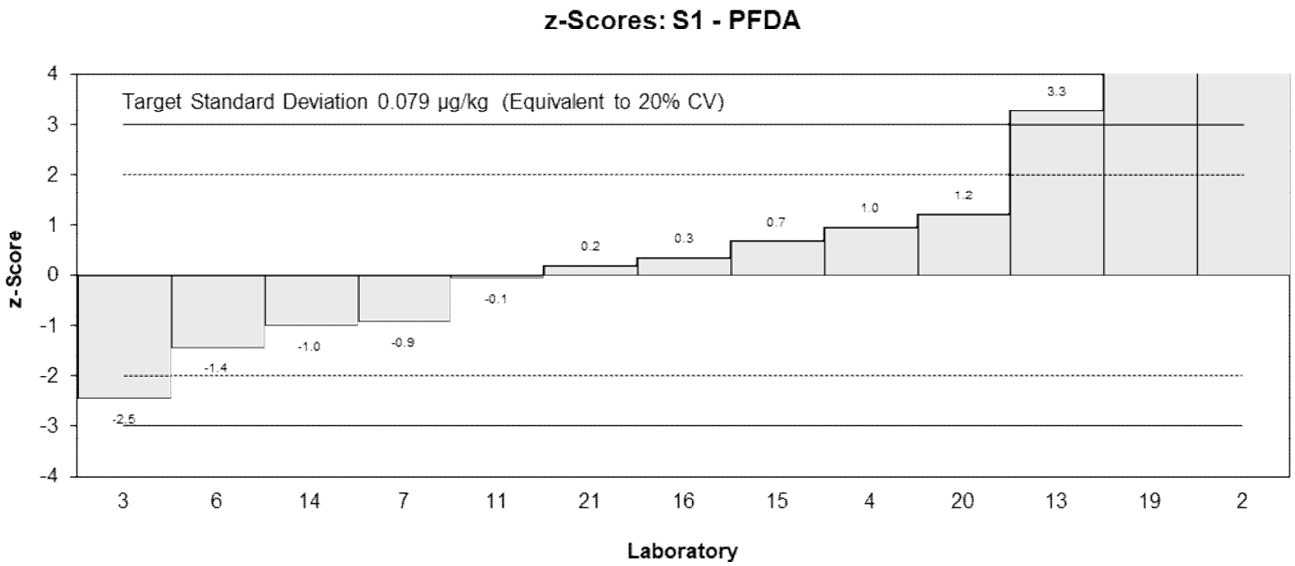
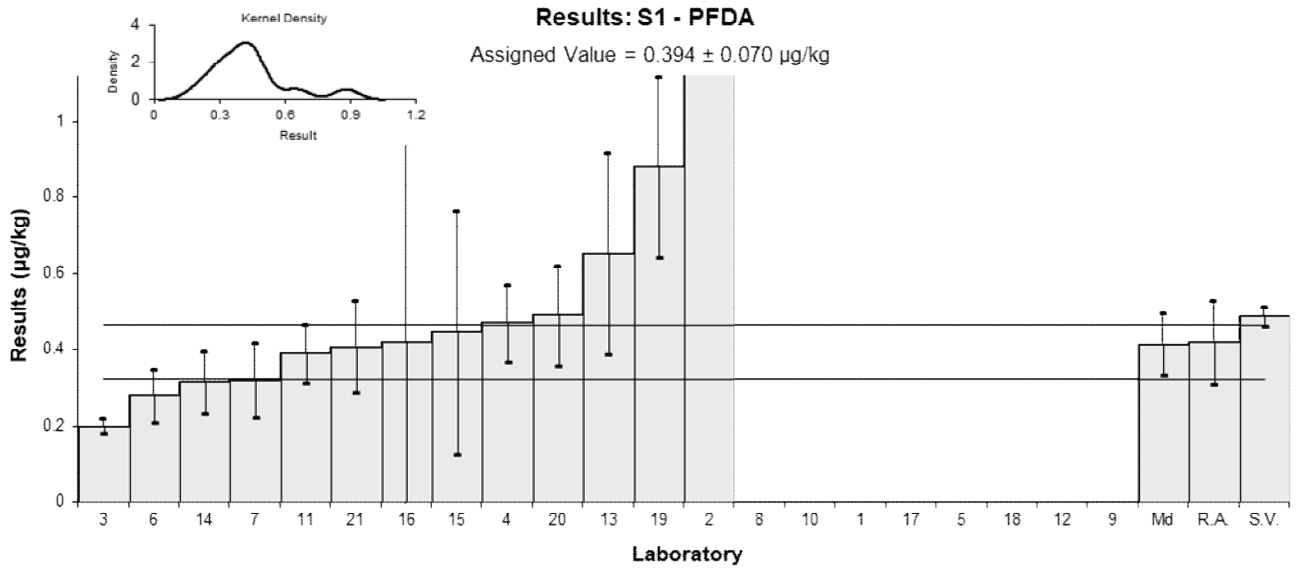


Figure 14

Table 18

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFUdA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	<0.5	NR	74.1		
2	1.17	0.03	80	13.22	10.63
3	0.2	0.01	NR	-1.88	-1.62
4	0.384	0.1	76	0.98	0.51
5	<2	NR	108		
6	0.21	0.06	68	-1.73	-1.17
7	0.272	0.0810	81	-0.76	-0.45
8	< 1.0	NR	89		
9	NT	NT	NT		
10	<0.2	0.04	68		
11	0.392	0.059	67	1.11	0.75
12	NT	NT	NT		
13	0.438	0.097	61.7	1.82	0.96
14	0.258	0.061	95	-0.98	-0.66
15	0.366	0.109	NR	0.70	0.34
16	0.382	23.9	94.2	0.95	0.00
17	<1	NR	144		
18	0.182	0.020	101	-2.17	-1.81
19	NR	NR	73		
20	0.39	0.16	71.7	1.07	0.39
21	0.378	0.11	64.4	0.89	0.43

## Statistics\*

<b>Assigned Value</b>	0.321	0.074
<b>Spike</b>	0.399	0.020
<b>Robust Average</b>	0.321	0.074
<b>Median</b>	0.372	0.041
<b>Mean</b>	0.321	
<b>N</b>	12	
<b>Max.</b>	0.438	
<b>Min.</b>	0.182	
<b>Robust SD</b>	0.10	
<b>Robust CV</b>	32%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

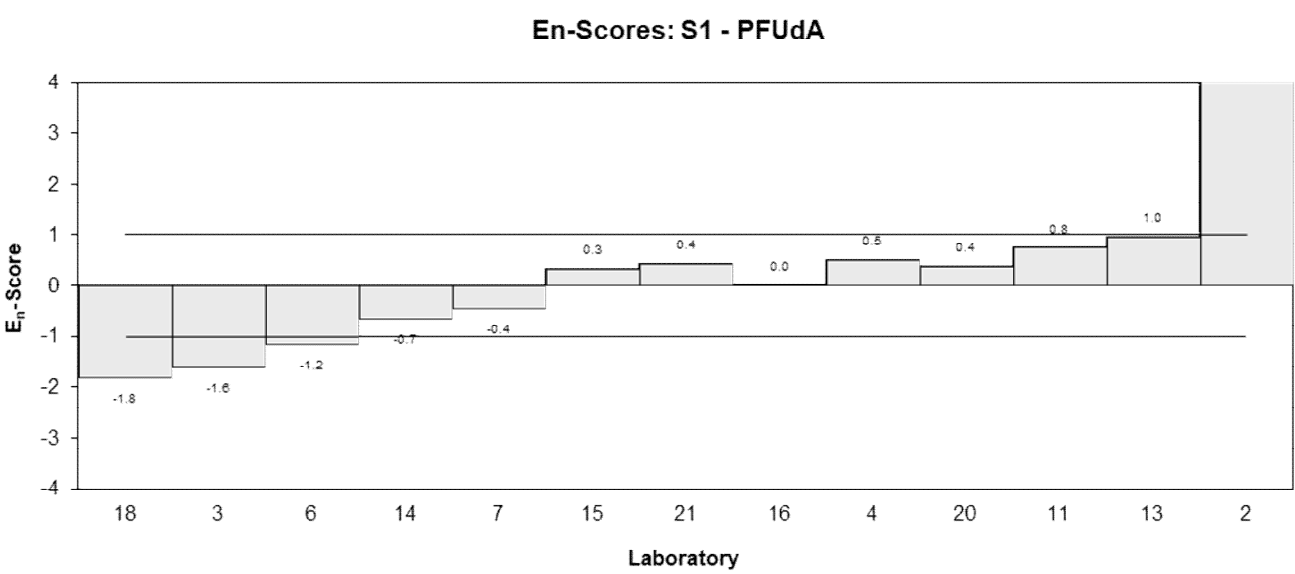
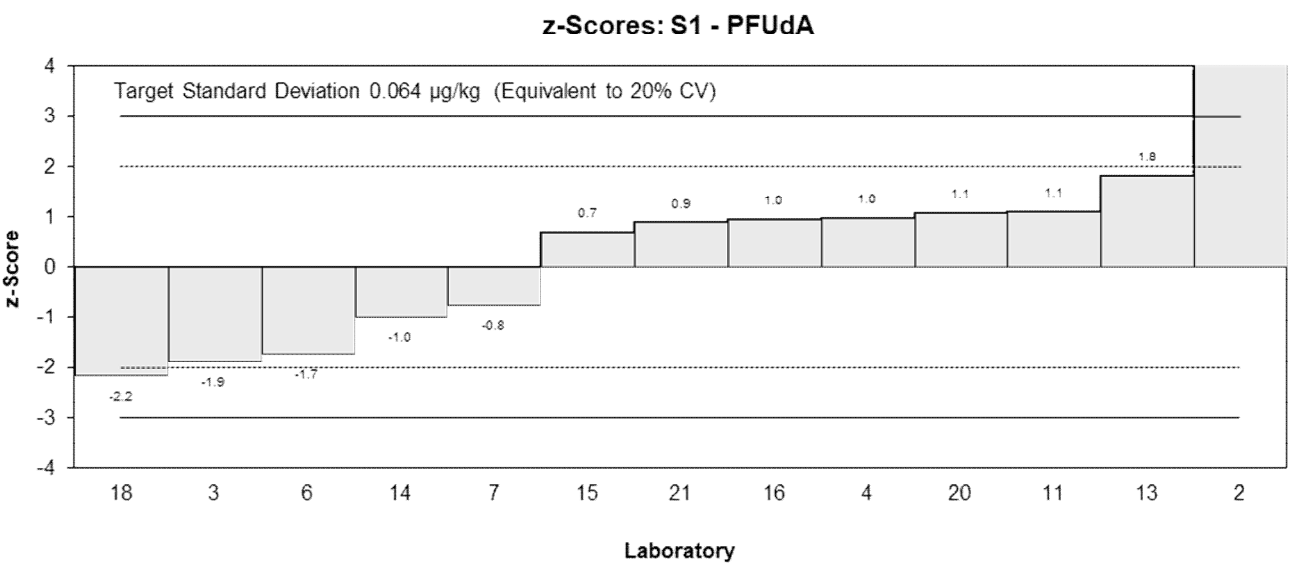
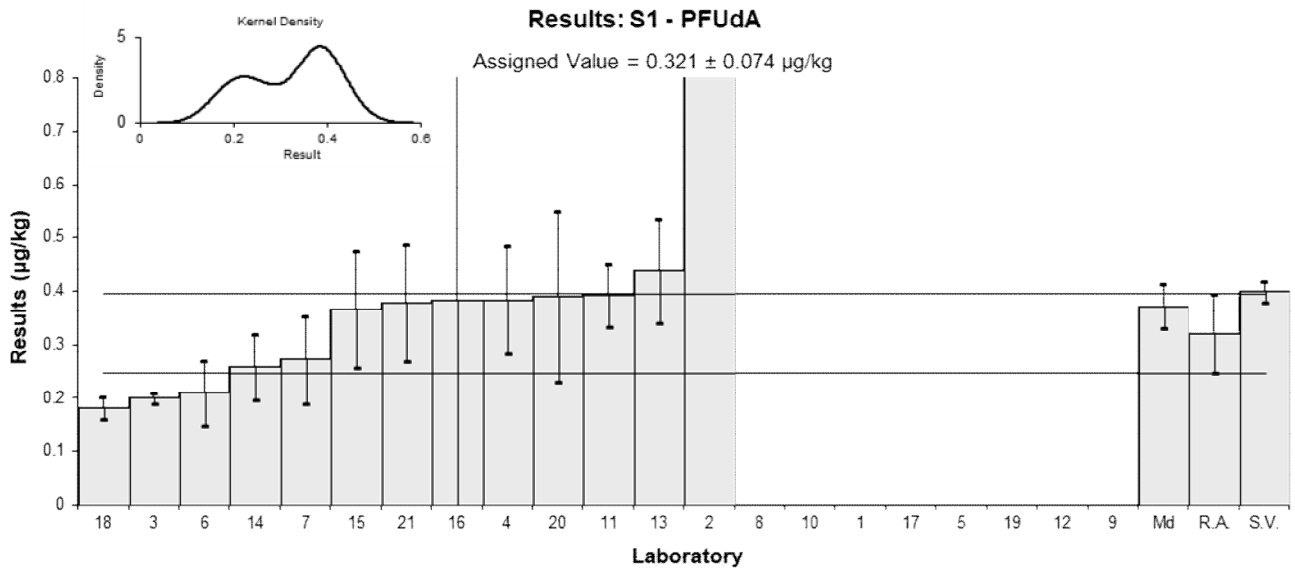


Figure 15

Table 19

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	PFOSA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	5.6	0.5768	74.1	-0.04	-0.06
2	12.94	1.05	NR	6.45	6.03
3	58	8.3	NR	46.33	6.29
4	6.08	2	76	0.38	0.21
5	6	2	105	0.31	0.17
6	NT	NT	NT		
7	3.98	1.19	85	-1.48	-1.25
8	6.0	0.51	92	0.31	0.44
9	NT	NT	NT		
10	2.01	0.49	61	-3.22	-4.70
11	6.14	1.6	69	0.43	0.29
12	NT	NT	NT		
13	6.440	0.744	73.6	0.70	0.83
14	4.37	1.1	95	-1.13	-1.02
15	5.561	0.982	NR	-0.08	-0.08
16	6.1	26.1	108	0.40	0.02
17	6.8	2.7	136	1.02	0.42
18	5.62	0.0087	116	-0.03	-0.05
19	10.5	2.7	66	4.29	1.75
20	5.73	0.93	24	0.07	0.07
21	3.55	1.07	41	-1.86	-1.71

## Statistics\*\*

<b>Assigned Value**</b>	5.65	0.60
<b>Spike</b>	6.93	0.35
<b>Robust Average</b>	5.60	0.77
<b>Median</b>	5.87	0.23
<b>Mean</b>	5.66	
<b>N</b>	16	
<b>Max.</b>	10.5	
<b>Min.</b>	2.01	
<b>Robust SD</b>	1.2	
<b>Robust CV</b>	22%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received. Laboratory 3 was also omitted from all statistical calculations.

\*\* Robust average excluding Laboratories 10 and 19.

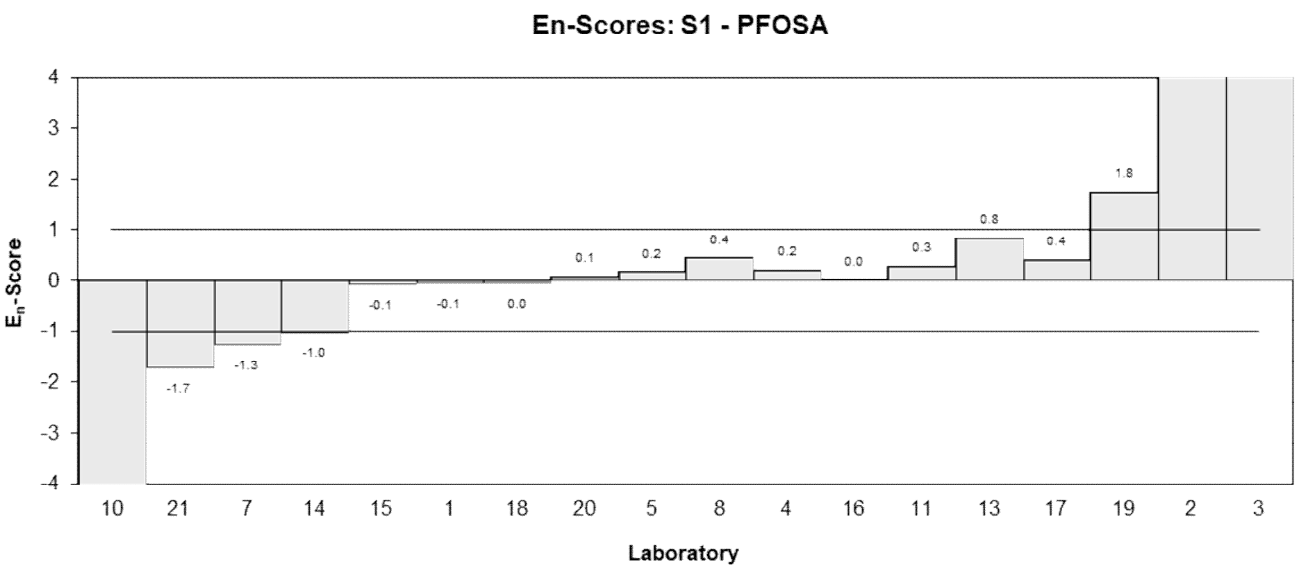
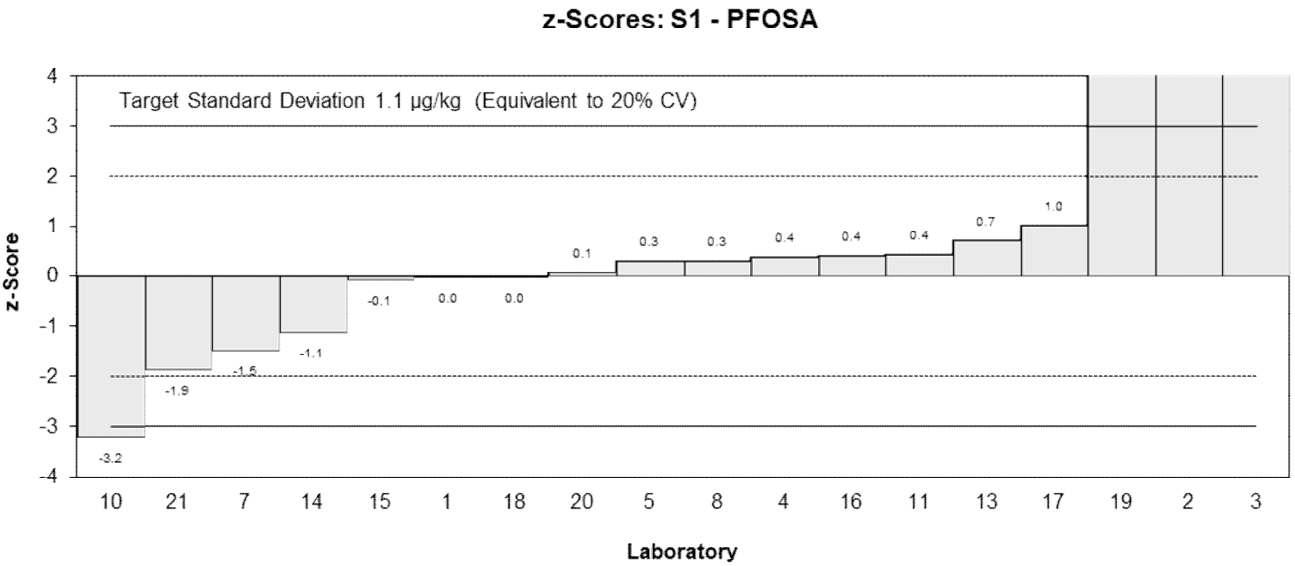
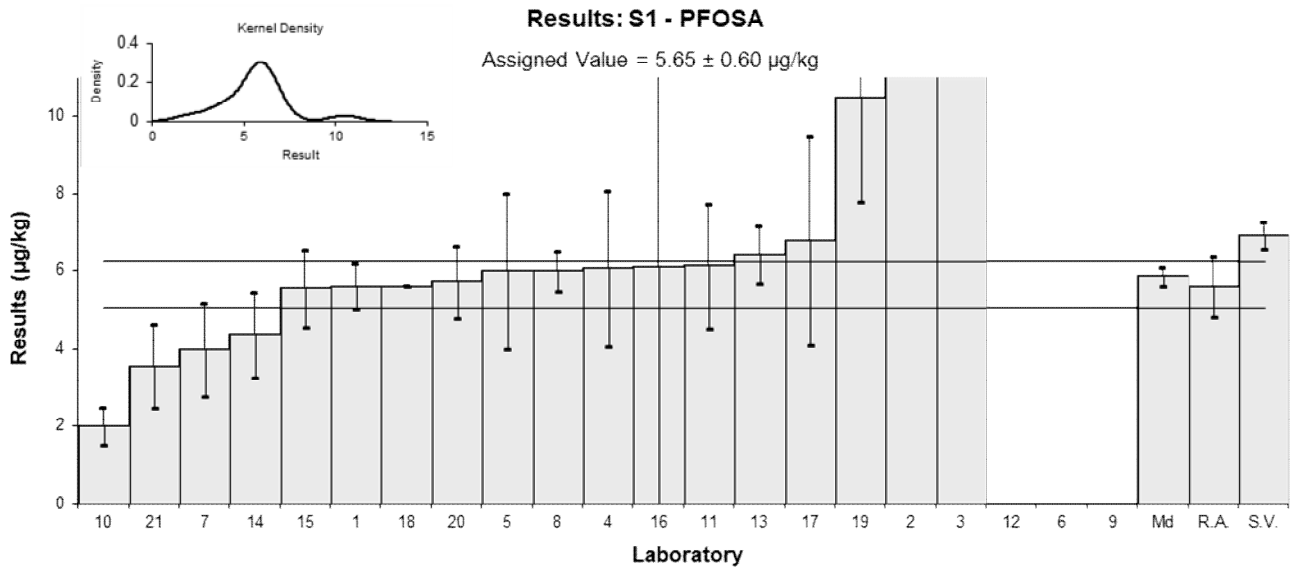


Figure 16

Table 20

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	ADONA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	5.4	NR	74.1	-1.40	-1.62
2	0.57	0.09	NR	-4.62	-5.32
3	NT	NT	NT		
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	7.23	2.17	91	-0.18	-0.11
8	NT	NT	NT		
9	NT	NT	NT		
10	7.29	2.19	NR	-0.14	-0.08
11	8.56	2.19	77	0.71	0.42
12	NT	NT	NT		
13	11.226	1.490	67.8	2.48	1.88
14	6.75	1.7	95	-0.50	-0.35
15	7.962	0.818	NR	0.31	0.30
16	7.14	40.8	118	-0.24	-0.01
17	NT	NT	NT		
18	NT	NT	NT		
19	15.1	5.4	81	5.07	1.37
20	NT	NT	NT		
21	NT	NT	NT		

## Statistics\*

<b>Assigned Value**</b>	7.5	1.3
<b>Spike</b>	9.35	0.47
<b>Robust Average</b>	8.1	1.9
<b>Median</b>	7.29	0.77
<b>Mean</b>	8.52	
<b>N</b>	9	
<b>Max.</b>	15.1	
<b>Min.</b>	5.4	
<b>Robust SD</b>	2.3	
<b>Robust CV</b>	29%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratory 19.



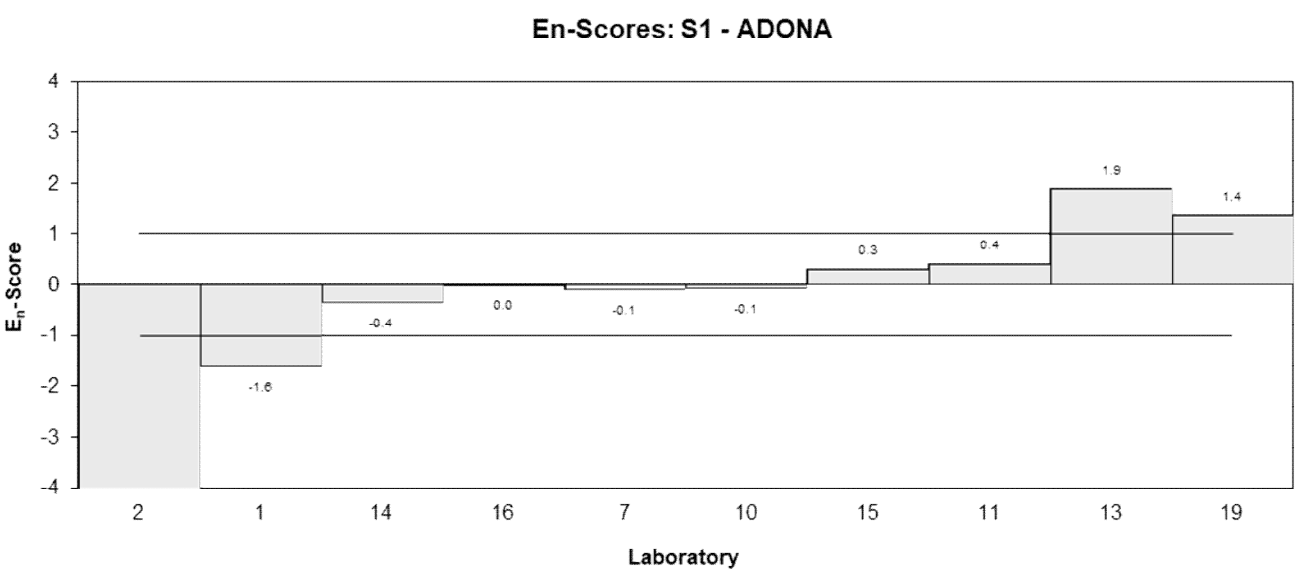
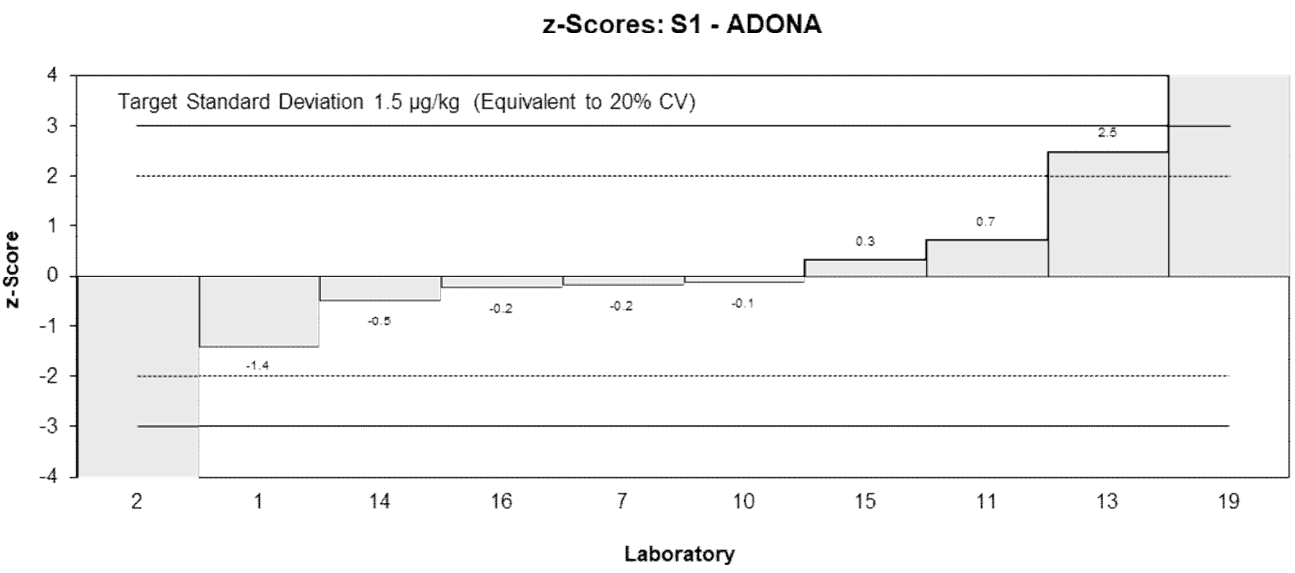
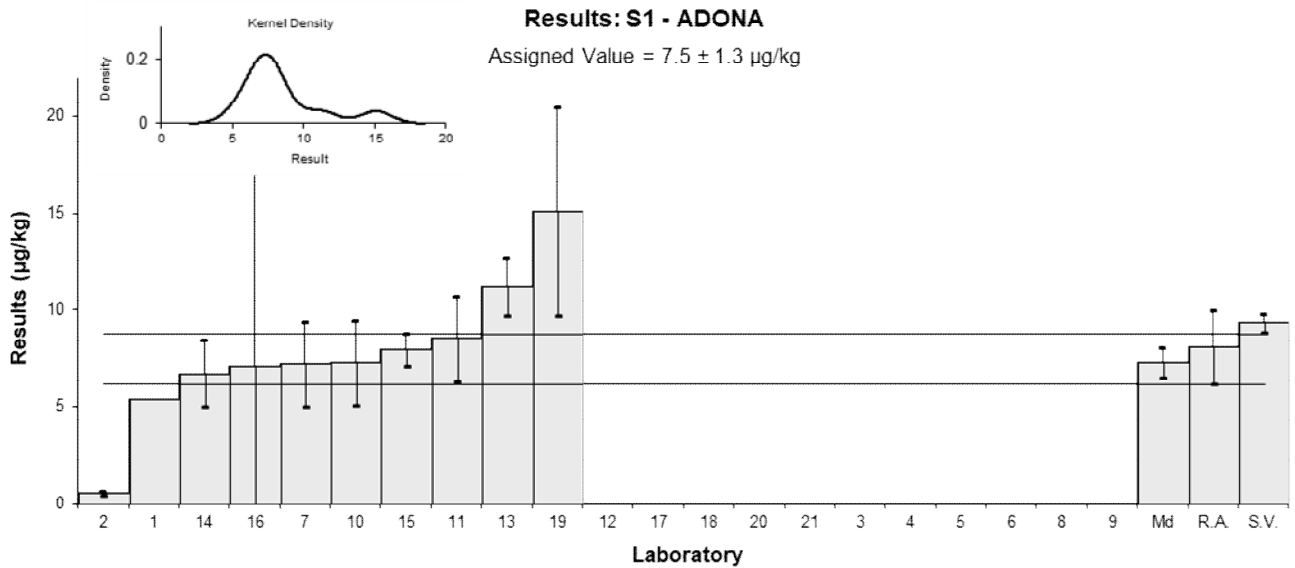


Figure 17

Table 21

**Sample Details**

<b>Sample No.</b>	S1
<b>Matrix</b>	Prawn
<b>Analyte</b>	GenX
<b>Units</b>	µg/kg

**Participant Results**

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	<0.5	NR	74.1		
2	NT	NT	NT		
3	NT	NT	NT		
4	NT	NT	NT		
5	NT	NT	NT		
6	NR	NR	30		
7	8.68	2.60	79	-0.48	-0.27
8	NT	NT	NT		
9	NT	NT	NT		
10	7.05	2.11	43	-1.33	-0.84
11	10.5	2.1	68	0.47	0.30
12	NT	NT	NT		
13	12.595	3.054	47.7	1.56	0.80
14	9.21	2.3	95	-0.20	-0.12
15	NT	NT	NT		
16	9.54	24	118	-0.03	0.00
17	NT	NT	NT		
18	NT	NT	NT		
19	17.1	7.5	75	3.91	0.96
20	NT	NT	NT		
21	NT	NT	NT		

**Statistics**

<b>Assigned Value*</b>	9.6	2.2
<b>Spike</b>	12.4	0.6
<b>Robust Average</b>	10.3	2.7
<b>Median</b>	9.5	1.3
<b>Mean</b>	11	
<b>N</b>	7	
<b>Max.</b>	17.1	
<b>Min.</b>	7.05	
<b>Robust SD</b>	2.9	
<b>Robust CV</b>	28%	

\* Robust average excluding Laboratory 19.

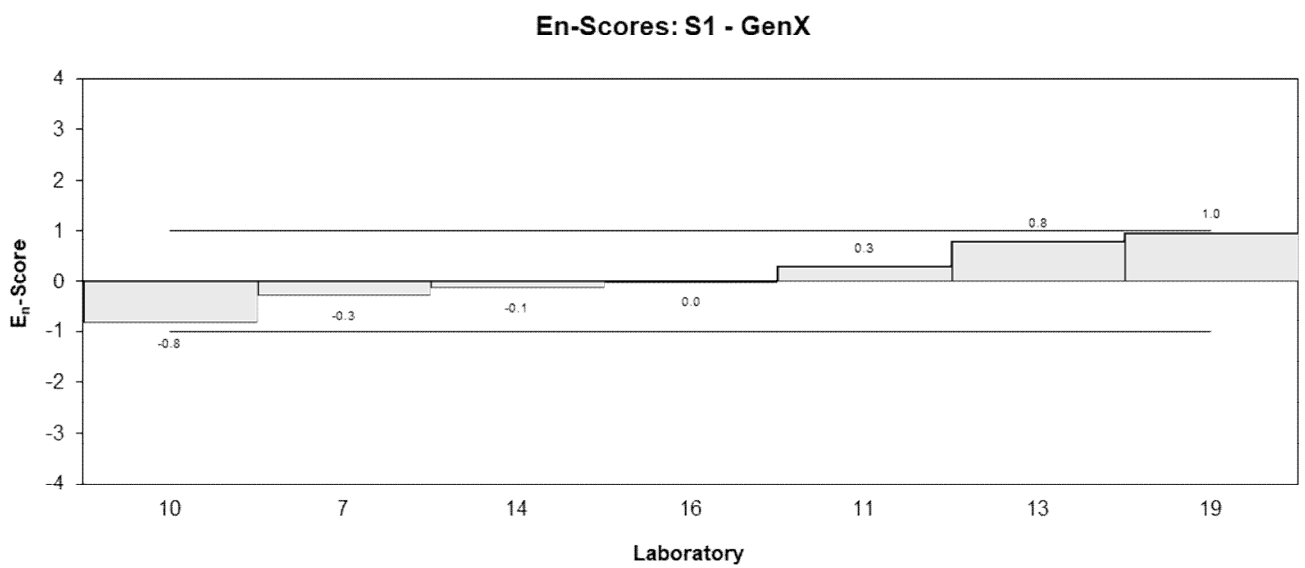
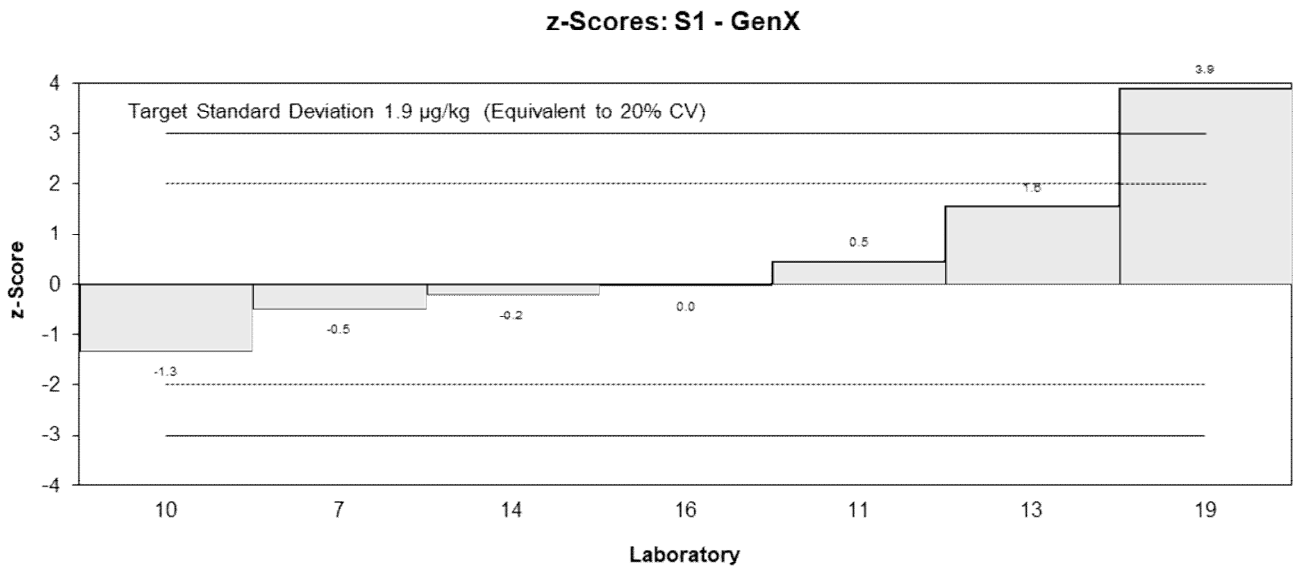
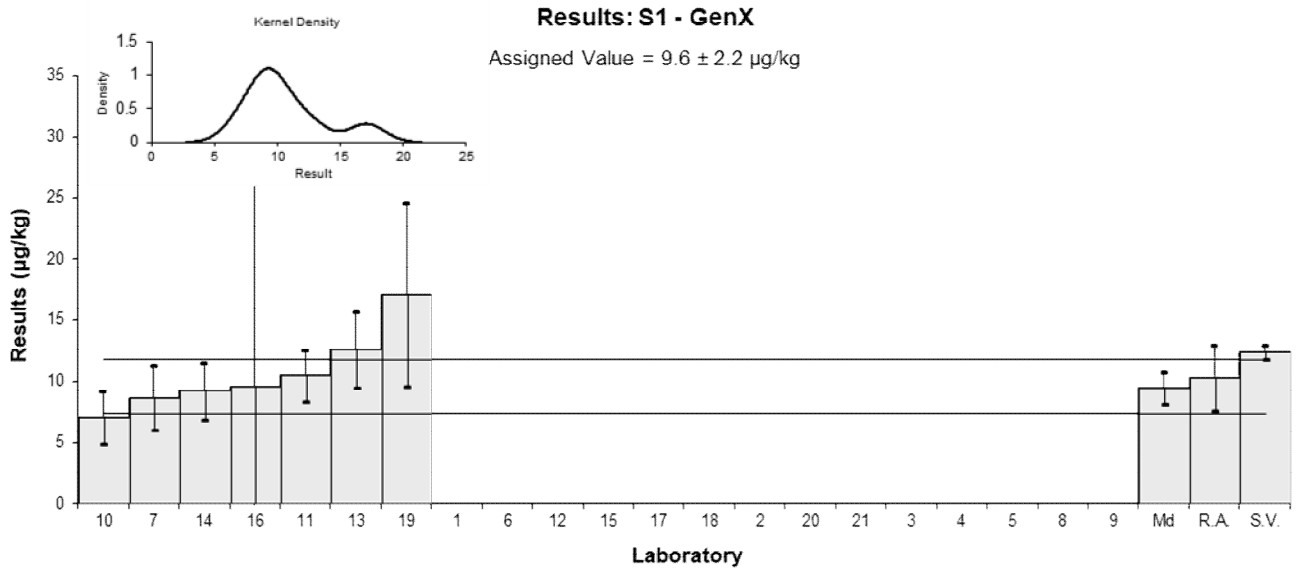


Figure 18

Table 22

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	PFBS
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	3.4	0.493	75.9	1.44	1.24
2	115.08	3.91	NR	212.95	28.64
3	NT	NT	NT		
4	2.79	1	78	0.28	0.14
5	3	1	80	0.68	0.34
6	2.6	0.65	122	-0.08	-0.05
7	2.03	0.609	77	-1.16	-0.86
8	2.7	0.031	104	0.11	0.17
9	1.71	NR	NR	-1.76	-2.58
10	2.44	0.32	81	-0.38	-0.42
11	2.18	0.4	89	-0.87	-0.85
12	NT	NT	NT		
13	3.444	0.451	98.6	1.52	1.39
14	2.29	0.054	97	-0.66	-0.96
15	3.274	0.449	NR	1.20	1.10
16	NT	NT	NT		
17	2.62	0.9	125	-0.04	-0.02
18	NT	NT	NT		
19	2.79	0.25	103	0.28	0.34
20	2.25	0.51	72.3	-0.74	-0.62
21	4.24	1.27	76.3	3.03	1.21

## Statistics\*

<b>Assigned Value**</b>	2.64	0.36
<b>Spike</b>	3.05	0.15
<b>Homogeneity Value</b>	2.82	0.85
<b>Robust Average</b>	2.70	0.38
<b>Median</b>	2.66	0.31
<b>Mean</b>	2.73	
<b>N</b>	16	
<b>Max.</b>	4.24	
<b>Min.</b>	1.71	
<b>Robust SD</b>	0.61	
<b>Robust CV</b>	23%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratory 21.

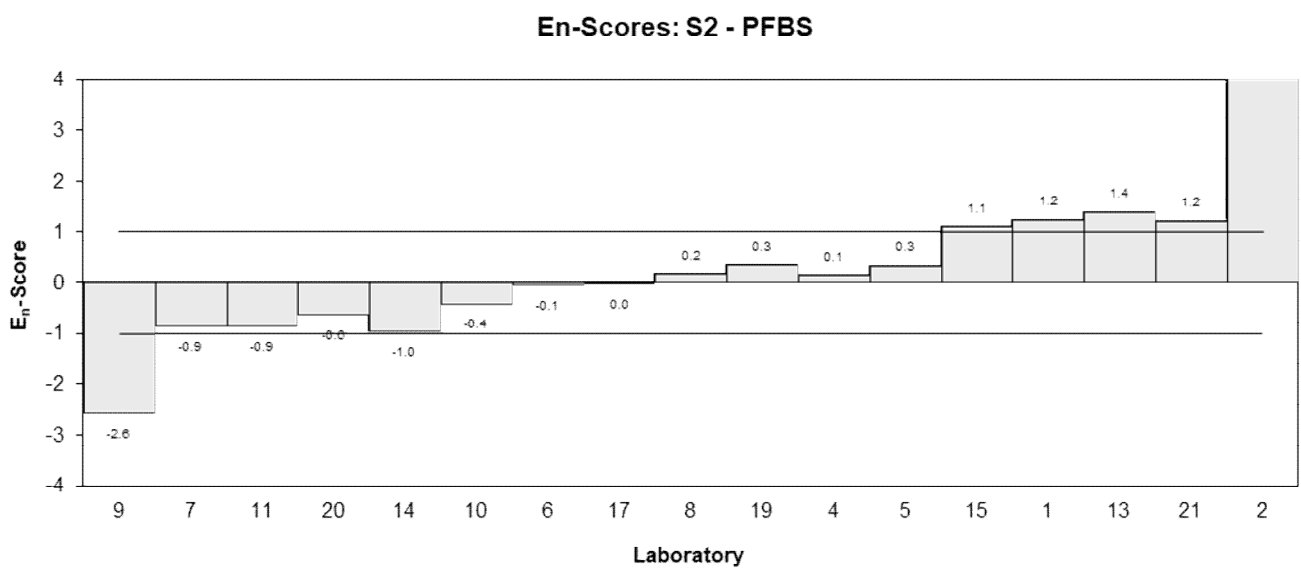
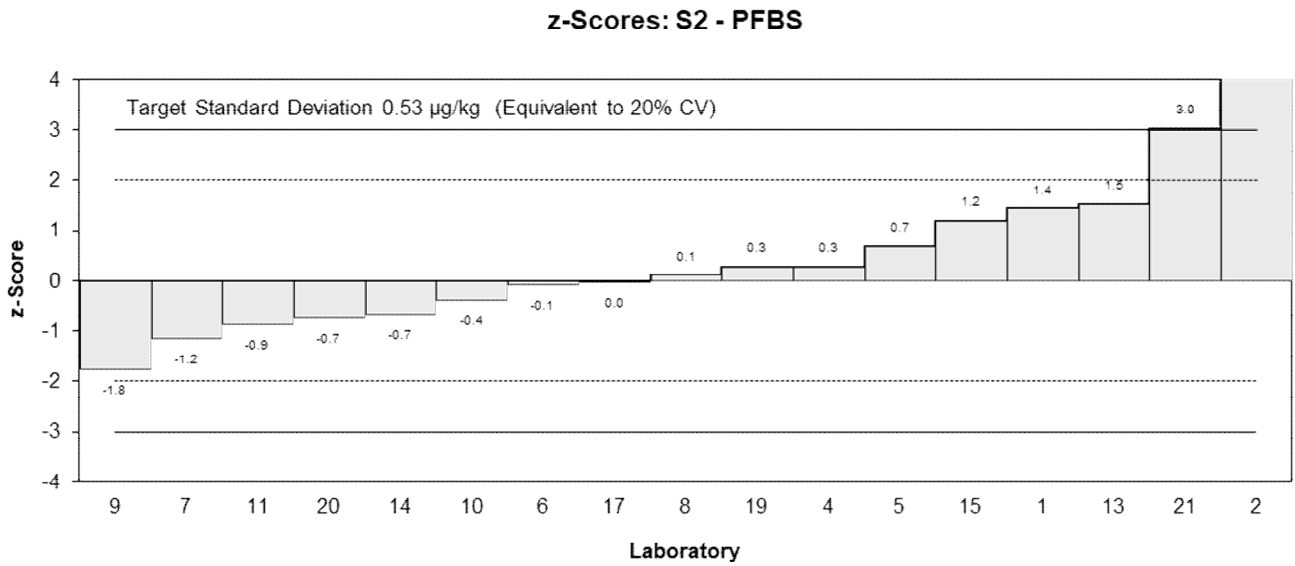
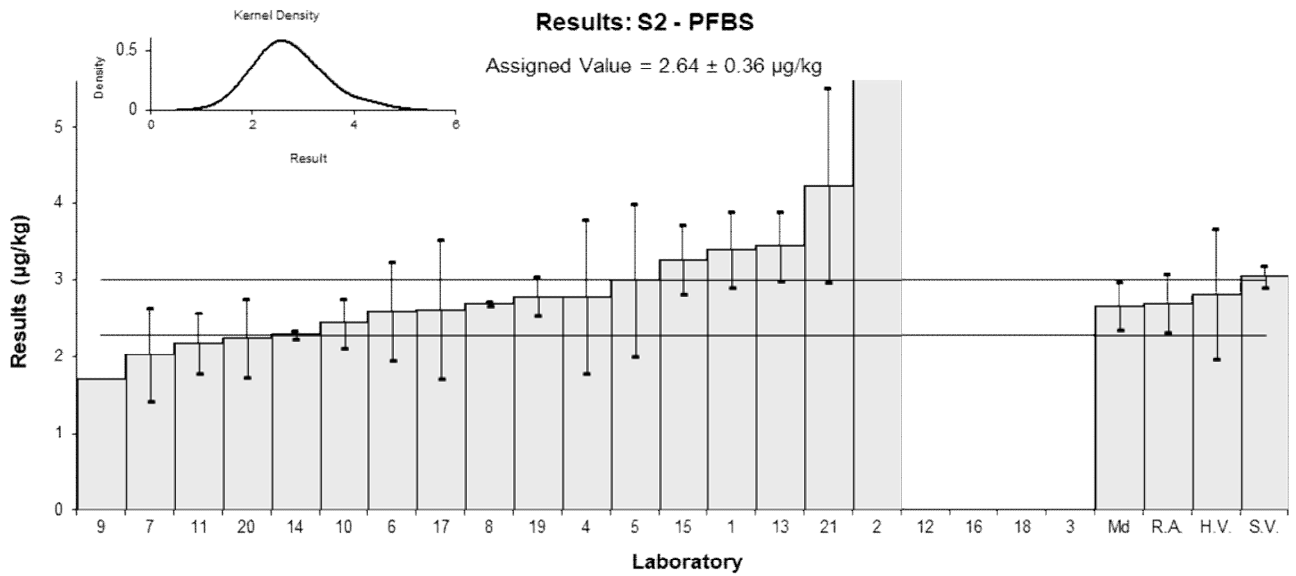


Figure 19

Table 23

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	PFHxS
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	4.6	0.92	75.9	-0.90	-0.96
2	234.17	5.72	NR	203.71	39.79
3	NT	NT	NT		
4	5.69	1	NR	0.07	0.07
5	6	2	90	0.35	0.19
6	5	1.25	NR	-0.54	-0.45
7	5.20	1.56	82	-0.37	-0.25
8	5.7	0.15	106	0.08	0.17
9	2.91	NR	NR	-2.41	-5.19
10	5.70	0.81	81	0.08	0.09
11	6.22	0.69	97	0.54	0.71
12	NT	NT	NT		
13	6.638	0.510	92.2	0.92	1.41
14	5.21	0.12	97	-0.36	-0.75
15	NT	NT	NT		
16	NT	NT	NT		
17	5.94	2	122	0.29	0.16
18	NT	NT	NT		
19	5.44	0.26	79	-0.15	-0.29
20	NR	NR	72.3		
21	7.59	2.28	80.5	1.76	0.85

## Statistics\*

<b>Assigned Value</b>	5.61	0.52
<b>Spike</b>	6.08	0.30
<b>Homogeneity Value</b>	5.6	1.7
<b>Robust Average</b>	5.61	0.52
<b>Median</b>	5.70	0.42
<b>Mean</b>	5.56	
<b>N</b>	14	
<b>Max.</b>	7.59	
<b>Min.</b>	2.91	
<b>Robust SD</b>	0.79	
<b>Robust CV</b>	14%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

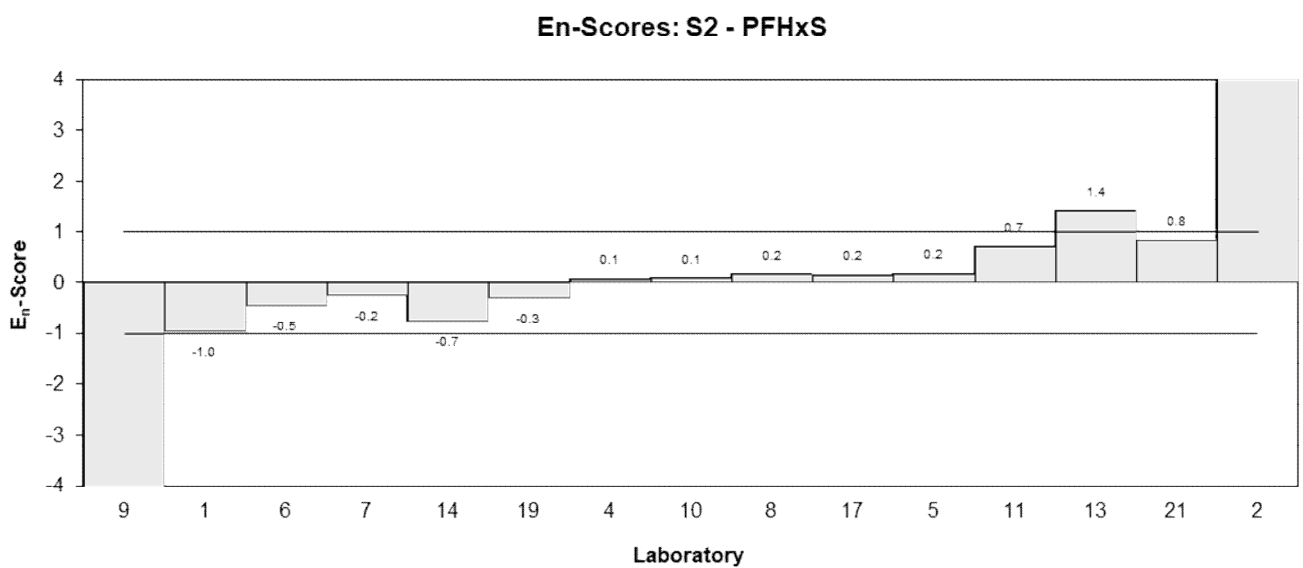
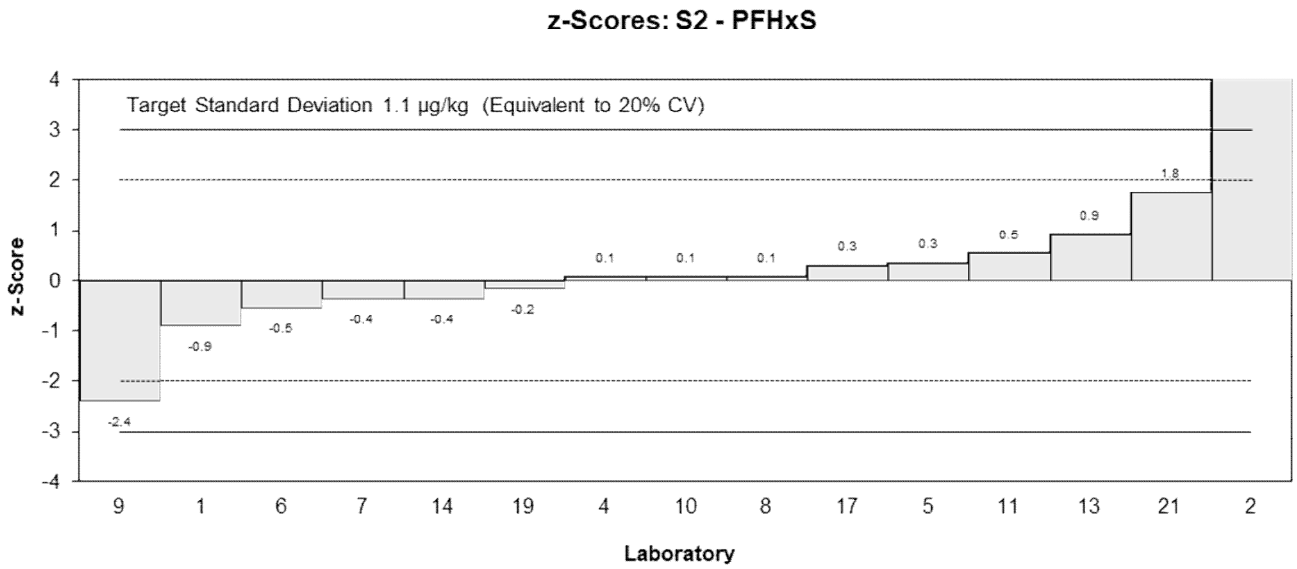
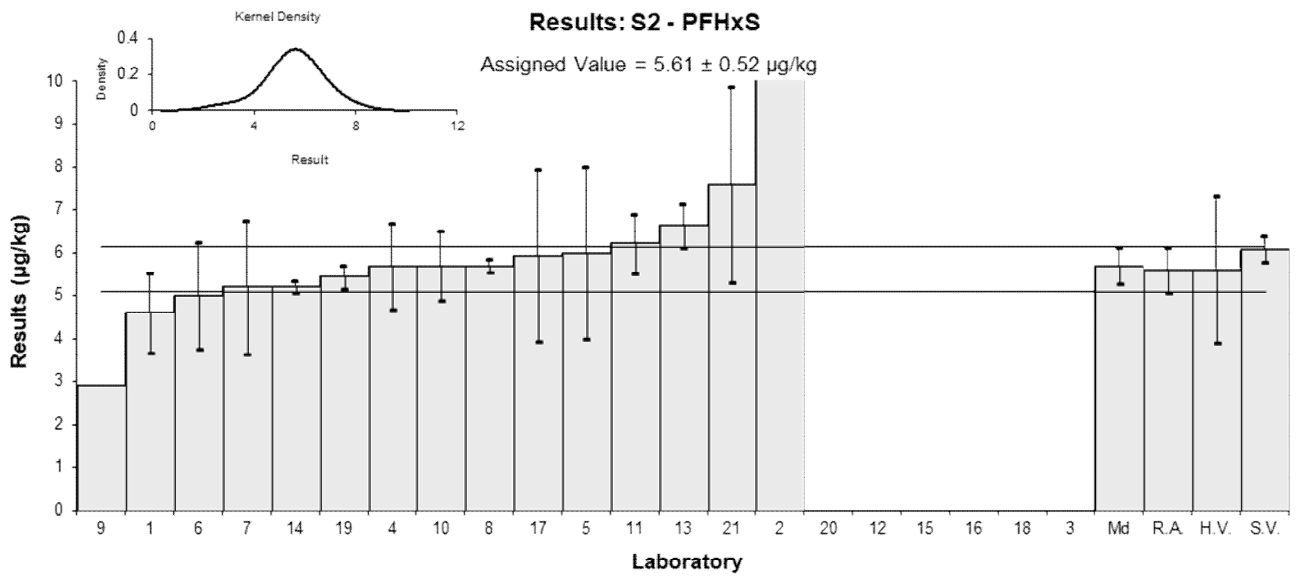


Figure 20

Table 24

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	PFHxS (linear)
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	NT	NT	NT		
2	228.00	5.60	70	193.95	39.58
3	NT	NT	NT		
4	5.09	1	69	-0.56	-0.59
5	6	2	90	0.24	0.13
6	5	1.25	119	-0.64	-0.55
7	5.20	1.56	82	-0.46	-0.33
8	5.7	0.15	106	-0.03	-0.07
9	NT	NT	NT		
10	5.70	0.81	81	-0.03	-0.03
11	6.22	0.69	97	0.43	0.61
12	NT	NT	NT		
13	6.521	0.514	92.2	0.69	1.19
14	5.21	0.13	97	-0.45	-1.18
15	6.003	0.662	NR	0.24	0.35
16	NT	NT	NT		
17	NT	NT	NT		
18	NT	NT	NT		
19	5.36	0.26	79	-0.32	-0.75
20	5.8	2	72.3	0.06	0.03
21	7.45	2.24	80.5	1.50	0.75

## Statistics\*

<b>Assigned Value</b>	5.73	0.42
<b>Spike</b>	5.86	0.29
<b>Homogeneity Value</b>	5.4	1.6
<b>Robust Average</b>	5.73	0.42
<b>Median</b>	5.70	0.44
<b>Mean</b>	5.79	
<b>N</b>	13	
<b>Max.</b>	7.45	
<b>Min.</b>	5	
<b>Robust SD</b>	0.61	
<b>Robust CV</b>	11%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.



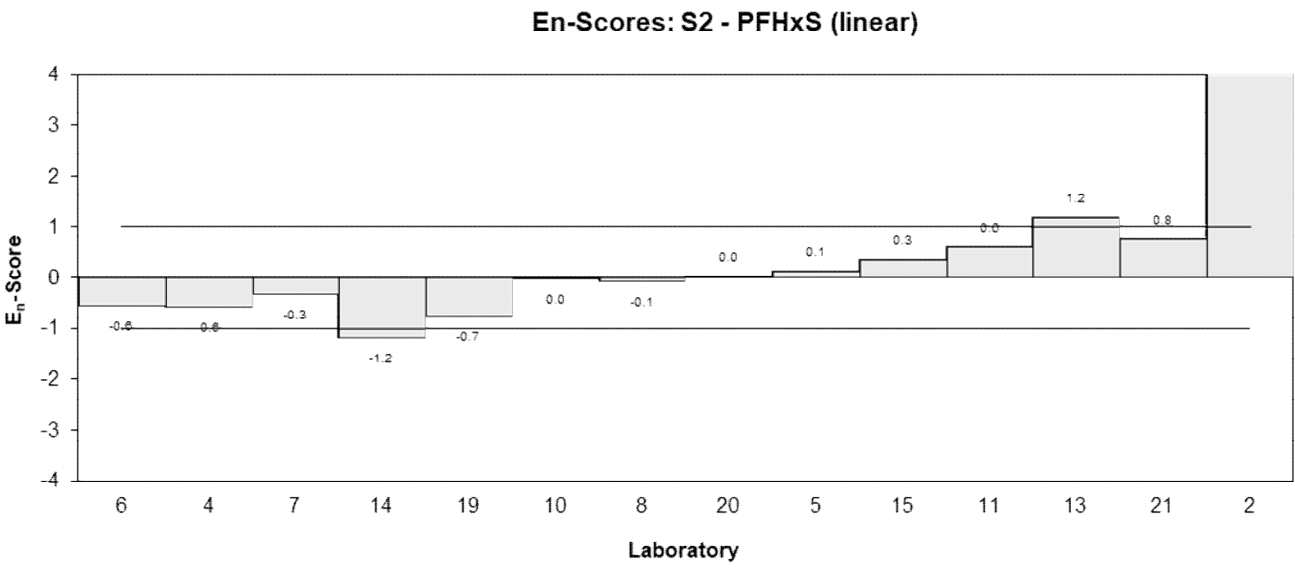
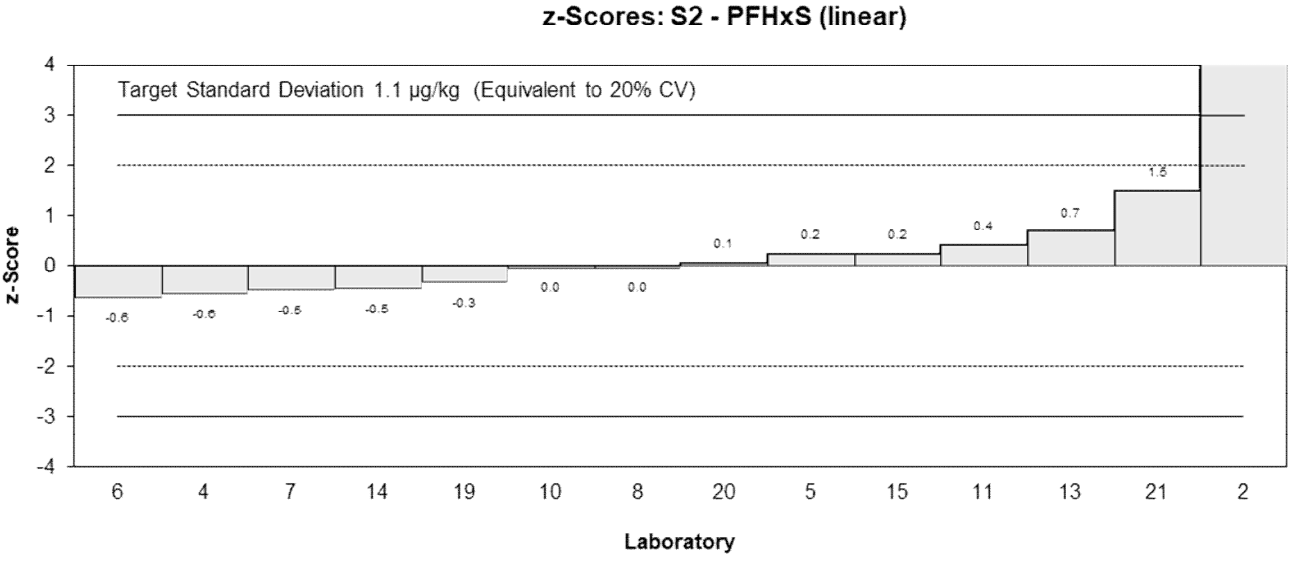
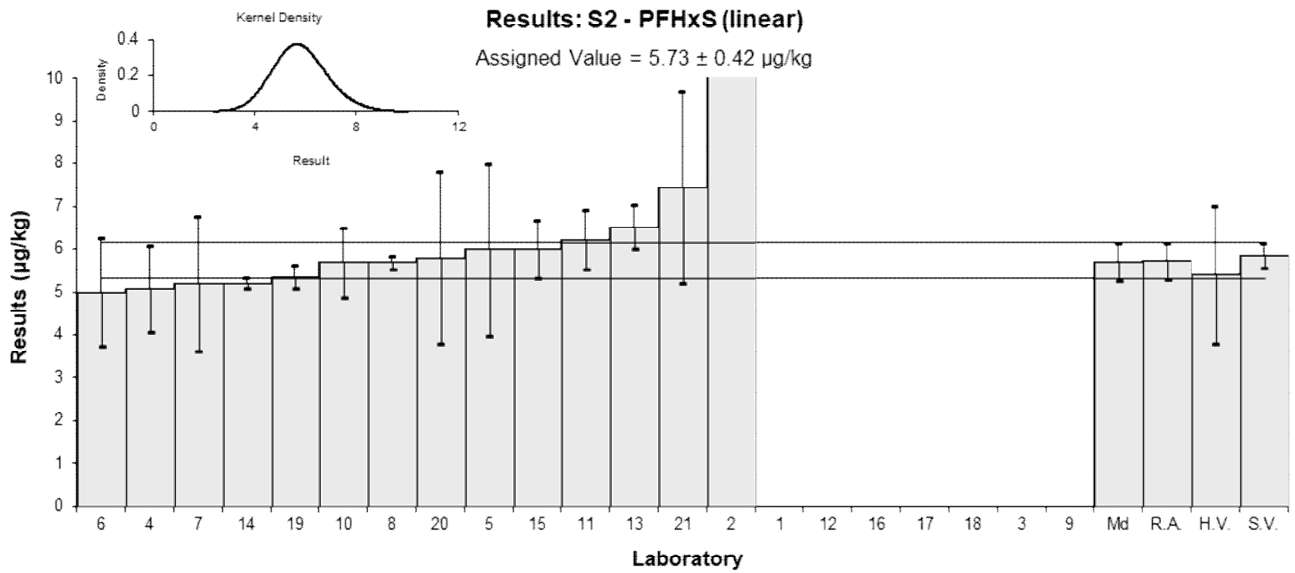


Figure 21

Table 25

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	PFHpS
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	4.4	NR	75.9	-1.57	-4.30
2	210.06	16.5	NR	158.60	12.34
3	NT	NT	NT		
4	7.48	2	NR	0.83	0.52
5	7	2	89	0.45	0.28
6	3.97	0.99	NR	-1.91	-2.24
7	6.21	1.86	71	-0.16	-0.11
8	5.9	0.11	106	-0.40	-1.08
9	3.147	NR	NR	-2.55	-6.96
10	7.04	1.93	NR	0.48	0.31
11	7.05	1.52	97	0.49	0.40
12	NT	NT	NT		
13	6.648	0.655	92.2	0.18	0.28
14	6.26	0.15	97	-0.12	-0.32
15	6.081	0.671	NR	-0.26	-0.41
16	NT	NT	NT		
17	6.62	2.3	127	0.16	0.09
18	NT	NT	NT		
19	6.55	0.23	79	0.10	0.25
20	6.3	1.7	72.3	-0.09	-0.07
21	9.89	2.97	80.5	2.70	1.15

## Statistics\*

<b>Assigned Value**</b>	6.42	0.47
<b>Spike</b>	7.33	0.37
<b>Homogeneity Value</b>	6.8	2.1
<b>Robust Average</b>	6.32	0.69
<b>Median</b>	6.43	0.43
<b>Mean</b>	6.28	
<b>N</b>	16	
<b>Max.</b>	9.89	
<b>Min.</b>	3.147	
<b>Robust SD</b>	1.1	
<b>Robust CV</b>	18%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 9 and 21.

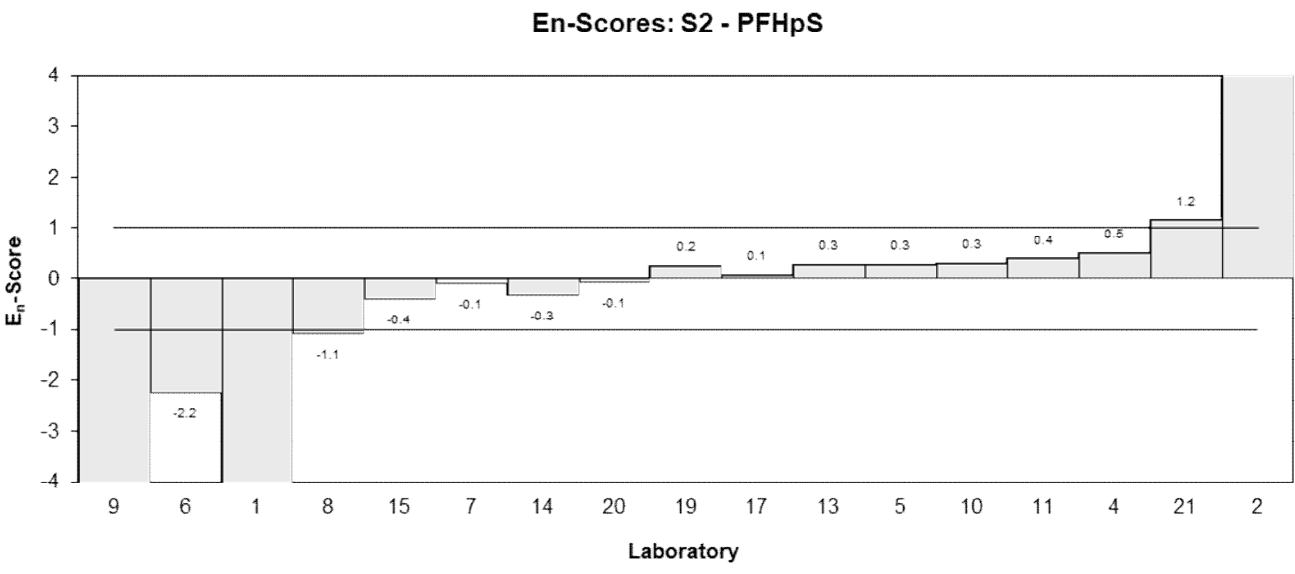
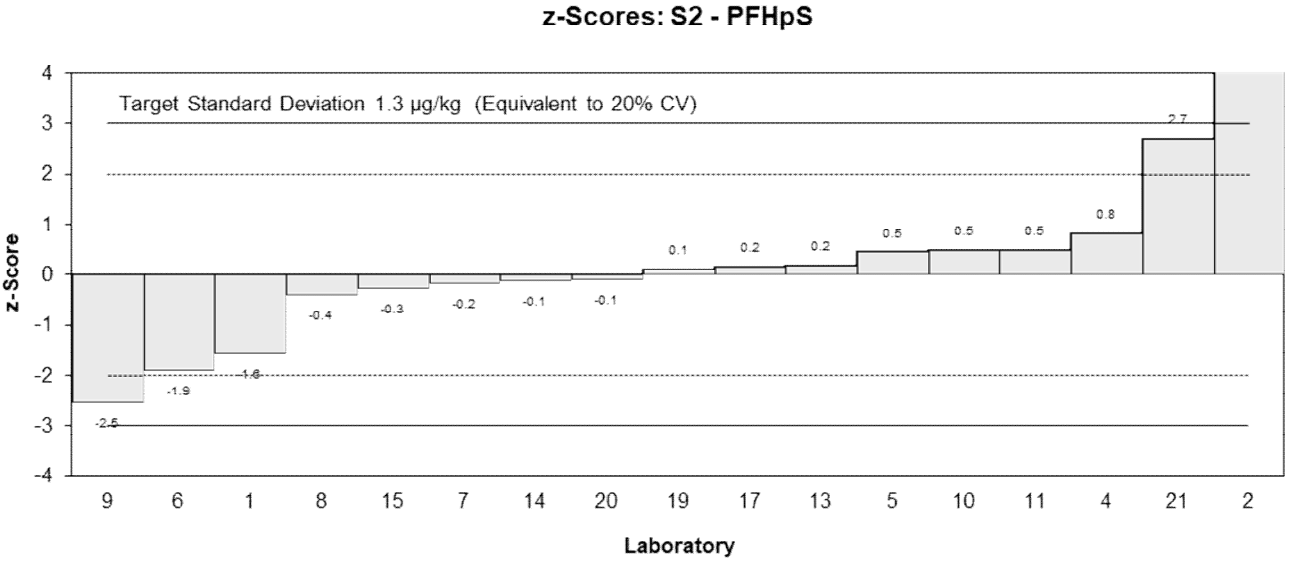
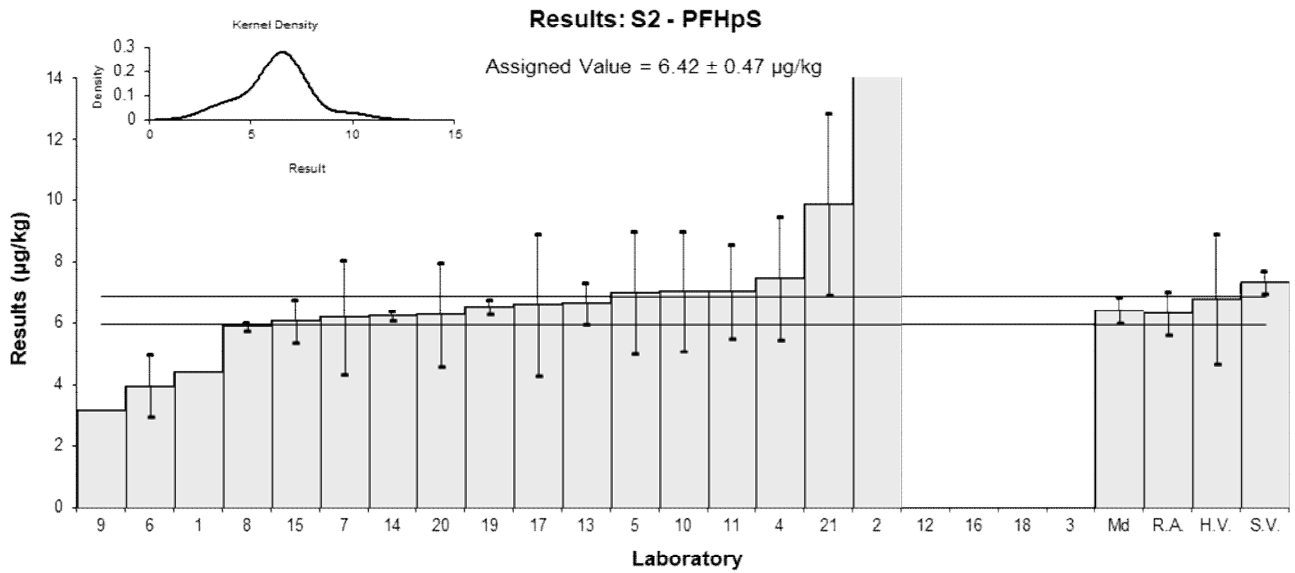


Figure 22

Table 26

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	PFOS
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	22.3	8.0503	75.9	-0.93	-0.58
2	1071.85	56.9	NR	190.59	18.32
3	NT	NT	NT		
4	24	6	NR	-0.62	-0.49
5	29	9	89	0.29	0.17
6	28.2	7.1	NR	0.15	0.10
7	23.8	7.14	71	-0.66	-0.46
8	25	0.55	103	-0.44	-0.70
9	15.9	NR	68.7	-2.10	-3.38
10	33.9	4.50	98	1.19	1.15
11	35.3	19.1	103	1.44	0.41
12	98.6	19.70	NR	12.99	3.56
13***	39.688	4.274	107.3	2.00	1.00
14	24.1	0.56	97	-0.60	-0.96
15	27.003	3.499	NR	-0.07	-0.08
16	NT	NT	NT		
17	28.1	12.0	127	0.13	0.06
18	NT	NT	NT		
19	27.9	0.29	90	0.09	0.15
20	28.1	7.4	74	0.13	0.09
21	54.6	16.4	100	4.96	1.62

## Statistics\*

<b>Assigned Value**</b>	27.4	3.4
<b>Spike</b>	37.2	1.9
<b>Max. Acceptable Conc.***</b>	48.2	
<b>Homogeneity Value</b>	23.5	7.0
<b>Robust Average</b>	29.1	4.6
<b>Median</b>	28.1	3.1
<b>Mean</b>	33.3	
<b>N</b>	17	
<b>Max.</b>	98.6	
<b>Min.</b>	15.9	
<b>Robust SD</b>	7.5	
<b>Robust CV</b>	26%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 12 and 21.

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

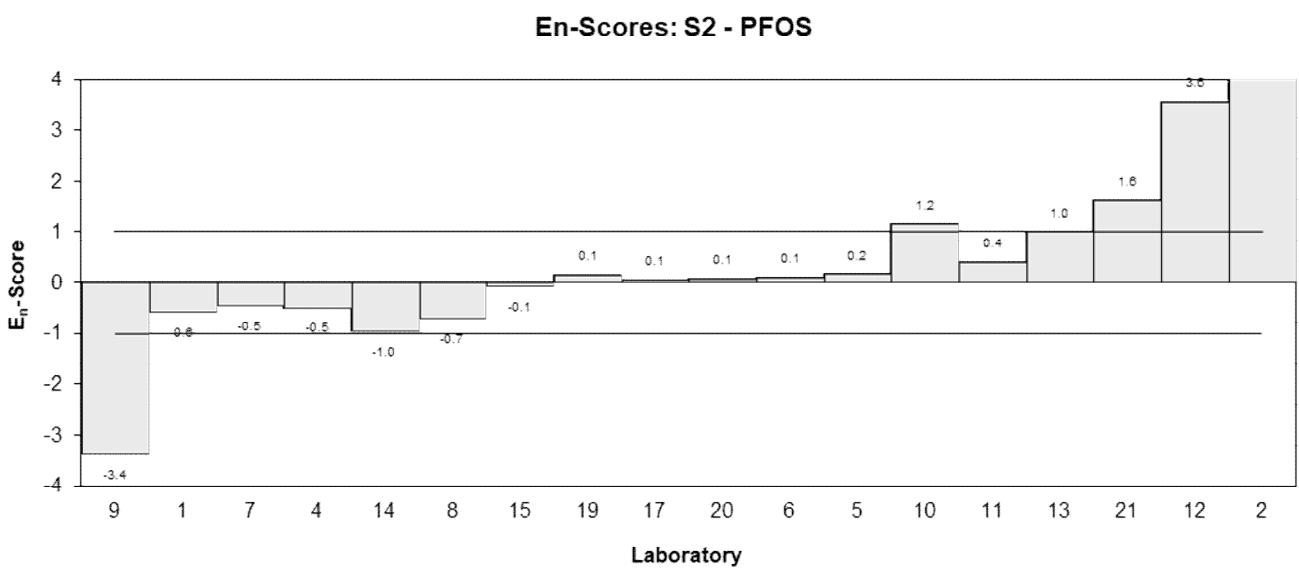
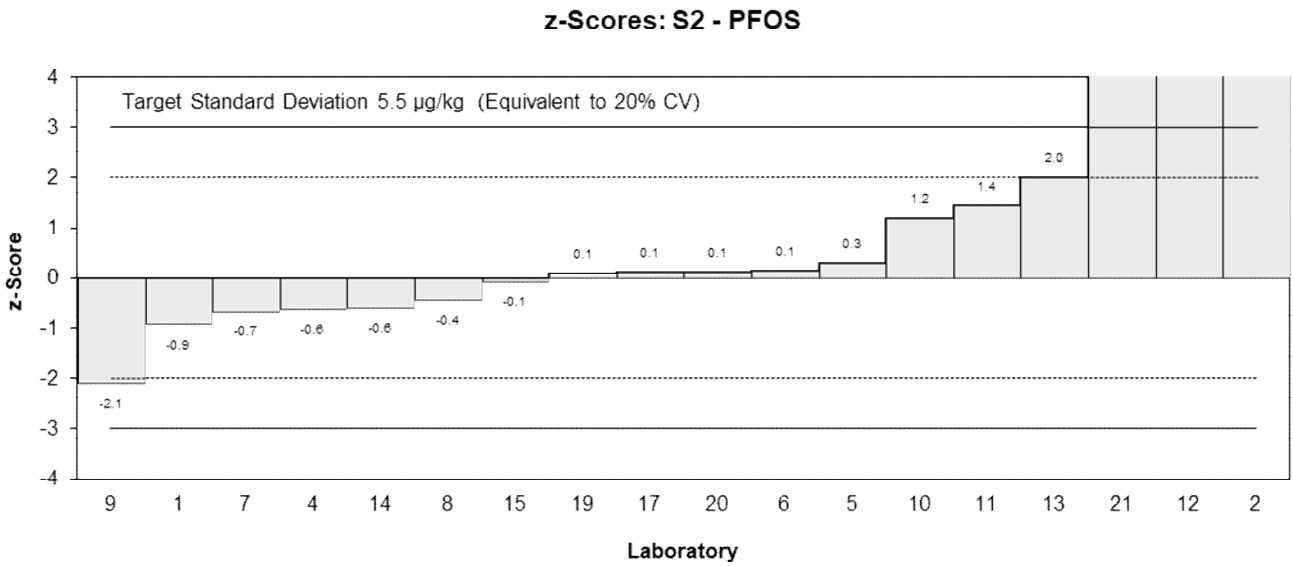
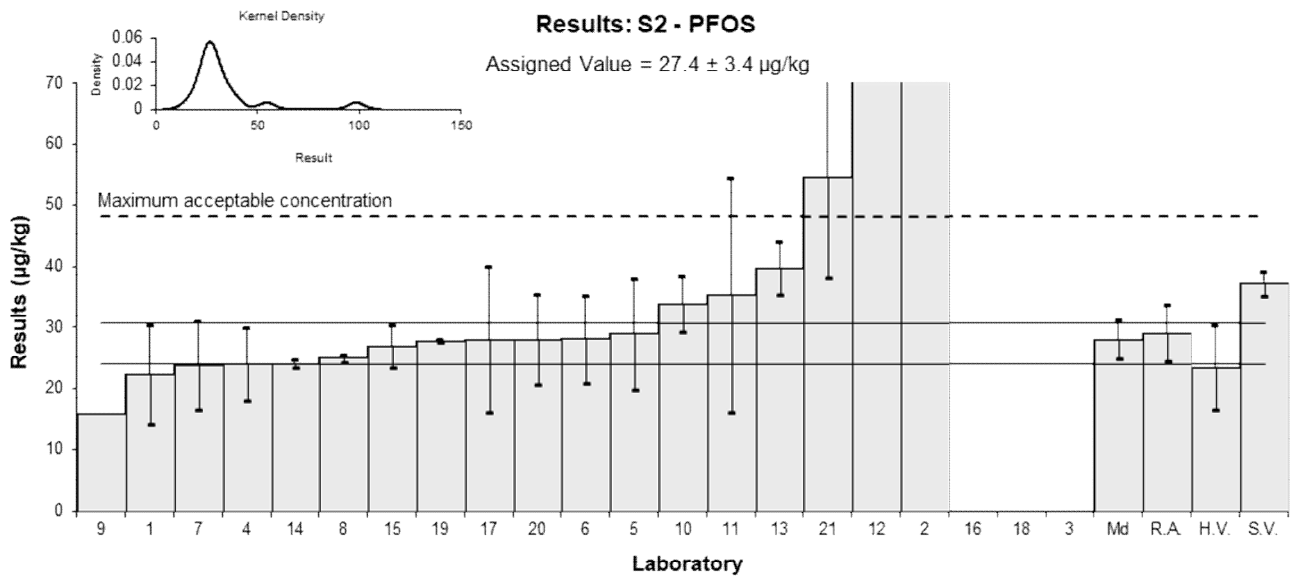


Figure 23

Table 27

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	PFOS (linear)
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	15.8	NR	75.9	-0.78	-1.71
2	664.05	18.3	64	172.55	35.11
3	NT	NT	NT		
4	18.2	5	65	-0.13	-0.09
5	18	5	89	-0.19	-0.13
6	19.8	5	83	0.29	0.21
7	15.6	4.68	71	-0.83	-0.62
8	18	0.18	103	-0.19	-0.41
9	NT	NT	NT		
10	19.6	2.60	98	0.24	0.29
11	22.7	12.3	103	1.07	0.32
12	59.1	11.8	NR	10.80	3.39
13	21.919	3.083	107.3	0.86	0.91
14	15.6	0.48	97	-0.83	-1.75
15	20.568	2.797	NR	0.50	0.57
16	NT	NT	NT		
17	17.54	7.5	127	-0.31	-0.15
18	NT	NT	NT		
19	19.07	0.29	90	0.10	0.21
20	20.3	4.5	74	0.43	0.33
21	35.9	10.8	100	4.60	1.57

## Statistics\*

<b>Assigned Value**</b>	18.7	1.7
<b>Spike</b>	25.8	1.3
<b>Homogeneity Value</b>	17.6	5.3
<b>Robust Average</b>	19.4	2.0
<b>Median</b>	19.3	1.2
<b>Mean</b>	22.4	
<b>N</b>	16	
<b>Max.</b>	59.1	
<b>Min.</b>	15.6	
<b>Robust SD</b>	3.1	
<b>Robust CV</b>	16%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 12 and 21.

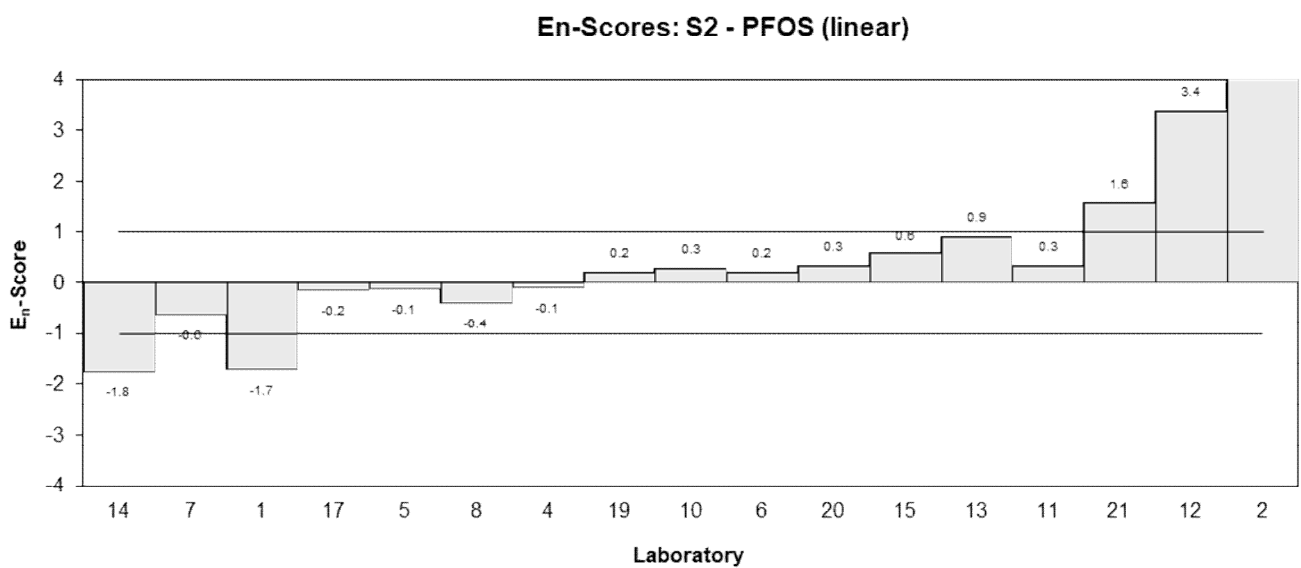
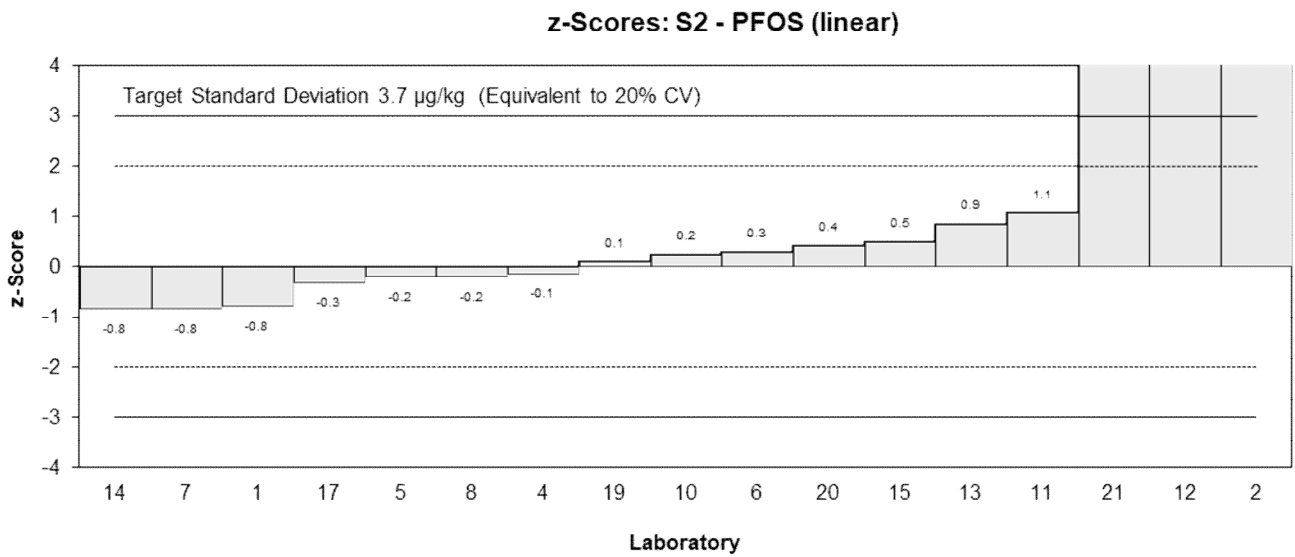
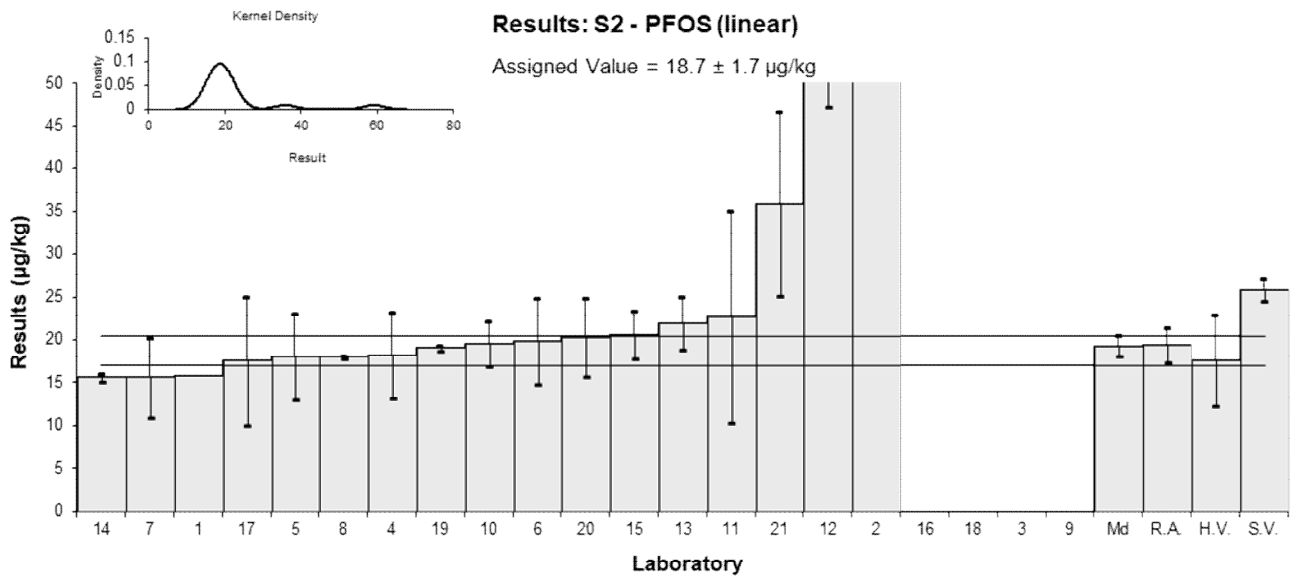


Figure 24

Table 28

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	PFDS
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	11.2	NR	75.9	-0.52	-0.62
2	470.63	37.6	NR	183.25	12.17
3	NT	NT	NT		
4	15.8	4	NR	1.32	0.73
5	13	4	89	0.20	0.11
6	2.6	0.7	NR	-3.96	-4.47
7	14.7	4.41	80	0.88	0.45
8	14	0.44	103	0.60	0.70
9	7.4	NR	NR	-2.04	-2.43
10	8.98	3.15	NR	-1.41	-0.93
11	17.1	4.02	103	1.84	1.01
12	NT	NT	NT		
13	1.256	2.594	107.3	-4.50	-3.37
14	13.9	0.32	97	0.56	0.66
15	10.382	1.845	NR	-0.85	-0.76
16	NT	NT	NT		
17	10.6	4.0	127	-0.76	-0.42
18	NT	NT	NT		
19	13.2	0.27	90	0.28	0.33
20	11.2	7.3	74	-0.52	-0.17
21	17.9	5.37	97.3	2.16	0.94

## Statistics\*

<b>Assigned Value**</b>	12.5	2.1
<b>Spike</b>	14.4	0.7
<b>Robust Average</b>	11.9	2.7
<b>Median</b>	12.1	1.8
<b>Mean</b>	11.5	
<b>N</b>	16	
<b>Max.</b>	17.9	
<b>Min.</b>	1.256	
<b>Robust SD</b>	4.3	
<b>Robust CV</b>	36%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 6, 13 and 21.



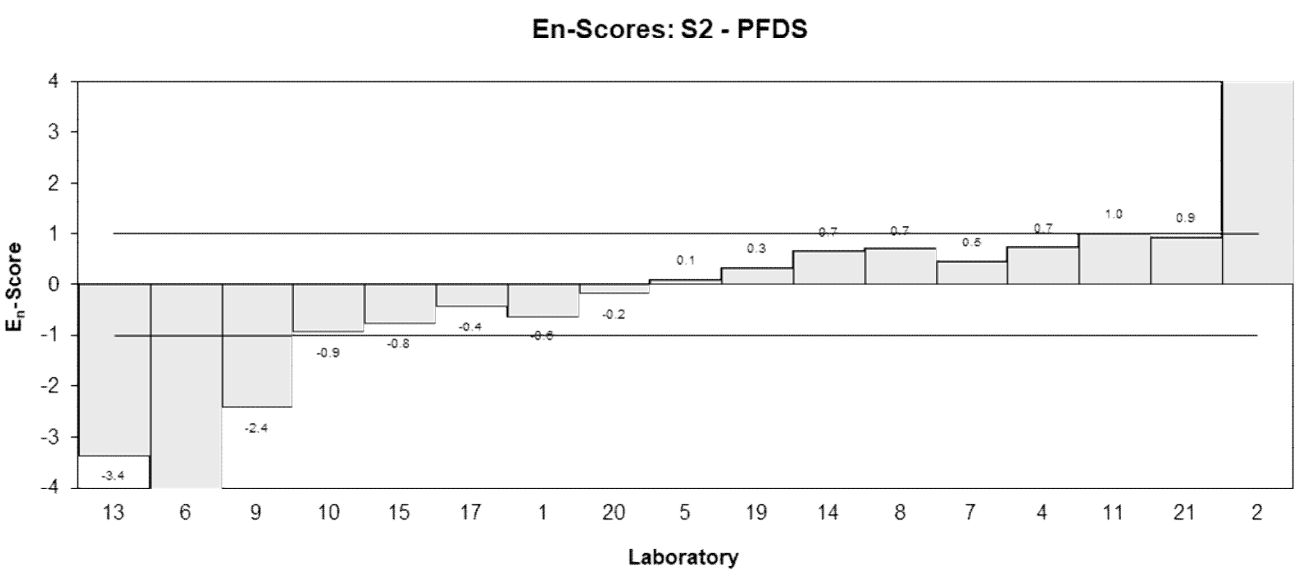
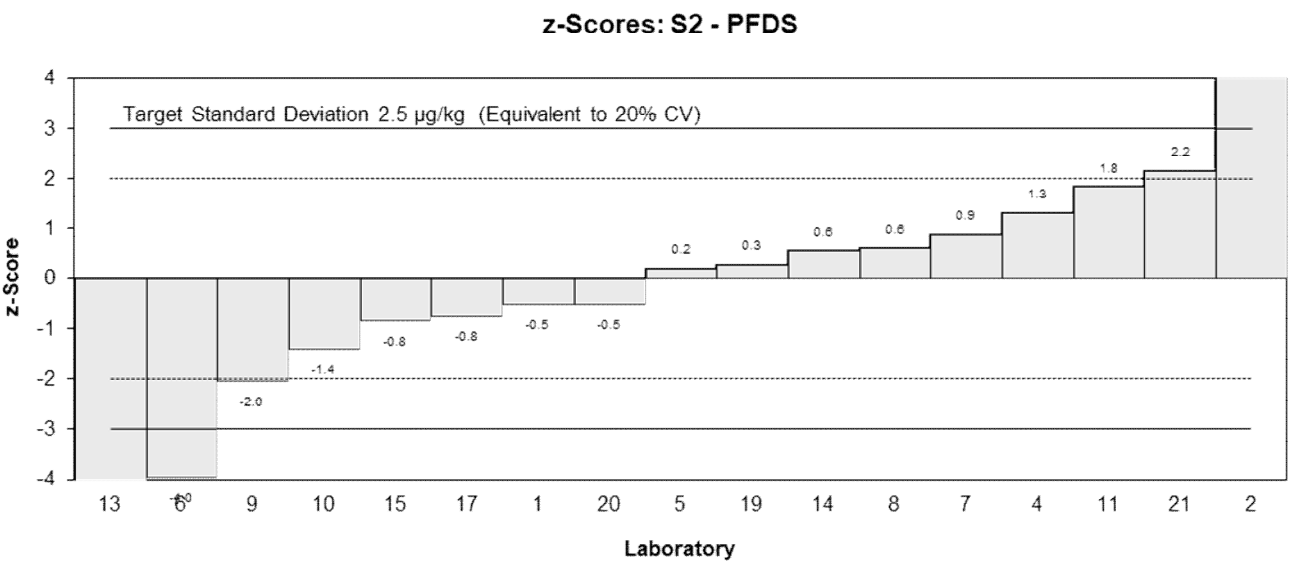
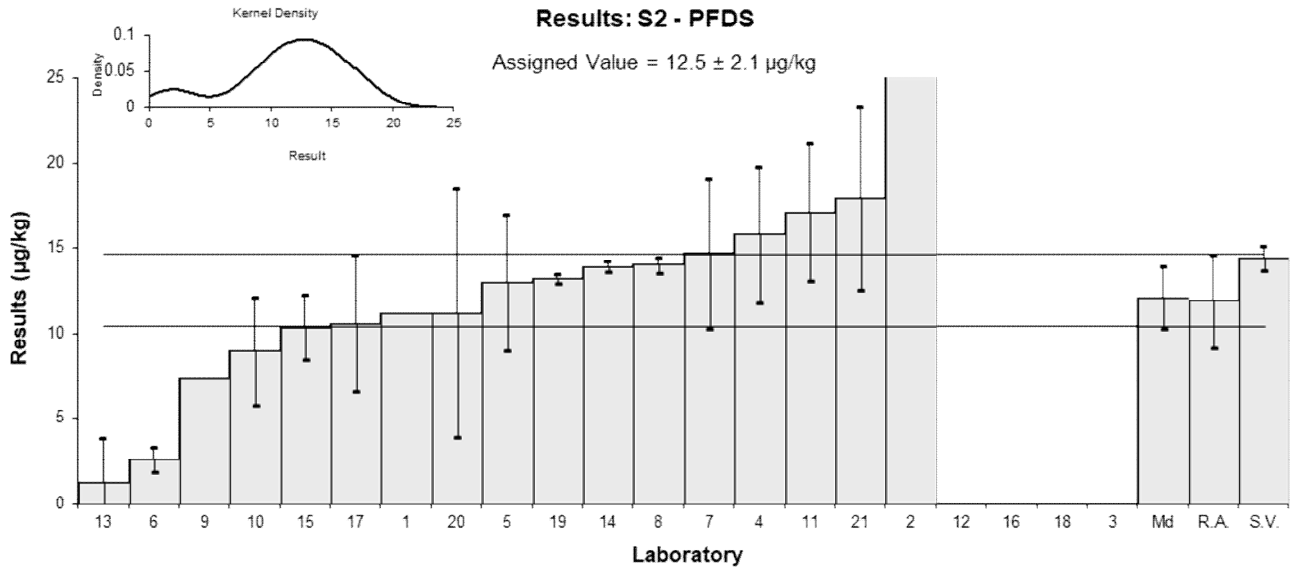


Figure 25

Table 29

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	PFBA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	40.5	3.564	75.9	6.25	5.88
2	682.48	71.13	75	184.58	9.34
3	NT	NT	NT		
4	20.6	5	71	0.72	0.50
5	18	5	90	0.00	0.00
6	17.4	4.4	59	-0.17	-0.13
7	15.3	4.59	79	-0.75	-0.56
8	18	0.16	111	0.00	0.00
9	NR	NR	NR		
10	17.4	2.39	95	-0.17	-0.22
11	13.2	3.03	75	-1.33	-1.44
12	NT	NT	NT		
13	19.786	1.065	83.0	0.50	1.02
14	16.6	0.36	97	-0.39	-0.97
15	17.615	1.901	NR	-0.11	-0.16
16	NT	NT	NT		
17	18.4	8.0	131	0.11	0.05
18	NT	NT	NT		
19	20.4	0.45	94	0.67	1.63
20	20	5.1	60	0.56	0.38
21	31.4	9.42	58.2	3.72	1.41

## Statistics\*

<b>Assigned Value**</b>	18.0	1.4
<b>Spike</b>	19.3	1.0
<b>Homogeneity Value</b>	20.6	6.2
<b>Robust Average</b>	18.6	1.7
<b>Median</b>	18.0	1.5
<b>Mean</b>	20.3	
<b>N</b>	15	
<b>Max.</b>	40.5	
<b>Min.</b>	13.2	
<b>Robust SD</b>	2.7	
<b>Robust CV</b>	15%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 1 and 21.

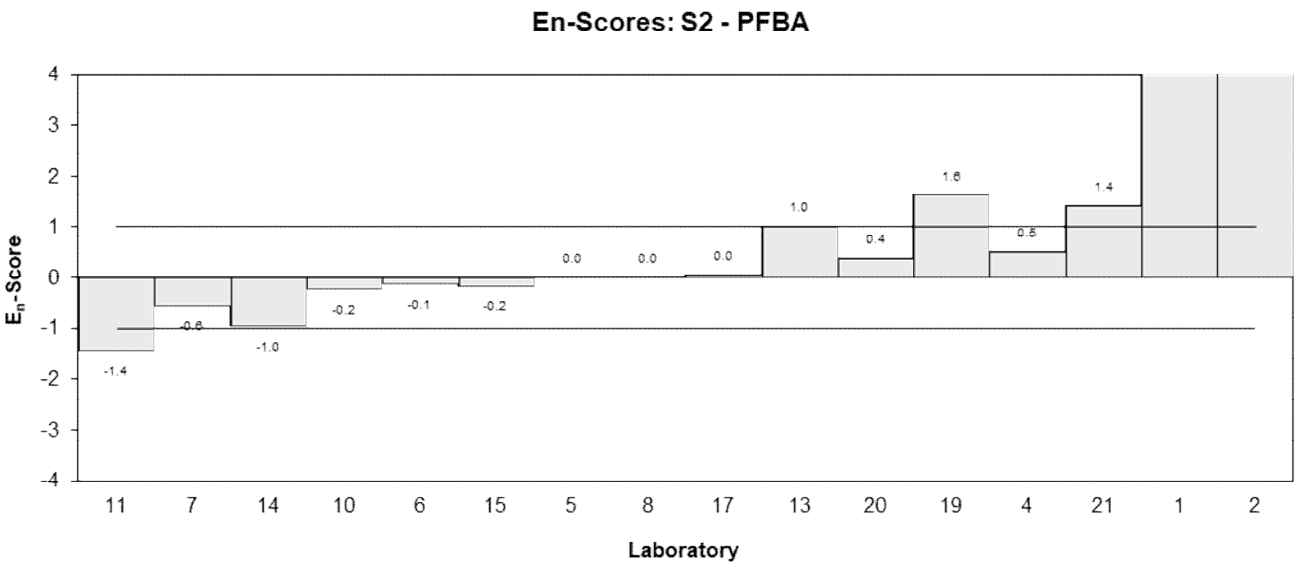
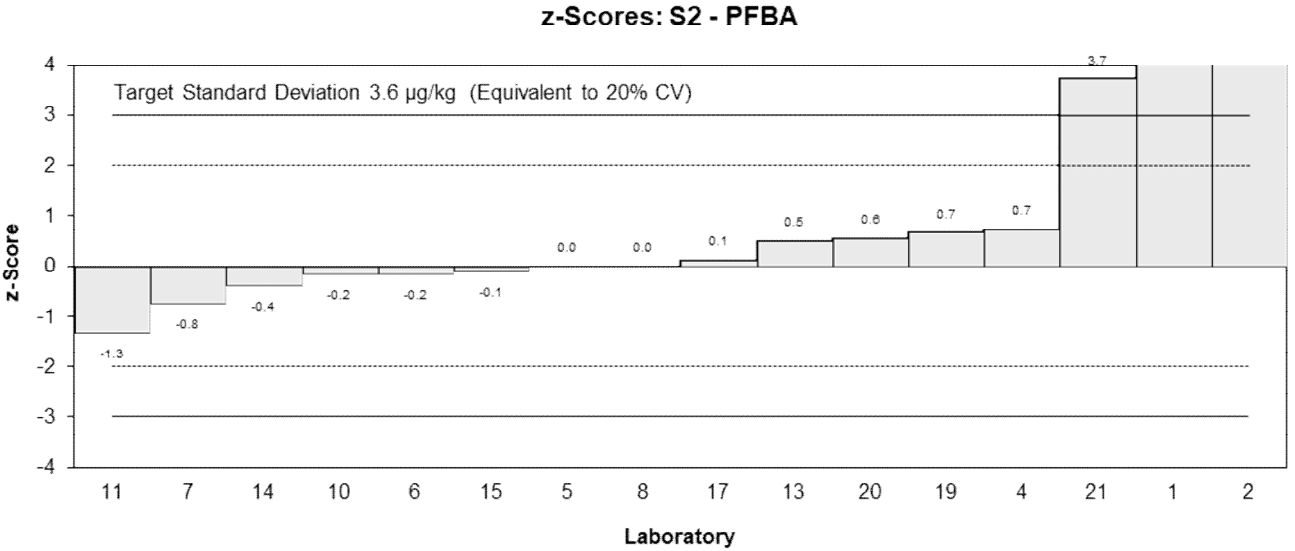
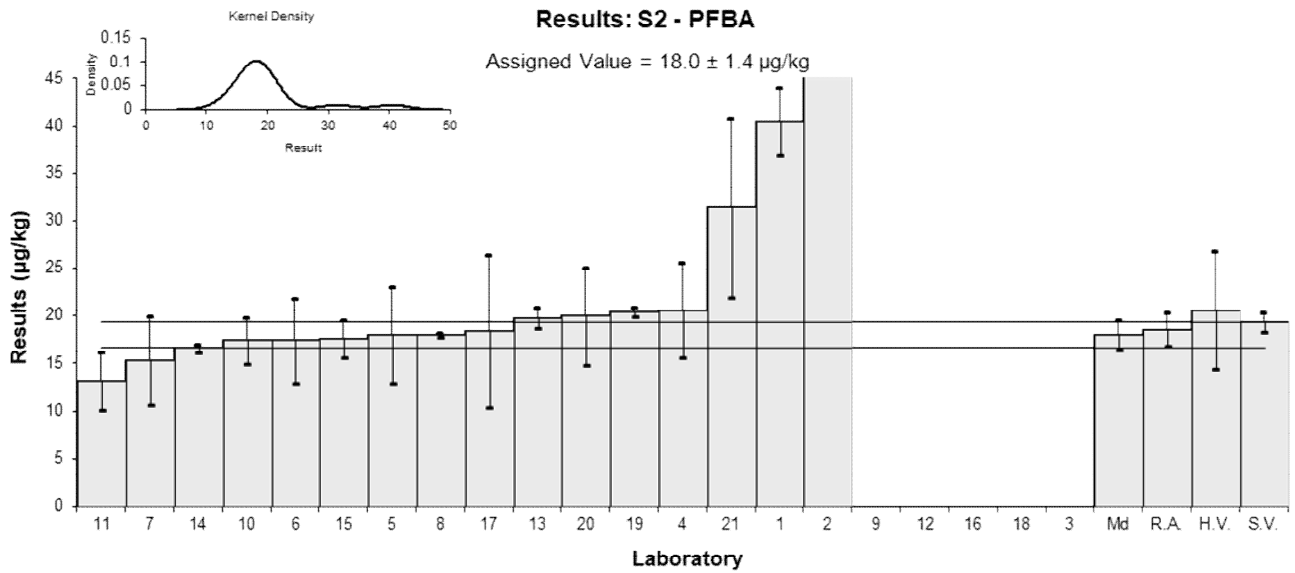


Figure 26

Table 30

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	PFPeA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	28.4	3.976	75.9	0.36	0.41
2	1052.74	97.47	51	193.63	10.53
3	NT	NT	NT		
4	28.2	7	66	0.32	0.23
5	31	10	74	0.85	0.44
6	21	5.3	75	-1.04	-0.95
7	24.7	7.41	82	-0.34	-0.23
8	26	0.18	108	-0.09	-0.21
9	23.4	NR	NR	-0.58	-1.29
10	26.1	3.58	106	-0.08	-0.09
11	17.9	1.6	72	-1.62	-2.98
12	NT	NT	NT		
13	31.597	3.340	77.4	0.96	1.24
14	25.6	0.58	97	-0.17	-0.36
15	23.864	2.551	NR	-0.50	-0.75
16	NT	NT	NT		
17	29.0	10.0	126	0.47	0.24
18	NT	NT	NT		
19	28.3	0.25	94	0.34	0.75
20	28.8	4.7	57.1	0.43	0.44
21	45.3	0.9	67.8	3.55	7.33

## Statistics\*

<b>Assigned Value**</b>	26.5	2.4
<b>Spike</b>	30.1	1.5
<b>Homogeneity Value</b>	27.1	8.1
<b>Robust Average</b>	26.9	2.5
<b>Median</b>	27.2	1.7
<b>Mean</b>	27.4	
<b>N</b>	16	
<b>Max.</b>	45.3	
<b>Min.</b>	17.9	
<b>Robust SD</b>	4.0	
<b>Robust CV</b>	15%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratory 21.

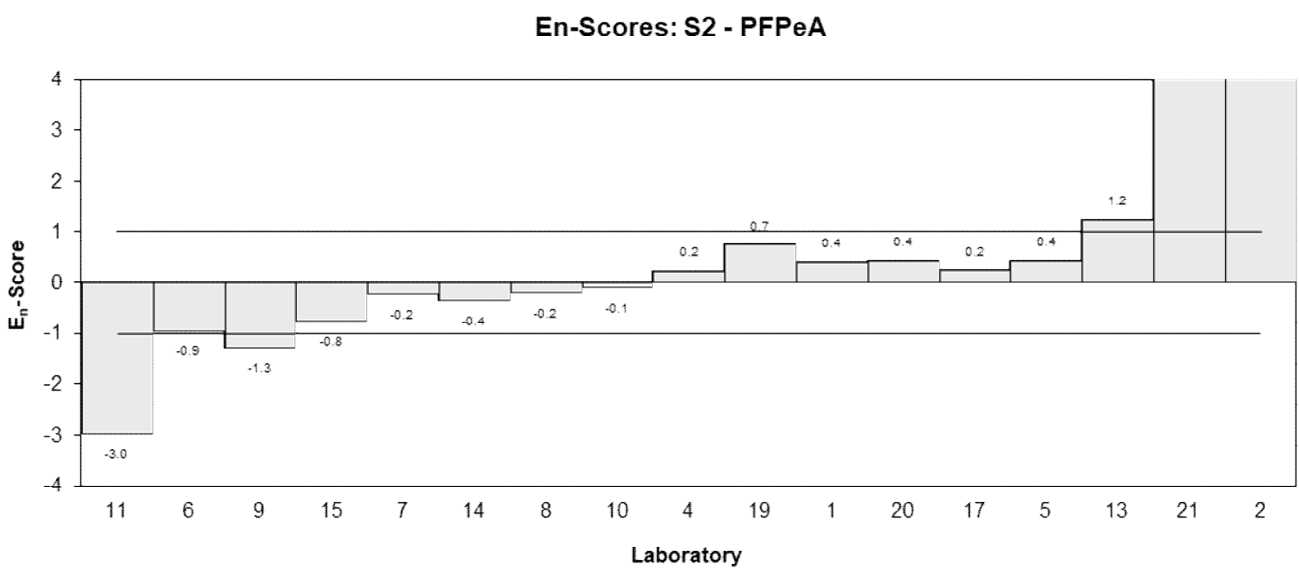
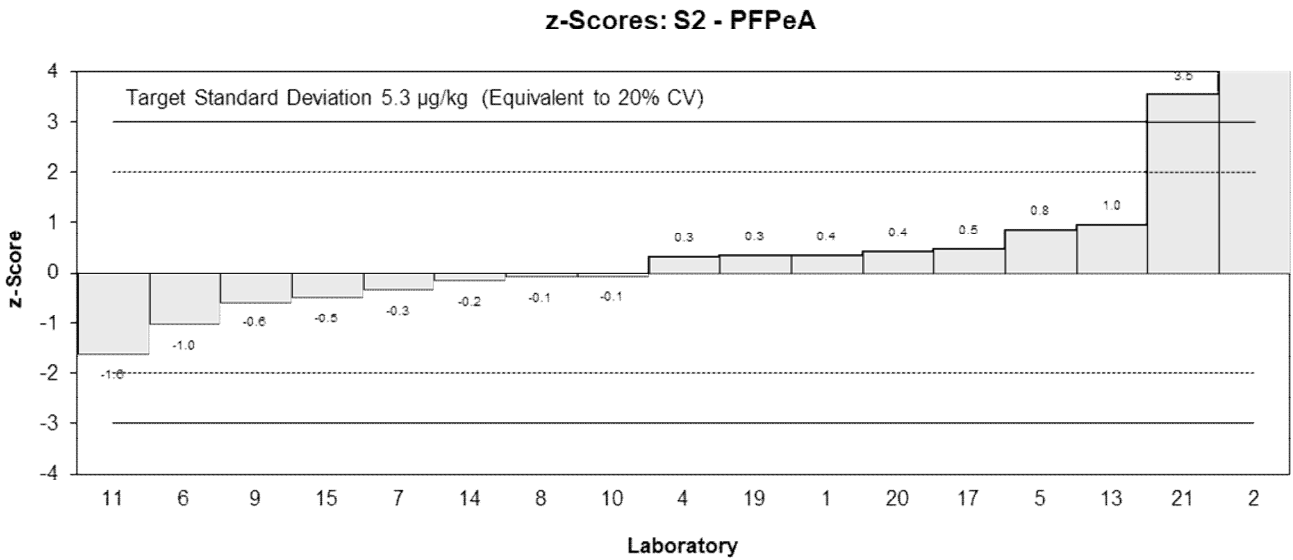
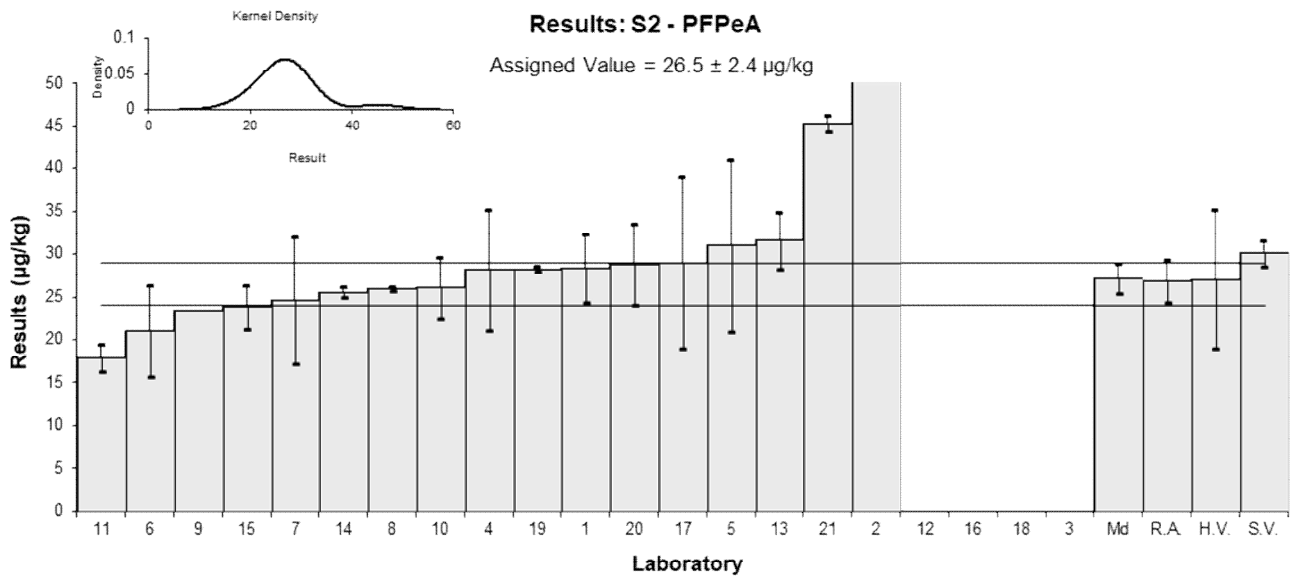


Figure 27

Table 31

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	PFHxA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	29.9	NR	75.9	-0.70	-1.29
2	1095.27	29.39	41	152.37	35.78
3	NT	NT	NT		
4	39.6	10	63	0.69	0.45
5	35	10	96	0.03	0.02
6	35.6	8.9	75	0.11	0.08
7	31.6	9.49	89	-0.46	-0.31
8	36	1.3	108	0.17	0.30
9	30	NR	NR	-0.69	-1.26
10	36.7	0.03	98	0.27	0.50
11	27.7	4.4	93	-1.02	-1.22
12	NT	NT	NT		
13	41.710	2.835	70.0	0.99	1.46
14	31.4	0.74	97	-0.49	-0.88
15	24.083	2.747	NR	-1.54	-2.29
16	NT	NT	NT		
17	41.1	16.0	115	0.91	0.38
18	NT	NT	NT		
19	42.4	0.29	73	1.09	1.99
20	38	10	65.5	0.46	0.30
21	62.8	18.8	80.8	4.02	1.46

## Statistics\*

<b>Assigned Value**</b>	34.8	3.8
<b>Spike</b>	33.9	1.7
<b>Homogeneity Value</b>	38	11
<b>Robust Average</b>	35.5	4.0
<b>Median</b>	35.8	3.8
<b>Mean</b>	36.5	
<b>N</b>	16	
<b>Max.</b>	62.8	
<b>Min.</b>	24.083	
<b>Robust SD</b>	6.4	
<b>Robust CV</b>	18%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratory 21.

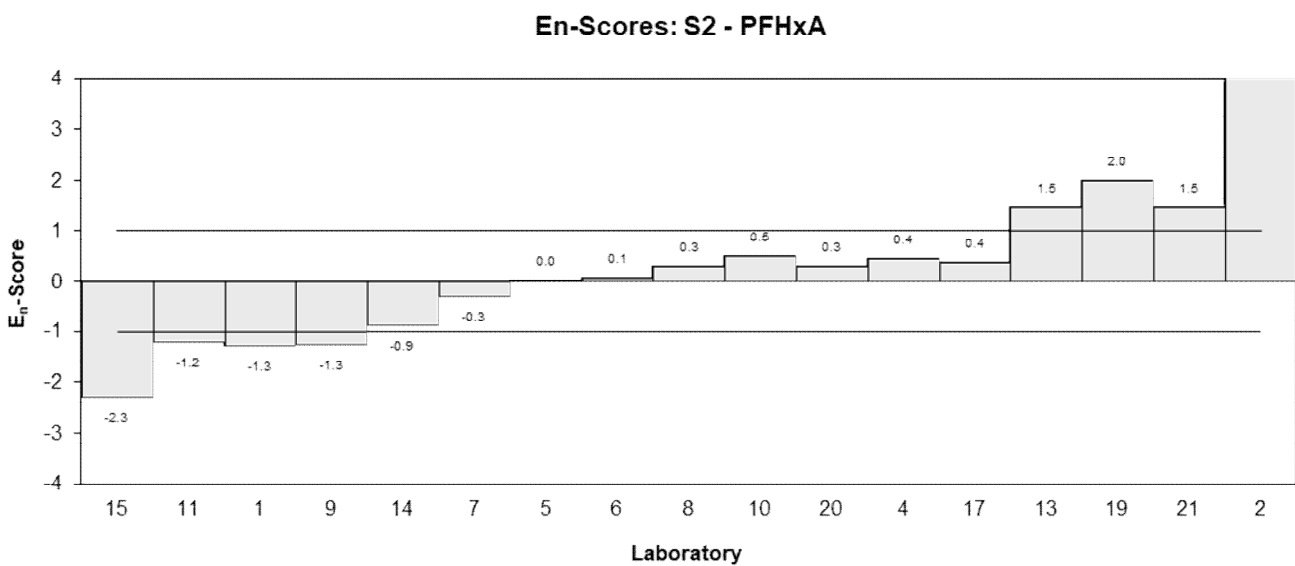
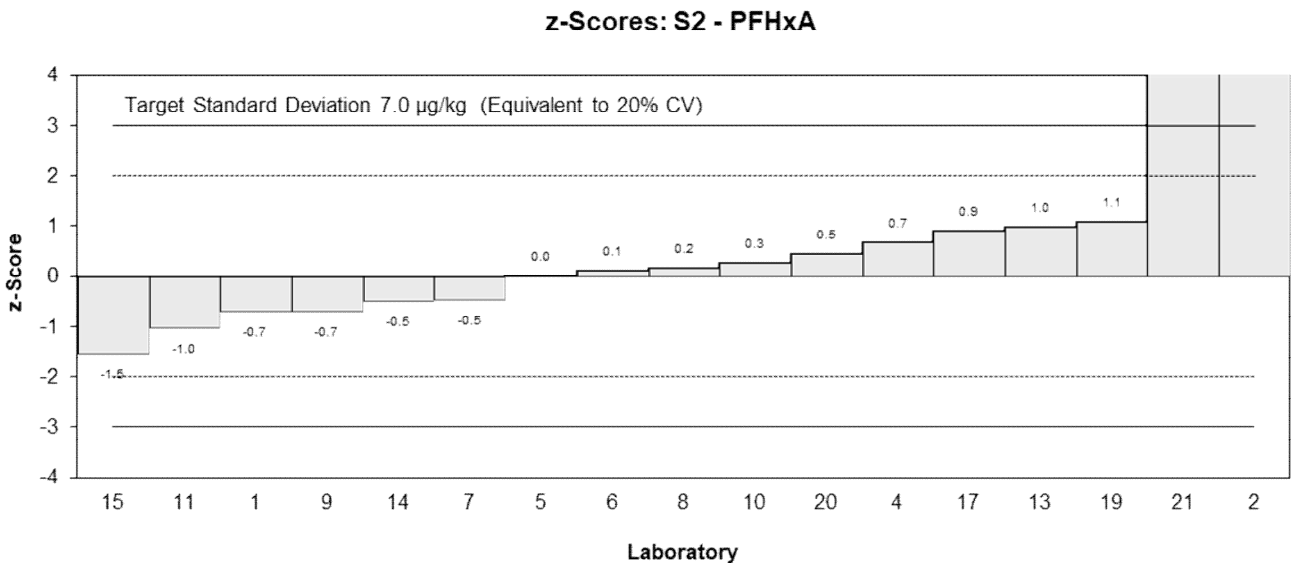
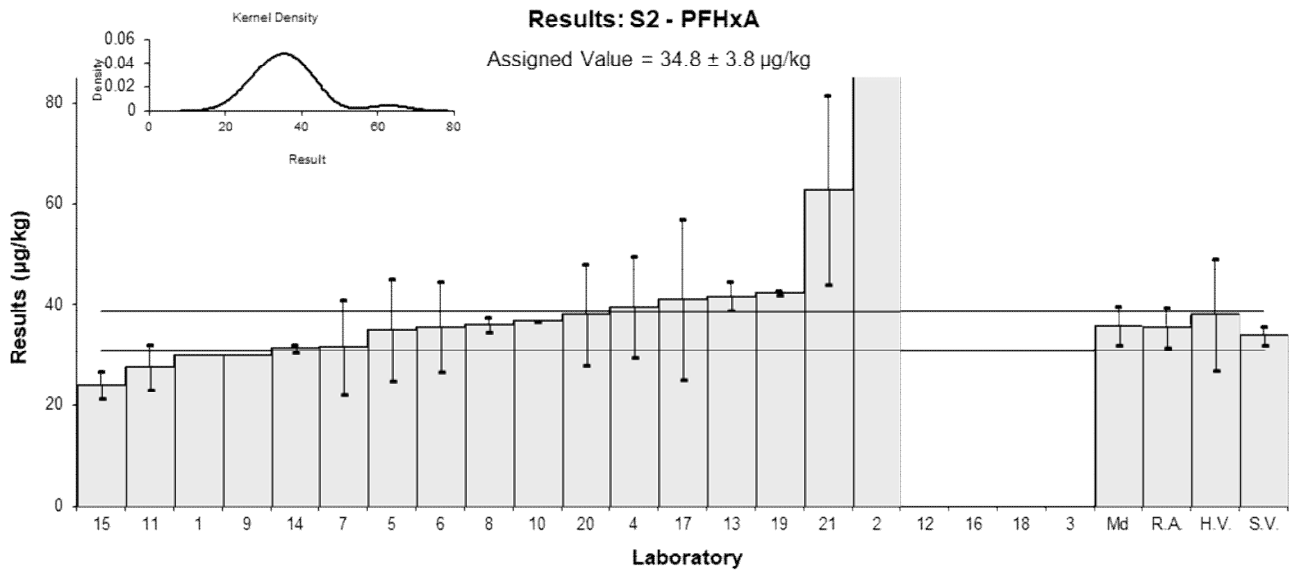


Figure 28

Table 32

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	PFOA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	11.7	2.4921	75.9	-1.05	-1.07
2	452.75	41.5	81	147.96	10.55
3	NT	NT	NT		
4	14.8	4	79	0.00	0.00
5	15	4	101	0.07	0.05
6	13.5	3.38	72	-0.44	-0.35
7	13.0	3.91	76	-0.61	-0.43
8	15	1.8	101	0.07	0.09
9	10.6	NR	NR	-1.42	-2.80
10	14.40	2.67	85	-0.14	-0.13
11	17.3	2.3	92	0.84	0.91
12	40.4	8.1	NR	8.65	3.11
13	16.565	1.994	36.0	0.60	0.71
14	13.2	0.31	97	-0.54	-1.04
15	14.504	1.484	NR	-0.10	-0.14
16	NT	NT	NT		
17	17.0	6.2	125	0.74	0.34
18	NT	NT	NT		
19	18.3	0.27	85	1.18	2.30
20	16.8	3.2	75.4	0.68	0.57
21	30.9	9.27	86.6	5.44	1.71

## Statistics\*

<b>Assigned Value**</b>	14.8	1.5
<b>Spike</b>	18.2	0.9
<b>Homogeneity Value</b>	14.4	4.3
<b>Robust Average</b>	15.4	1.8
<b>Median</b>	15.0	1.4
<b>Mean</b>	17.2	
<b>N</b>	17	
<b>Max.</b>	40.4	
<b>Min.</b>	10.6	
<b>Robust SD</b>	2.9	
<b>Robust CV</b>	19%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratories 12 and 21.



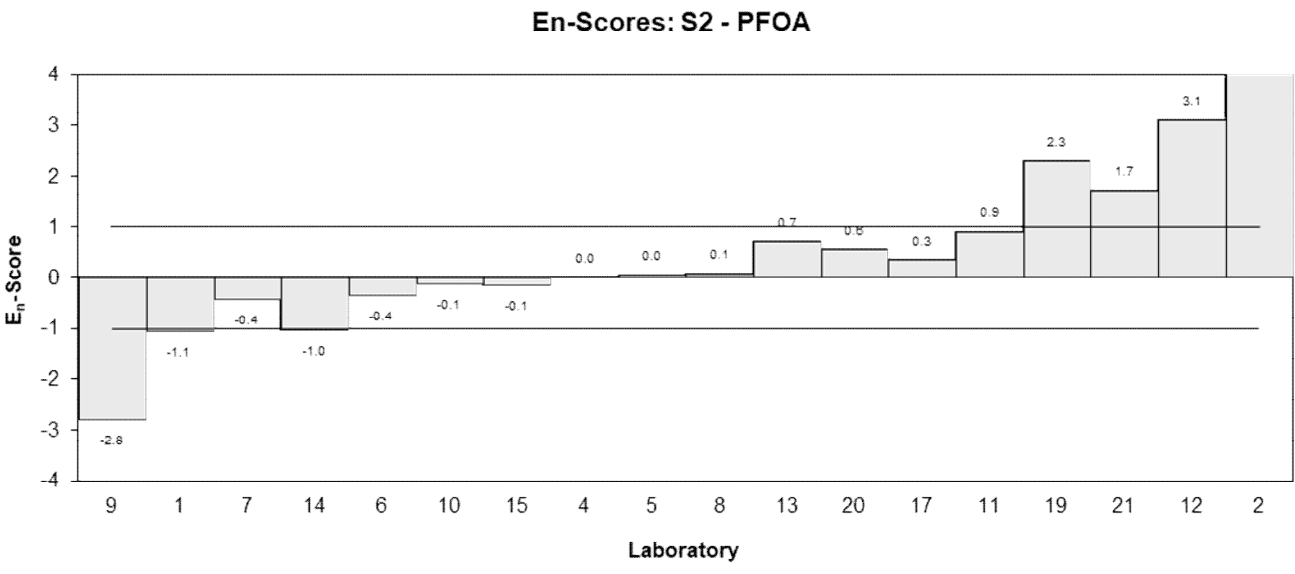
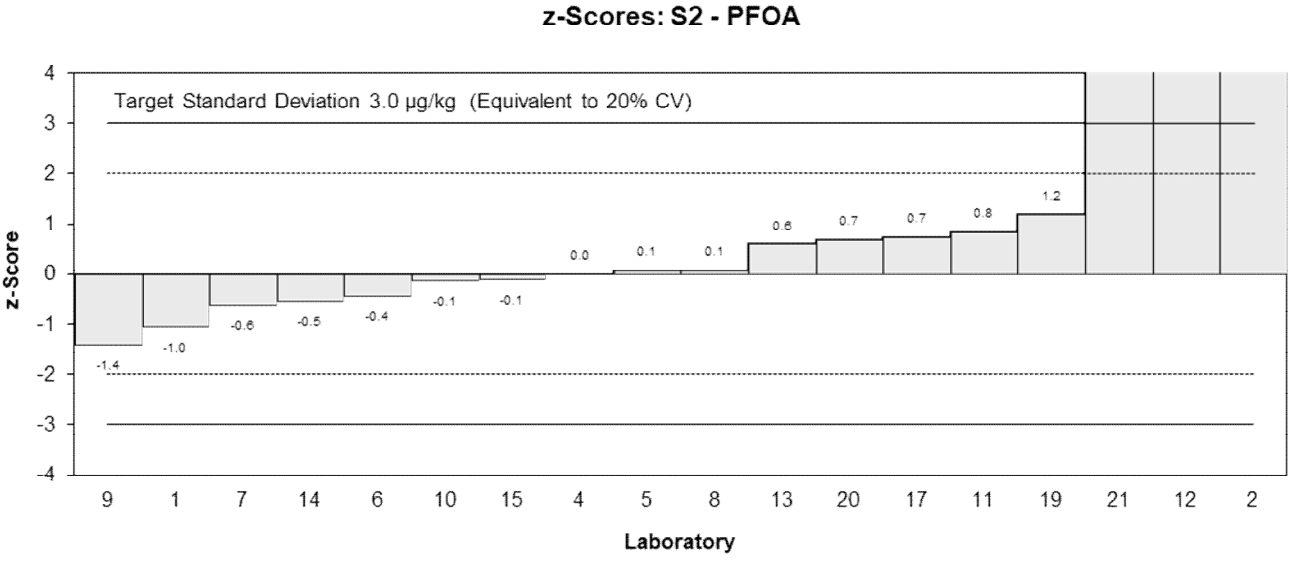
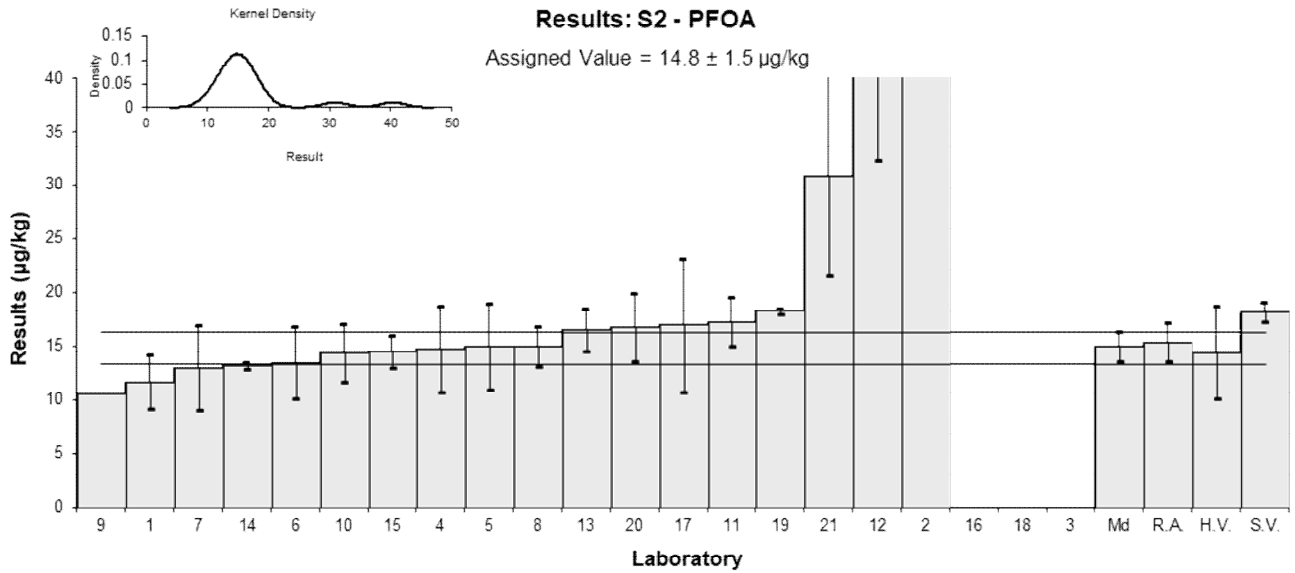


Figure 29

Table 33

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	PFNA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	3.9	0.585	75.9	0.45	0.43
2	104.96	1.42	69	141.59	67.92
3	NT	NT	NT		
4	3.66	1	70	0.11	0.07
5	3	1	94	-0.81	-0.53
6	2.65	0.66	71	-1.30	-1.16
7	2.90	0.871	80	-0.95	-0.69
8	3.8	0.008	125	0.31	0.48
9	1.88	NR	NR	-2.37	-3.70
10	3.49	0.59	114	-0.13	-0.12
11	4.92	1	97	1.87	1.22
12	NT	NT	NT		
13	4.365	1.069	13.4	1.10	0.67
14	3.31	0.079	97	-0.38	-0.58
15	3.63	0.42	NR	0.07	0.08
16	NT	NT	NT		
17	3.58	1.3	140	0.00	0.00
18	NT	NT	NT		
19	4.35	0.29	111	1.08	1.42
20	3.9	1.2	76.6	0.45	0.25
21	6.25	1.88	97.7	3.73	1.38

## Statistics\*

<b>Assigned Value**</b>	3.58	0.46
<b>Spike</b>	4.53	0.23
<b>Homogeneity Value</b>	3.5	1.1
<b>Robust Average</b>	3.67	0.51
<b>Median</b>	3.65	0.39
<b>Mean</b>	3.72	
<b>N</b>	16	
<b>Max.</b>	6.25	
<b>Min.</b>	1.88	
<b>Robust SD</b>	0.82	
<b>Robust CV</b>	22%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratory 21.

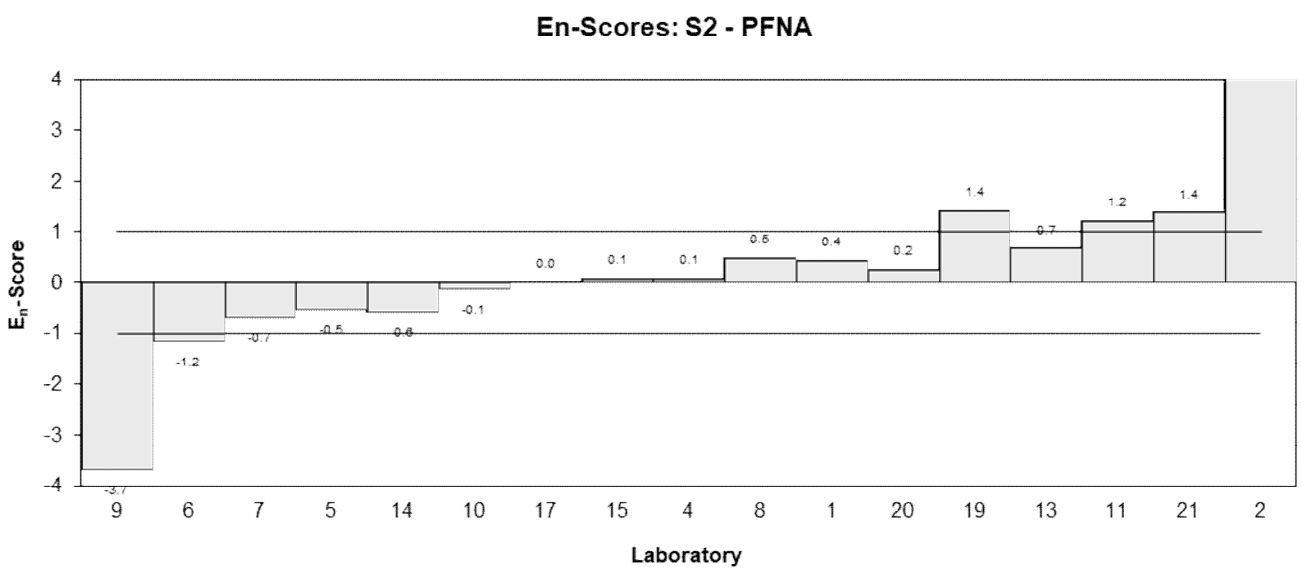
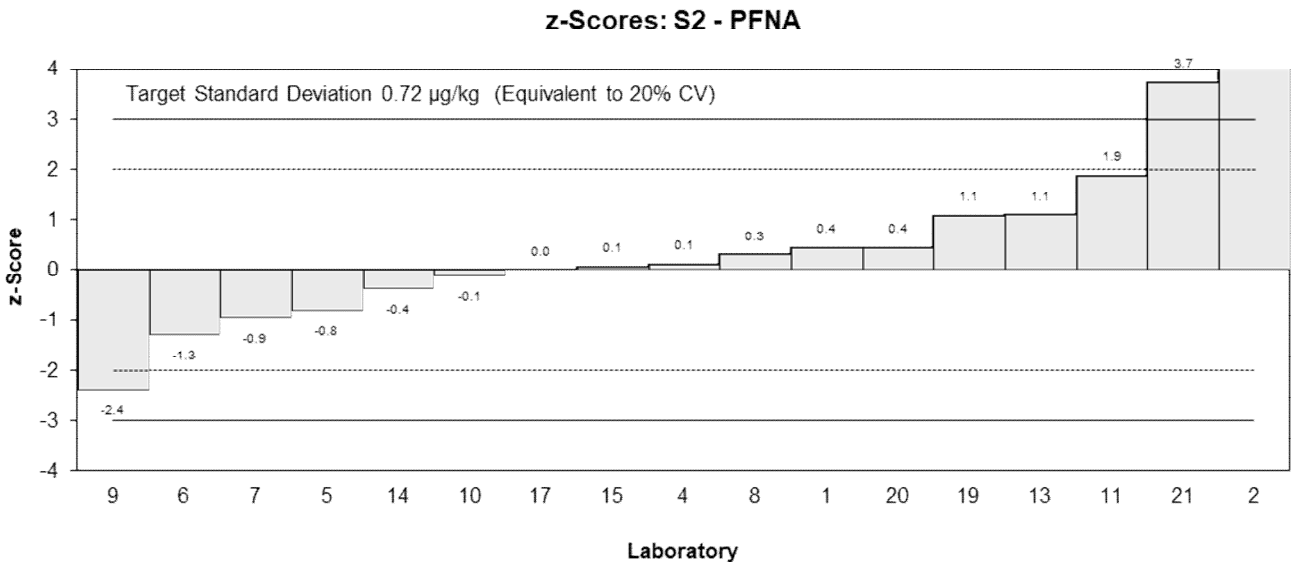
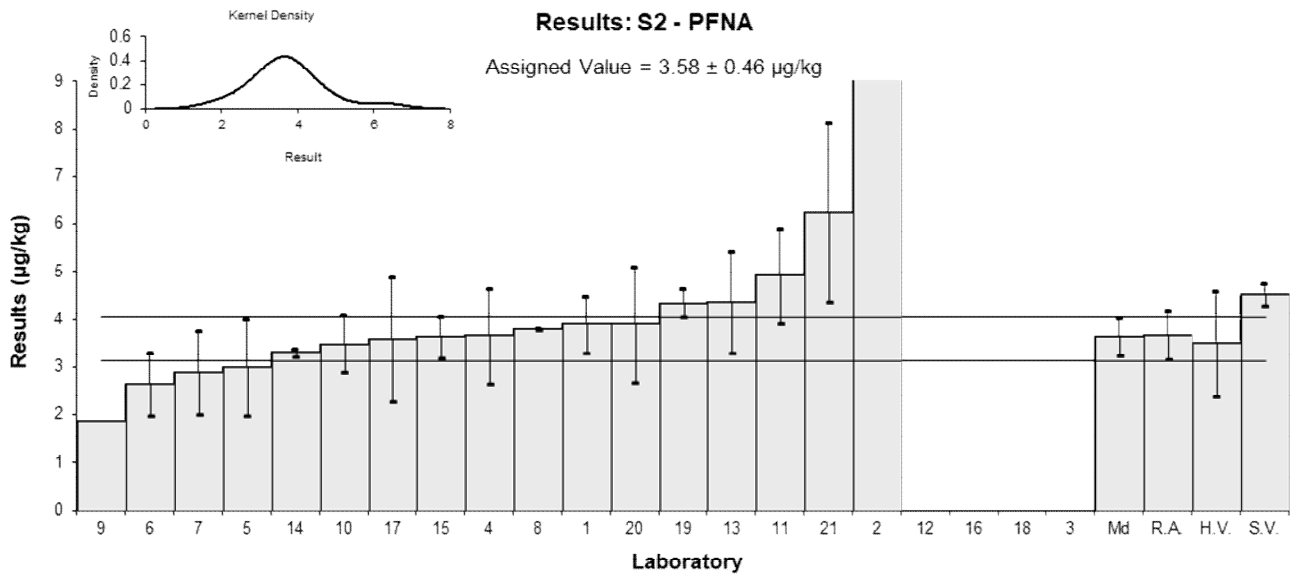


Figure 30

Table 34

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	PFDA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	2.6	0.2158	75.9	-0.50	-0.78
2	85.64	2.92	107	143.17	28.19
3	NT	NT	NT		
4	2.97	1	67	0.14	0.08
5	3	1	95	0.19	0.11
6	2.65	0.66	45	-0.42	-0.33
7	2.58	0.773	79	-0.54	-0.37
8	2.8	0.25	112	-0.16	-0.23
9	1.53	NR	NR	-2.35	-4.53
10	2.45	0.43	121	-0.76	-0.84
11	4.12	0.8	99	2.13	1.44
12	NT	NT	NT		
13	3.434	1.317	4.0	0.94	0.40
14	2.61	0.061	97	-0.48	-0.91
15	2.743	0.344	NR	-0.25	-0.32
16	NT	NT	NT		
17	3.18	1.2	147	0.50	0.23
18	NT	NT	NT		
19	3.55	0.27	81	1.14	1.64
20	3.06	0.84	72.6	0.29	0.19
21	4.91	1.47	112	3.49	1.35

## Statistics\*

<b>Assigned Value**</b>	2.89	0.30
<b>Spike</b>	3.33	0.17
<b>Homogeneity Value</b>	3.09	0.93
<b>Robust Average</b>	2.96	0.34
<b>Median</b>	2.89	0.23
<b>Mean</b>	3.01	
<b>N</b>	16	
<b>Max.</b>	4.91	
<b>Min.</b>	1.53	
<b>Robust SD</b>	0.54	
<b>Robust CV</b>	18%	

\* Laboratory 2 was omitted from all statistical calculations for all analytes, as they reported results based on the dry sample instead of as received.

\*\* Robust average excluding Laboratory 21.

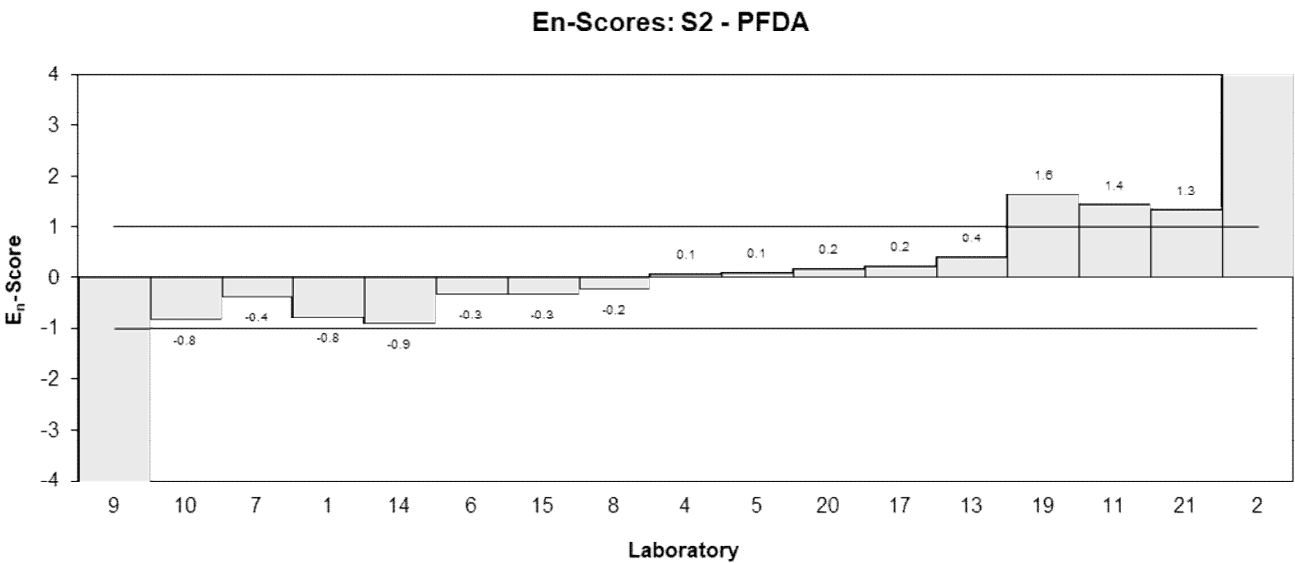
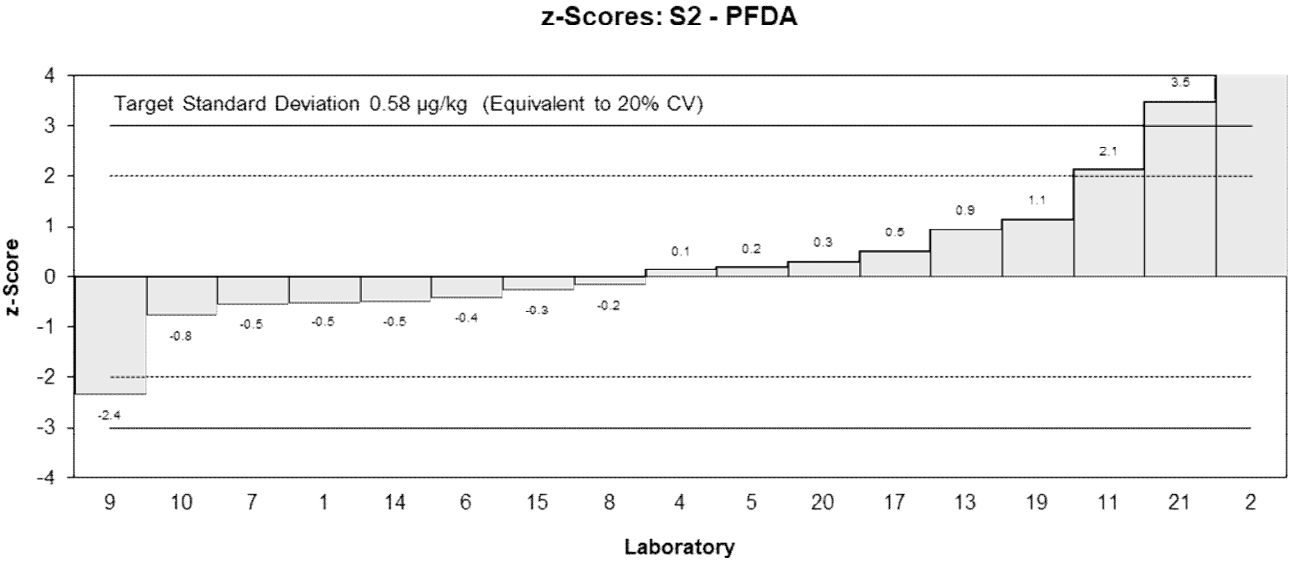
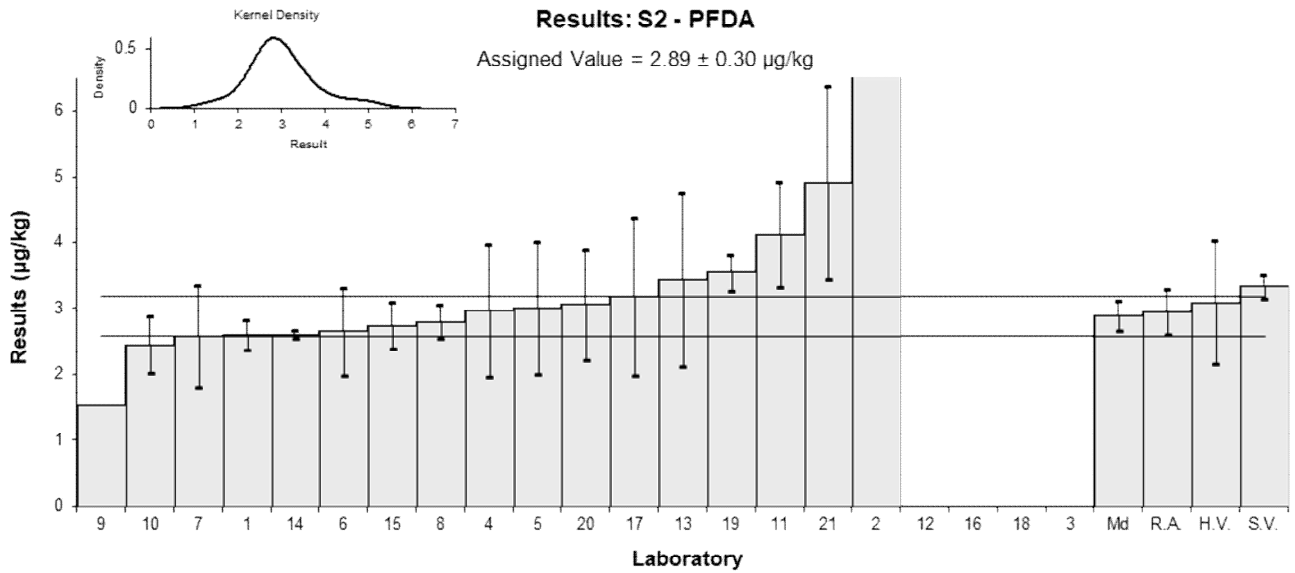


Figure 31

Table 35

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	ADONA
<b>Units</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	20	NR	75.9	-0.50	-0.88
2	<0.25	NR	NR		
3	NT	NT	NT		
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	20.6	4.12	82	-0.36	-0.33
8	NT	NT	NT		
9	NT	NT	NT		
10	21.24	6.37	NR	-0.22	-0.14
11	22.7	5.8	92	0.11	0.08
12	NT	NT	NT		
13	23.706	4.571	73.9	0.34	0.29
14	24.2	0.55	97	0.45	0.78
15	19.071	2	NR	-0.70	-0.98
16	NT	NT	NT		
17	NT	NT	NT		
18	NT	NT	NT		
19	28.2	0.36	86	1.35	2.38
20	NT	NT	NT		
21	NT	NT	NT		

## Statistics

<b>Assigned Value</b>	22.2	2.5
<b>Spike</b>	27.4	1.4
<b>Robust Average</b>	22.2	2.5
<b>Median</b>	22.0	2.3
<b>Mean</b>	22.5	
<b>N</b>	8	
<b>Max.</b>	28.2	
<b>Min.</b>	19.071	
<b>Robust SD</b>	2.8	
<b>Robust CV</b>	13%	

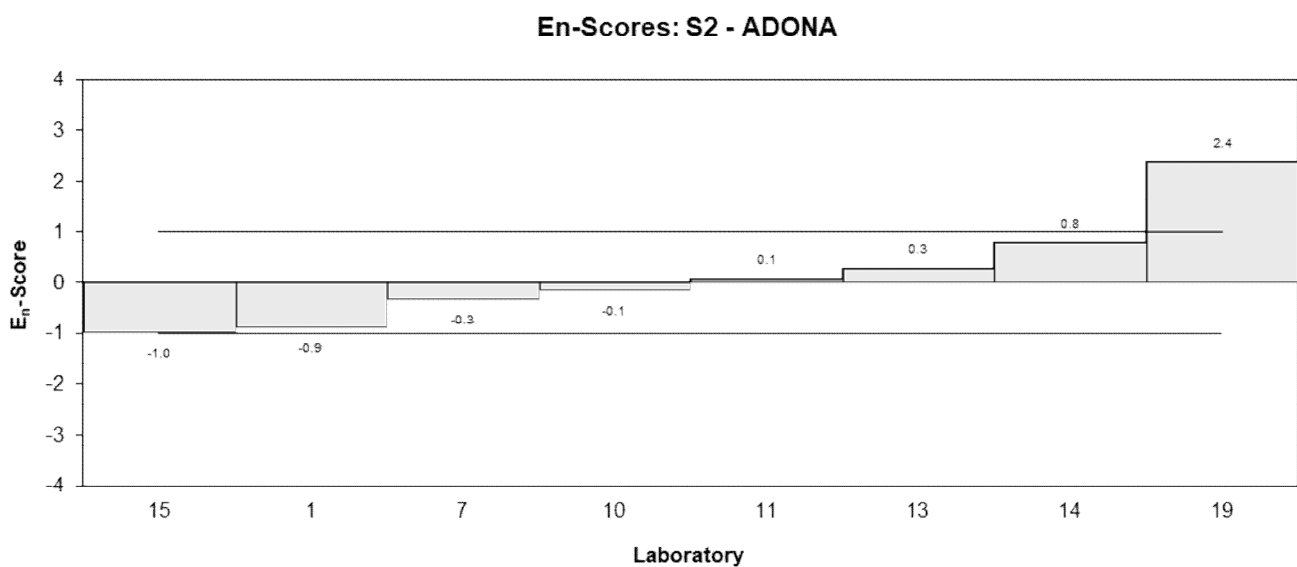
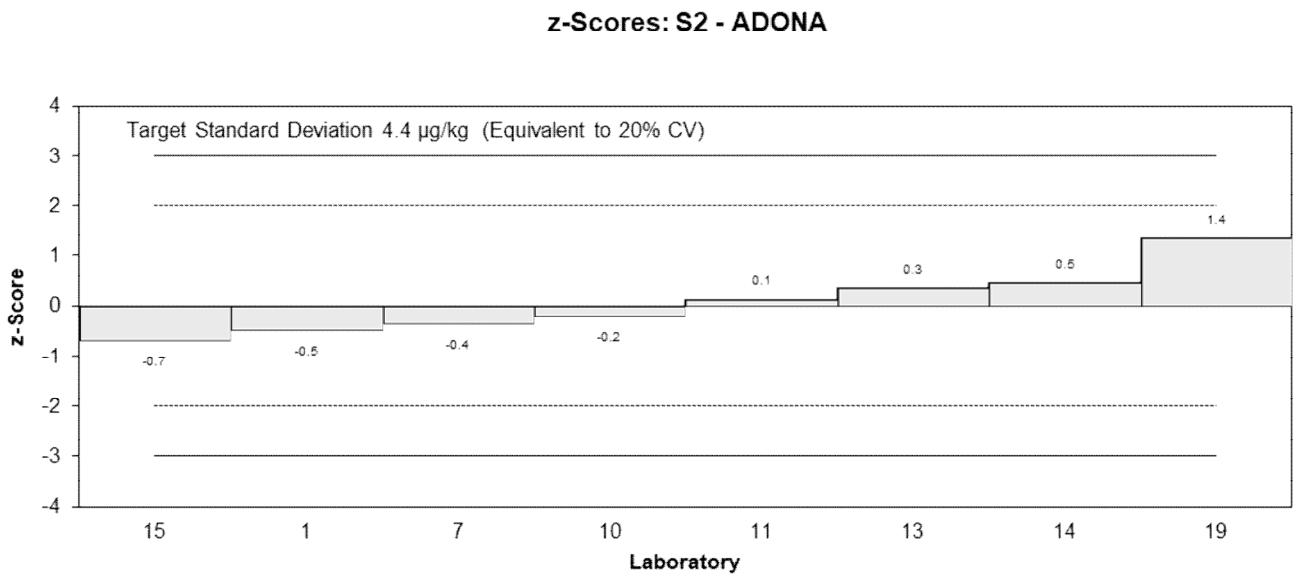
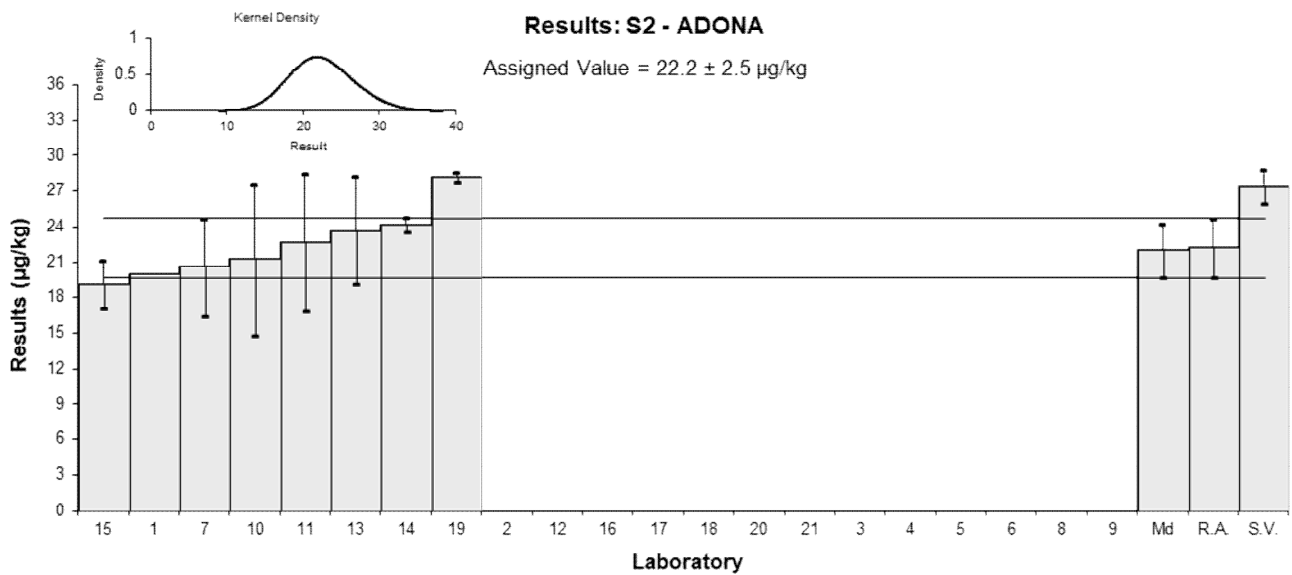


Figure 32

Table 36

**Sample Details**

<b>Sample No.</b>	S2
<b>Matrix</b>	Lettuce
<b>Analyte</b>	GenX
<b>Units</b>	µg/kg

**Participant Results**

Lab. Code	Result	Uncertainty	Recovery	z-Score	E <sub>n</sub> -Score
1	15.8	NR	75.9	-1.05	-1.56
2	NT	NT	NT		
3	NT	NT	NT		
4	NT	NT	NT		
5	NT	NT	NT		
6	18	4.5	51	-0.50	-0.38
7	21.0	4.20	84	0.25	0.20
8	NT	NT	NT		
9	NT	NT	NT		
10	22.36	6.71	124	0.59	0.33
11	16.9	3.3	87	-0.78	-0.73
12	NT	NT	NT		
13	22.297	9.090	45.5	0.57	0.24
14	22.4	0.44	97	0.60	0.88
15	NT	NT	NT		
16	NT	NT	NT		
17	NT	NT	NT		
18	NT	NT	NT		
19	21.1	0.44	71	0.28	0.40
20	NT	NT	NT		
21	NT	NT	NT		

**Statistics**

<b>Assigned Value</b>	20.0	2.7
<b>Spike</b>	20.8	1.0
<b>Robust Average</b>	20.0	2.7
<b>Median</b>	21.1	1.6
<b>Mean</b>	20.0	
<b>N</b>	8	
<b>Max.</b>	22.4	
<b>Min.</b>	15.8	
<b>Robust SD</b>	3.0	
<b>Robust CV</b>	15%	



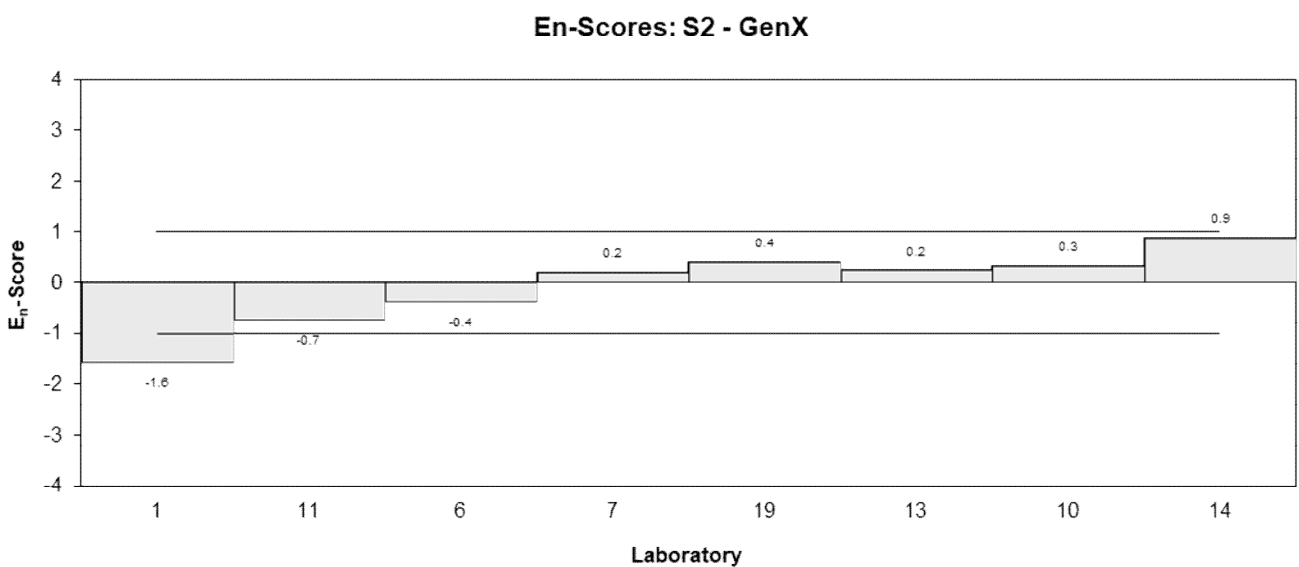
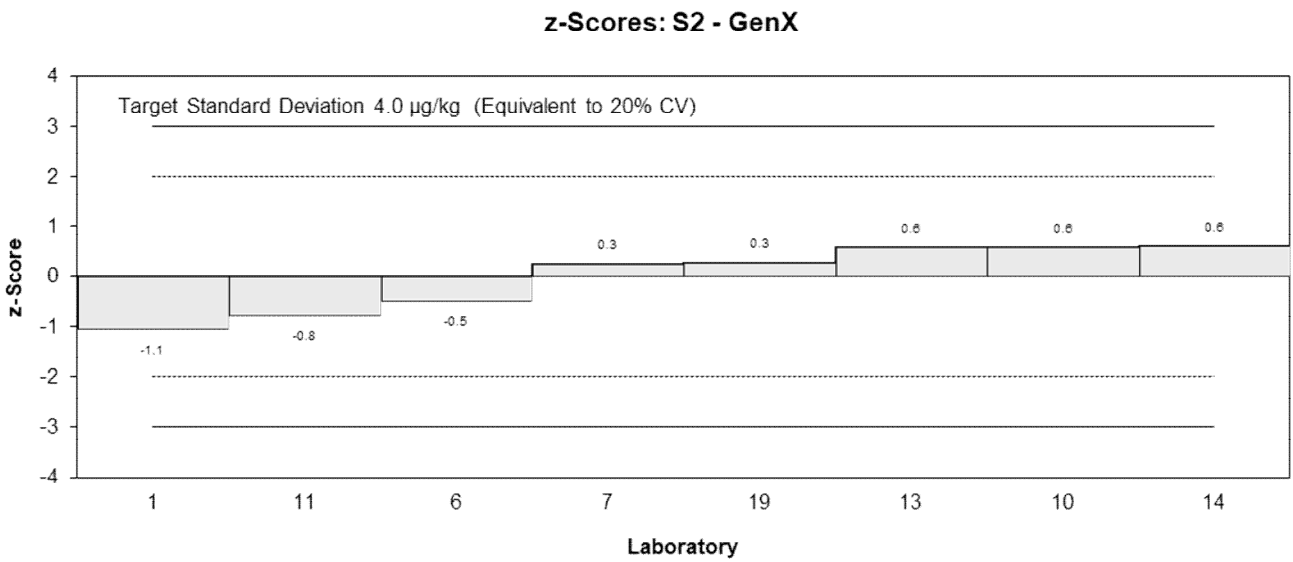
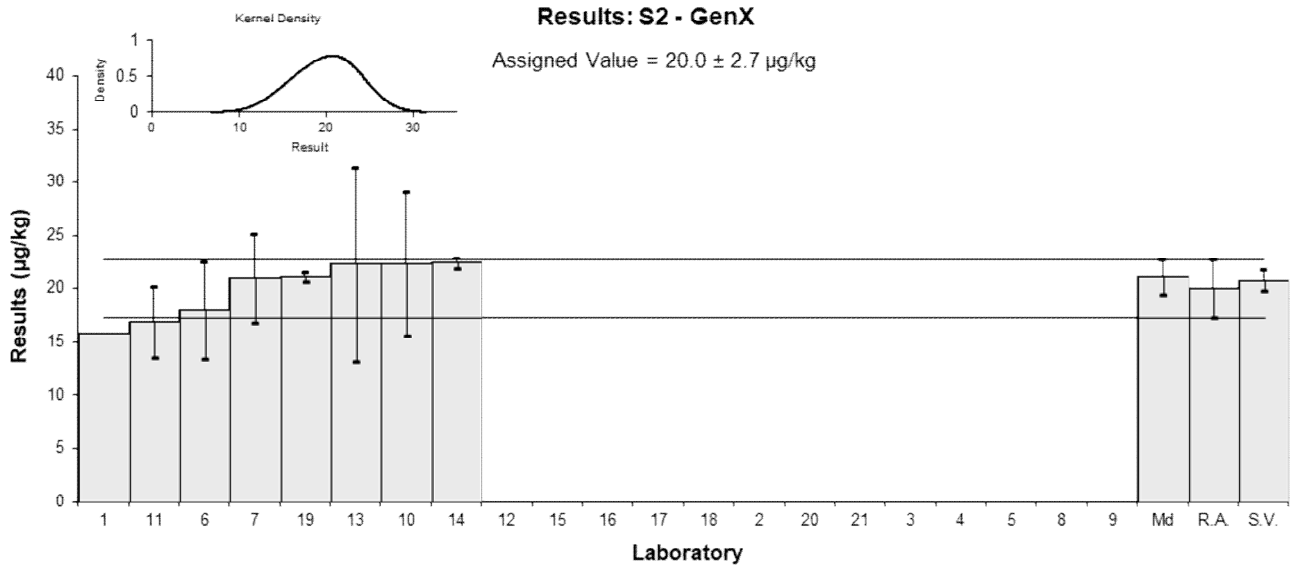


Figure 33

## 6 DISCUSSION OF RESULTS

### 6.1 Assigned Value

The robust average of participants' results was used as the assigned value for all analytes. The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528:2015.<sup>5</sup> Results less than 50% and greater than 150% of the robust average were removed before the calculation of the assigned value.<sup>3,4</sup> The calculation of the expanded uncertainty for the robust average is presented in Appendix 3, using PFHxS (linear) in Sample S2 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.

A comparison of the assigned and spiked values of analytes is presented in Table 37. For this study, the assigned value for analytes was within the range 71 – 103% of the spiked value, providing good support for the assigned values and the analyte stability.

Table 37 Comparison of Assigned and Spiked Values

Sample	Analyte	Assigned Value (µg/kg)	Spiked Value (µg/kg)	Assigned Value / Spiked Value (%)
S1 (Prawn)	PFBS	0.158	0.201	79
	PFHxS	5.35	6.53	82
	PFHxS (linear)	5.29	6.39	83
	PFHpS	4.38	5.54	79
	PFOS	8.7	12.3	71
	PFOS (linear)	6.1	8.54	71
	PFBA	0.85	0.983	86
	PFPeA	0.89	1.01	88
	PFHxA	1.1	1.19	92
	PFHpA	1.61	1.92	84
	PFOA	3.87	4.94	78
	PFNA	0.78	0.976	80
	PFDA	0.394	0.488	81
	PFUdA	0.321	0.399	80
	PFOSA	5.65	6.93	82
	ADONA	7.5	9.35	80
	GenX	9.6	12.4	77
S2 (Lettuce)	PFBS	2.64	3.05	87
	PFHxS	5.61	6.08	92
	PFHxS (linear)	5.73	5.86	98
	PFHpS	6.42	7.33	88
	PFOS	27.4	37.2	74
	PFOS (linear)	18.7	25.8	72
	PFDS	12.5	14.4	87

Sample	Analyte	Assigned Value (µg/kg)	Spiked Value (µg/kg)	Assigned Value / Spiked Value (%)
	PFBA	18	19.3	93
	PFPeA	26.5	30.1	88
	PFHxA	34.8	33.9	103
	PFOA	14.8	18.2	81
	PFNA	3.58	4.53	79
	PFDA	2.89	3.33	87
	ADONA	22.2	27.4	81
	GenX	20	20.8	96

## 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.<sup>7</sup>

Of 496 numerical results reported for analytes of interest in this study, 476 (96%) were reported with an associated expanded MU. Participants used a wide variety of procedures to estimate their uncertainty (Table 2).

Laboratory **1** stated they were accredited to ISO/IEC 17025, but reported uncertainties for some analytes only. Laboratory **9** did not report any uncertainties, stating that they were not accredited.

Laboratory **19** reported their Sample S1 results as a percentage rather than in µg/kg. These values were modified accordingly by the study coordinator for this report.

The magnitude of the reported MUs for analytes in this study was within the range 0.03% to 10529% of the reported value. In general, an expanded uncertainty of less than 10% relative is likely to be unrealistically small for the routine measurement of PFAS, while over 50% is likely too large. Of the 476 MUs, 96 were less than 10% relative and 26 were greater than 50% relative. Laboratory **16**'s uncertainties were all greater than 250% relative of their result – this participant may have reported uncertainties as relative instead of absolute values.

Laboratories having a satisfactory z-score but an unsatisfactory E<sub>n</sub>-score may have underestimated the expanded MU associated with their result.

Laboratories **10** and **21** attached estimates of expanded MU for some non-value results reported. An uncertainty expressed as a value should not be attached to a non-value result.<sup>8</sup>

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 21.919 ± 3.083 µg/kg, it is better to report this as 21.9 ± 3.1 µg/kg).<sup>8</sup>

## 6.3 z-Score

Target SDs equivalent to 20% PCV were used to calculate z-scores. CVs predicted by the Thompson-Horwitz Equation,<sup>6</sup> target SDs (as PCVs), and the between laboratories CVs obtained in this study for scored analytes are presented for comparison in Table 38.

Table 38 Comparison of Thompson-Horwitz CVs, Target SDs and Between Laboratories CVs

Sample	Analyte	Assigned Value (µg/kg)	Thompson-Horwitz CV (%)	Target SD (as PCV, %)	Between Laboratories CV* (%)
S1	PFBS	0.158	22	20	34
	PFHxS	5.35	22	20	17
	PFHxS (linear)	5.29	22	20	21
	PFHpS	4.38	22	20	18
	PFOS	8.7	22	20	19
	PFOS (linear)	6.1	22	20	18
	PFBA	0.85	22	20	25
	PFPeA	0.89	22	20	27
	PFHxA	1.1	22	20	23
	PFHpA	1.61	22	20	23
	PFOA	3.87	22	20	19
	PFNA	0.78	22	20	29
	PFDA	0.394	22	20	21
	PFUdA	0.321	22	20	32
	PFOSA	5.65	22	20	16
	ADONA	7.5	22	20	19
GenX	9.6	22	20	22	
S2	PFBS	2.64	22	20	21
	PFHxS	5.61	22	20	14
	PFHxS (linear)	5.73	22	20	11
	PFHpS	6.42	22	20	11
	PFOS	27.4	22	20	19
	PFOS (linear)	18.7	22	20	13
	PFDS	12.5	22	20	24
	PFBA	18	22	20	11
	PFPeA	26.5	22	20	14
	PFHxA	34.8	22	20	17
	PFOA	14.8	22	20	16
	PFNA	3.58	22	20	20
	PFDA	2.89	22	20	16
	ADONA	22.2	22	20	13
GenX	20	22	20	15	

\* Robust between laboratories CV with outliers removed, if applicable. Shaded cells are between laboratories CVs which were higher than the target SD established by the study coordinator and the coefficient of variation from predictive mathematical model (Thompson-Horwitz equation).

To account for possible low bias in the consensus value due to laboratories using inefficient analytical or extraction techniques, a total of 7 z-scores were adjusted for the following analytes: S1 PFBS, PFHpS, PFOS and PFOS (linear), and S2 PFOS. 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 also not adjusted.

Of 496 results for which z-scores were calculated, 404 (81%) returned  $|z| \leq 2.0$ , indicating a satisfactory performance.

Laboratories **7**, **11**, **13** and **14** reported results for all 32 scored analytes.

Sixteen participants analysed both matrices. Laboratories **7** and **14** returned satisfactory z-scores for all 32 scored analytes. Laboratories **4** (28), **20** (25), **5** (22), **8** (22), and **17** (21) returned satisfactory z-scores for all reported results.

Five participants analysed only one matrix. Of these participants, Laboratory **16** returned satisfactory z-scores for all scored analytes in the biota matrix (17).

Laboratories **2** (29) and **12** (3) returned unsatisfactory z-scores for all reported results, with all results significantly higher than the assigned value (except for ADONA for Laboratory **2**, where results were significantly lower instead). These participants should check for laboratory or methodology bias.

The dispersal of participants' z-scores is presented graphically by laboratory in Figure 34 and by analyte in Figure 35.

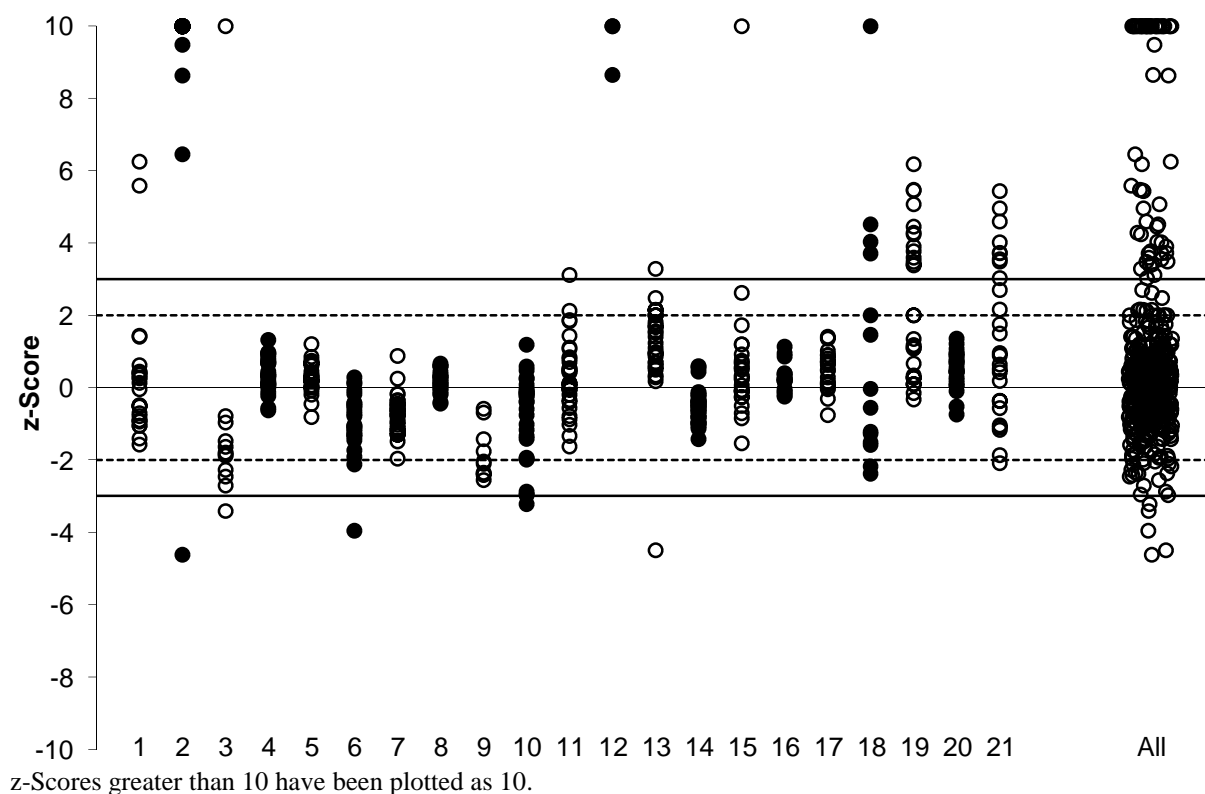
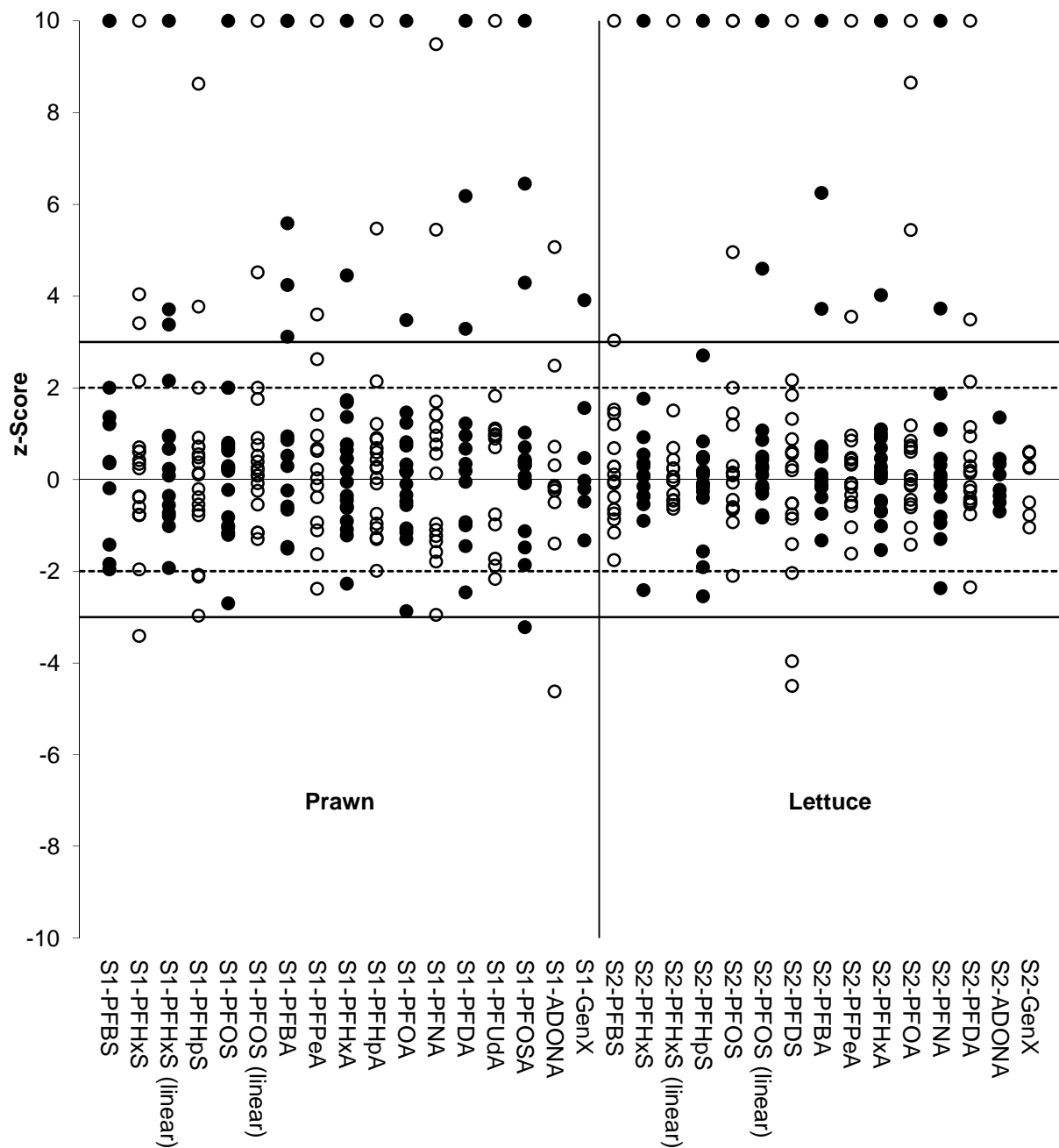


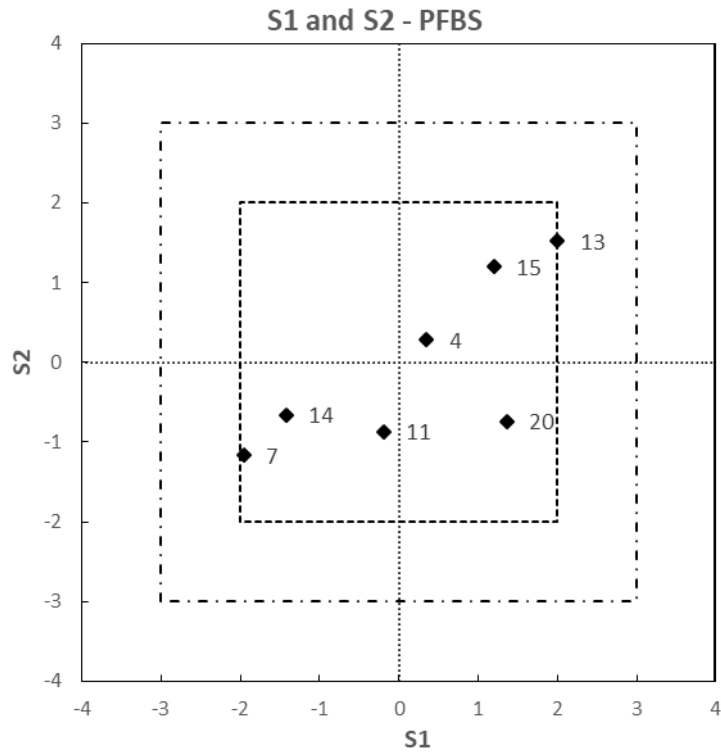
Figure 34 z-Score Dispersal by Laboratory



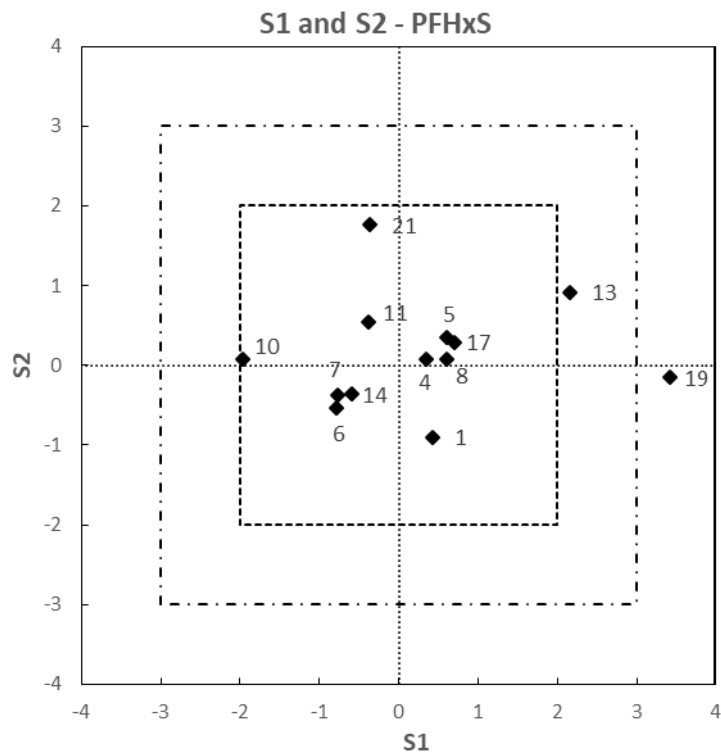
z-Scores greater than 10 have been plotted as 10.

Figure 35 z-Score Dispersal by Analyte

Scatter plots of z-scores for analytes present in both Samples S1 (Prawn) and S2 (Lettuce) are presented in Figures 36 to 49. 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 2 is off-scale.  
**Figure 36 z-Score Scatter Plot – PFBS**



Laboratory 2 is off-scale.  
**Figure 37 z-Score Scatter Plot – PFHxS**

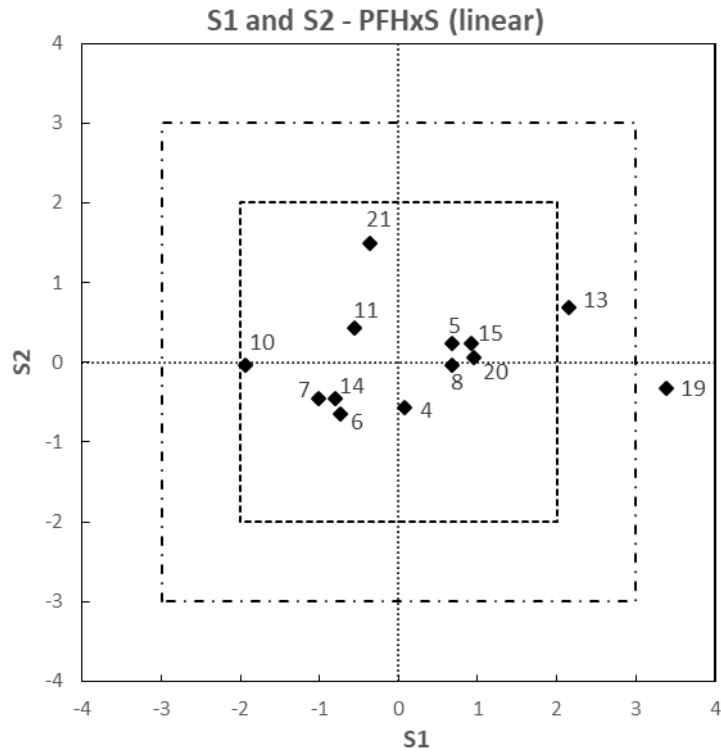


Figure 38 z-Score Scatter Plot – PFHxS (linear)

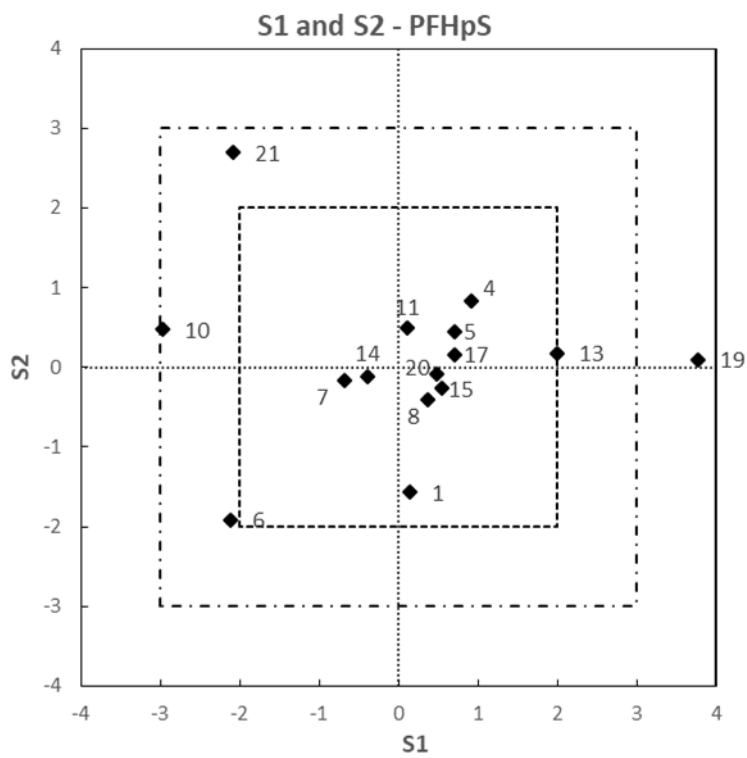
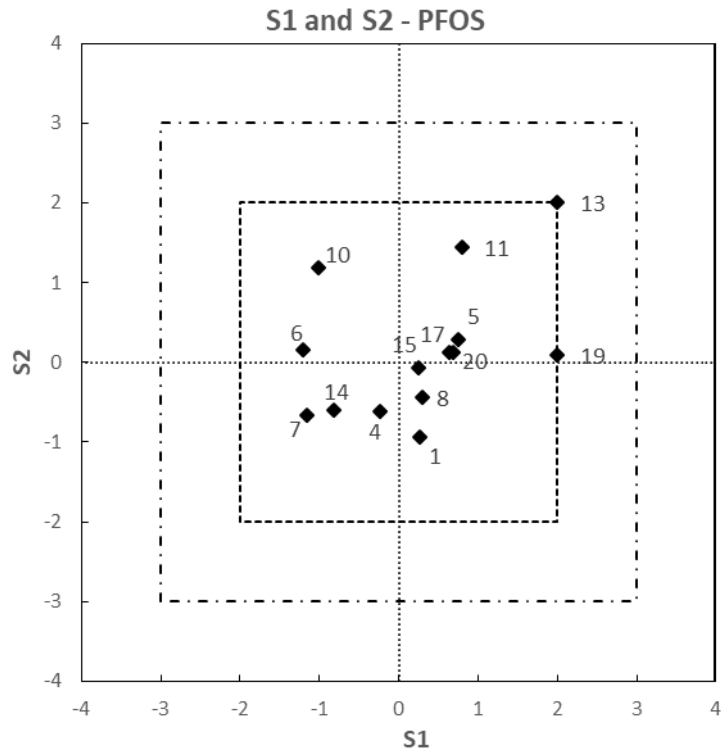


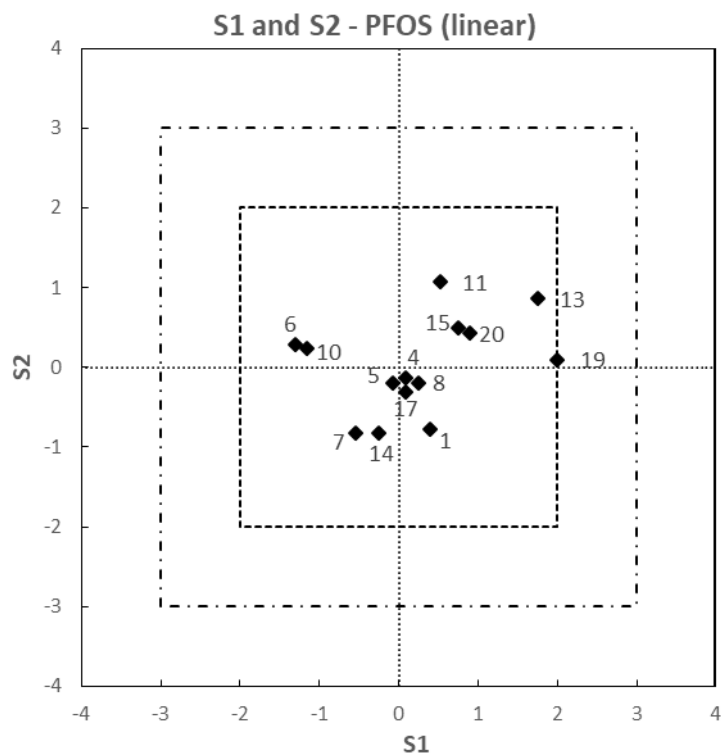
Figure 39 z-Score Scatter Plot – PFHpS





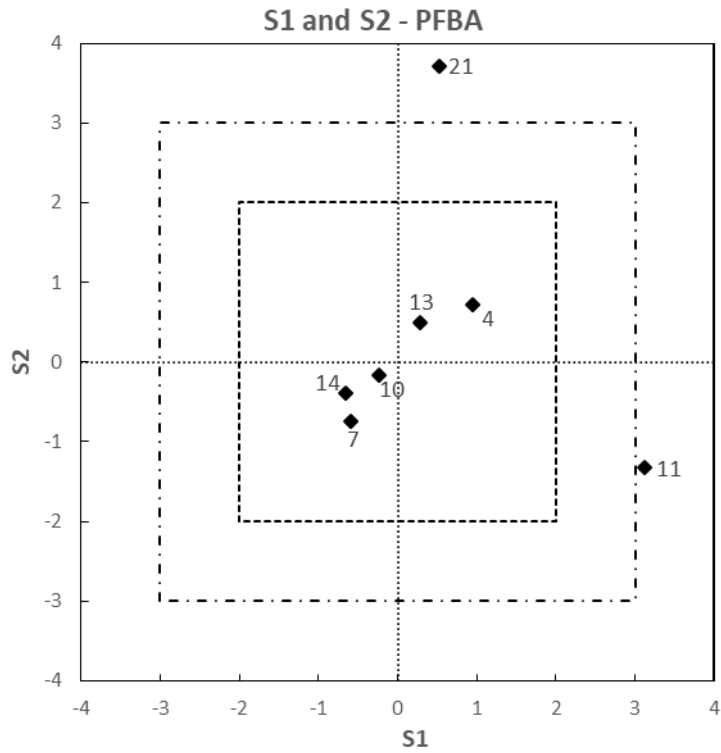
Laboratories 2 and 21 are off-scale.

Figure 40 z-Score Scatter Plot – PFOS



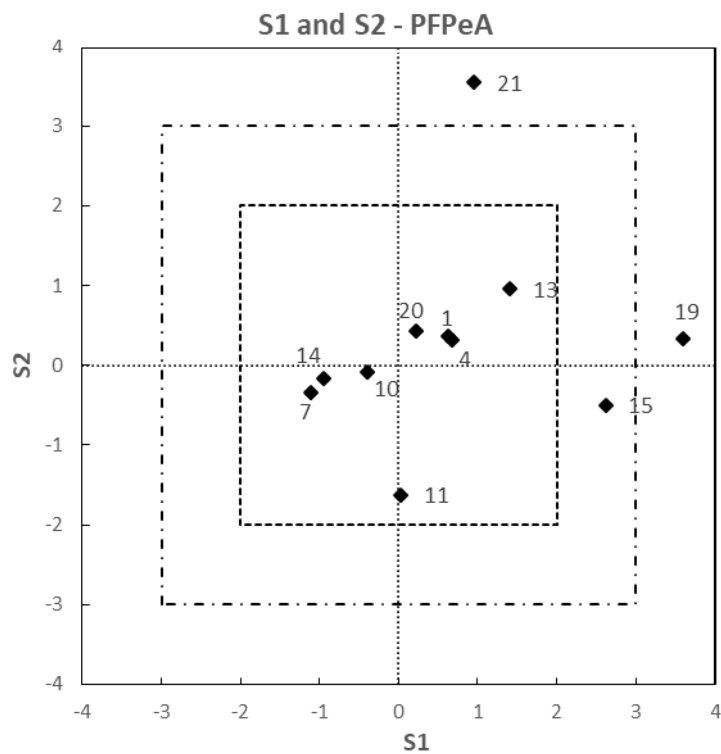
Laboratories 2 and 21 are off-scale.

Figure 41 z-Score Scatter Plot – PFOS (linear)



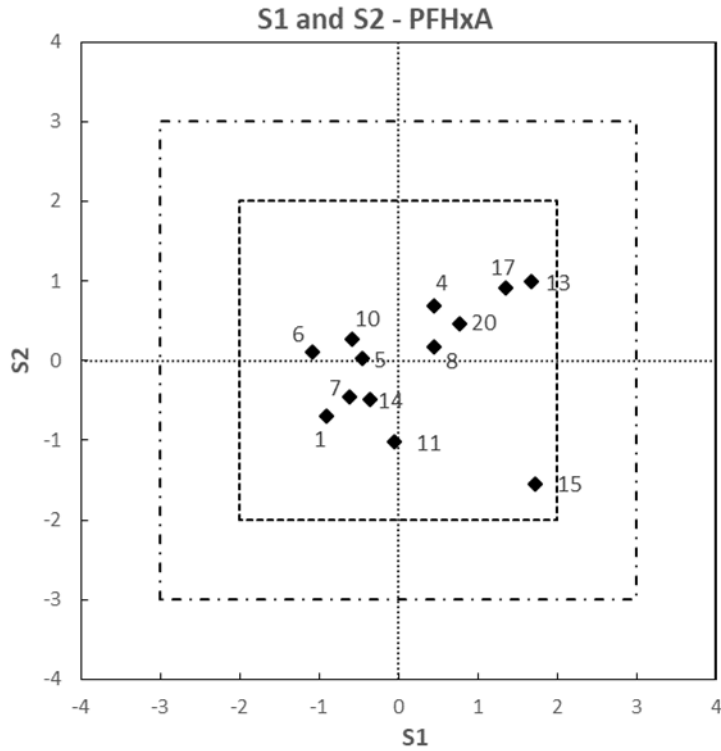
Laboratories 1, 2, 15 and 19 are off-scale.

Figure 42 z-Score Scatter Plot – PFBA

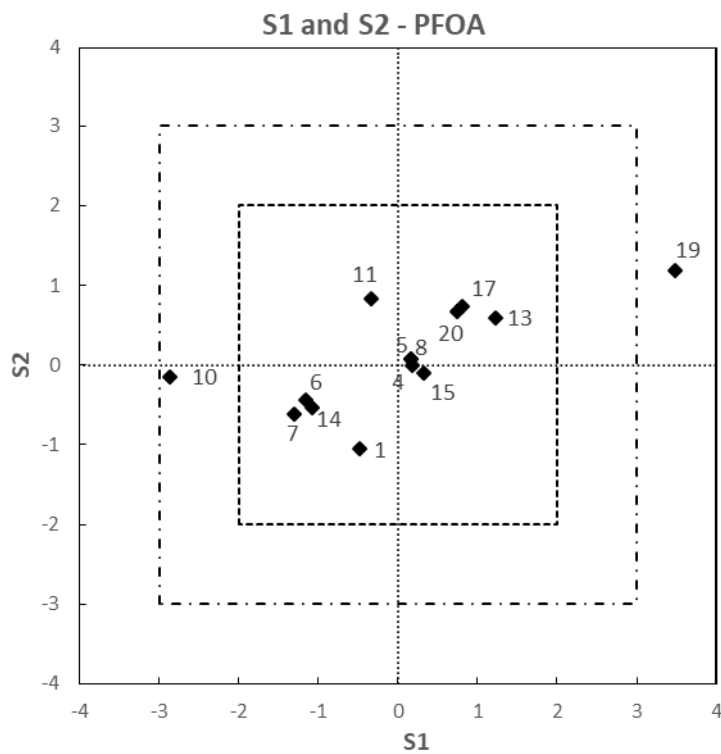


Laboratory 2 is off-scale.

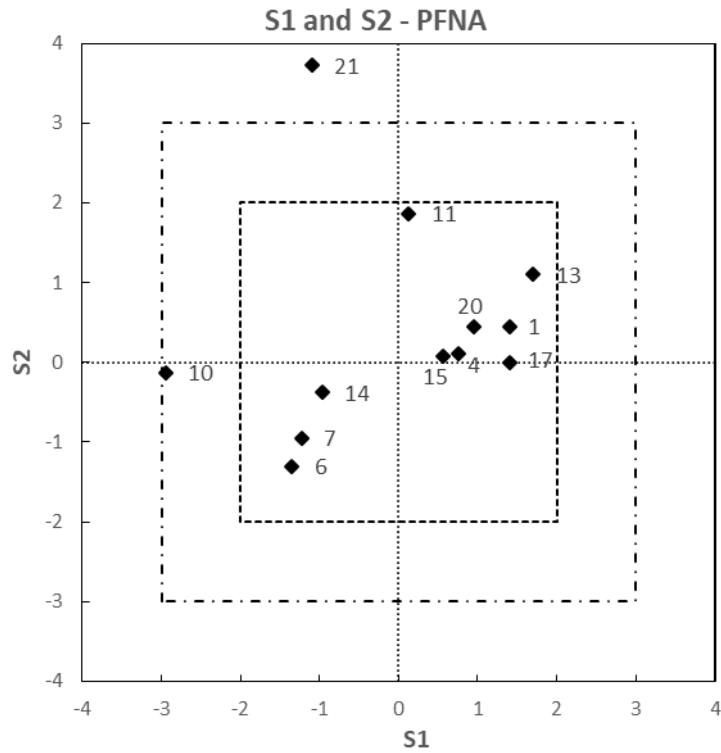
Figure 43 z-Score Scatter Plot – PFPeA



Laboratories 2, 19 and 21 are off-scale.  
 Figure 44 z-Score Scatter Plot – PFHxA

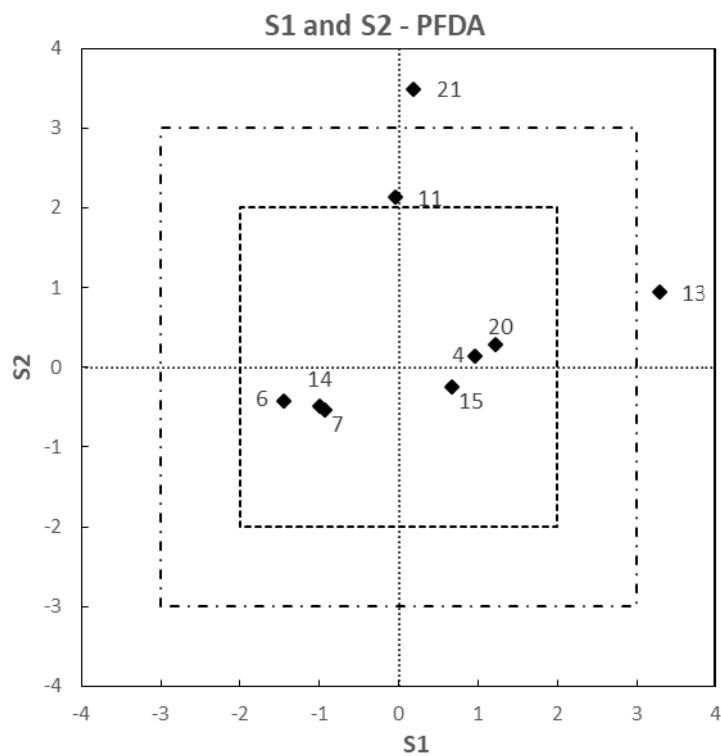


Laboratories 2 and 21 are off-scale.  
 Figure 45 z-Score Scatter Plot – PFOA



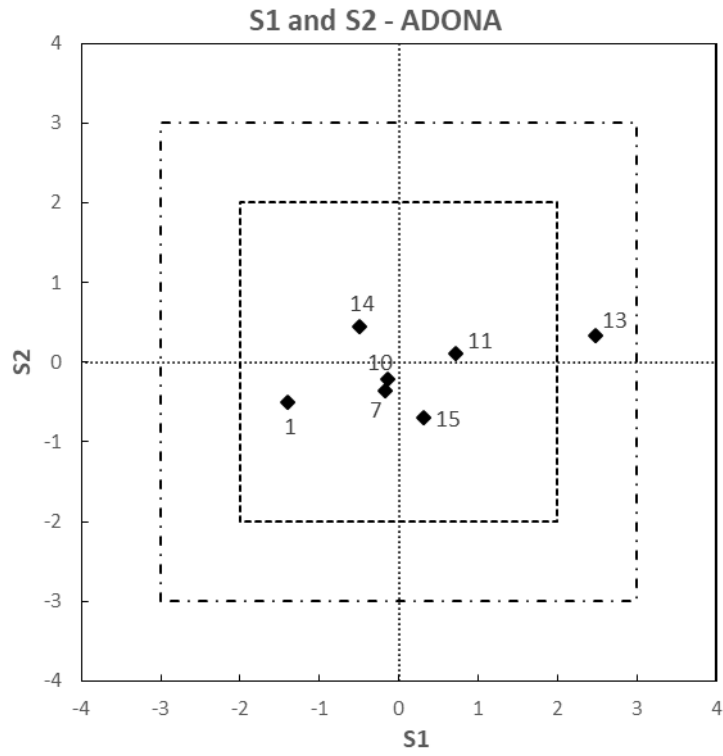
Laboratories 2 and 19 are off-scale.

Figure 46 z-Score Scatter Plot – PFNA



Laboratories 2 and 19 are off-scale.

Figure 47 z-Score Scatter Plot – PFDA



Laboratory 19 is off-scale.

Figure 48 z-Score Scatter Plot – ADONA

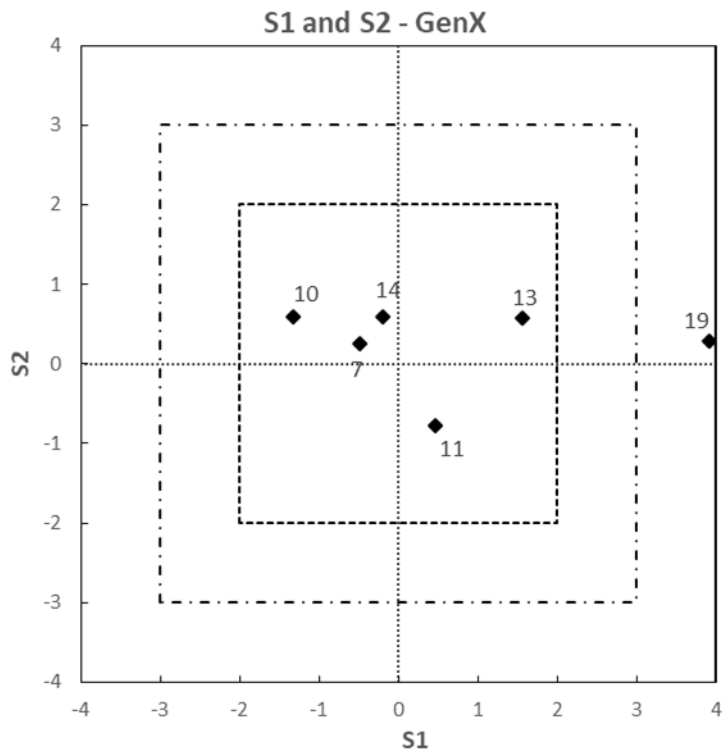


Figure 49 z-Score Scatter Plot – GenX

## 6.4 E<sub>n</sub>-Score

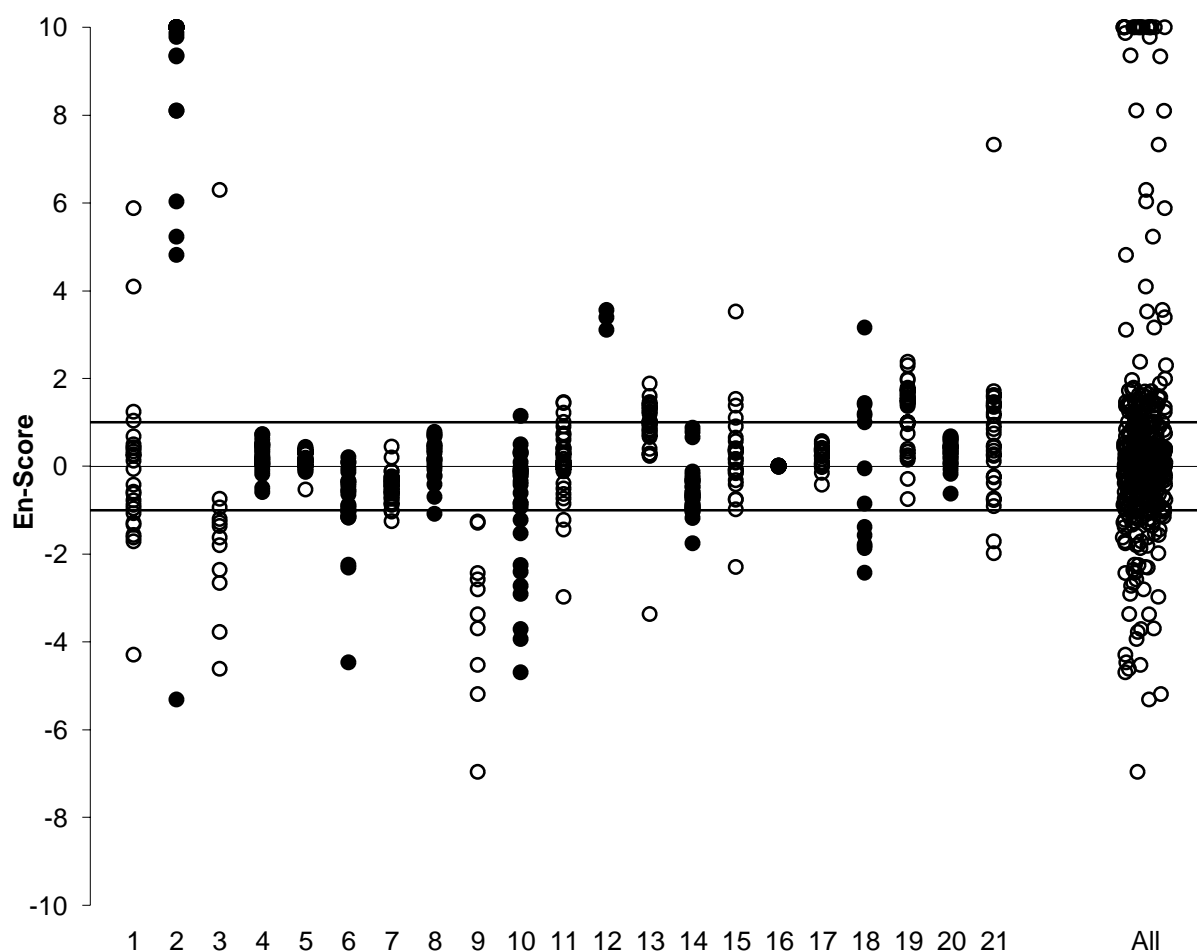
Where a laboratory did not report an uncertainty with a result, an uncertainty of zero (0) was used to calculate the E<sub>n</sub>-score. E<sub>n</sub>-scores greater than 1.0 were set to 1.0 for results with z-scores that were adjusted as discussed in Section 6.3 z-Scores.

Of 496 results for which E<sub>n</sub>-scores were calculated, 339 (68%) 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<sub>n</sub>-scores for all analytes of interest in this study. Of the participants analysing both matrices, Laboratories **4** (28), **20** (25), **5** (22) and **17** (21) returned satisfactory E<sub>n</sub>-scores for all reported results. Of the participants analysing only one matrix, Laboratory **16** (17) returned satisfactory E<sub>n</sub>-scores for all biota matrix results.

Laboratories **2** (29), **9** (10) and **12** (3) returned unsatisfactory E<sub>n</sub>-scores for all reported results.

The dispersal of participants' E<sub>n</sub>-scores is presented graphically in Figure 50.



E<sub>n</sub>-Scores greater than 10 have been plotted as 10.

Figure 50 E<sub>n</sub>-Score Dispersal by Laboratory

## 6.5 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. Results reported in this way reflect the true variability of results reported to clients. The method descriptions provided by participants are presented in Tables 57 to 78. The study coordinator thanks all laboratories

that completed the method questionnaire. A summary is presented below as technique (number of participants):

- Pre-treatment: homogenisation (15 for both, 1 for S2 only), freeze-drying (1), base digestion (1 for S1 only), addition of drying agent (1 for S2 only), no pre-treatment (3, 1 for S1 only).
- Extraction technique: SLE (8), SPE (6), alkaline digestion (5), sonication (3), LLE (1).
- Extraction process: shaking (11), sonication (10), tumbling (2), vortex (2), SPE (1), QuEChERS (1).
- Extraction solvent: acetonitrile (7, 1 for S1 only), methanol/base, e.g. KOH, NaOH (7), methanol (3), acetonitrile/acid(/water) (2), acetonitrile/base (1), acetonitrile/methanol (1), NaOH (1), MTBE (1).
- Extraction temperature: room temperature (18, 1 for S1 only), cooled then room (1 for S2 only), heated (1).
- Extraction time: 30 minutes and under (9, 2 for S1 only), >30 minutes to 1 hour (4, 1 for S2 only), >1 hour to 10 hours (1), >10hours (4, 1 for S2 only).
- Extraction clean-up: carbon (8, 1 for S1 only), other SPE (12, 1 for S2 only), centrifugation (2), filtration (2), LLE (1, 1 for S1 only), no clean-up (2).
- Instrument: LC-MS/MS or LC-QQQ (18), Orbitrap (3).
- Dilution: Yes (2), No/Not Reported (19)
- Guard column: Yes (11), No (10)
- Delay column: Yes (17), No (4)
- Blank Correction: Yes (3), No (18)
- Labelled Standard: Wellington Laboratories (20), Cambridge Isotope Laboratories (1)
- Recovery correction: Yes (18), No (3).

Laboratory 2 reported significantly higher results for all analytes (approximately by a factor of 3.6 for Sample S1 and 36 for Sample S2), except ADONA which had significantly lower results. This was the only participant to use freeze-drying as a pre-treatment, and they commented that their results were based on the dry sample. For this study, participants were instructed to report their results on as received basis, so all results from Laboratory 2 were excluded from statistical calculations. Laboratory 12's results were all significantly higher than the assigned value (on average, by a factor of approximately 3); this was the only participant that reported using MTBE as the extraction solvent.

Laboratory 19's Sample S1 results and Laboratory 21's Sample S2 results were also all biased high compared to the assigned value (approximately by a factor of 1.8 and 1.7 respectively), though some of their results did return satisfactory z-scores.

These participants may need to review their sample or standard preparation procedure, or consider if there was a calculation error causing all their results to be increased by a similar factor.

A comparison of z-scores vs sample mass used for analysis is presented in Figure 51 (all scores greater than 10 have been plotted as 10, and results from the laboratories discussed above have been excluded). For Sample S1 Prawn, 4 participants reported using a sample mass of 0.2, 0.3 or 0.5 g, and the results reported by these participants were either biased high

or low. Caution should be exercised when a small sample size (e.g. < 1 g) is taken for analysis as this may not be a suitable representation of the whole sample.

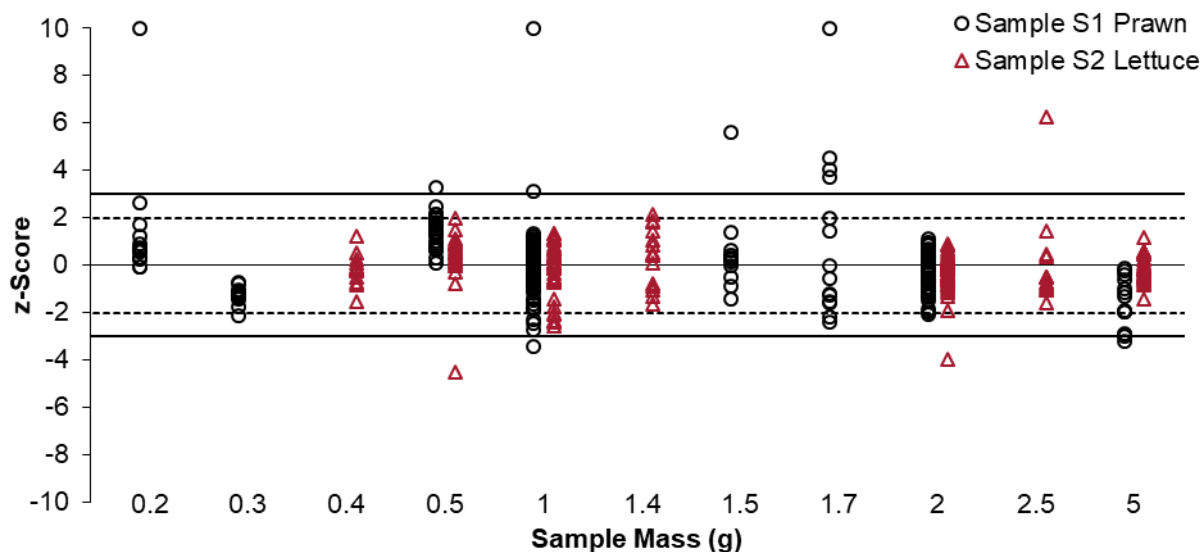


Figure 51 z-Score vs Sample Mass Used for Analysis

Graphs of z-scores compared to a number of extraction and analysis parameters are provided in Figures 52 to 56 (all scores greater than 10 have been plotted as 10, and results from the laboratories discussed previously have been excluded). For the majority of these methodology parameters, no significant trend was identified for when more than one participant used a particular technique. It is noted that one participant reported an extraction time of one minute, and all results reported by this participant except one was biased low. The shorter extraction time employed by this participant may have contributed to a less efficient extraction of analytes.



Figure 52 z-Score vs Pre-Treatment



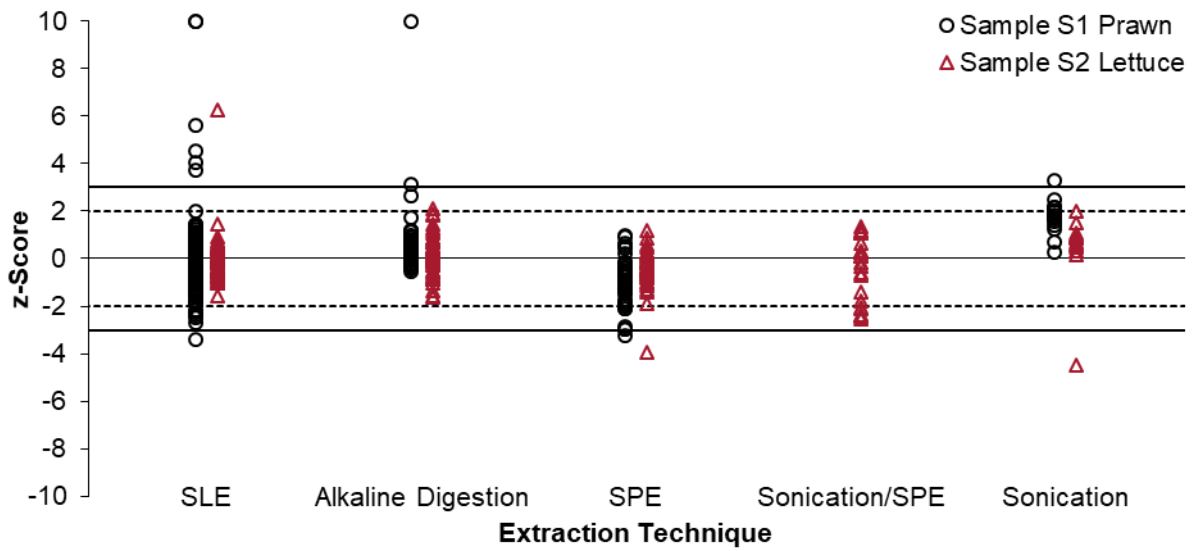


Figure 53 z-Score vs Extraction Technique

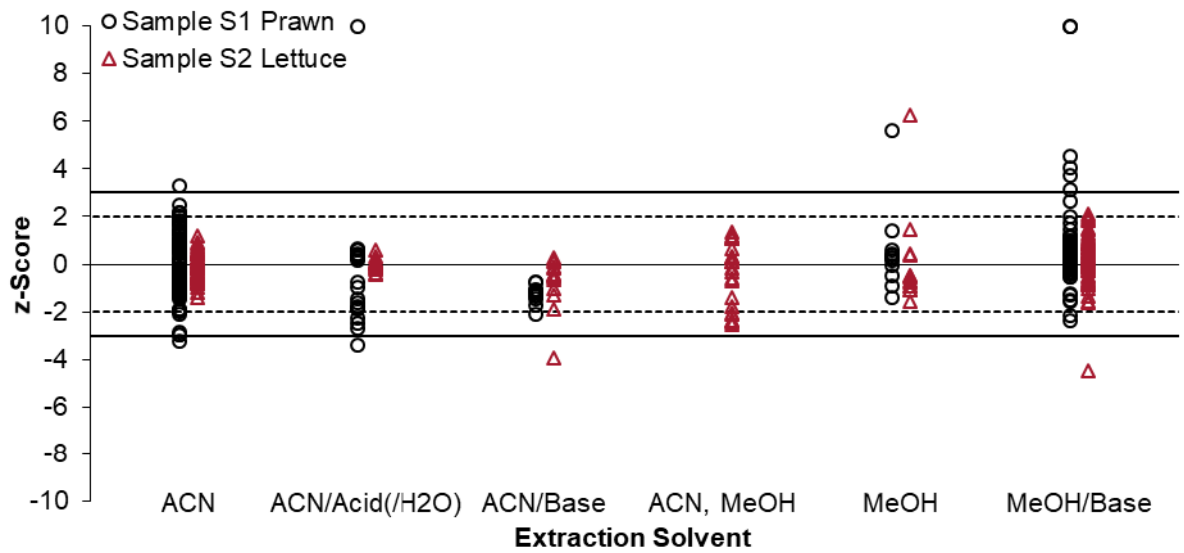


Figure 54 z-Score vs Extraction Solvent

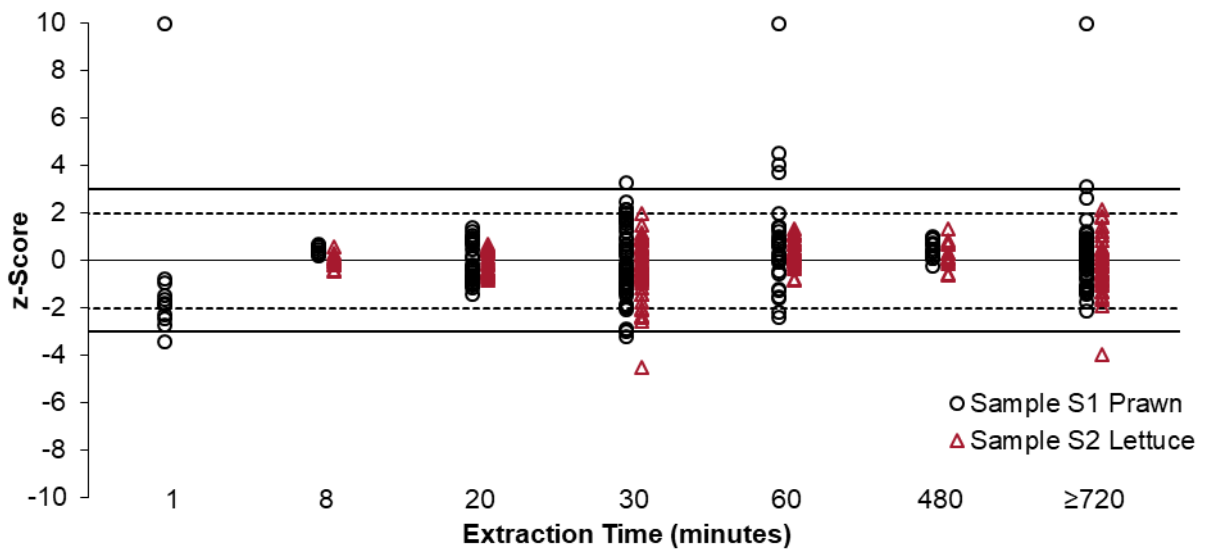


Figure 55 z-Score vs Extraction Time

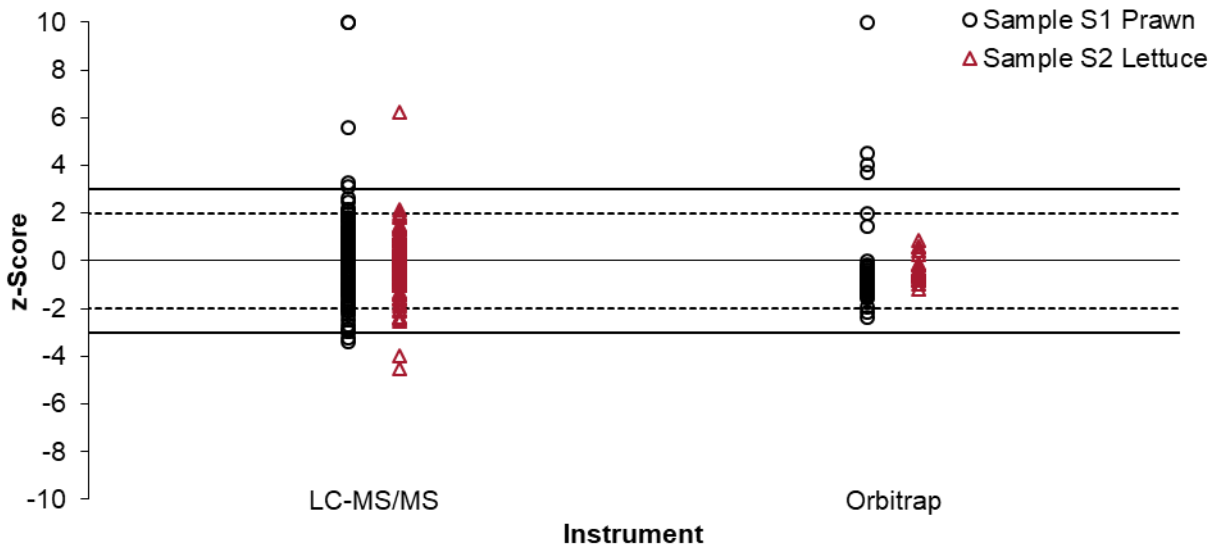


Figure 56 z-Score vs Measurement Instrument

### 6.6 Linear and Branched Isomers – PFHxS and PFOS

In this study, 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 39.

Table 39 Number of Participants Reporting PFHxS and PFOS Results

Sample	PFHxS			PFOS		
	Linear and Total	Linear Only	Total Only	Linear and Total	Linear Only	Total Only
S1	14	2	3	18	0	1
S2	12	2	3	17	0	1

#### PFHxS

No substantial difference was expected between the PFHxS total results and PFHxS linear only results as the contribution from branched isomers to total PFHxS was minimal (estimated spiked ratio of linear to total PFHxS was 98% and 96% for Sample S1 and S2 respectively). Overall, participants reported similar ratios of linear to total PFHxS, being on average 98% for both Samples S1 and S2.

For Sample S1, Laboratories **2, 5, 6, 8, 10, 13, 16, 19** and **21** reported linear to total PFHxS ratios of 99 – 100%, while Laboratories **4, 7, 11, 14** and **18** reported slightly lower ratios of 93 – 95%.

For Sample S2, Laboratories **2, 5, 6, 7, 8, 10, 11, 13, 14, 19** and **21** reported linear to total PFHxS ratios of 97 – 100%, while Laboratory **4** reported a lower ratio of 89%.

A summary of participants’ proportions of linear to total PFHxS is presented in Figure 57.

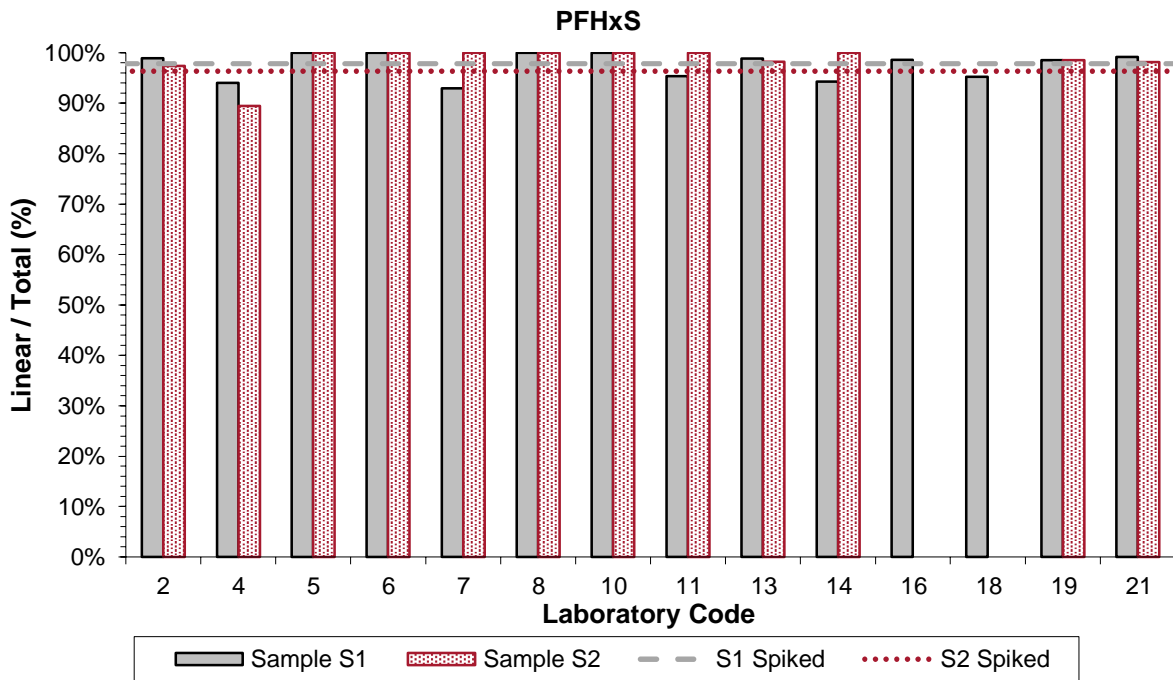


Figure 57 Participants' Reported Proportions of PFHxS Linear Isomers

**PFOS**

The spiked ratio of linear isomers to total PFOS was approximately 69% for both samples. Taken as a group, participants reported more varying PFOS isomer ratios, with the proportion of linear isomers ranging from 63 – 81% for Sample S1 (average of 71%) and 55 – 76% for Sample S2 (average of 66%). Of participants reporting PFOS results in both matrices, Laboratories **2, 7, 10, 13** and **14** reported results where the difference of the ratios of linear to total PFOS was greater than 5% for these two samples which were spiked with similar PFOS isomer proportions.

A summary of participants' proportions of linear to total PFOS is presented in Figure 58.

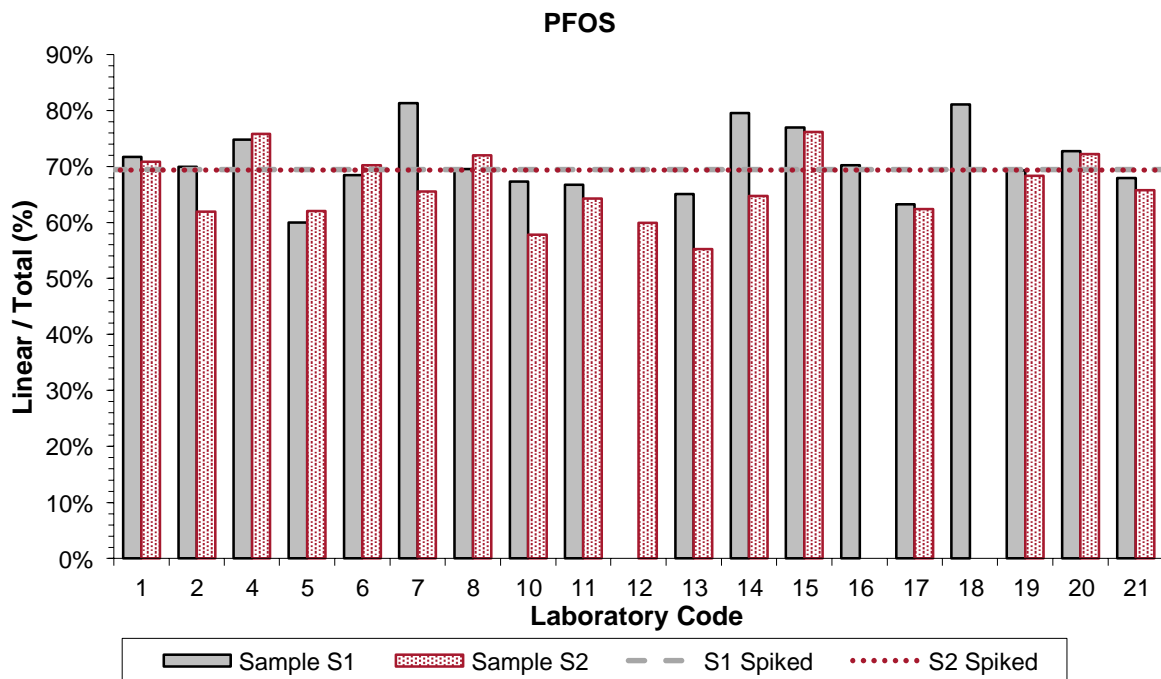


Figure 58 Participants' Reported Proportions of PFOS Linear Isomers

## 6.7 Effects of Sample Matrix

The samples in this study were prawn (Sample S1) and lettuce (Sample S2). A summary of the results reported and z-scores obtained by matrix is presented in Table 40.

Participants overall performed better with the food (lettuce) matrix, with a higher proportion of results reported and a higher proportion of satisfactory z-scores.

Table 40 Result Comparison by Matrix

Sample	Matrix	Expected Number of Results	Results Reported	z-Scores Calculated	Satisfactory z-Scores
S1	Prawn (spiked)	323	263 (81%)	263	208 (79%)
S2	Lettuce (spiked)	270	233 (86%)	233	196 (84%)

## 6.8 False Negatives

Table 41 presents false negative results – an analyte present in the sample for which a participant tested for but did not report a result (e.g. laboratories reporting a ‘<’ or NR result when the assigned and spiked value was higher than the participants’ reporting limit, or participants that did not report any value).

Table 41 False Negatives

Lab. Code	Sample	Analyte	Assigned Value (µg/kg)	Spiked Value (µg/kg)	Reported Result (µg/kg)
1	S1	GenX	9.6	12.4	<0.5
2	S2	ADONA	22.2	27.4	<0.25
6	S1	PFBS	0.158	0.201	NR*
		PFBA	0.85	0.983	NR*
		PFPeA	0.89	1.01	NR*
		GenX	9.6	12.4	NR*
9	S2	PFBA	18.0	19.3	NR
10	S1	PFDA	0.394	0.488	<0.2
		PFUdA	0.321	0.399	<0.2
18	S1	PFDA	0.394	0.488	NR**
19	S1	PFBS	0.158	0.201	NR
		PFUdA	0.321	0.399	NR
20	S1	PFHxS	5.35	6.53	NR
		PFBA	0.85	0.983	<0.366
	S2	PFHxS	5.61	6.08	NR

\* Laboratory 6 reported these results as “<LOD”. Depending on actual LOD, this result may or may not be a false negative.

\*\* Laboratory 18 reported this result as “below limit of quantification”. Depending on actual LOQ, this result may or may not be a false negative.

## 6.9 Reporting of Additional Analytes

Fourteen 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 42.

Table 42 Non-Spiked Analytes Reported by Participants

Lab. Code	Sample	Analyte	Result (µg/kg)	Uncertainty (µg/kg)	Recovery (%)
1	S2	4:2 FTS	15.1	NR	75.9
2	S1	PFPeS	0.05	0.02	NR
		PFNS	0.05	0.01	NR
		PFDS	0.05	0.01	NR
		N-MeFOSAA	0.03	0.01	NR
	S2	PFHpA	7.80	0.60	102
3	S1	N-MeFOSE	0.11	0.02	NR
4	S2	PFHpA	0.232	0.06	75
6	S2	PFHpA	0.15	0.05	71
7	S2	PFHpA	0.181	0.0905	72
11	S2	PFHpA	0.222	0.04	95
13	S2	PFHpA	0.265	0.008	73.9
14	S2	PFHpA	0.211	0.0051	97
15	S1	PFPeS	0.027	0.022	NR
		PFTTrDA	0.017	0.036	NR
		PFTeDA	0.051	0.042	NR
	S2	PFPeS	0.024	0.043	NR
		PFHpA	0.132	0.084	NR
		PFUdA	0.011	0.066	NR
		6:2 FTS	0.438	0.555	NR
17	S2	4:2 FTS	3.0	1.1	348
19	S1	N-EtFOSA	0.822	0.222	51
	S2	PFHpA	0.278	0.3	86
20	S1	PFNS	0.041	0.018	76.4
		PFDS	0.04	0.026	76.4
		PFTTrDA	0.046	0.019	46.5
	S2	PFNS	0.042	0.018	74
		PFHpA	0.222	0.062	72.7
21	S2	PFHpA	0.315	0.09	63.1
		4:2 FTS	1.93	0.58	73.9
		6:2 FTS	0.723	0.22	185

### 6.10 Summary of Participants' Results and Performances

Summaries of participants' results and performances for scored analytes in this PT study are presented in Tables 43 and 44, and Figure 59.

Table 43 Summary of Participants' Sample S1 Results (all values are in µg/kg)\*

Lab. Code	PFBS	PFHxS	PFHxS (linear)	PFHpS	PFOS	PFOS (linear)	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUdA	PFOSA	ADONA	GenX
A.V.	0.158	5.35	5.29	4.38	8.7	6.10	0.85	0.89	1.10	1.61	3.87	0.78	0.394	0.321	5.65	7.5	9.6
1	<0.5	5.8	NT	4.5	9.16	6.57	1.8	1	0.9	1.7	3.5	1	<0.5	<0.5	5.6	5.4	<0.5
2	0.55	19.24	19.03	11.94	33.1	23.16	6.67	2.93	3.75	5.93	11.81	2.26	1.19	1.17	12.94	0.57	NT
3	0.1	1.7	NT	3.7	4	NT	0.6	0.6	0.6	1.3	NT	0.5	0.2	0.2	58	NT	NT
4	0.169	5.71	5.37	5.18	8.3	6.21	1.01	1.01	1.2	1.83	4.01	0.899	0.47	0.384	6.08	NT	NT
5	<1	6	6	5	10	6	<1	<1	1	2	4	<1	<2	<2	6	NT	NT
6	NR	4.52	4.52	2.52	6.6	4.52	NR	NR	0.86	1.27	2.98	0.57	0.28	0.21	NT	NT	NR
7	0.0960	4.53	4.21	3.78	6.68	5.43	0.749	0.693	0.963	1.19	2.86	0.588	0.321	0.272	3.98	7.23	8.68
8	< 1.0	6.0	6.0	4.7	9.2	6.4	< 1.0	< 1.0	1.2	1.8	4.0	< 1.0	< 1.0	< 1.0	6.0	NT	NT
9	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
10	<0.2	3.25	3.25	1.78	6.95	4.68	0.81	0.82	0.97	0.97	1.65	0.32	<0.2	<0.2	2.01	7.29	7.05
11	0.152	4.93	4.7	4.48	10.1	6.74	1.38	0.895	1.09	1.62	3.61	0.801	0.390	0.392	6.14	8.56	10.5
12	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
13	0.227	7.647	7.561	6.245	12.641	8.231	0.900	1.141	1.469	2.299	4.825	1.045	0.653	0.438	6.440	11.226	12.595
14	0.113	4.71	4.44	4.04	7.28	5.79	0.737	0.722	1.02	1.37	3.04	0.629	0.315	0.258	4.37	6.75	9.21
15	0.196	NT	6.26	4.865	9.117	7.019	5.583	1.356	1.481	1.582	4.122	0.868	0.447	0.366	5.561	7.962	NT
16	0.17	5.61	5.53	4.2	9.03	6.34	0.996	0.867	1.14	1.69	3.79	0.958	0.421	0.382	6.1	7.14	9.54
17	<1	6.1	NT	5.0	9.8	6.2	<5	<2	1.4	1.9	4.5	1.0	<1	<1	6.8	NT	NT
18	0.529	9.67	9.21	3.90	14.32	11.61	0.594	0.467	0.831	1.20	5.00	0.534	NR	0.182	5.62	NT	NT
19	NR	9.00	8.87	7.68	14.6	10.12	1.57	1.53	2.08	3.37	6.56	1.63	0.881	NR	10.5	15.1	17.1
20	0.201	NR	6.3	4.8	9.9	7.2	<0.366	0.93	1.27	1.89	4.44	0.93	0.49	0.39	5.73	NT	NT
21	< 0.300	4.95	4.91	2.56	6.89	4.68	0.938	1.06	1.24	1.75	3.44	0.608	0.409	0.378	3.55	NT	NT

\* A.V. = Assigned Value. Shaded cells are results which returned a questionable or unsatisfactory z-score.

Table 44 Summary of Participants' Sample S2 Results (all values are in µg/kg)\*

Lab. Code	PFBS	PFHxS	PFHxS (linear)	PFHpS	PFOS	PFOS (linear)	PFDS	PFBA	PFPeA	PFHxA	PFOA	PFNA	PFDA	ADONA	GenX
A.V.	2.64	5.61	5.73	6.42	27.4	18.7	12.5	18.0	26.5	34.8	14.8	3.58	2.89	22.2	20.0
1	3.4	4.6	NT	4.4	22.3	15.8	11.2	40.5	28.4	29.9	11.7	3.9	2.6	20	15.8
2	115.08	234.17	228	210.06	1071.85	664.05	470.63	682.48	1052.74	1095.27	452.75	104.96	85.64	<0.25	NT
3	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
4	2.79	5.69	5.09	7.48	24	18.2	15.8	20.6	28.2	39.6	14.8	3.66	2.97	NT	NT
5	3	6	6	7	29	18	13	18	31	35	15	3	3	NT	NT
6	2.6	5	5	3.97	28.2	19.8	2.6	17.4	21	35.6	13.5	2.65	2.65	NT	18
7	2.03	5.2	5.2	6.21	23.8	15.6	14.7	15.3	24.7	31.6	13	2.9	2.58	20.6	21
8	2.7	5.7	5.7	5.9	25	18	14	18	26	36	15	3.8	2.8	NT	NT
9	1.71	2.91	NT	3.147	15.9	NT	7.4	NR	23.4	30	10.6	1.88	1.53	NT	NT
10	2.44	5.70	5.70	7.04	33.9	19.6	8.98	17.4	26.1	36.7	14.40	3.49	2.45	21.24	22.36
11	2.18	6.22	6.22	7.05	35.3	22.7	17.1	13.2	17.9	27.7	17.3	4.92	4.12	22.7	16.9
12	NT	NT	NT	NT	98.6	59.1	NT	NT	NT	NT	40.4	NT	NT	NT	NT
13	3.444	6.638	6.521	6.648	39.688	21.919	1.256	19.786	31.597	41.710	16.565	4.365	3.434	23.706	22.297
14	2.29	5.21	5.21	6.26	24.1	15.6	13.9	16.6	25.6	31.4	13.2	3.31	2.61	24.2	22.4
15	3.274	NT	6.003	6.081	27.003	20.568	10.382	17.615	23.864	24.083	14.504	3.63	2.743	19.071	NT
16	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
17	2.62	5.94	NT	6.62	28.1	17.54	10.6	18.4	29.0	41.1	17.0	3.58	3.18	NT	NT
18	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
19	2.79	5.44	5.36	6.55	27.9	19.07	13.2	20.4	28.3	42.4	18.3	4.35	3.55	28.2	21.1
20	2.25	NR	5.8	6.3	28.1	20.3	11.2	20	28.8	38	16.8	3.9	3.06	NT	NT
21	4.24	7.59	7.45	9.89	54.6	35.9	17.9	31.4	45.3	62.8	30.9	6.25	4.91	NT	NT

\* A.V. = Assigned Value. Shaded cells are results which returned a questionable or unsatisfactory z-score.

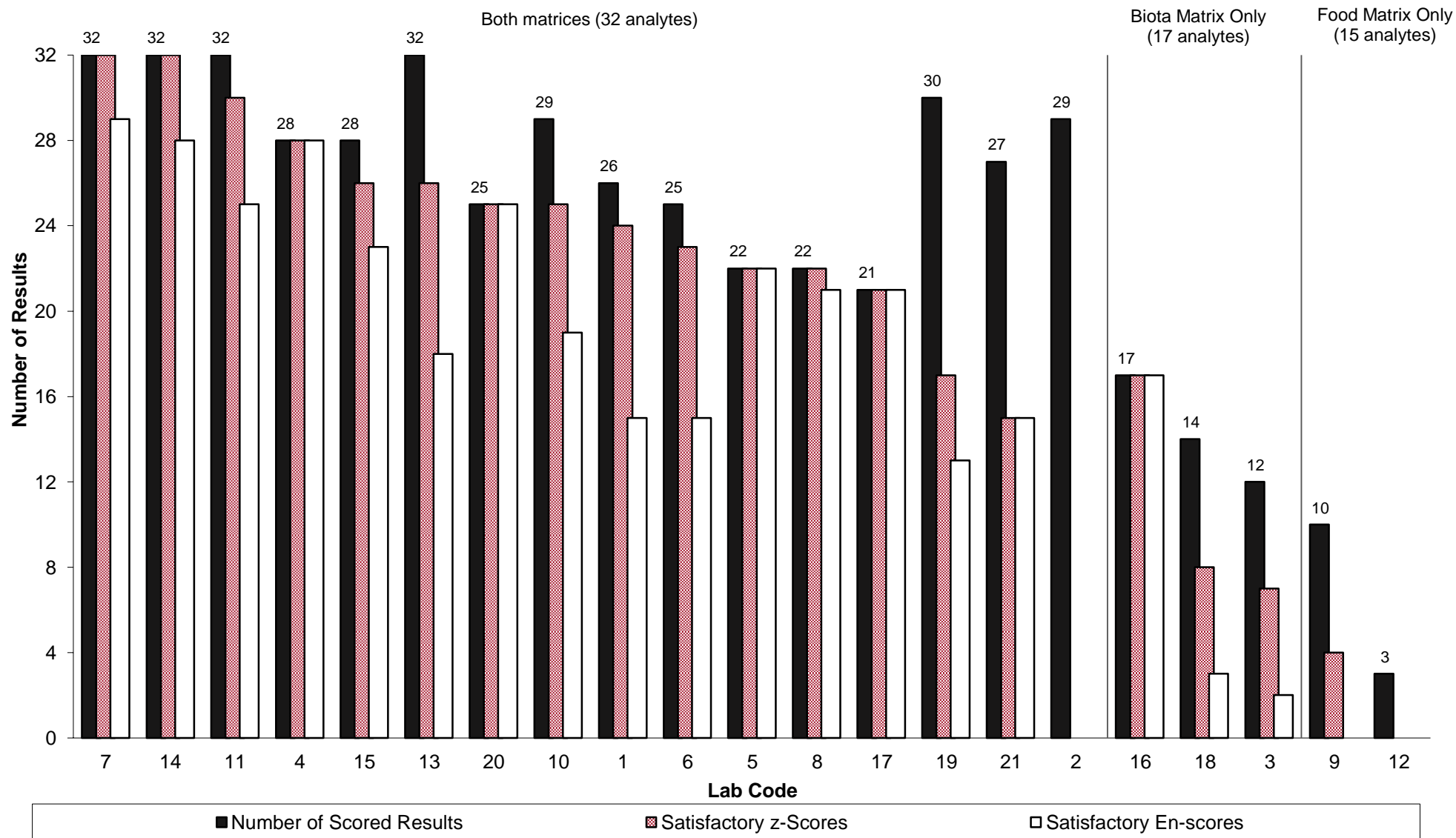


Figure 59 Summary of Participants' Performance



### 6.11 Comparison with Previous PFAS in Biota and Food Studies

NMI has coordinated PFAS in Biota and Food PT studies since 2016. AQA 18-09 and prior PT studies had PFAS in biota and food run in conjunction with soil and water matrices; only PFAS in biota and food results are presented here.

A summary of participation and reported results rates in PFAS in Biota and Food PT studies over the last 5 studies (2016 to 2020) is presented in Figure 60.

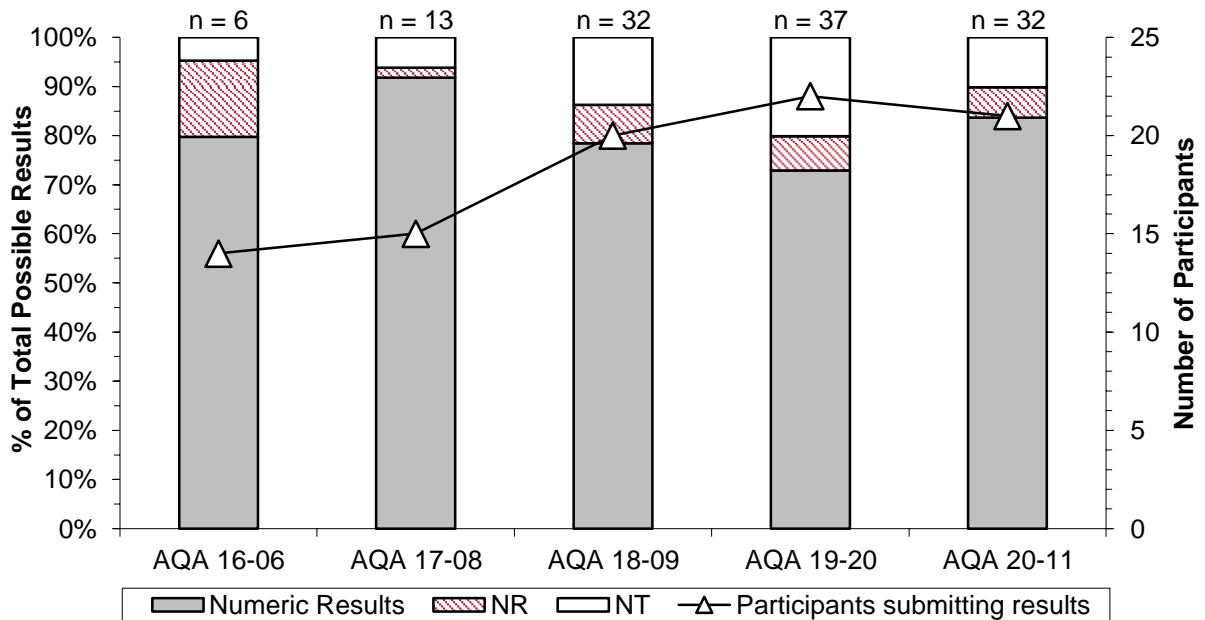


Figure 60 Summary of Participation and Reported Results in PFAS in Biota and Food PT Studies (n = number of spiked analytes).

A summary of the satisfactory performance (presented as a percentage of the total number of scores for each study) in PFAS in Biota and Food PT studies over the last 5 studies (2016 to 2020) is presented in Figure 61. The target SD used to calculate z-scores has been kept constant at 20% PCV. Over this period, the average proportion of satisfactory scores was 89% for z-scores and 77% for E<sub>n</sub>-scores.

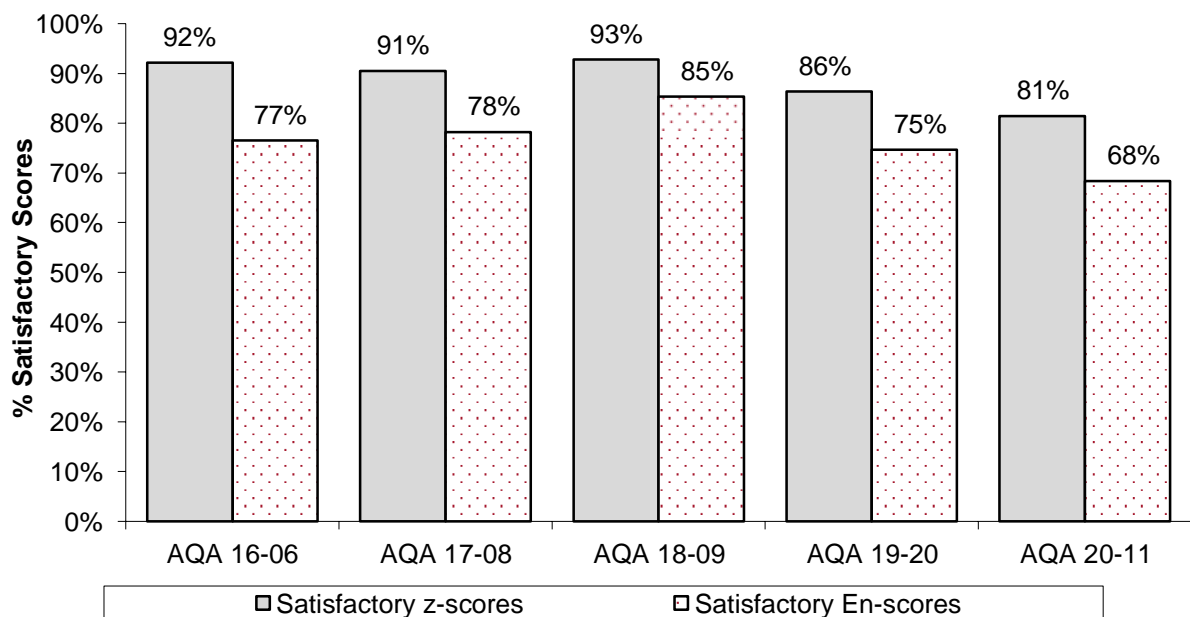


Figure 61 Summary of Participants' Performance for PFAS in Biota and Food PT Studies

The number of analytes assessed in each study has increased significantly as compared to the initial PFAS in Biota and 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 5 studies is presented in Figure 62.

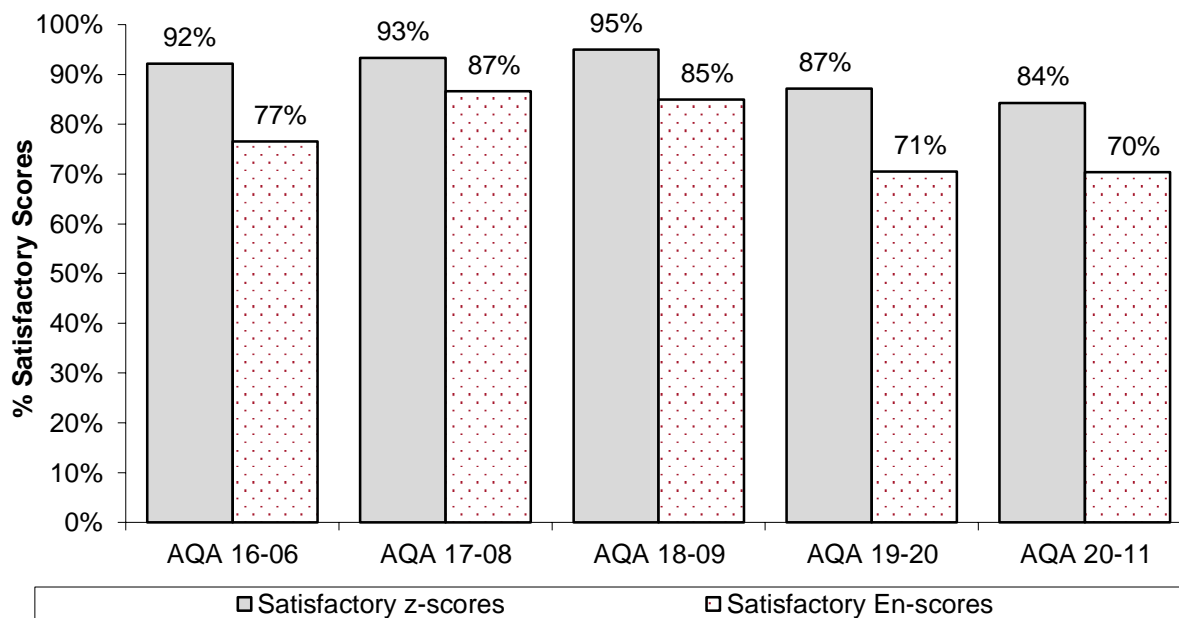


Figure 62 Summary of Participants' Performance for PFOS and PFOA in Biota and Food PT Studies

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## APPENDIX 1 – SAMPLE PREPARATION, HOMOGENEITY TESTING AND STABILITY CHECKING

### A1.1 Sample Preparation

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

**Sample S1:** Three kilograms of peeled and deveined raw prawns were bought from a local Sydney store. The prawns were blended to yield a puree. The pureed prawns were sprayed with a spiking solution containing PFAS analytes in methanol. This was thoroughly mixed, before being divided into patties of no more than 6 cm in diameter, covered, and placed into the freezer overnight at -80°C. The patties were then ground using a Retsch SM2000 Knife Mill; the set-up 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 prawn were packed into sample tubes. The tubes were labelled, shrink-wrapped, and then stored at -80°C prior to dispatch.

**Sample S2:** Fifteen kilograms of organic lettuce were bought from a Sydney organic fruit and vegetable wholesaler. The lettuces were rinsed, cut, blended, and then passed through an 850 µm sieve. The resultant puree was continuously stirred while 40 mL aliquots were dispersed into sample tubes to provide unspiked samples. The remaining lettuce 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.

### A1.2 Homogeneity Testing for Sample S2 Lettuce

Homogeneity testing was based on that described in the International Harmonized Protocol.<sup>4</sup>

Spiked lettuce samples were analysed at NMI North Ryde. The measurements were made under repeatability conditions in random order. Samples were prepared in duplicate by accurately weighing 1 g of the sample then spiking with 30 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 10 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. 1 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, Canada.

Results of the Sample S2 lettuce homogeneity testing are presented in Tables 45 to 56. For each analyte, the mean result of measurements was used as the homogeneity value. Samples were found to be sufficiently homogeneous for use in this PT study.

Table 45 Sample S2 PFBS Homogeneity Testing Results

Bottle Fill Number	PFBS ( $\mu\text{g}/\text{kg}$ )	
	Replicate 1	Replicate 2
3	2.63	2.90
14	2.75	2.75
20	2.76	2.97
25	2.89	2.82
32	2.89	2.92
38	2.83	2.80
48	2.83	2.77
Mean	2.82	
CV	3.1%	

Thompson and Fearn Homogeneity Tests<sup>9</sup>

Test	Value	Critical	Result
Cochran	0.57	0.73	<b>Pass</b>
$S_{\text{an}}/\sigma$	0.17	0.5	<b>Pass</b>
$s^2_{\text{sam}}$	0.000	0.07	<b>Pass</b>

Table 46 Sample S2 PFHxS Homogeneity Testing Results

Bottle Fill Number	PFHxS ( $\mu\text{g}/\text{kg}$ )	
	Replicate 1	Replicate 2
3	5.1	5.7
14	5.0	5.4
20	6.3	5.8
25	5.7	6.4
32	5.6	5.6
38	5.1	5.8
48	5.3	5.6
Mean	5.6	
CV	7.1%	

Thompson and Fearn Homogeneity Tests<sup>9</sup>

Test	Value	Critical	Result
Cochran	0.27	0.73	<b>Pass</b>
$S_{\text{an}}/\sigma$	0.32	0.5	<b>Pass</b>
$s^2_{\text{sam}}$	0.035	0.42	<b>Pass</b>

Table 47 Sample S2 PFHxS (linear) Homogeneity Testing Results

Bottle Fill Number	PFHxS (linear) (µg/kg)	
	Replicate 1	Replicate 2
3	5.0	5.6
14	4.9	5.2
20	6.0	5.6
25	5.5	5.8
32	5.4	5.4
38	5.0	5.6
48	5.1	5.5
Mean	5.4	
CV	6.1%	

Thompson and Fearn Homogeneity Tests<sup>9</sup>

Test	Value	Critical	Result
Cochran	0.35	0.73	<b>Pass</b>
$S_{an}/\sigma$	0.28	0.5	<b>Pass</b>
$s^2_{sam}$	0.021	0.35	<b>Pass</b>

Table 48 Sample S2 PFHpS Homogeneity Testing Results

Bottle Fill Number	PFHpS (µg/kg)	
	Replicate 1	Replicate 2
3	6.9	7.5
14	7.1	6.9
20	7.1	6.7
25	7.5	6.7
32	7.0	6.4
38	6.7	7.3
48	6.6	5.4
Mean	6.8	
CV	7.7%	

Thompson and Fearn Homogeneity Tests<sup>9</sup>

Test	Value	Critical	Result
Cochran	0.42	0.73	<b>Pass</b>
$S_{an}/\sigma$	0.35	0.5	<b>Pass</b>
$s^2_{sam}$	0.047	0.69	<b>Pass</b>

Table 49 Sample S2 PFOS Homogeneity Testing Results

Bottle Fill Number	PFOS ( $\mu\text{g}/\text{kg}$ )	
	Replicate 1	Replicate 2
3	23.2	26.5
14	23.2	24.8
20	26.9	23.4
25	24.5	21.9
32	22.6	21.7
38	23.6	25.0
48	22.1	19.1
Mean	23.5	
CV	8.7%	

Thompson and Fearn Homogeneity Tests<sup>9</sup>

Test	Value	Critical	Result
Cochran	0.28	0.73	<b>Pass</b>
$S_{\text{an}}/\sigma$	0.38	0.5	<b>Pass</b>
$s^2_{\text{sam}}$	1.066	8.65	<b>Pass</b>

Table 50 Sample S2 PFOS (linear) Homogeneity Testing Results

Bottle Fill Number	PFOS (linear) ( $\mu\text{g}/\text{kg}$ )	
	Replicate 1	Replicate 2
3	16.9	20.2
14	17.4	19.0
20	20.6	17.1
25	18.5	16.3
32	16.6	16.2
38	17.8	18.9
48	16.8	14.2
Mean	17.6	
CV	10%	

Thompson and Fearn Homogeneity Tests<sup>9</sup>

Test	Value	Critical	Result
Cochran	0.31	0.73	<b>Pass</b>
$S_{\text{an}}/\sigma$	0.47	0.5	<b>Pass</b>
$s^2_{\text{sam}}$	0.163	6.24	<b>Pass</b>

Table 51 Sample S2 PFBA Homogeneity Testing Results

Bottle Fill Number	PFBA ( $\mu\text{g}/\text{kg}$ )	
	Replicate 1	Replicate 2
3	20.0	21.6
14	21.1	20.4
20	22.9	21.1
25	22.1	18.9
32	18.9	20.6
38	21.1	19.5
48	19.6	20.6
Mean	20.6	
CV	5.7%	

Thompson and Fearn Homogeneity Tests<sup>9</sup>

Test	Value	Critical	Result
Cochran	0.44	0.73	<b>Pass</b>
$S_{\text{an}}/\sigma$	0.31	0.5	<b>Pass</b>
$s^2_{\text{sam}}$	0.000	5.59	<b>Pass</b>

Table 52 Sample S2 PFPeA Homogeneity Testing Results

Bottle Fill Number	PFPeA ( $\mu\text{g}/\text{kg}$ )	
	Replicate 1	Replicate 2
3	26.7	27.4
14	26.6	26.9
20	27.7	27.2
25	26.1	27.8
32	26.5	27.4
38	26.9	27.4
48	26.9	27.2
Mean	27.1	
CV	1.8%	

Thompson and Fearn Homogeneity Tests<sup>9</sup>

Test	Value	Critical	Result
Cochran	0.60	0.73	<b>Pass</b>
$S_{\text{an}}/\sigma$	0.11	0.5	<b>Pass</b>
$s^2_{\text{sam}}$	0.000	6.05	<b>Pass</b>



Table 53 Sample S2 PFHxA Homogeneity Testing Results

Bottle Fill Number	PFHxA (µg/kg)	
	Replicate 1	Replicate 2
3	38	39
14	39	39
20	40	39
25	35	40
32	39	38
38	39	37
48	38	36
Mean	38	
CV	3.6%	

Thompson and Fearn Homogeneity Tests<sup>9</sup>

Test	Value	Critical	Result
Cochran	0.64	0.73	<b>Pass</b>
$S_{an}/\sigma$	0.21	0.5	<b>Pass</b>
$s^2_{sam}$	0.000	14.74	<b>Pass</b>

Table 54 Sample S2 PFOA Homogeneity Testing Results

Bottle Fill Number	PFOA (µg/kg)	
	Replicate 1	Replicate 2
3	15.5	14.8
14	13.5	14.8
20	14.8	15.2
25	15.6	14.1
32	14.2	13.8
38	12.8	15.7
48	14.0	13.4
Mean	14.4	
CV	6.3%	

Thompson and Fearn Homogeneity Tests<sup>9</sup>

Test	Value	Critical	Result
Cochran	0.62	0.73	<b>Pass</b>
$S_{an}/\sigma$	0.34	0.5	<b>Pass</b>
$s^2_{sam}$	0.000	2.98	<b>Pass</b>

Table 55 Sample S2 PFNA Homogeneity Testing Results

Bottle Fill Number	PFNA ( $\mu\text{g}/\text{kg}$ )	
	Replicate 1	Replicate 2
3	3.3	3.8
14	3.7	3.4
20	3.6	4.1
25	3.7	3.6
32	3.5	3.1
38	3.7	3.6
48	3.3	3.0
Mean	3.5	
CV	7.8%	

Thompson and Fearn Homogeneity Tests<sup>9</sup>

Test	Value	Critical	Result
Cochran	0.31	0.73	<b>Pass</b>
$S_{\text{an}}/\sigma$	0.34	0.5	<b>Pass</b>
$s^2_{\text{sam}}$	0.019	0.18	<b>Pass</b>

Table 56 Sample S2 PFDA Homogeneity Testing Results

Bottle Fill Number	PFDA ( $\mu\text{g}/\text{kg}$ )	
	Replicate 1	Replicate 2
3	3.22	3.49
14	3.29	3.06
20	3.30	3.17
25	3.30	3.36
32	2.98	2.84
38	2.74	2.91
48	2.83	2.83
Mean	3.09	
CV	7.8%	

Thompson and Fearn Homogeneity Tests<sup>9</sup>

Test	Value	Critical	Result
Cochran	0.37	0.73	<b>Pass</b>
$S_{\text{an}}/\sigma$	0.19	0.5	<b>Pass</b>
$s^2_{\text{sam}}$	0.048	0.09	<b>Pass</b>

### A1.3 Stability Testing for Sample S2 Lettuce

As lettuce has not previously been used as a matrix for PFAS in NMI PT studies, a stability study was conducted for Sample S2. Short term stability was assessed by comparing the average of duplicate measurements of samples starting at the beginning of October 2020 (approximately sample dispatch date) and finishing in December 2020 (approximately results due date). Results were in good agreement with each other and the assigned value (Figure 63).

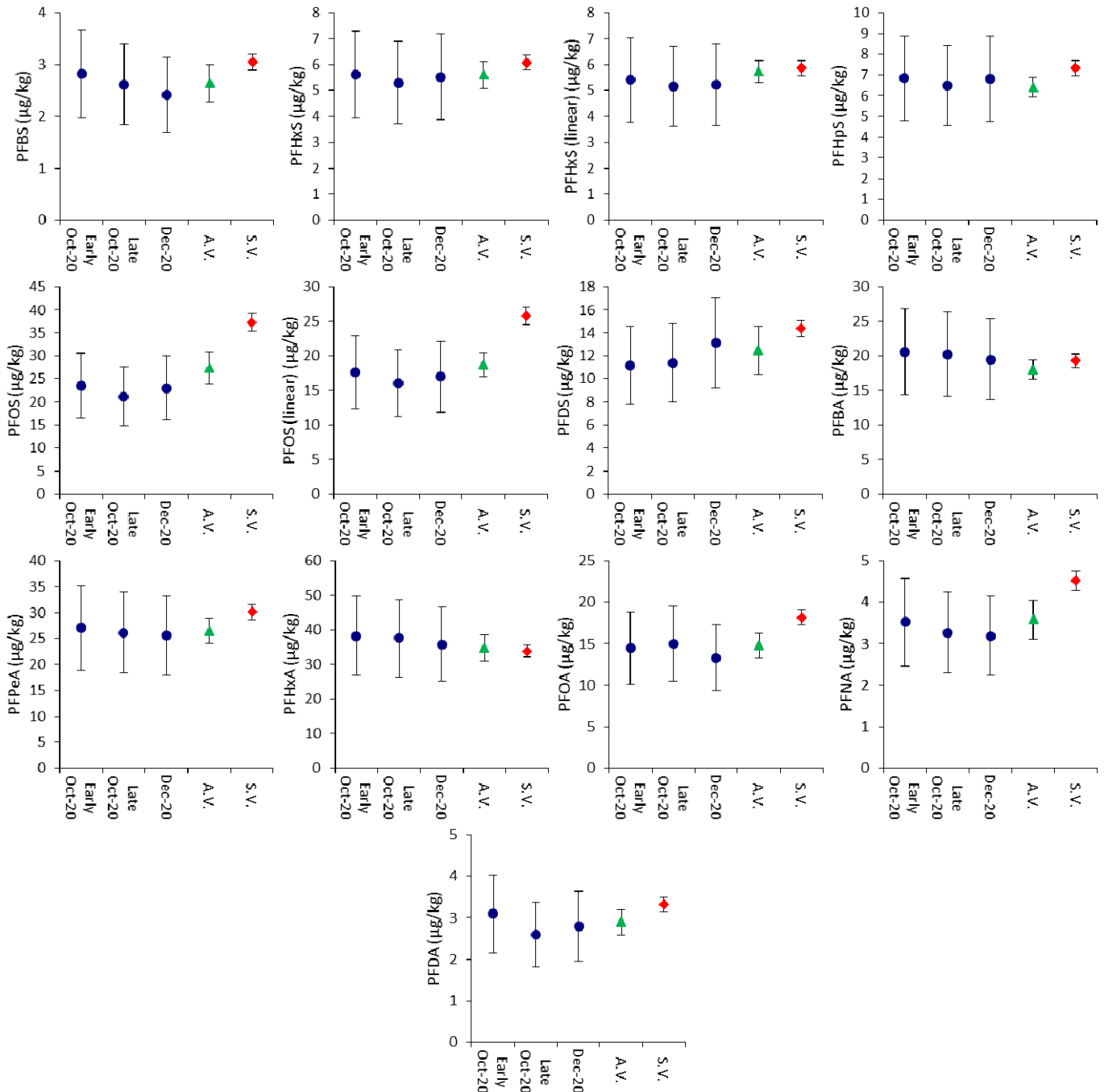


Figure 63 Stability Results, Assigned Value (A.V.) and Spiked Value (S.V.) for Sample S2 Lettuce

To confirm the stability during transportation, results returned by participants were compared to the number of days the samples spent in transit, and subjected to a trend analysis (Figure 64; all results from Laboratory 2 have been excluded as extreme outliers). No trend was evident, providing further evidence of sample stability.

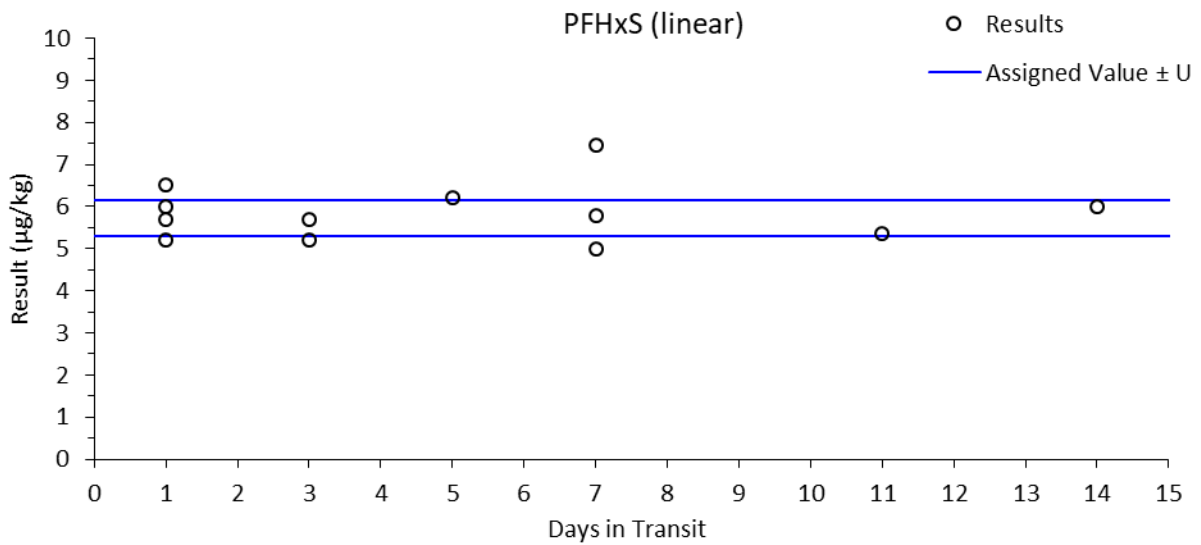
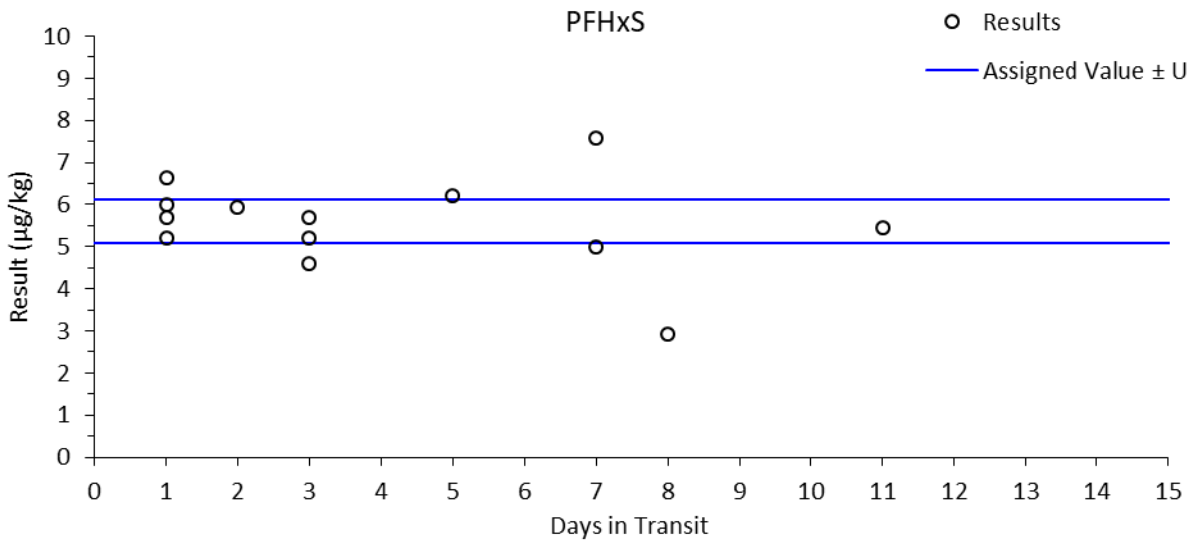
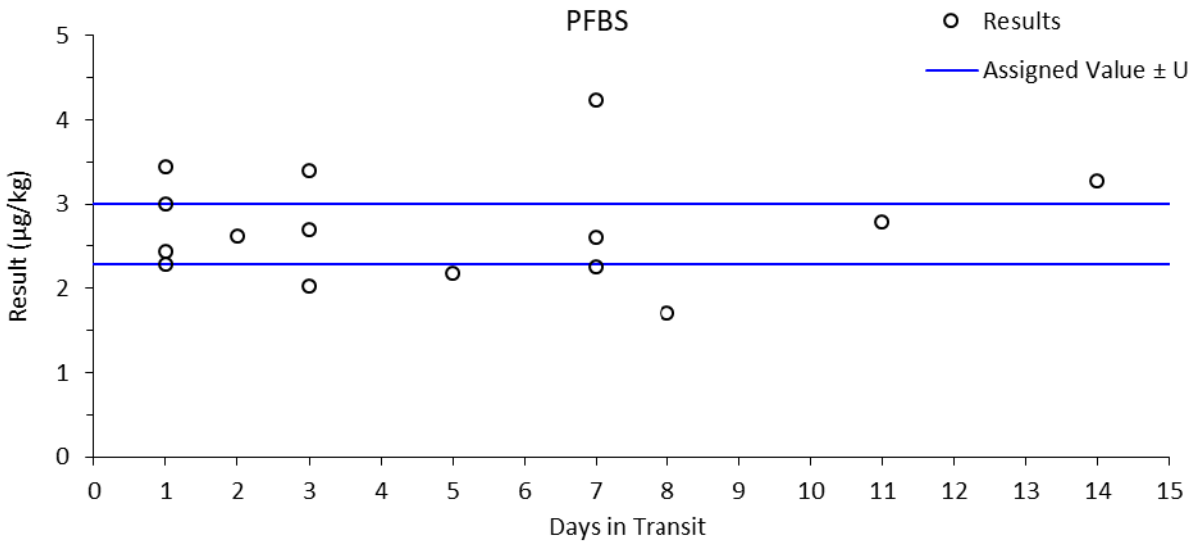


Figure 64 Participant Results vs Days in Transit for Sample S2 Lettuce

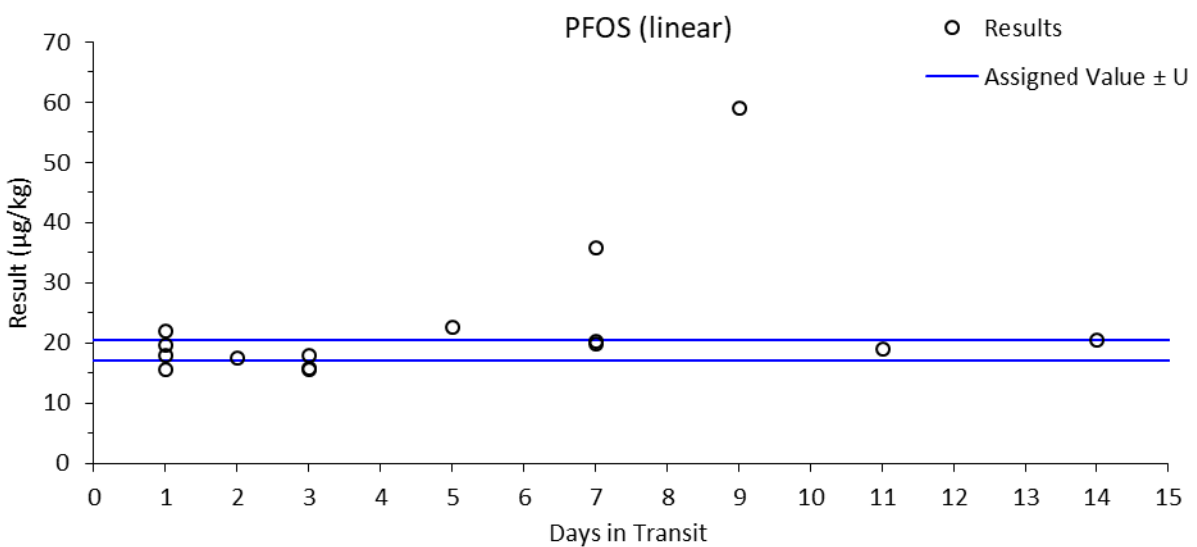
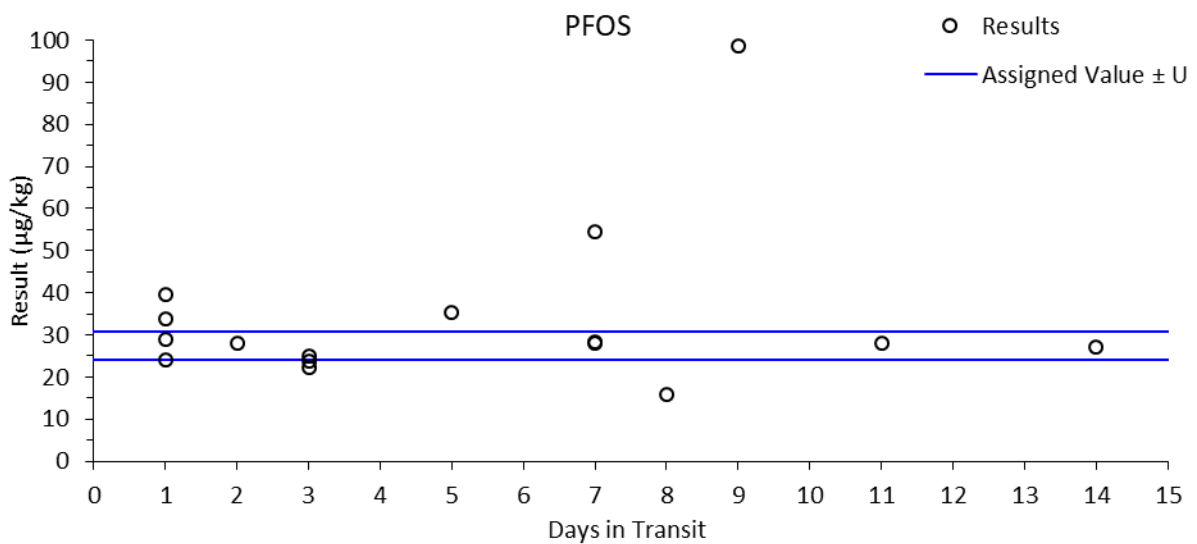
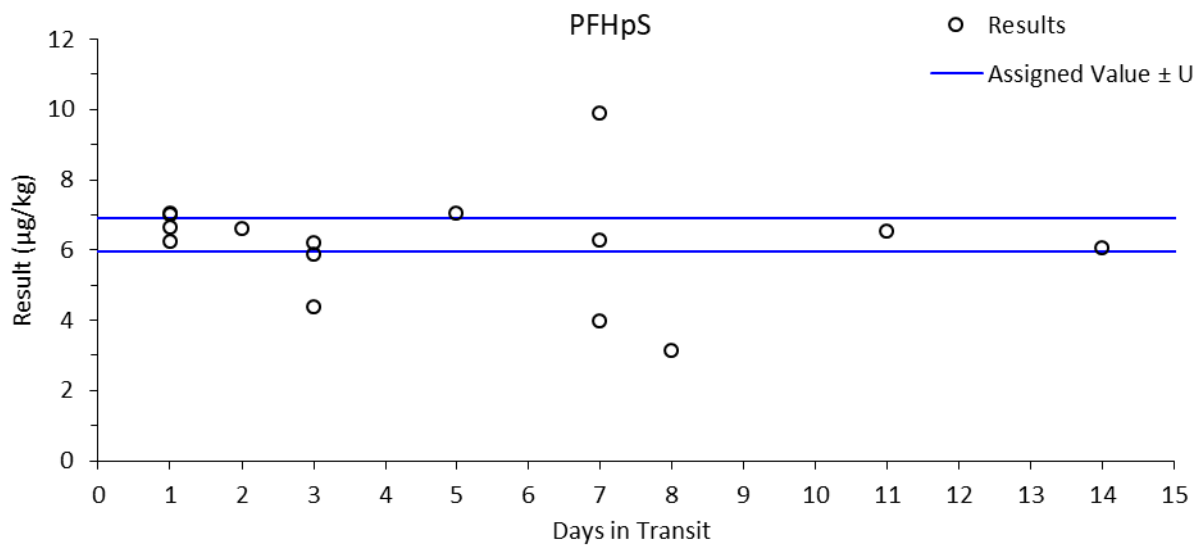


Figure 64 (continued) Participant Results vs Days in Transit for Sample S2 Lettuce

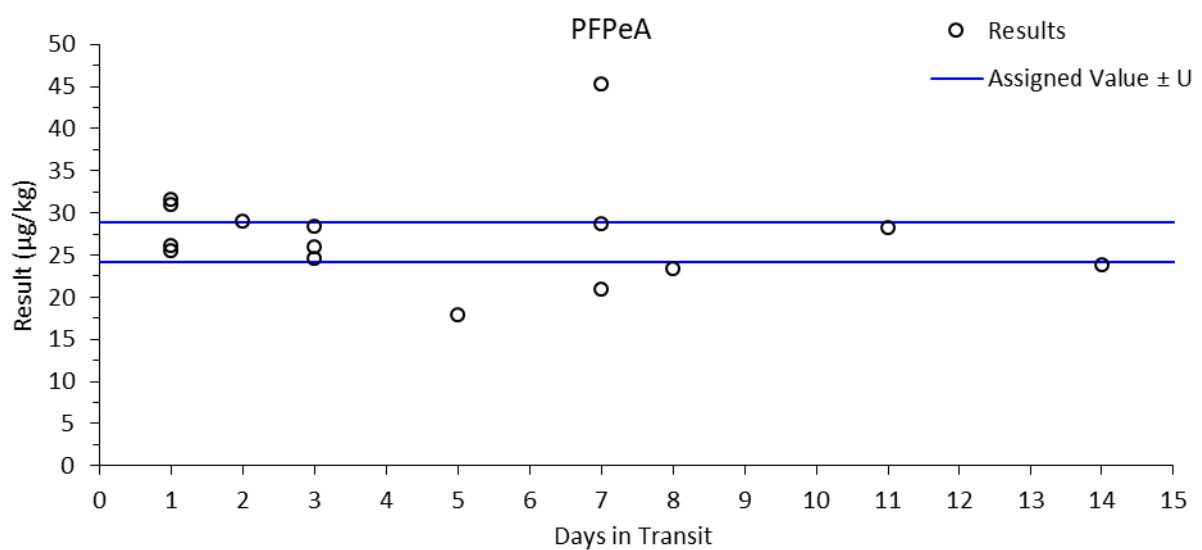
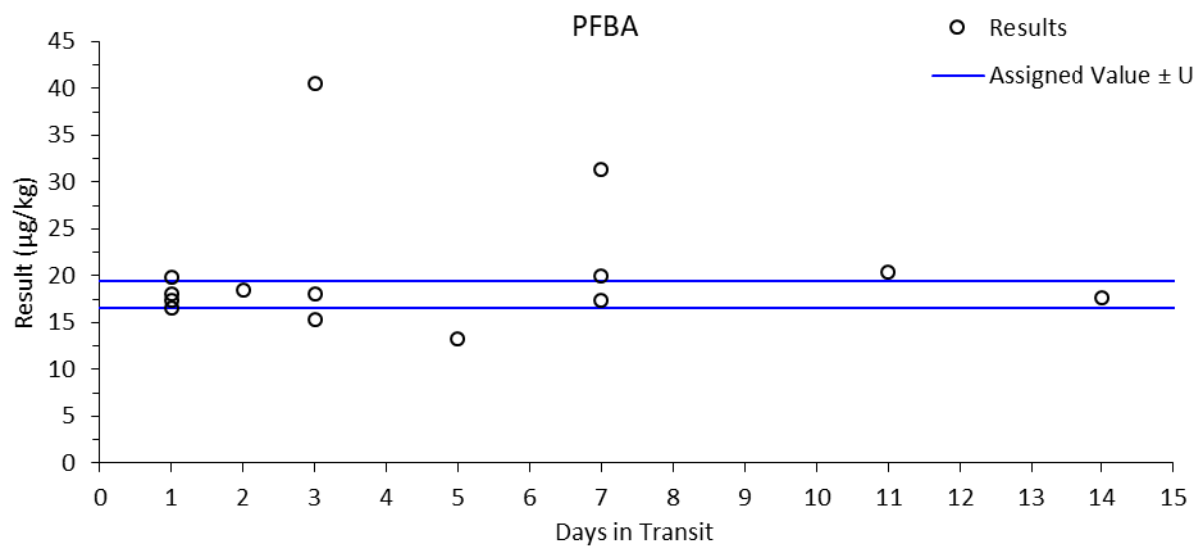
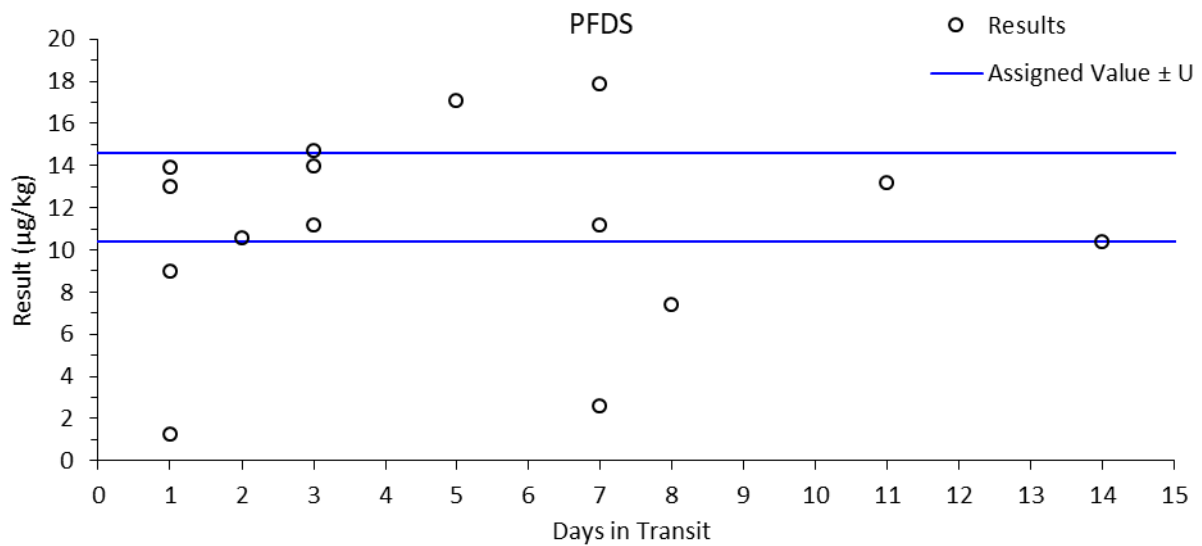


Figure 64 (continued) Participant Results vs Days in Transit for Sample S2 Lettuce

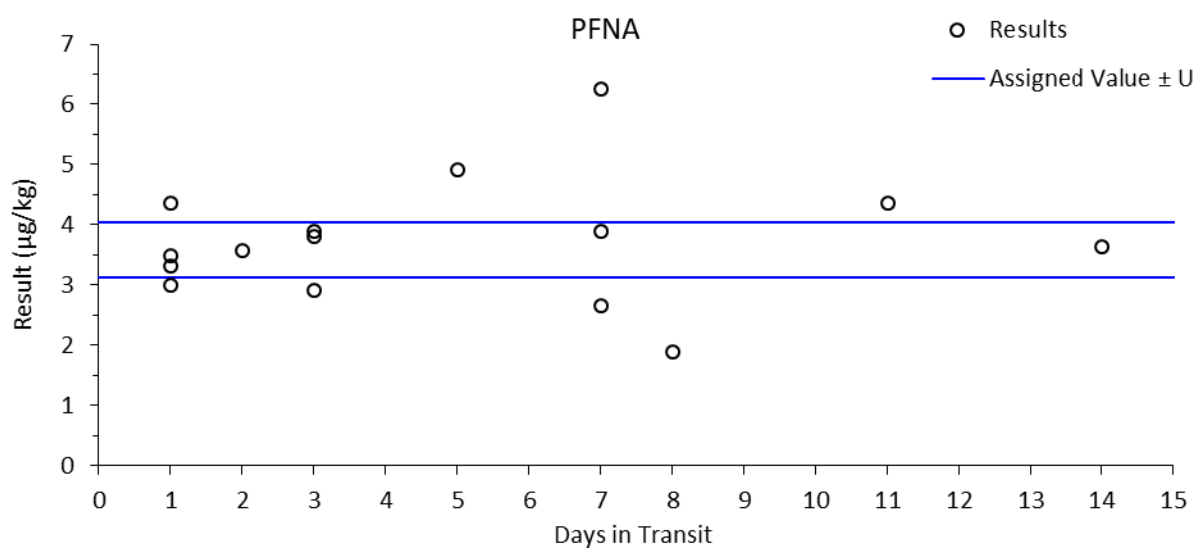
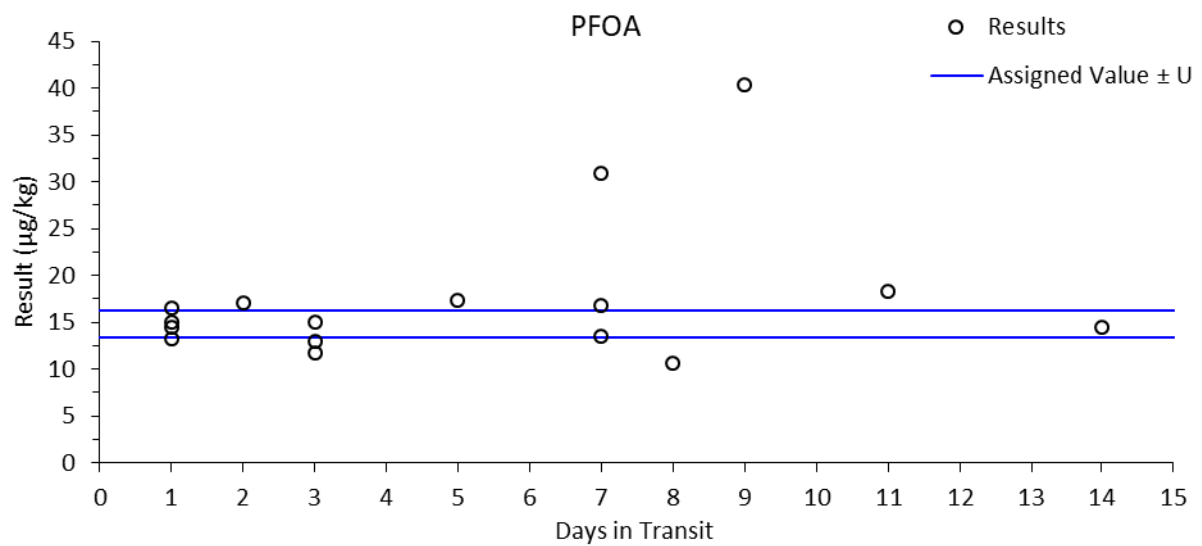
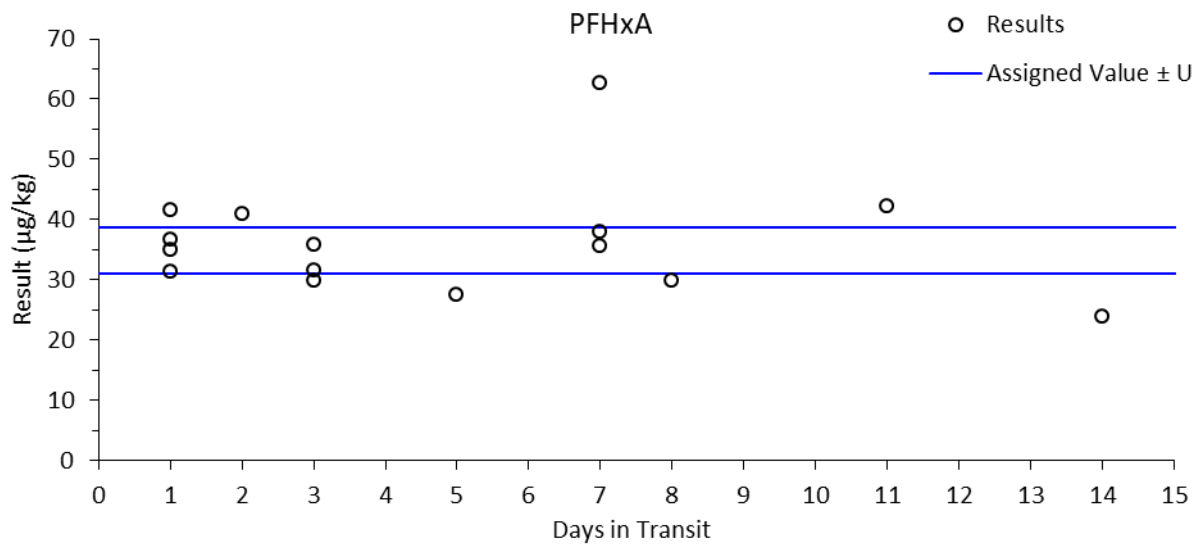


Figure 64 (continued) Participant Results vs Days in Transit for Sample S2 Lettuce

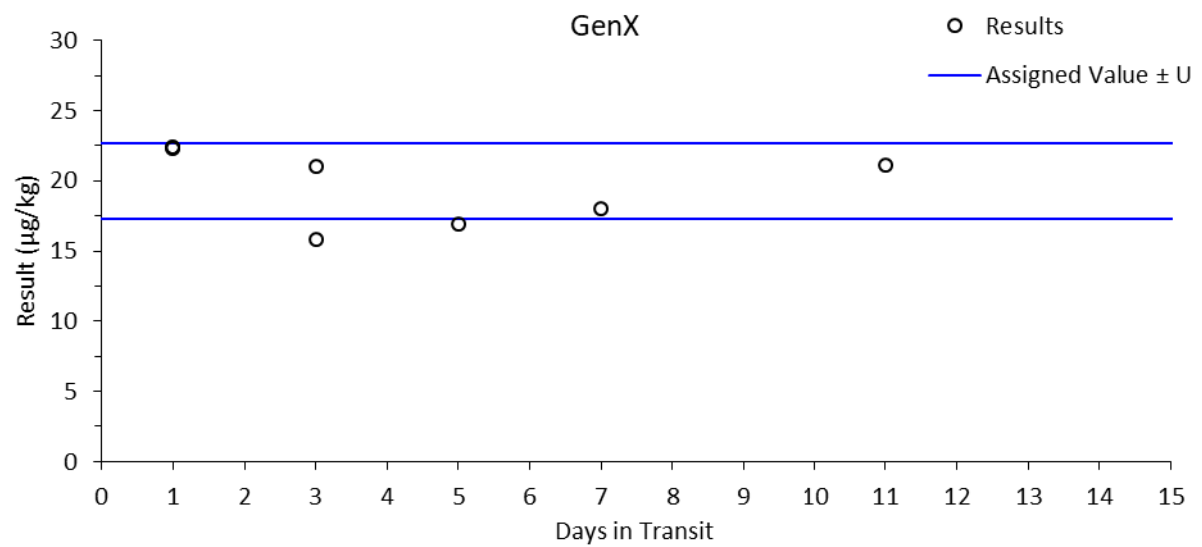
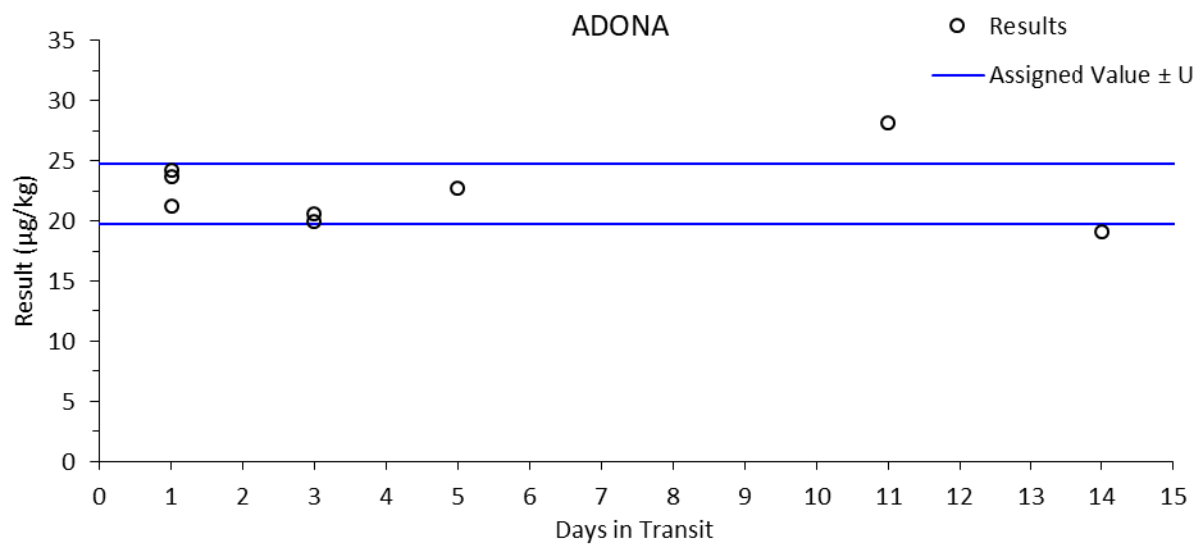
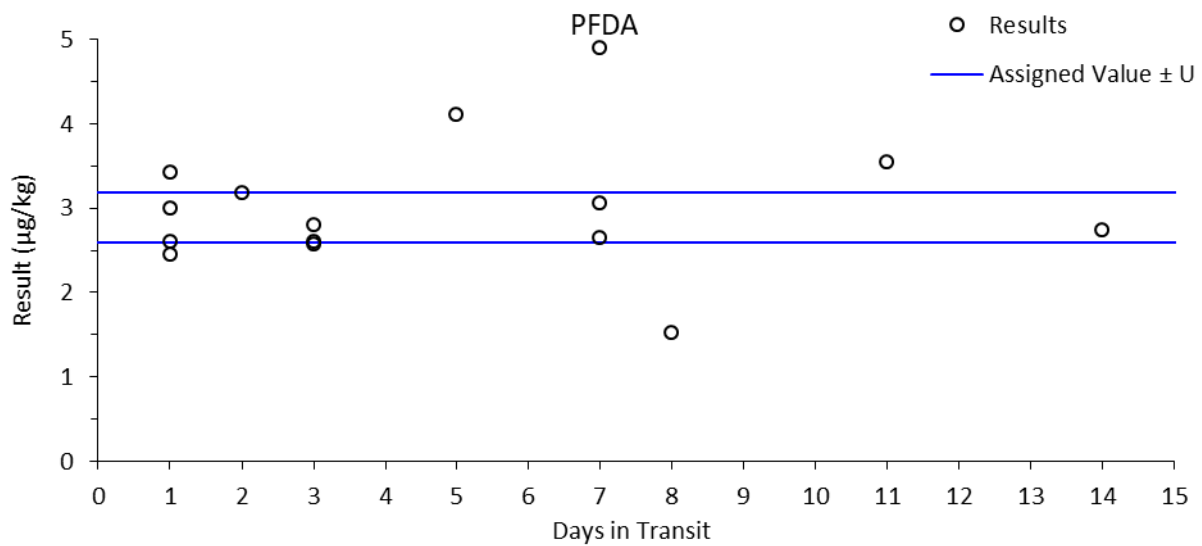


Figure 64 (continued) Participant Results vs Days in Transit Sample S2 Lettuce



## APPENDIX 2 – PARTICIPANTS’ TEST METHODS

Participants’ responses to the methodology questionnaire are presented in Tables 57 to 78. Some responses have been modified so that the participant cannot be identified.

Table 57 Participant Methodology – Extraction

Lab. Code	S1 Sample Weight (g)	S2 Sample Weight (g)	Sample Pre-treatment	Extraction Technique	Extraction Solvent	Extraction Process	Extraction Temperature	Extraction Time	Extraction Clean Up
1	1.5	2.5	Homogenisation	Solid-Liquid Extraction (SLE)	Methanol	Sonication			Filtration
2	0.5	0.1	Freezer dry	Alkaline Digestion	NaOH	Sonication	Room T	S1: half hour S2: over night	Carbon cartridge
3	1	NT	Homogenisation	Solid-Liquid Extraction (SLE)	Acidified ACN/H <sub>2</sub> O	Shaking	Room	1 min	Clean up with Hexane pre-saturated ACN
4	1	1	Homogenisation	Alkaline Digestion	KOH-methanol	Tumbling	Room temp	8 hrs	Active carbon SPE
5	1	2	Homogenisation	Alkaline Digestion	Basified MeOH	Sonication / Shaking / SPE	Room	60 mins	Envicarb
6	0.3	2	-	Solid Phase Extraction (SPE)	Acetonitrile (+ 0.1 % Ammonia)	Shaking	RT	> 12 h	S1: SPE Column: Envi-Carb <sup>TM</sup> (Supelco) S2: SPE Column: Strata-X-AW (Phenomenex)
7	2	2	Homogenisation	Solid Phase Extraction (SPE)	Acetonitrile	Shaking	Room Temperature	30 mins	SPE
8	2.073	2.027	NA	Solid-Liquid Extraction (SLE)	2% formic acid in acetonitrile	Merris-Minimix shaker	Room temperature	8 min	dSPE (C18, Envicarb, MgSO <sub>4</sub> )
9	NT	0.9937	Homogenisation	ACN Sonication followed by SPE	Extracted with Acetonitrile, SPE eluted with Methanol	Sonication	Room Temp	2x 15 minutes	EMR-dSPE followed by SPE

Lab. Code	S1 Sample Weight (g)	S2 Sample Weight (g)	Sample Pre-treatment	Extraction Technique	Extraction Solvent	Extraction Process	Extraction Temperature	Extraction Time	Extraction Clean Up
10	5	5	Homogenisation	Solid Phase Extraction (SPE)	Acetonitrile	Tumbling	Ambient	30min	SPE
11	1.06	1.36	Homogenisation	Alkaline Digestion	KOH / Methanol	Combination of Shaker / Sonication	Room temperature	3 hour shake, 12 hour sonication bath	SPE (WAX 150mg/6cc)
12	NT	2	Homogenisation	Liquid-Liquid Extraction (LLE)	Methyl tert butylether MTBE	Shaking	room	35 min	No clean up
13	0.5	0.5	S1: Base Digestion S2: Homogenisation	Sonication	S1:Acetonitrile S2:Methanol and 0.2M Sodium Hydroxide in Methanol	Sonication	S1: Room Temp S2: 4°C overnight and then room temperature	30 Min	S1: centrifugation, liquid liquid extraction(n-hexane) followed by active carbon S2: Centrifugation and carbon cartridge
14	1	5	Homogenisation	Solid-Liquid Extraction (SLE)	Acetonitrile	Shaking	Room	20 minutes	C18
15	0.2	0.4	Homogenisation	Alkaline Digestion	NaOH-methanol	Shaking	RT	16h	SPE
16	2.03	NT	Homogenisation	Solid-Liquid Extraction (SLE)	KOH-Methanol	Shaking	Ambient/ Room	16 hours	Activated Carbon/SPE/Filtration
17	0.5	0.5	Homogenisation	Solid-Liquid Extraction (SLE)	Acetonitrile	Shaking	Room	60 minutes	C18 & Activated Carbon
18	1.7	NT	Homogenisation	Solid-Liquid Extraction (SLE)	10 ml of NaOH 10 mM in methanol	Sonication	Room temperature (20°C)	1 h	SPE with Oasis WAX 3cc
19	1.1	1	S1: none S2: drying agent added	sonication/SPE	Acetonitrile : MeOH	Sonication / vortex	ambient	S1: 30minutes S2: 1 hour	None

Lab. Code	S1 Sample Weight (g)	S2 Sample Weight (g)	Sample Pre-treatment	Extraction Technique	Extraction Solvent	Extraction Process	Extraction Temperature	Extraction Time	Extraction Clean Up
20	1	1	no	Solid-Liquid Extraction (SLE)	Acetonitrile	Vortex, polytron, sonication	Room temp	20 min	SPE-WAX, ultracentrifugation
21	1.95	2.67	Homogenisation	Solid Phase Extraction (SPE)	Acetonitrile	Sonication + Quechers	40°C	30 min	active carbon /SPE

Table 58 Participant Methodology – Extraction Additional Information

Lab. Code	Extraction Additional Information
2	The results were based on the dry sample -The sample was freezer dried before it was analysed.
9	1g of sample is sonicated twice in ACN, extract added to EMD dSPE and polishing salts to remove lipids, extract is then added to water and put through SPE. Elutant in Methanol is evaporated and final extract in 1:1 methanol:water is viald.
12	Concentration under N2
16	Isotopically labelled surrogate standards were spiked into sample prior to extraction
18	After extraction with methanol (NaOH) the samples were centrifuged. The supernatant was evaporated under N2 current and reconstituted in 100 mL of HPLC-Water before SPE purification

Table 59 Participant Methodology – 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		BEH C18 50 mm x 2.1 mm x 1.7 µm		No	No	
2	LC-MSMS or LC-QQQ	C18,	C18, 100*2.1 3ul		Yes	No	No
3	LC-MSMS or LC-QQQ		C18 2.0mmx50mm(1.6um)		Yes	No	
4	LC-MSMS or LC-QQQ	No	C18, 50 mm	No	Yes	No	
5	LC-MSMS or LC-QQQ	Pre-column Filter 0.2µm	C18 50mm x 2.1 mm x 1.8µm	50	Yes	No	No. In-house
6	LC-MSMS or LC-QQQ	XBridge BEH C18; 3.5 µm; 2.1mmx5mm	XBridge C 18; 3.5 µm; 2.1mmx150mm	No	Yes	No	

Lab. Code	Instrument	Guard Column	Instrument Column	Dilution Factor	Delay Column?	Blank Correction?	Standard Method?
7	Orbitrap	C18	C18		Yes	No	
8	LC-MSMS or LC-QQQ	NA	Zorbax XDB-C18, 100 mm x 2.1 mm, 1.8µm	NA	Yes	No	No
9	LC-MSMS or LC-QQQ	Pinnacle DB Aqueous C18, 10 x 2.1mm, 5.0 µm	Pinnacle DB AQ C18, 50 x 2.1mm, 1.9 µm	No	Yes	No	
10	LC-MSMS or LC-QQQ	UHPLC guard column; AU; InfinityLabPoroshell 120; EC-C18; 4.6 mm; 4 µm	LC column; AU; Poroshell 120 HPH C18; 2.1x50 mm; 2.7 µm; narrow bore	NO	Yes	No	Isotope dilutions
11	LC-MSMS or LC-QQQ		C18 10cm x 3.0 mm x 3 µm	No	Yes	No	No
12	LC-MSMS or LC-QQQ	No	Raptor - Biphenyl, 100mm, 2.7µm, 2.1mm	No	No	No	Isotope dilution
13	LC-MSMS or LC-QQQ	Evo C18 2 x 2.1mm	Evo C18 2.6 µ 100x2.1 mm	No	Yes	No	No
14	Orbitrap	C18	C18 50 x 2.1 mm	NO	C18 50 x 4.6 mm	Yes	
15	LC-MSMS or LC-QQQ	PFP 5mm×2.1mm×1.8µm	PFP 150mm×2.1mm×1.8µm	No	Yes	Yes	
16	LC-MSMS or LC-QQQ	Phenomenex Evo C18	BEH C18; 1.7µm, 50 x 2.1 mm	None	Yes	No	No
17	LC-MSMS or LC-QQQ	nil	C18 1.6µm, 2.0mm x 50mm	No	Yes	No	QuEChERS
18	Orbitrap	Not used	ACQUITY UPLC BEH C18 (2.1 x 50 mm; 1.7 µm)	Yes, 2/10	No	No	Calibration curve with surrogate internal standards.
19	LC/MS/MS			No	No	NO	Isotope dilution
20	LC-MSMS or LC-QQQ	Gemini NX-C18; 4 mm x 2.0 mm ID	NX-C18; 15 cm x 2 mm x 3 µm	no	Yes	Yes	No
21	LC-MSMS or LC-QQQ		Nucleodur Sphinx RP C18, isis 3µm		Yes	No	

Table 60 Participant Methodology – Labelled Standards

Lab. Code	Labelled Standard Source	Recovery Correction?	Labelled Standards Additional Information
1	Wellington	Yes	
2	Wellington	No	
3	Wellington	No	
4	Wellington	Yes	
5	Wellington	Yes	
6	Wellington	Yes	
7	Wellington	Yes	
8	Wellington Laboratory	Yes	NA
9	Wellington	Yes	PFOS-13C8 is used a surrogate
10	Wellington Laboratories	Yes	
11	Wellington	Yes	
12	Cambridge Isotope Laboratories, ES-5571	Yes	
13	Wellington	Yes	
14	Wellington	Yes	Results calibrated by ISTD added before instrumentation
15	Wellington	No	
16	Wellington	Yes	
17	Wellington Laboratories	Yes	
18	Wellington	Yes	
19	Wellington	Yes	
20	Wellington	Yes	
21	Wellington	Yes	

Table 61 Labelled Standards for PFBS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2		
3	18O2 PFHxS	
4	13C3 PFBS	
5	13C3-PFBS	N/A
6	M3PFBS	
7	PFBS-13C3	PFOS-13C8
8	M3PFBS	NA
9		
10	13C3-PFBS	
11	13C3 PFBS	
12		
13	13C3-PFBS	13C3-PFHxS
14	PFOS-13C8	PFBS-13C3
15	13C3-PFBS	
16	13C3-PFBS	1802-PFHxS
17	13C3-PFBS	
18	x	
19	13C3-PFBS	13C3-PFBA
20	18O2-PFHxS	18O2-PFOS
21	13C3 PFBS	13C4 PFOA

Table 62 Labelled Standards for PFHxS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2		
3	18O2PFHxS ISTD	
4	13C3 PFHxS	
5	18O2-PFHxS	N/A
6	M3PFHxS	
7	PFHxS-18O2	PFOS-13C8
8	M3PFHxS	NA
9		
10	18O2-PFHxS	
11	18O2 PFHxS	
12		
13	18O2-PFHxS	13C3-PFHxS
14	PFOS-13C8	PFHxS-18O2
15	13C3-PFHxS	
16	13C3-PFHxS	1802-PFHxS
17	16O2-PFHxS	
18		
19	13C3-PFHxS	13C2-PFOA
20		
21	18O2 PFHxS	13C4 PFOA

Table 63 Labelled Standards for PFHxS (linear)

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2	Yes	
3	18O2PFHxS ISTD	
4	13C3 PFHxS	
5	18O2-PFHxS	N/A
6	M3PFHxS	
7	PFHxS-18O2	PFOS-13C8
8	M3PFHxS	NA
9		
10	18O2-PFHxS	
11	18O2 PFHxS	
12		
13	18O2-PFHxS	13C3-PFHxS
14	PFOS-13C8	PFHxS-18O2
15	13C3-PFHxS	
16	13C3-PFHxS	1802-PFHxS
17		
18	x	
19	13C3-PFHxS	13C2-PFOA
20	18O2-PFHxS	18O2-PFOS
21	18O2 PFHxS	13C4 PFOA

Table 64 Labelled Standards for PFHpS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2		
3	18O2PFHxS ISTD	
4	13C3 PFHxS	
5	13C4-PFOS	N/A
6	M3PFHxS	
7	PFOS-13C4	PFOS-13C8
8	M3PFHxS	NA
9		
10		
11	18O2 PFHxS	
12		
13	18O2-PFHxS	13C3-PFHxS
14	PFOS-13C8	PFOS-C4
15	13C3-PFHxS	
16	13C8-PFOS	13C4-PFOS
17	13C8-PFOS	
18		
19	13C3-PFHxS	13C2-PFOA
20	18O2-PFHxS	18O2-PFOS
21	18O2 PFHxS	13C4 PFOA

Table 65 Labelled Standards for PFOS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2		
3	13C4 PFOS ISTD	
4	13C8 PFOS	13C4 PFOS
5	13C4-PFOS	N/A
6	M8PFOS	
7	PFOS-13C4	PFOS-13C8
8	M8PFOS	NA
9	PFOS-13C8	MPFOS-13C4
10	13C8-PFOS	
11	13C4 PFOS	
12		
13	13C4-PFOS	13C8-PFOS
14	PFOS-13C8	PFOS-C4
15	13C8-PFOS	
16	13C8-PFOS	13C4-PFOS
17	13C8-PFOS	
18		
19	13C8-PFOS	13C4-PFOS
20	13C4-PFOS	18O2-PFOS
21	13C4 PFOS	13C4 PFOA

Table 66 Labelled Standards for PFOS (linear)

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2	Yes	
3	13C4 PFOS ISTD	
4	13C8 PFOS	
5	13C4-PFOS	N/A
6	M8PFOS	
7	PFOS-13C4	PFOS-13C8
8	M8PFOS	NA
9		
10	13C8-PFOS	
11	13C4 PFOS	
12	yes	
13	13C4-PFOS	13C8-PFOS
14	PFOS-13C8	PFOS-C4
15	13C8-PFOS	
16	13C8-PFOS	13C4-PFOS
17	13C8-PFOS	
18	x	
19	13C8-PFOS	13C4-PFOS
20	13C4-PFOS	18O2-PFOS
21	13C4 PFOS	13C4 PFOA

Table 67 Labelled Standards for PFDS

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2		
3	13C4 PFOS ISTD	
4	13C8 PFOS	
5	13C4-PFOS	N/A
6	M8PFOS	
7	6:2 FTS-13C2	PFOS-13C8
8	M8PFOS	NA
9		
10		
11	13C4 PFOS	
12		
13	13C4-PFOS	13C8-PFOS
14	PFOS-13C8	PFBA-13C4
15	13C8-PFOS	
16	13C8-PFOS	13C4-PFOS
17	13C8-PFOS	
18		
19	13C8-PFOS	13C4-PFOS
20	13C4-PFOS	18O2-PFOS
21	13C2 PFUnA	13C4 PFOA

Table 68 Labelled Standards for PFBA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2	yes	
3	13C4 PFBA ISTD	
4	13C4 PFBA	13C3 PFBA
5	13C4-PFBA	N/A
6	M4PFBA	
7	PFBA-13C4	PFOS-13C8
8	M4PFBA	NA
9		MPFBA
10	13C4-PFBA	
11	13C2 PFHxA	
12		
13	13C4-PFBA	13C3-PFBA
14	PFOS-13C8	PFBA-13C4
15	13C4-PFBA	
16	13C4-PFBA	13C3-PFBA
17	13C4-PFBA	
18	x	
19	13C4-PFBA	13C3-PFBA
20	13C4-PFBA	13C8-PFOA
21	13C4 PFBA	13C4 PFOA

Table 69 Labelled Standards for PFPeA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2	yes	
3	13C4 PFBA ISTD	
4	13C5 PFPeA	
5	13C3-PFPeA	N/A
6	M5PFPA	
7	PFPeA-13C3	PFOS-13C8
8	M5PFPeA	NA
9		
10	13C5-PFPeA	
11	13C4 PFHpA	
12		
13	13C4-PFPeA	13C5 -PFPeA
14	PFOS-13C8	PFPeA-13C3
15	13C5-PFPeA	
16	13C5-PFPeA	13C2-PFHxA
17	13C5-PFPeA	
18	x	
19	13C5-PFPeA	13C3-PFBA
20	13C5-PFPeA	13C8-PFOA
21	13C5 PFPeA	13C4 PFOA



Table 70 Labelled Standards for PFHxA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2	yes	
3	13C2 PFHxA ISTD	
4	13C5 PFHxA	
5	13C2-PFHxA	N/A
6	M5PFHxA	M2PFHxA
7	PFHxA-13C2	PFOS-13C8
8	M5PFHxA	NA
9		
10	13C2-PFHxA	
11	13C2 PFHxA	
12		
13	13C2-PFHxA	13C5 -PFPeA
14	PFOS-13C8	PFHxA=13C2
15	13C5-PFHxA	
16	13C5-PFHxA	13C2-PFHxA
17	13C5-PFHxA	
18	x	
19	13C5-PFHxA	13C2-PFOA
20	13C5-PFHxA	13C8-PFOA
21	13C12 PFHxA	13C4 PFOA

Table 71 Labelled Standards for PFHpA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2	yes	
3	13C4 PFOA	
4	13C4 PFHpA	
5	13C4-PFHpA	N/A
6	M4PFHpA	
7	PFHpA-13C4	PFOS-13C8
8	MPFHpA	NA
9		
10	13C4-PFHpA	
11	13C4 PFHpA	
12		
13	13C3-PFHpA	13C8-PFOA
14	PFOS-13C8	PFHpA-13C4
15	13C4-PFHpA	
16	13C4-PFHpA	13C4-PFOA
17	13C4-PFHpA	
18	x	
19	13C4-PFHpA	13C2-PFOA
20	13C4-PFHpA	13C8-PFOA
21	13C4 PFHpA	13C4 PFOA

Table 72 Labelled Standards for PFOA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2	yes	
3	13C4 PFOA	
4	13C8 PFOA	13C2 PFOA
5	13C4-PFOA	N/A
6	M8PFOA	
7	PFOA-13C4	PFOS-13C8
8	M8PFOA	NA
9		
10	13C8-PFOA	
11	13C4 PFOA	
12	yes	
13	13C4-PFOA	13C8-PFOA
14	PFOS-13C8	PFOA-13C4
15	13C8-PFOA	
16	13C8-PFOA	13C4-PFOA
17	13C4-PFOA	
18	x	
19	13C8-PFOA	13C2-PFOA
20	13C4-PFOA	13C8-PFOA
21	13C8 PFOA	13C4 PFOA

Table 73 Labelled Standards for PFNA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2	yes	
3	13C5 PFNA ISTD	
4	13C9 PFNA	
5	13C5-PFNA	N/A
6	M9PFNA	M5PFNA
7	PFNA-13C5	PFOS-13C8
8	M9PFNA	NA
9		
10	13C5-PFNA	
11	13C5 PFNA	
12		
13	13C5-PFNA	13C8-PFOA
14	PFOS-13C8	PFNA-13C5
15	13C9-PFNA	
16	13C9-PFNA	13C5-PFNA
17	13C5-PFNA	
18	x	
19	13C9-PFNA	13C4-PFOS
20	13C9-PFNA	13C5-PFNA
21	13C5 PFNA	13C4 PFOA

Table 74 Labelled Standards for PFDA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2	yes	
3	13C2 PFDA ISTD	
4	13C6 PFDA	13C2 PFDA
5	13C2-PFDA	N/A
6	M6PFDA	
7	PFDA-13C2	PFOS-13C8
8	M6PFDA	NA
9		
10	13C6-PFDA	
11	13C2 PFDA	
12		
13	13C2-PFDA	13C8-PFOA
14	PFOS-13C8	PFDA-13C2
15	13C6-PFDA	
16	13C6-PFDA	13C2-PFDA
17	13C6-PFDA	
18	x	
19	13C6-PFDA	13C2-PFDA
20	13C2-PFDA	13C5-PFNA
21	13C2 PFDA	13C4 PFOA

Table 75 Labelled Standards for PFUdA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2	yes	
3	13C2 PFUdA ISTD	
4	13C7 PFUnA	
5	13C2-PFUdA	N/A
6	M7PFUdA	
7	PFUnDA-13C2	PFOS-13C8
8	M7PFUnDA	NA
9		
10	13C2-PFUdA	
11	13C2 PFUnA	
12		
13	13C2-PFUdA	13C8-PFOA
14	PFOS-13C8	PFUNDA-13C2
15	13C7-PFUdA	
16	13C7-PFUdA	13C2-PFDA
17	13C2-PFUdA	
18	x	
19	13C7-PFUdA	13C2-PFDA
20	13C2-PFUdA	13C5-PFNA
21	13C2 PFUnA	13C4 PFOA

Table 76 Labelled Standards for PFOSA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2		
3	D5-EtFOSAA-M	
4	13C8 PFOSA	
5	13C8-FOSA	N/A
6	NT	
7	FOSA-13C8	PFOS-13C8
8	MPFOA	NA
9		
10	13C8-FOSA	
11	13C8 PFOSA	
12		
13	13C8-FOSA	none
14	PFOS-13C8	FOSA-13C8
15	13C8-FOSA	
16	13C8-PFOA	13C4-PFOS
17	13C8-FOSA	
18	x	
19	13C8-PFOA	13C2-PFDA
20	13C8-PFOA	13C2-PFTeDA
21	13C8 PFOSA	13C4 PFOA

Table 77 Labelled Standards for ADONA

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2		
3		
4	NT	
5	Not Tested	N/A
6	NT	
7	PFHxS-18O2	PFOS-13C8
8	NT	NA
9		
10		
11	13C4 PFOS	
12		
13	13C3-PFHpA	13C8-PFOA
14	PFOS-13C8	PFOA-13C4
15	13C8-PFOA	
16	13C3-HFPO-DA	13C2-PFHxA
17		
18		
19	13C4-PFHpA	13C2-PFOA
20	NT	NT
21	NT	NT

Table 78 Labelled Standards for GenX

Lab. Code	Before Extraction	Before Instrument Analysis
1	13C4-PFOA	
2		
3		
4	NT	
5	Not Tested	N/A
6		
7	HFPO-DA-13C3	PFOS-13C8
8	NT	NA
9		
10		
11	13C3 HFPO-DA	
12		
13	13C3-GenX	none
14	PFOS-13C8	PFHxS-18O2
15		
16	13C3-HFPO-DA	13C2-PFHxA
17		
18		
19	13C3-HFPODA	13C2-PFOA
20	NT	NT
21	NT	NT

## APPENDIX 3 – ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, Z-SCORE AND E<sub>n</sub>-SCORE CALCULATIONS

### A3.1 Robust Average and Associated Uncertainty

The robust average was calculated using the procedure described in ISO 13528:2015 Annex C.<sup>5</sup> The uncertainty was estimated as:

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

where:

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

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

$p$  is the number of results

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

A worked example is set out below in Table 79.

Table 79 Uncertainty Estimate for PFHxS (linear) in Sample S2

No. results (p)	13
Robust Average	5.73 µg/kg
$S_{rob\ average}$	0.61 µg/kg
$u_{rob\ average}$	0.21 µg/kg
$K$	2
$U_{rob\ average}$	0.42 µg/kg

Therefore, the robust average for PFHxS (linear) in Sample S2 is  $5.73 \pm 0.42$  µg/kg.

### A3.2 z-Score and E<sub>n</sub>-Score Calculations

For each participant's result, a z-score and E<sub>n</sub>-score are calculated according to Equations 2 and 3 respectively.

A worked example is set out below in Table 80.

Table 80 z-Score and E<sub>n</sub>-Score for Sample S1 PFBS Result Reported by Laboratory 2

Participant Result (µg/kg)	Assigned Value (µg/kg)	Target Standard Deviation	z-Score	E <sub>n</sub> -Score
0.55 ± 0.02	0.158 ± 0.044	20% as PCV, or: 0.2 × 0.158 = 0.0316 µg/kg	$z\text{-Score} = \frac{0.55 - 0.158}{0.0316}$ = 12.41	$E_n\text{-Score} = \frac{0.55 - 0.158}{\sqrt{0.02^2 + 0.044^2}}$ = 8.11

## APPENDIX 4 – ACRONYMS AND ABBREVIATIONS

4:2 FTS	4:2 Fluorotelomer sulfonic acid
6:2 FTS	6:2 Fluorotelomer sulfonic acid
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
EPA	Environment Protection Authority
EtFOSA	N-Ethyl perfluorooctane sulfonamide
EtFOSE	N-Ethyl perfluorooctane sulfonamidoethanol
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
LOD	Limit of Detection
LOQ	Limit of Quantification
Max	Maximum value in a set of results
Md	Median
MeFOSAA	N-Methyl perfluorooctane sulfonamidoacetic acid
MeFOSE	N-Methyl perfluorooctane sulfonamidoethanol
Min	Minimum value in a set of results
MS	Mass Spectrometry
MS/MS	Tandem Mass Spectrometry
MU	Measurement Uncertainty
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 polyfluorinated alkyl substances
PFBA	Perfluorobutanoic acid
PFBS	Perfluorobutanesulfonic acid
PFDA	Perfluorodecanoic acid
PFDS	Perfluorodecanesulfonic acid

PFHpA	Perfluoroheptanoic acid
PFHpS	Perfluoroheptanesulfonic acid
PFHxA	Perfluorohexanoic acid
PFHxS	Perfluorohexanesulfonic acid
PFNA	Perfluorononanoic acid
PFNS	Perfluorononanesulfonic acid
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctanesulfonic acid
PFOSA	Perfluorooctanesulfonamide
PFPeA	Perfluoropentanoic acid
PFPeS	Perfluoropentanesulfonic acid
PFTeDA	Perfluorotetradecanoic acid
PFTrDA	Perfluorotridecanoic acid
PFUdA	Perfluoroundecanoic acid
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
SPE	Solid Phase Extraction
SS	Spiked Samples

**END OF REPORT**