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Department of Industry,  
Science and Resources

National  
Measurement  
Institute

# Proficiency Test Final Report AQA 23-15 PFAS in Biota and Food

March 2024

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## TABLE OF CONTENTS

SUMMARY	1
1 INTRODUCTION	3
1.1 NMI Proficiency Testing Program	3
1.2 Study Aims	3
1.3 Study Conduct	3
2 STUDY INFORMATION	4
2.1 Study Timetable	4
2.2 Participation and Laboratory Code	4
2.3 Selection of PFAS Analytes and Test Material Preparation	4
2.4 Homogeneity and Stability of Test Materials	5
2.5 Test Material Storage and Dispatch	6
2.6 Instructions to Participants	6
2.7 Interim Report and Preliminary Report	7
3 PARTICIPANT LABORATORY INFORMATION	8
3.1 Participants' Test Methods	8
3.2 Basis of Participants' Measurement Uncertainty Estimates	8
3.3 Participants' Comments	10
4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS	12
4.1 Results Summary	12
4.2 Outliers and Extreme Outliers	12
4.3 Assigned Value	12
4.4 Robust Average and Robust Between-Laboratory Coefficient of Variation	12
4.5 Performance Coefficient of Variation (PCV)	12
4.6 Target Standard Deviation for Proficiency Assessment	13
4.7 z-Score	13
4.8 E <sub>n</sub> -Score	13
4.9 Traceability and Measurement Uncertainty	13
5 TABLES AND FIGURES	14
6 DISCUSSION OF RESULTS	174
6.1 Assigned Value	174
6.2 Measurement Uncertainty Reported by Participants	176
6.3 z-Score	177
6.4 E <sub>n</sub> -Score	186
6.5 Range of PFAS Analysed by Participants	187
6.6 PFAS in Food Trigger Points	189
6.7 False Negatives	195
6.8 Reporting of Additional Analytes	196
6.9 Participants' Methods	197
6.10 Total vs Linear Isomers – PFHxS and PFOS	204
6.11 Effects of Sample Matrix	207

6.12	Summary of Participants' Results and Performances	208
6.13	Comparison with Previous PFAS in Biota and Food Studies	215
7	REFERENCES	218
APPENDIX 1	SAMPLE PREPARATION	219
APPENDIX 2	HOMOGENEITY AND STABILITY ASSESSMENT	220
A2.1	Sample S1 Fish Homogeneity and Stability Assessment	220
A2.2	Sample S2 Spinach Homogeneity and Stability Assessment	228
A2.3	Sample S3 Bovine Liver Homogeneity and Stability Assessment	242
APPENDIX 3	ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, z-SCORE AND E <sub>n</sub> -SCORE CALCULATIONS	257
A3.1	Robust Average and Associated Uncertainty	257
A3.2	z-Score and E <sub>n</sub> -Score Calculations	257
APPENDIX 4	PARTICIPANTS' TEST METHODS	258
APPENDIX 5	ACRONYMS AND ABBREVIATIONS	317

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## SUMMARY

AQA 23-15 PFAS in Biota and Food commenced in July 2023. Twenty-two laboratories registered to participate. Twenty-one participants submitted results by the due date.

The sample set consisted of a spiked fish sample (Sample S1), a spiked spinach sample (Sample S2) and a spiked bovine liver sample (Sample S3). The per- and polyfluoroalkyl substances (PFAS) analytes assessed in this study were: PFBS, PFPeS, PFHxS, PFHpS, PFOS, PFNS, PFDS, PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUdA, PFDoA, PFTTrDA, PFTeDA, PFOSA, EtFOSA, EtFOSAA, 6:2FTS, 8:2FTS, GenX, ADONA, 9Cl-PF3ONS and 11Cl-PF3OUdS.

Of 1465 possible results, 1168 (80%) were numeric results. Fifty-seven were a 'less than' value ( $< x$ ) or Not Reported (NR), and 240 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.*

Of the participants who returned results, 14 participants analysed all three matrices, six participants analysed fish and spinach only, and one participant analysed fish only.

Laboratory **21** reported numeric results for all 79 scored analytes across all matrices.

Seven participants did not report results for analytes that they tested for and were spiked into the samples (total of 17 results), while five participants reported analytes that were not spiked into the samples (total of 12 results).

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

Of 1161  $z$ -scores, 1091 (94%) returned  $|z| \leq 2.0$ , indicating an acceptable performance.

Of 1159  $E_n$ -scores, 959 (83%) returned  $|E_n| \leq 1.0$ , indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Laboratory **21** returned acceptable  $z$ -scores for all 79 scored analytes, and all their  $E_n$ -scores (78 results assessed) were also acceptable.

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

Participants used a wide variety of methods for extraction and analysis. Most participants did not base their methodology on a standard method.

The most popular for this study was alkaline digestion with basified methanol and solid-phase extraction clean-up, and then analysis using LC-MS/MS or LC-QQQ.

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

Of 1168 numeric results reported for analytes of interest in this study, 1153 (99%) were reported with an associated expanded measurement uncertainty.

Although it has been a requirement of ISO/IEC 17025 that laboratories have procedures to estimate the uncertainty, a large number of laboratories are still reporting potentially unrealistically small or large relative uncertainties for routine PFAS. The magnitude of the

reported measurement uncertainties for spiked analytes in this study was within the range 0.06% to 100% of the reported value.

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

NMI has been conducting PFAS in biota and food PT studies since 2016.

The proportion of total possible results being reported by participants as numeric results has remained fairly consistent, even with the significantly increased number of PFAS analytes over the last few studies, indicating that participants have the capacity to analyse a wide range of PFAS, including novel analytes, at relevant mass fractions.

This study returned the highest proportion of acceptable  $z$ -scores, and the second highest proportion of acceptable  $E_n$ -scores, across all NMI PFAS in biota and food PT studies.

- *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 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 soil, water, fruit, vegetables and herbs;
- petroleum hydrocarbons in soil and water;
- per- and polyfluoroalkyl substances (PFAS) in soil, biosolid, water, biota and food;
- inorganic analytes in soil, water, filters, food and pharmaceuticals;
- controlled drug assay, drugs in wipes and clandestine laboratory; and
- allergens in food.

### **1.2 Study 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 participants' test methods for PFAS in biota and food analysis;
- develop the practical application of traceability and measurement uncertainty;
- compare the performance of participants with their past performance; and
- produce materials that can be used in method validation and as control samples.

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

### **1.3 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. This study falls within the scope of NMI's accreditation.

## 2 STUDY INFORMATION

### 2.1 Study Timetable

The timetable of the study was:

Invitations sent	10/07/2023
Samples sent	4/09/2023
Results due	10/11/2023
Interim Report	14/11/2023
Preliminary Report	1/12/2023

The results due date was extended to accommodate sample delivery delays to an international participant.

### 2.2 Participation and Laboratory Code

Twenty-two laboratories registered to participate in this study. All participants were assigned a confidential laboratory code number for this study. Twenty-one participants submitted results by the results due date.

### 2.3 Selection of PFAS Analytes and Test Material Preparation

Participants were provided with a list of potential PFAS analytes that were spiked into the study's samples, as presented in Table 1.

Table 1 Potential Spiked PFAS Analytes

PFBS	PFBA	PFTeDA	8:2FTS
PFPeS	PFPeA	PFODA	10:2FTS
PFHxS	PFHxA	PFOSA	8:2diPAP
PFHpS	PFHpA	MeFOSA	GenX
PFOS	PFOA	EtFOSA	ADONA
PFNS	PFNA	MeFOSAA	9Cl-PF3ONS
PFDS	PFDA	EtFOSAA	11Cl-PF3OUdS
PFUdS	PFUdA	MeFOSE	3:3FTCA
PFDoS	PFDoA	EtFOSE	5:3FTCA
PFTTrDS	PFTTrDA	6:2FTS	7:3FTCA

Three samples were prepared in July and August 2023. Care was taken to avoid any PFAS contamination during sample preparation. The prepared samples were:

- Sample S1: Fish paste (5 g portions) spiked with 25 different PFAS analytes.
- Sample S2: Spinach puree (30 g portions) spiked with 24 different PFAS analytes.
- Sample S3: Bovine liver paste (5 g portions) spiked with 25 different PFAS analytes.

Details of spiked analytes and values are presented in Table 2. Further sample preparation details can be found in Appendix 1.

Table 2 Spiked Values of Test Samples

Analyte	Sample S1 Fish (µg/kg)	Sample S2 Spinach (µg/kg)	Sample S3 Bovine Liver (µg/kg)
PFBS	1.41	4.62	1.46
PFPeS	3.08	3.47	3.22
PFHxS*	6.24	2.20	6.50
PFHpS	1.34	1.76	1.41
PFOS*	1.79	2.50	1.87
PFNS	1.80	3.55	1.88
PFDS	1.80	2.68	1.89
PFBA	6.57	10.7	6.77
PFPeA	2.39	4.60	2.44
PFHxA	6.54	4.61	6.89
PFHpA	7.48	2.77	7.84
PFOA	0.943	5.57	0.951
PFNA	1.89	4.64	1.96
PFDA	1.59	2.09	1.64
PFUdA	0.756	3.73	0.781
PFDoA	3.70	Not Spiked	3.92
PFTrDA	Not Spiked	2.78	Not Spiked
PFTeDA	1.68	Not Spiked	1.76
PFOSA	5.39	12.3	5.58
EtFOSA	4.67	7.46	4.89
EtFOSAA	4.67	9.26	4.89
6:2FTS	Not Spiked	4.39	Not Spiked
8:2FTS	9.00	Not Spiked	9.39
GenX	14.1	8.40	14.7
ADONA	17.6	15.8	18.4
9Cl-PF3ONS	21.8	19.0	22.8
11Cl-PF3OUdS	26.4	22.6	27.7

\* Participants were requested to report both linear isomers only and total value. The samples in this study were spiked with linear standards only for these analytes.

## 2.4 Homogeneity and Stability of Test Materials

The process used to prepare, store and dispatch Samples S1 and S2 has been demonstrated in previous NMI PFAS in biota and food PT studies to produce sufficiently homogeneous and stable samples.

Furthermore, homogeneity testing was conducted for all samples in this PT study, and stability testing was conducted for Samples S2 and S3. The samples were demonstrated to be sufficiently homogeneous and stable for the evaluation of participants' performance in this study.

The stability of the samples was also assessed by comparing the results returned by participants with the spiked values. Assigned values for scored analytes were within 61% to 100%, 87% to 115% and 75% to 105% of the spiked values for Samples S1, S2 and S3 respectively. These values are similar to those observed in previous NMI PFAS in biota and food PT studies, and provides support for the stability of these analytes.

Further details on the homogeneity and stability assessment are given in Appendix 2.

## **2.5 Test Material Storage and Dispatch**

After preparation, the test materials were dispensed into sample tubes, labelled and shrink-wrapped. Prior to sample dispatch, Samples S1 and S3 were stored at -80 °C, and Sample S2 was stored at -20 °C.

Samples were packed into insulated polystyrene foam boxes with cooler bricks and sent by courier on 4 September 2023.

The following items were packaged with the samples:

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

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

## **2.6 Instructions to Participants**

Participants were instructed as follows:

- Quantitatively analyse the samples for PFAS, using your routine test method.
  - For PFAS analytes that contain linear and branched isomers, report total (the sum of linear and branched isomers).
  - For PFOS and PFHxS you are asked to report total (the sum of linear and branched isomers) and linear (the linear isomers only).
- Report results using the electronic results sheet emailed to you.
- For each analyte report a single result expressed as if reporting to a client (i.e. corrected for recovery or not, according to your standard procedure, but state if results are corrected on the result sheet). This figure will be used in all statistical analysis in the study report. Report results in units of  $\mu\text{g}/\text{kg}$  on an as received basis for all samples.
- For each analyte report the associated expanded measurement uncertainty as  $\mu\text{g}/\text{kg}$  (e.g.  $0.50 \pm 0.02 \mu\text{g}/\text{kg}$ ), if determined.
- No limit of reporting has been set for this study. Report results as you would to a client, applying the limit of reporting of the method used for analysis.
- Report any listed analyte not tested as NT.
- Please complete the method details and report the basis of your uncertainty estimates as required by the results sheet.
- If determined, report your internal standard percentage recovery. This will be presented in the report for information only.
- Return the completed results sheet by email ([proficiency@measurement.gov.au](mailto:proficiency@measurement.gov.au)) by 6 October 2023.

Due to significant sample delivery delays to an international participant caused by customs clearance issues, the results due date was extended to 10 November 2023.

## 2.7 Interim Report and Preliminary Report

An Interim Report was emailed to all participants on 14 November 2023.

A Preliminary Report was emailed to all participants on 1 December 2023. This report included a summary of the results reported by participants, assigned values, performance coefficients of variation (PCVs),  $z$ -scores and  $E_n$ -scores for each analyte in this study.

No data from the Preliminary Report has been changed in the present Final Report with the exception of the spiked values for PFOSA in Samples S1, S2 and S3 (Table 3), and subsequently the assigned value and scoring of results for this analyte.

A high ratio of participant results' robust averages to the spiked values was noticed for PFOSA across all three samples in this study (166% for Sample S1, 188% for Sample S2 and 178% for Sample S3). Similar ratios were observed in the PT study prepared at the same time as AQA 23-15, AQA 23-14 PFAS in Soil and Water. An investigation was conducted and identified problems with the standard used for spiking. The spiked values have been adjusted accordingly. Participants' results reported for this study in all samples were in excellent agreement with each other and with the adjusted spiked value. An assigned value was set for this analyte and results were scored across all samples.

Table 3 PFOSA Expected Spiked Values and Adjusted Spiked Values

Sample	Analyte	Expected Spike Value ( $\mu\text{g}/\text{kg}$ )	Adjusted Spike Value ( $\mu\text{g}/\text{kg}$ )
S1	PFOSA	$2.82 \pm 0.14$	$5.39 \pm 0.27$
S2	PFOSA	$6.43 \pm 0.32$	$12.3 \pm 0.6$
S3	PFOSA	$2.92 \pm 0.15$	$5.58 \pm 0.28$

### 3 PARTICIPANT LABORATORY INFORMATION

#### 3.1 Participants' Test Methods

Participants were requested to provide information about their methodology. Responses are presented in Appendix 4.

#### 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 4 and 5. Responses may be modified so that the participant cannot be identified.

Table 4 Basis of Participants' Uncertainty Estimate

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
1	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS Duplicate analysis	CRM Instrument calibration Recoveries of SS Standard purity	NATA GAG Estimating and Reporting MU
2	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS		Statistics and Chemometrics for Analytical Chemistry, Miller and Miller, 5th Edition
3	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
4		Duplicate analysis Instrument calibration	Instrument calibration	
5	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)	Control samples - SS Duplicate analysis	Recoveries of SS Standard purity	ISO/GUM
6	Top Down - precision and estimates of the method and laboratory bias	Control samples Duplicate analysis Instrument calibration	Instrument calibration Laboratory bias from PT studies	
7	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	
8	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS	Recoveries of SS	ISO/GUM
9	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS Standard purity	ISO/GUM
10	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM		Eurachem/CITAC Guide

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
11	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis Instrument calibration	Recoveries of SS	NEN7779
12	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)	Duplicate analysis Instrument calibration	Instrument calibration Standard purity	ISO/GUM
13	Standard deviation of replicate analyses multiplied by 2 or 3	Standard deviation from PT studies only		ISO/GUM
		Duplicate analysis Instrument calibration	CRM Recoveries of SS	
14		Duplicate analysis	CRM Laboratory bias from PT studies Recoveries of SS	Eurachem/CITAC Guide
15				
16	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS	Laboratory bias from PT studies Recoveries of SS	ISO/GUM
17	standard deviation of triplicate measurements	Standard deviation from PT studies only		
19	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS	Recoveries of SS	USEPA SW-846
20	Top Down - precision and estimates of the method and laboratory bias	Control samples - RM Duplicate analysis	CRM Laboratory bias from PT studies Recoveries of SS	Nordtest Report TR537
21	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
22	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples - SS		ISO/GUM

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

Table 5 Uncertainty Estimate Additional Comments

Lab. Code	Uncertainty Estimate Comments
1	Recovery and uncertainty data given for analytes at method limit of reporting.
2	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
7	Uncertainty calculated as 3xSD of replicate analysis.

Lab. Code	Uncertainty Estimate Comments
14	<p>MU (k = 2) = 2 x %RSDR</p> <p>Between laboratory reproducibility (%RSDR) cannot be evaluated and will be estimated as:            %RSDR = %RSDWLR x 1.5 or %RSDr x 2 where</p> <ul style="list-style-type: none"> <li>• The estimate of repeatability (%RSDr) is obtained from spiked samples analysed in the same batch</li> <li>• The estimate of within-laboratory reproducibility (%RSRWLR) is obtained from proficiency test samples analysed on different batches</li> </ul> <p>The measurement of uncertainty for all validated analytes in fish/meat is 35%, so in lieu of accurate data for spinach this has been applied in this case.</p>
19	Standard practice for laboratories utilizing US EPA's SW-846 document.
22	The expanded measurement uncertainty values were calculated using a coverage factor (K) value of 2.00 and at the 95% confidence limit.

### 3.3 Participants' Comments

Participants were invited to make comments on the samples, this PT study, or suggestions for future studies. Such feedback may be useful in improving future studies. Participants' comments are presented in Table 6, along with the study coordinator's response where applicable. Responses may be modified so that the participant cannot be identified.

Table 6 Participants' Comments

Lab. Code	Sample	Participant's Comments	Study Coordinator's Response
2	S1	PFTrDA is not reported (NR) because of poor recovery of our QC sample and 10:2 FTS is not reported (NR) because of high recovery of our QC sample.	
	S3	N-EtFOSAA and NMeFOSAA are not reported (NR) because of a high internal standard recovery. N-EtFOSA and NMeFOSA, N-EtFOSE and NMeFOSE are not reported (NR) because of poor recovery of our QC sample	
5	All	Due to high concentration of this study, 1g of sample is used instead of method 10g. Suggestion: Send more sample weight for future studies (especially for fish sample)	The amount per sample has been selected to balance the preparation method requirements while allowing participants to perform some replicates. For this study, most participants used around 1 g or less per analysis for Sample S1 Fish. Participants can also order additional samples at time of enrolment in the PT study, if larger amounts of sample are required for their analyses.
11	S1	Only linear measured PFUnDa 0.69±0.35 µg/kg PFDoDA 3.06±1.53 µg/kg	
	S2	Only linear measured PFUnDA 3.43±1.72 µg/kg	



Lab. Code	Sample	Participant's Comments	Study Coordinator's Response
14	All	ISO/IEC 17025 accreditation by INAB for fish and liver matrices. Method has not been validated for spinach. PFBS/PFDS not currently validated but results included.	
22	S1 and S2	Sample was received at a temperature of 10°C which was above the method recommended sample storage temperature (less than or equal to 6°C).	<p>For Sample S1 fish, stability testing conducted in a previous PFAS PT study showed that similar analytes in fish were sufficiently stable for at least a month at ambient temperatures.<sup>5</sup></p> <p>For Sample S2, stability testing was conducted in this study, and analytes were found to also be sufficiently stable for at least a month at ambient temperatures (Appendix 2).</p>

## 4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

### 4.1 Results Summary

Participant results are presented in Tables 7 to 86 with 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), as well as other estimates of analyte mass fraction. Bar charts of results and performance scores are presented in Figures 2 to 81. An example chart with interpretation guide is shown in Figure 1.

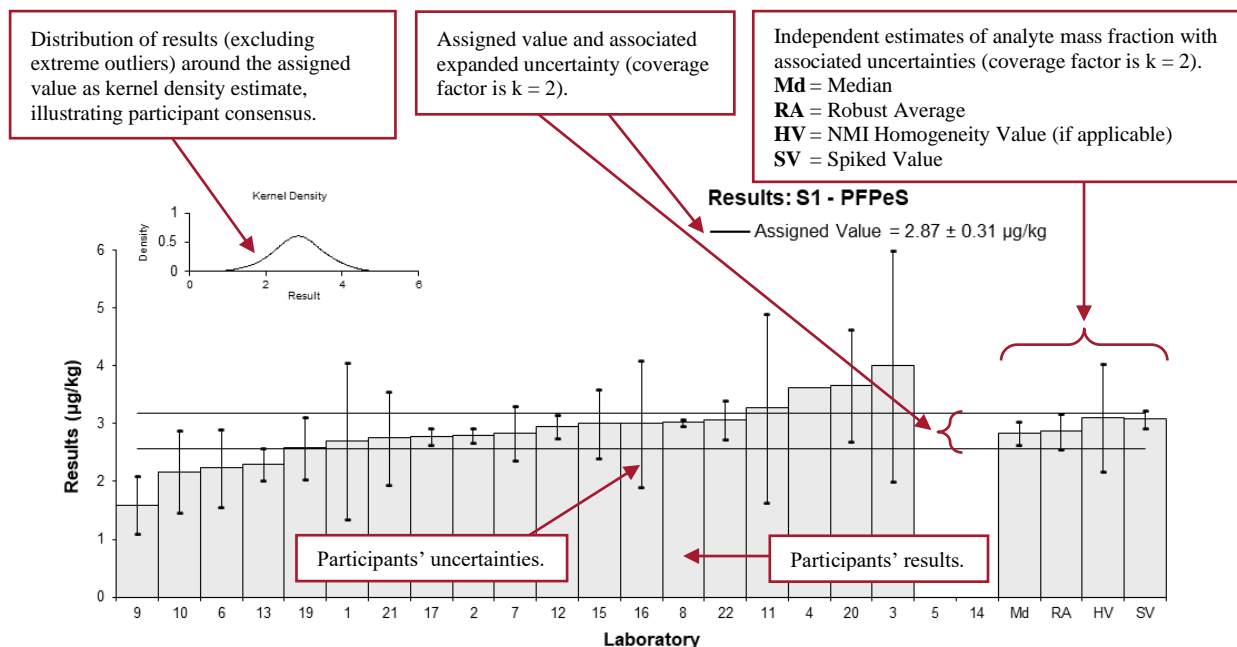


Figure 1 Guide to Presentation of Results

### 4.2 Outliers and Extreme Outliers

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

### 4.3 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 were the robust averages of participants’ results, and the expanded uncertainties were estimated from the associated robust SDs (Appendix 3).

### 4.4 Robust Average and Robust Between-Laboratory Coefficient of Variation

The robust averages and expanded MUs, and robust CVs (a measure of the variability of participants’ results) were calculated using the procedure described in ISO 13528.<sup>6</sup>

### 4.5 Performance Coefficient of Variation (PCV)

The PCV is a fixed measure of the between-laboratory variation that in the judgement of the study coordinator would be expected from participants given the levels of analytes present. The PCV is not the CV of participants’ results; it is set by the study coordinator and is based on the mass fraction of the analytes and experience from previous studies, and is supported by mathematical models such as the Thompson-Horwitz equation.<sup>7</sup> By setting a fixed and realistic value for the PCV, a participant’s performance does not depend on the performance of other participants and can be compared from study to study.

#### 4.6 Target Standard Deviation for Proficiency Assessment

The target standard deviation for proficiency assessment ( $\sigma$ ) is the product of the assigned value ( $X$ ) and the PCV, as presented in Equation 1.

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

#### 4.7 z-Score

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

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

where:

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

For the absolute value of a z-score:

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

To account for potential low bias in consensus values due to inefficient methodologies, z-scores may be adjusted for a 'maximum acceptable result' (see also Section 6.3).

#### 4.8 $E_n$ -Score

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

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

where:

- $E_n$  is  $E_n$ -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 the absolute value of an  $E_n$ -score:

- $|E_n| \leq 1.0$  is acceptable;
- $|E_n| > 1.0$  is unacceptable.

#### 4.9 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC 17025 must establish and demonstrate the traceability and measurement uncertainty associated with their test results.<sup>8</sup>

Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.<sup>9</sup>

## 5 TABLES AND FIGURES

Table 7

### Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFBS
<b>Unit</b>	µg/kg

### Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	1.1	0.55	106	-0.13	-0.05
2	1.3	0.021	101	0.75	1.52
3	1	1	109	-0.58	-0.13
4	1.6804	NR	NR	2.00▼	
5	NT	NT	NT		
6	0.932	0.28	86	-0.88	-0.66
7	1.28	0.09	95	0.66	1.06
8	1.20	0.056	107.7	0.31	0.57
9	0.74	0.22	107	-1.73	-1.59
10	0.948	0.32	72.88	-0.81	-0.54
11	1.19	0.595	NR	0.27	0.10
12	1.54	0.11	NR	1.81	2.64
13	1.1	0.25	NR	-0.13	-0.11
14	1.0878	0.3807	67	-0.19	-0.11
15	< 1	NR	NR		
16	1.1	0.4	NR	-0.13	-0.07
17	1.191	0.039	94	0.27	0.52
19	0.968	0.274	94	-0.72	-0.55
20	1.36	0.304	91.3	1.02	0.71
21	1.06	0.3	83	-0.31	-0.22
22	1.02	0.143	93.9	-0.49	-0.61

▼ Adjusted Score, see Section 6.3

### Statistics

<b>Assigned Value</b>	1.13	0.11
<b>Spike Value</b>	1.41	0.07
<b>Homogeneity Value</b>	1.05	0.32
<b>Robust Average</b>	1.13	0.11
<b>Max Acceptable Result</b>	1.98	
<b>Median</b>	1.10	0.09
<b>Mean</b>	1.15	
<b>N</b>	19	
<b>Max</b>	1.6804	
<b>Min</b>	0.74	
<b>Robust SD</b>	0.19	
<b>Robust CV</b>	17%	

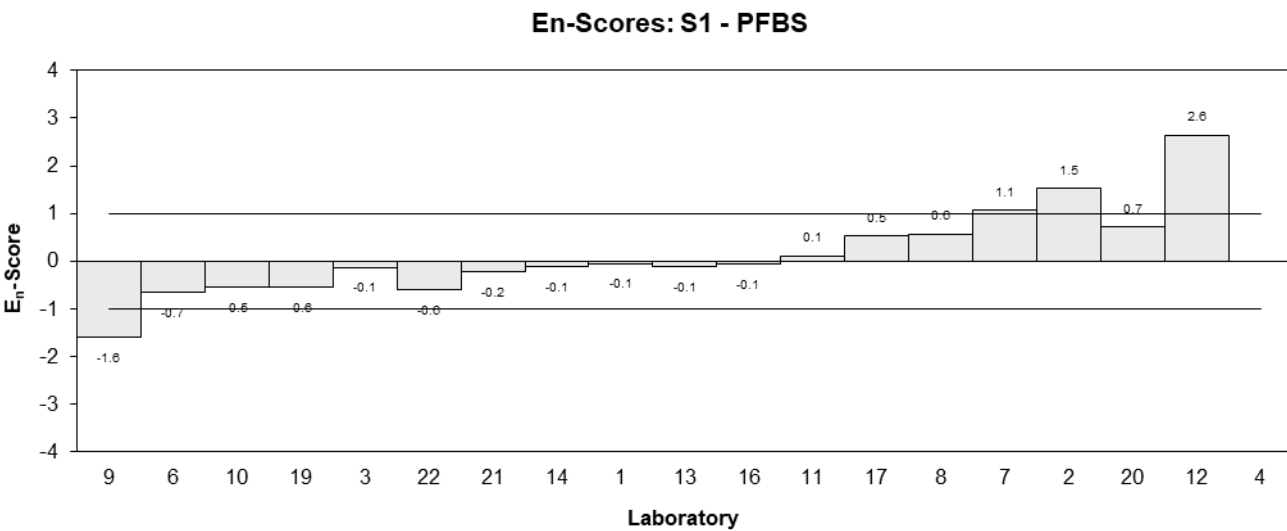
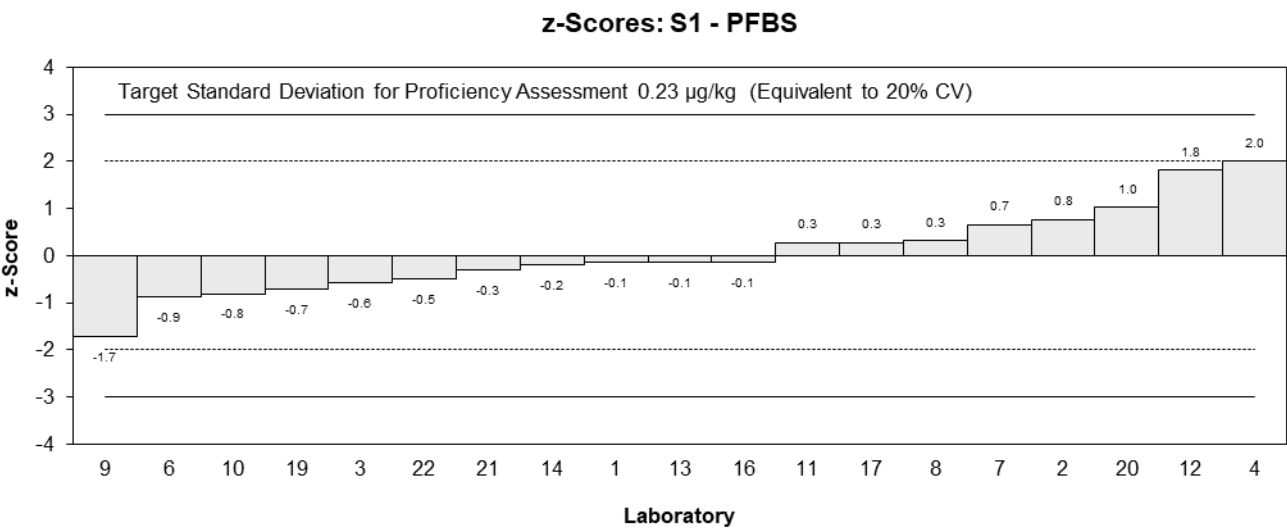
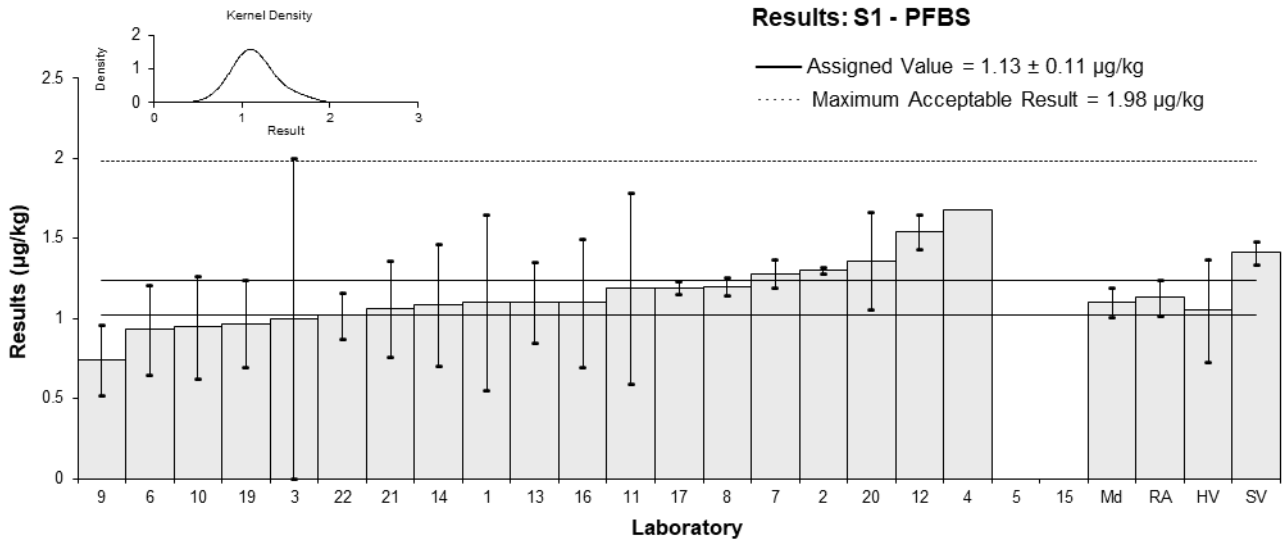


Figure 2

Table 8

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFPeS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	2.7	1.35	NR	-0.30	-0.12
2	2.8	0.13	116	-0.12	-0.21
3	4	2	113	1.97	0.56
4	3.616	NR	NR	1.30	2.41
5	NT	NT	NT		
6	2.237	0.671	84	-1.10	-0.86
7	2.83	0.47	95	-0.07	-0.07
8	3.02	0.062	NR	0.26	0.47
9	1.59	0.5	107	-2.23	-2.18
10	2.172	0.72	78.42	-1.22	-0.89
11	3.27	1.635	NR	0.70	0.24
12	2.95	0.21	NR	0.14	0.21
13	2.3	0.28	NR	-0.99	-1.36
14	NT	NT	NT		
15	3.0	0.6	NR	0.23	0.19
16	3.0	1.1	NR	0.23	0.11
17	2.778	0.145	92	-0.16	-0.27
19	2.58	0.536	94	-0.51	-0.47
20	3.66	0.967	91.3	1.38	0.78
21	2.75	0.8	NR	-0.21	-0.14
22	3.06	0.336	93.1	0.33	0.42

## Statistics

<b>Assigned Value</b>	2.87	0.31
<b>Spike Value</b>	3.08	0.15
<b>Homogeneity Value</b>	3.11	0.93
<b>Robust Average</b>	2.87	0.31
<b>Median</b>	2.83	0.20
<b>Mean</b>	2.86	
<b>N</b>	19	
<b>Max</b>	4	
<b>Min</b>	1.59	
<b>Robust SD</b>	0.54	
<b>Robust CV</b>	19%	

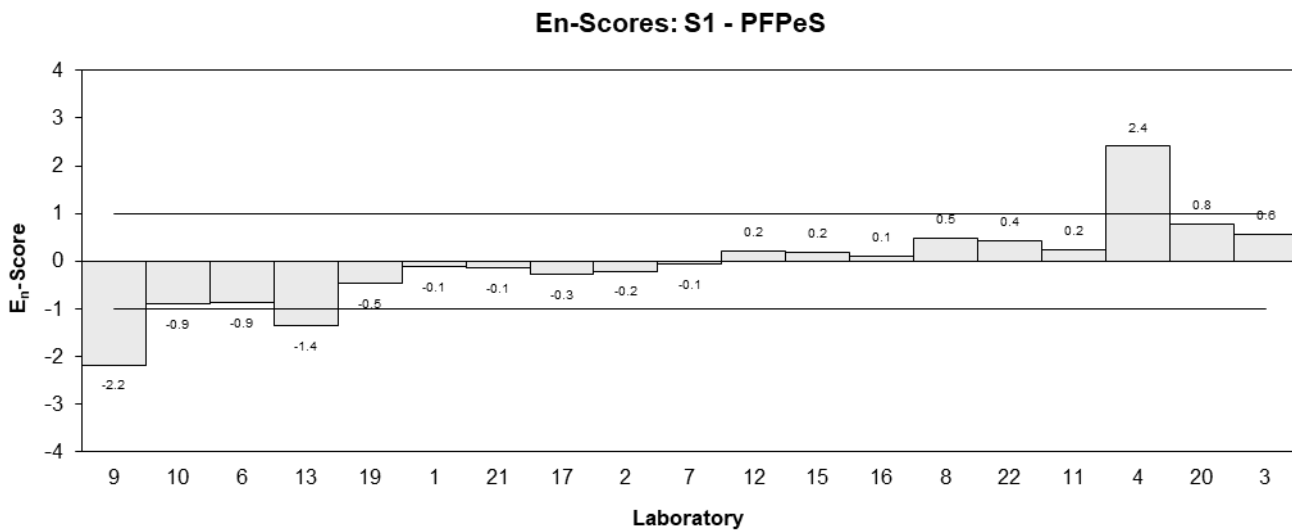
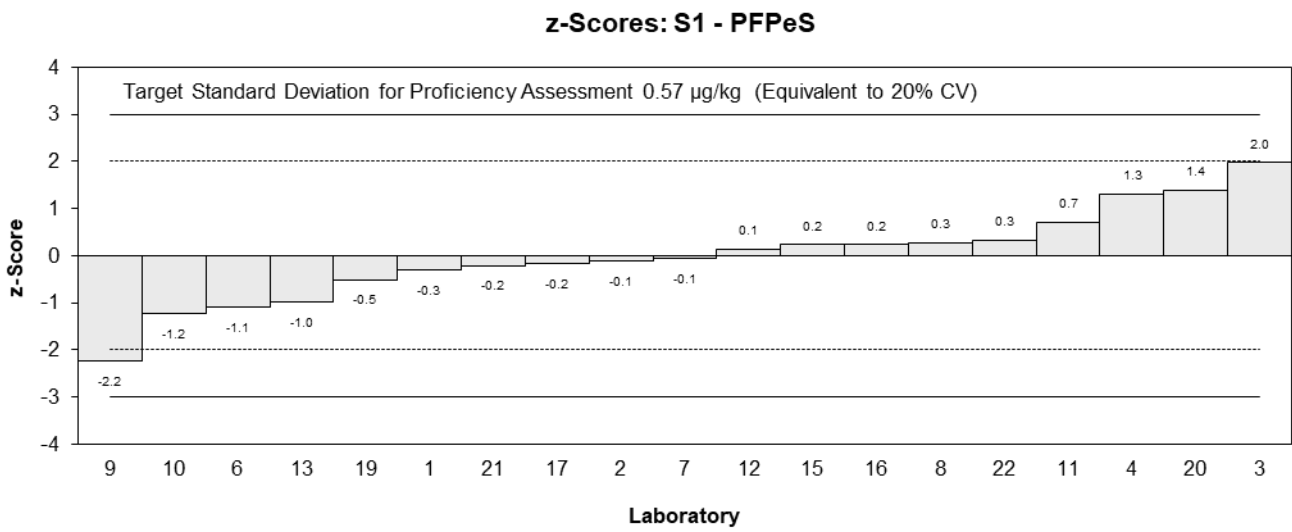
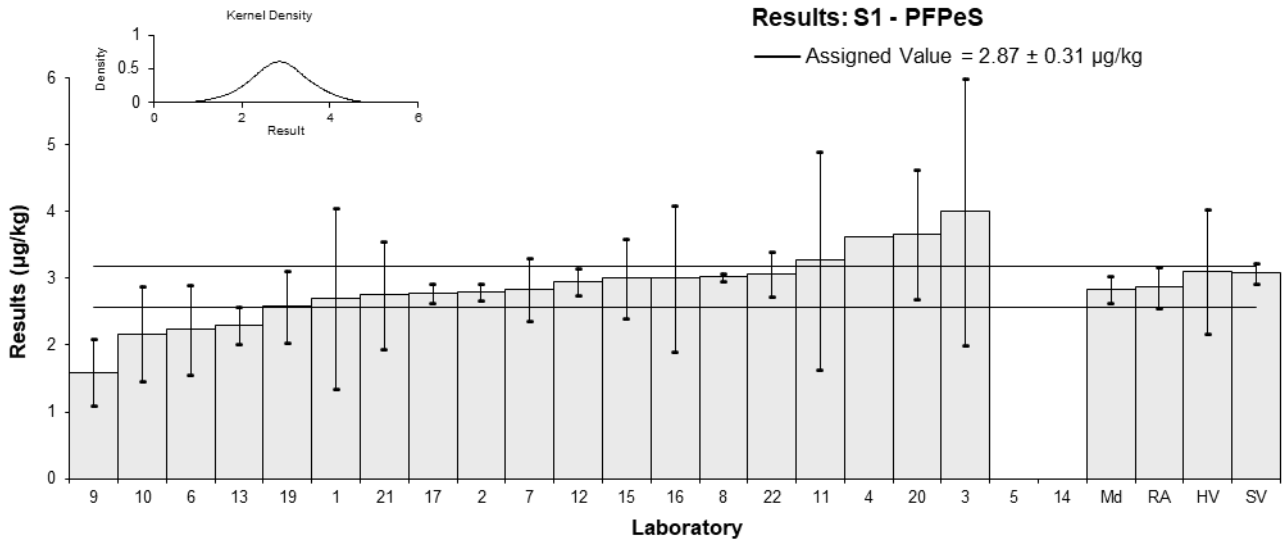


Figure 3

Table 9

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFHxS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	5.6	2.8	108	-0.04	-0.02
2	6.5	0.17	101	0.75	1.41
3	8	3	113	2.08	0.77
4	NT	NT	NT		
5	5.65	0.069	NT	0.00	0.00
6	4.723	1.417	91	-0.82	-0.61
7	6.37	1.08	NR	0.64	0.59
8	5.74	0.068	NR	0.08	0.15
9	4.52	1.35	104	-1.00	-0.77
10	4.562	1.59	78.42	-0.96	-0.64
11	NT	NT	NT		
12	6.8	0.48	NR	1.02	1.53
13	5.0	0.35	NR	-0.58	-0.96
14	5.7392	2.0087	84	0.08	0.04
15	4.6	0.92	NR	-0.93	-0.97
16	7.7	2.7	NR	1.81	0.74
17	5.563	0.241	92	-0.08	-0.14
19	5.09	0.881	93	-0.50	-0.53
20	NT	NT	NT		
21	5.99	2	80	0.30	0.16
22	4.97	0.695	93.1	-0.60	-0.75

## Statistics

<b>Assigned Value</b>	5.65	0.58
<b>Spike Value</b>	6.24	0.31
<b>Homogeneity Value</b>	5.4	1.6
<b>Robust Average</b>	5.65	0.58
<b>Median</b>	5.63	0.61
<b>Mean</b>	5.73	
<b>N</b>	18	
<b>Max</b>	8	
<b>Min</b>	4.52	
<b>Robust SD</b>	0.98	
<b>Robust CV</b>	17%	



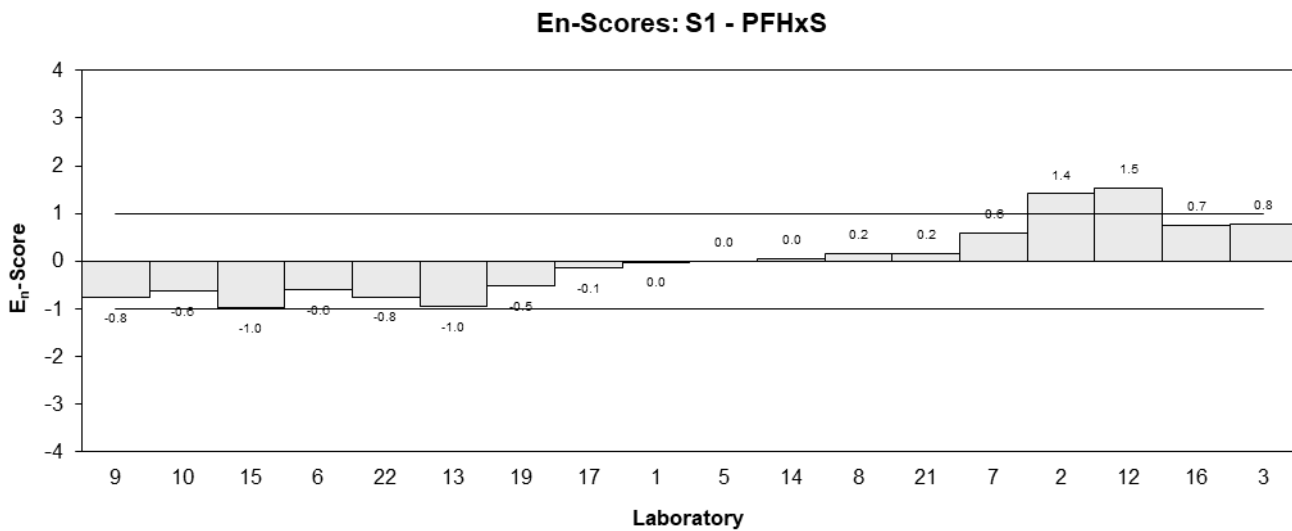
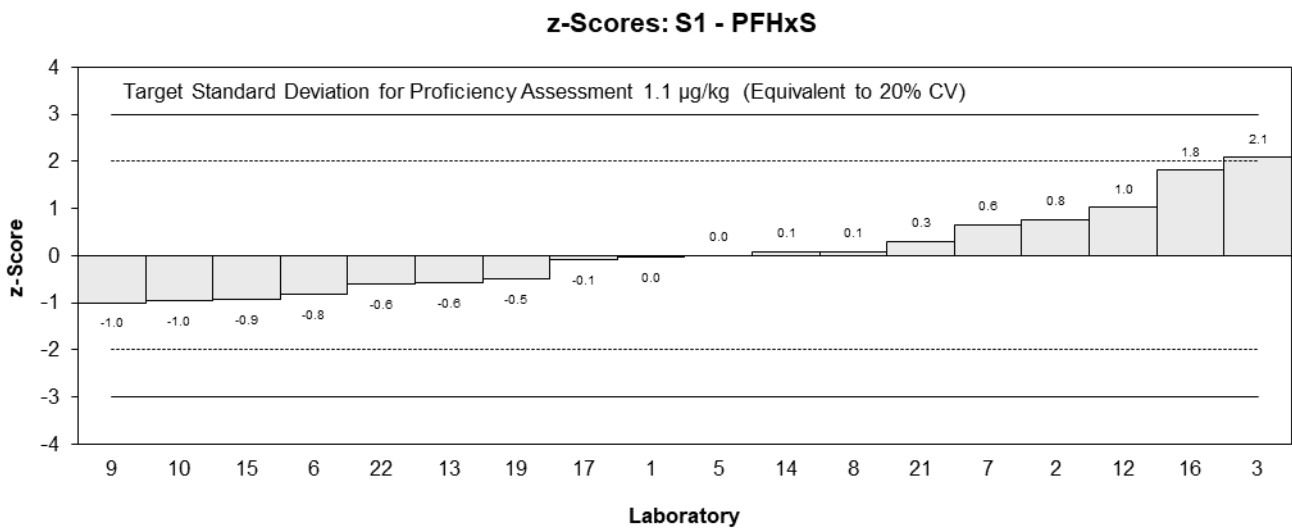
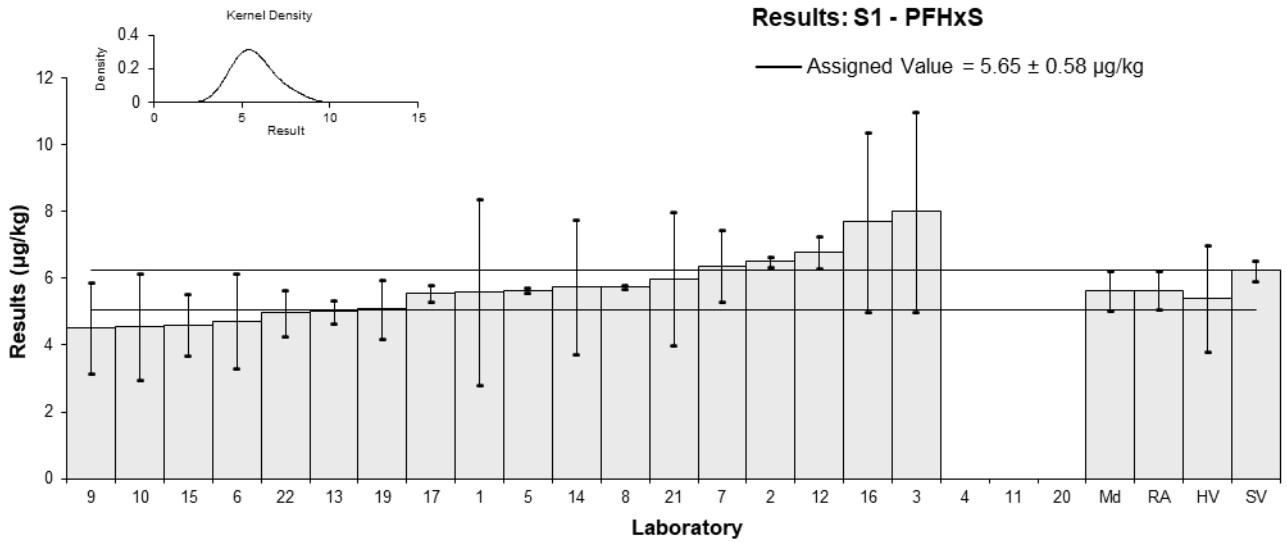


Figure 4

Table 10

## Sample Details

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

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	5.6	2.8	NR	-0.31	-0.13
2	6.5	0.17	101	0.44	0.72
3	8	3	113	1.70	0.66
4	8.2013	NR	NR	1.87	3.10
5	NT	NT	NT		
6	4.723	1.417	91	-1.04	-0.78
7	6.37	1.08	95	0.34	0.31
8	5.74	0.068	108.9	-0.19	-0.32
9	4.52	1.35	104	-1.21	-0.95
10	NT	NT	NT		
11	6.52	3.26	NR	0.46	0.16
12	6.8	0.48	NR	0.70	0.96
13	4.7	0.33	NR	-1.06	-1.60
14	5.7392	2.0087	84	-0.19	-0.11
15	NT	NT	NT		
16	NT	NT	NT		
17	NR	NR	NR		
19	5.09	0.021	93	-0.74	-1.22
20	6.88	2.15	91.3	0.76	0.40
21	5.99	2	NR	0.02	0.01
22	4.97	0.695	93.1	-0.84	-1.00

## Statistics

<b>Assigned Value</b>	5.97	0.72
<b>Spike Value</b>	6.24	0.31
<b>Homogeneity Value</b>	5.4	1.6
<b>Robust Average</b>	5.97	0.72
<b>Median</b>	5.87	0.77
<b>Mean</b>	6.02	
<b>N</b>	16	
<b>Max</b>	8.2013	
<b>Min</b>	4.52	
<b>Robust SD</b>	1.2	
<b>Robust CV</b>	19%	

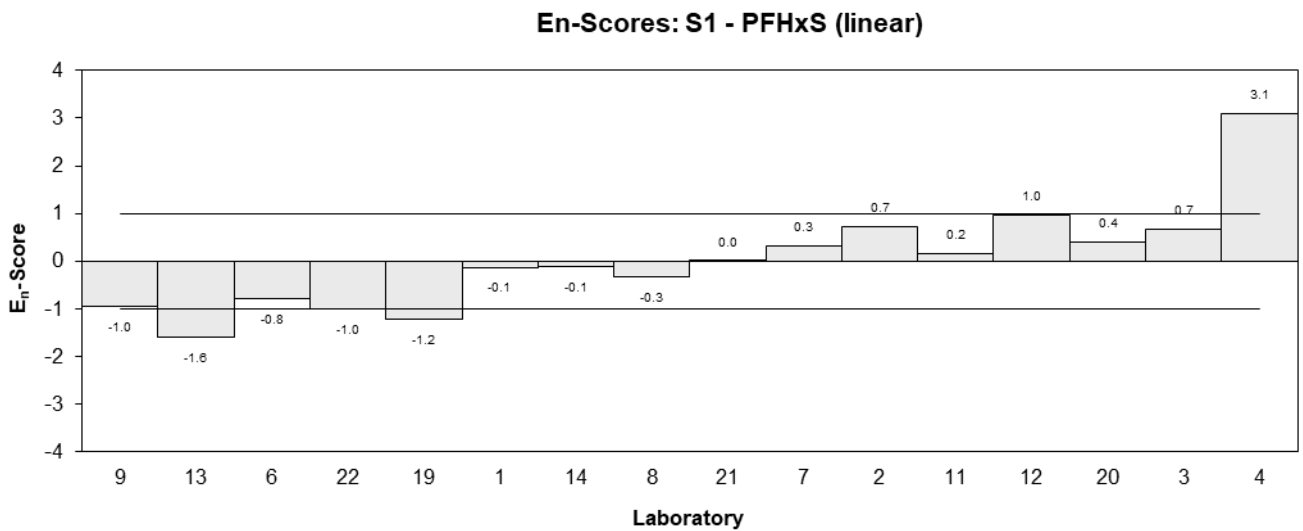
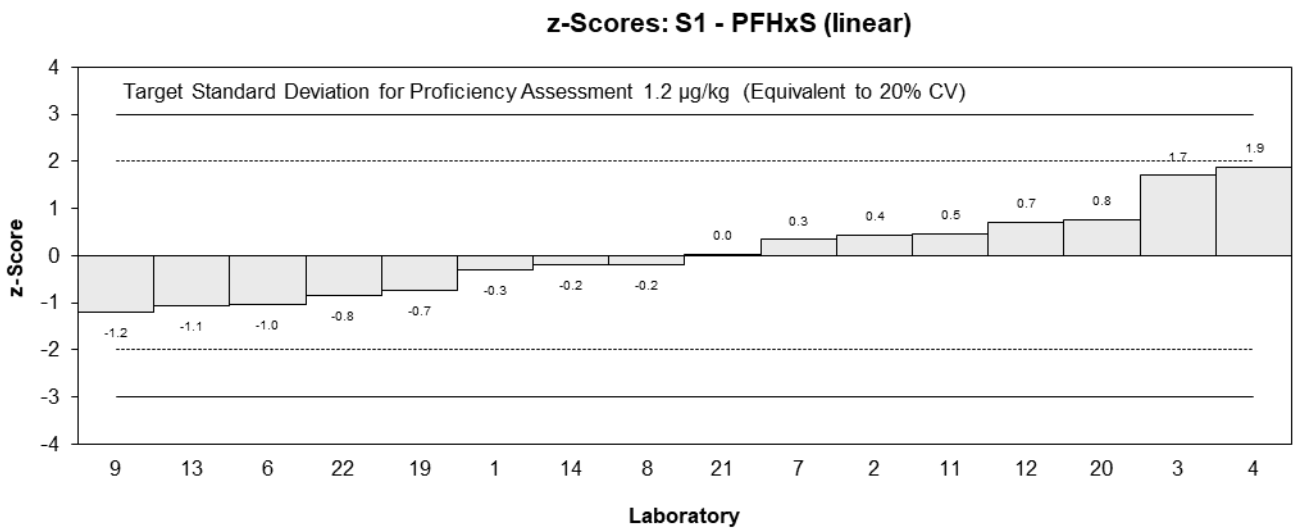
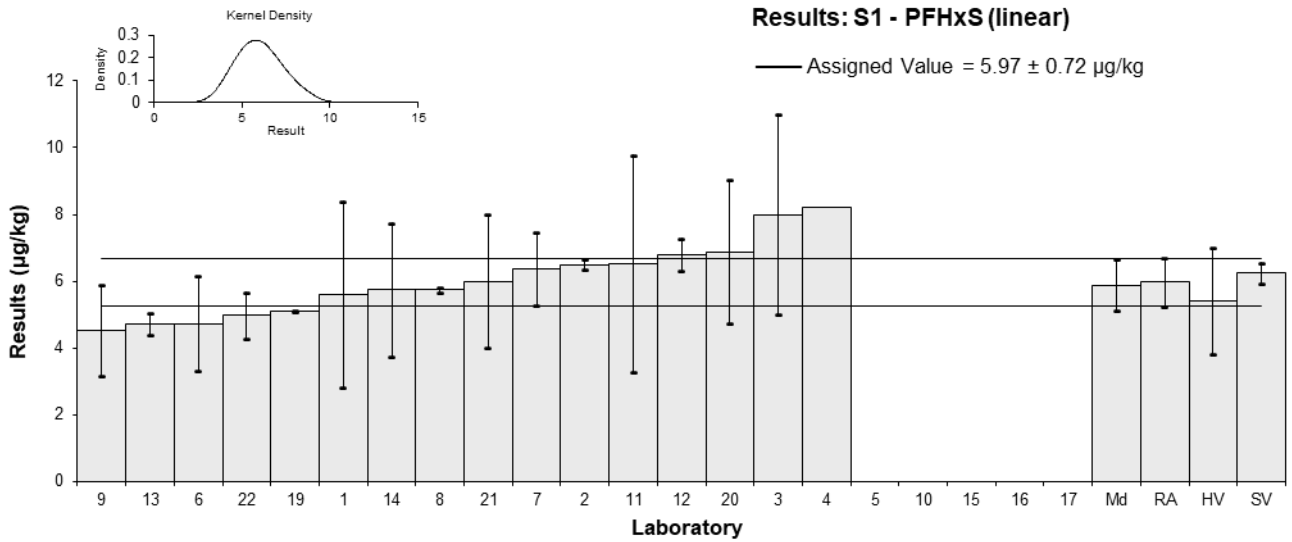


Figure 5

Table 11

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFHpS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	1.1	0.55	NR	-0.70	-0.32
2	1.4	0.078	101	0.47	0.89
3*	2	1	100	2.81	0.72
4	1.964	NR	NR	2.67	6.22
5	NT	NT	NT		
6	1.062	0.318	83	-0.85	-0.65
7	1.35	0.30	95	0.27	0.22
8	1.31	0.093	NR	0.12	0.21
9	1.05	0.3	101	-0.90	-0.72
10	1.07	0.36	80.23	-0.82	-0.56
11	1.34	0.67	NR	0.23	0.09
12	1.61	0.12	NR	1.29	2.03
13*	2.3	0.52	NR	3.98	1.92
14	1.1774	0.4121	86	-0.40	-0.24
15	1.2	0.24	NR	-0.31	-0.30
16	1.4	0.5	NR	0.47	0.23
17	1.334	0.07	92	0.21	0.41
19	1.12	0.222	93	-0.62	-0.65
20	1.41	0.345	91.3	0.51	0.36
21	1.24	0.4	NR	-0.16	-0.10
22	1.39	0.21	96.8	0.43	0.46

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	1.28	0.11
<b>Spike Value</b>	1.34	0.07
<b>Homogeneity Value</b>	1.17	0.35
<b>Robust Average</b>	1.33	0.13
<b>Median</b>	1.34	0.12
<b>Mean</b>	1.39	
<b>N</b>	20	
<b>Max</b>	2.3	
<b>Min</b>	1.05	
<b>Robust SD</b>	0.24	
<b>Robust CV</b>	18%	

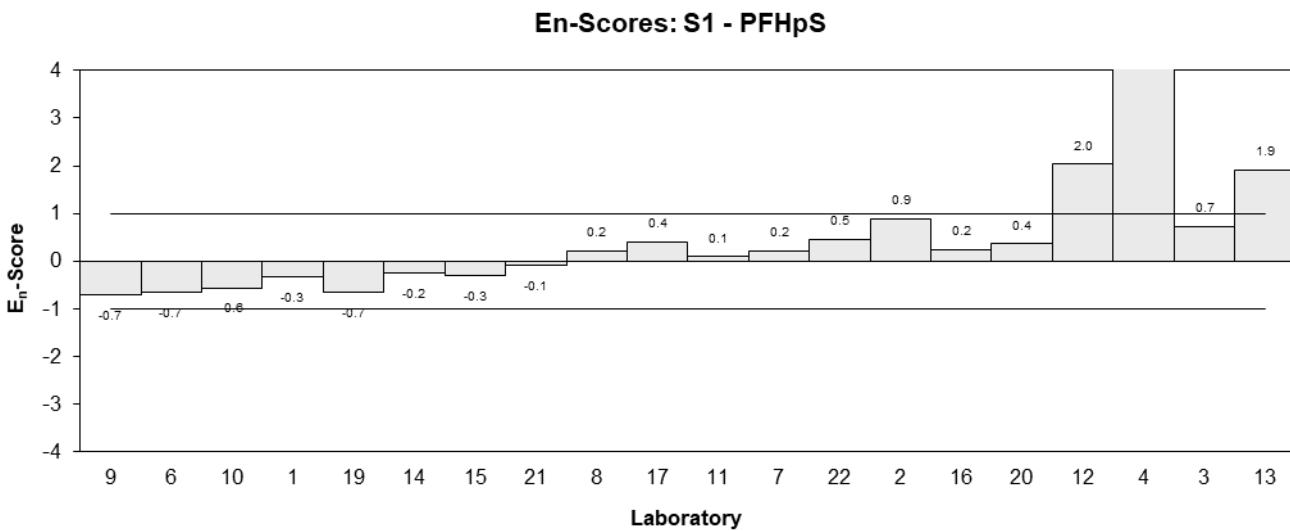
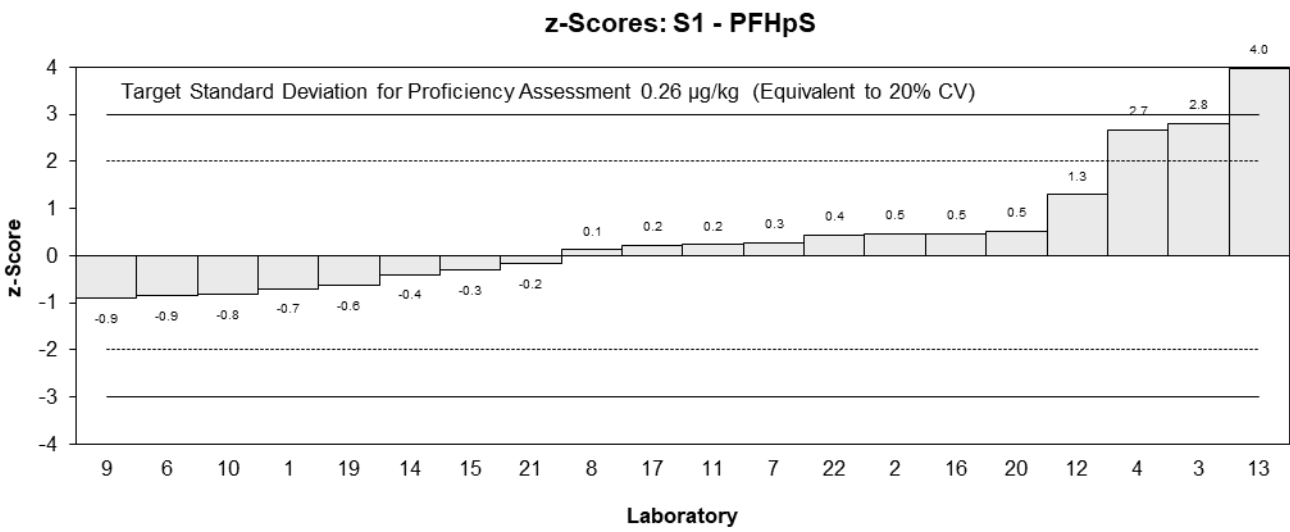
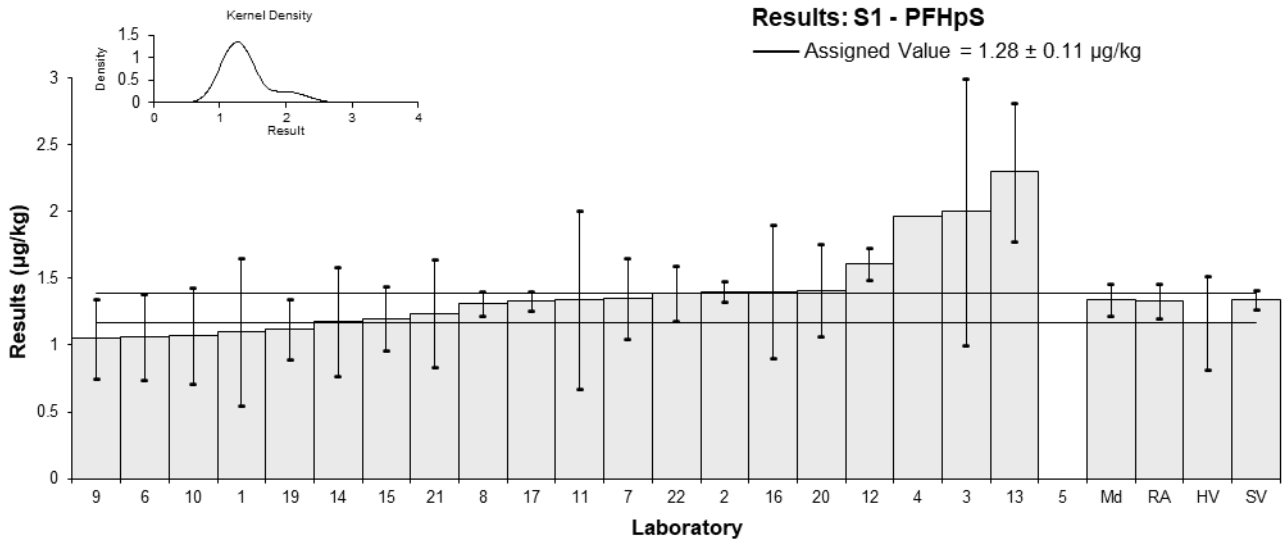


Figure 6

Table 12

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFOS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	1.6	0.8	107	0.00	0.00
2	1.8	0.36	102	0.62	0.52
3	2	1	100	1.25	0.40
4	NT	NT	NT		
5	2.06	0.036	NT	1.44	3.41
6	1.362	0.408	94	-0.74	-0.56
7	1.66	0.31	100	0.19	0.18
8	1.55	0.18	NR	-0.16	-0.23
9	1.3	0.4	112	-0.94	-0.71
10	1.492	0.54	80.23	-0.34	-0.19
11	NT	NT	NT		
12	1.81	0.13	NR	0.66	1.14
13	1.7	0.24	NR	0.31	0.37
14	1.5750	0.5512	86	-0.08	-0.04
15	1.4	0.28	NR	-0.63	-0.65
16	0.91	0.3	NR	-2.16	-2.11
17	1.707	0.093	91	0.33	0.67
19	1.40	0.414	92	-0.63	-0.46
20	1.70	0.959	95.3	0.31	0.10
21	1.44	0.4	74	-0.50	-0.38
22	1.71	0.29	96.8	0.34	0.35

## Statistics

<b>Assigned Value</b>	1.60	0.13
<b>Spike Value</b>	1.79	0.09
<b>Homogeneity Value</b>	1.55	0.46
<b>Robust Average</b>	1.60	0.13
<b>Median</b>	1.60	0.14
<b>Mean</b>	1.59	
<b>N</b>	19	
<b>Max</b>	2.06	
<b>Min</b>	0.91	
<b>Robust SD</b>	0.23	
<b>Robust CV</b>	15%	

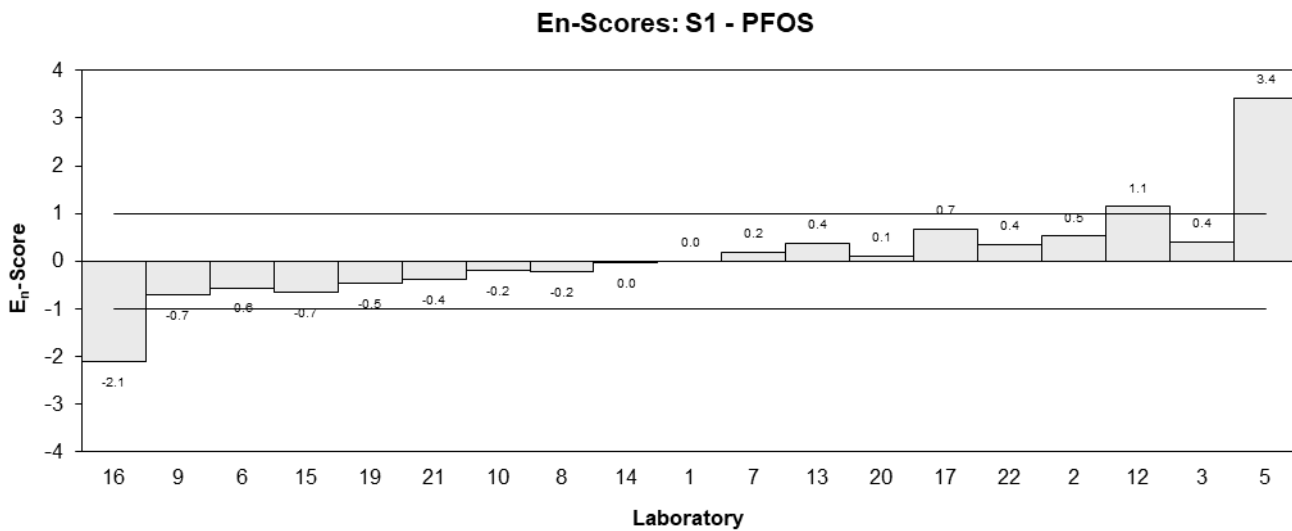
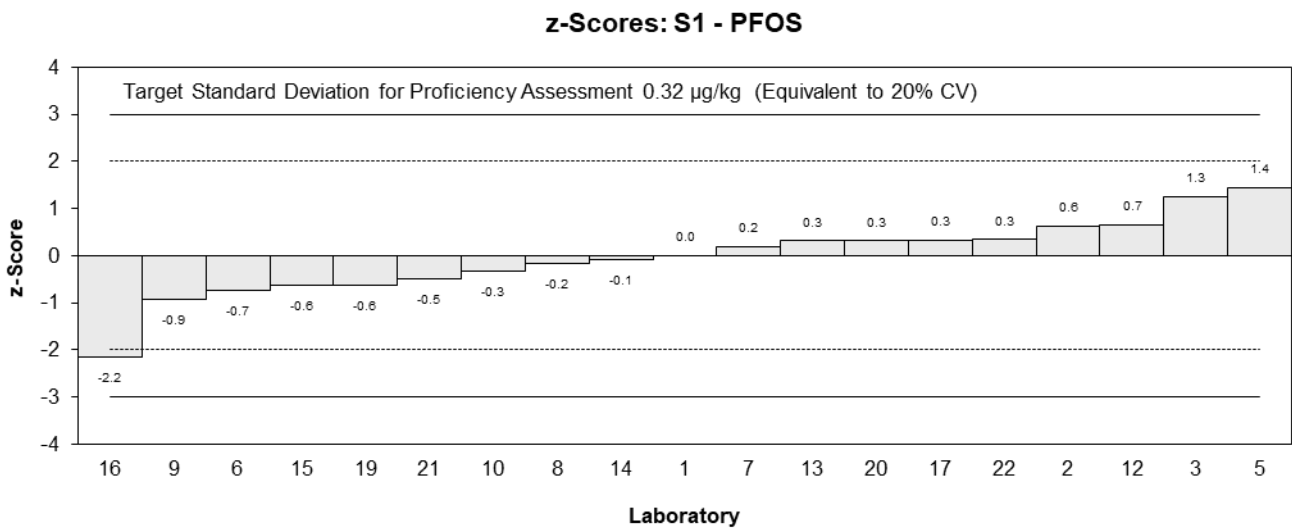
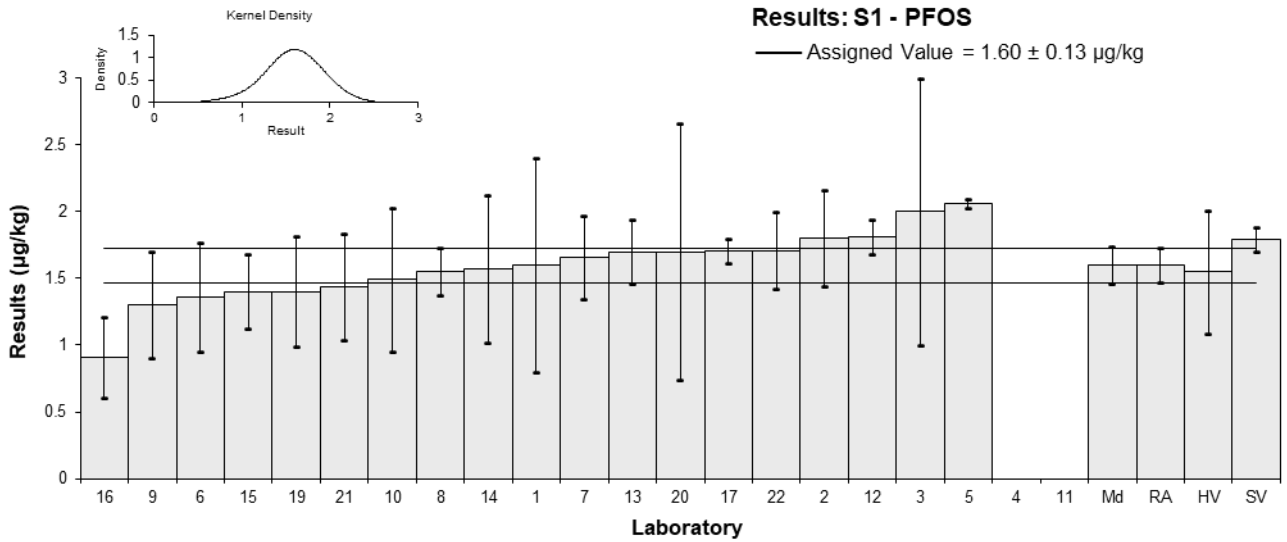


Figure 7

Table 13

## Sample Details

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

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	1.6	0.8	NR	-0.03	-0.01
2	1.8	0.36	102	0.59	0.50
3	2	1	100	1.21	0.39
4*	2.892	NR	NR	3.98	9.86
5	NT	NT	NT		
6	1.362	0.408	94	-0.77	-0.58
7	1.66	0.31	100	0.16	0.15
8	1.55	0.078	111.6	-0.19	-0.40
9	1.3	0.4	112	-0.96	-0.74
10	1.492	0.54	80.23	-0.37	-0.21
11	1.86	0.93	NR	0.78	0.27
12	1.81	0.13	NR	0.62	1.09
13	1.6	0.27	NR	-0.03	-0.03
14	1.5750	0.5512	86	-0.11	-0.06
15	NT	NT	NT		
16	NT	NT	NT		
17	NR	NR	NR		
19	1.40	0.016	92	-0.65	-1.60
20	1.70	0.375	95.3	0.28	0.23
21	1.44	0.4	NR	-0.53	-0.40
22	1.71	0.29	96.8	0.31	0.31

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	1.61	0.13
<b>Spike Value</b>	1.79	0.09
<b>Homogeneity Value</b>	1.55	0.46
<b>Robust Average</b>	1.64	0.14
<b>Median</b>	1.60	0.14
<b>Mean</b>	1.69	
<b>N</b>	17	
<b>Max</b>	2.892	
<b>Min</b>	1.3	
<b>Robust SD</b>	0.23	
<b>Robust CV</b>	14%	



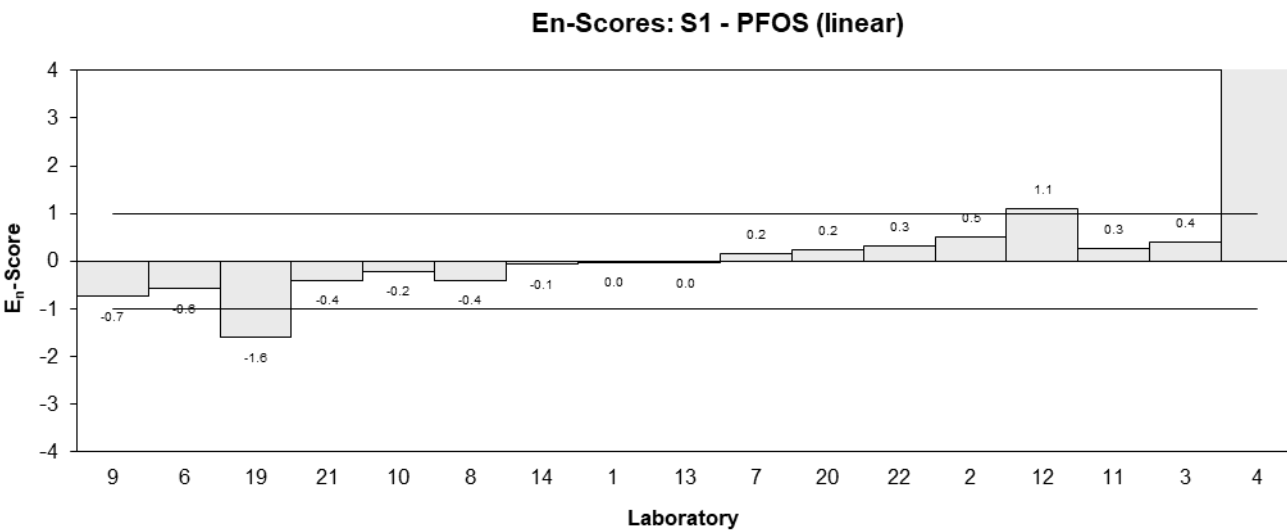
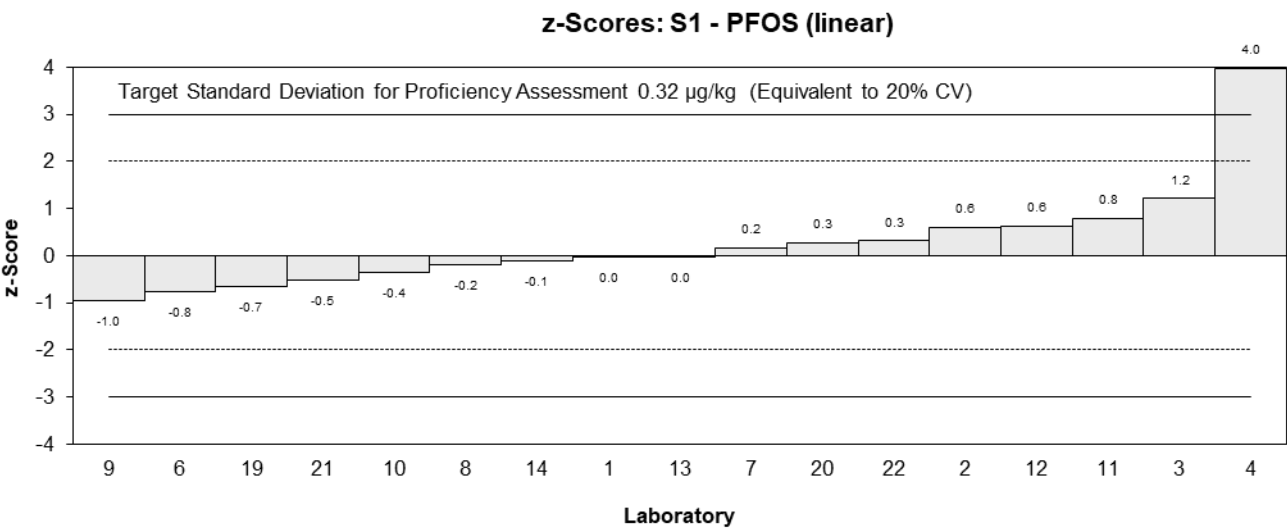
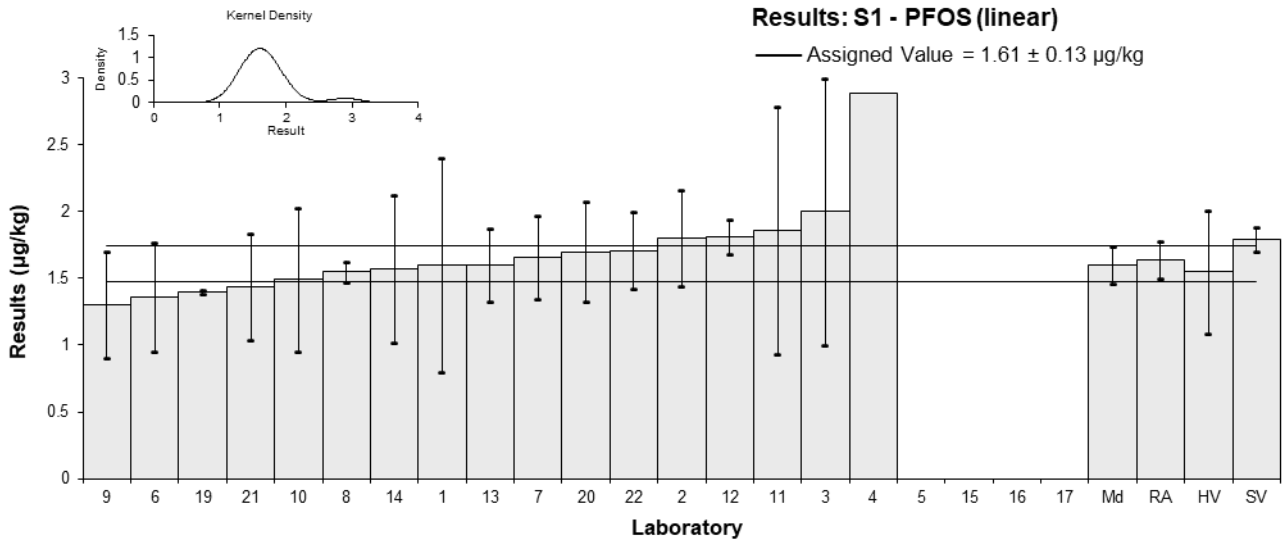


Figure 8

Table 14

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFNS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 2	1	NR		
2	1.6	0.085	102	0.03	0.06
3	2	1	100	1.29	0.41
4	NT	NT	NT		
5	NT	NT	NT		
6	1.338	0.401	85	-0.79	-0.60
7	1.58	0.06	100	-0.03	-0.07
8	1.59	0.062	NR	0.00	0.00
9	1.19	0.35	110	-1.26	-1.07
10	NT	NT	NT		
11	1.63	0.815	NR	0.13	0.05
12	1.63	0.12	NR	0.13	0.23
13	1.8	0.20	NR	0.66	0.88
14	NT	NT	NT		
15	NT	NT	NT		
16*	2.6	0.9	NR	3.18	1.11
17	1.668	0.079	91	0.25	0.51
19	1.34	0.222	92	-0.79	-0.97
20	1.77	0.965	95.3	0.57	0.18
21	1.63	0.5	NR	0.13	0.08
22	1.53	0.321	96.8	-0.19	-0.17

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	1.59	0.13
<b>Spike Value</b>	1.80	0.09
<b>Homogeneity Value</b>	1.51	0.45
<b>Robust Average</b>	1.62	0.15
<b>Median</b>	1.63	0.10
<b>Mean</b>	1.66	
<b>N</b>	15	
<b>Max</b>	2.6	
<b>Min</b>	1.19	
<b>Robust SD</b>	0.23	
<b>Robust CV</b>	14%	

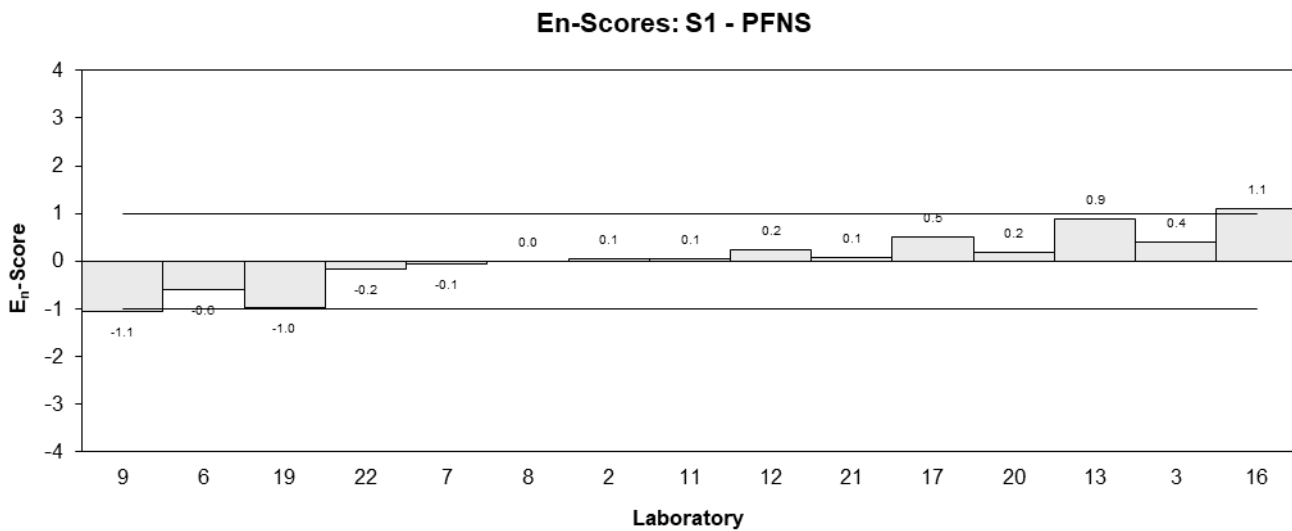
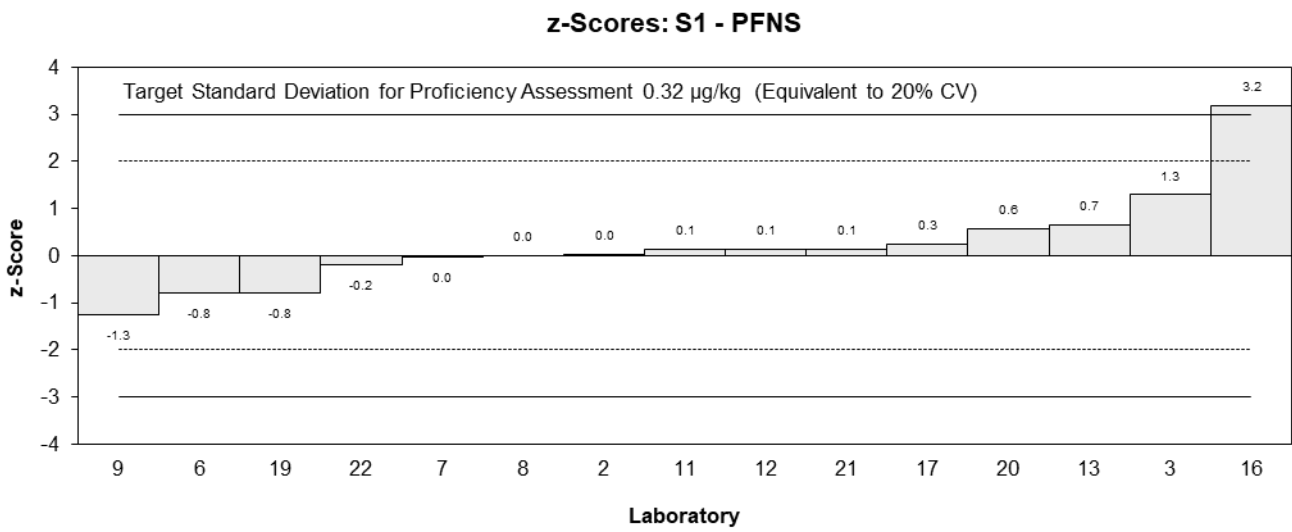
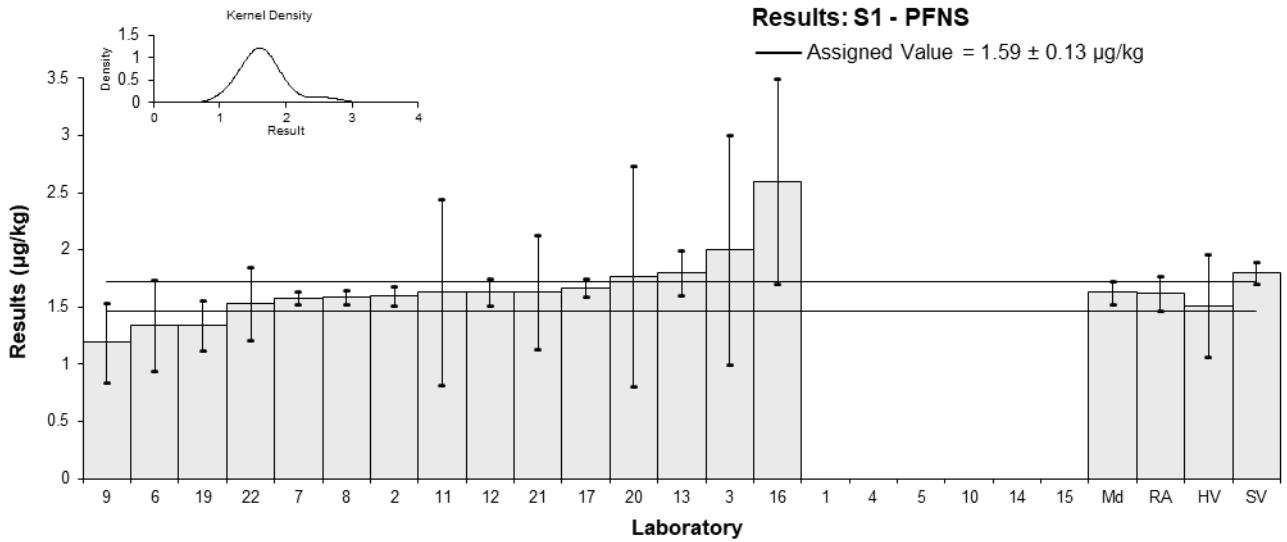


Figure 9

Table 15

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFDS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 2	1	NR		
2	1.3	0.26	102	-0.94	-0.90
3	2	1	100	1.25	0.39
4	NT	NT	NT		
5	NT	NT	NT		
6	1.247	0.374	88	-1.10	-0.82
7	1.33	0.43	100	-0.84	-0.56
8	1.66	0.066	NR	0.19	0.27
9	1.06	0.32	110	-1.69	-1.41
10	<2	NR	80.23		
11	1.91	0.955	NR	0.97	0.32
12	1.71	0.12	NR	0.34	0.45
13*	3.5	1.06	NR	5.94	1.76
14	1.6938	0.5928	86	0.29	0.15
15	NT	NT	NT		
16	2.1	0.7	NR	1.56	0.68
17	1.716	0.11	91	0.36	0.49
19	1.32	0.271	92	-0.87	-0.82
20	1.65	0.969	95.3	0.16	0.05
21	1.57	0.6	NR	-0.09	-0.05
22	1.74	0.383	96.8	0.44	0.32

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	1.60	0.21
<b>Spike Value</b>	1.80	0.09
<b>Homogeneity Value</b>	1.44	0.58
<b>Robust Average</b>	1.64	0.22
<b>Median</b>	1.68	0.26
<b>Mean</b>	1.72	
<b>N</b>	16	
<b>Max</b>	3.5	
<b>Min</b>	1.06	
<b>Robust SD</b>	0.36	
<b>Robust CV</b>	22%	

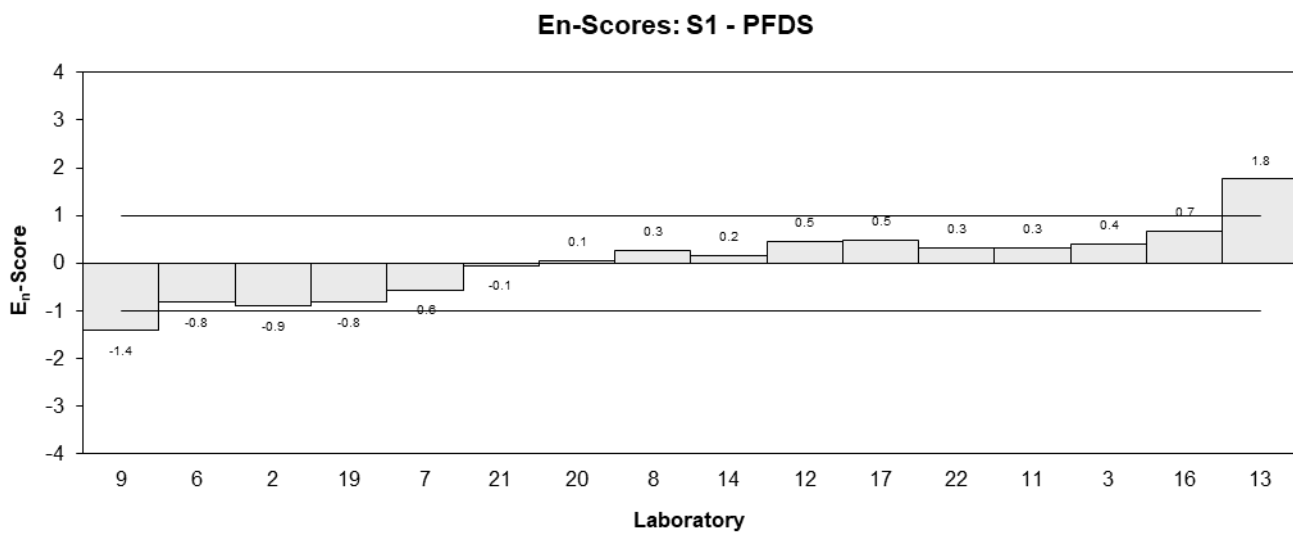
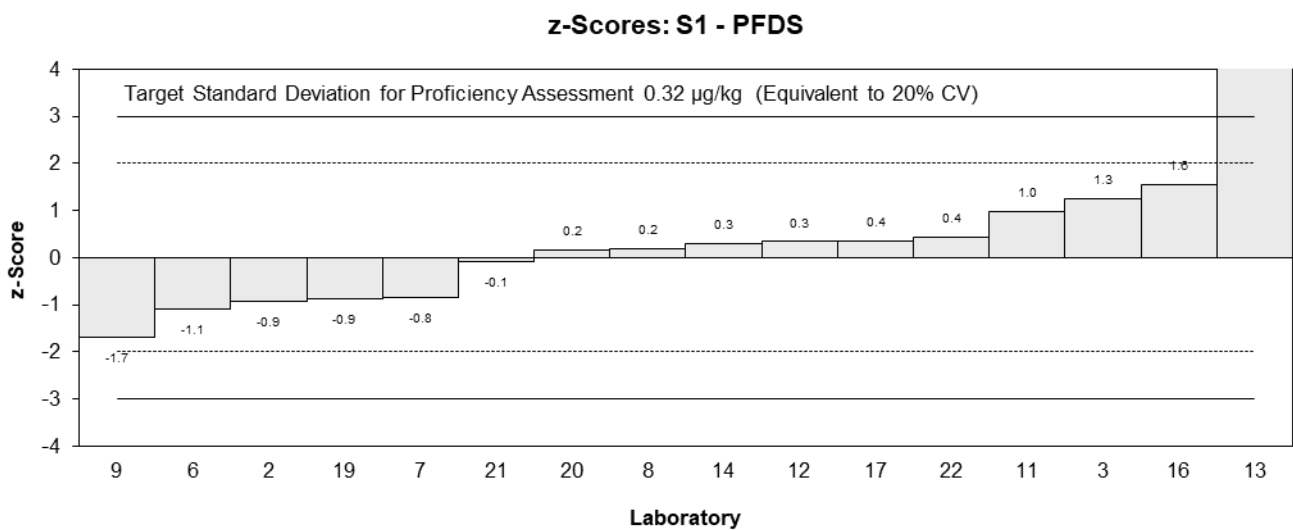
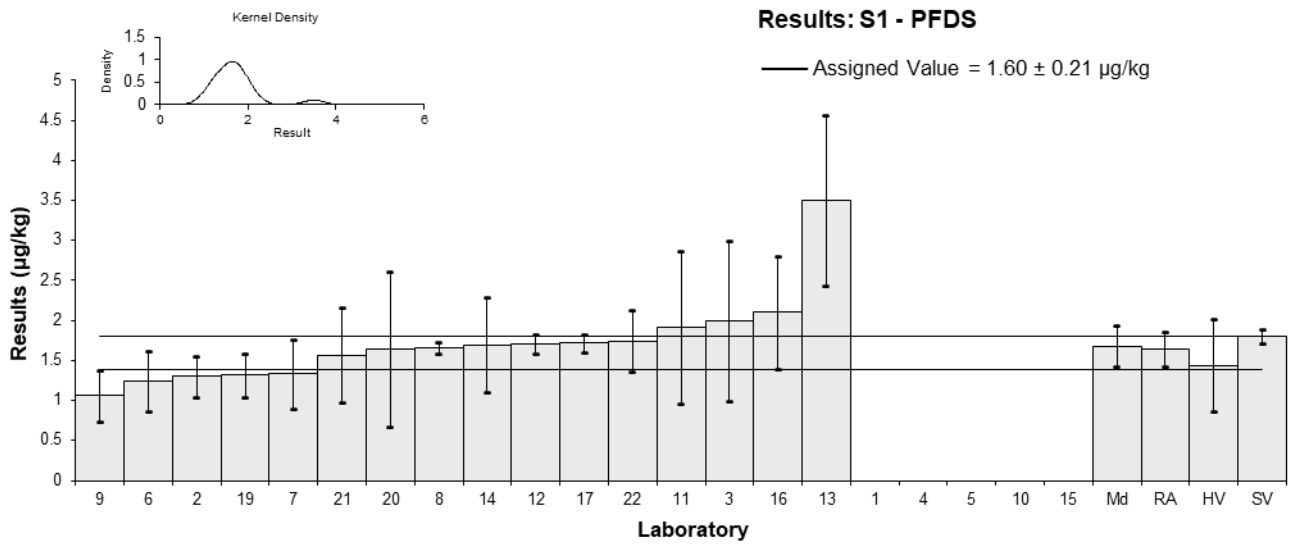


Figure 10

Table 16

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFBA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	5.5	2.75	104	-0.56	-0.25
2	6.5	0.13	112	0.24	0.43
3	8	3	107	1.45	0.59
4*	10.192	NR	NR	3.22	5.87
5	NT	NT	NT		
6	4.134	1.24	81	-1.67	-1.46
7	6.42	0.25	65	0.18	0.30
8	6.29	0.068	77.6	0.07	0.13
9	5.51	1.65	58	-0.56	-0.39
10	5.712	2.4	32.51	-0.39	-0.20
11	6.07	3.035	NR	-0.10	-0.04
12	7.22	0.52	NR	0.82	1.19
13	NT	NT	NT		
14	NT	NT	NT		
15	4.6	0.92	NR	-1.29	-1.40
16	8.3	2.9	NR	1.69	0.71
17	5.873	0.057	65	-0.26	-0.48
19	5.41	0.969	46	-0.64	-0.67
20	7.39	1.69	98.8	0.96	0.65
21	6.19	2	74	-0.01	0.00
22	6.38	0.574	92.3	0.15	0.20

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	6.20	0.68
<b>Spike Value</b>	6.57	0.33
<b>Homogeneity Value</b>	5.8	2.3
<b>Robust Average</b>	6.33	0.75
<b>Median</b>	6.24	0.64
<b>Mean</b>	6.43	
<b>N</b>	18	
<b>Max</b>	10.192	
<b>Min</b>	4.134	
<b>Robust SD</b>	1.3	
<b>Robust CV</b>	20%	

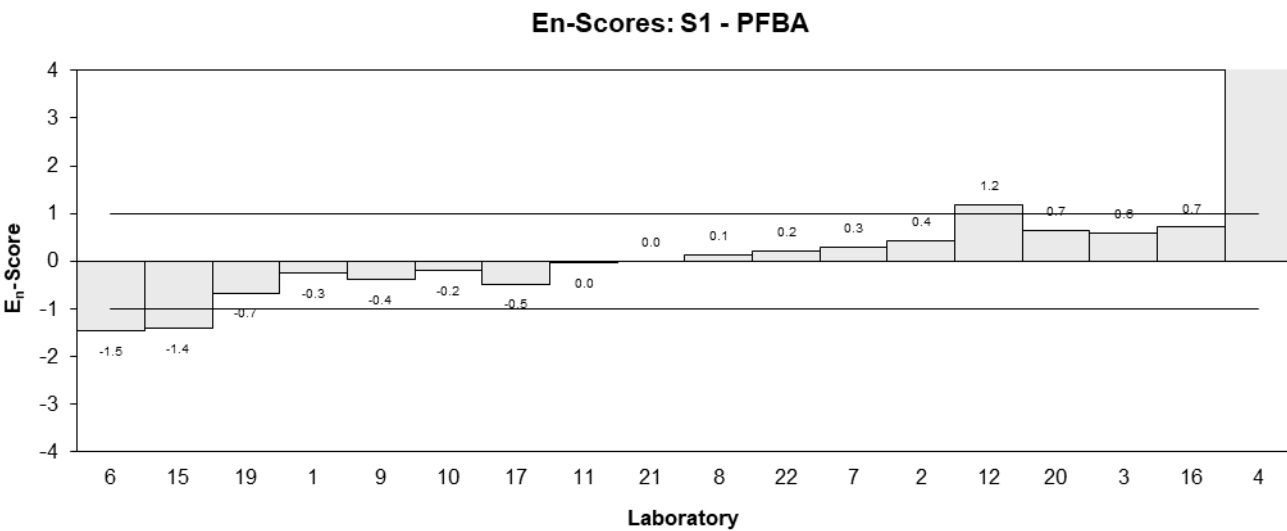
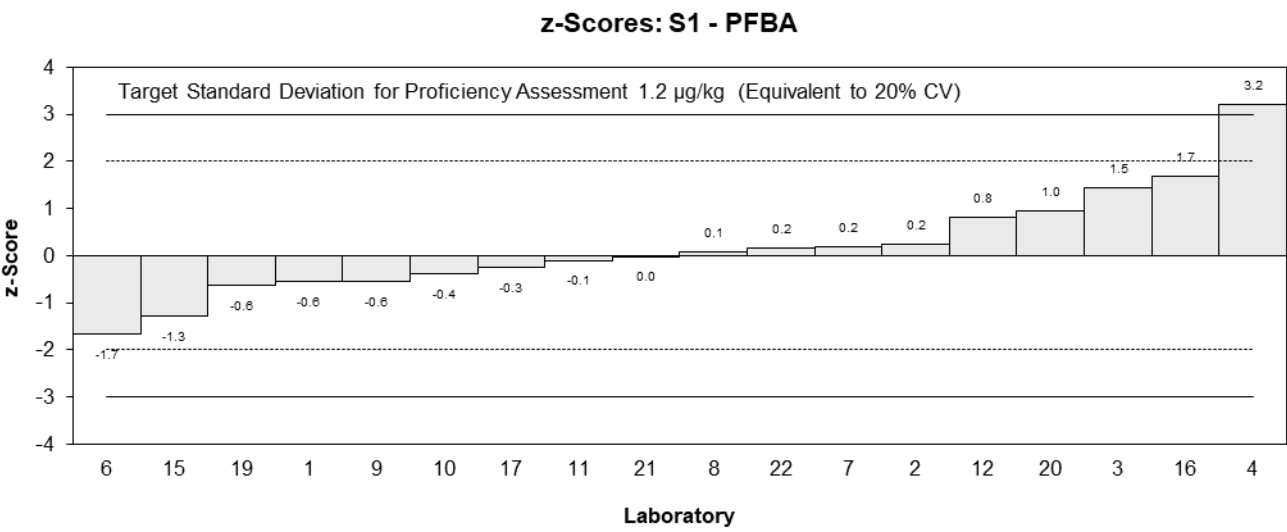
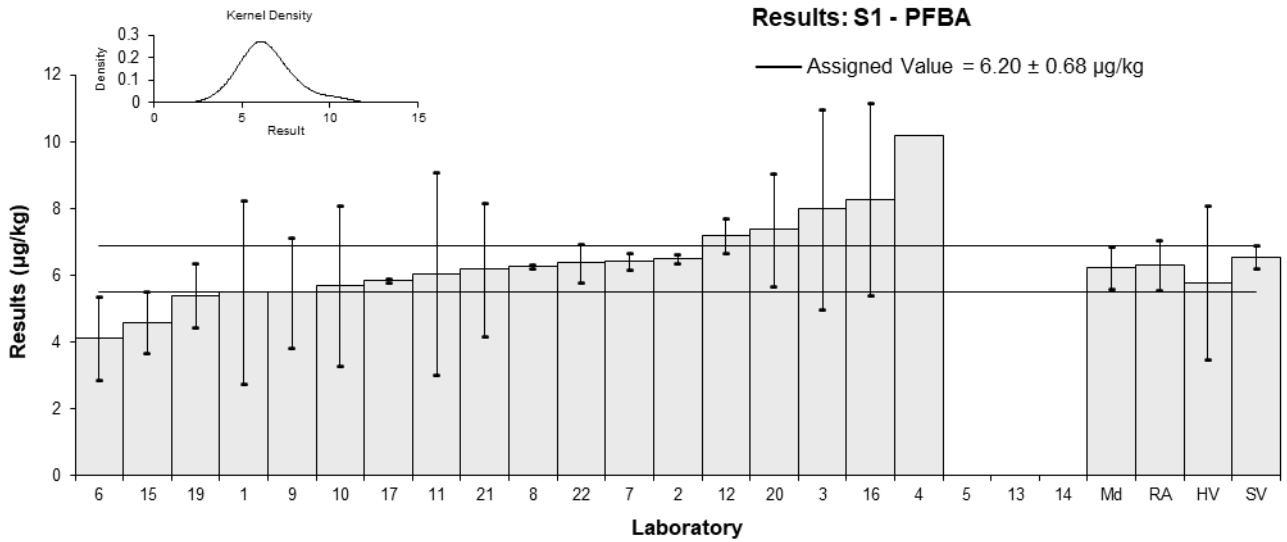


Figure 11

Table 17

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFPeA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	2	1	112	-0.61	-0.28
2	2.4	0.0042	111	0.26	0.63
3	3	1	96	1.58	0.71
4	2.77	NR	NR	1.07	2.58
5	NT	NT	NT		
6	1.943	0.583	85	-0.74	-0.55
7	2.32	0.01	68	0.09	0.21
8	2.21	0.075	87.9	-0.15	-0.34
9	2	0.6	89	-0.61	-0.44
10	<2	NR	88		
11	2.66	1.33	NR	0.83	0.28
12	2.22	0.16	NR	-0.13	-0.24
13	2.1	0.40	NR	-0.39	-0.41
14	2.2760	0.7966	9	-0.01	0.00
15	1.8	0.36	NR	-1.05	-1.18
16	2.5	0.9	NR	0.48	0.24
17	2.169	0.052	69	-0.24	-0.56
19	2.07	0.516	87	-0.46	-0.38
20	2.79	2.66	100.3	1.12	0.19
21	2.06	0.6	78	-0.48	-0.35
22	2.17	0.217	105	-0.24	-0.38

## Statistics

<b>Assigned Value</b>	2.28	0.19
<b>Spike Value</b>	2.39	0.12
<b>Homogeneity Value</b>	2.21	0.66
<b>Robust Average</b>	2.28	0.19
<b>Median</b>	2.21	0.16
<b>Mean</b>	2.29	
<b>N</b>	19	
<b>Max</b>	3	
<b>Min</b>	1.8	
<b>Robust SD</b>	0.34	
<b>Robust CV</b>	15%	



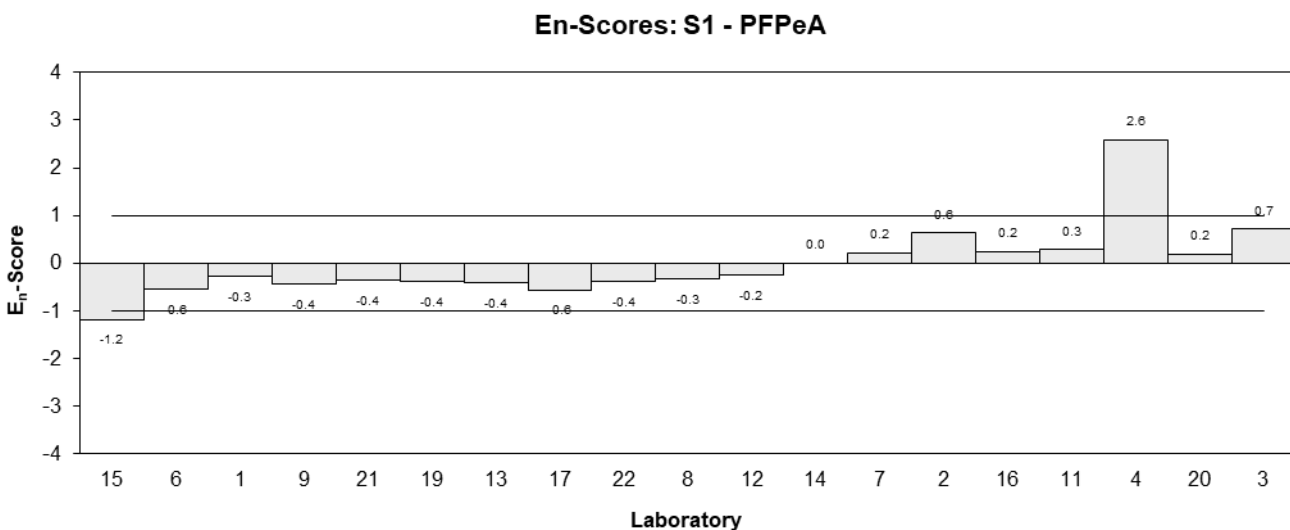
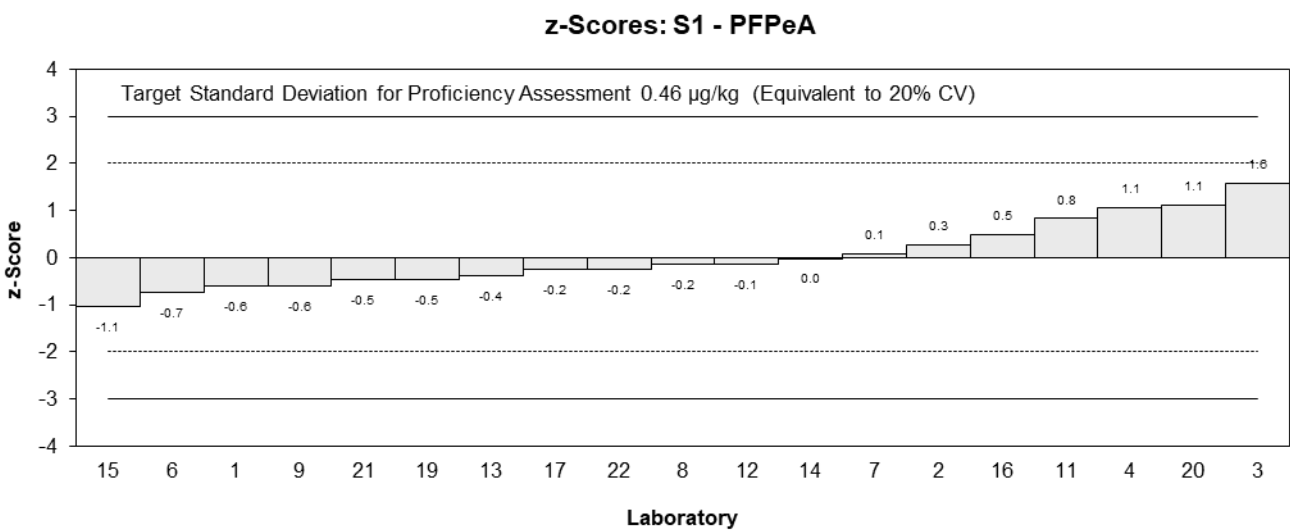
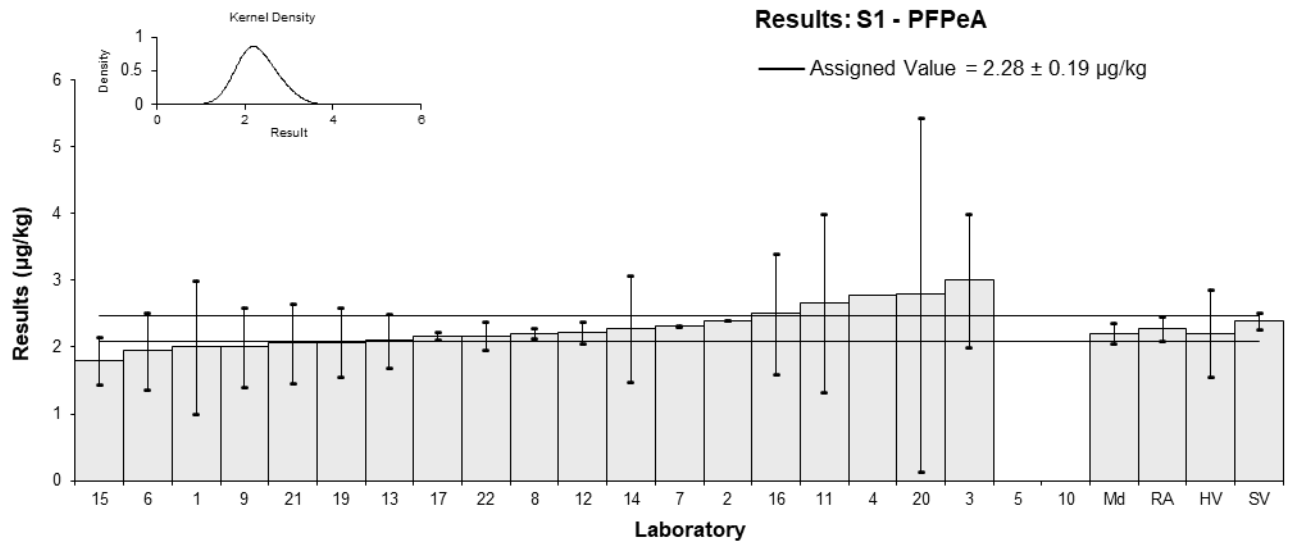


Figure 12

Table 18

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFHxA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	5.8	2.9	101	-0.33	-0.14
2	7.3	0.24	116	0.88	1.74
3	8	3	104	1.44	0.59
4*	9.524	NR	NR	2.67	5.71
5	NT	NT	NT		
6	5.207	1.562	96	-0.81	-0.60
7	7.09	0.08	64	0.71	1.50
8	6.18	0.074	92.2	-0.02	-0.05
9	4.83	1.45	105	-1.11	-0.88
10	4.552	1.7	82.45	-1.33	-0.92
11	7.06	3.53	NR	0.68	0.24
12	6.74	0.48	NR	0.43	0.70
13	6.2	1.41	NR	-0.01	-0.01
14	6.4614	2.2615	17	0.20	0.11
15	4.8	0.96	NR	-1.14	-1.26
16	6.8	2.4	NR	0.48	0.24
17	6.246	0.318	73	0.03	0.05
19	5.67	1	89	-0.43	-0.47
20	7.21	1.69	115.9	0.81	0.56
21	5.83	2	78	-0.31	-0.18
22	6.09	0.852	85.2	-0.10	-0.12

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	6.21	0.58
<b>Spike Value</b>	6.54	0.33
<b>Homogeneity Value</b>	6.3	1.9
<b>Robust Average</b>	6.30	0.62
<b>Median</b>	6.22	0.59
<b>Mean</b>	6.38	
<b>N</b>	20	
<b>Max</b>	9.524	
<b>Min</b>	4.552	
<b>Robust SD</b>	1.1	
<b>Robust CV</b>	18%	

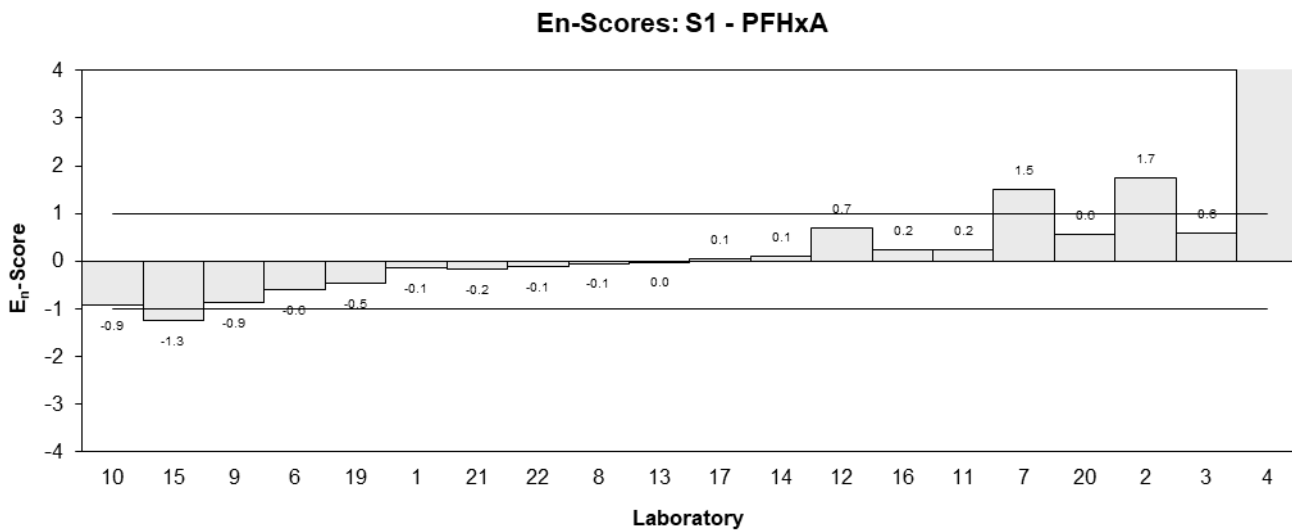
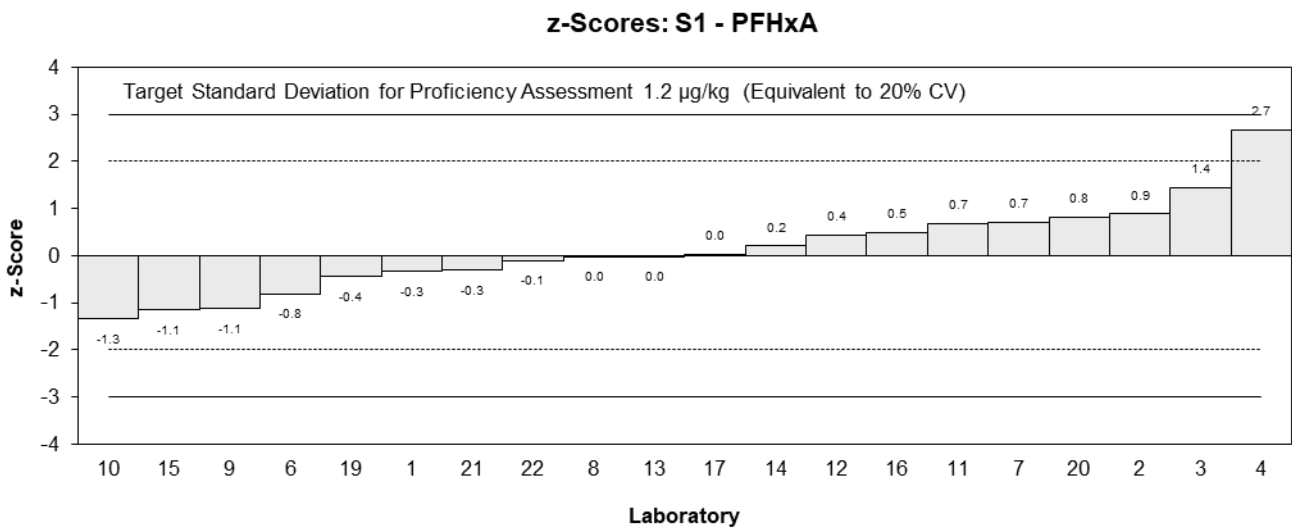
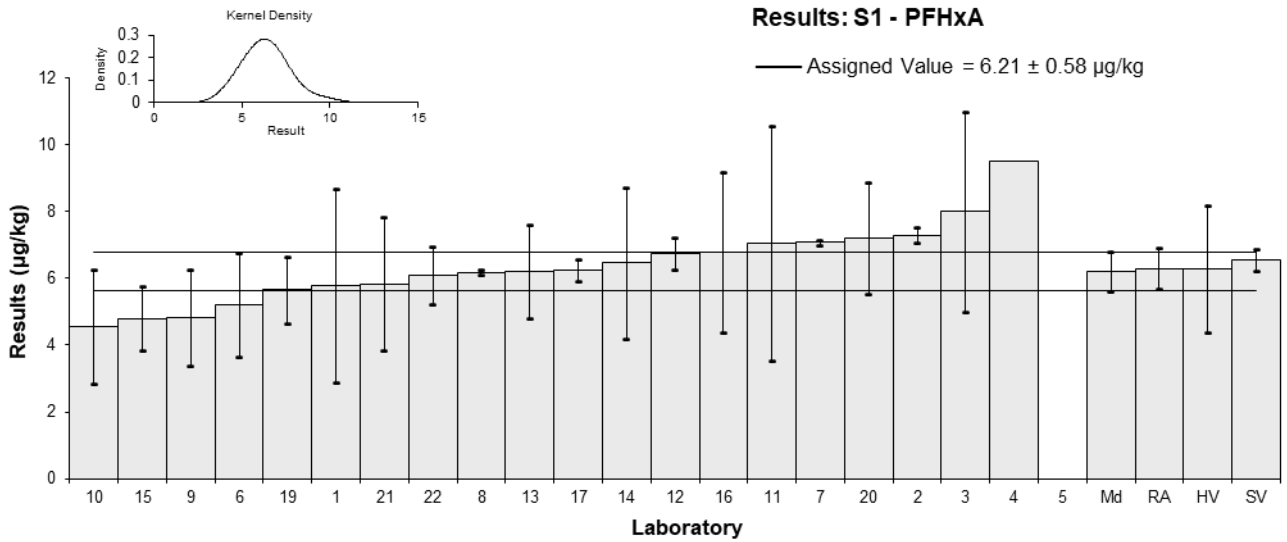


Figure 13

Table 19

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFHpA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	6.4	3.2	101	-0.21	-0.09
2	7.5	0.68	112	0.61	0.83
3	8	3	99	0.99	0.43
4*	12.348	NR	NR	4.24	7.98
5	NT	NT	NT		
6	5.55	1.665	94	-0.85	-0.62
7	8.71	1.26	58	1.52	1.40
8	6.94	0.089	NR	0.19	0.36
9	4.57	1.37	91	-1.58	-1.37
10	5.49	2	82.06	-0.89	-0.56
11	6.89	3.445	NR	0.16	0.06
12	7.26	0.52	NR	0.43	0.66
13	5.4	0.57	NR	-0.96	-1.41
14	6.5226	2.2829	39	-0.12	-0.07
15	5.3	1.06	NR	-1.03	-1.08
16	6.5	2.3	NR	-0.13	-0.07
17	7.854	0.404	73	0.88	1.44
19	6.43	1.06	90	-0.19	-0.20
20	8.79	2.44	109.0	1.58	0.83
21	6.87	2	77	0.14	0.09
22	6.14	0.922	84	-0.40	-0.46

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	6.68	0.71
<b>Spike Value</b>	7.48	0.37
<b>Homogeneity Value</b>	7.2	2.2
<b>Robust Average</b>	6.81	0.77
<b>Median</b>	6.70	0.81
<b>Mean</b>	6.97	
<b>N</b>	20	
<b>Max</b>	12.348	
<b>Min</b>	4.57	
<b>Robust SD</b>	1.4	
<b>Robust CV</b>	20%	

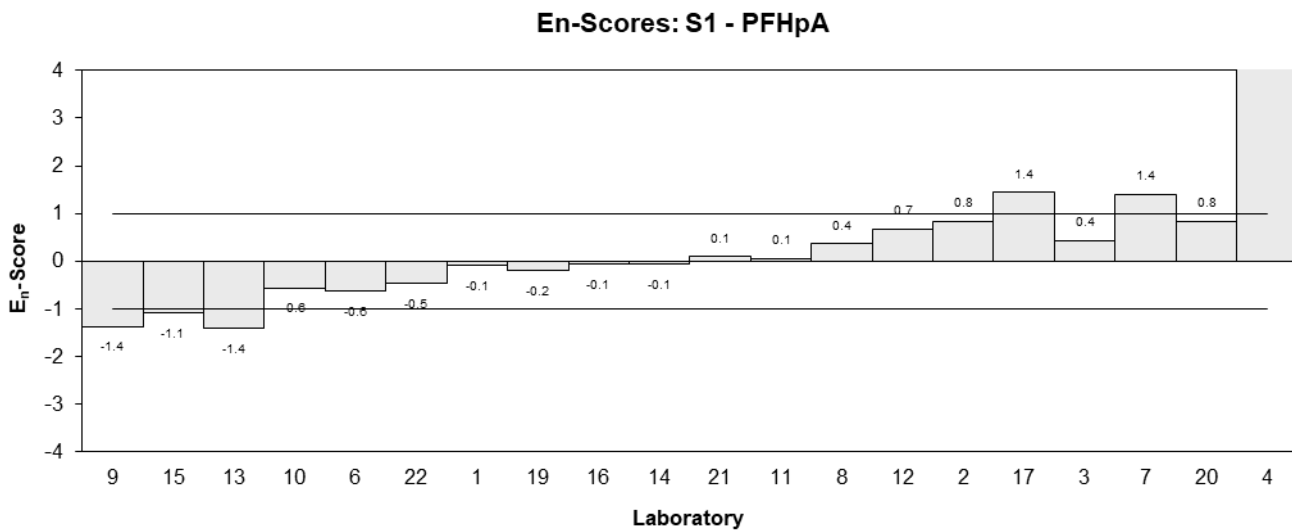
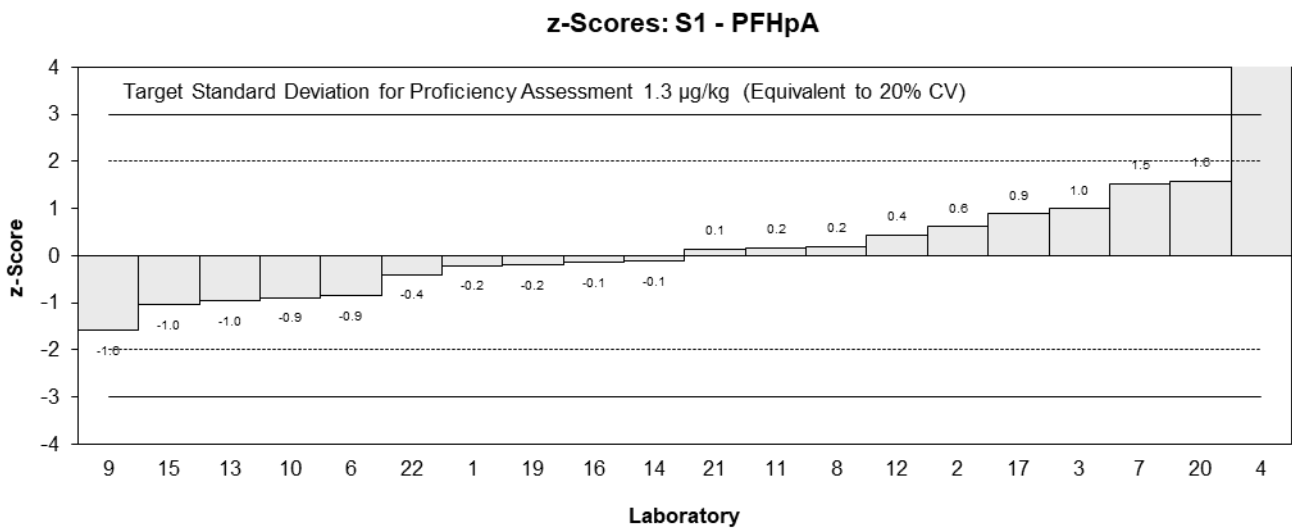
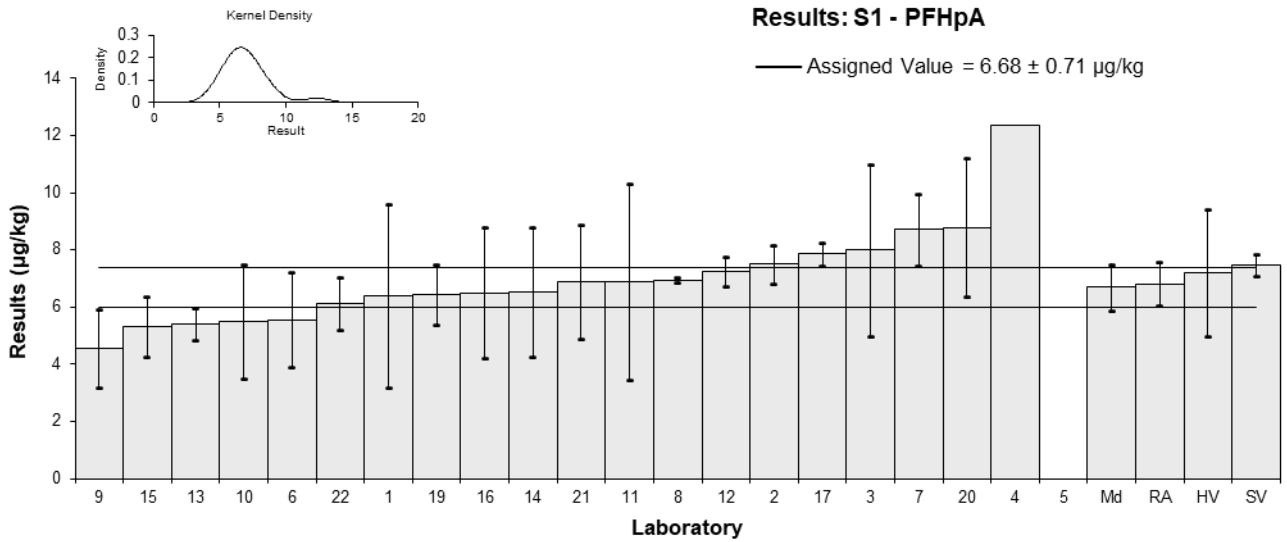


Figure 14

Table 20

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFOA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 5	2.5	96		
2	< 1.0	NR	112		
3	1	1	99	0.32	0.06
4*	1.567	NR	NR	3.34	7.94
5	0.84	0.042	NT	-0.53	-1.12
6	0.756	0.227	89	-0.98	-0.77
7	0.94	0.05	60	0.00	0.00
8	0.942	0.074	100.6	0.01	0.02
9	0.73	0.22	104	-1.12	-0.90
10	<1	NR	83.26		
11	1	0.5	NR	0.32	0.12
12	0.91	0.06	NR	-0.16	-0.30
13	1.1	0.26	NR	0.85	0.59
14	0.8466	0.2963	65	-0.50	-0.30
15	< 1	NR	NR		
16	1.3	0.5	NR	1.91	0.71
17	1.015	0.017	76	0.40	0.93
19	0.868	0.16	91	-0.38	-0.40
20	1.07	0.201	105.8	0.69	0.60
21	0.90	0.3	78	-0.21	-0.13
22	0.966	0.155	85.7	0.14	0.15

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	0.940	0.079
<b>Spike Value</b>	0.943	0.047
<b>Homogeneity Value</b>	1.03	0.31
<b>Robust Average</b>	0.955	0.088
<b>Median</b>	0.942	0.067
<b>Mean</b>	0.985	
<b>N</b>	17	
<b>Max</b>	1.567	
<b>Min</b>	0.73	
<b>Robust SD</b>	0.14	
<b>Robust CV</b>	15%	

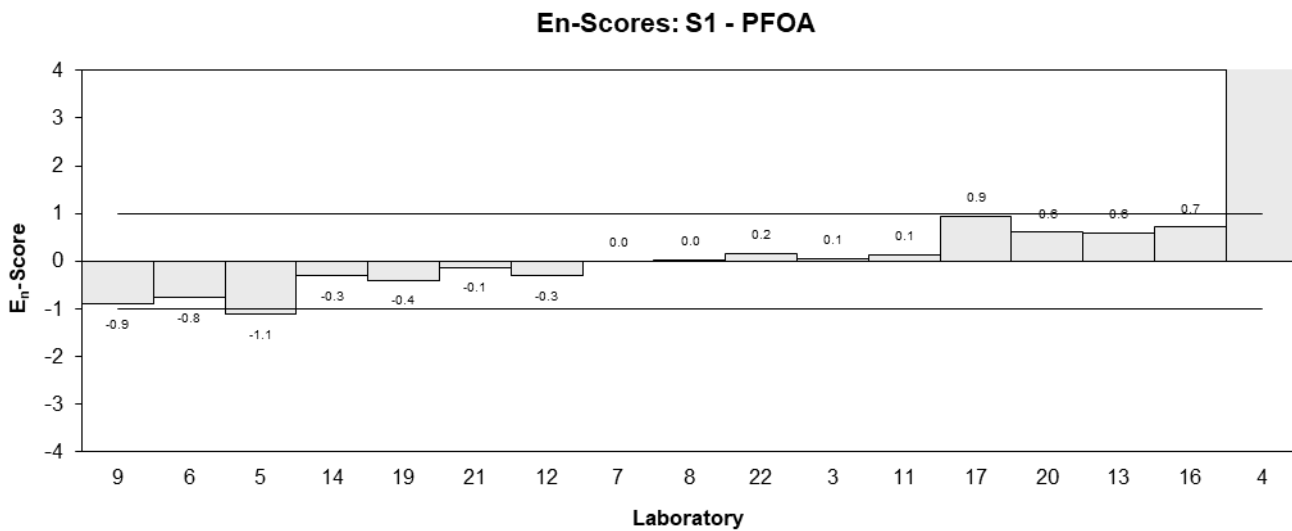
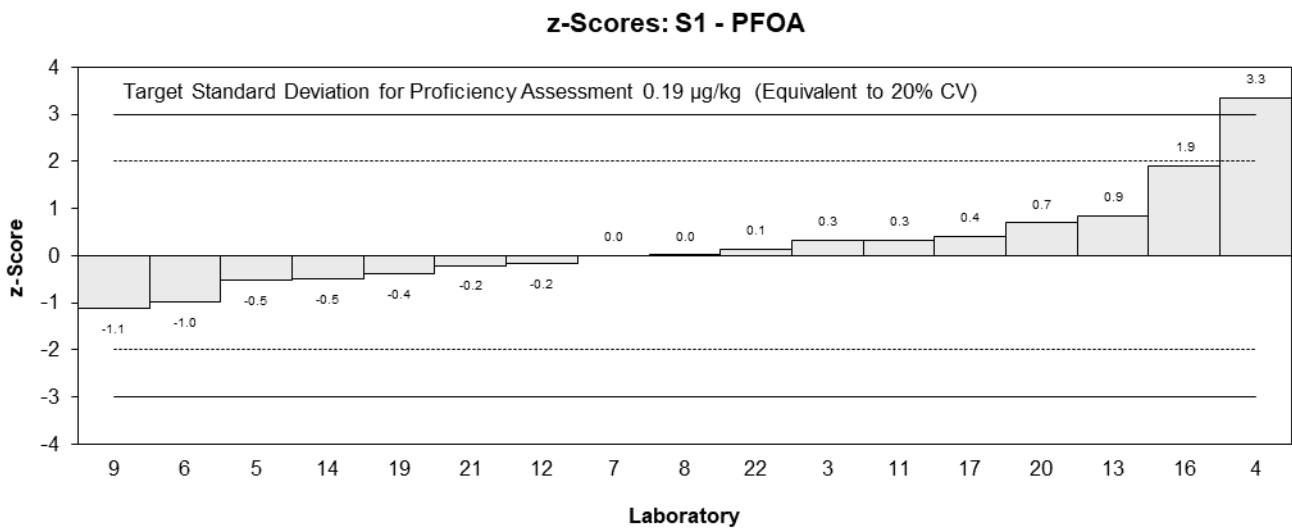
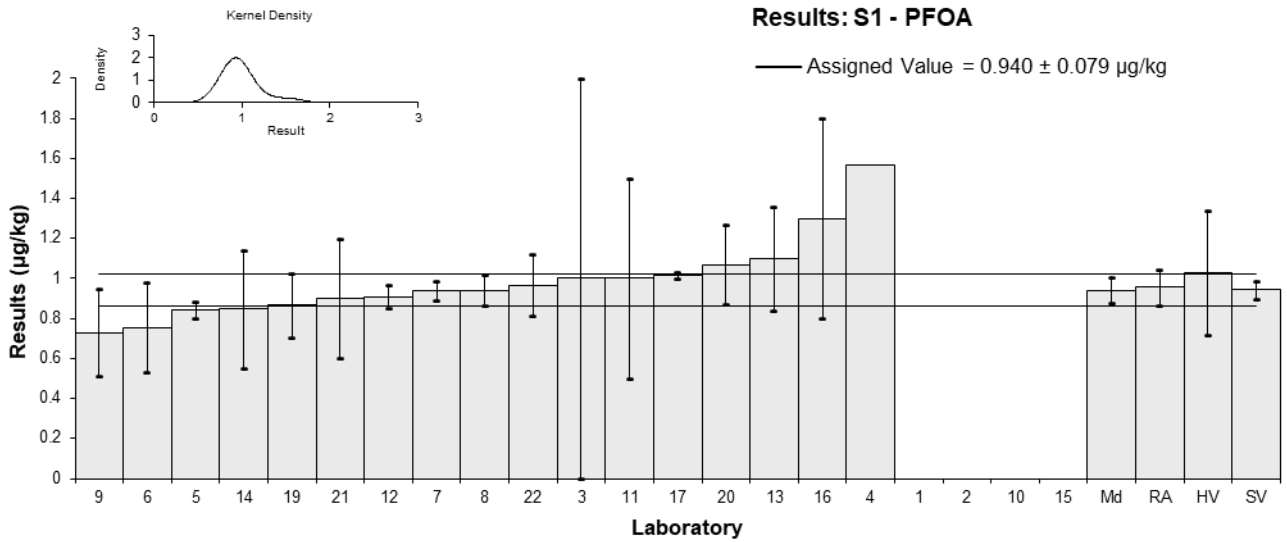


Figure 15

Table 21

**Sample Details**

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFNA
<b>Unit</b>	µg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>Uncertainty</b>	<b>Rec</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	< 2	1	96		
2	2.0	0.15	111	0.59	0.84
3	2	1	104	0.59	0.21
4	2.656	NR	NR	2.42	4.33
5*	3.36	0.2	NT	4.39	5.55
6	1.505	0.452	88	-0.80	-0.58
7	1.97	0.43	59	0.50	0.38
8	1.88	0.070	NR	0.25	0.42
9	1.35	0.4	113	-1.23	-0.98
10	1.228	0.43	92.71	-1.57	-1.19
11	2.29	1.145	NR	1.40	0.43
12	1.87	0.13	NR	0.22	0.34
13	1.6	0.16	NR	-0.53	-0.74
14	1.7093	0.5983	72	-0.23	-0.13
15	1.2	0.24	NR	-1.65	-1.89
16	2.1	0.7	NR	0.87	0.43
17	1.667	0.09	76	-0.34	-0.56
19	1.66	0.251	92	-0.36	-0.41
20	1.91	0.543	101.3	0.34	0.21
21	1.8	0.5	79	0.03	0.02
22	1.82	0.291	96.6	0.08	0.08

\* Outlier, see Section 4.2

**Statistics**

<b>Assigned Value</b>	1.79	0.20
<b>Spike Value</b>	1.89	0.09
<b>Homogeneity Value</b>	1.91	0.57
<b>Robust Average</b>	1.82	0.21
<b>Median</b>	1.85	0.15
<b>Mean</b>	1.88	
<b>N</b>	20	
<b>Max</b>	3.36	
<b>Min</b>	1.2	
<b>Robust SD</b>	0.38	
<b>Robust CV</b>	21%	



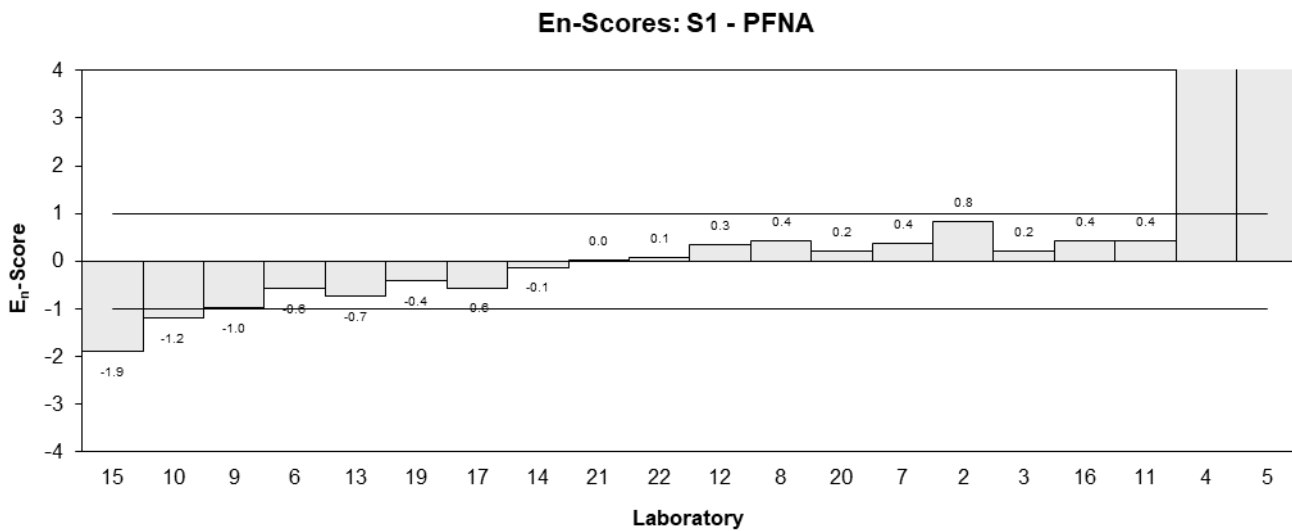
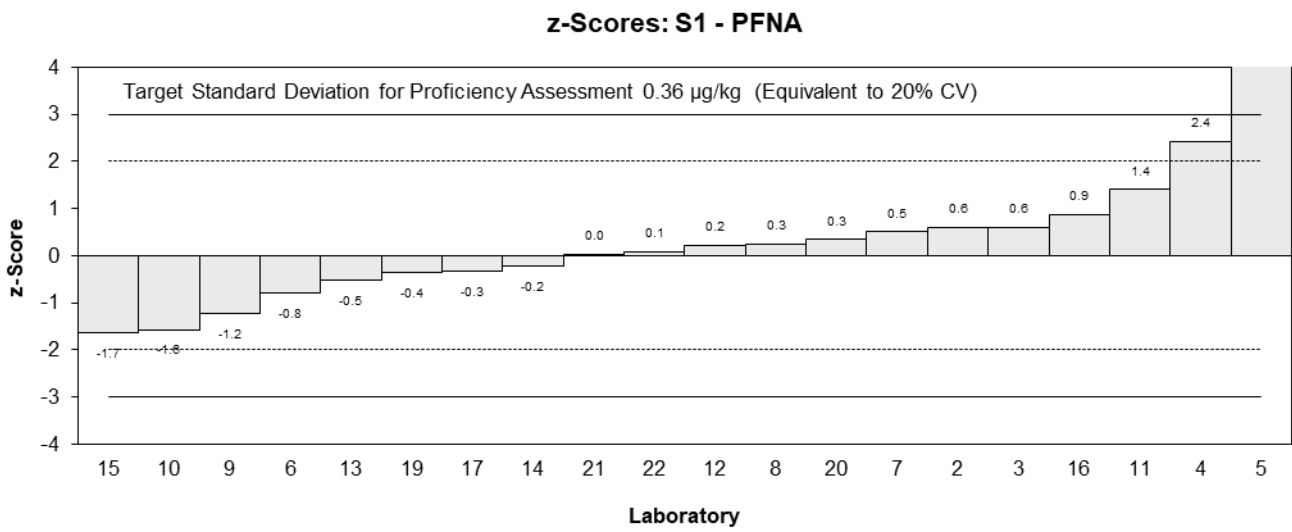
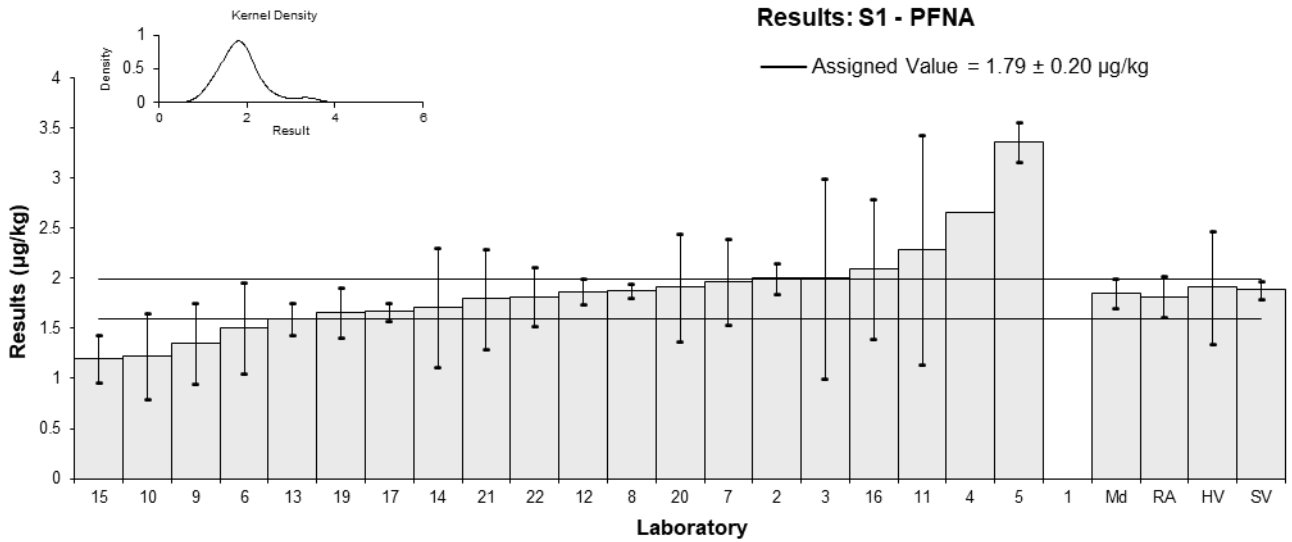


Figure 16

Table 22

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFDA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 5	2.5	104		
2	1.7	0.26	101	0.38	0.40
3	2	1	95	1.33	0.42
4	1.85	NR	NR	0.85	1.80
5	NT	NT	NT		
6	1.295	0.389	97	-0.90	-0.68
7	1.78	0.23	49	0.63	0.73
8	1.58	0.042	NR	0.00	0.00
9	1.18	0.35	100	-1.27	-1.05
10	1.104	0.39	95.42	-1.51	-1.14
11	1.61	0.805	NR	0.09	0.04
12	1.71	0.13	NR	0.41	0.65
13	1.3	0.13	NR	-0.89	-1.41
14	1.7502	0.6126	82	0.54	0.27
15	< 1	NR	NR		
16	1.7	0.6	NR	0.38	0.19
17	1.501	0.121	76	-0.25	-0.41
19	1.37	0.223	91	-0.66	-0.78
20	1.70	0.451	99.8	0.38	0.25
21	1.54	0.5	74	-0.13	-0.08
22	1.69	0.254	88.4	0.35	0.37

## Statistics

<b>Assigned Value</b>	1.58	0.15
<b>Spike Value</b>	1.59	0.08
<b>Homogeneity Value</b>	1.66	0.50
<b>Robust Average</b>	1.58	0.15
<b>Median</b>	1.65	0.10
<b>Mean</b>	1.58	
<b>N</b>	18	
<b>Max</b>	2	
<b>Min</b>	1.104	
<b>Robust SD</b>	0.26	
<b>Robust CV</b>	16%	

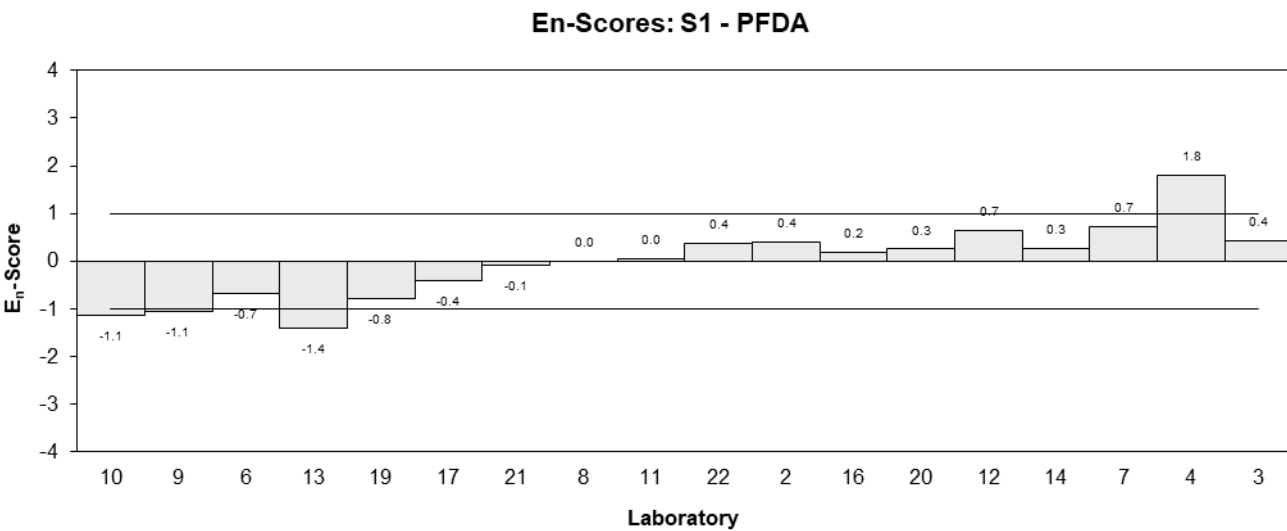
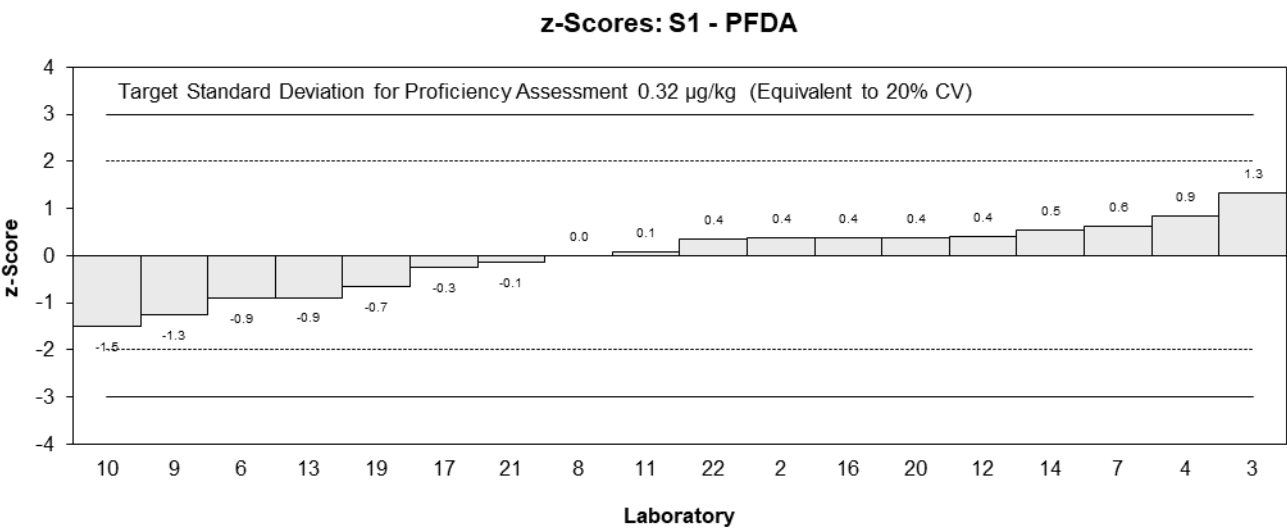
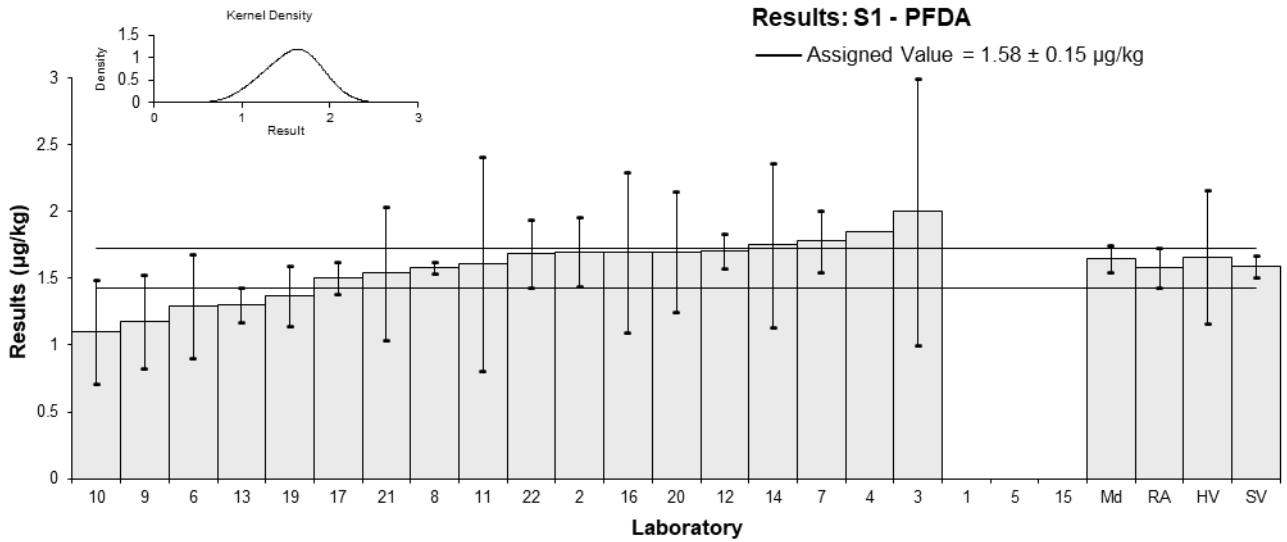


Figure 17

Table 23

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFUdA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 2	1	116		
2	< 1.0	NR	96		
3	< 2	NR	99		
4	NT	NT	NT		
5	NT	NT	NT		
6	0.581	0.174	99	-0.98	-0.75
7	<0.1	NR	49		
8	0.776	0.039	99.7	0.37	0.68
9	0.53	0.16	120	-1.33	-1.10
10	<1	NR	96.93		
11	NT	NT	NT		
12	0.76	0.05	NR	0.26	0.45
13	0.65	0.07	NR	-0.50	-0.73
14	0.6533	0.2287	105	-0.48	-0.29
15	NT	NT	NT		
16	0.76	0.3	NR	0.26	0.12
17	0.785	0.057	76	0.44	0.70
19	0.732	0.113	81	0.07	0.08
20	0.777	0.307	95.8	0.38	0.17
21	0.785	0.2	73	0.44	0.30
22	0.823	0.139	88.4	0.70	0.65

## Statistics

<b>Assigned Value</b>	0.722	0.069
<b>Spike Value</b>	0.756	0.038
<b>Homogeneity Value</b>	0.81	0.24
<b>Robust Average</b>	0.722	0.069
<b>Median</b>	0.760	0.028
<b>Mean</b>	0.718	
<b>N</b>	12	
<b>Max</b>	0.823	
<b>Min</b>	0.53	
<b>Robust SD</b>	0.095	
<b>Robust CV</b>	13%	

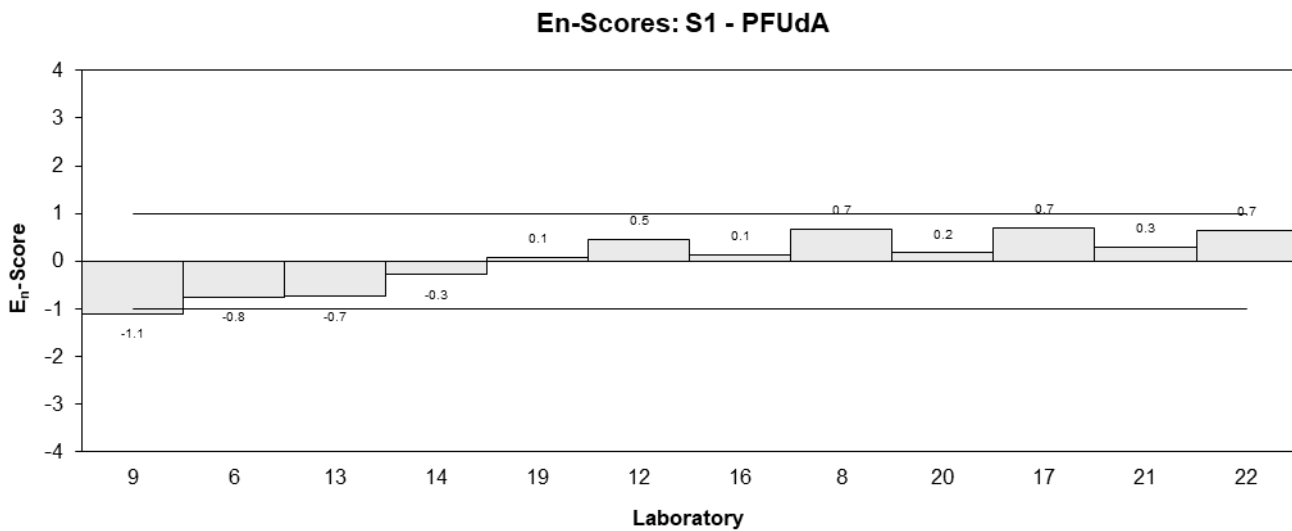
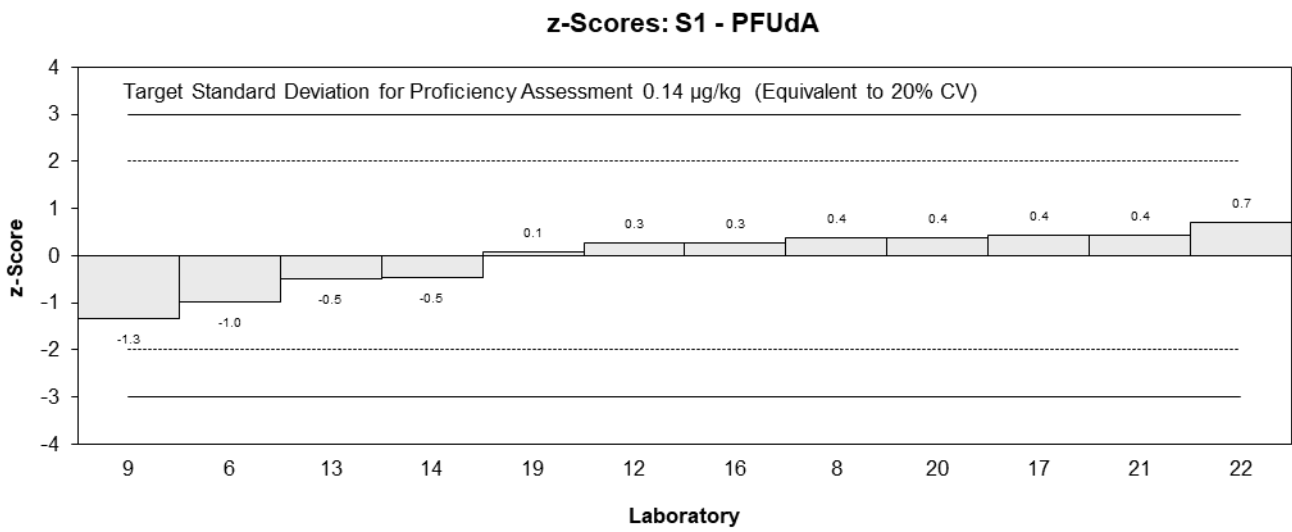
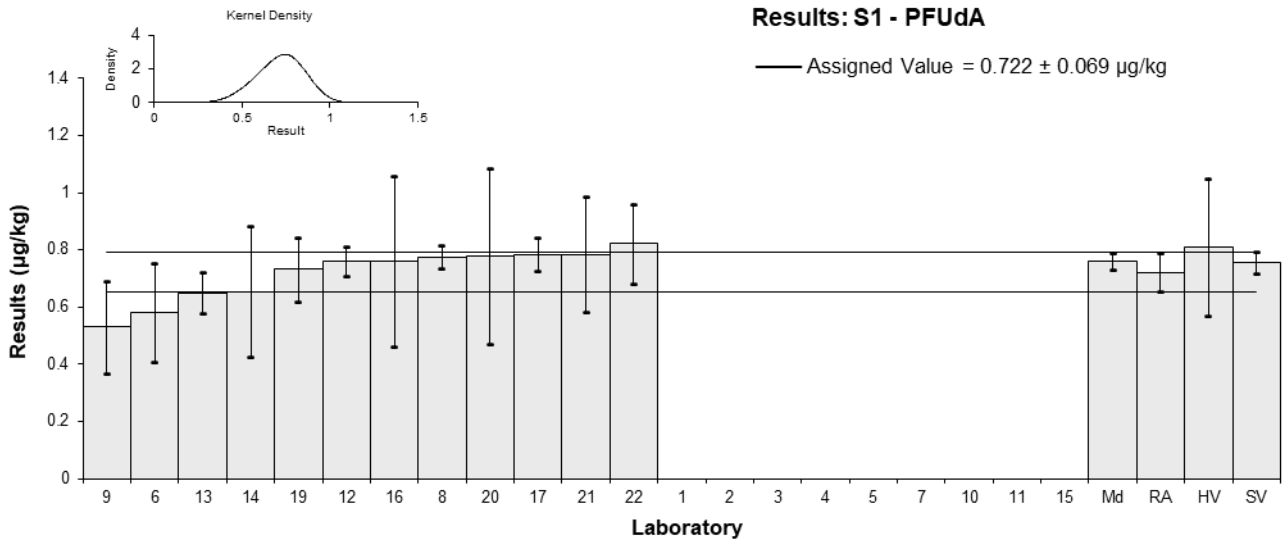


Figure 18

Table 24

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFDoA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 5	2.5	115		
2	3.4	0.14	77	0.48	0.78
3	4	2	101	1.45	0.44
4	NT	NT	NT		
5	NT	NT	NT		
6	2.379	0.714	104	-1.16	-0.90
7	3.16	1.12	31	0.10	0.05
8	3.35	0.057	102.5	0.40	0.69
9	2.14	0.65	115	-1.55	-1.29
10	2.448	0.73	88.48	-1.05	-0.80
11	NT	NT	NT		
12	3.32	0.23	NR	0.35	0.51
13	NT	NT	NT		
14	3.1792	1.1127	83	0.13	0.07
15	NT	NT	NT		
16	3.7	1.3	NR	0.97	0.44
17	3.197	0.247	76	0.16	0.22
19	2.59	0.324	91	-0.82	-1.05
20	3.22	0.736	100.8	0.19	0.15
21	2.88	0.9	63	-0.35	-0.23
22	3.47	0.763	78.2	0.60	0.44

## Statistics

<b>Assigned Value</b>	3.10	0.36
<b>Spike Value</b>	3.70	0.18
<b>Homogeneity Value</b>	3.09	0.93
<b>Robust Average</b>	3.10	0.36
<b>Median</b>	3.20	0.26
<b>Mean</b>	3.10	
<b>N</b>	15	
<b>Max</b>	4	
<b>Min</b>	2.14	
<b>Robust SD</b>	0.56	
<b>Robust CV</b>	18%	

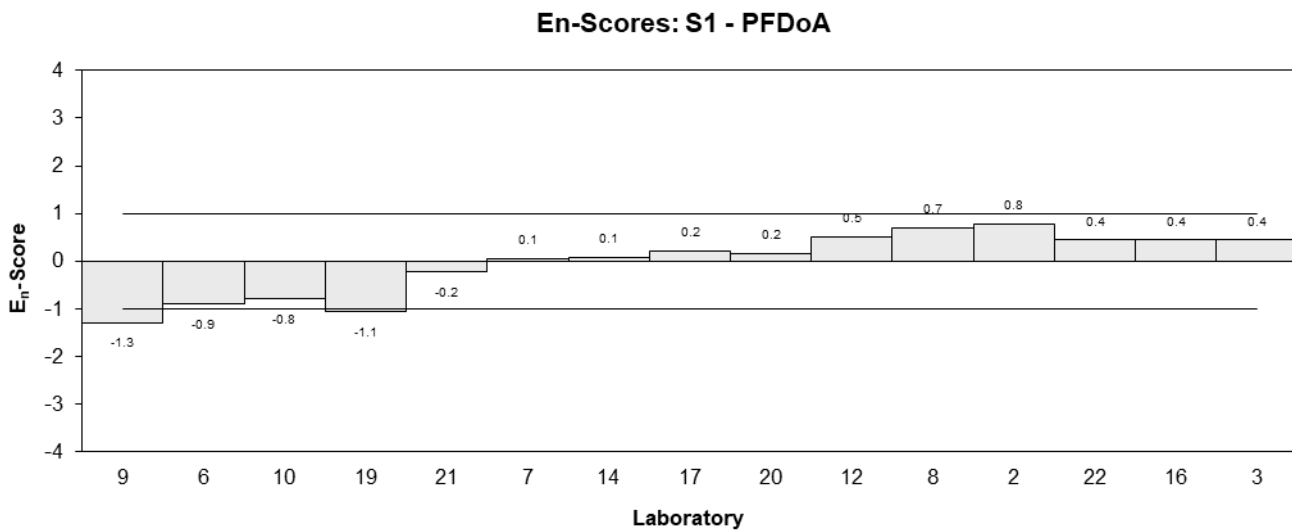
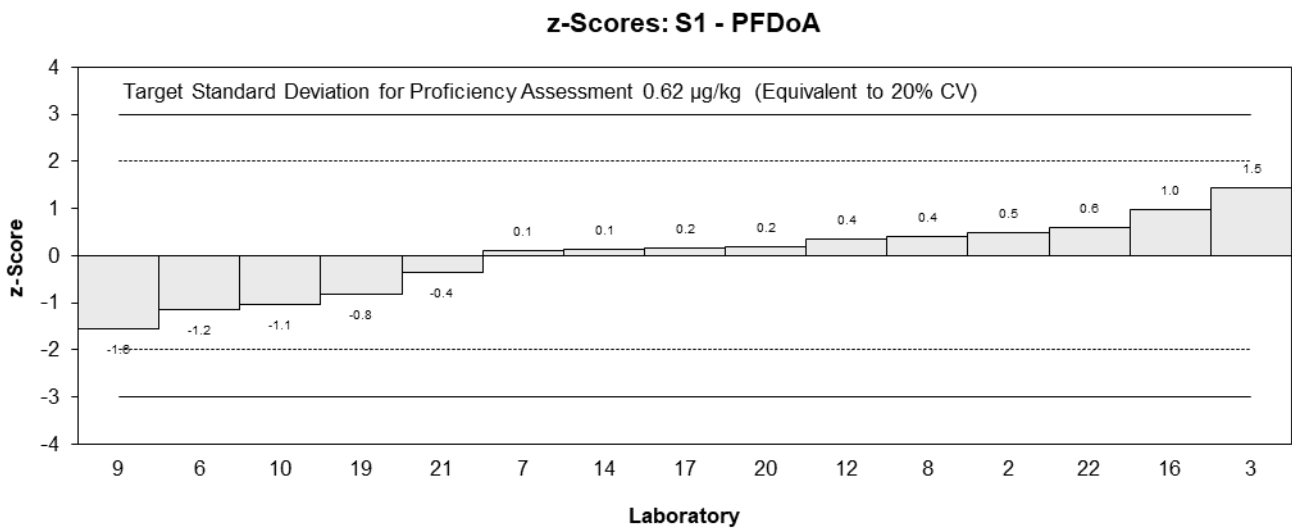
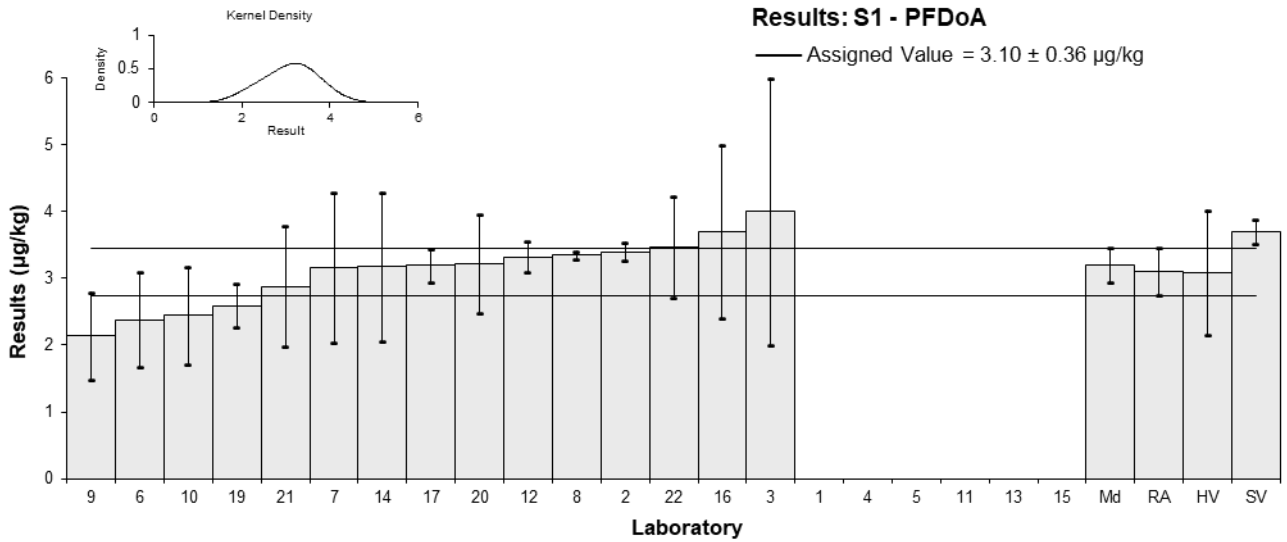


Figure 19

Table 25

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFTeDA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 5	2.5	120		
2	1.1	0.079	180	-0.83	-1.02
3	< 5	NR	113		
4	NT	NT	NT		
5	NT	NT	NT		
6	1.056	0.317	107	-1.00	-0.70
7	1.53	0.05	7	0.80	1.02
8	1.60	0.061	94.1	1.06	1.34
9	0.94	0.28	109	-1.44	-1.10
10	<2	NR	94.08		
11	1.39	0.695	NR	0.27	0.10
12	1.73	0.12	NR	1.55	1.76
13	1.2	0.70	NR	-0.45	-0.16
14	1.2525	0.4384	103	-0.26	-0.14
15	NT	NT	NT		
16	1.2	0.4	NR	-0.45	-0.27
17	1.079	0.041	54	-0.91	-1.18
19	1.07	0.163	90	-0.95	-0.97
20	1.14	0.423	130.3	-0.68	-0.38
21	1.72	0.7	34	1.52	0.55
22	1.74	0.227	26.5	1.59	1.39

## Statistics

<b>Assigned Value</b>	1.32	0.20
<b>Spike Value</b>	1.68	0.08
<b>Homogeneity Value</b>	1.91	0.76
<b>Robust Average</b>	1.32	0.20
<b>Median</b>	1.20	0.14
<b>Mean</b>	1.32	
<b>N</b>	15	
<b>Max</b>	1.74	
<b>Min</b>	0.94	
<b>Robust SD</b>	0.32	
<b>Robust CV</b>	24%	



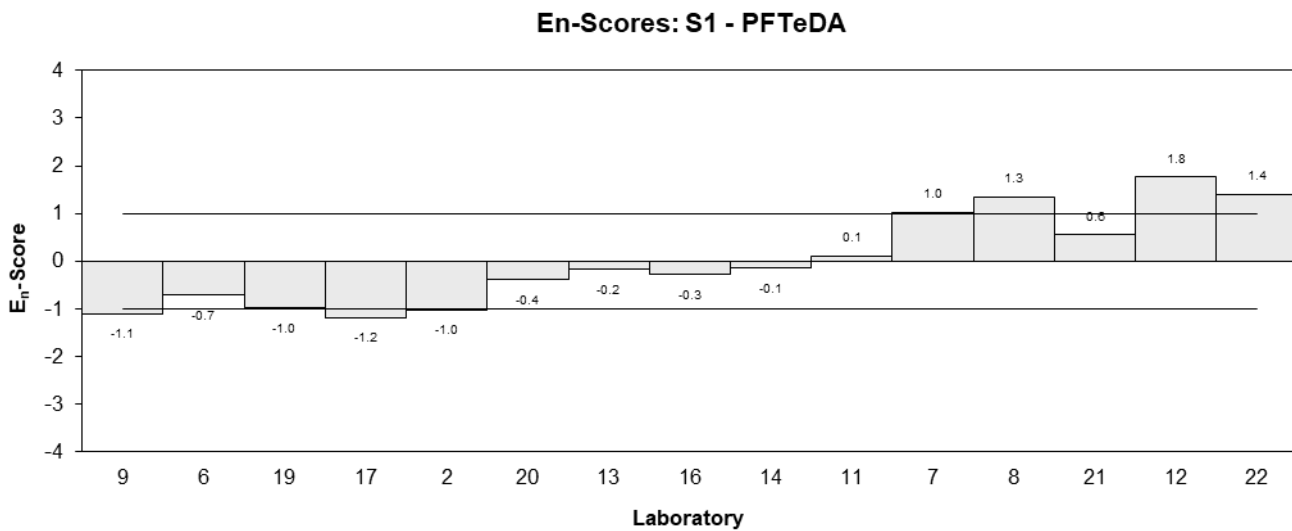
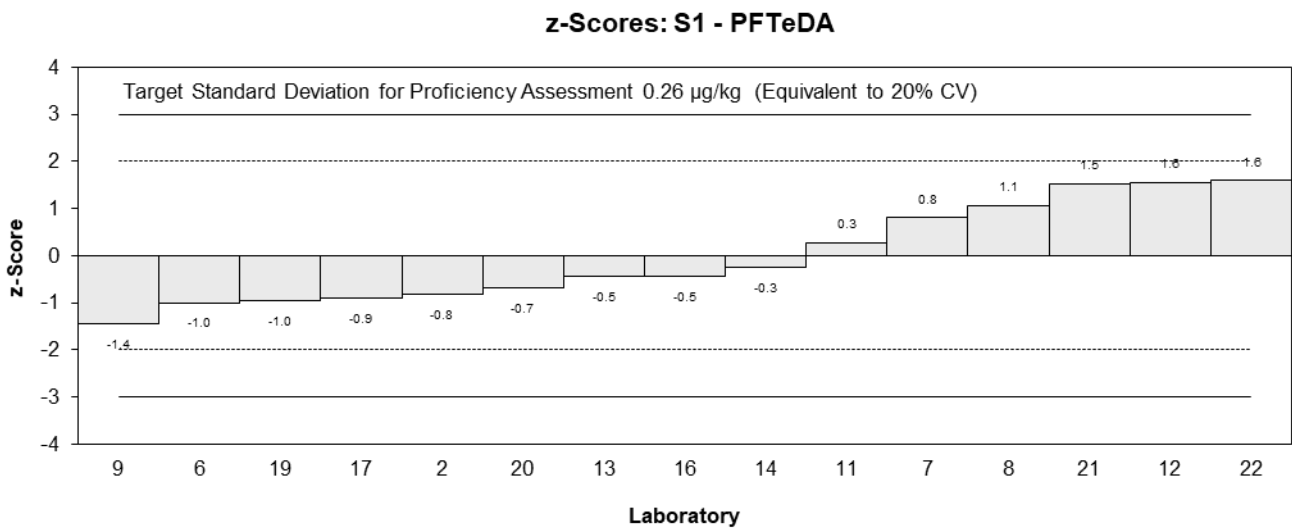
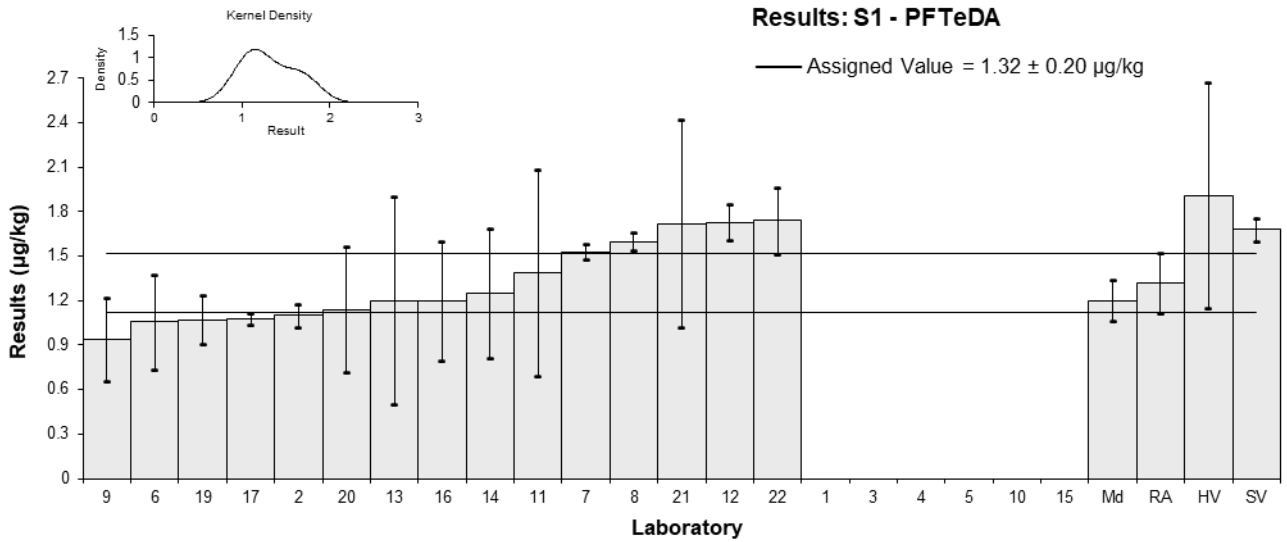


Figure 20

Table 26

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	PFOSA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	4.8	2.4	104	0.12	0.05
2	4.9	0.078	102	0.22	0.45
3	6	5	113	1.40	0.26
4	NT	NT	NT		
5	NT	NT	NT		
6	4.097	1.229	89	-0.63	-0.45
7	4.64	0.13	71	-0.05	-0.10
8	4.74	0.065	115.8	0.05	0.11
9	3.32	0.99	108	-1.46	-1.25
10	<5	NR	95.55		
11	NT	NT	NT		
12	NT	NT	NT		
13	4.4	0.75	NR	-0.31	-0.33
14	NT	NT	NT		
15	NT	NT	NT		
16	5.2	1.8	NR	0.54	0.27
17	5.521	0.283	17	0.89	1.54
19	3.67	1.08	87	-1.09	-0.87
20	4.71	0.893	31.3	0.02	0.02
21	4.51	1	72	-0.19	-0.16
22	5.12	0.717	107	0.46	0.50

## Statistics

<b>Assigned Value</b>	4.69	0.46
<b>Spike Value</b>	5.39	0.27
<b>Homogeneity Value</b>	4.3	1.3
<b>Robust Average</b>	4.69	0.46
<b>Median</b>	4.73	0.36
<b>Mean</b>	4.69	
<b>N</b>	14	
<b>Max</b>	6	
<b>Min</b>	3.32	
<b>Robust SD</b>	0.69	
<b>Robust CV</b>	15%	

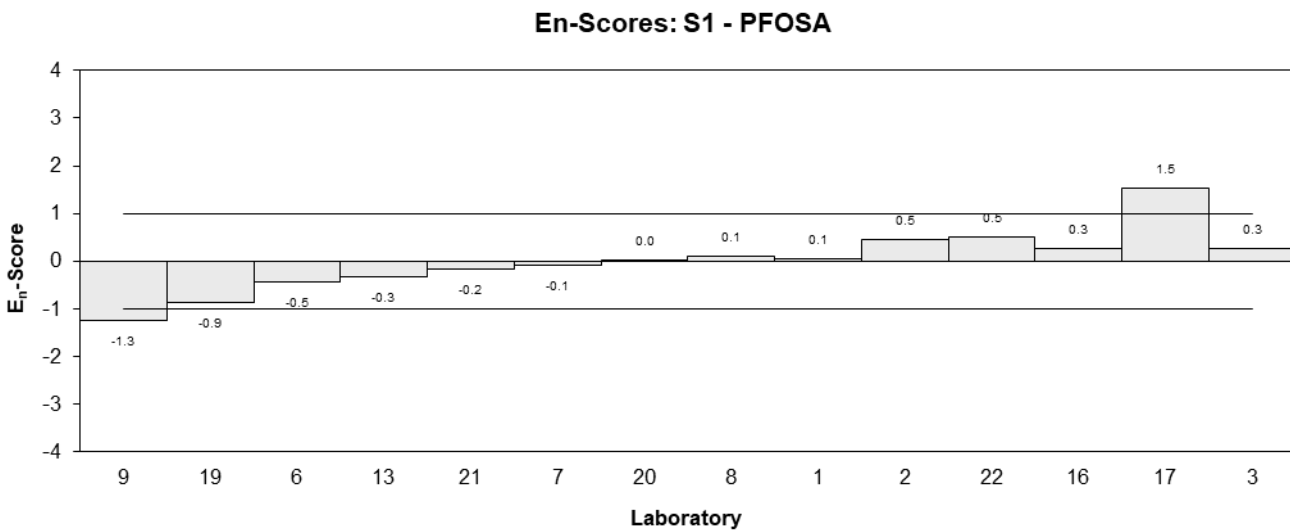
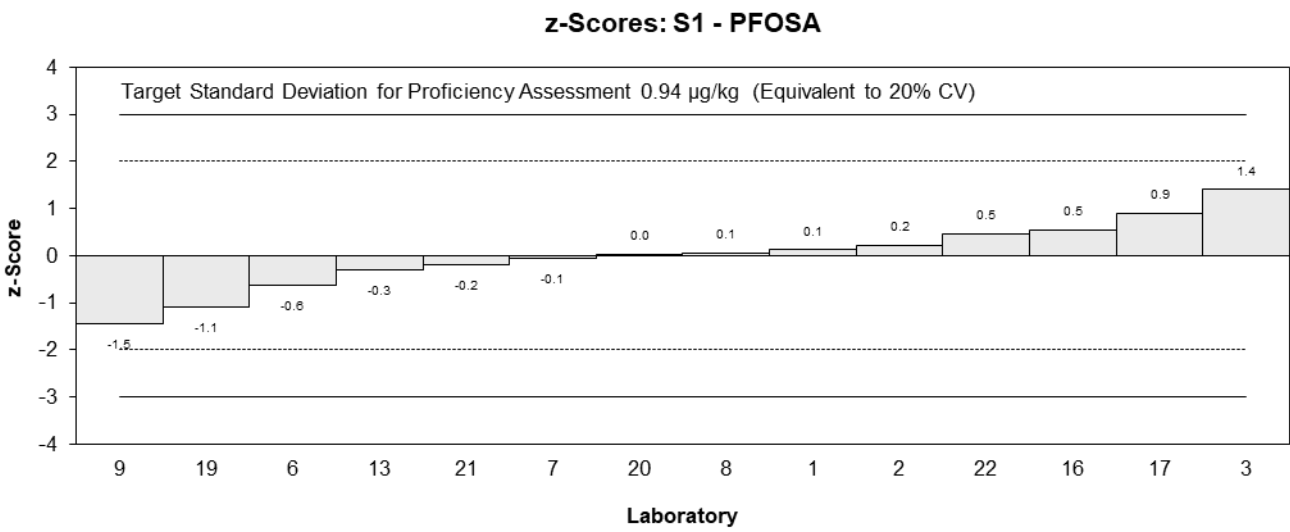
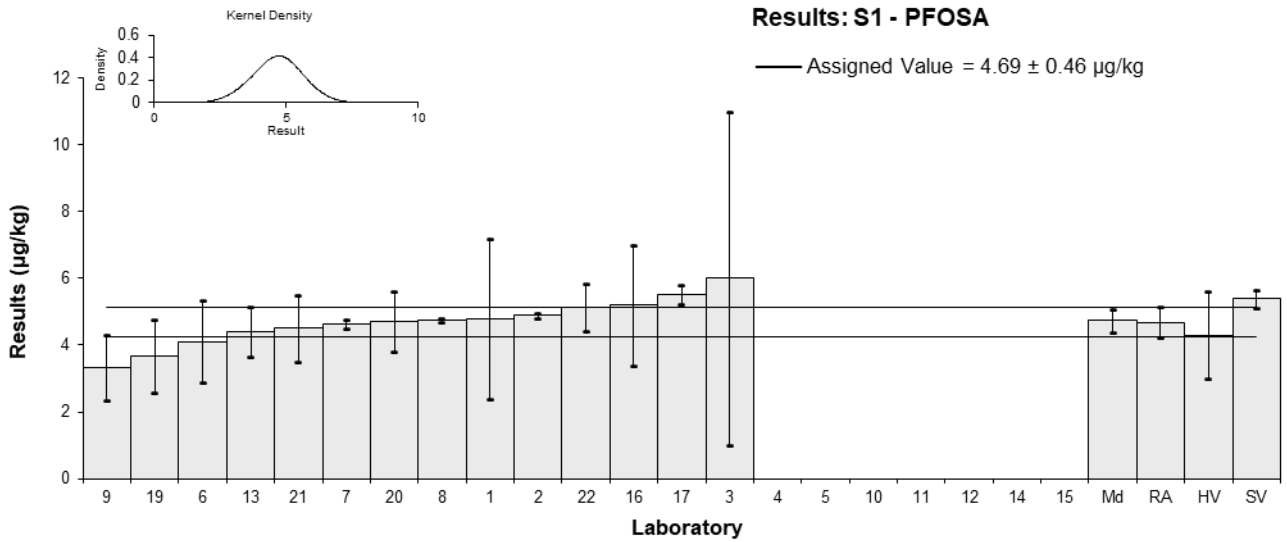


Figure 21

Table 27

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	EtFOSA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 5	2.5	119		
2	2.9	0.39	101	0.09	0.08
3	< 5	NR	91		
4	NT	NT	NT		
5	NT	NT	NT		
6	2.297	0.689	100	-0.97	-0.63
7	NT	NT	NT		
8	NT	NT	NT		
9	2.76	0.83	58	-0.16	-0.09
10	2.724	1	81.81	-0.22	-0.11
11	NT	NT	NT		
12	NT	NT	NT		
13	2.8	1.47	NR	-0.09	-0.03
14	NT	NT	NT		
15	NT	NT	NT		
16	NT	NT	NT		
17	NT	NT	NT		
19	3.44	0.832	91	1.04	0.59
20	2.15	0.213	31.3	-1.23	-1.21
21	4.59	2	26	2.00▼	
22*	8.05	1.29	15.6	9.12	3.72

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

## Statistics

<b>Assigned Value</b>	2.85	0.54
<b>Spike Value</b>	4.67	0.23
<b>Homogeneity Value</b>	4.1	1.6
<b>Robust Average</b>	3.15	0.88
<b>Max Acceptable Result</b>	6.54	
<b>Median</b>	2.80	0.62
<b>Mean</b>	3.5	
<b>N</b>	9	
<b>Max</b>	8.05	
<b>Min</b>	2.15	
<b>Robust SD</b>	1.1	
<b>Robust CV</b>	33%	

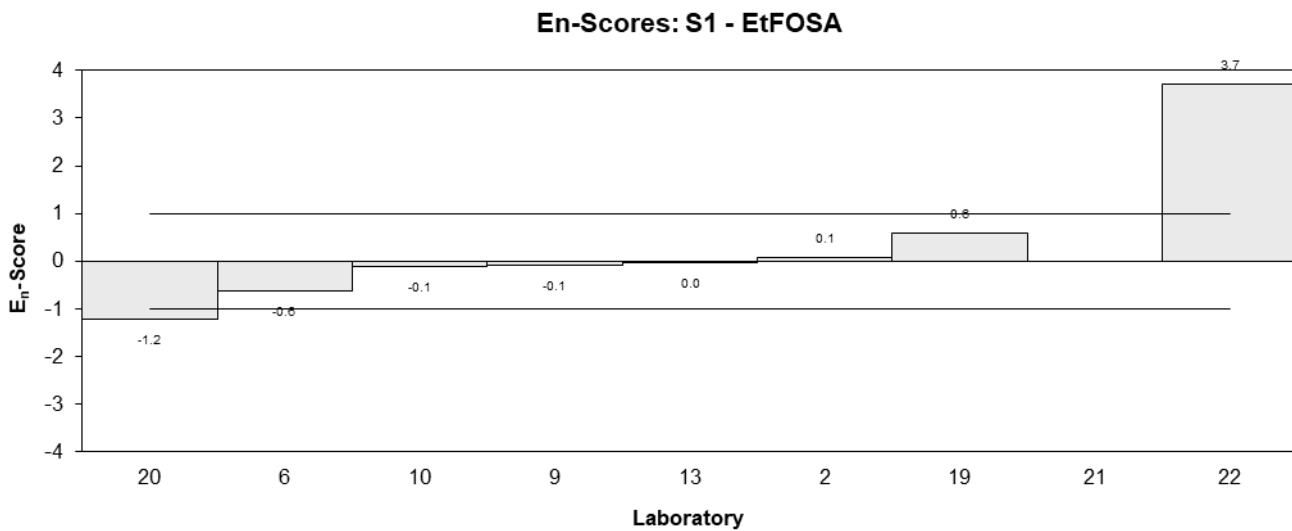
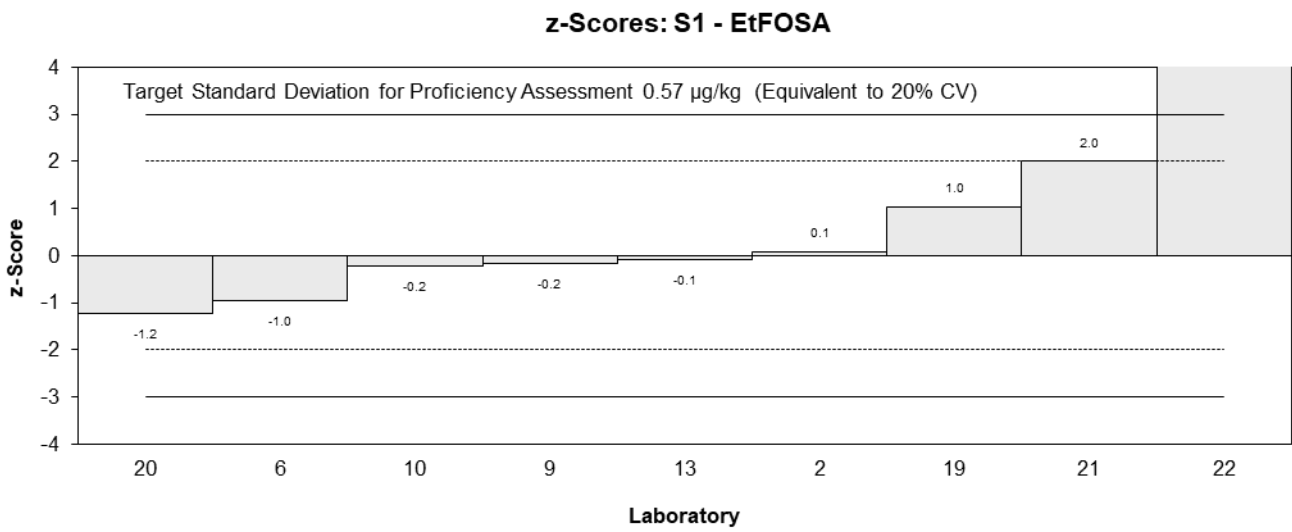
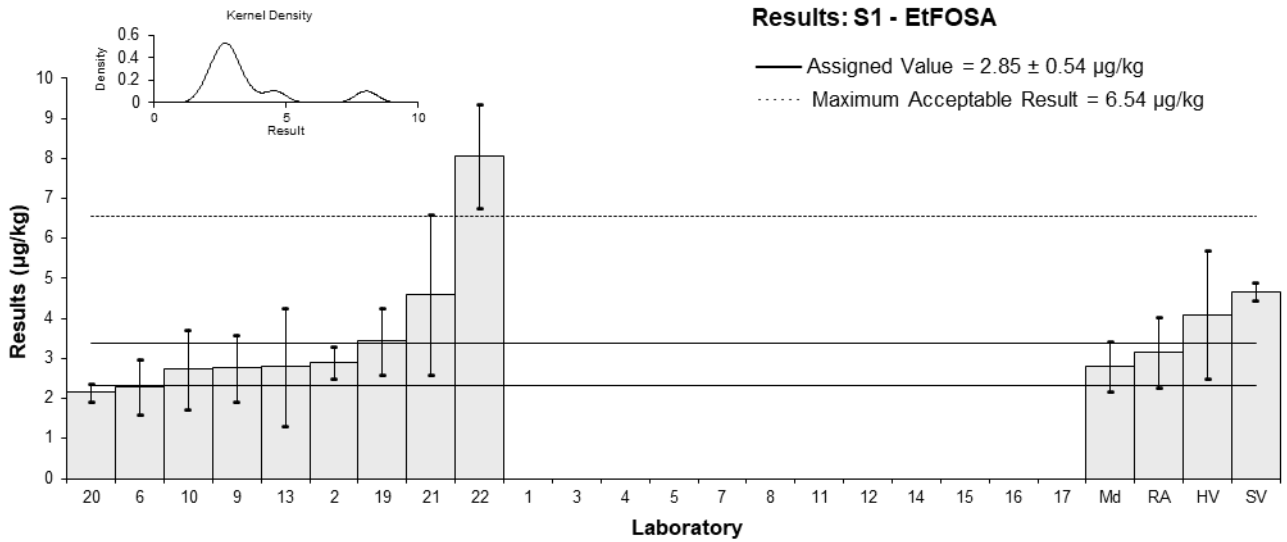


Figure 22

Table 28

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	EtFOSAA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 5	2.5	120		
2	4.4	0.28	99	0.15	0.14
3	6	2	105	2.03	0.79
4	NT	NT	NT		
5	NT	NT	NT		
6	3.507	1.052	91	-0.89	-0.56
7	3.76	0.46	35	-0.60	-0.52
8	NT	NT	NT		
9	3.05	0.92	148	-1.43	-0.96
10	3.168	0.94	133.74	-1.29	-0.86
11	NT	NT	NT		
12	NT	NT	NT		
13	5.7	1.04	NR	1.67	1.05
14	NT	NT	NT		
15	NT	NT	NT		
16	NT	NT	NT		
17	NT	NT	NT		
19	4.39	1.04	111	0.14	0.09
20	NT	NT	NT		
21	4.15	1	70	-0.14	-0.09
22	4.63	1.018	95.7	0.42	0.27

## Statistics

<b>Assigned Value</b>	4.27	0.87
<b>Spike Value</b>	4.67	0.23
<b>Homogeneity Value</b>	4.5	1.3
<b>Robust Average</b>	4.27	0.87
<b>Median</b>	4.27	0.75
<b>Mean</b>	4.28	
<b>N</b>	10	
<b>Max</b>	6	
<b>Min</b>	3.05	
<b>Robust SD</b>	1.1	
<b>Robust CV</b>	26%	

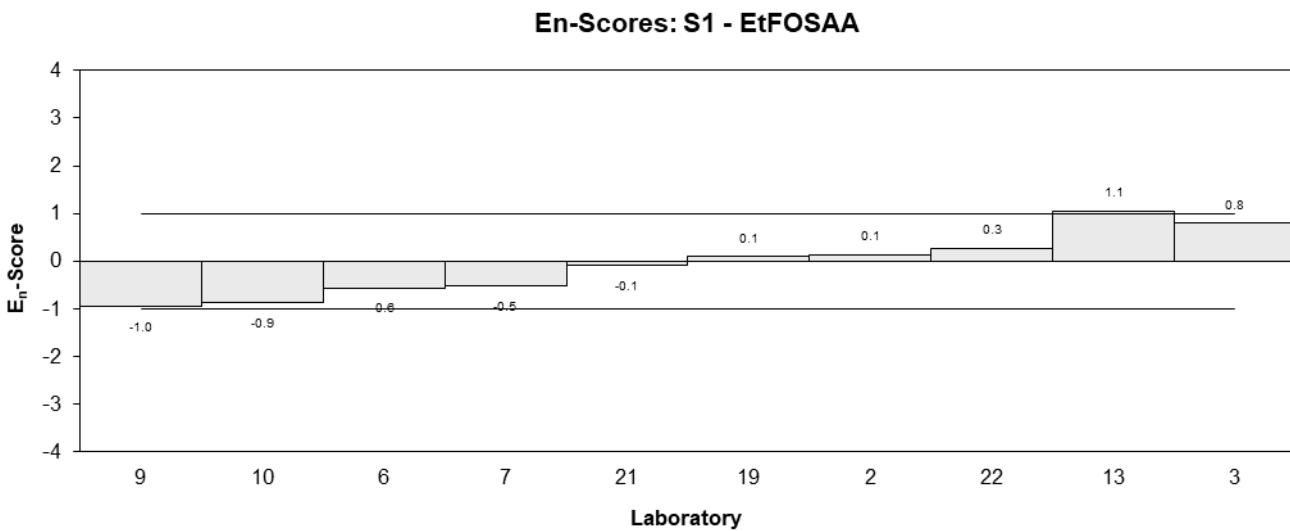
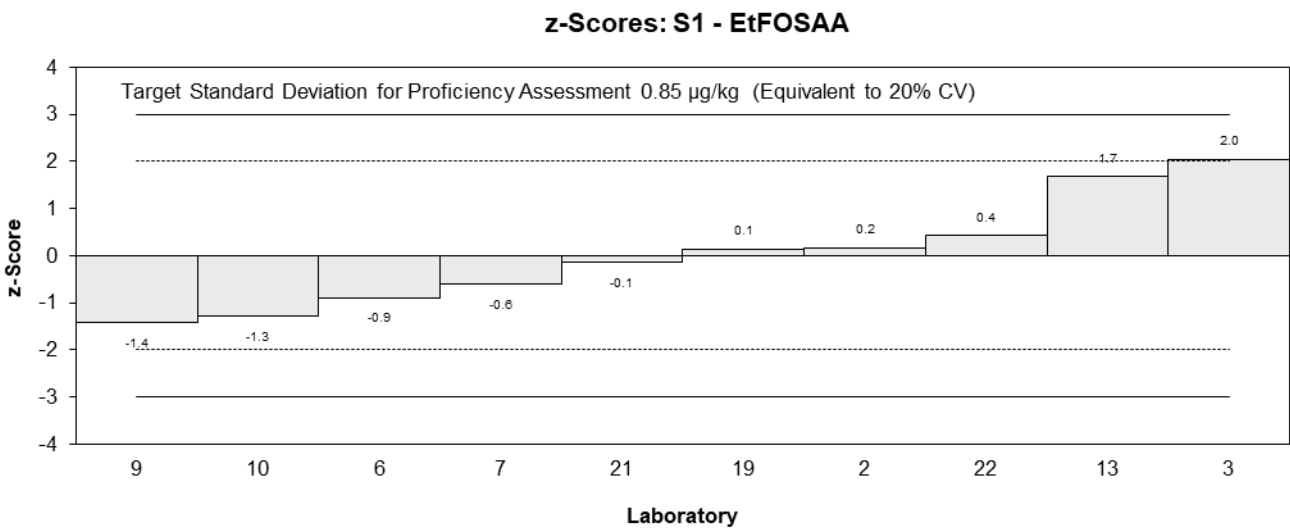
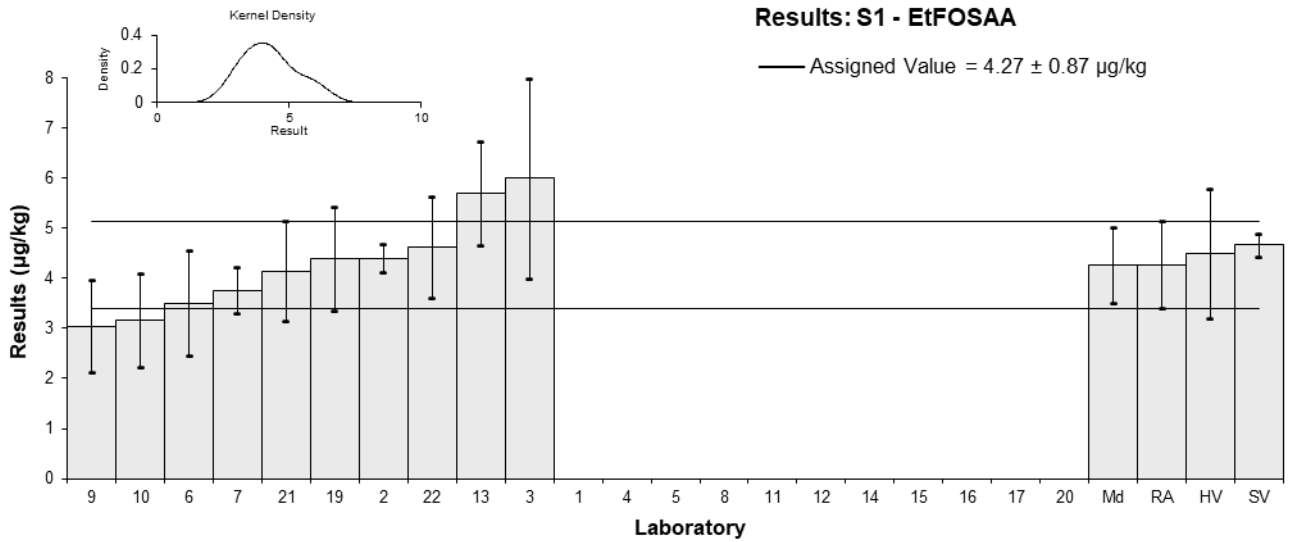


Figure 23

Table 29

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	8:2FTS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 1	0.5	106		
2	8.7	1.4	111	0.27	0.27
3	11	4	91	1.66	0.67
4	NT	NT	NT		
5	NT	NT	NT		
6	7.256	2.177	102	-0.61	-0.43
7	8.52	0.96	73	0.16	0.20
8	8.92	0.15	118.2	0.40	0.78
9	5.42	1.63	108	-1.72	-1.55
10	7.562	2.7	133.93	-0.42	-0.25
11	9.79	4.895	NR	0.93	0.31
12	NT	NT	NT		
13	8.4	0.38	NR	0.08	0.15
14	NT	NT	NT		
15	7.0	1.4	NR	-0.76	-0.77
16*	16	5.6	NR	4.69	1.37
17	8.637	0.653	66	0.23	0.36
19	6.99	0.978	115	-0.77	-0.99
20	NT	NT	NT		
21	9.13	3	50	0.53	0.28
22	8.25	1.15	67.3	-0.01	-0.01

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	8.26	0.83
<b>Spike Value</b>	9.00	0.45
<b>Homogeneity Value</b>	10.3	3.1
<b>Robust Average</b>	8.45	0.95
<b>Median</b>	8.52	0.92
<b>Mean</b>	8.8	
<b>N</b>	15	
<b>Max</b>	16	
<b>Min</b>	5.42	
<b>Robust SD</b>	1.5	
<b>Robust CV</b>	17%	



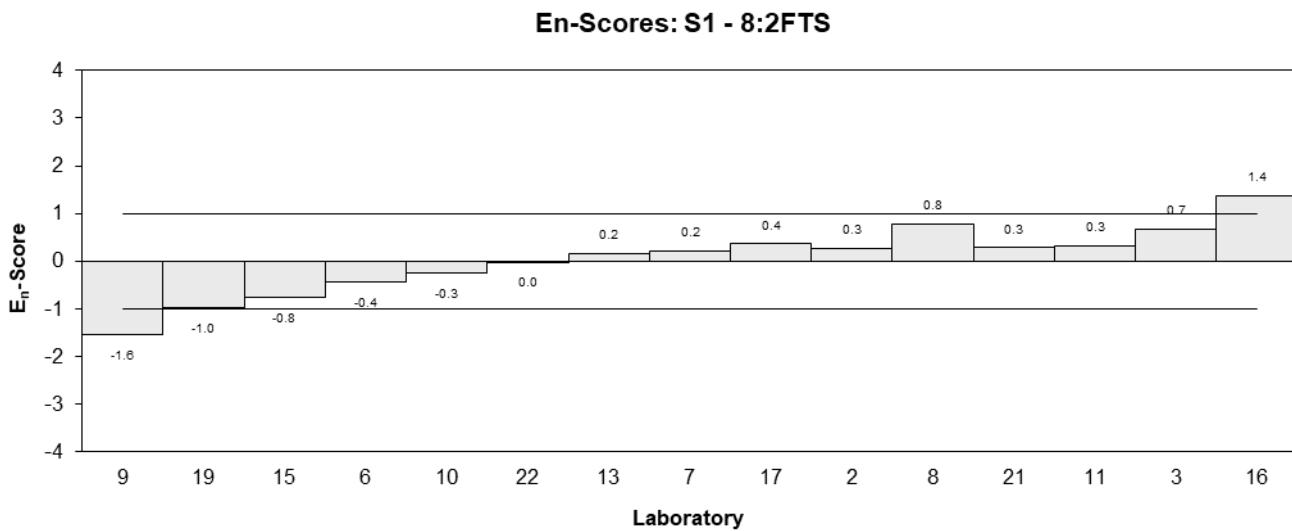
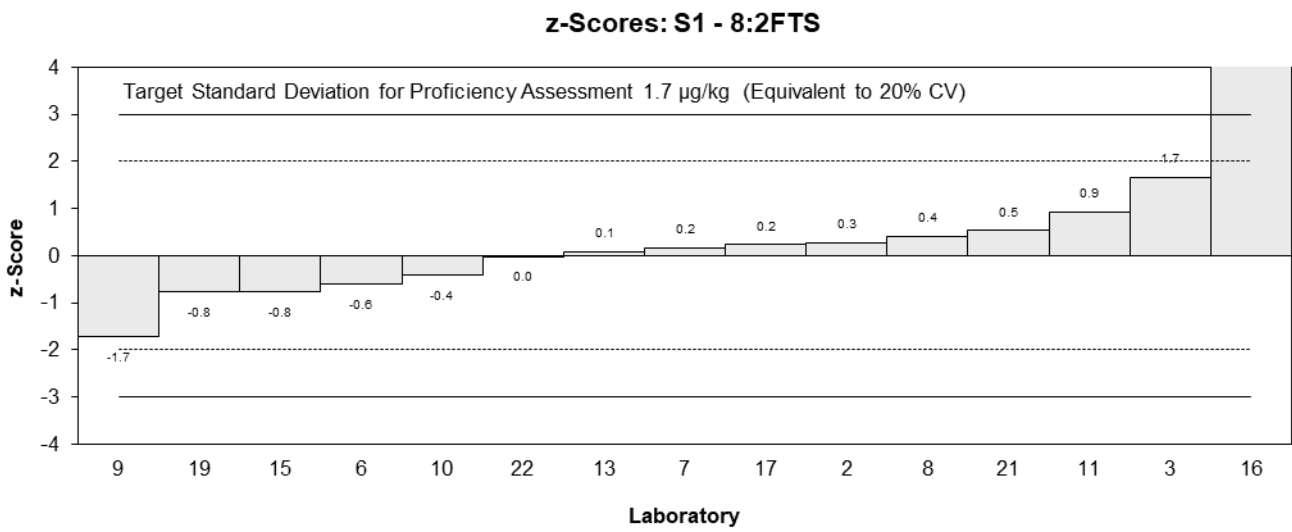
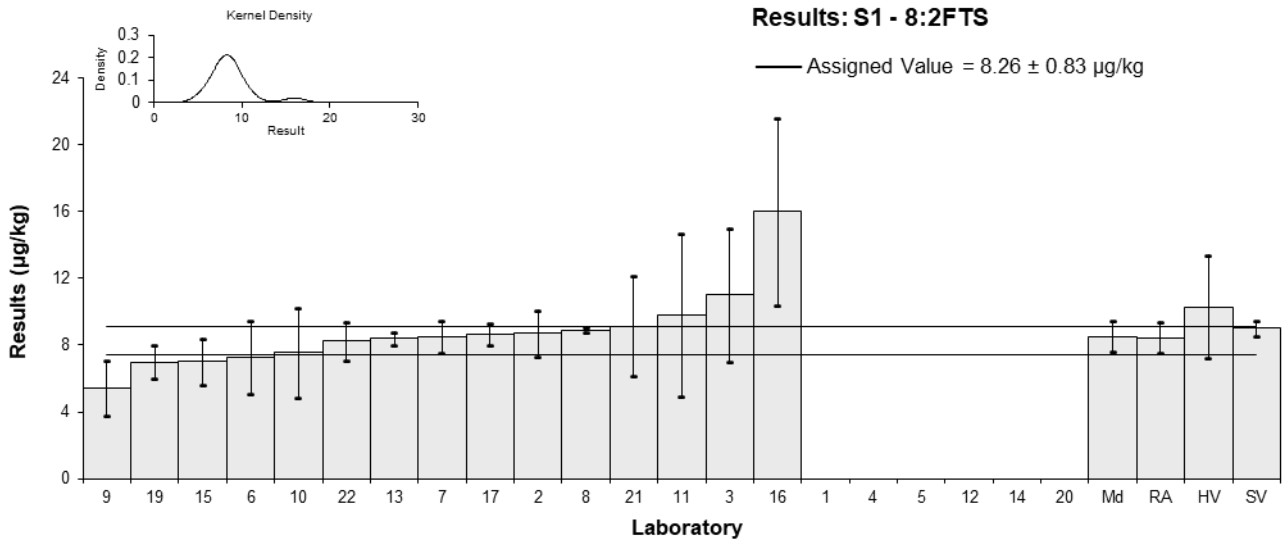


Figure 24

Table 30

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	GenX
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	NT	NT	NT		
2	16	3.1	105	1.30	0.85
3	< 50	NR	112		
4	NT	NT	NT		
5	NT	NT	NT		
6	8.637	2.591	99	-1.60	-1.17
7	NT	NT	NT		
8	13.3	0.057	95.9	0.24	0.26
9	8.03	2.4	60	-1.84	-1.40
10	NT	NT	NT		
11	11.19	5.595	NR	-0.59	-0.25
12	12.86	0.92	NR	0.06	0.06
13	17	7.26	NR	1.69	0.56
14	NT	NT	NT		
15	NT	NT	NT		
16	NT	NT	NT		
17	13.753	0.612	78	0.41	0.44
19	11.6	3.1	69	-0.43	-0.28
20	NT	NT	NT		
21	12.9	4	69	0.08	0.04
22	14.3	3.01	63.5	0.63	0.42

## Statistics

<b>Assigned Value</b>	12.7	2.3
<b>Spike Value</b>	14.1	0.7
<b>Homogeneity Value</b>	12.4	3.7
<b>Robust Average</b>	12.7	2.3
<b>Median</b>	12.9	1.6
<b>Mean</b>	12.7	
<b>N</b>	11	
<b>Max</b>	17	
<b>Min</b>	8.03	
<b>Robust SD</b>	3.1	
<b>Robust CV</b>	25%	

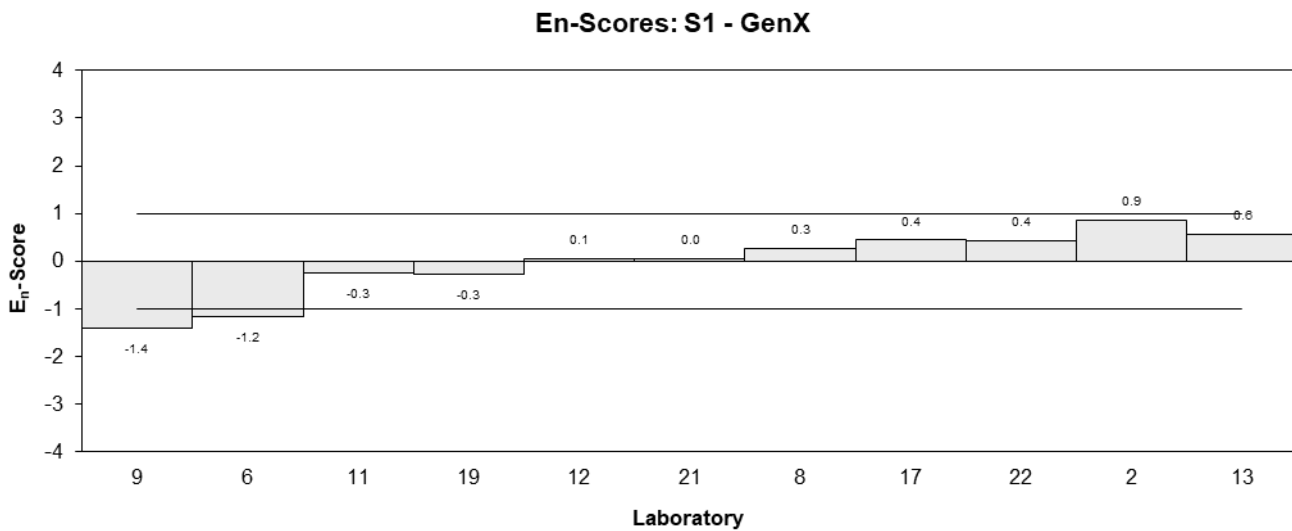
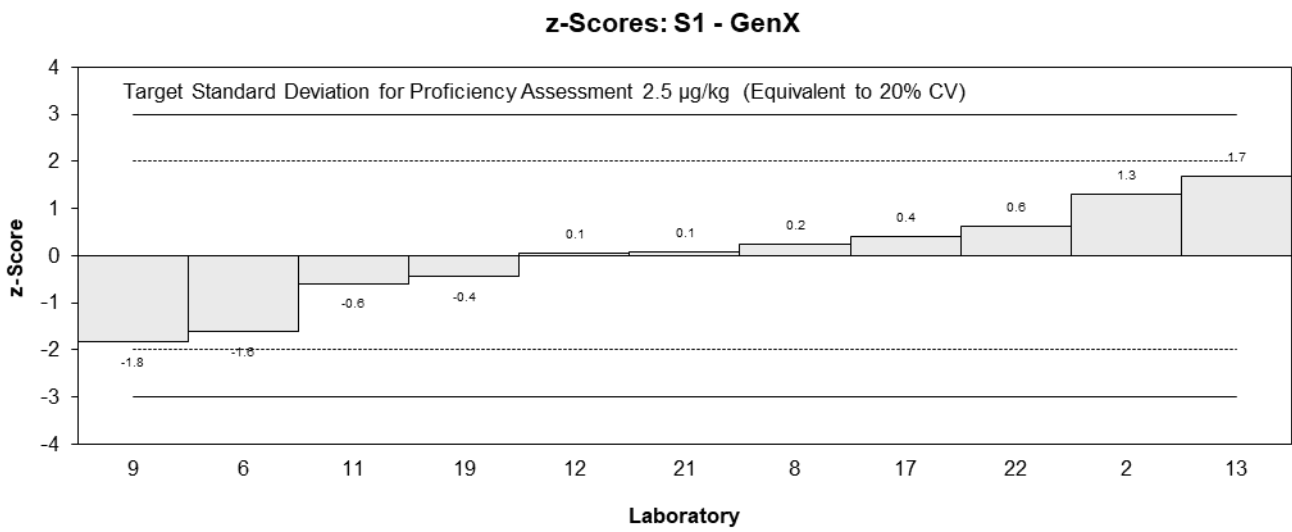
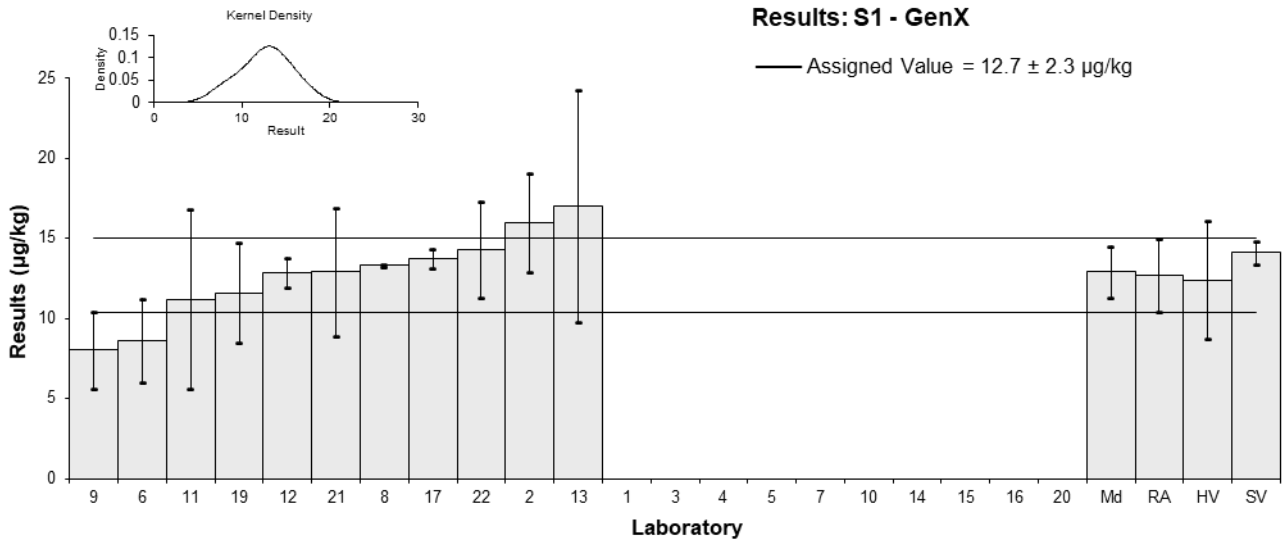


Figure 25

Table 31

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	ADONA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	NT	NT	NT		
2	15	0.54	112	-0.19	-0.22
3	19	6	99	1.09	0.52
4	20.679	NR	NR	1.63	1.88
5	NT	NT	NT		
6	11.158	3.347	88	-1.42	-1.03
7	16.28	2.55	60	0.22	0.18
8	17.8	0.052	NR	0.71	0.81
9	9.91	2.98	112	-1.82	-1.41
10	NT	NT	NT		
11	NT	NT	NT		
12	12.65	0.9	NR	-0.95	-1.04
13	10	0.52	NR	-1.79	-2.04
14	NT	NT	NT		
15	NT	NT	NT		
16	20	7.0	NR	1.41	0.59
17	17.693	0.341	76	0.67	0.77
19	14.8	4.23	92	-0.26	-0.16
20	NT	NT	NT		
21	17.6	5	NR	0.64	0.35
22	16.1	7.01	63.5	0.16	0.07

## Statistics

<b>Assigned Value</b>	15.6	2.7
<b>Spike Value</b>	17.6	0.9
<b>Homogeneity Value</b>	14.5	4.4
<b>Robust Average</b>	15.6	2.7
<b>Median</b>	16.2	2.2
<b>Mean</b>	15.6	
<b>N</b>	14	
<b>Max</b>	20.679	
<b>Min</b>	9.91	
<b>Robust SD</b>	4.0	
<b>Robust CV</b>	26%	

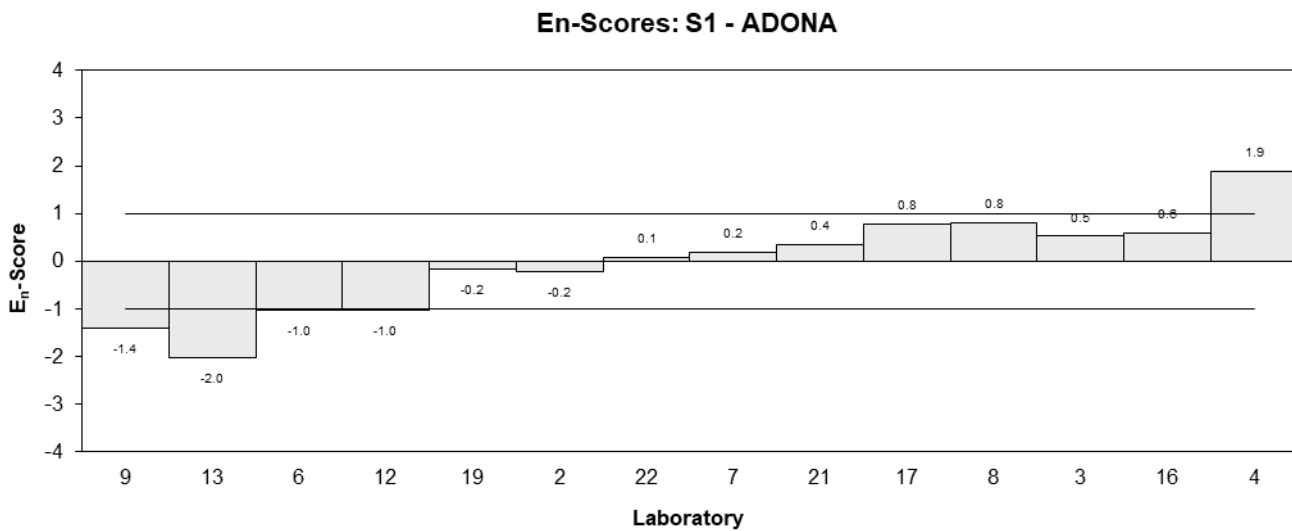
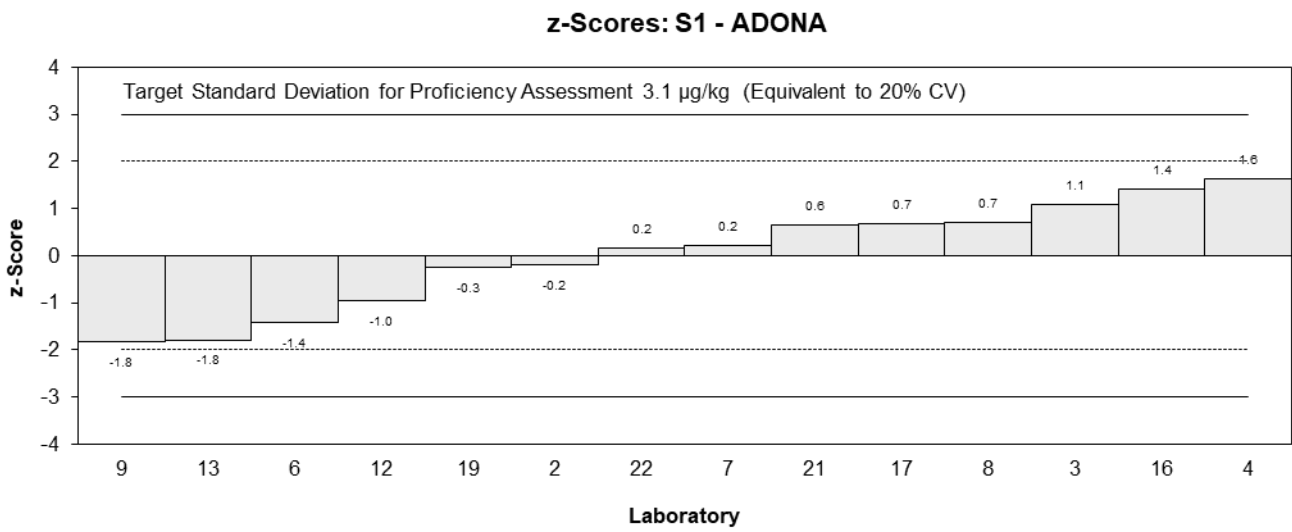
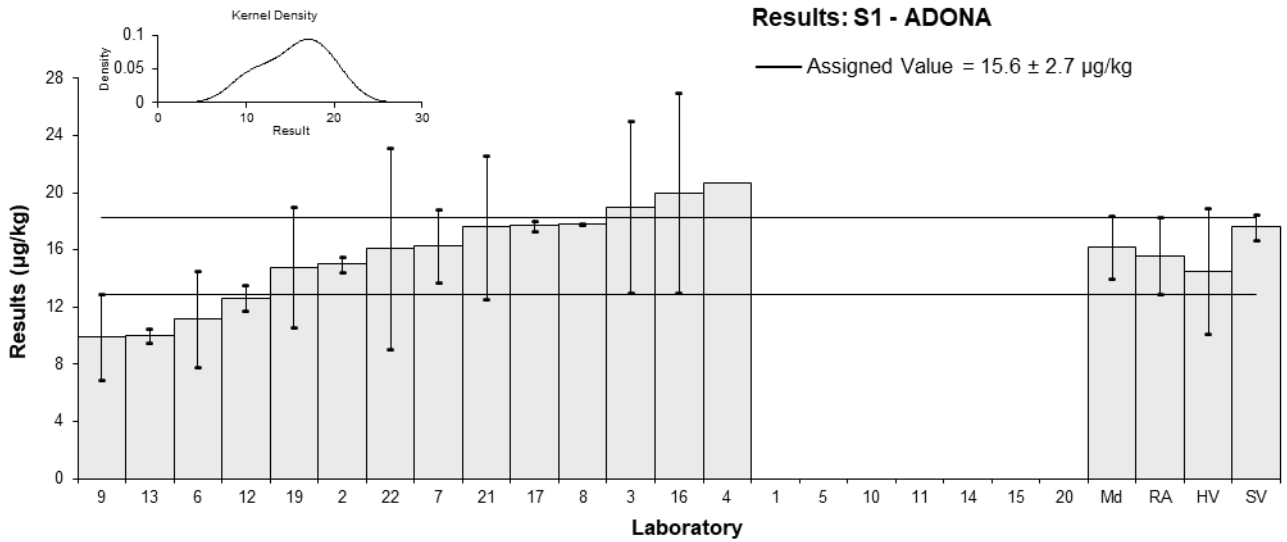


Figure 26

Table 32

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	9CI-PF3ONS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	NT	NT	NT		
2	20	0.45	102	0.13	0.18
3	21	6	100	0.38	0.23
4	25.6	NR	NR	1.56	2.26
5	NT	NT	NT		
6	14.591	4.377	100	-1.26	-0.95
7	19.02	2.62	100	-0.12	-0.13
8	19.0	0.080	NR	-0.13	-0.19
9	10.4	3.12	105	-2.33	-2.21
10	NT	NT	NT		
11	NT	NT	NT		
12	19.93	1.42	NR	0.11	0.14
13*	31	2.36	NR	2.95	3.21
14	NT	NT	NT		
15	NT	NT	NT		
16	NT	NT	NT		
17	20.789	0.959	92	0.33	0.45
19	16.8	3.9	92	-0.69	-0.57
20	NT	NT	NT		
21	20.3	8	NR	0.21	0.09
22	23.8	6.66	63.5	1.10	0.60

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	19.5	2.7
<b>Spike Value</b>	21.8	1.1
<b>Homogeneity Value</b>	19.7	7.9
<b>Robust Average</b>	20.1	3.0
<b>Median</b>	20.0	1.0
<b>Mean</b>	20.2	
<b>N</b>	13	
<b>Max</b>	31	
<b>Min</b>	10.4	
<b>Robust SD</b>	4.3	
<b>Robust CV</b>	21%	

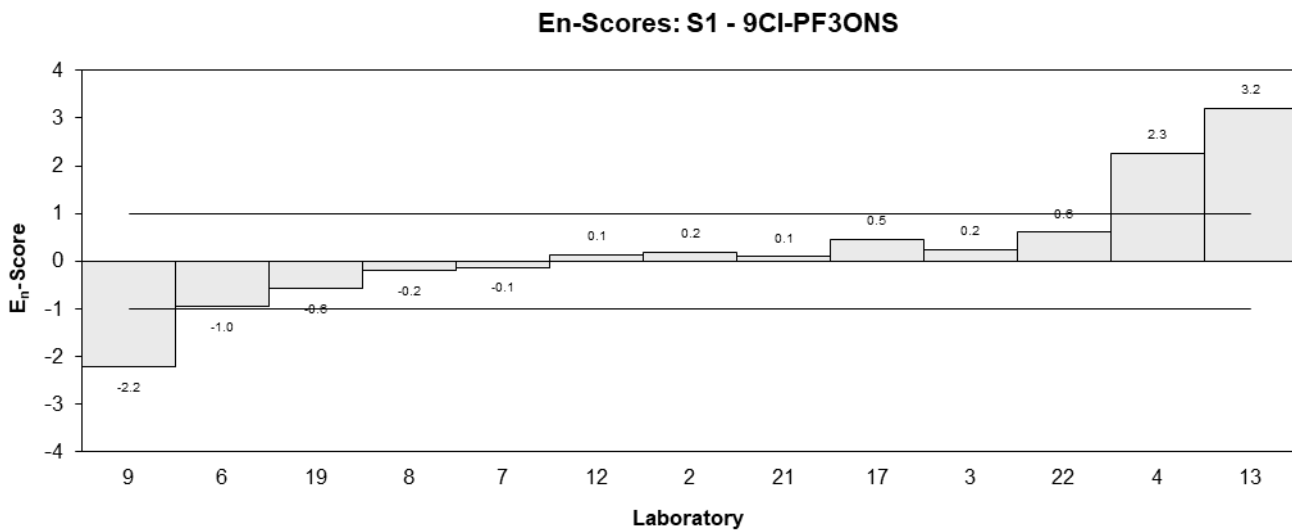
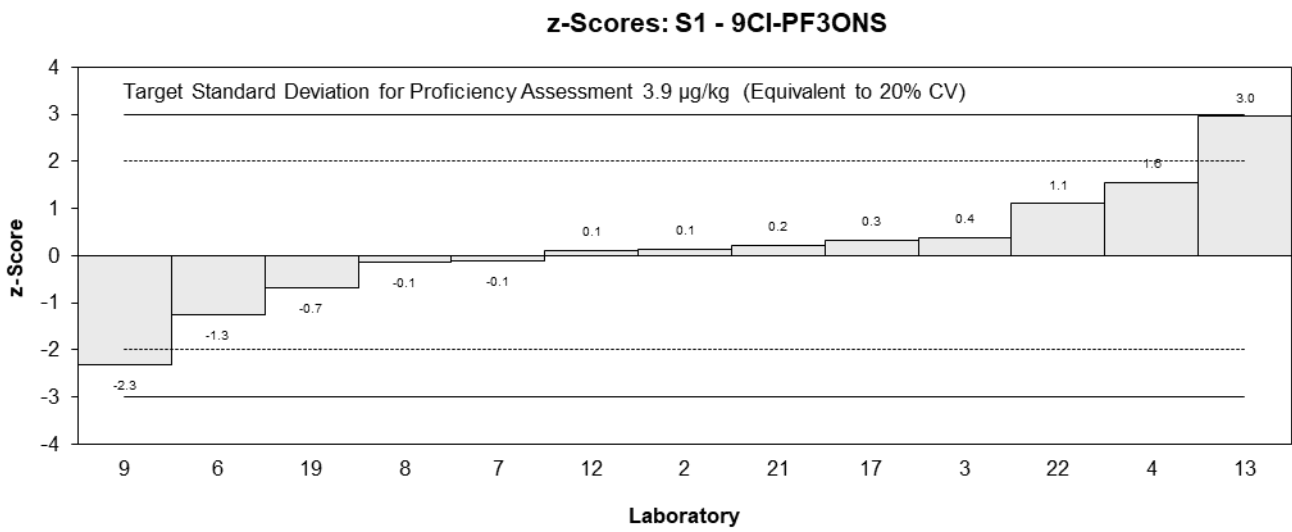
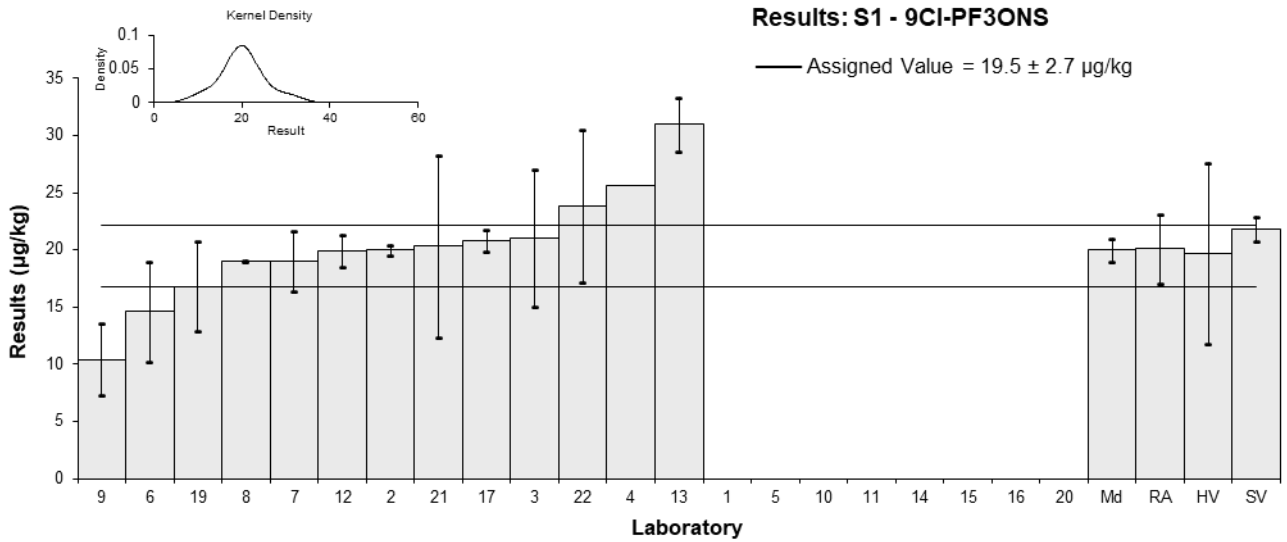


Figure 27

Table 33

## Sample Details

<b>Sample No.</b>	S1
<b>Matrix</b>	Fish
<b>Analyte</b>	11Cl-PF3OUdS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	NT	NT	NT		
2	25	0.014	96	0.36	0.39
3	29	7	100	1.22	0.69
4	31.829	NR	NR	1.83	1.94
5	NT	NT	NT		
6	19.045	5.714	88	-0.91	-0.59
7	17.35	4.85	100	-1.28	-0.91
8	24.0	0.091	NR	0.15	0.16
9	11.94	3.58	105	-2.44	-2.00
10	NT	NT	NT		
11	NT	NT	NT		
12	19.91	1.42	NR	-0.73	-0.73
13	31	4.01	NR	1.65	1.29
14	NT	NT	NT		
15	NT	NT	NT		
16	NT	NT	NT		
17	22.731	1.476	92	-0.12	-0.12
19	18.2	6.89	92	-1.09	-0.62
20	NT	NT	NT		
21	23	9	NR	-0.06	-0.03
22	28.5	9.4	63.5	1.12	0.50

## Statistics

<b>Assigned Value</b>	23.3	4.4
<b>Spike Value</b>	26.4	1.3
<b>Homogeneity Value</b>	22.1	8.8
<b>Robust Average</b>	23.3	4.4
<b>Median</b>	23.0	4.9
<b>Mean</b>	23.2	
<b>N</b>	13	
<b>Max</b>	31.829	
<b>Min</b>	11.94	
<b>Robust SD</b>	6.4	
<b>Robust CV</b>	27%	



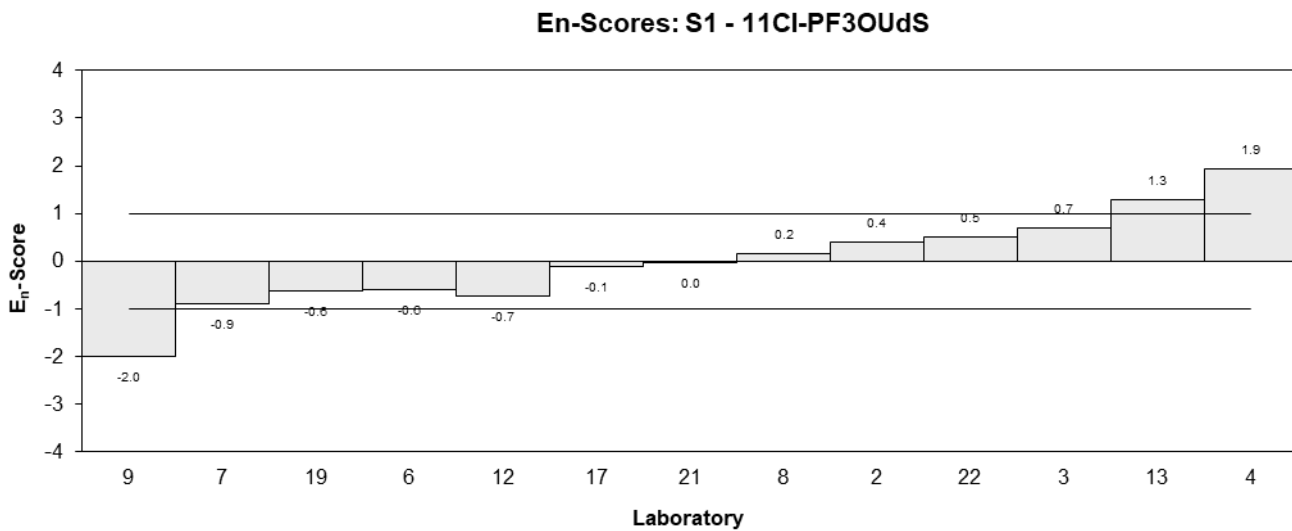
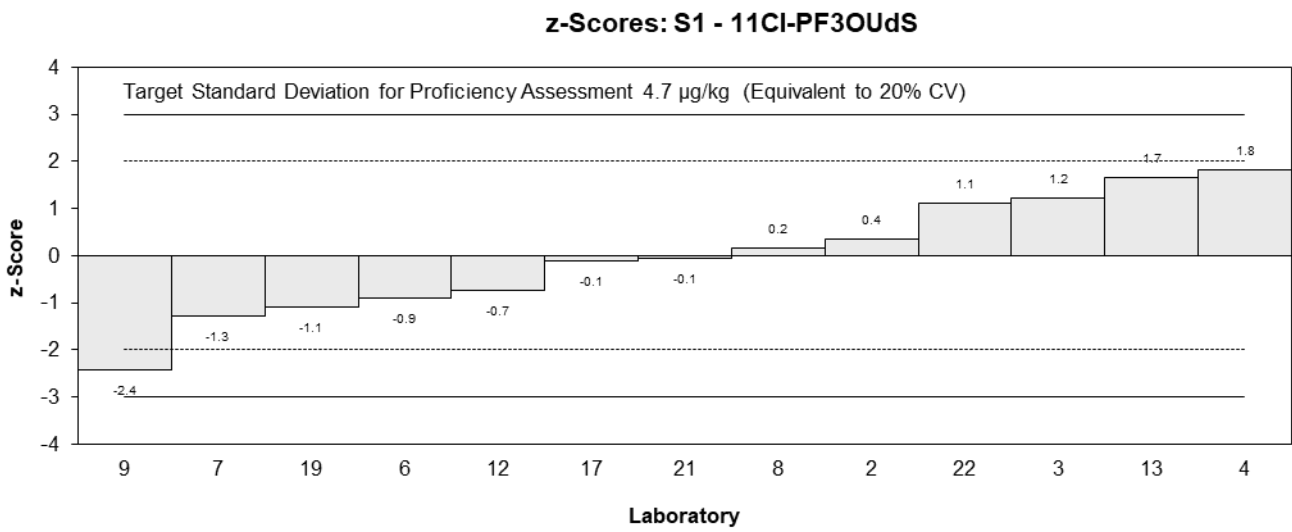
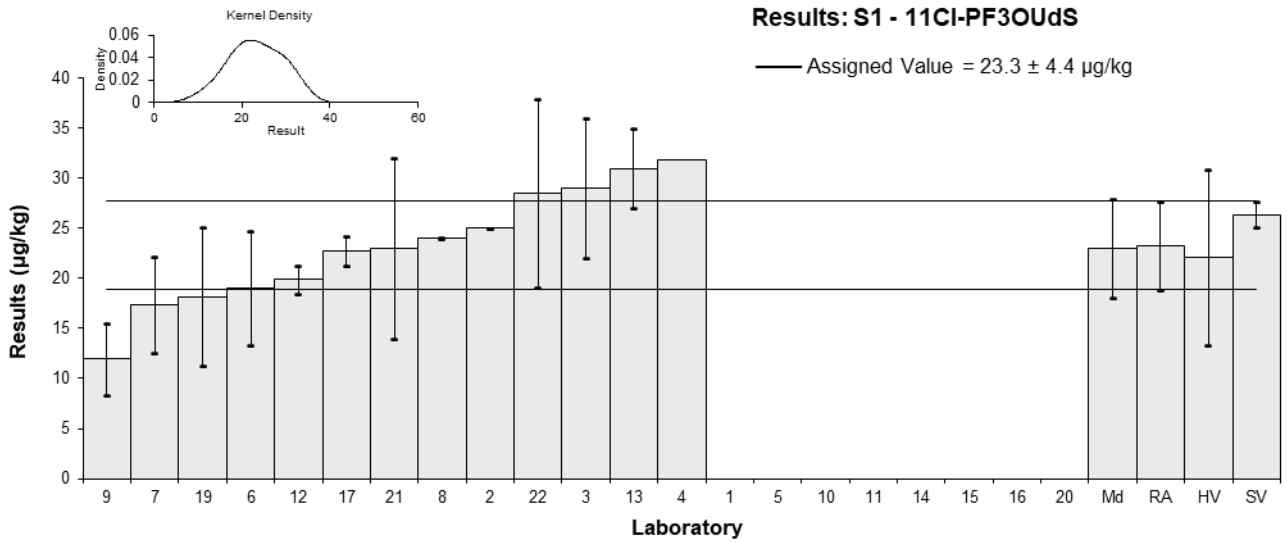


Figure 28

Table 34

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFBS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	3.7	1.9	100	-0.40	-0.17
2	4.4	0.38	110	0.47	0.77
3	4	2	113	-0.02	-0.01
4	NS	NS	NS		
5	NT	NT	NT		
6	3.527	1.058	84	-0.61	-0.45
7	4.82	0.53	80	1.00	1.30
8	4.10	0.071	101.4	0.10	0.25
9	4.01	1.2	63	-0.01	-0.01
10	4.08	1.39	81.57	0.07	0.04
11	4.28	2.14	NR	0.32	0.12
12	5.48	0.39	NR	1.82	2.93
13	3.5	0.574	NR	-0.65	-0.80
14	4.1321	1.4462	78	0.14	0.08
15	3.1	0.62	NR	-1.14	-1.33
16	4.5	1.6	NR	0.60	0.29
17	4.417	0.096	99	0.49	1.22
19	4.29	1.22	88	0.34	0.21
20	2.33	0.521	95.6	-2.10	-2.79
21	3.73	1	90	-0.36	-0.28
22	3.68	0.516	93.4	-0.42	-0.56

## Statistics

<b>Assigned Value</b>	4.02	0.31
<b>Spike Value</b>	4.62	0.23
<b>Homogeneity Value</b>	3.7	1.1
<b>Robust Average</b>	4.02	0.31
<b>Median</b>	4.08	0.30
<b>Mean</b>	4.00	
<b>N</b>	19	
<b>Max</b>	5.48	
<b>Min</b>	2.33	
<b>Robust SD</b>	0.54	
<b>Robust CV</b>	14%	

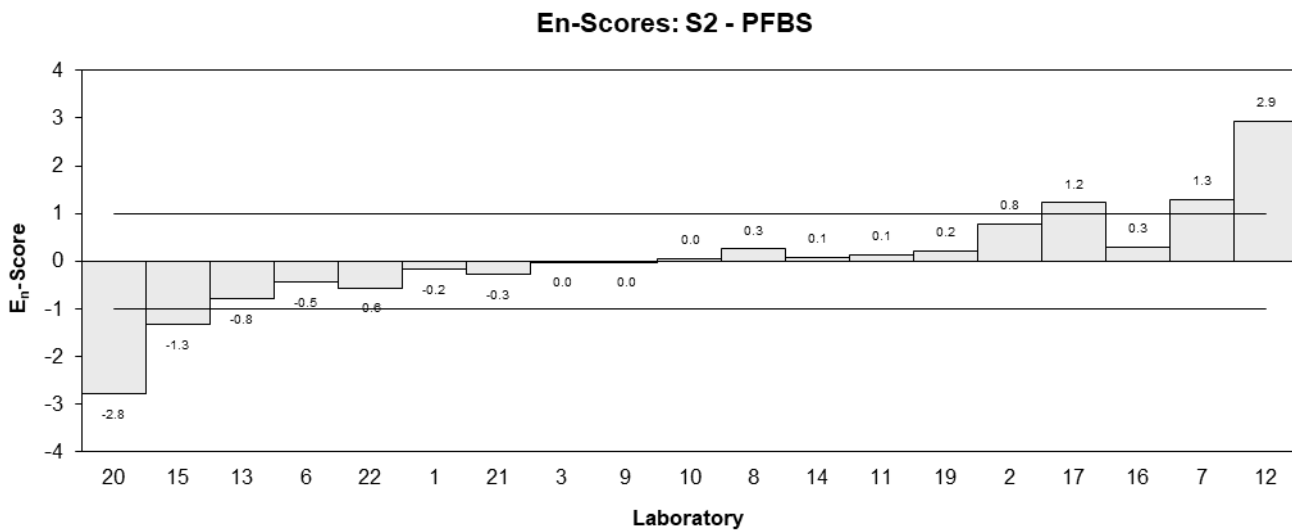
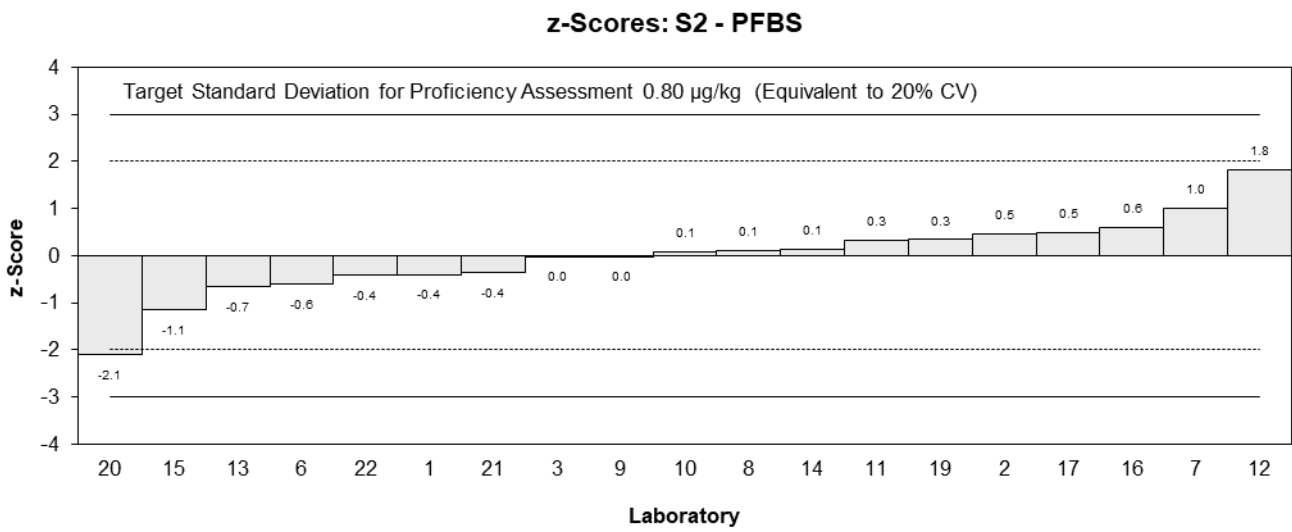
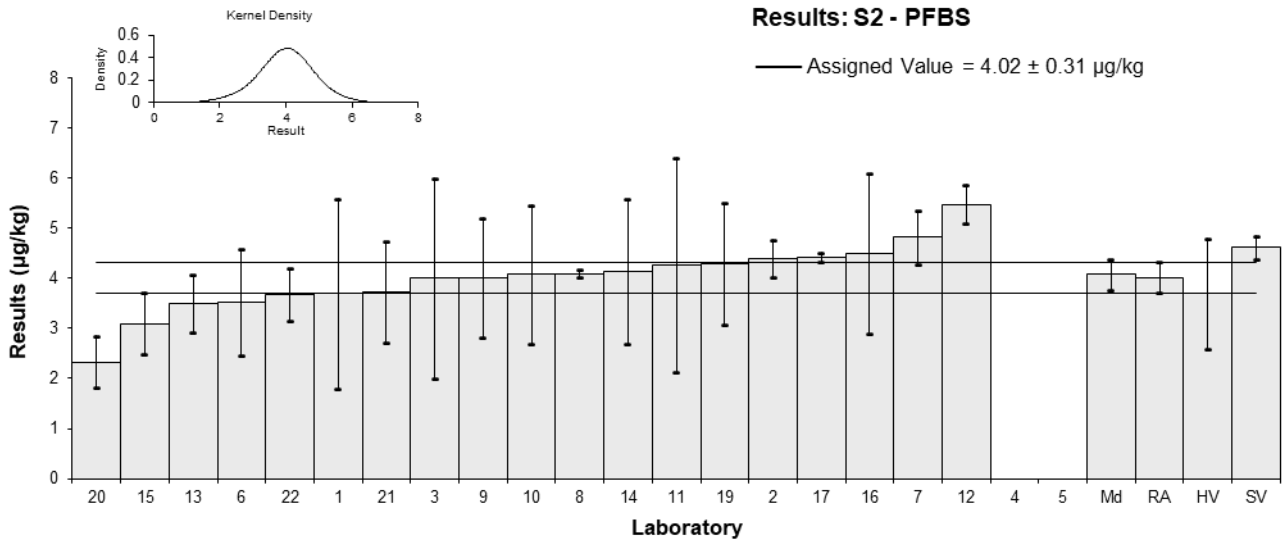


Figure 29

Table 35

**Sample Details**

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFPeS
<b>Unit</b>	µg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>Uncertainty</b>	<b>Rec</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	2.5	1.3	NR	-1.30	-0.65
2	3.8	0.41	123	0.62	0.76
3	4	2	116	0.92	0.30
4	NS	NS	NS		
5	NT	NT	NT		
6	3.034	0.91	82	-0.51	-0.35
7	2.97	0.18	87	-0.61	-1.00
8	3.35	0.068	NR	-0.04	-0.08
9	<0.5	NR	63		
10	4.136	1.38	89.99	1.12	0.53
11	3.39	1.695	NR	0.01	0.01
12*	5.92	0.42	NR	3.76	4.54
13	2.3	0.714	NR	-1.60	-1.34
14	NT	NT	NT		
15*	5.3	1.06	NR	2.84	1.71
16	3.4	1.2	NR	0.03	0.02
17	3.144	0.167	98	-0.35	-0.58
19	4.16	0.865	88	1.15	0.83
20	3.23	0.853	95.6	-0.22	-0.16
21	3.52	1	NR	0.21	0.13
22	3.55	0.39	98.2	0.25	0.32

\* Outlier, see Section 4.2

**Statistics**

<b>Assigned Value</b>	3.38	0.37
<b>Spike Value</b>	3.47	0.17
<b>Homogeneity Value</b>	3.5	1.0
<b>Robust Average</b>	3.52	0.44
<b>Median</b>	3.40	0.36
<b>Mean</b>	3.63	
<b>N</b>	17	
<b>Max</b>	5.92	
<b>Min</b>	2.3	
<b>Robust SD</b>	0.72	
<b>Robust CV</b>	20%	

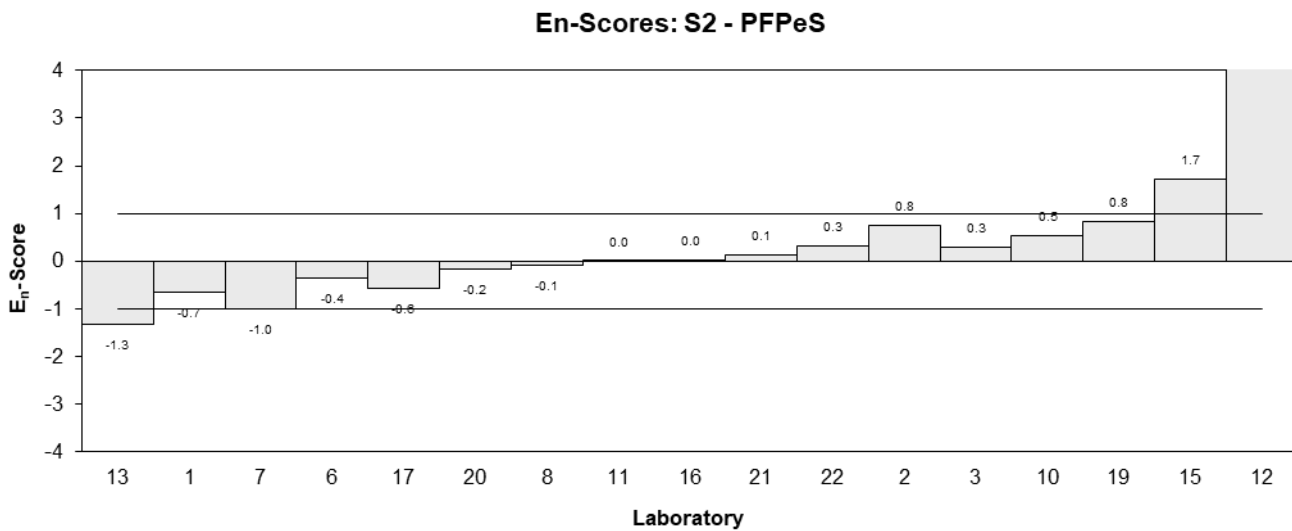
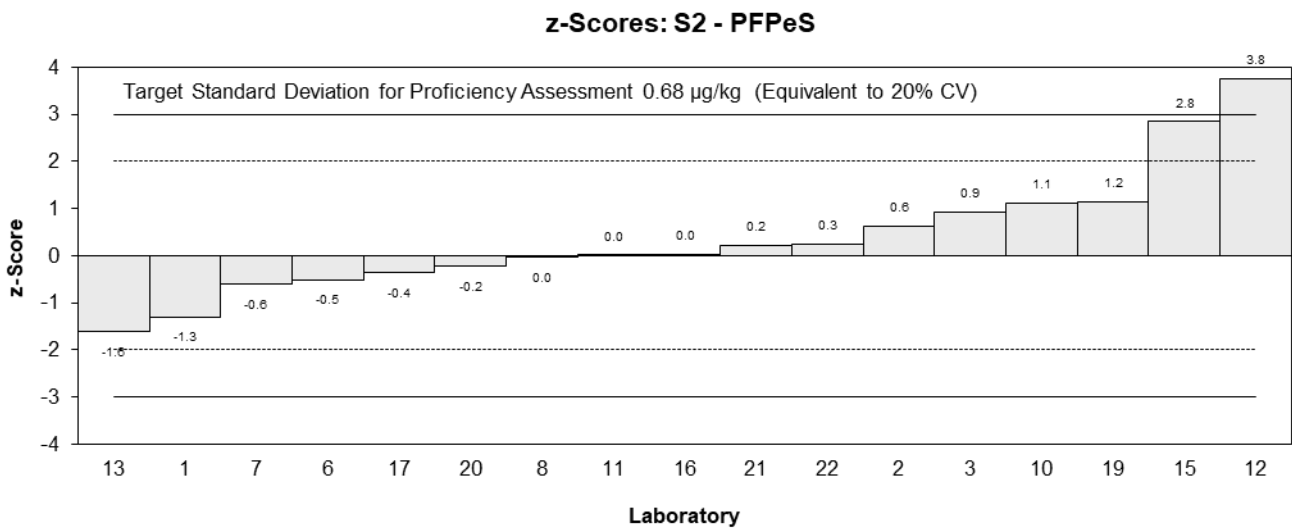
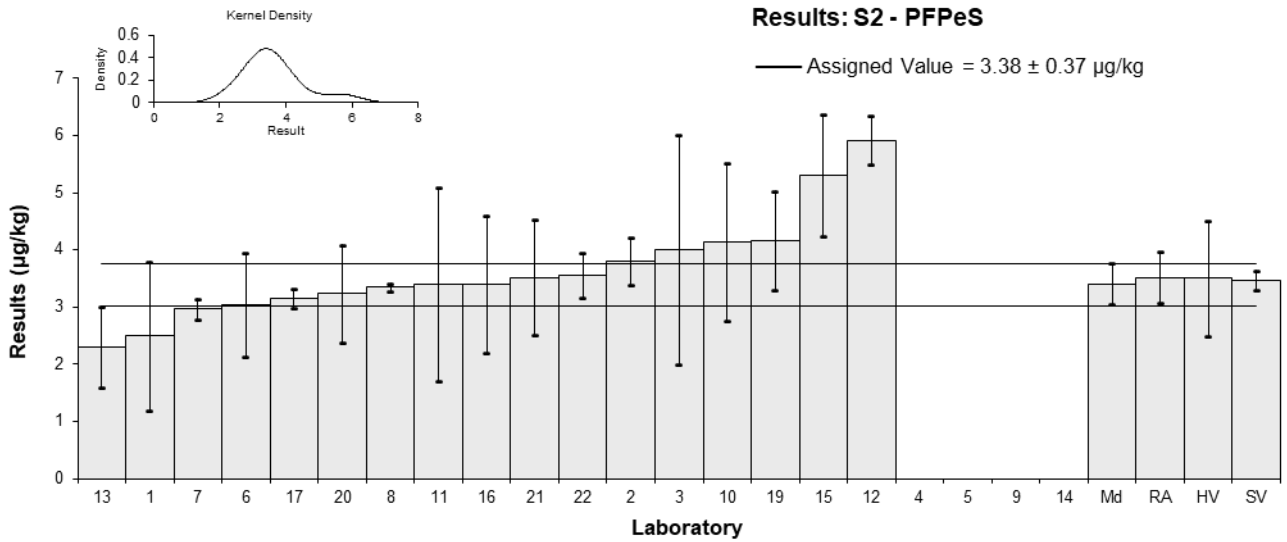


Figure 30

Table 36

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFHxS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	2	1	113	-0.58	-0.25
2	2.2	0.057	109	-0.13	-0.21
3	3	1	116	1.64	0.71
4	NS	NS	NS		
5	2.12	0.026	NT	-0.31	-0.50
6	1.9	0.57	89	-0.80	-0.57
7	2.48	0.10	NR	0.49	0.74
8	1.94	0.060	105.2	-0.71	-1.12
9	2.01	0.6	70	-0.55	-0.38
10	2.55	0.89	89.99	0.64	0.31
11	NT	NT	NT		
12	2.37	0.17	NR	0.24	0.34
13	1.8	0.979	NR	-1.02	-0.45
14	1.7933	0.6276	96	-1.03	-0.68
15	3.3	0.66	NR	2.30	1.45
16	2.9	1.0	NR	1.42	0.62
17	1.831	0.061	98	-0.95	-1.50
19	2.80	0.485	92	1.19	0.96
20	NT	NT	NT		
21	2.18	0.7	86	-0.18	-0.11
22	1.89	0.265	98.2	-0.82	-0.96

## Statistics

<b>Assigned Value</b>	2.26	0.28
<b>Spike Value</b>	2.20	0.11
<b>Homogeneity Value</b>	2.19	0.66
<b>Robust Average</b>	2.26	0.28
<b>Median</b>	2.15	0.25
<b>Mean</b>	2.28	
<b>N</b>	18	
<b>Max</b>	3.3	
<b>Min</b>	1.7933	
<b>Robust SD</b>	0.48	
<b>Robust CV</b>	21%	

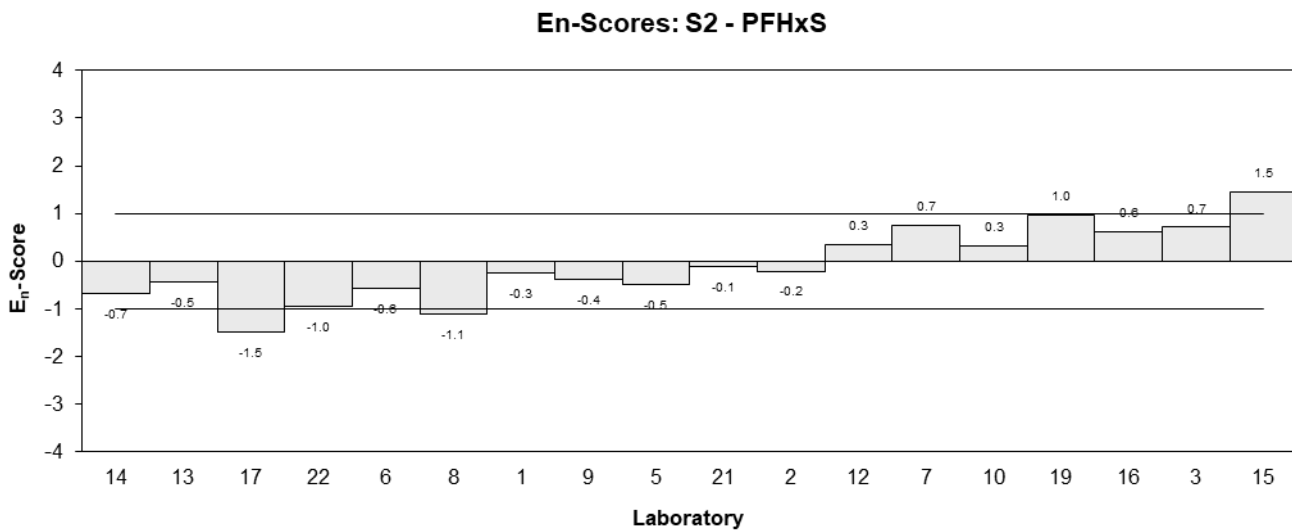
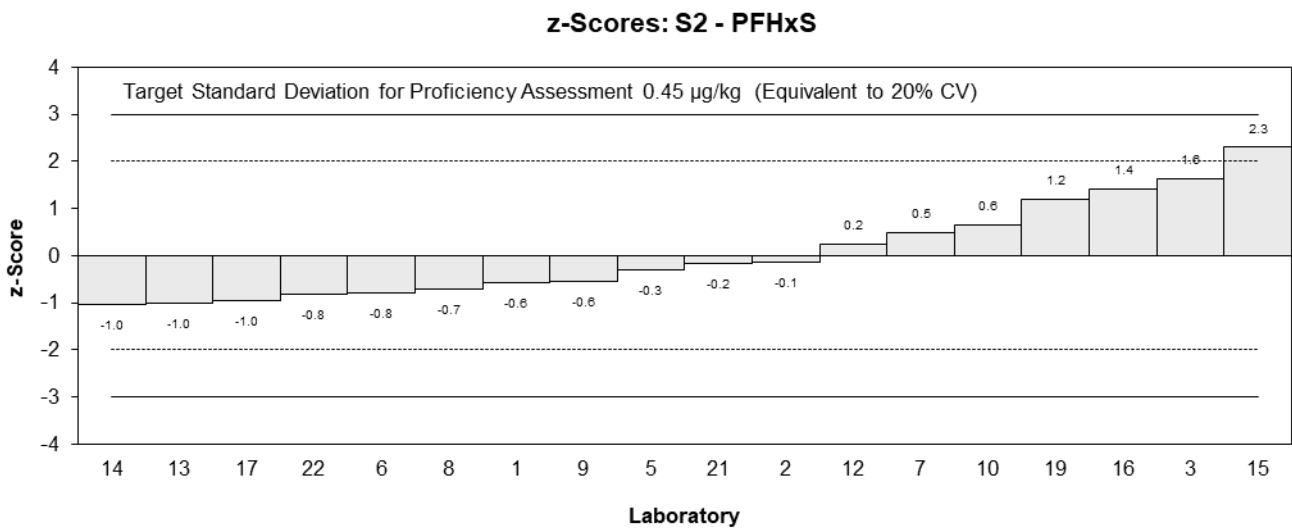
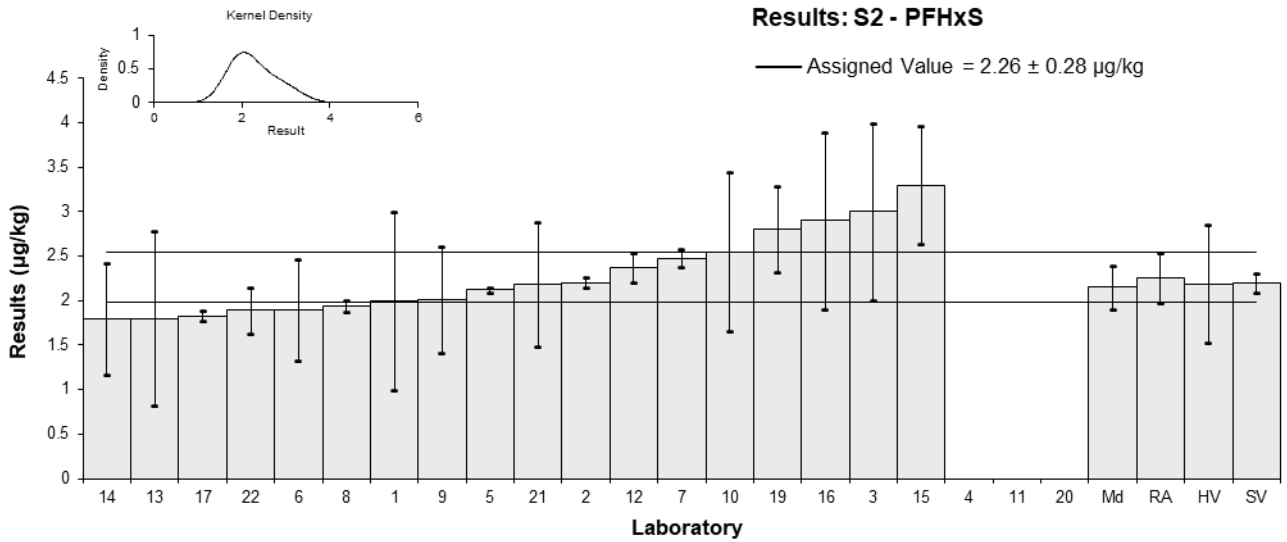


Figure 31

Table 37

## Sample Details

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

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	2	1	NR	-0.43	-0.18
2	2.2	0.057	109	0.02	0.04
3	3	1	116	1.85	0.79
4	NS	NS	NS		
5	NT	NT	NT		
6	1.9	0.57	89	-0.66	-0.47
7	2.48	0.10	87	0.66	1.08
8	1.94	0.061	NR	-0.57	-0.97
9	2.01	0.6	70	-0.41	-0.28
10	NT	NT	NT		
11	2.44	1.22	NR	0.57	0.20
12	2.37	0.17	NR	0.41	0.60
13	1.7	0.921	NR	-1.12	-0.51
14	1.7933	0.6276	96	-0.91	-0.59
15	NT	NT	NT		
16	NT	NT	NT		
17	NR	NR	NR		
19	2.80	0.012	92	1.39	2.44
20	2.43	0.760	95.6	0.55	0.30
21	2.18	0.7	NR	-0.02	-0.01
22	1.89	0.265	98.2	-0.68	-0.82

## Statistics

<b>Assigned Value</b>	2.19	0.25
<b>Spike Value</b>	2.20	0.11
<b>Homogeneity Value</b>	2.19	0.66
<b>Robust Average</b>	2.19	0.25
<b>Median</b>	2.18	0.25
<b>Mean</b>	2.21	
<b>N</b>	15	
<b>Max</b>	3	
<b>Min</b>	1.7	
<b>Robust SD</b>	0.38	
<b>Robust CV</b>	18%	



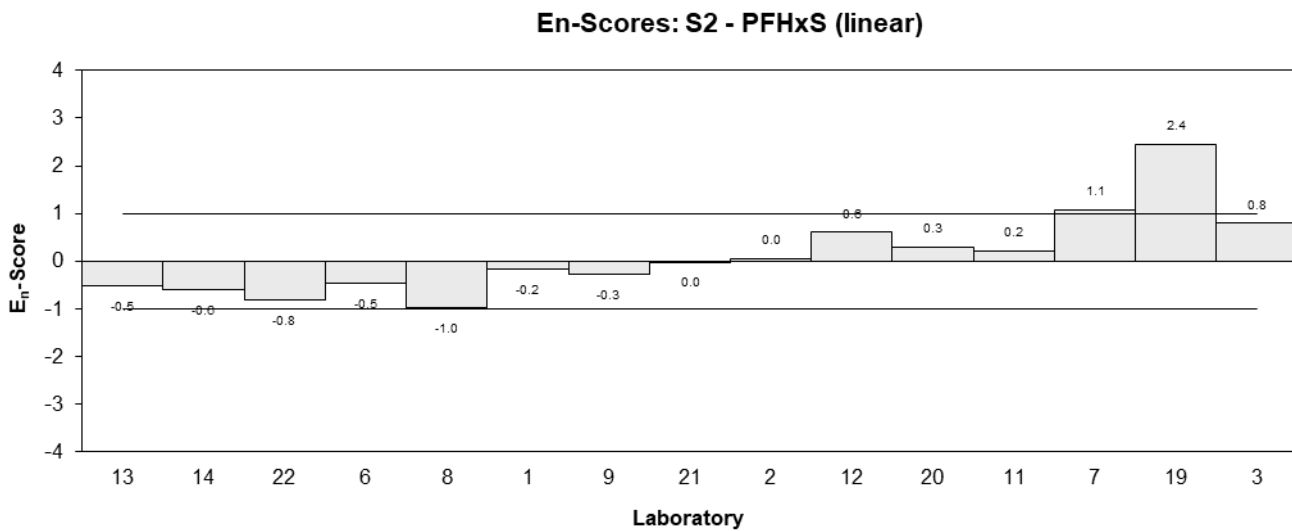
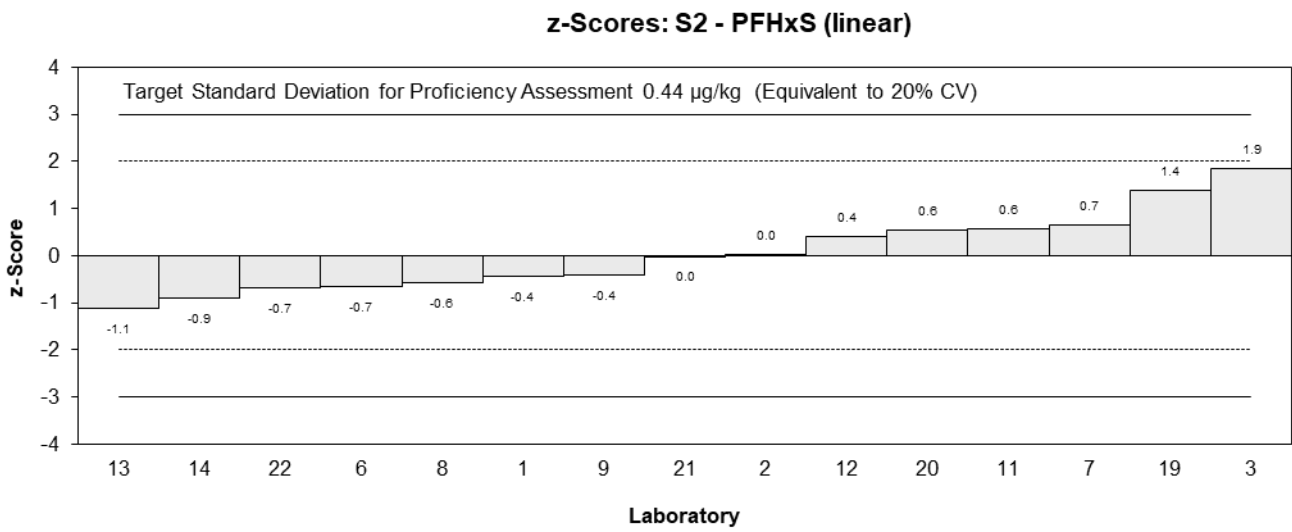
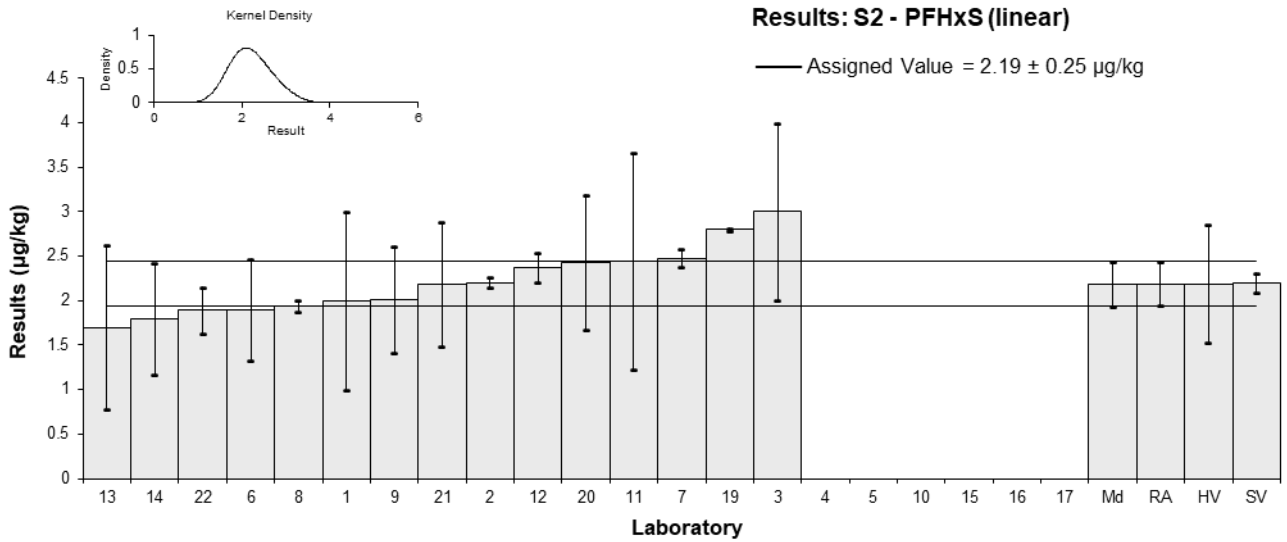


Figure 32

Table 38

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFHpS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	1.6	0.8	NR	-0.29	-0.12
2	1.7	0.16	109	0.00	0.00
3	2	1	107	0.88	0.30
4	NS	NS	NS		
5	NT	NT	NT		
6	1.407	0.422	81	-0.86	-0.64
7	1.95	0.15	87	0.74	1.07
8	1.58	0.057	NR	-0.35	-0.64
9	1.67	0.5	70	-0.09	-0.06
10	2.038	0.69	93.37	0.99	0.47
11	1.88	0.94	NR	0.53	0.19
12*	3.79	0.27	NR	6.15	6.44
13*	3.1	2.327	NR	4.12	0.60
14	1.4971	0.5240	100	-0.60	-0.37
15*	3.4	0.68	NR	5.00	2.42
16	1.3	0.5	NR	-1.18	-0.75
17	1.589	0.065	98	-0.33	-0.58
19	2.18	0.431	92	1.41	1.03
20	1.40	0.343	95.6	-0.88	-0.77
21	1.6	0.5	NR	-0.29	-0.19
22	1.91	0.286	97.4	0.62	0.62

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	1.70	0.18
<b>Spike Value</b>	1.76	0.09
<b>Homogeneity Value</b>	1.66	0.50
<b>Robust Average</b>	1.82	0.23
<b>Median</b>	1.70	0.21
<b>Mean</b>	1.98	
<b>N</b>	19	
<b>Max</b>	3.79	
<b>Min</b>	1.3	
<b>Robust SD</b>	0.41	
<b>Robust CV</b>	22%	

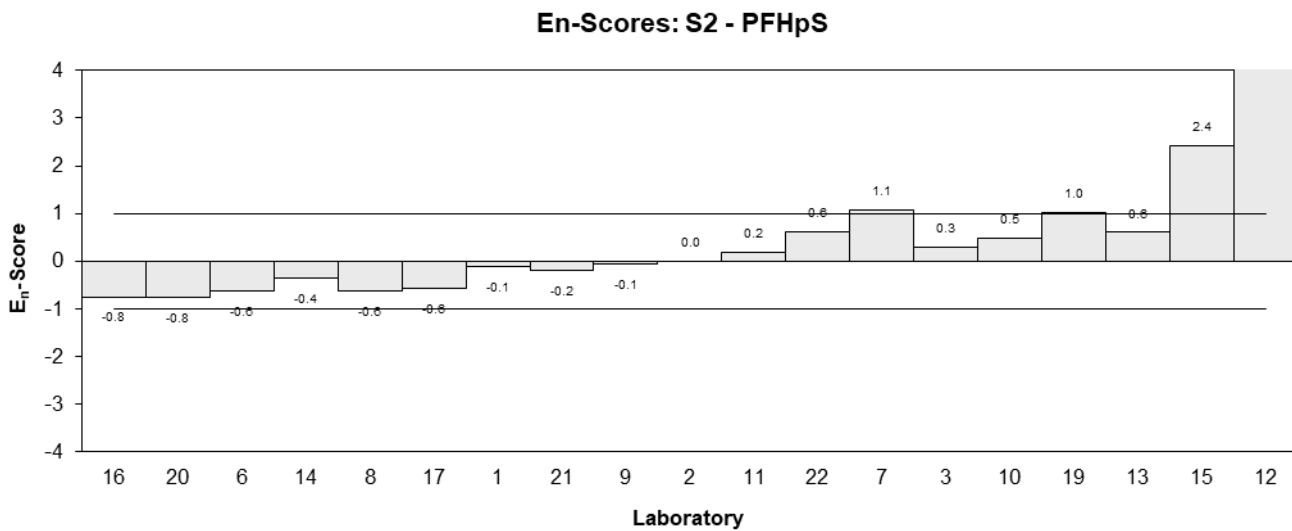
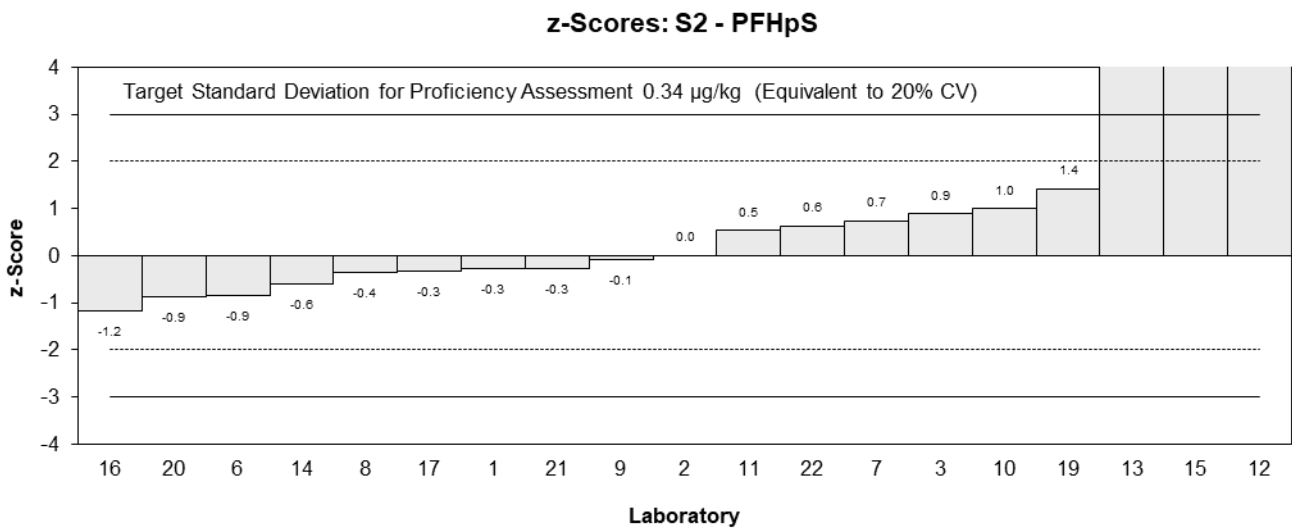
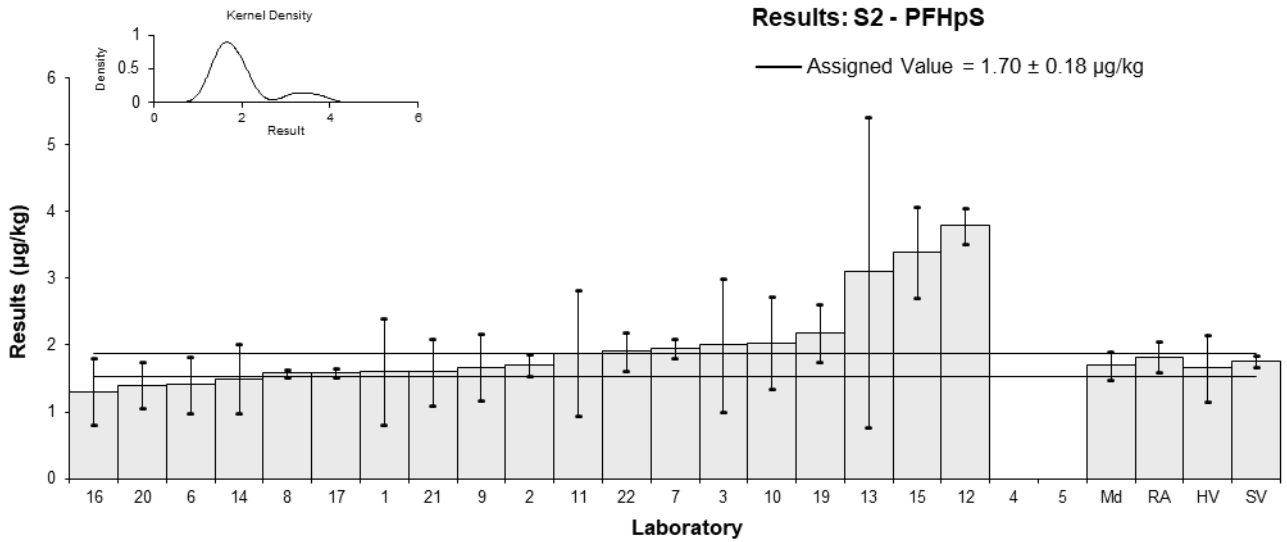


Figure 33

Table 39

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFOS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	2.2	1.1	103	-0.70	-0.32
2	2.6	0.27	112	0.08	0.11
3	2	1	107	-1.09	-0.54
4	NS	NS	NS		
5	3.09	0.054	NT	1.04	2.00
6	2.191	0.657	92	-0.72	-0.52
7	2.66	0.08	79	0.20	0.37
8	2.11	0.22	103.3	-0.88	-1.32
9	2.84	0.85	79	0.55	0.32
10	3.036	1.11	93.37	0.93	0.42
11	NT	NT	NT		
12	2.54	0.18	NR	-0.04	-0.06
13	2.9	1.633	NR	0.66	0.21
14	2.0827	0.7289	100	-0.93	-0.62
15*	4.2	0.84	NR	3.20	1.87
16	3	1.1	NR	0.86	0.39
17	2.211	0.094	95	-0.68	-1.26
19	3.04	0.898	97	0.94	0.51
20	2.88	1.63	77.9	0.62	0.19
21	2.2	0.7	82	-0.70	-0.48
22	2.54	0.432	97.4	-0.04	-0.04

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	2.56	0.26
<b>Spike Value</b>	2.50	0.12
<b>Homogeneity Value</b>	2.31	0.69
<b>Robust Average</b>	2.60	0.27
<b>Median</b>	2.60	0.34
<b>Mean</b>	2.65	
<b>N</b>	19	
<b>Max</b>	4.2	
<b>Min</b>	2	
<b>Robust SD</b>	0.46	
<b>Robust CV</b>	18%	

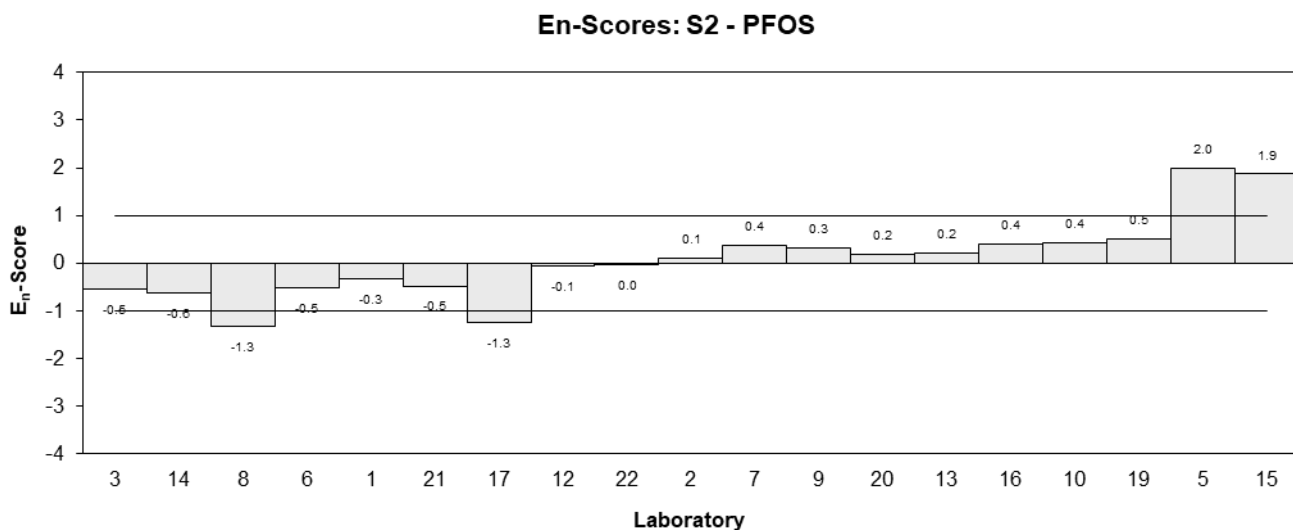
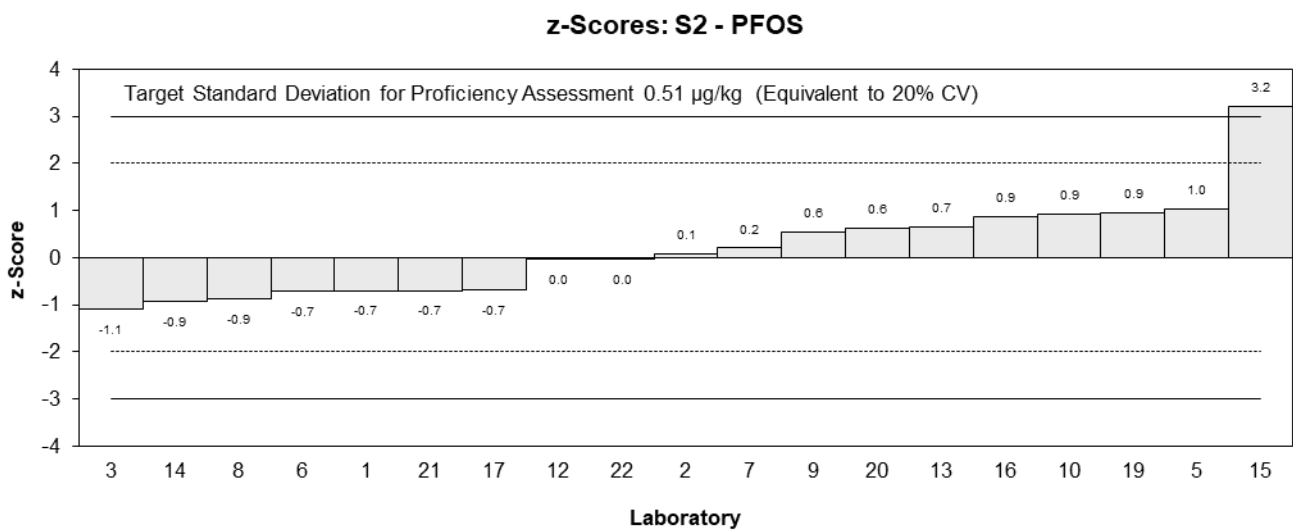
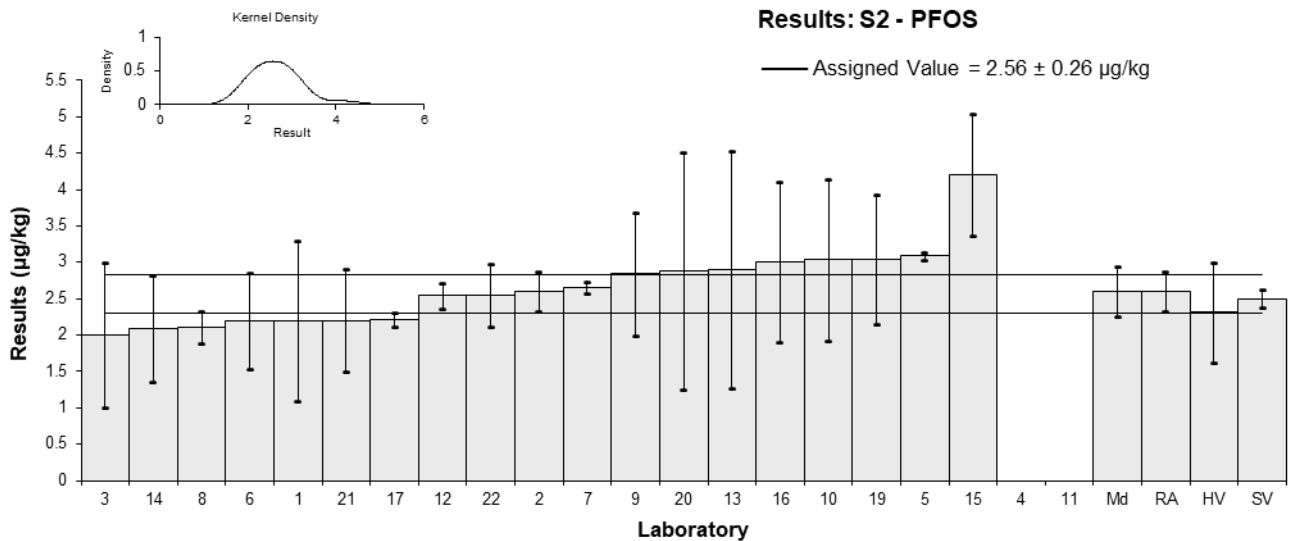


Figure 34

Table 40

## Sample Details

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

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	2.2	1.1	NR	-0.67	-0.30
2	2.6	0.27	112	0.12	0.16
3	2	1	107	-1.06	-0.52
4	NS	NS	NS		
5	NT	NT	NT		
6	2.191	0.657	92	-0.69	-0.49
7	2.66	0.08	79	0.24	0.44
8	2.11	0.057	NR	-0.85	-1.62
9	2.84	0.85	79	0.59	0.34
10	3.036	1.11	93.37	0.98	0.44
11	2.88	1.44	NR	0.67	0.23
12	2.54	0.18	NR	0.00	0.00
13	2.9	1.647	NR	0.71	0.22
14	2.0827	0.7289	100	-0.90	-0.59
15	NT	NT	NT		
16	NT	NT	NT		
17	NR	NR	NR		
19	3.04	0.035	97	0.98	1.91
20	2.88	0.638	77.9	0.67	0.49
21	2.2	0.7	NR	-0.67	-0.46
22	2.54	0.432	97.4	0.00	0.00

## Statistics

<b>Assigned Value</b>	2.54	0.26
<b>Spike Value</b>	2.50	0.12
<b>Homogeneity Value</b>	2.31	0.69
<b>Robust Average</b>	2.54	0.26
<b>Median</b>	2.57	0.32
<b>Mean</b>	2.54	
<b>N</b>	16	
<b>Max</b>	3.04	
<b>Min</b>	2	
<b>Robust SD</b>	0.41	
<b>Robust CV</b>	16%	

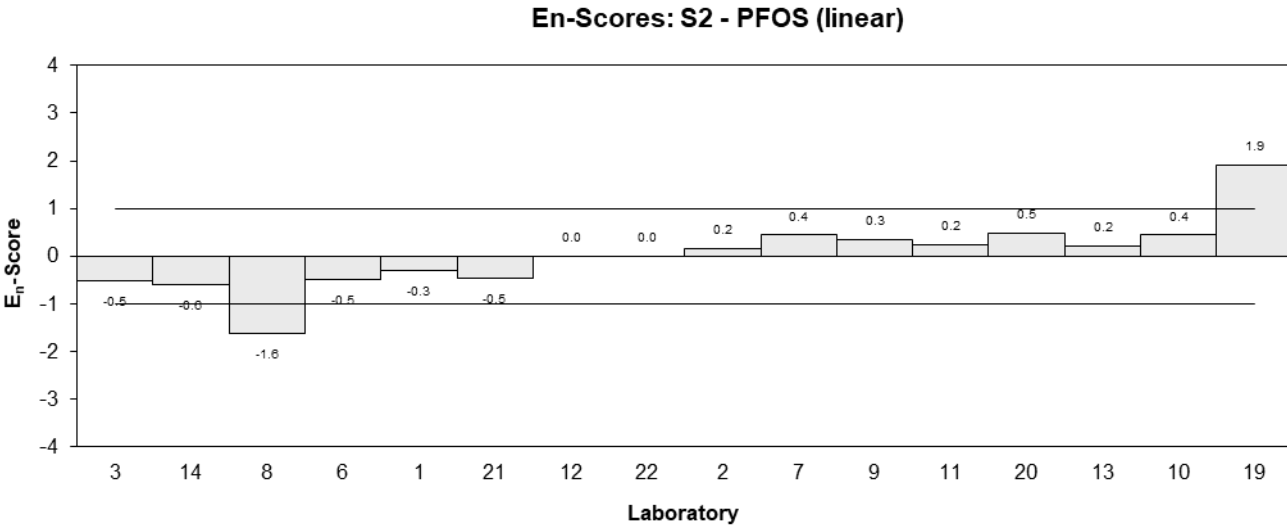
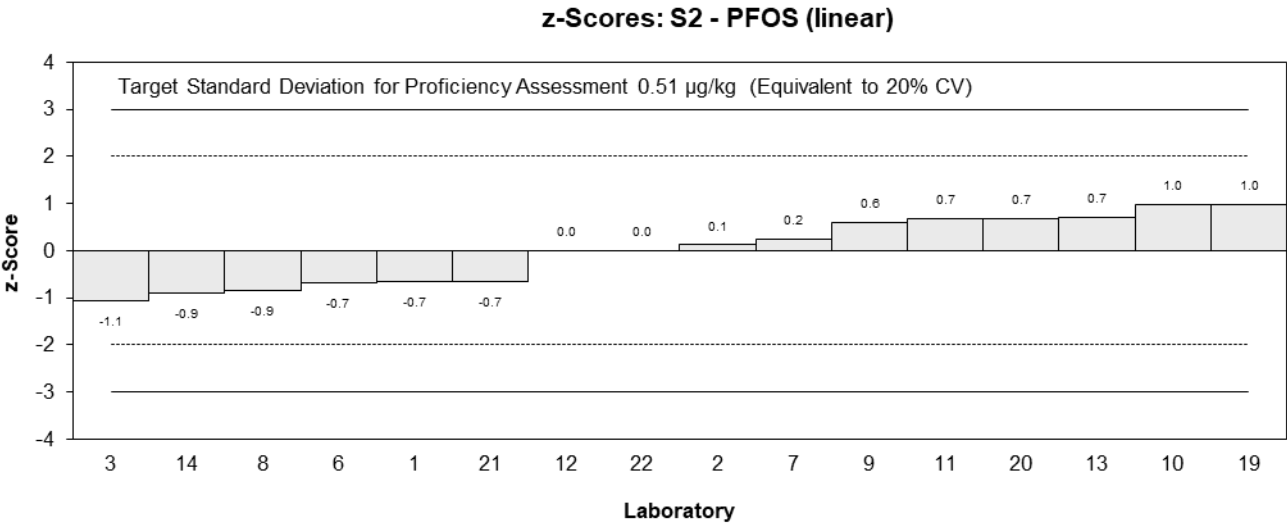
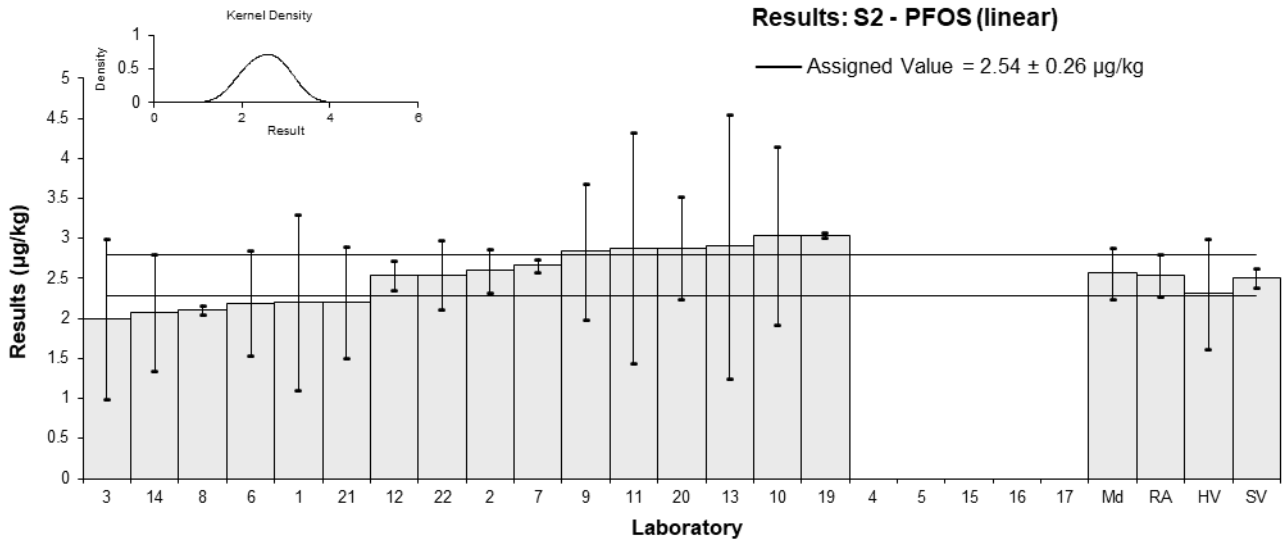


Figure 35

Table 41

**Sample Details**

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFNS
<b>Unit</b>	µg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>Uncertainty</b>	<b>Rec</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	2.7	1.4	NR	-1.05	-0.49
2	3.8	0.15	112	0.56	0.87
3	4	2	107	0.85	0.28
4	NS	NS	NS		
5	NT	NT	NT		
6	3.145	0.944	83	-0.40	-0.27
7	2.55	0.48	79	-1.27	-1.38
8	3.11	0.080	NR	-0.45	-0.74
9	3.69	1.1	79	0.39	0.23
10	NT	NT	NT		
11	3.87	1.935	NR	0.66	0.23
12	3.11	0.22	NR	-0.45	-0.67
13	2.6	1.529	NR	-1.20	-0.52
14	NT	NT	NT		
15	NT	NT	NT		
16*	9	3.2	NR	8.16	1.73
17	3.161	0.173	95	-0.38	-0.58
19	4.67	0.774	97	1.83	1.43
20	4.03	2.20	77.9	0.89	0.27
21	3.57	1	NR	0.22	0.14
22	3.56	0.749	97.4	0.20	0.16

\* Outlier, see Section 4.2

**Statistics**

<b>Assigned Value</b>	3.42	0.41
<b>Spike Value</b>	3.55	0.18
<b>Homogeneity Value</b>	3.6	1.1
<b>Robust Average</b>	3.50	0.45
<b>Median</b>	3.57	0.41
<b>Mean</b>	3.79	
<b>N</b>	16	
<b>Max</b>	9	
<b>Min</b>	2.55	
<b>Robust SD</b>	0.72	
<b>Robust CV</b>	21%	



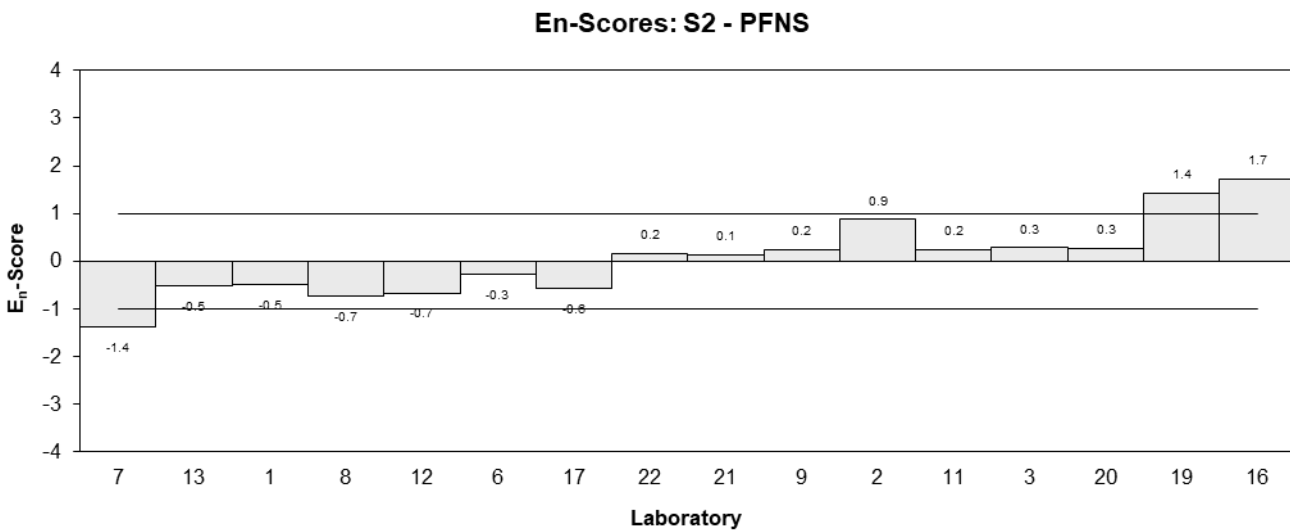
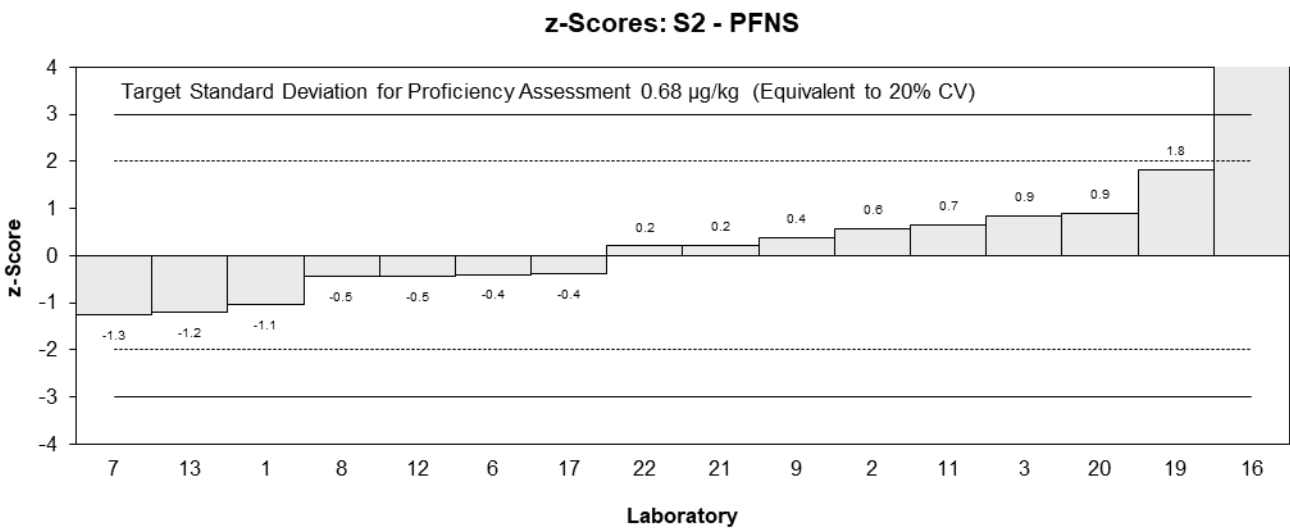
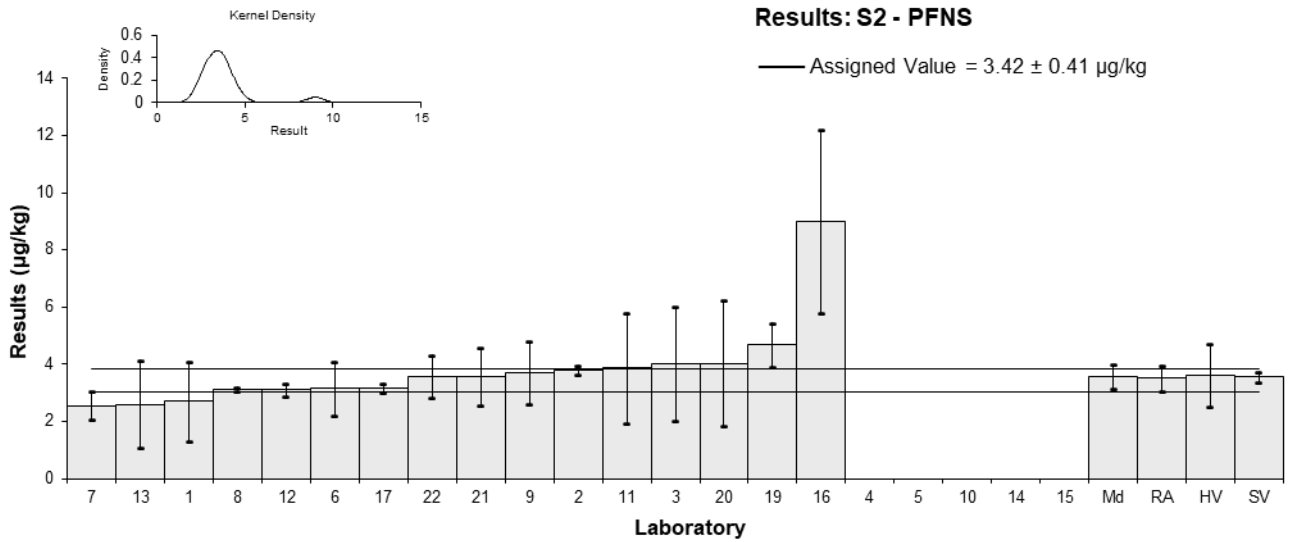


Figure 36

Table 42

**Sample Details**

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFDS
<b>Unit</b>	µg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>Uncertainty</b>	<b>Rec</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT	NT		
2	2.6	0.11	112	0.16	0.25
3	2	1	107	-1.03	-0.50
4	NS	NS	NS		
5	NT	NT	NT		
6	2.084	0.625	86	-0.87	-0.63
7*	0.83	0.63	79	-3.35	-2.42
8	2.41	0.065	NR	-0.22	-0.36
9	2.52	0.76	79	0.00	0.00
10	3.122	1.19	93.37	1.19	0.49
11	2.58	1.29	NR	0.12	0.05
12	2.34	0.17	NR	-0.36	-0.52
13*	6.4	4.116	NR	7.70	0.94
14	1.9229	0.6730	100	-1.18	-0.81
15	NT	NT	NT		
16*	6.2	2.2	NR	7.30	1.66
17	2.357	0.084	95	-0.32	-0.52
19	3.09	0.633	97	1.13	0.81
20	3.09	1.82	77.9	1.13	0.31
21	2.35	0.9	NR	-0.34	-0.18
22	2.79	0.614	97.4	0.54	0.40

\* Outlier, see Section 4.2

**Statistics**

<b>Assigned Value</b>	2.52	0.30
<b>Spike Value</b>	2.68	0.13
<b>Homogeneity Value</b>	2.5	1.0
<b>Robust Average</b>	2.58	0.37
<b>Median</b>	2.52	0.39
<b>Mean</b>	2.86	
<b>N</b>	17	
<b>Max</b>	6.4	
<b>Min</b>	0.83	
<b>Robust SD</b>	0.60	
<b>Robust CV</b>	23%	

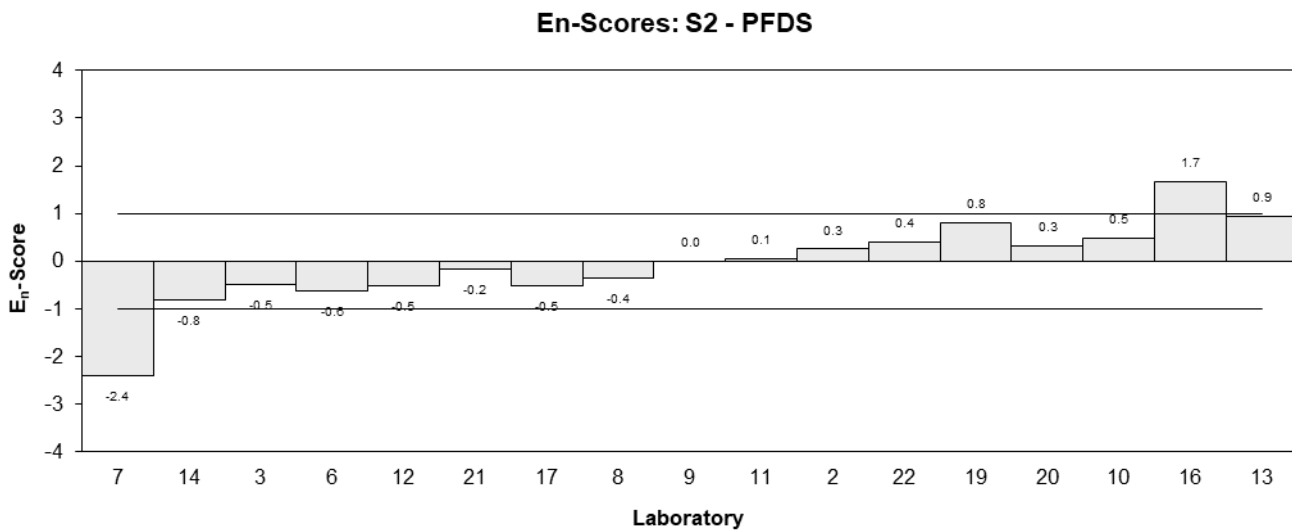
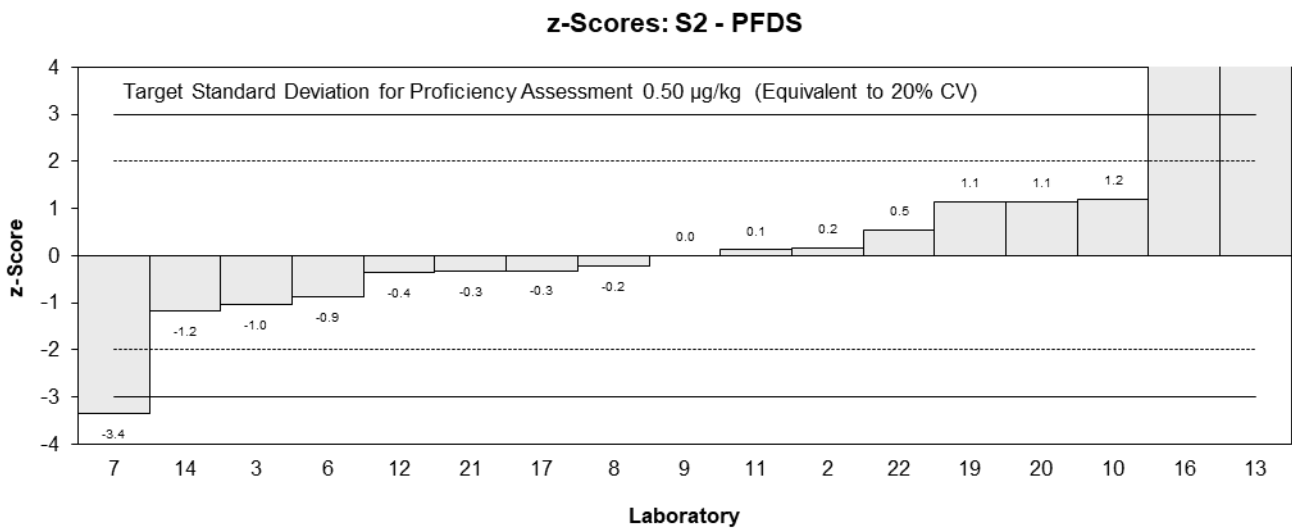
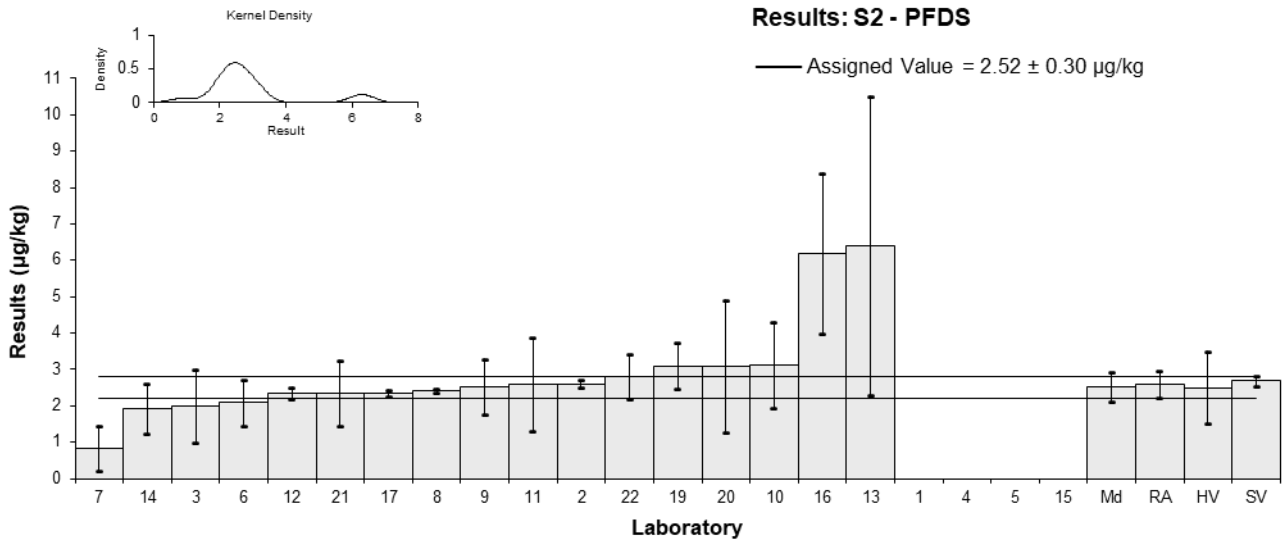


Figure 37

Table 43

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFBA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	10	5	67	-0.87	-0.41
2	12	0.25	86	-0.04	-0.08
3	12	4	103	-0.04	-0.02
4	NS	NS	NS		
5	NT	NT	NT		
6	7.393	2.218	79	-1.95	-1.83
7	17.75	2.52	17	2.33	1.99
8	11.9	0.026	62.4	-0.08	-0.15
9	10.44	3.13	98	-0.69	-0.49
10	17.96	7.7	76.64	2.42	0.75
11	12.17	6.085	NR	0.03	0.01
12	12.82	0.91	NR	0.30	0.45
13	NT	NT	NT		
14	NT	NT	NT		
15	NT	NT	NT		
16	15	5.3	NR	1.20	0.53
17	10.892	0.24	79	-0.50	-0.91
19	10.6	1.9	14	-0.62	-0.65
20	13.0	2.96	49.5	0.37	0.28
21	11.7	5	80	-0.17	-0.08
22	12.2	1.1	94.6	0.04	0.06

## Statistics

<b>Assigned Value</b>	12.1	1.3
<b>Spike Value</b>	10.7	0.5
<b>Homogeneity Value</b>	11.4	4.6
<b>Robust Average</b>	12.1	1.3
<b>Median</b>	12.0	1.0
<b>Mean</b>	12.4	
<b>N</b>	16	
<b>Max</b>	17.96	
<b>Min</b>	7.393	
<b>Robust SD</b>	2.1	
<b>Robust CV</b>	17%	

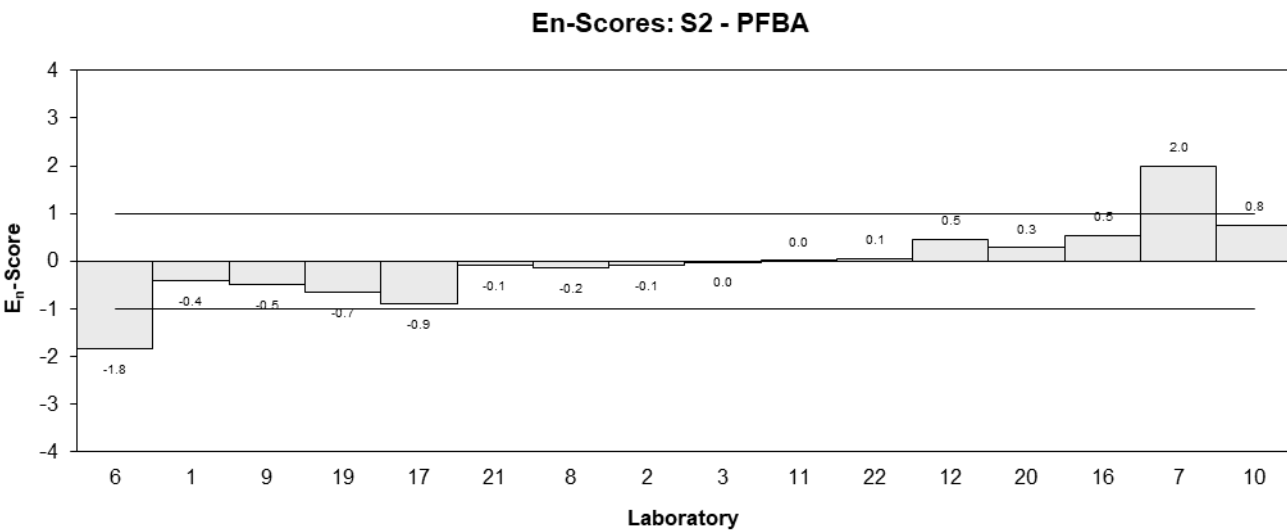
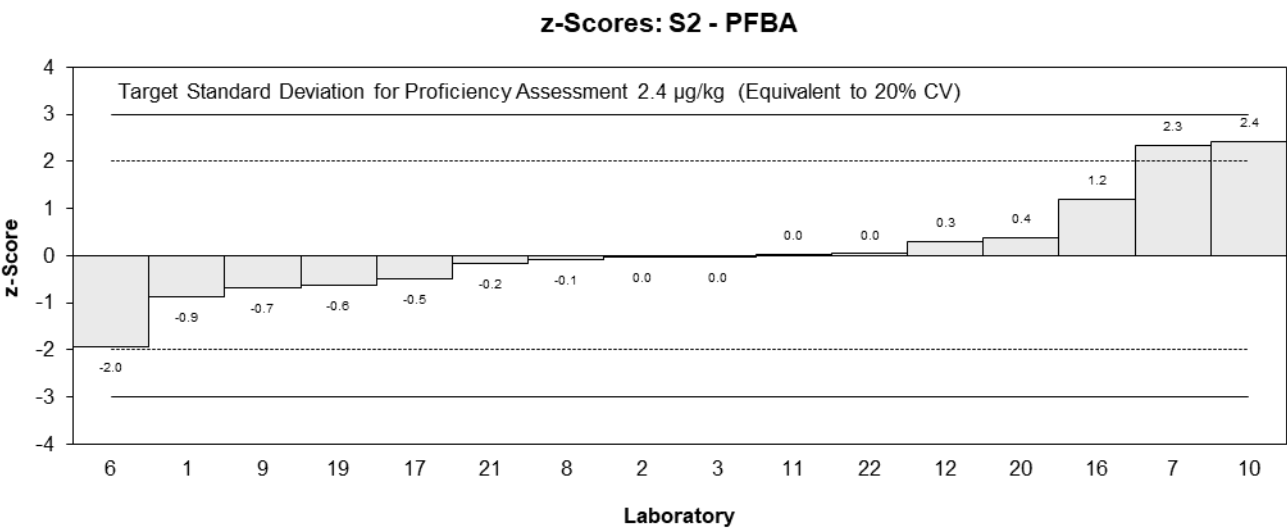
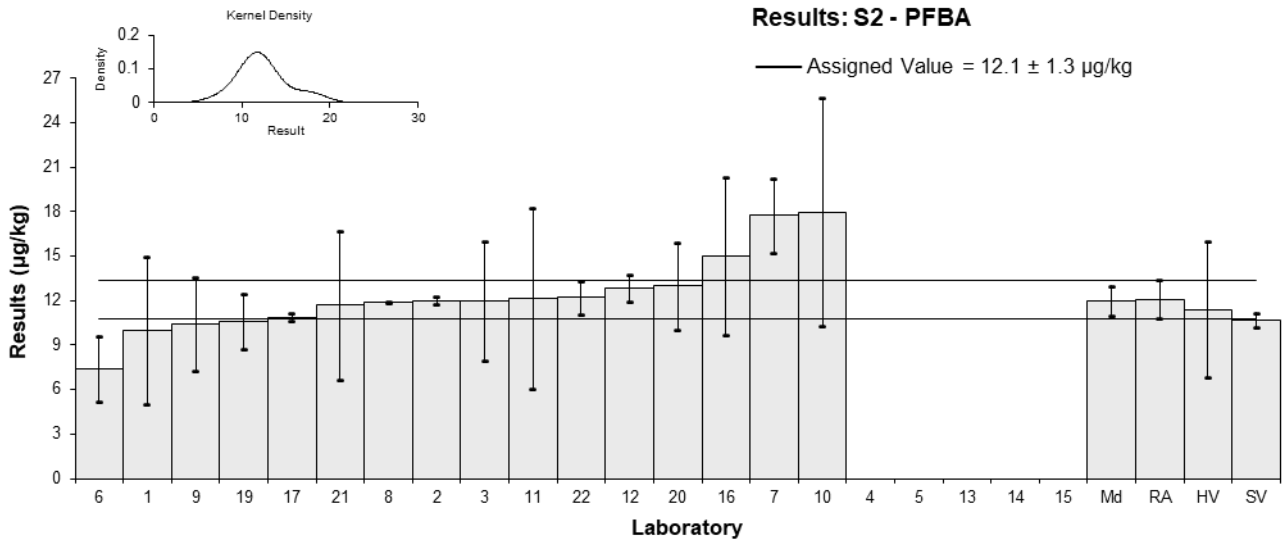


Figure 38

Table 44

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFPeA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	3.9	2.0	107	-0.82	-0.37
2	4.8	0.071	108	0.15	0.30
3	6	3	104	1.44	0.44
4	NS	NS	NS		
5	NT	NT	NT		
6	3.518	1.055	83	-1.23	-0.99
7	5.39	0.72	45	0.78	0.85
8	4.30	0.053	99.8	-0.39	-0.78
9	4.25	1.28	71	-0.44	-0.30
10	4.384	1.58	106.85	-0.30	-0.17
11	5.72	2.86	NR	1.14	0.37
12	4.55	0.32	NR	-0.12	-0.20
13	3.6	0.388	NR	-1.14	-1.76
14	6.6309	2.3208	31	2.11	0.83
15	4.4	0.88	NR	-0.28	-0.26
16	5.0	1.8	NR	0.36	0.18
17	4.499	0.069	86	-0.17	-0.35
19	4.42	1.1	75	-0.26	-0.20
20	5.34	5.10	30.7	0.73	0.13
21	4.08	1	83	-0.62	-0.53
22	4.68	0.468	94.4	0.02	0.03

## Statistics

<b>Assigned Value</b>	4.66	0.46
<b>Spike Value</b>	4.60	0.23
<b>Homogeneity Value</b>	4.5	1.4
<b>Robust Average</b>	4.66	0.46
<b>Median</b>	4.50	0.36
<b>Mean</b>	4.71	
<b>N</b>	19	
<b>Max</b>	6.6309	
<b>Min</b>	3.518	
<b>Robust SD</b>	0.80	
<b>Robust CV</b>	17%	

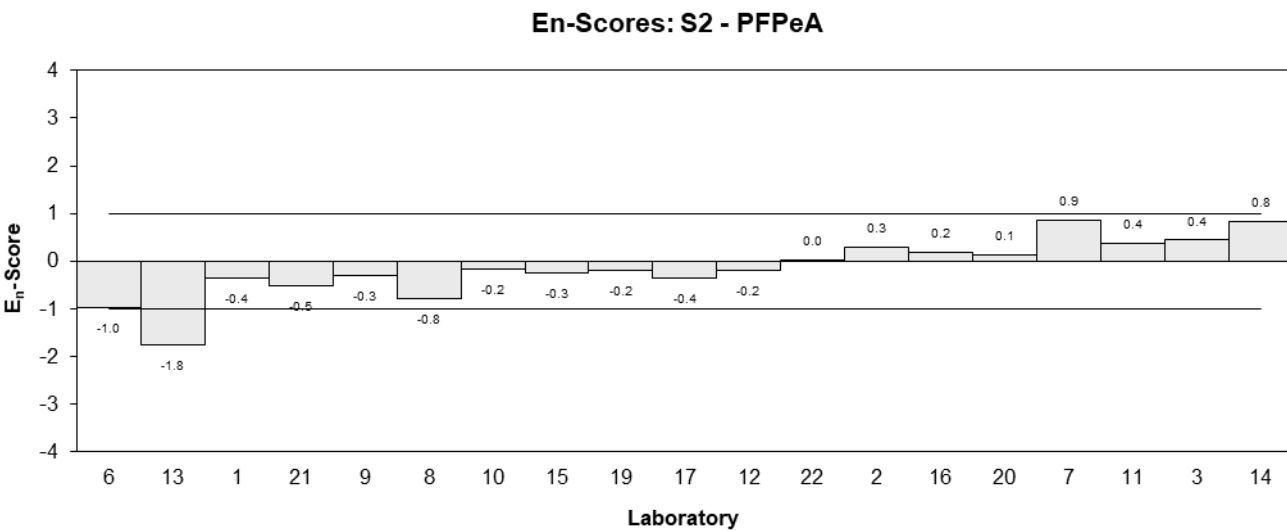
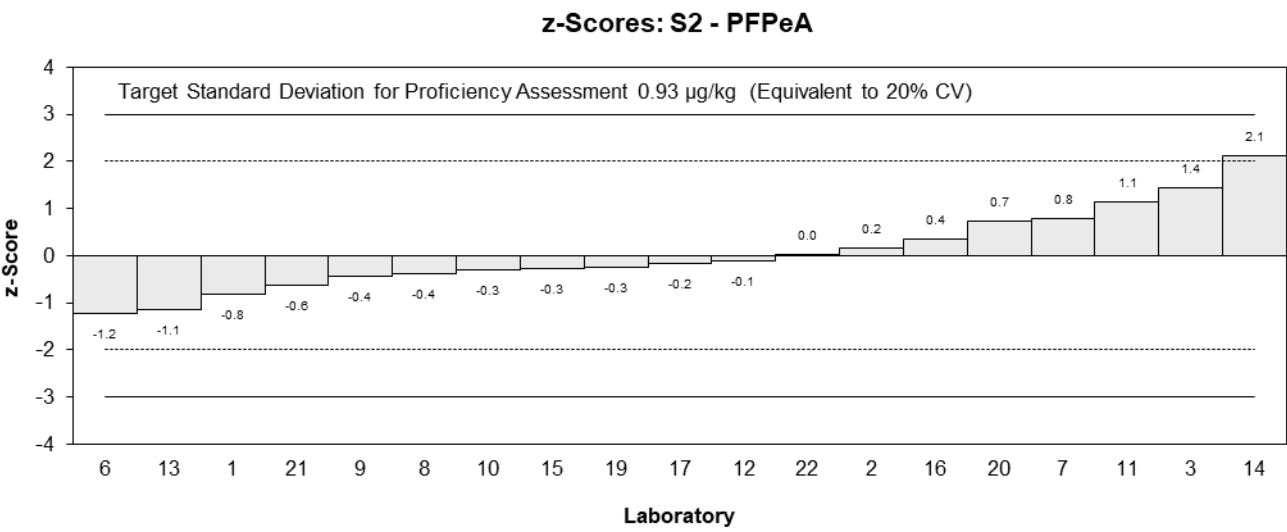
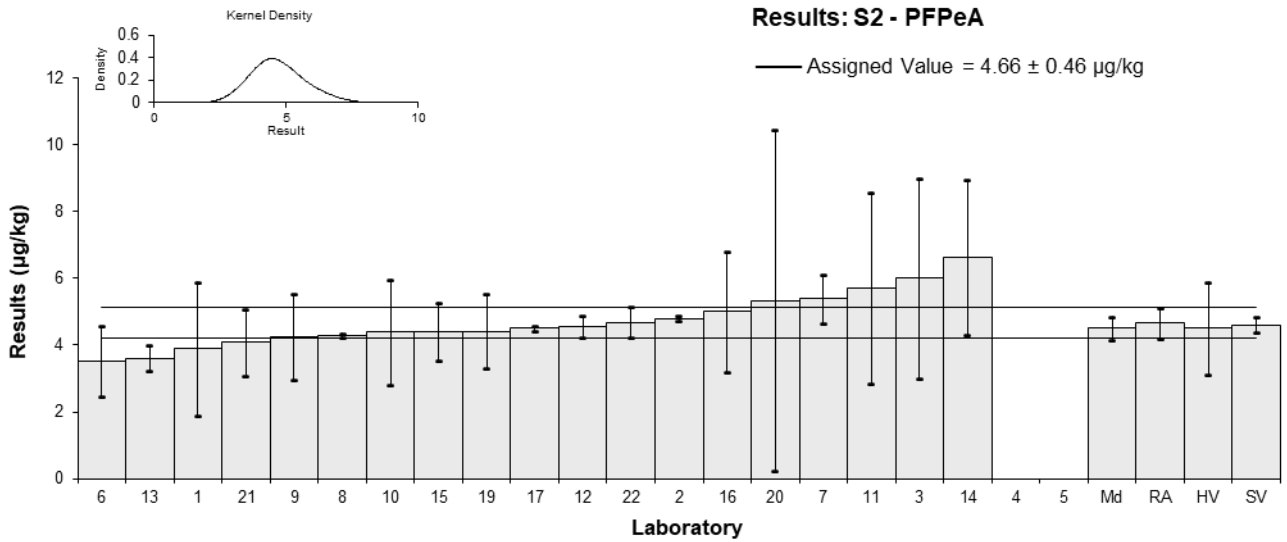


Figure 39

Table 45

**Sample Details**

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFHxA
<b>Unit</b>	µg/kg

**Participant Results**

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	5	2.5	58	-0.27	-0.11
2	5.8	1.1	123	0.49	0.44
3	6	3	110	0.68	0.24
4	NS	NS	NS		
5	NT	NT	NT		
6	4.529	1.359	94	-0.71	-0.53
7	6.57	1.02	58	1.22	1.17
8	4.61	0.051	106.9	-0.63	-1.58
9	4.81	1.47	99	-0.45	-0.31
10	5.298	2	95.65	0.02	0.01
11	6.42	3.21	NR	1.08	0.35
12	5.22	0.37	NR	-0.06	-0.11
13*	26	5.000	NR	19.62	4.13
14	4.6530	1.6285	54	-0.59	-0.37
15	4.9	0.98	NR	-0.36	-0.36
16	6.3	2.2	NR	0.97	0.46
17	4.889	0.034	87	-0.37	-0.93
19	5.35	0.946	86	0.07	0.07
20	5.46	1.28	52.4	0.17	0.13
21	4.77	1	81	-0.48	-0.47
22	4.76	0.666	91.3	-0.49	-0.66

\* Outlier, see Section 4.2

**Statistics**

<b>Assigned Value</b>	5.28	0.42
<b>Spike Value</b>	4.61	0.23
<b>Homogeneity Value</b>	5.0	1.5
<b>Robust Average</b>	5.36	0.45
<b>Median</b>	5.22	0.39
<b>Mean</b>	6.4	
<b>N</b>	19	
<b>Max</b>	26	
<b>Min</b>	4.529	
<b>Robust SD</b>	0.79	
<b>Robust CV</b>	15%	



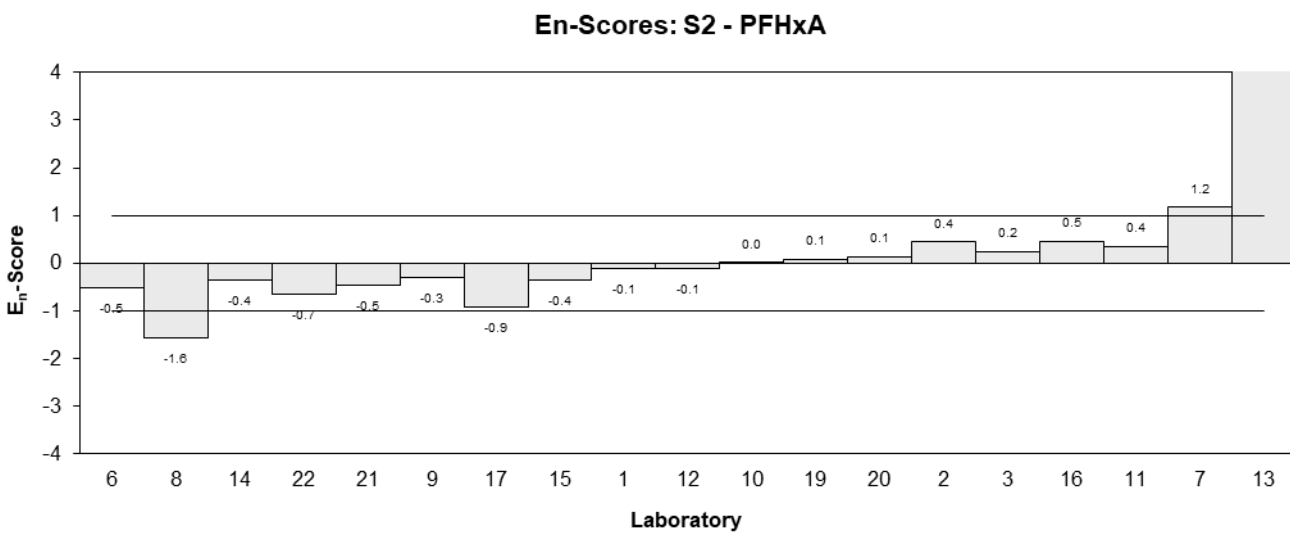
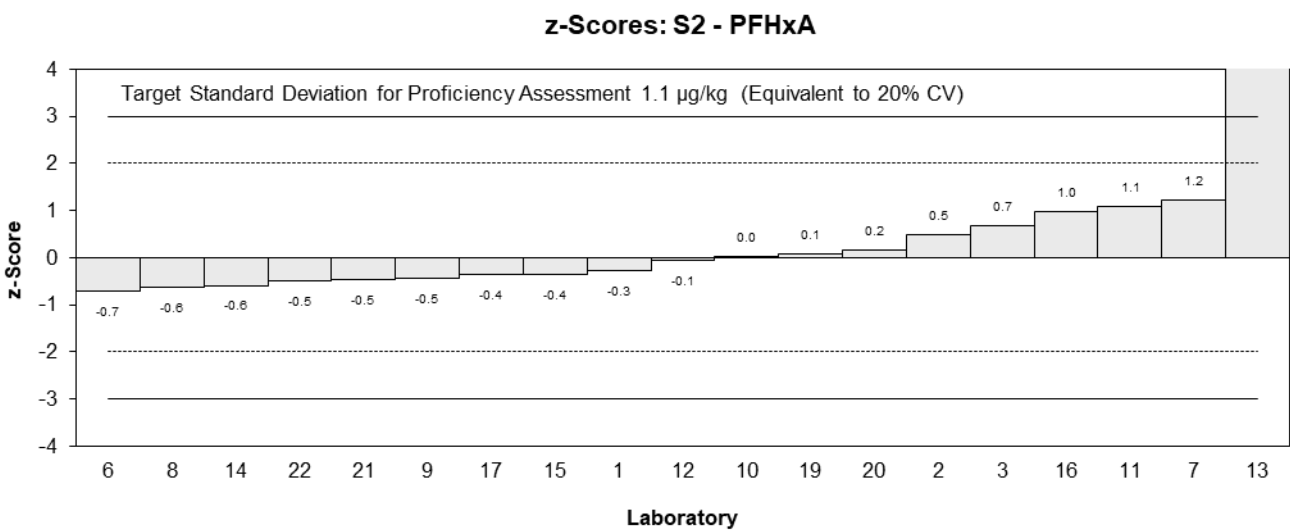
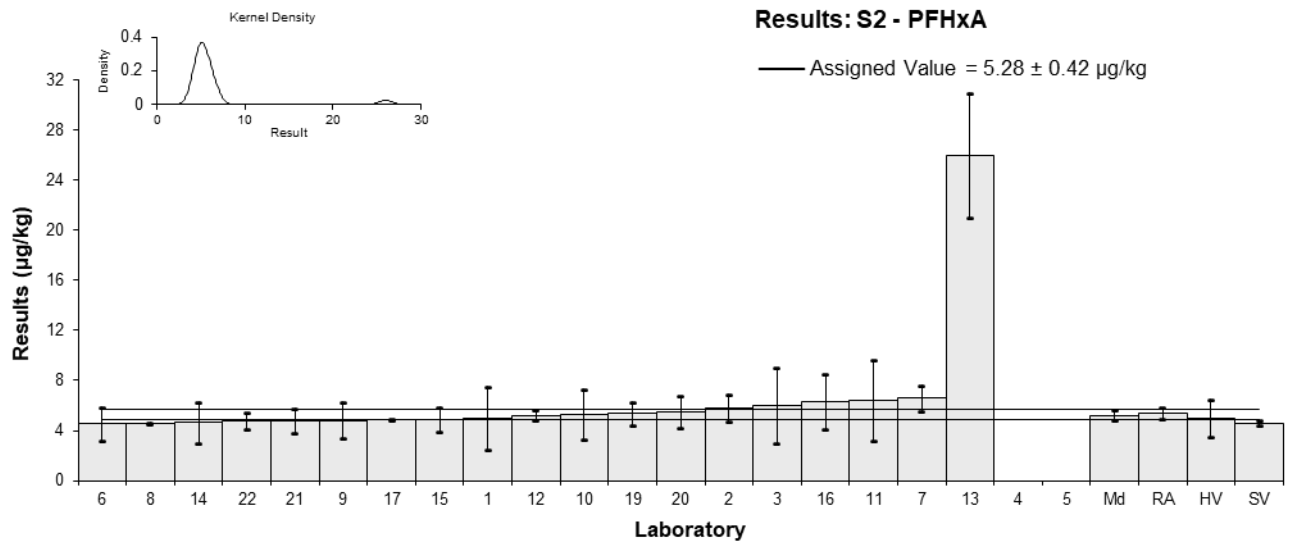


Figure 40

Table 46

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFHpA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	2.7	1.4	125	-0.34	-0.14
2	3.1	0.047	105	0.34	0.73
3	3	1	103	0.17	0.10
4	NS	NS	NS		
5	NT	NT	NT		
6	1.831	0.549	92	-1.84	-1.75
7	3.76	0.87	69	1.48	0.94
8	2.88	0.086	NR	-0.03	-0.07
9	2.62	0.77	86	-0.48	-0.34
10	3.246	1.2	100.96	0.60	0.28
11	2.65	1.325	NR	-0.43	-0.18
12	2.95	0.21	NR	0.09	0.15
13	2.0	0.532	NR	-1.55	-1.51
14	3.10285	1.0860	43	0.35	0.18
15	3.2	0.64	NR	0.52	0.43
16	2.1	0.7	NR	-1.38	-1.07
17	3.005	0.022	87	0.18	0.39
19	3.45	0.569	94	0.95	0.87
20	3.43	0.952	62.6	0.91	0.54
21	2.83	0.8	84	-0.12	-0.08
22	2.77	0.415	88.9	-0.22	-0.26

## Statistics

<b>Assigned Value</b>	2.90	0.27
<b>Spike Value</b>	2.77	0.14
<b>Homogeneity Value</b>	3.03	0.91
<b>Robust Average</b>	2.90	0.27
<b>Median</b>	2.95	0.21
<b>Mean</b>	2.87	
<b>N</b>	19	
<b>Max</b>	3.76	
<b>Min</b>	1.831	
<b>Robust SD</b>	0.47	
<b>Robust CV</b>	16%	

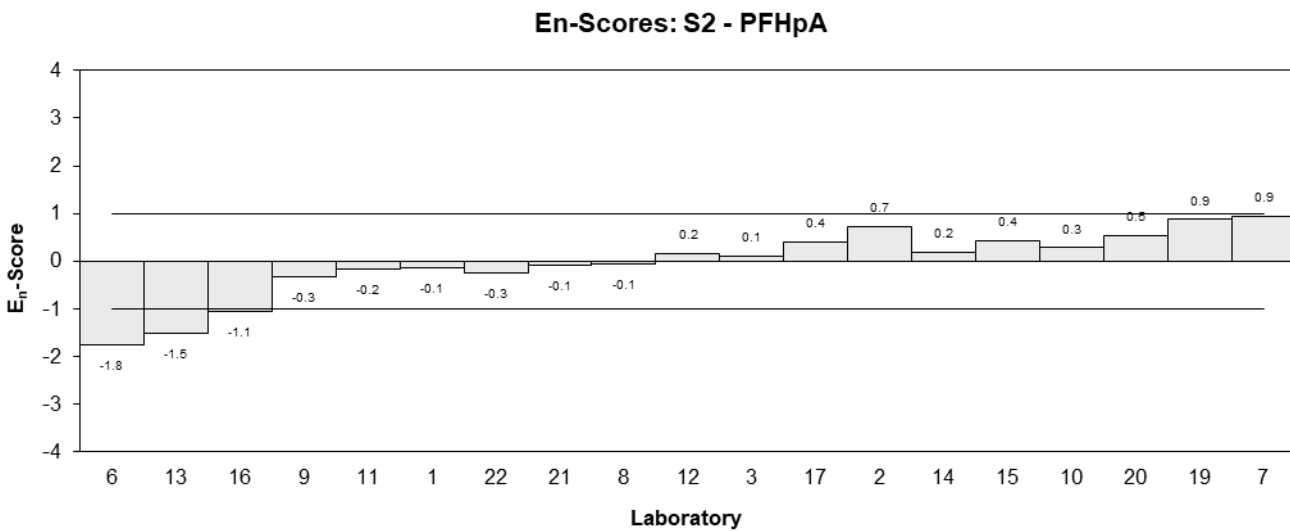
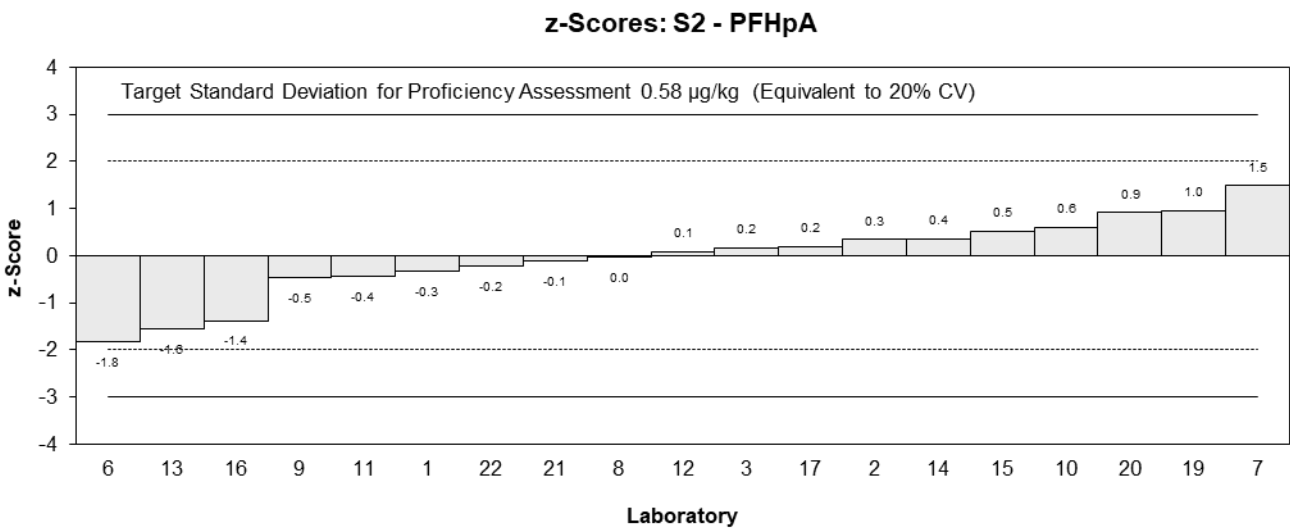
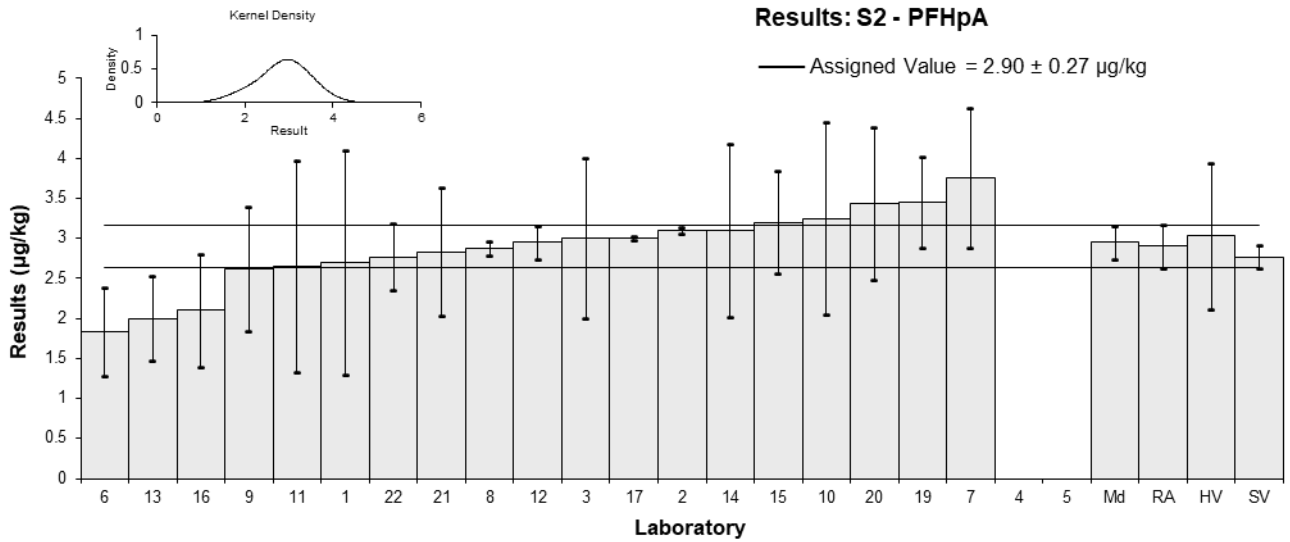


Figure 41

Table 47

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFOA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	5	2.5	126	-0.88	-0.42
2	5.4	0.045	110	-0.55	-1.28
3	7	3	94	0.77	0.31
4	NS	NS	NS		
5	5.08	0.26	NT	-0.82	-1.70
6	5.091	1.527	87	-0.81	-0.61
7	6.37	0.77	83	0.25	0.32
8	5.37	0.072	104.9	-0.58	-1.33
9	5.86	1.76	54	-0.17	-0.11
10	7.474	2.72	91.9	1.16	0.51
11	6.71	3.355	NR	0.53	0.19
12	5.68	0.4	NR	-0.32	-0.59
13	6.1	1.000	NR	0.02	0.03
14	5.3815	1.8835	58	-0.57	-0.35
15	7.3	1.46	NR	1.01	0.79
16	6.0	2.1	NR	-0.06	-0.03
17	5.451	0.186	88	-0.51	-1.12
19	7.89	1.46	91	1.50	1.17
20	6.72	1.26	89.1	0.54	0.48
21	5.52	2	81	-0.45	-0.27
22	6.42	1.03	93.7	0.29	0.30

## Statistics

<b>Assigned Value</b>	6.07	0.52
<b>Spike Value</b>	5.57	0.28
<b>Homogeneity Value</b>	6.0	1.8
<b>Robust Average</b>	6.07	0.52
<b>Median</b>	5.93	0.46
<b>Mean</b>	6.09	
<b>N</b>	20	
<b>Max</b>	7.89	
<b>Min</b>	5	
<b>Robust SD</b>	0.92	
<b>Robust CV</b>	15%	

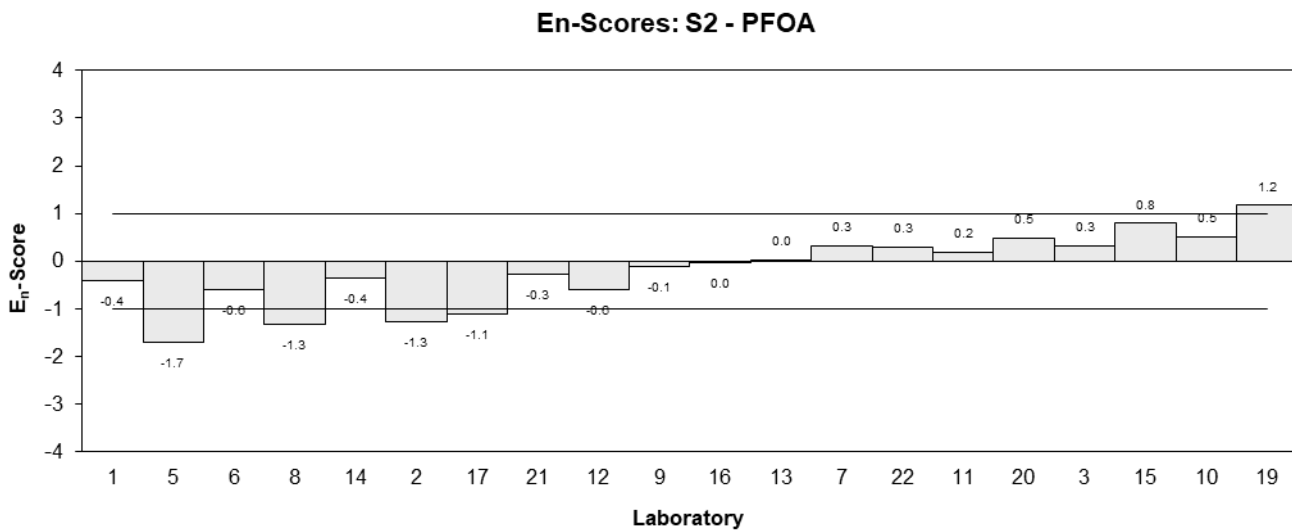
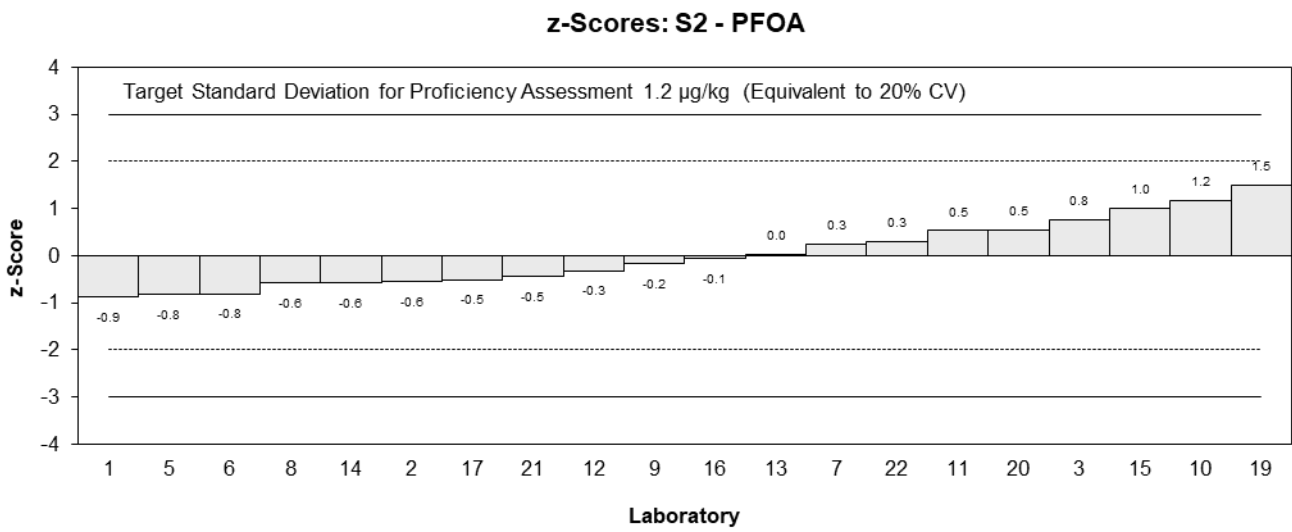
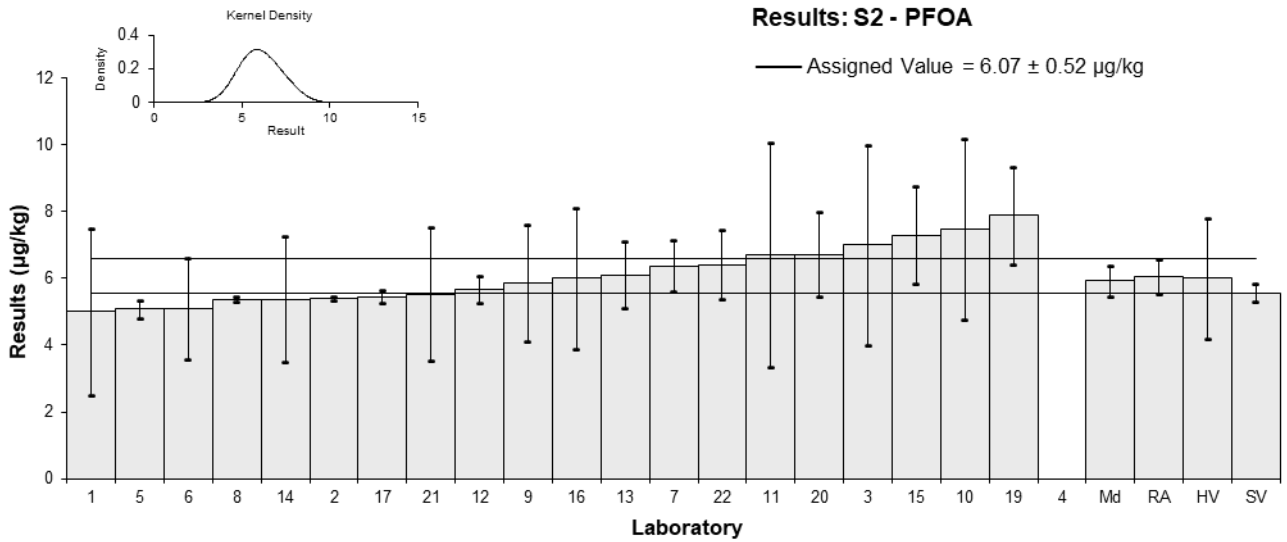


Figure 42

Table 48

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFNA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	4.3	2.2	125	-0.61	-0.27
2	4.7	0.11	118	-0.20	-0.46
3	5	2	104	0.10	0.05
4	NS	NS	NS		
5*	11.79	0.71	NT	7.03	8.35
6	2.691	0.807	86	-2.25	-2.43
7	5.59	0.80	69	0.70	0.76
8	4.91	0.066	NR	0.01	0.02
9	4.32	1.3	37	-0.59	-0.42
10	4.93	1.73	112.64	0.03	0.02
11	6.35	3.175	NR	1.48	0.45
12	4.7	0.33	NR	-0.20	-0.37
13	4.8	0.500	NR	-0.10	-0.15
14	4.283	1.4991	57	-0.63	-0.40
15	7.5	1.5	NR	2.65	1.67
16	5.2	1.8	NR	0.31	0.16
17	4.001	0.192	88	-0.92	-1.95
19	6.47	0.977	93	1.60	1.48
20	5.15	1.47	83.9	0.26	0.16
21	4.62	1	77	-0.29	-0.26
22	4.78	0.765	100	-0.12	-0.14

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	4.90	0.42
<b>Spike Value</b>	4.64	0.23
<b>Homogeneity Value</b>	4.9	1.5
<b>Robust Average</b>	5.03	0.53
<b>Median</b>	4.86	0.36
<b>Mean</b>	5.30	
<b>N</b>	20	
<b>Max</b>	11.79	
<b>Min</b>	2.691	
<b>Robust SD</b>	0.96	
<b>Robust CV</b>	19%	

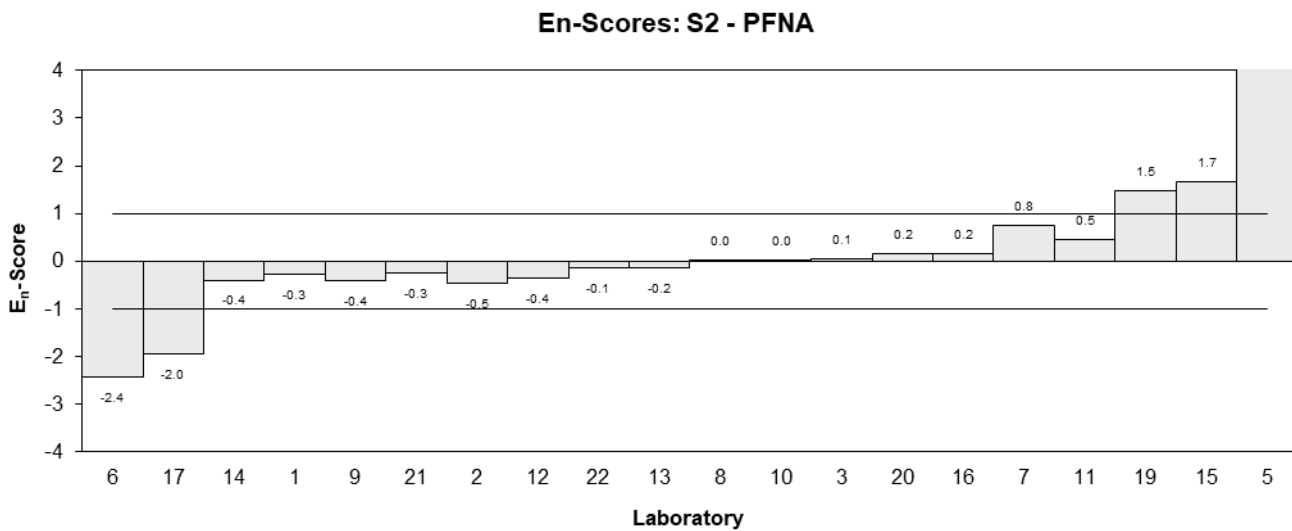
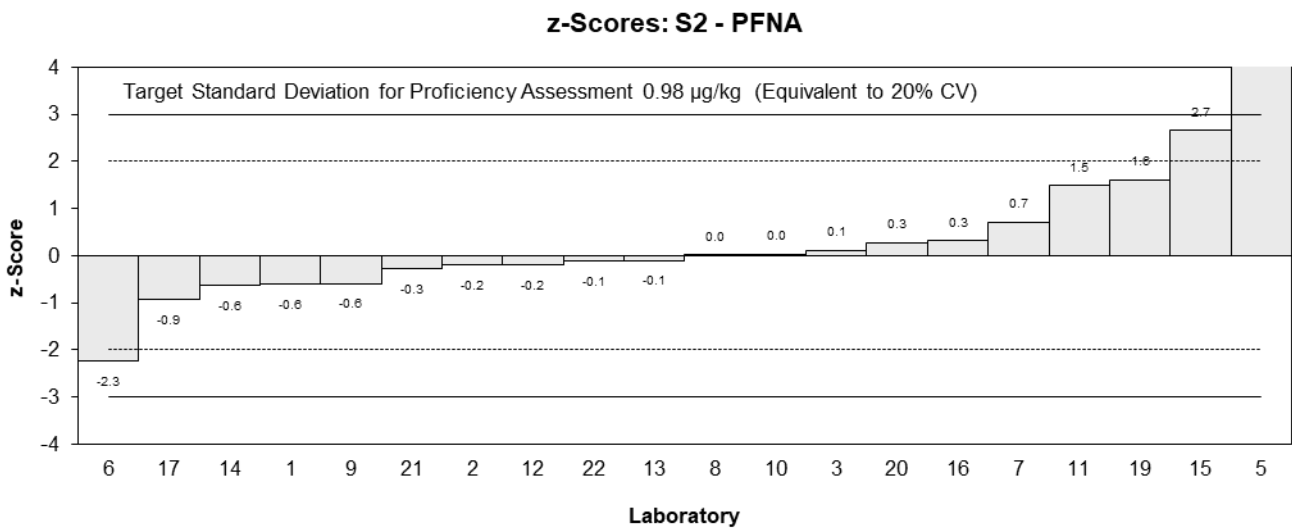
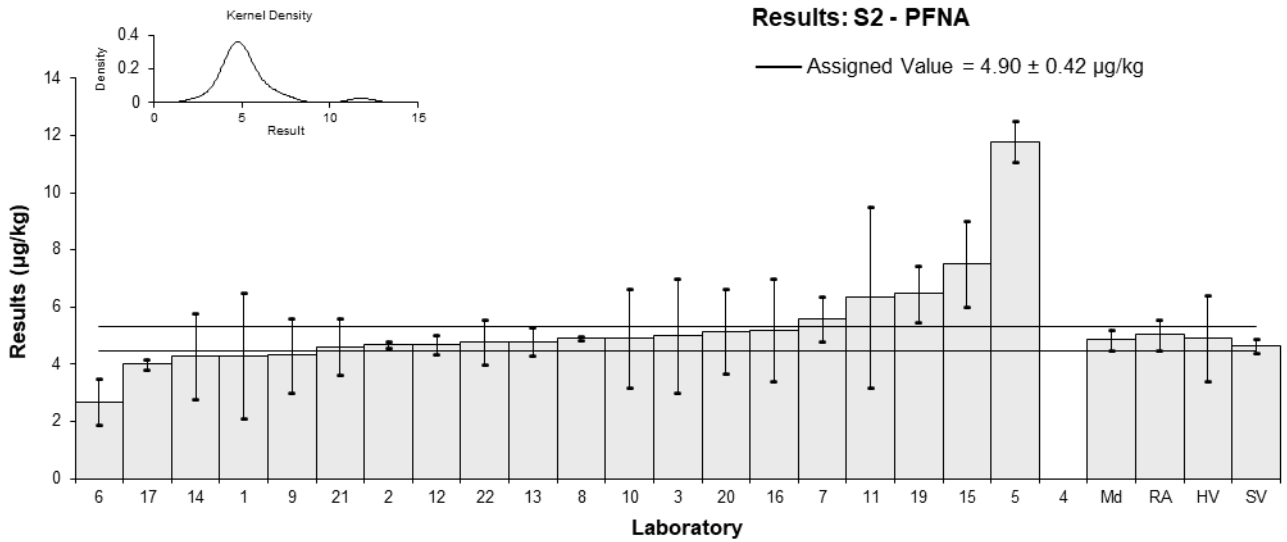


Figure 43

Table 49

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFDA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	2	1	98	-0.76	-0.35
2	2.0	0.0057	103	-0.76	-1.44
3	3	1	100	1.36	0.62
4	NS	NS	NS		
5	NT	NT	NT		
6	1.821	0.546	95	-1.14	-0.90
7	2.72	0.06	41	0.76	1.40
8	2.02	0.045	NR	-0.72	-1.34
9	2.2	0.66	76	-0.34	-0.23
10	2.824	1	103.37	0.98	0.45
11	2.48	1.24	NR	0.25	0.09
12	2.31	0.17	NR	-0.11	-0.17
13	2.1	0.300	NR	-0.55	-0.67
14	2.0614	0.7215	67	-0.63	-0.39
15	3.4	0.68	NR	2.20	1.44
16	2.1	0.7	NR	-0.55	-0.35
17	2.157	0.103	88	-0.43	-0.75
19	3.03	0.493	93	1.42	1.21
20	2.33	0.619	166.5	-0.06	-0.04
21	2.24	0.7	80	-0.25	-0.16
22	2.51	0.376	95	0.32	0.33

## Statistics

<b>Assigned Value</b>	2.36	0.25
<b>Spike Value</b>	2.09	0.10
<b>Homogeneity Value</b>	2.24	0.67
<b>Robust Average</b>	2.36	0.25
<b>Median</b>	2.24	0.20
<b>Mean</b>	2.38	
<b>N</b>	19	
<b>Max</b>	3.4	
<b>Min</b>	1.821	
<b>Robust SD</b>	0.43	
<b>Robust CV</b>	18%	



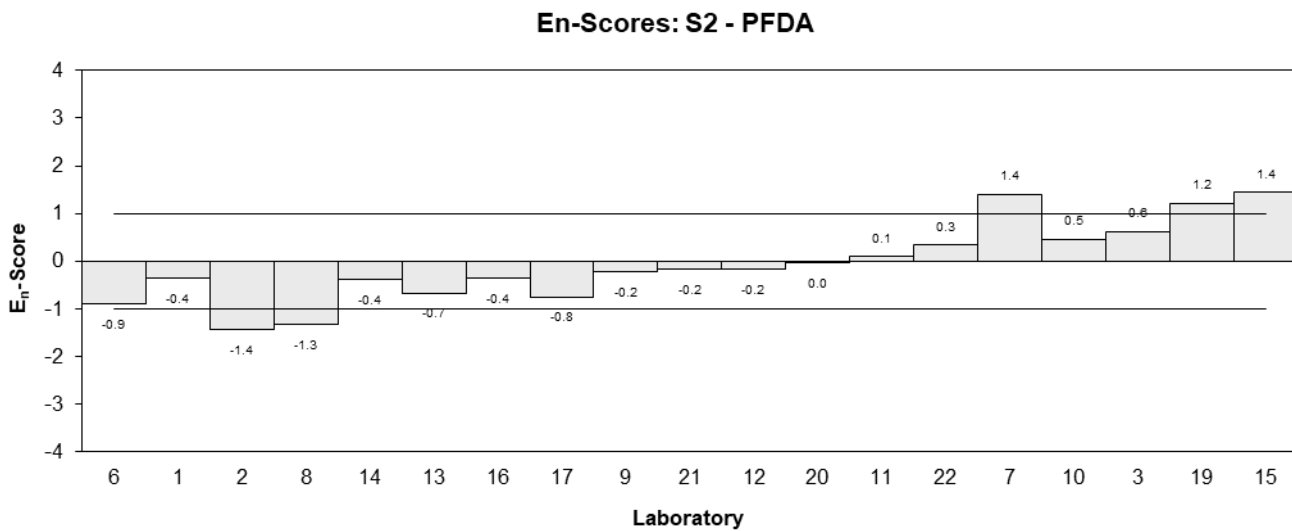
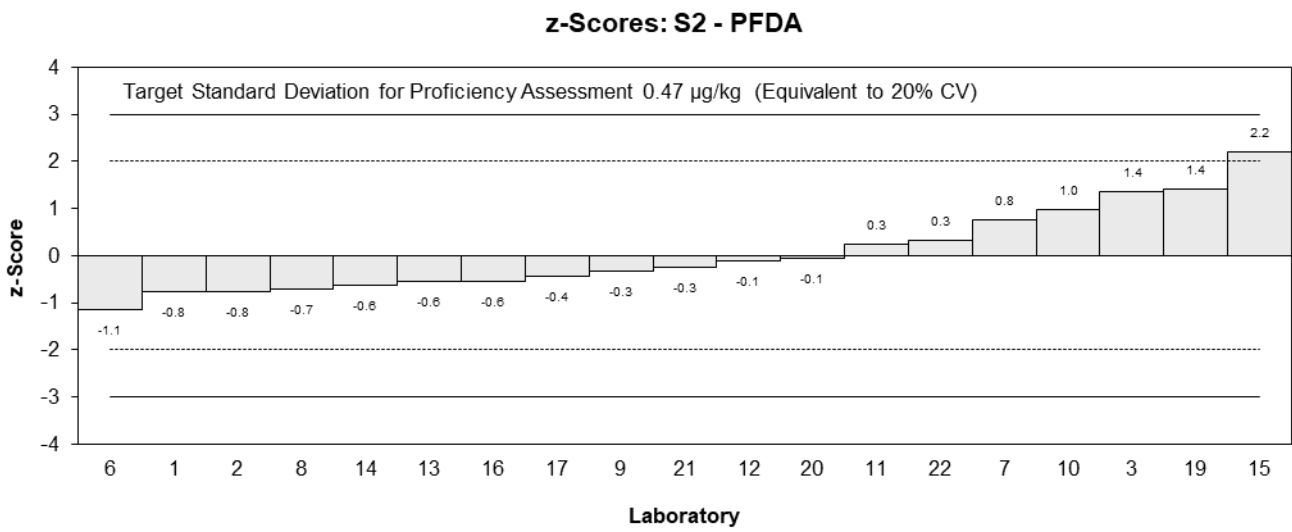
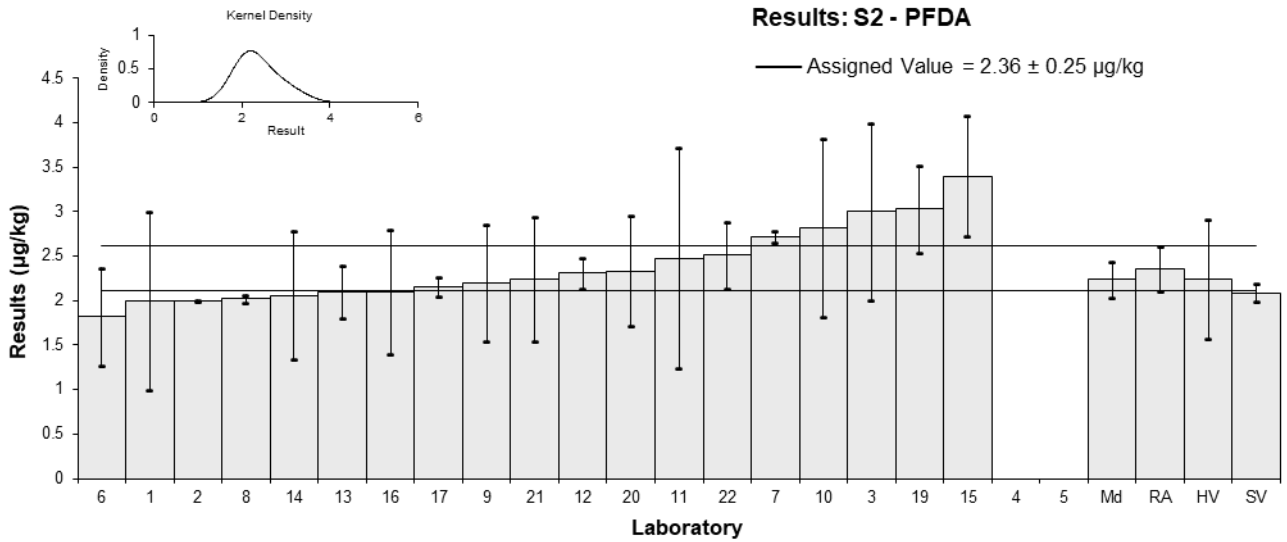


Figure 44

Table 50

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFUdA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	3.5	1.8	158	-0.69	-0.31
2	4.6	1.2	99	0.67	0.43
3	5	3	90	1.16	0.31
4	NS	NS	NS		
5	NT	NT	NT		
6	3.587	1.076	97	-0.58	-0.42
7	2.49	0.22	41	-1.93	-3.72
8	3.73	0.074	102.5	-0.41	-0.90
9	4.26	1.28	84	0.25	0.15
10	4.616	1.63	109.64	0.68	0.33
11	NT	NT	NT		
12	3.9	0.27	NR	-0.20	-0.36
13*	6.5	0.500	NR	3.00	3.96
14	3.7318	1.3061	70	-0.40	-0.24
15	NT	NT	NT		
16	3.8	1.3	NR	-0.32	-0.19
17	3.785	0.186	88	-0.34	-0.68
19	5.89	0.907	87	2.25	1.88
20	4.26	1.69	156.4	0.25	0.12
21	3.99	1	71	-0.09	-0.07
22	4.16	0.707	101	0.12	0.13

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	4.06	0.36
<b>Spike Value</b>	3.73	0.19
<b>Homogeneity Value</b>	4.2	1.3
<b>Robust Average</b>	4.14	0.41
<b>Median</b>	3.99	0.24
<b>Mean</b>	4.22	
<b>N</b>	17	
<b>Max</b>	6.5	
<b>Min</b>	2.49	
<b>Robust SD</b>	0.67	
<b>Robust CV</b>	16%	

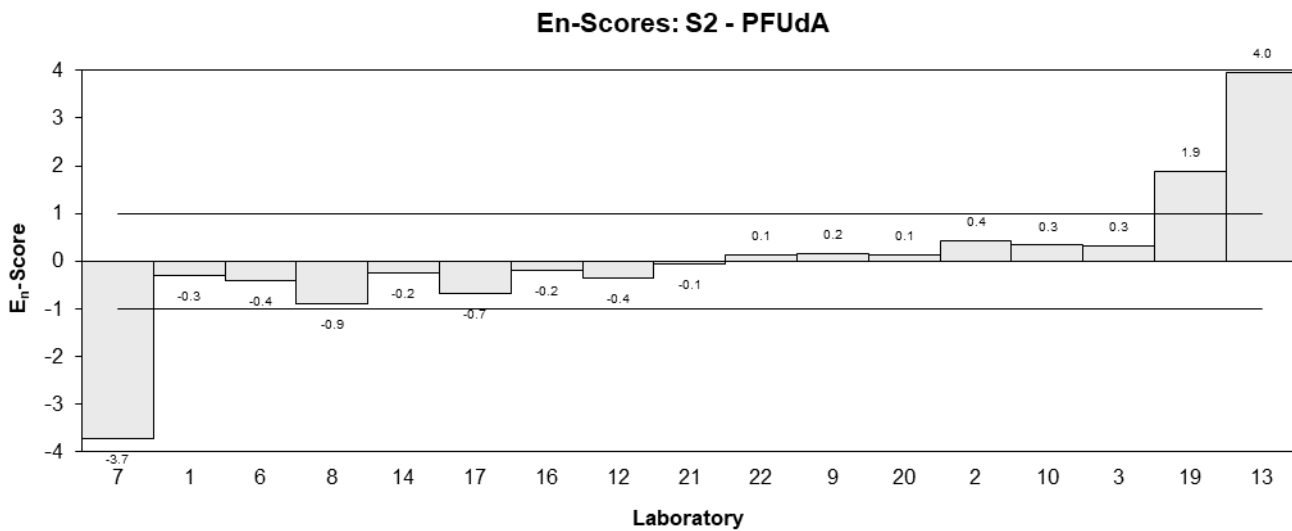
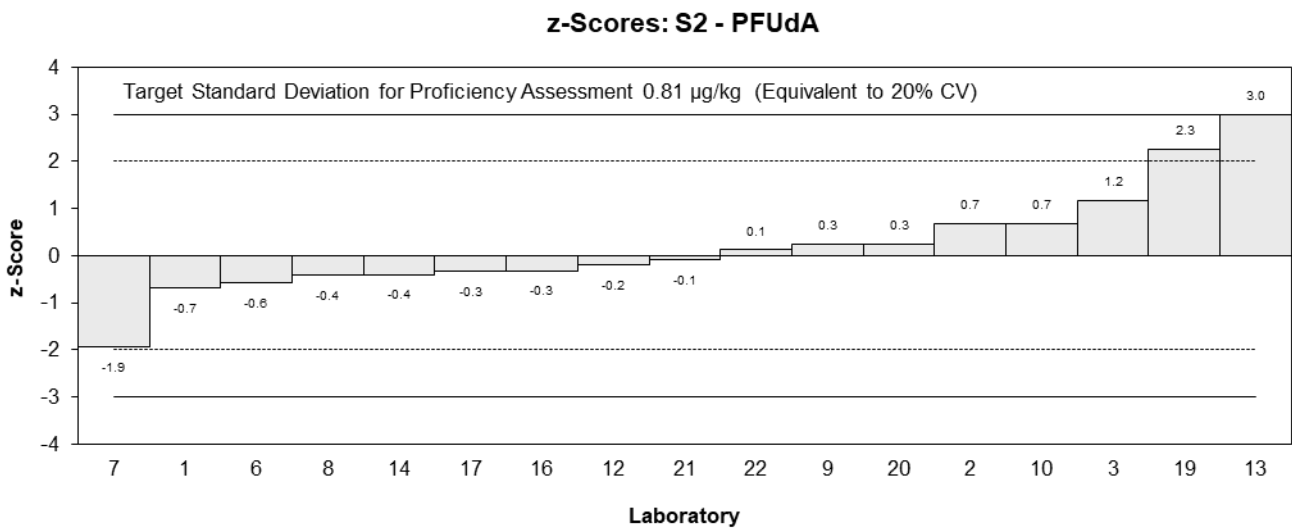
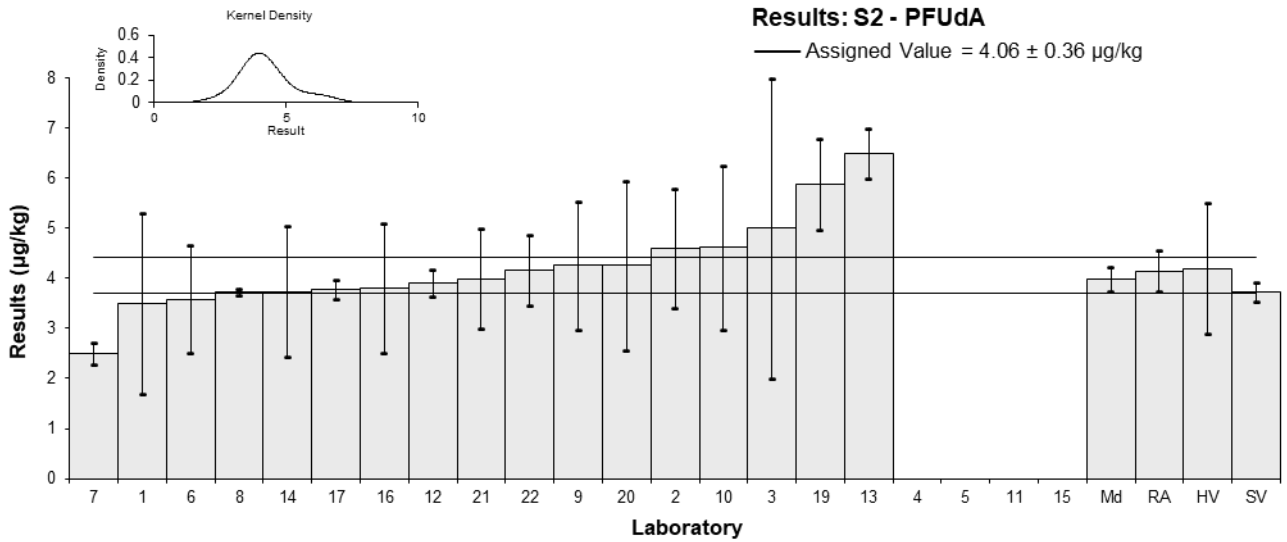


Figure 45

Table 51

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFTTrDA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	NT	NT	NT		
2	2.4	0.34	110	-0.86	-0.90
3	4	2	109	1.90	0.54
4	NS	NS	NS		
5	NT	NT	NT		
6	2.292	0.688	96	-1.05	-0.74
7	3.16	0.23	12	0.45	0.52
8	3.15	0.074	NR	0.43	0.56
9	3.03	0.9	41	0.22	0.13
10	3.242	1	111	0.59	0.31
11	2.8	1.4	NR	-0.17	-0.07
12	1.64	0.12	NR	-2.17	-2.76
13	2.6	0.300	NR	-0.52	-0.56
14	NT	NT	NT		
15	NT	NT	NT		
16	3.4	1.2	NR	0.86	0.39
17	1.979	0.04	97	-1.59	-2.08
19	3.59	0.72	90	1.19	0.82
20	2.40	0.855	106.8	-0.86	-0.52
21	2.79	0.8	NR	-0.19	-0.12
22	3.72	0.856	77.3	1.41	0.85

## Statistics

<b>Assigned Value</b>	2.90	0.44
<b>Spike Value</b>	2.78	0.14
<b>Homogeneity Value</b>	2.69	0.81
<b>Robust Average</b>	2.90	0.44
<b>Median</b>	2.92	0.46
<b>Mean</b>	2.89	
<b>N</b>	16	
<b>Max</b>	4	
<b>Min</b>	1.64	
<b>Robust SD</b>	0.70	
<b>Robust CV</b>	24%	

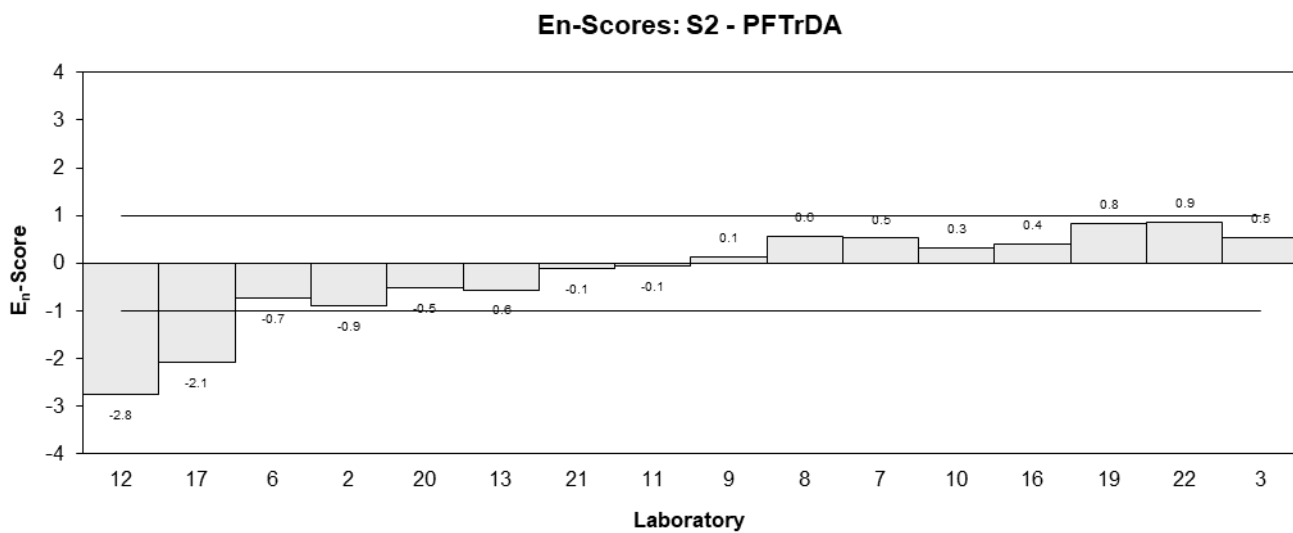
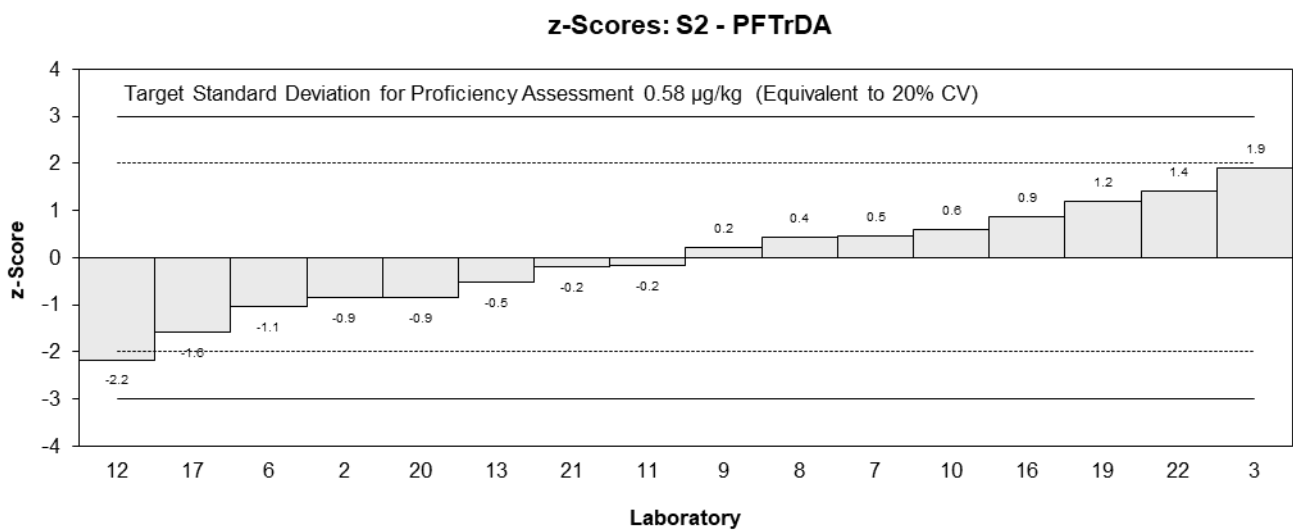
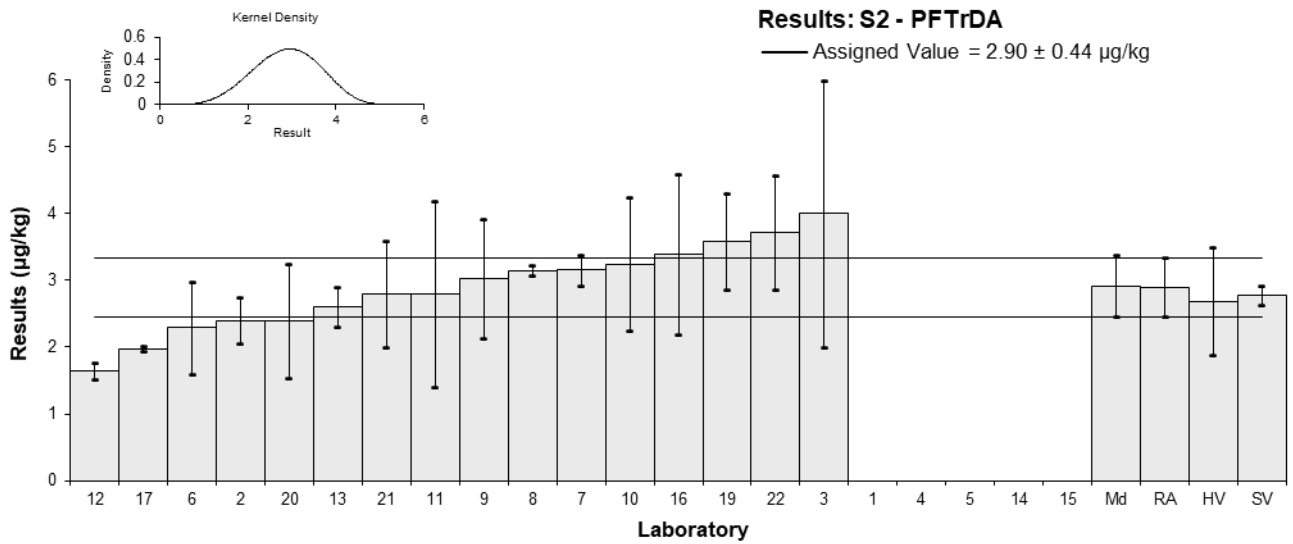


Figure 46

Table 52

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	PFOSA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	12	6	81	-0.04	-0.02
2	12	0.00	108	-0.04	-0.09
3	18	6	118	2.44	0.97
4	NS	NS	NS		
5	NT	NT	NT		
6	10.006	3.002	87	-0.87	-0.65
7	11.99	1.35	12	-0.05	-0.06
8	11.0	0.062	70.4	-0.45	-1.00
9	10.69	3.2	33	-0.58	-0.42
10	14.01	5.5	112.43	0.79	0.34
11	NT	NT	NT		
12	NT	NT	NT		
13	12	0.300	NR	-0.04	-0.09
14	NT	NT	NT		
15	NT	NT	NT		
16**	0.82	0.3	NR	-4.66	-9.89
17	11.258	0.55	35	-0.35	-0.68
19	15.6	4.61	91	1.45	0.74
20	10.9	2.08	29.6	-0.50	-0.51
21	10.8	3	82	-0.54	-0.41
22	13.2	1.84	130	0.45	0.51

\*\* Extreme Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	12.1	1.1
<b>Spike Value</b>	12.3	0.6
<b>Homogeneity Value</b>	11.7	3.5
<b>Robust Average</b>	12.1	1.1
<b>Median</b>	12.0	1.1
<b>Mean</b>	12.4	
<b>N</b>	14	
<b>Max</b>	18	
<b>Min</b>	10.006	
<b>Robust SD</b>	1.7	
<b>Robust CV</b>	14%	

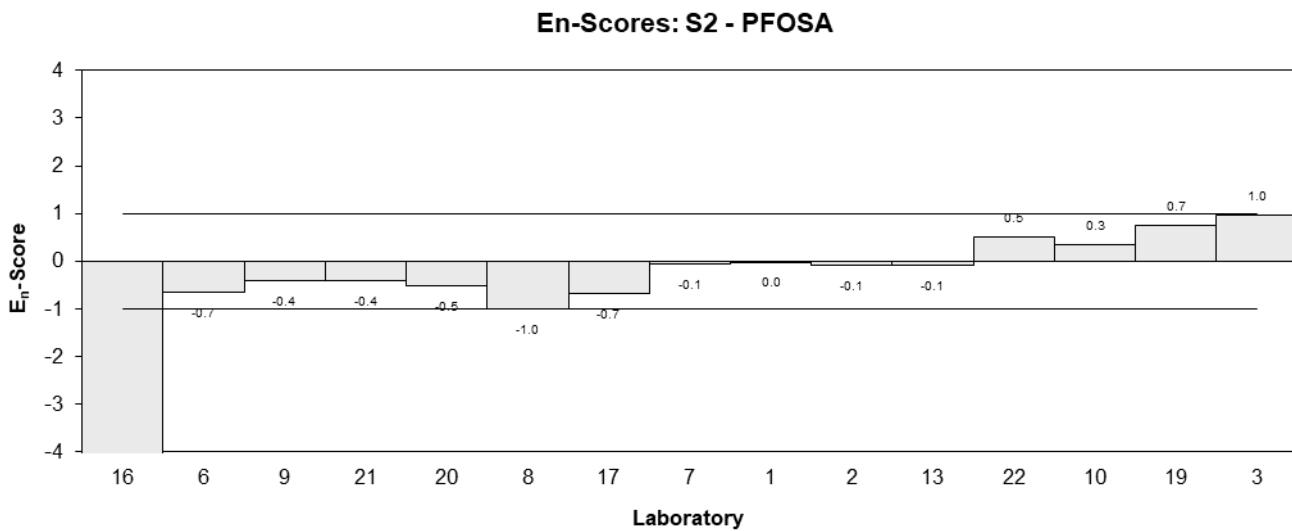
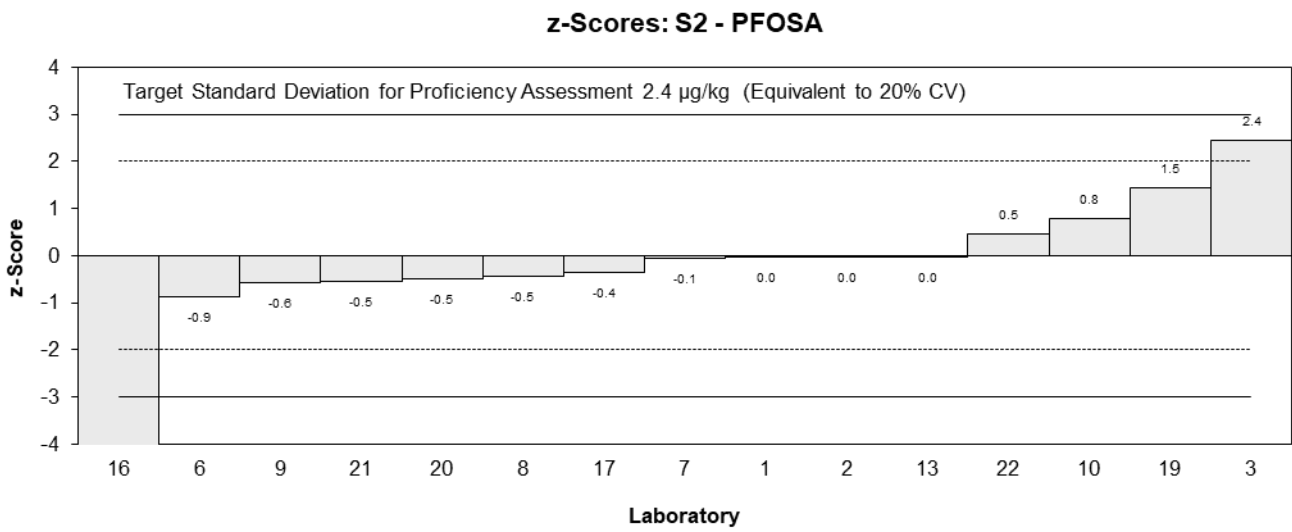
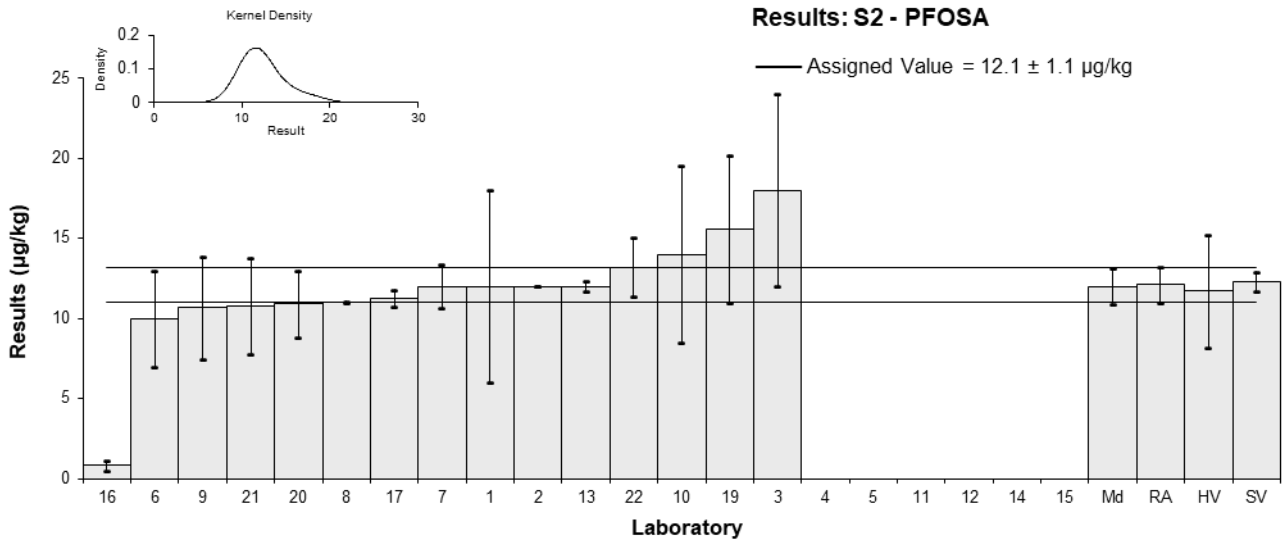


Figure 47

Table 53

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	EtFOSA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	6	3	23	-0.52	-0.20
2	7.0	0.27	103	0.22	0.17
3	8	3	90	0.97	0.38
4	NS	NS	NS		
5	NT	NT	NT		
6	3.802	1.141	98	-2.16	-1.42
7	NT	NT	NT		
8	NT	NT	NT		
9	7.69	2.3	17	0.74	0.35
10	8.942	3.5	87.05	1.67	0.58
11	NT	NT	NT		
12	NT	NT	NT		
13	5.9	0.300	NR	-0.60	-0.46
14	NT	NT	NT		
15	NT	NT	NT		
16	NT	NT	NT		
17	NT	NT	NT		
19*	11.7	2.83	64	3.73	1.51
20	3.69	0.187	29.6	-2.25	-1.76
21	8.01	3	14	0.98	0.38
22	8.36	1.34	52.2	1.24	0.77

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	6.7	1.7
<b>Spike Value</b>	7.46	0.37
<b>Homogeneity Value</b>	9.7	3.9
<b>Robust Average</b>	7.1	1.8
<b>Median</b>	7.7	1.4
<b>Mean</b>	7.2	
<b>N</b>	11	
<b>Max</b>	11.7	
<b>Min</b>	3.69	
<b>Robust SD</b>	2.4	
<b>Robust CV</b>	34%	



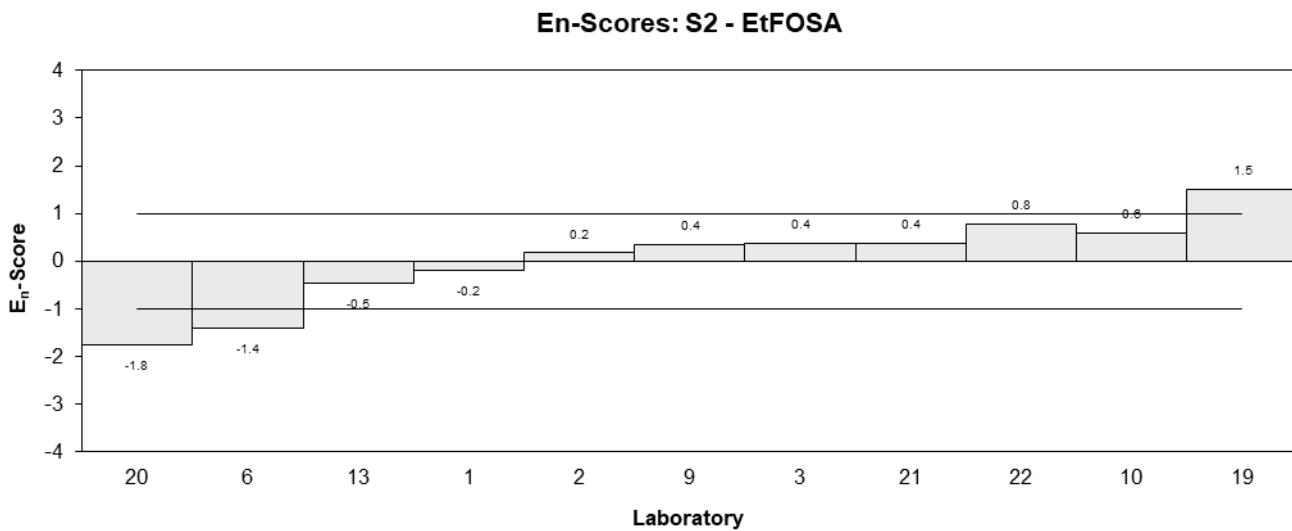
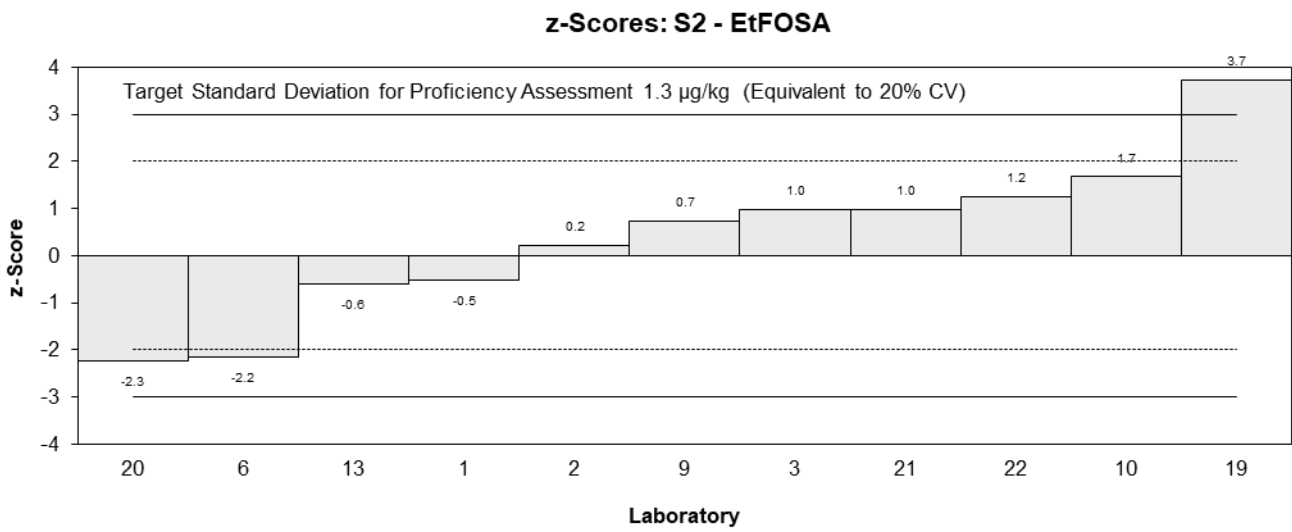
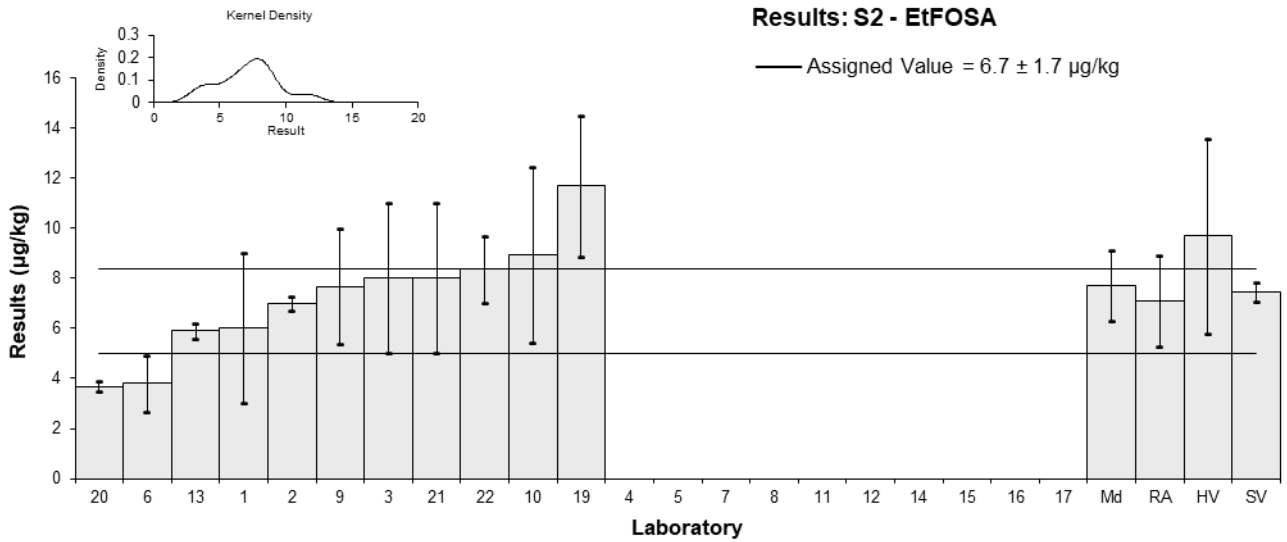


Figure 48

Table 54

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	EtFOSAA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	7	3.5	291	-1.07	-0.52
2	9.0	1.1	97	0.06	0.07
3	9	3	94	0.06	0.03
4	NS	NS	NS		
5	NT	NT	NT		
6	8.22	2.466	89	-0.38	-0.26
7	8.02	1.39	20	-0.49	-0.51
8	NT	NT	NT		
9	10.07	3.02	98	0.66	0.37
10	9.832	3.4	176.09	0.52	0.26
11	NT	NT	NT		
12	NT	NT	NT		
13	8.0	1.000	NR	-0.51	-0.64
14	NT	NT	NT		
15	NT	NT	NT		
16	NT	NT	NT		
17	NT	NT	NT		
19*	16.4	3.89	68	4.21	1.87
20	NT	NT	NT		
21	8.96	3	85	0.03	0.02
22	10.8	2.37	98.1	1.07	0.74

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	8.9	1.0
<b>Spike Value</b>	9.26	0.46
<b>Homogeneity Value</b>	8.2	2.5
<b>Robust Average</b>	9.1	1.1
<b>Median</b>	9.0	1.1
<b>Mean</b>	9.6	
<b>N</b>	11	
<b>Max</b>	16.4	
<b>Min</b>	7	
<b>Robust SD</b>	1.5	
<b>Robust CV</b>	16%	

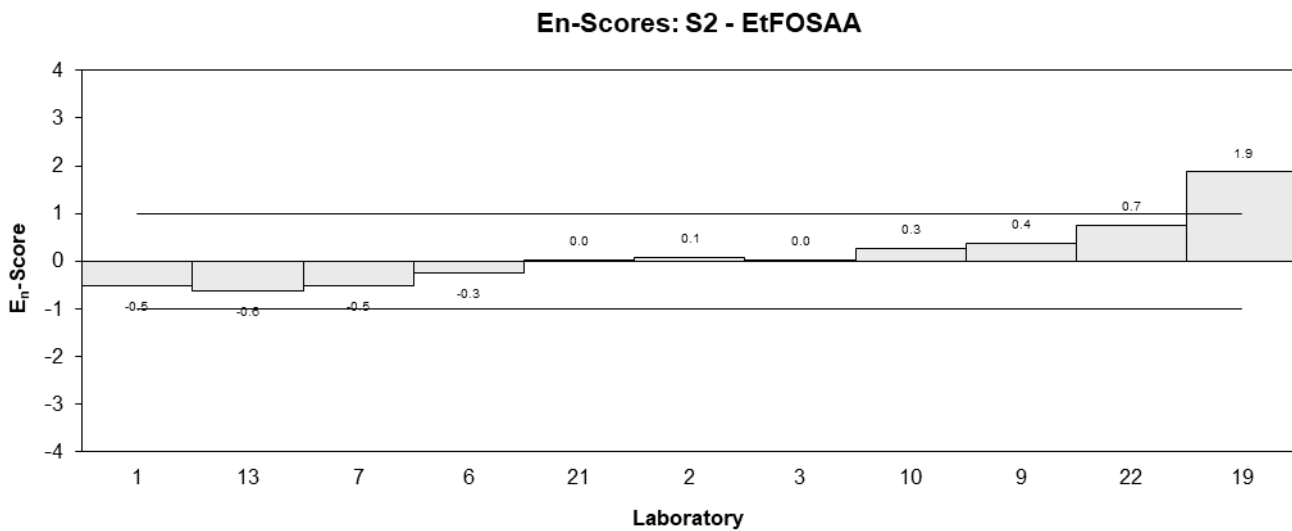
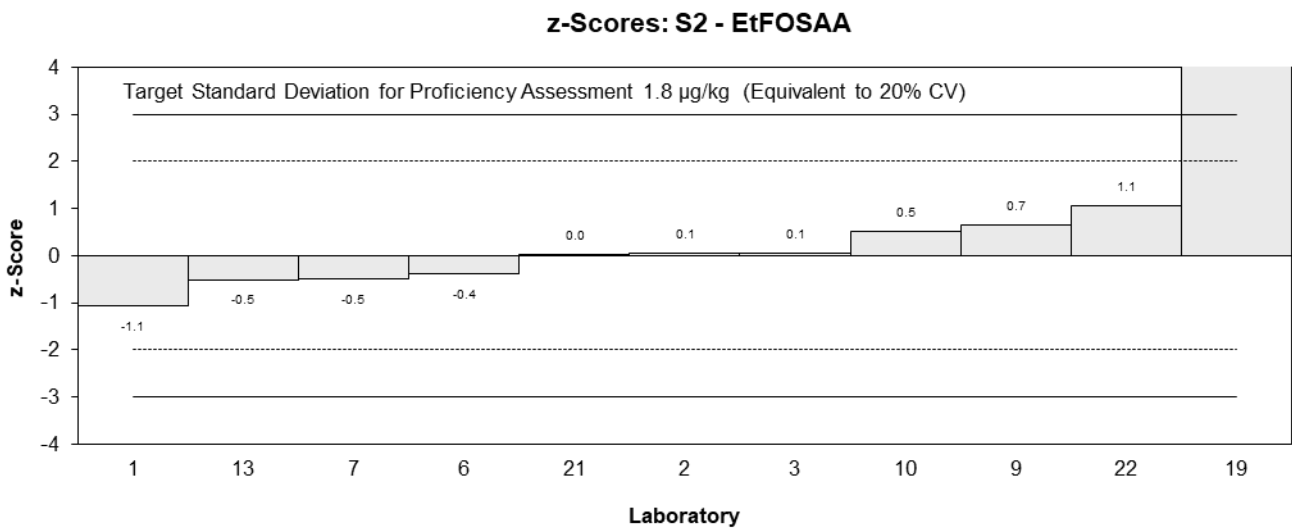
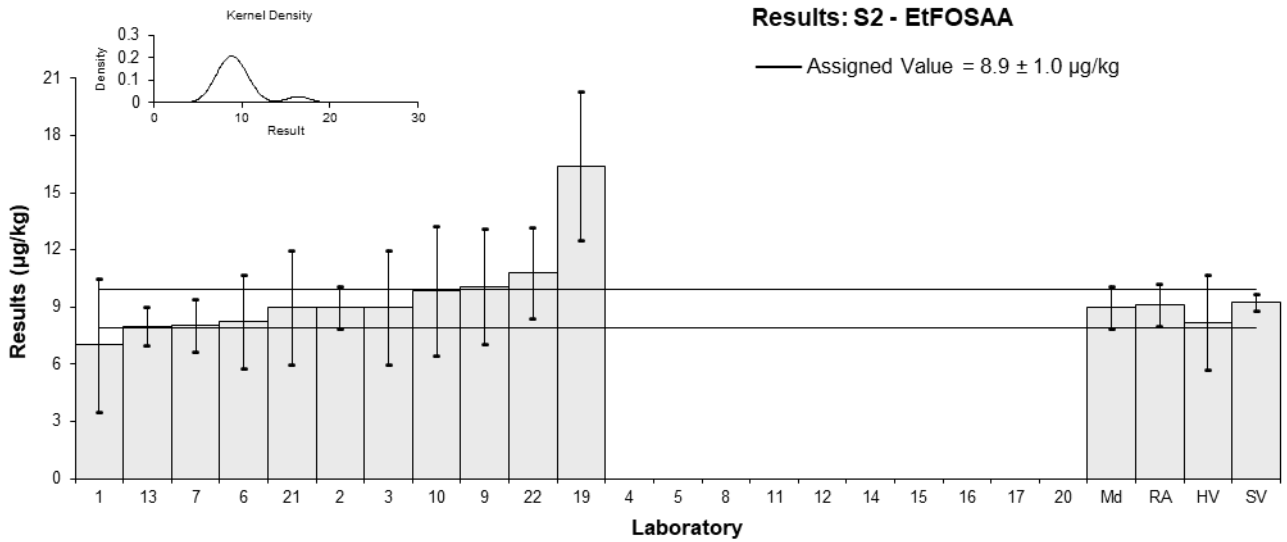


Figure 49

Table 55

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	6:2FTS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	4	2	280	-0.63	-0.28
2	4.3	0.27	90	-0.31	-0.56
3	5	2	101	0.46	0.21
4	NS	NS	NS		
5	NT	NT	NT		
6*	1.371	0.411	65	-3.50	-5.46
7	4.83	0.29	348	0.27	0.49
8	3.42	0.14	108.4	-1.27	-2.62
9	4.6	1.38	93	0.02	0.01
10	4.78	2	171.35	0.22	0.10
11	5	2.5	NR	0.46	0.17
12	NT	NT	NT		
13	4.3	0.500	NR	-0.31	-0.43
14	NT	NT	NT		
15*	7.4	1.48	NR	3.08	1.83
16*	2.0	0.7	NR	-2.82	-3.16
17	3.789	0.095	77	-0.86	-1.84
19	5.29	1.1	157	0.78	0.60
20	NT	NT	NT		
21	4.75	1	69	0.19	0.16
22	5.24	2.83	88.4	0.72	0.23

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	4.58	0.42
<b>Spike Value</b>	4.39	0.22
<b>Homogeneity Value</b>	5.0	1.5
<b>Robust Average</b>	4.46	0.57
<b>Median</b>	4.68	0.44
<b>Mean</b>	4.38	
<b>N</b>	16	
<b>Max</b>	7.4	
<b>Min</b>	1.371	
<b>Robust SD</b>	0.90	
<b>Robust CV</b>	20%	

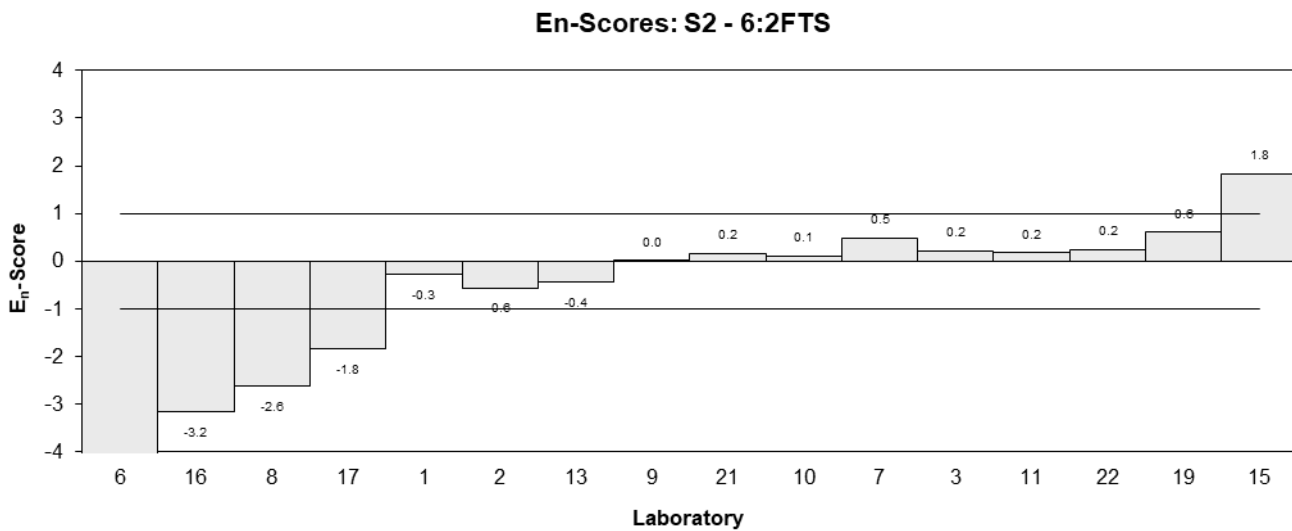
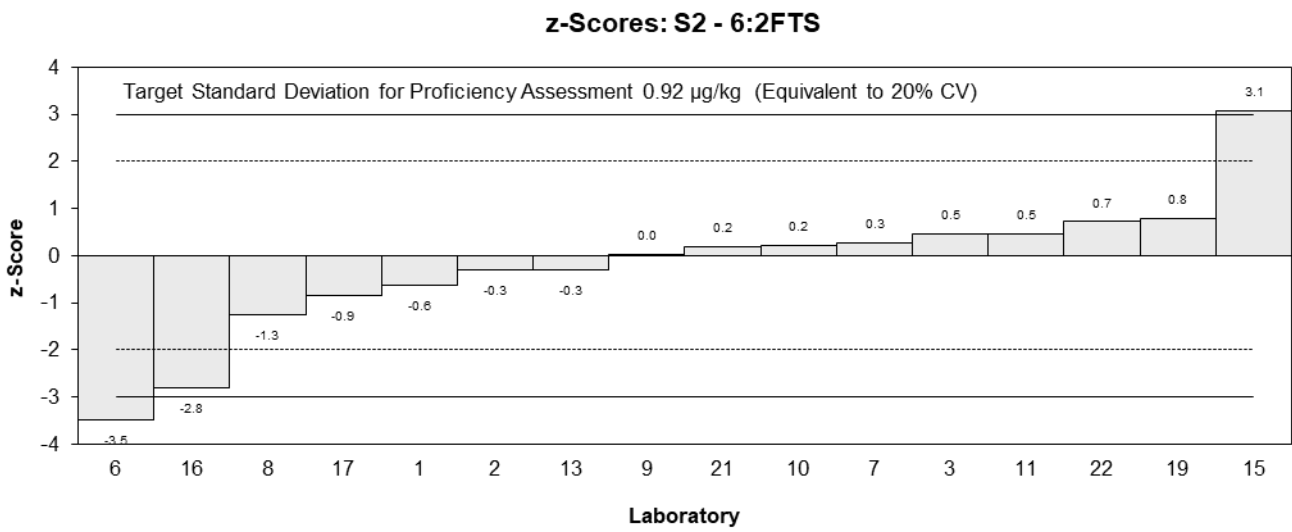
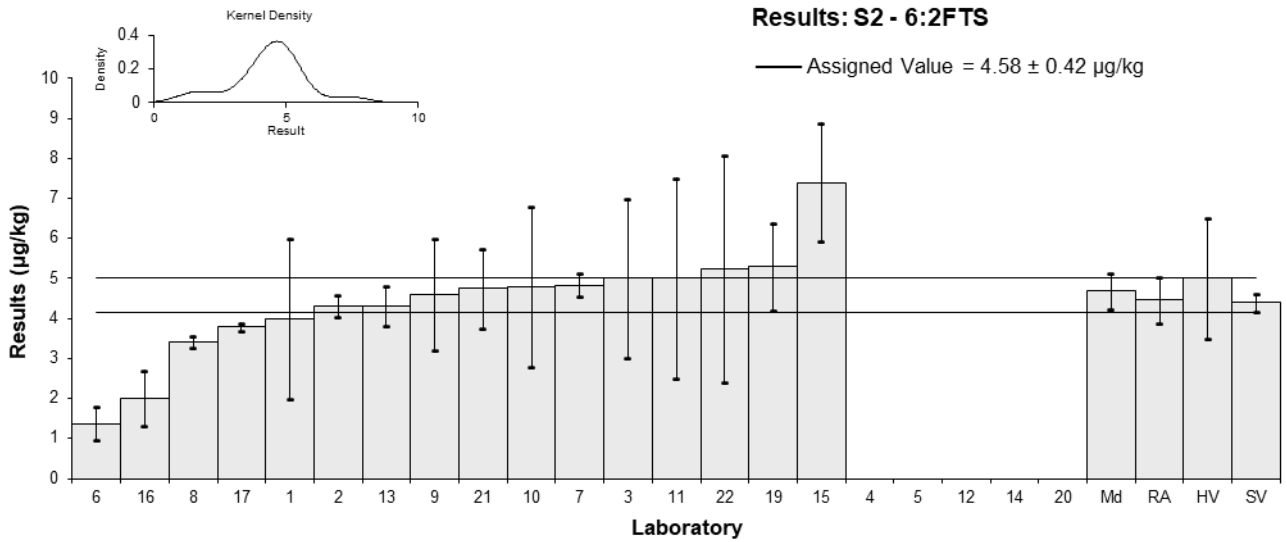


Figure 50

Table 56

**Sample Details**

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	GenX
<b>Unit</b>	µg/kg

**Participant Results**

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	NT	NT	NT		
2	9.3	0.67	121	0.46	0.99
3	< 50	NR	106		
4	NS	NS	NS		
5	NT	NT	NT		
6*	3.684	1.105	97	-2.84	-4.06
7	NT	NT	NT		
8	7.97	0.071	NR	-0.32	-1.21
9	8.91	2.67	31	0.24	0.15
10	NT	NT	NT		
11	8.73	4.365	NR	0.13	0.05
12	8.04	0.57	NR	-0.28	-0.65
13	8.8	0.500	NR	0.17	0.44
14	NT	NT	NT		
15	NT	NT	NT		
16	NT	NT	NT		
17	8.571	0.007	87	0.04	0.14
19	7.83	2.09	68	-0.40	-0.32
20	NT	NT	NT		
21	8.07	2	71	-0.26	-0.21
22	8.84	1.86	64	0.19	0.17

\* Outlier, see Section 4.2

**Statistics**

<b>Assigned Value</b>	8.51	0.44
<b>Spike Value</b>	8.40	0.42
<b>Homogeneity Value</b>	8.0	2.4
<b>Robust Average</b>	8.41	0.49
<b>Median</b>	8.57	0.56
<b>Mean</b>	8.07	
<b>N</b>	11	
<b>Max</b>	9.3	
<b>Min</b>	3.684	
<b>Robust SD</b>	0.64	
<b>Robust CV</b>	7.7%	

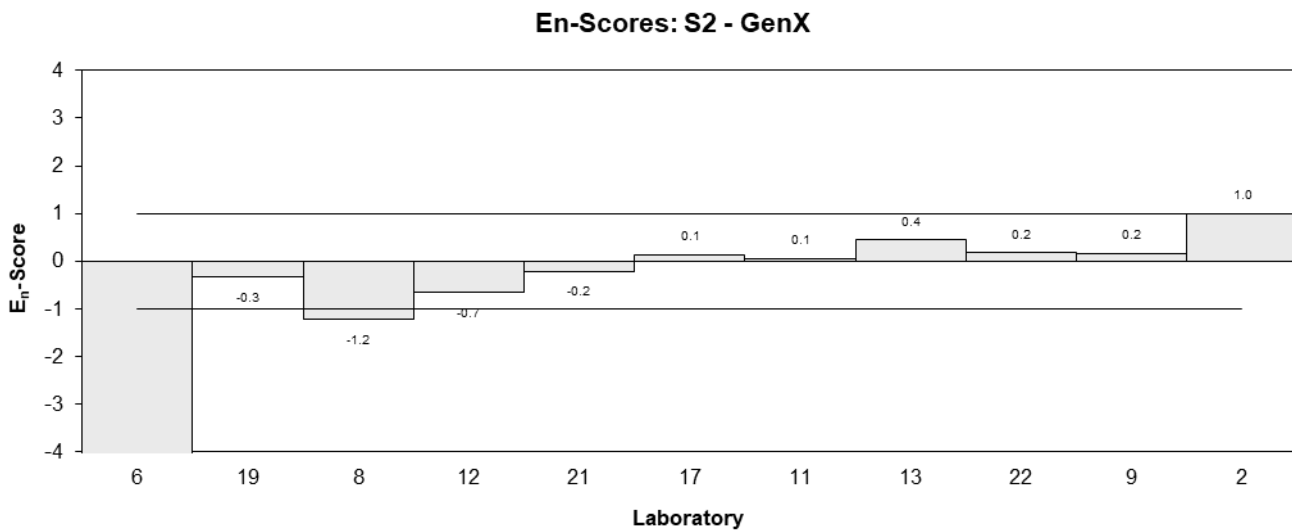
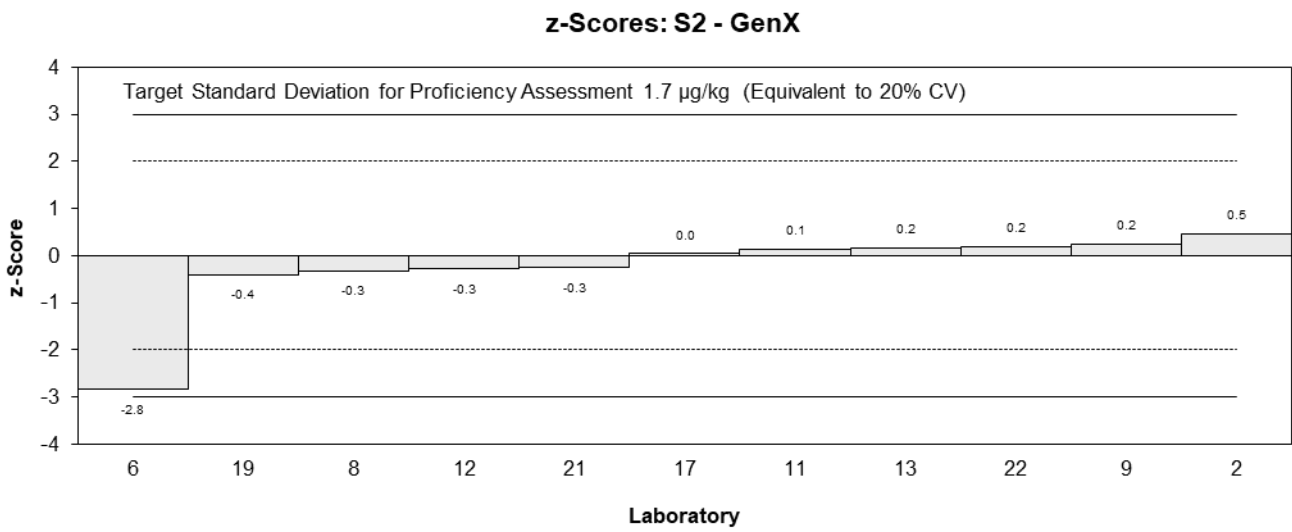
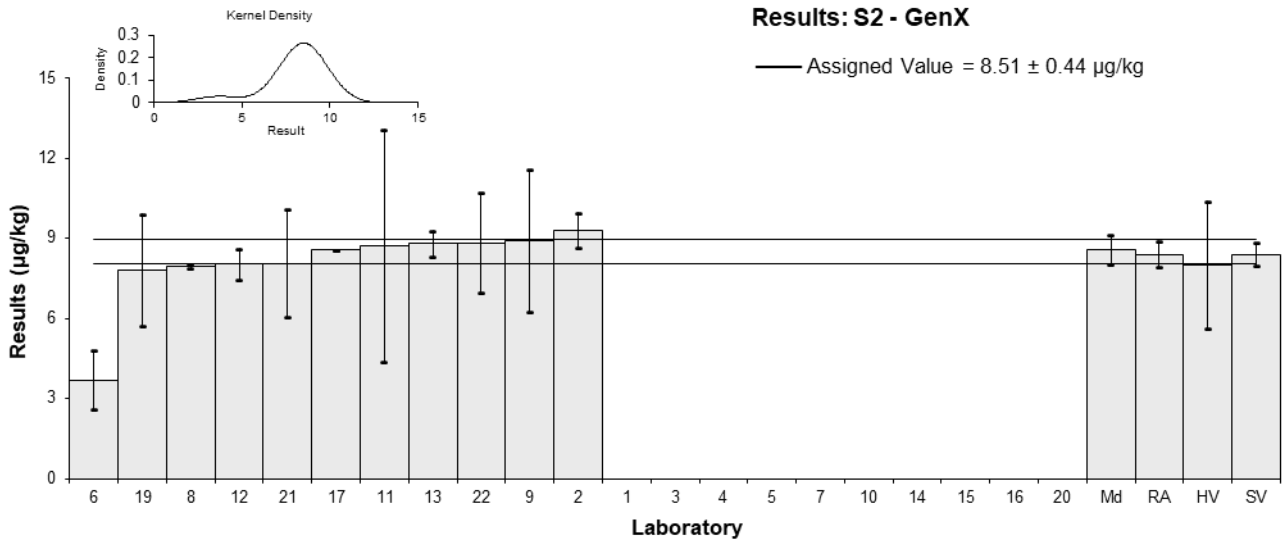


Figure 51

Table 57

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	ADONA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	NT	NT	NT		
2	14	0.62	105	0.04	0.04
3	16	6	103	0.76	0.33
4	NS	NS	NS		
5	NT	NT	NT		
6	8.693	2.608	86	-1.87	-1.50
7	13.94	1.19	83	0.01	0.02
8	16.4	0.053	NR	0.90	1.09
9	8.83	2.65	79	-1.82	-1.44
10	NT	NT	NT		
11	NT	NT	NT		
12	11.48	0.82	NR	-0.87	-0.99
13	11	0.800	NR	-1.04	-1.19
14	NT	NT	NT		
15	NT	NT	NT		
16	15	5.3	NR	0.40	0.19
17	15.601	0.386	88	0.61	0.73
19	17.4	4.98	97	1.26	0.64
20	NT	NT	NT		
21	15.3	5	NR	0.50	0.25
22	16.8	7.31	64	1.04	0.38

## Statistics

<b>Assigned Value</b>	13.9	2.3
<b>Spike Value</b>	15.8	0.8
<b>Homogeneity Value</b>	17.9	5.4
<b>Robust Average</b>	13.9	2.3
<b>Median</b>	15.0	1.4
<b>Mean</b>	13.9	
<b>N</b>	13	
<b>Max</b>	17.4	
<b>Min</b>	8.693	
<b>Robust SD</b>	3.2	
<b>Robust CV</b>	23%	



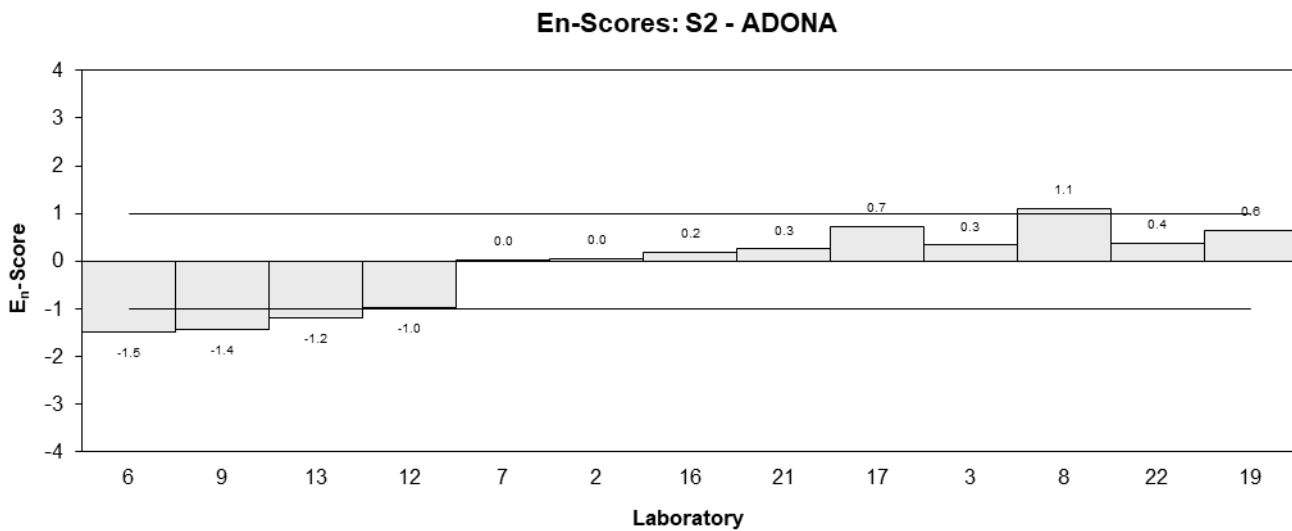
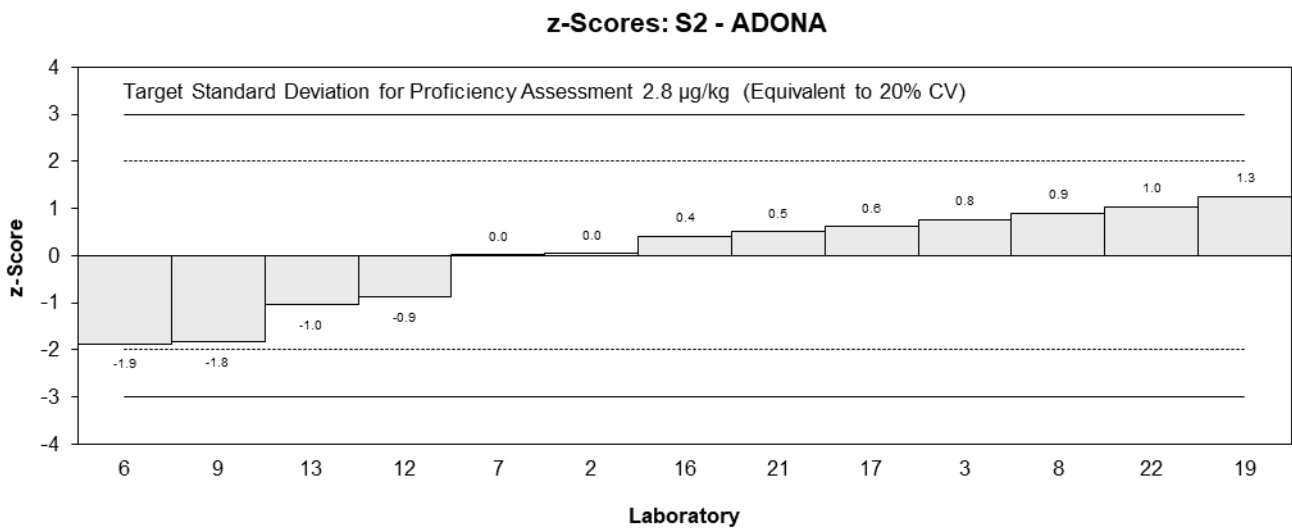
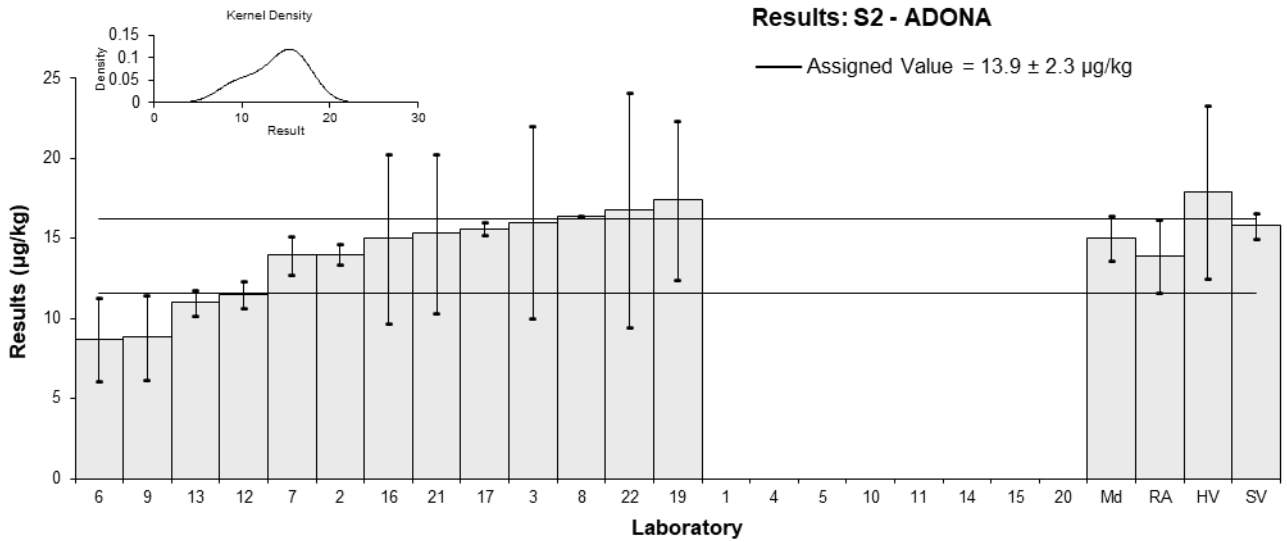


Figure 52

Table 58

**Sample Details**

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	9CI-PF3ONS
<b>Unit</b>	µg/kg

**Participant Results**

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	NT	NT	NT		
2	19	0.31	112	0.14	0.18
3	19	6	107	0.14	0.08
4	NS	NS	NS		
5	NT	NT	NT		
6	12.976	3.893	98	-1.49	-1.15
7	18.00	2.35	79	-0.14	-0.14
8	16.0	0.083	NR	-0.68	-0.89
9	15.78	4.73	99	-0.74	-0.49
10	NT	NT	NT		
11	NT	NT	NT		
12	18.85	1.34	NR	0.09	0.11
13*	38	5.000	NR	5.27	3.40
14	NT	NT	NT		
15	NT	NT	NT		
16	NT	NT	NT		
17	16.547	1.118	98	-0.53	-0.65
19	23.7	5.51	97	1.41	0.84
20	NT	NT	NT		
21	19.7	8	NR	0.32	0.14
22	25.2	7.04	64	1.81	0.88

\* Outlier, see Section 4.2

**Statistics**

<b>Assigned Value</b>	18.5	2.8
<b>Spike Value</b>	19.0	1.0
<b>Homogeneity Value</b>	20.4	8.1
<b>Robust Average</b>	19.2	3.2
<b>Median</b>	18.9	2.8
<b>Mean</b>	20.2	
<b>N</b>	12	
<b>Max</b>	38	
<b>Min</b>	12.976	
<b>Robust SD</b>	4.5	
<b>Robust CV</b>	23%	

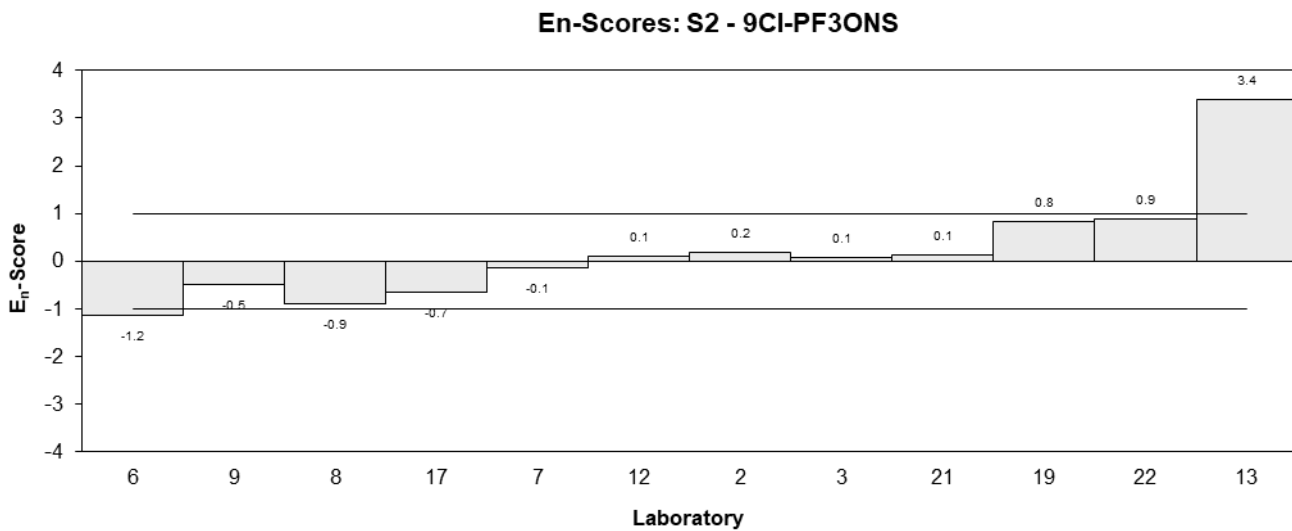
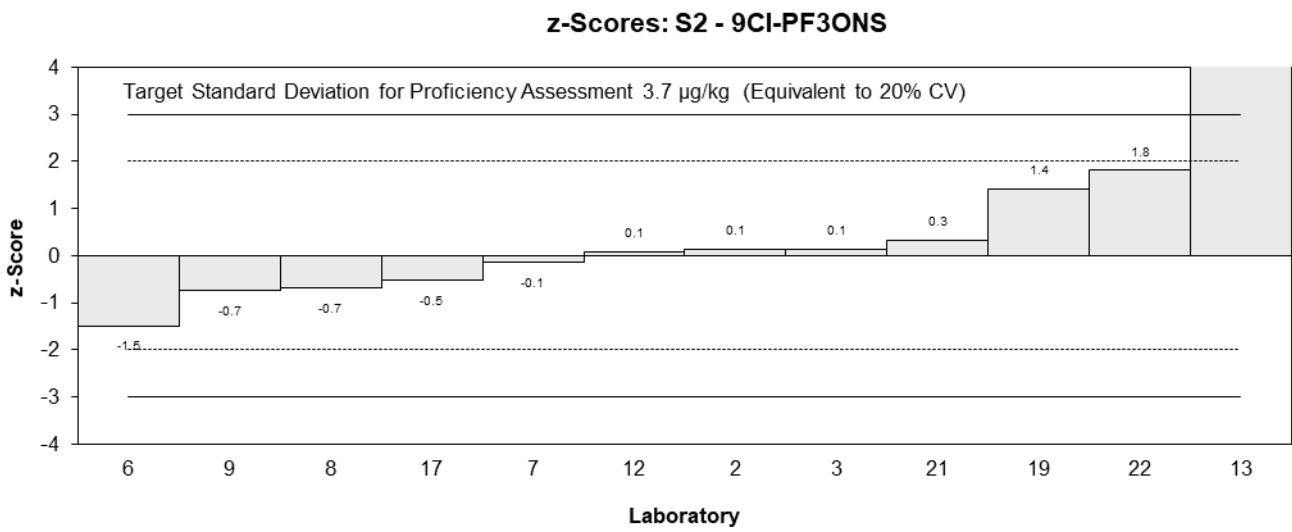
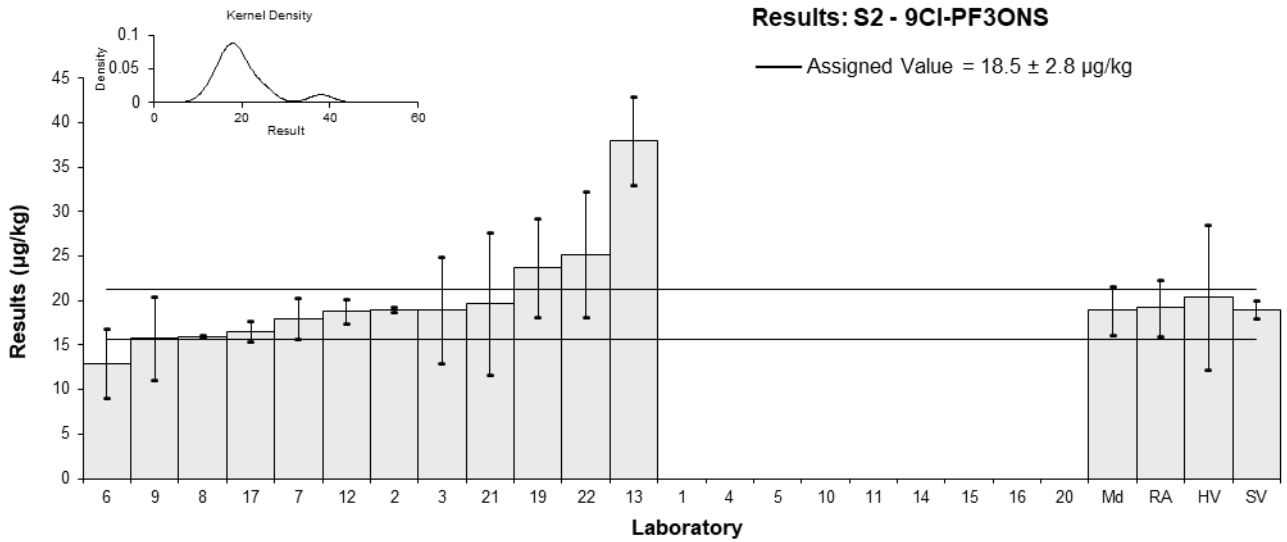


Figure 53

Table 59

## Sample Details

<b>Sample No.</b>	S2
<b>Matrix</b>	Spinach
<b>Analyte</b>	11Cl-PF3OUdS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	NT	NT	NT		
2	20	0.23	110	-0.17	-0.20
3	24	8	107	0.80	0.38
4	NS	NS	NS		
5	NT	NT	NT		
6	17.588	5.276	86	-0.75	-0.49
7*	7.44	0.83	79	-3.20	-3.69
8	20.7	0.085	NR	0.00	0.00
9	16.84	5.05	99	-0.93	-0.63
10	NT	NT	NT		
11	NT	NT	NT		
12	15.71	1.12	NR	-1.21	-1.36
13*	35	5.000	NR	3.45	2.34
14	NT	NT	NT		
15	NT	NT	NT		
16	NT	NT	NT		
17	19.315	0.983	98	-0.33	-0.38
19	26.0	9.85	97	1.28	0.51
20	NT	NT	NT		
21	19.9	8	NR	-0.19	-0.09
22	30.2	9.96	64	2.29	0.90

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	20.7	3.5
<b>Spike Value</b>	22.6	1.1
<b>Homogeneity Value</b>	24.5	9.8
<b>Robust Average</b>	21.0	4.8
<b>Median</b>	20.0	3.8
<b>Mean</b>	21.1	
<b>N</b>	12	
<b>Max</b>	35	
<b>Min</b>	7.44	
<b>Robust SD</b>	6.7	
<b>Robust CV</b>	32%	

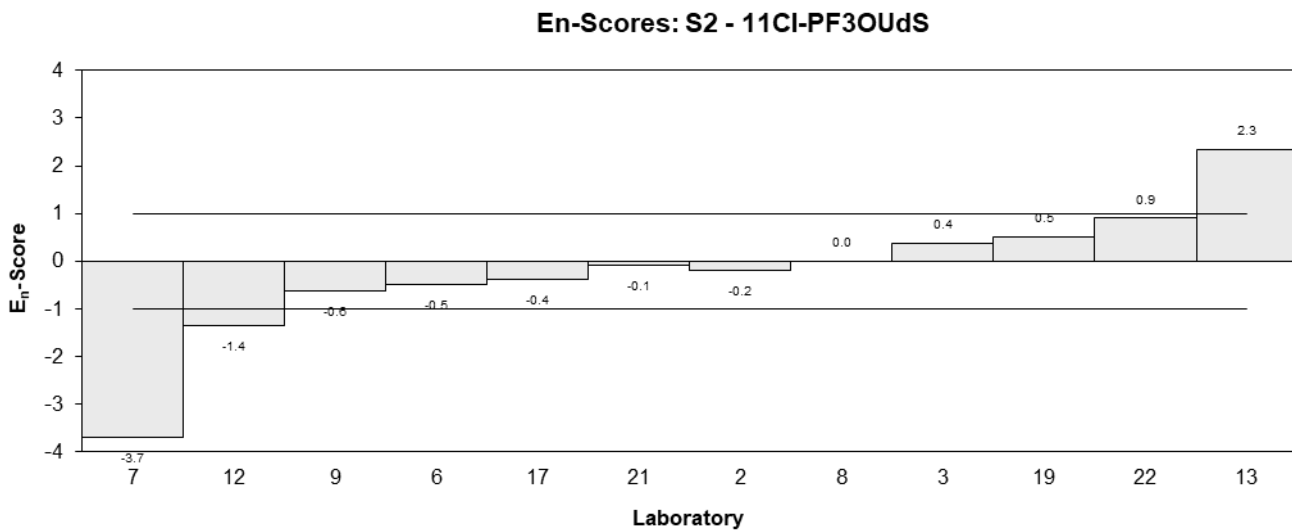
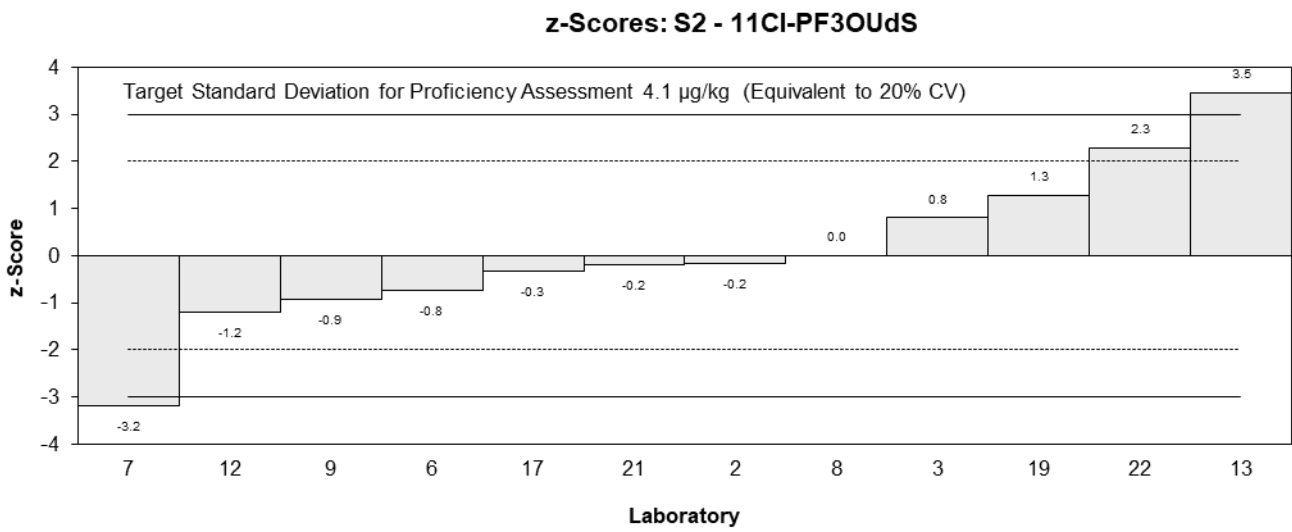
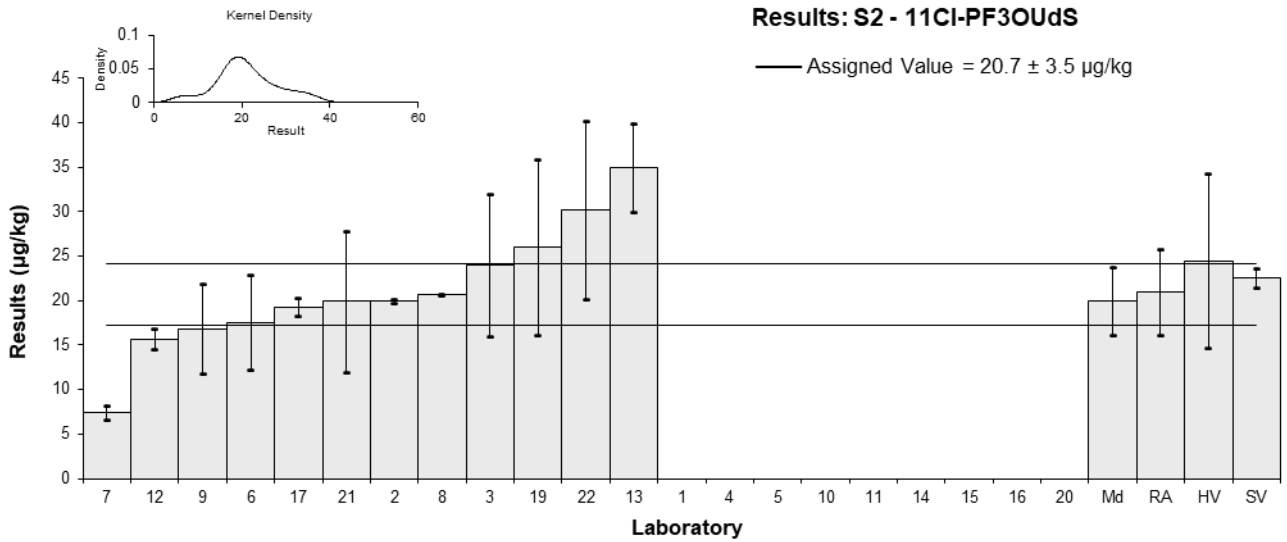


Figure 54

Table 60

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFBS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	1.1	0.55	106	-0.38	-0.16
2	1.4	0.0014	92	0.88	1.91
3	1	1	108	-0.80	-0.19
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	1.36	0.17	97	0.71	0.84
8	1.19	0.056	103.2	0.00	0.00
9	1.03	0.31	83	-0.67	-0.49
10	1.224	0.41	45.04	0.14	0.08
11	NS	NS	NS		
12	1.66	0.12	NR	1.97	2.89
13	1.2	0.50	NR	0.04	0.02
14	1.0868	0.3804	71	-0.43	-0.26
15	NS	NS	NS		
16	NS	NS	NS		
17	1.312	0.023	78	0.51	1.09
19	1.19	0.337	97	0.00	0.00
20	1.03	0.230	117.0	-0.67	-0.63
21	1.1	0.3	79	-0.38	-0.28
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	1.19	0.11
<b>Spike Value</b>	1.46	0.07
<b>Homogeneity Value</b>	1.06	0.32
<b>Robust Average</b>	1.19	0.11
<b>Median</b>	1.19	0.11
<b>Mean</b>	1.21	
<b>N</b>	14	
<b>Max</b>	1.66	
<b>Min</b>	1	
<b>Robust SD</b>	0.16	
<b>Robust CV</b>	14%	

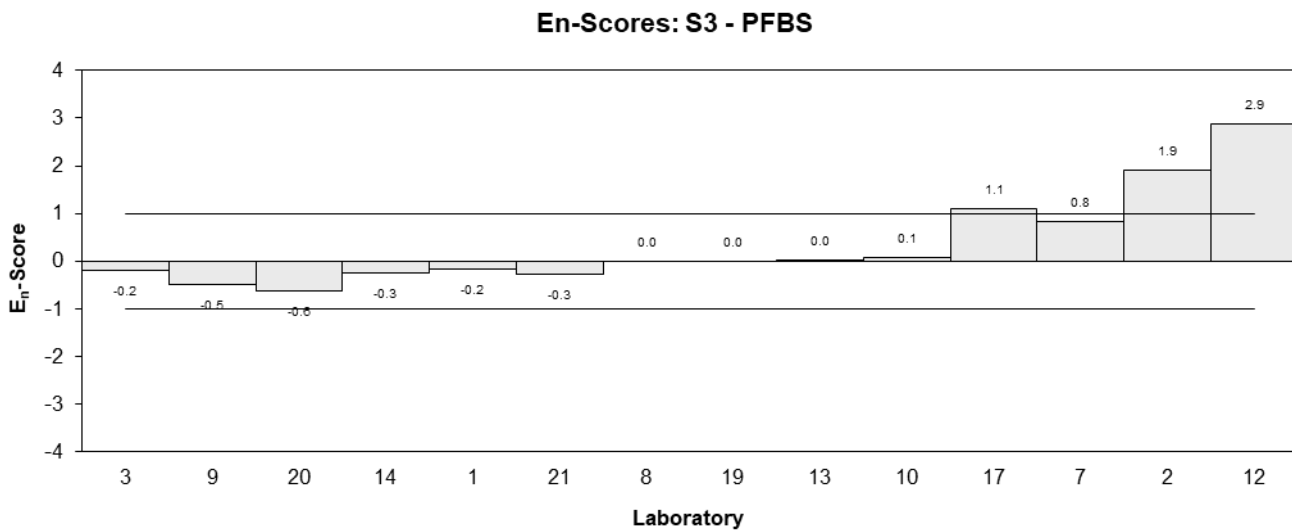
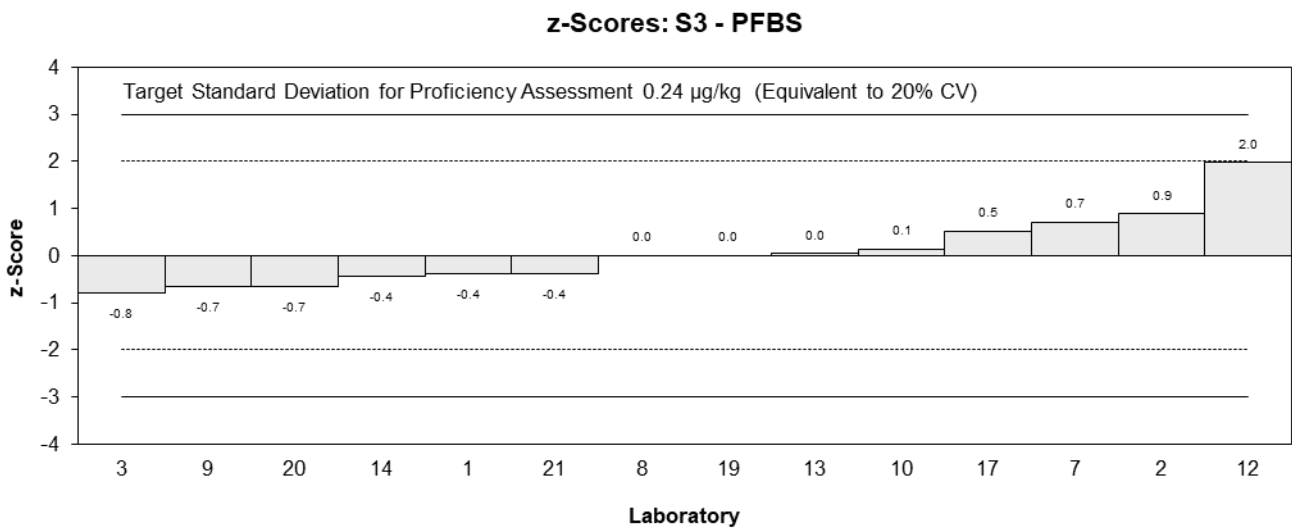
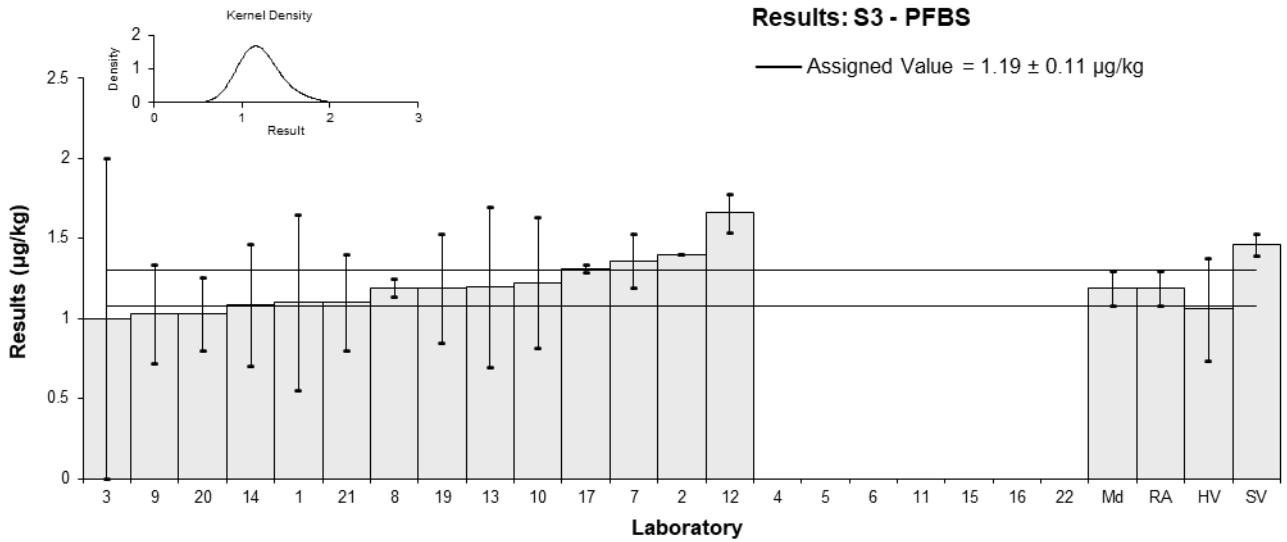


Figure 55

Table 61

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFPeS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	2.7	1.35	NR	-0.33	-0.14
2	3.6	0.082	107	1.23	1.92
3	4	2	113	1.92	0.55
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	2.82	0.40	100	-0.12	-0.13
8	2.85	0.062	NR	-0.07	-0.11
9	1.93	0.58	83	-1.66	-1.41
10	3.324	1.1	41.57	0.75	0.37
11	NS	NS	NS		
12	3.1	0.22	NR	0.36	0.50
13	2.2	0.50	NR	-1.19	-1.12
14	NT	NT	NT		
15	NS	NS	NS		
16	NS	NS	NS		
17	2.949	0.065	78	0.10	0.16
19	2.73	0.568	97	-0.28	-0.24
20	2.68	0.708	117.0	-0.36	-0.26
21	2.89	0.9	NR	0.00	0.00
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	2.89	0.36
<b>Spike Value</b>	3.22	0.16
<b>Homogeneity Value</b>	2.87	0.86
<b>Robust Average</b>	2.89	0.36
<b>Median</b>	2.85	0.17
<b>Mean</b>	2.91	
<b>N</b>	13	
<b>Max</b>	4	
<b>Min</b>	1.93	
<b>Robust SD</b>	0.52	
<b>Robust CV</b>	18%	



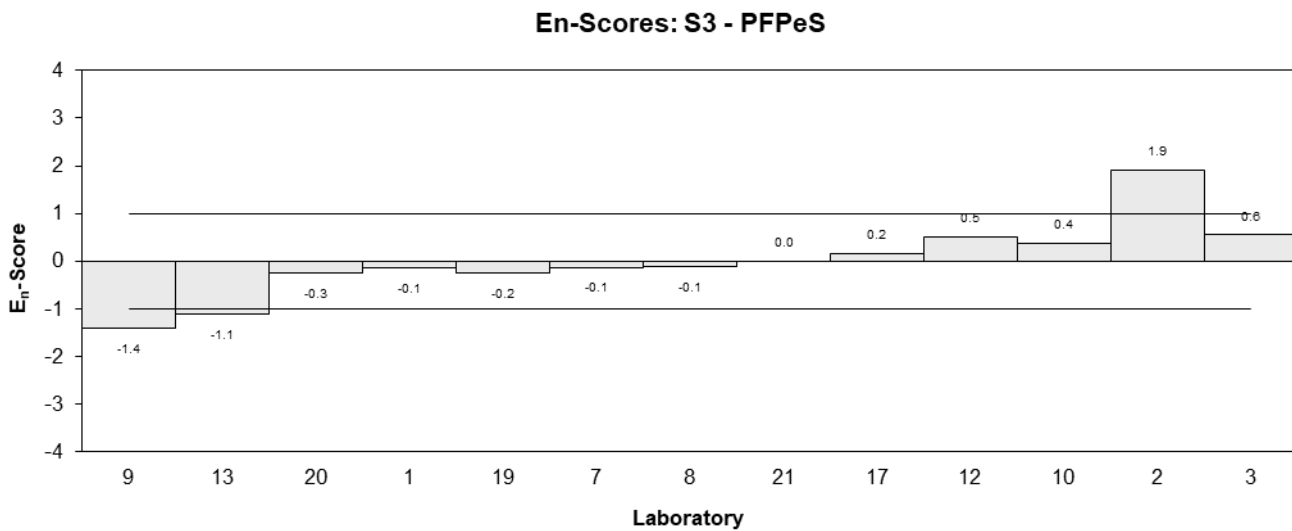
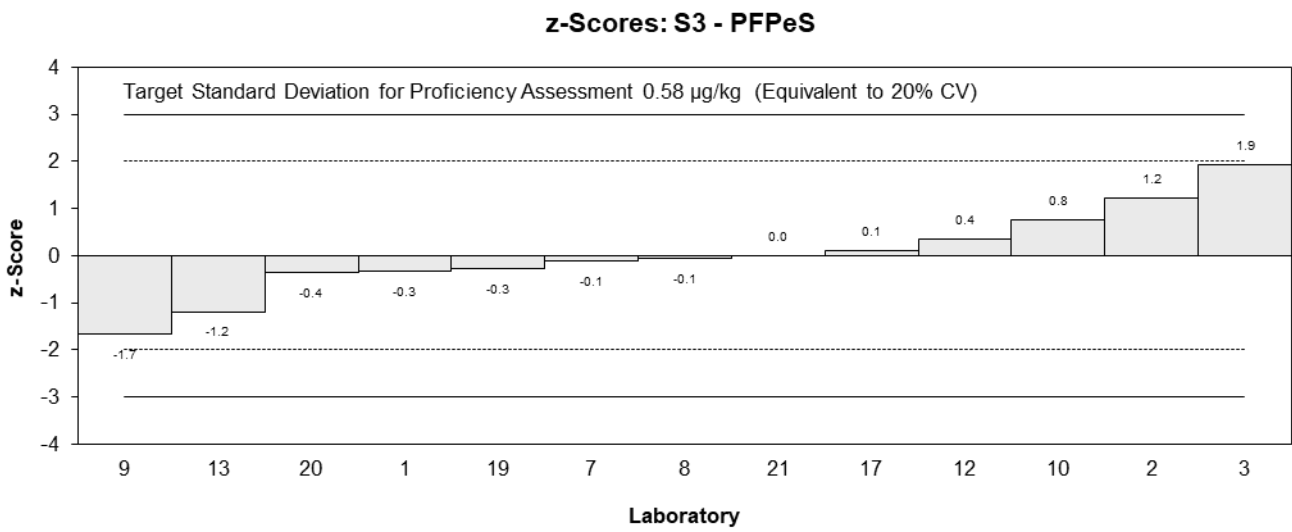
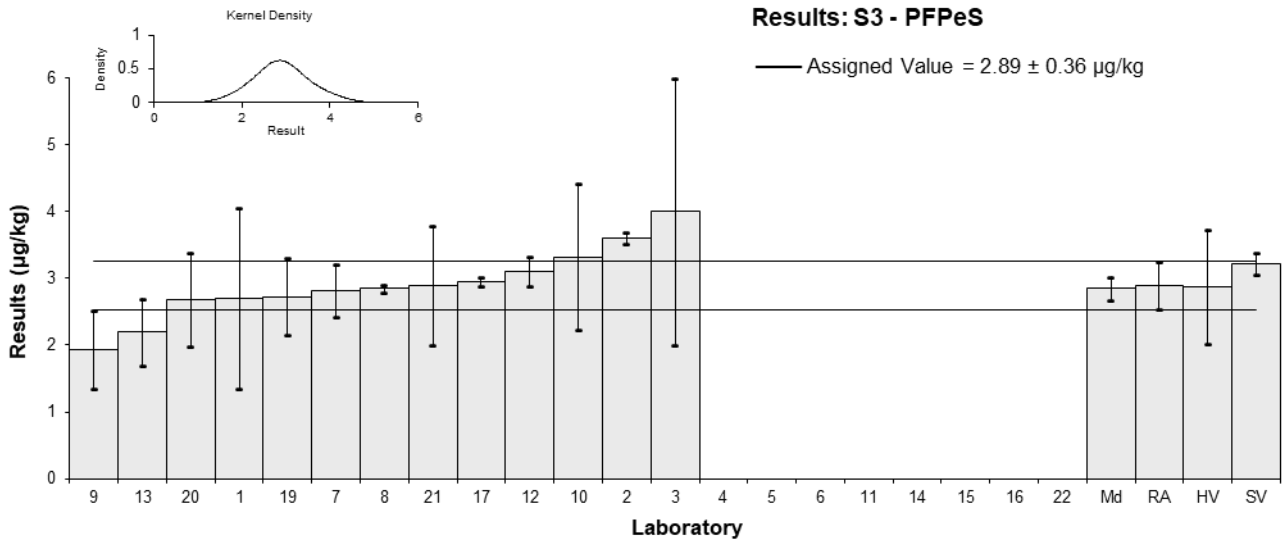


Figure 56

Table 62

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFHxS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	5.4	2.7	108	-0.45	-0.20
2	6.4	0.63	96	0.39	0.60
3	8	3	113	1.73	0.68
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	6.34	1.25	NR	0.34	0.30
8	5.76	0.068	101.3	-0.15	-0.40
9	5.25	1.58	76	-0.58	-0.42
10	6.34	2.2	41.57	0.34	0.18
11	NS	NS	NS		
12	6.51	0.46	NR	0.48	0.90
13	5.0	1.00	NR	-0.79	-0.86
14	5.5033	1.9262	74	-0.37	-0.22
15	NS	NS	NS		
16	NS	NS	NS		
17	5.96	0.217	78	0.02	0.04
19	5.72	0.99	90	-0.19	-0.20
20	NT	NT	NT		
21	6.18	2	76	0.20	0.12
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	5.94	0.44
<b>Spike Value</b>	6.50	0.32
<b>Homogeneity Value</b>	5.6	1.7
<b>Robust Average</b>	5.94	0.44
<b>Median</b>	5.96	0.45
<b>Mean</b>	6.03	
<b>N</b>	13	
<b>Max</b>	8	
<b>Min</b>	5	
<b>Robust SD</b>	0.63	
<b>Robust CV</b>	11%	

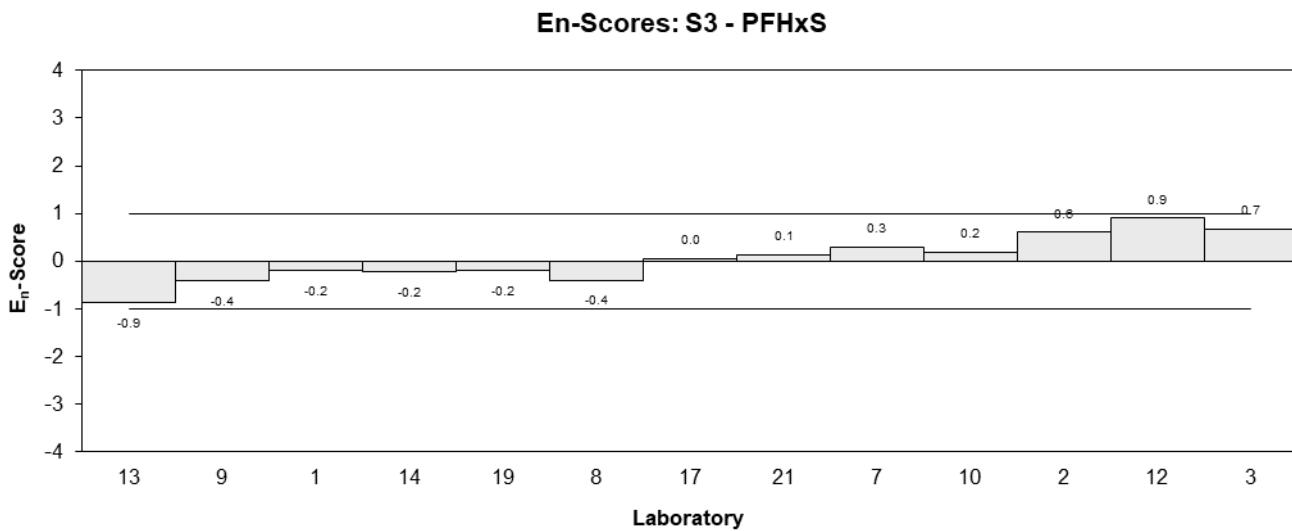
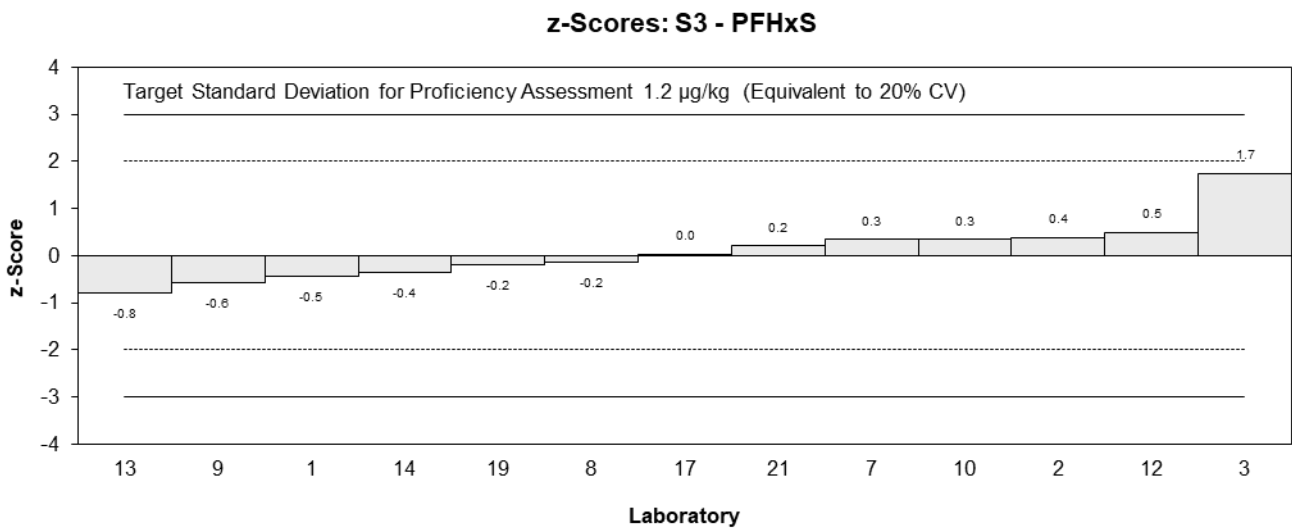
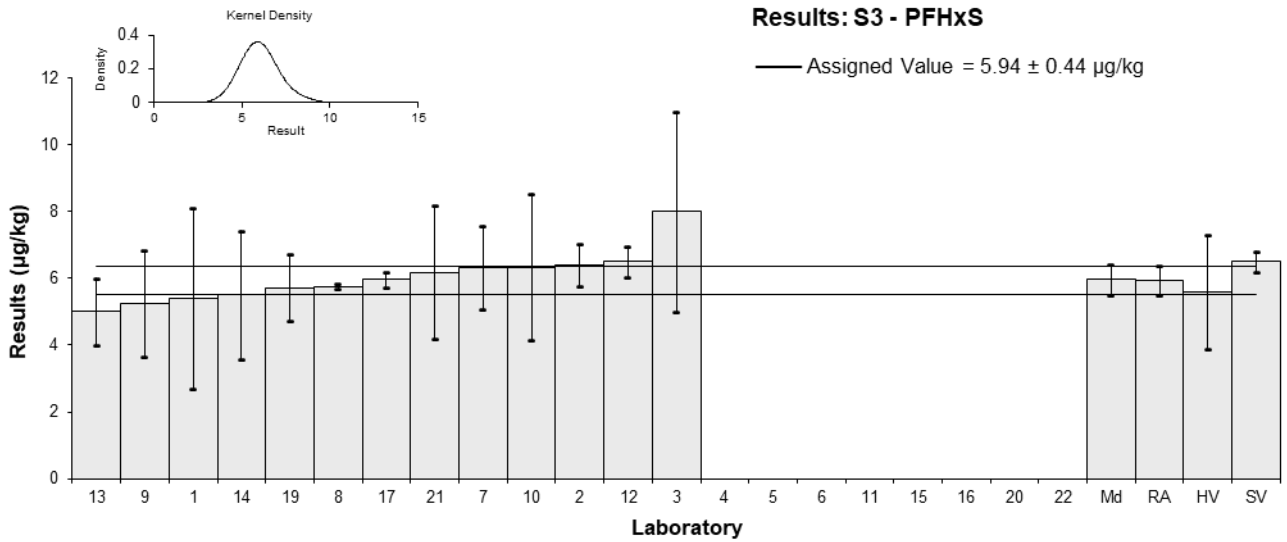


Figure 57

Table 63

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFHxS (linear)
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	5.4	2.7	NR	-0.47	-0.20
2	6.4	0.63	96	0.37	0.53
3	8	3	113	1.71	0.67
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	6.34	1.25	100	0.32	0.28
8	5.76	0.068	NR	-0.17	-0.37
9	5.25	1.58	76	-0.60	-0.43
10	NT	NT	NT		
11	NS	NS	NS		
12	6.51	0.46	NR	0.46	0.78
13	4.7	1.00	NR	-1.06	-1.11
14	5.5033	1.9262	74	-0.38	-0.23
15	NS	NS	NS		
16	NS	NS	NS		
17	NR	NR	NR		
19	5.72	0.0232	90	-0.20	-0.45
20	6.58	2.06	117.0	0.52	0.29
21	6.18	2	NR	0.18	0.11
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	5.96	0.53
<b>Spike Value</b>	6.50	0.32
<b>Homogeneity Value</b>	5.6	1.7
<b>Robust Average</b>	5.96	0.53
<b>Median</b>	5.97	0.54
<b>Mean</b>	6.03	
<b>N</b>	12	
<b>Max</b>	8	
<b>Min</b>	4.7	
<b>Robust SD</b>	0.74	
<b>Robust CV</b>	12%	

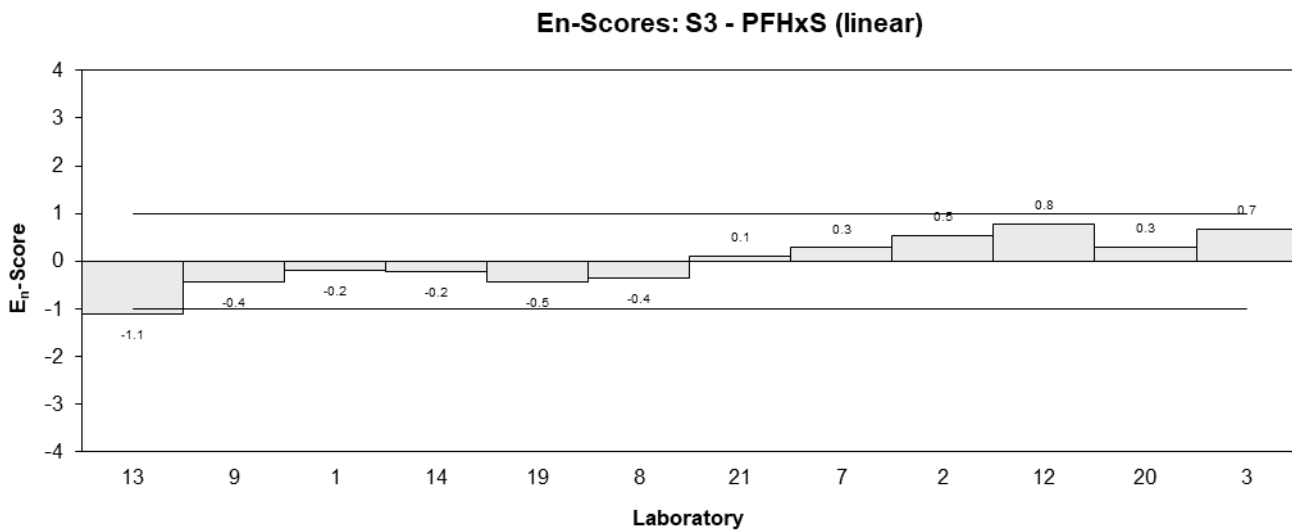
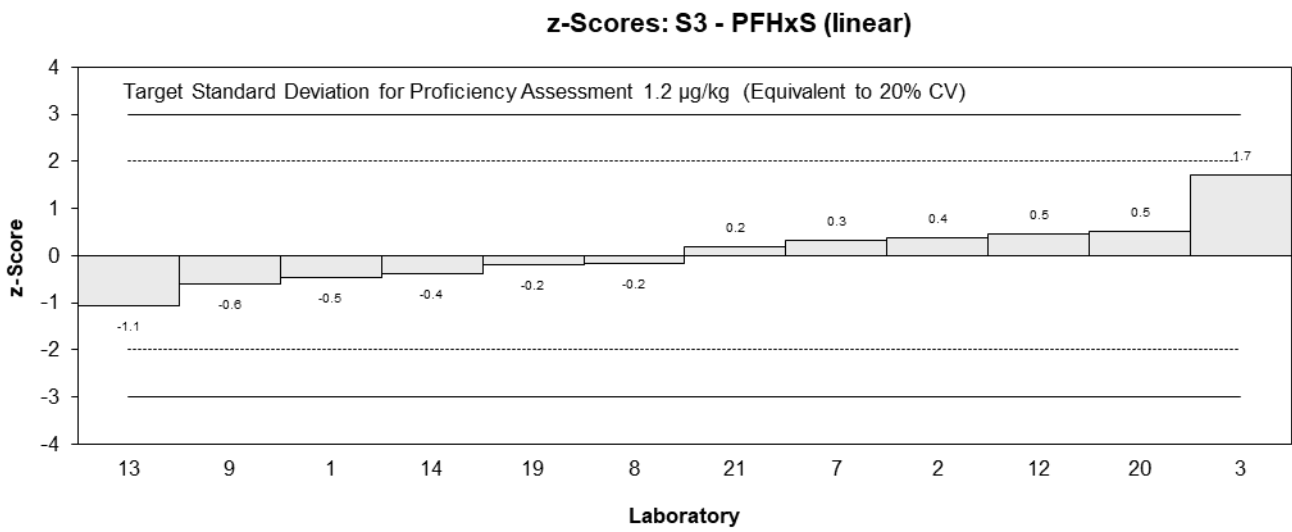
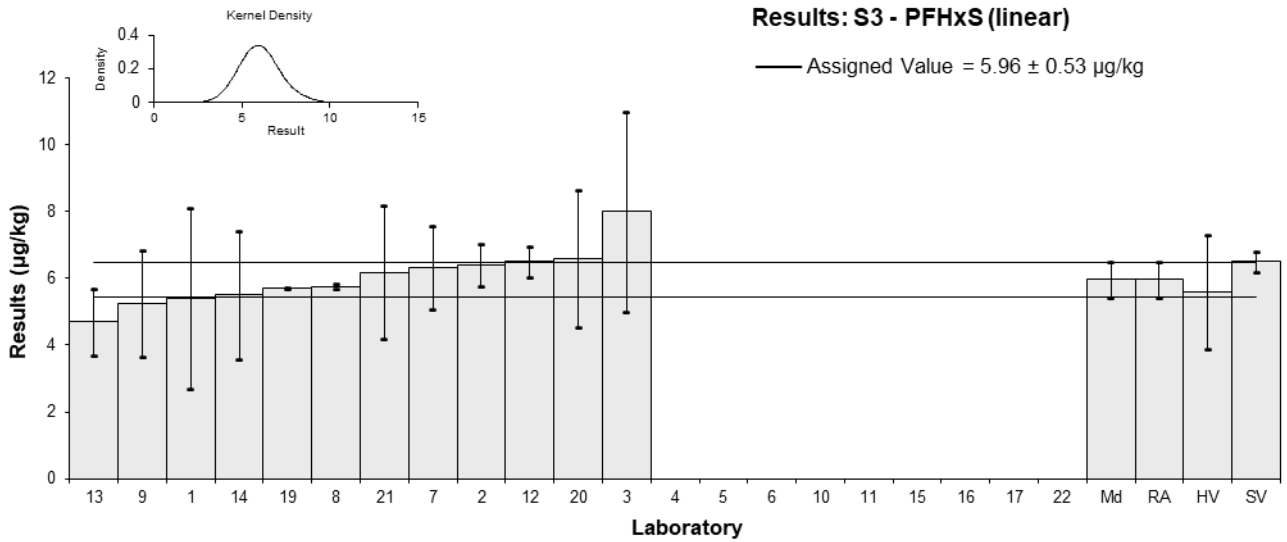


Figure 58

Table 64

**Sample Details**

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFHpS
<b>Unit</b>	µg/kg

**Participant Results**

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	1	0.5	NR	-1.12	-0.55
2	1.4	0.024	96	0.43	0.68
3	1	1	102	-1.12	-0.29
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	1.29	0.29	100	0.00	0.00
8	1.27	0.093	NR	-0.08	-0.11
9	1.66	0.5	76	1.43	0.70
10	1.446	0.49	57.04	0.60	0.30
11	NS	NS	NS		
12	1.44	0.11	NR	0.58	0.77
13*	2.3	0.50	NR	3.91	1.92
14	1.1228	0.3930	80	-0.65	-0.39
15	NS	NS	NS		
16	NS	NS	NS		
17	1.377	0.09	78	0.34	0.47
19	1.54	0.305	90	0.97	0.73
20	1.09	0.266	117.0	-0.78	-0.64
21	1.21	0.4	NR	-0.31	-0.19
22	NS	NS	NS		

\* Outlier, see Section 4.2

**Statistics**

<b>Assigned Value</b>	1.29	0.16
<b>Spike Value</b>	1.41	0.07
<b>Homogeneity Value</b>	1.18	0.35
<b>Robust Average</b>	1.33	0.17
<b>Median</b>	1.33	0.16
<b>Mean</b>	1.37	
<b>N</b>	14	
<b>Max</b>	2.3	
<b>Min</b>	1	
<b>Robust SD</b>	0.26	
<b>Robust CV</b>	19%	

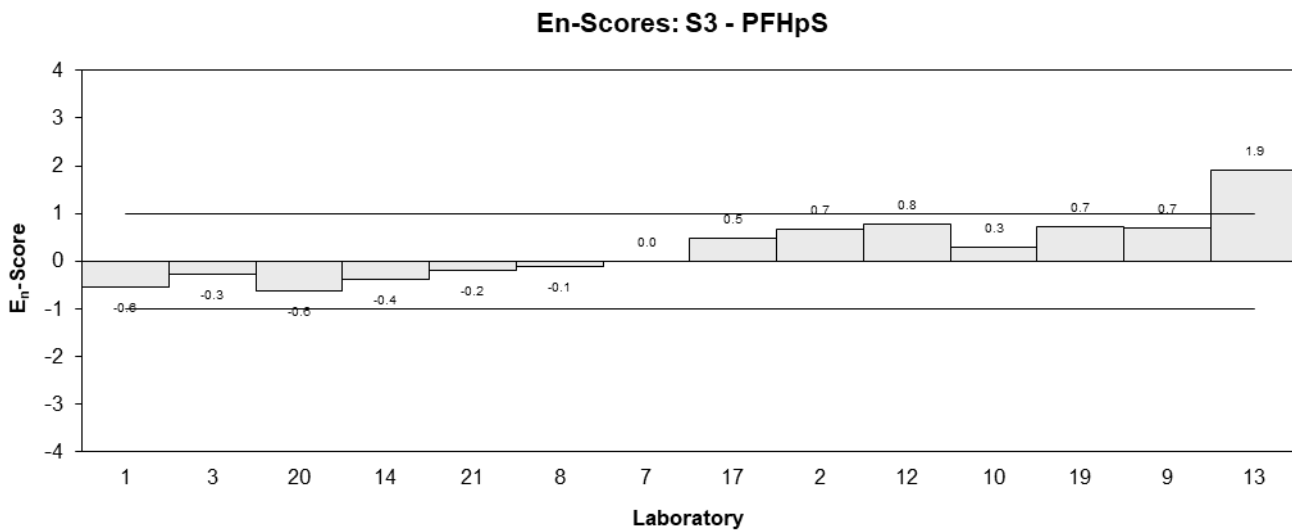
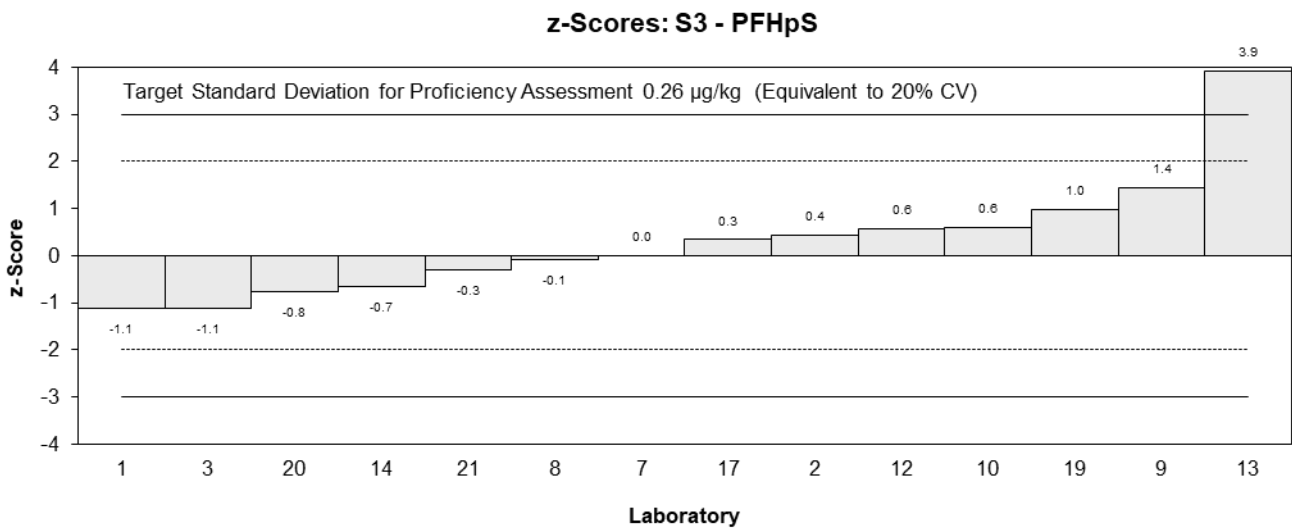
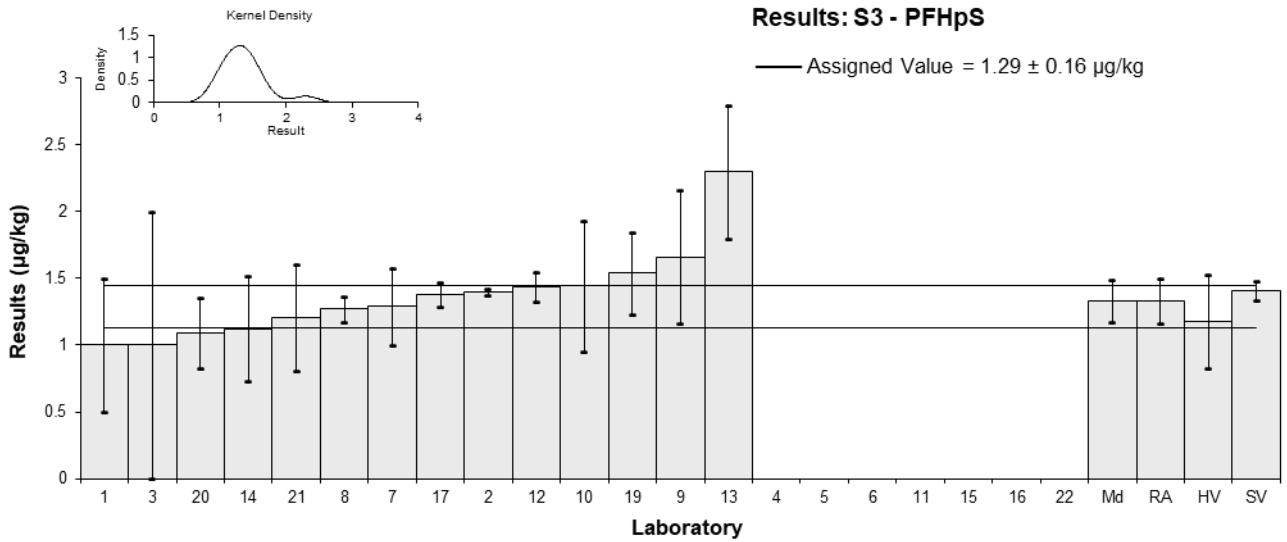


Figure 59

Table 65

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFOS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	1.4	0.7	99	-1.11	-0.55
2	2.1	0.071	97	0.83	1.35
3	2	1	102	0.56	0.20
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	1.83	0.19	97	0.08	0.11
8	1.50	0.18	109.0	-0.83	-1.08
9	1.91	0.57	103	0.31	0.18
10	2.112	0.77	57.04	0.87	0.39
11	NS	NS	NS		
12	1.77	0.13	NR	-0.08	-0.12
13*	3.0	0.50	NR	3.33	2.21
14	1.5304	0.5356	80	-0.75	-0.47
15	NS	NS	NS		
16	NS	NS	NS		
17	1.953	0.055	76	0.43	0.70
19	<2.38	NR	67		
20	1.95	1.10	89.4	0.42	0.13
21	1.49	0.4	73	-0.86	-0.69
22	NS	NS	NS		

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	1.80	0.21
<b>Spike Value</b>	1.87	0.09
<b>Homogeneity Value</b>	1.50	0.45
<b>Robust Average</b>	1.84	0.22
<b>Median</b>	1.91	0.20
<b>Mean</b>	1.89	
<b>N</b>	13	
<b>Max</b>	3	
<b>Min</b>	1.4	
<b>Robust SD</b>	0.32	
<b>Robust CV</b>	17%	



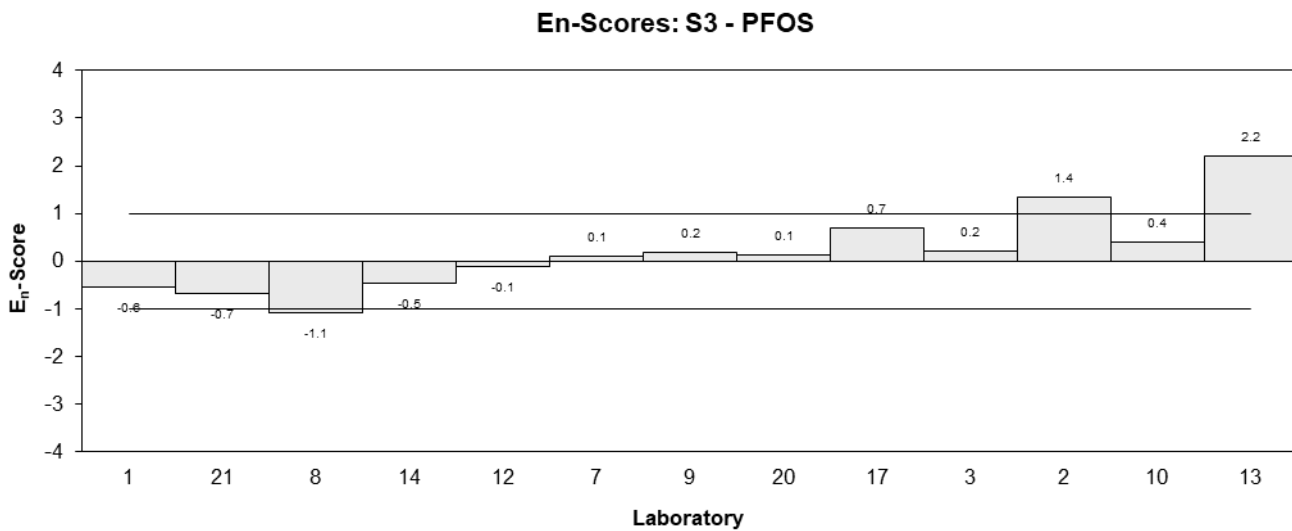
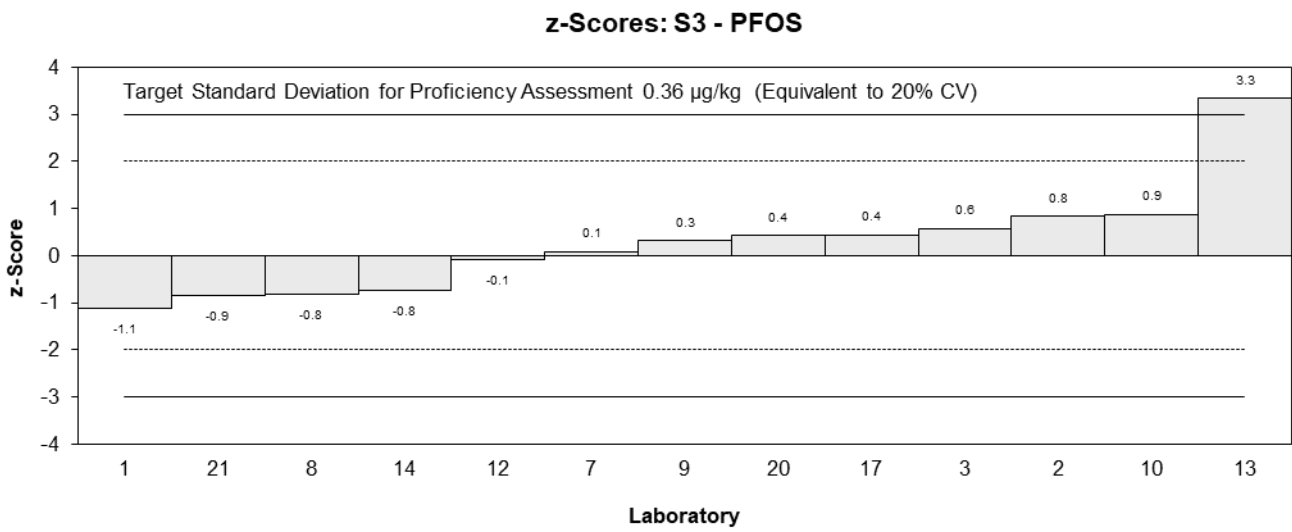
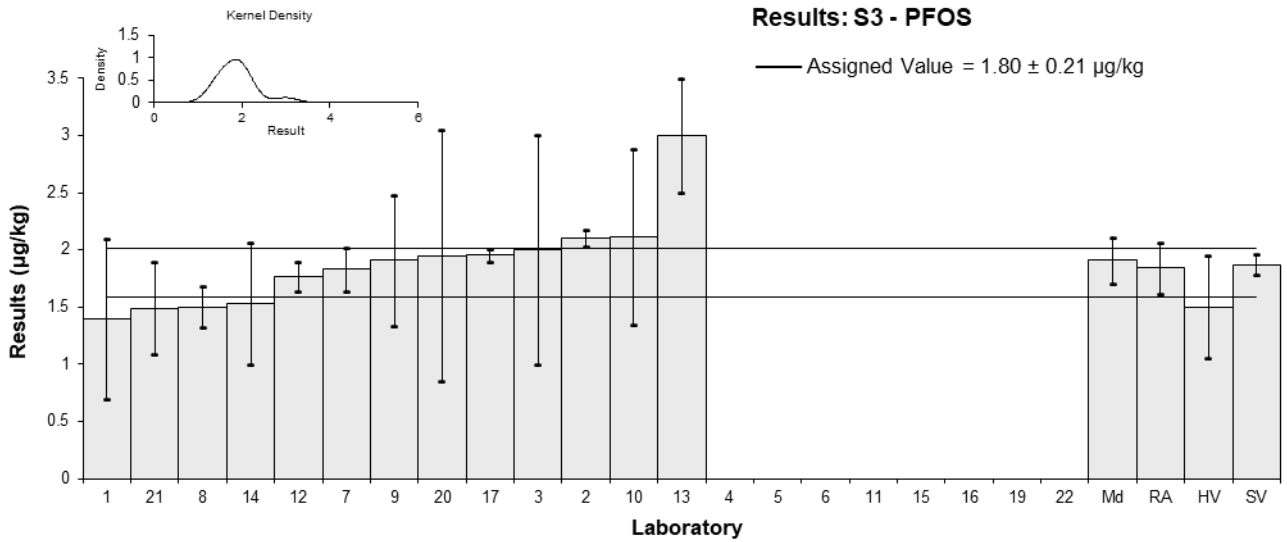


Figure 60

Table 66

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFOS (linear)
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	1.4	0.7	NR	-1.07	-0.52
2	2.1	0.071	97	0.90	1.38
3	2	1	102	0.62	0.21
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	1.79	0.46	97	0.03	0.02
8	1.50	0.078	NR	-0.79	-1.20
9	1.91	0.57	103	0.37	0.21
10	2.112	0.77	57.04	0.93	0.41
11	NS	NS	NS		
12	1.77	0.13	NR	-0.03	-0.04
13*	3.0	0.50	NR	3.43	2.23
14	1.5304	0.5356	80	-0.70	-0.43
15	NS	NS	NS		
16	NS	NS	NS		
17	NR	NR	NR		
19	<2.38	NR	67		
20	1.95	0.431	89.4	0.48	0.35
21	1.49	0.4	NR	-0.81	-0.64
22	NS	NS	NS		

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	1.78	0.22
<b>Spike Value</b>	1.87	0.09
<b>Homogeneity Value</b>	1.50	0.45
<b>Robust Average</b>	1.82	0.24
<b>Median</b>	1.85	0.27
<b>Mean</b>	1.88	
<b>N</b>	12	
<b>Max</b>	3	
<b>Min</b>	1.4	
<b>Robust SD</b>	0.33	
<b>Robust CV</b>	18%	

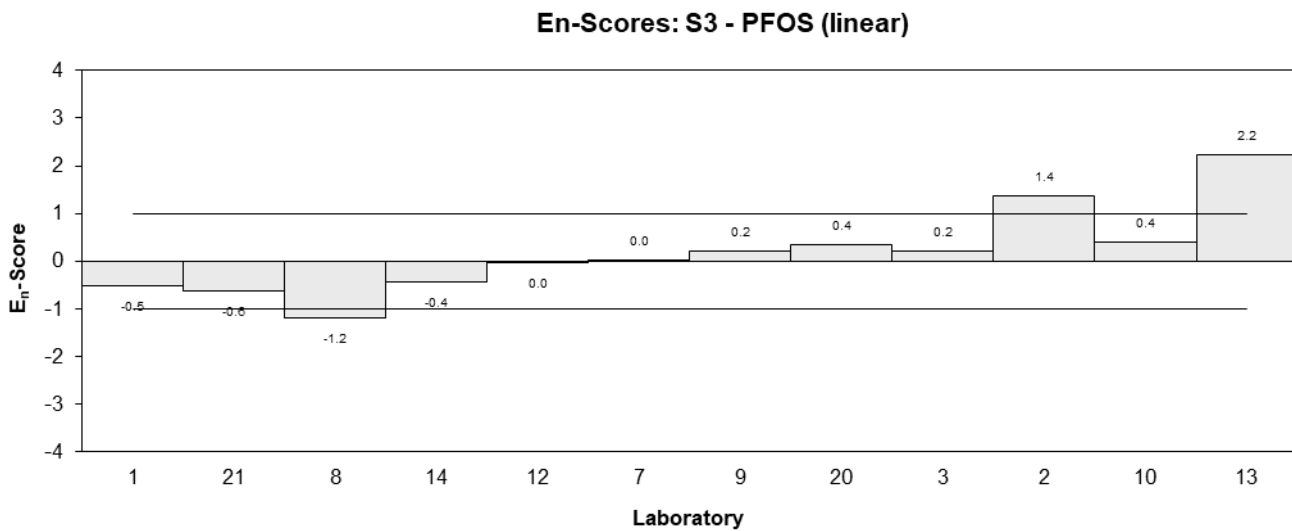
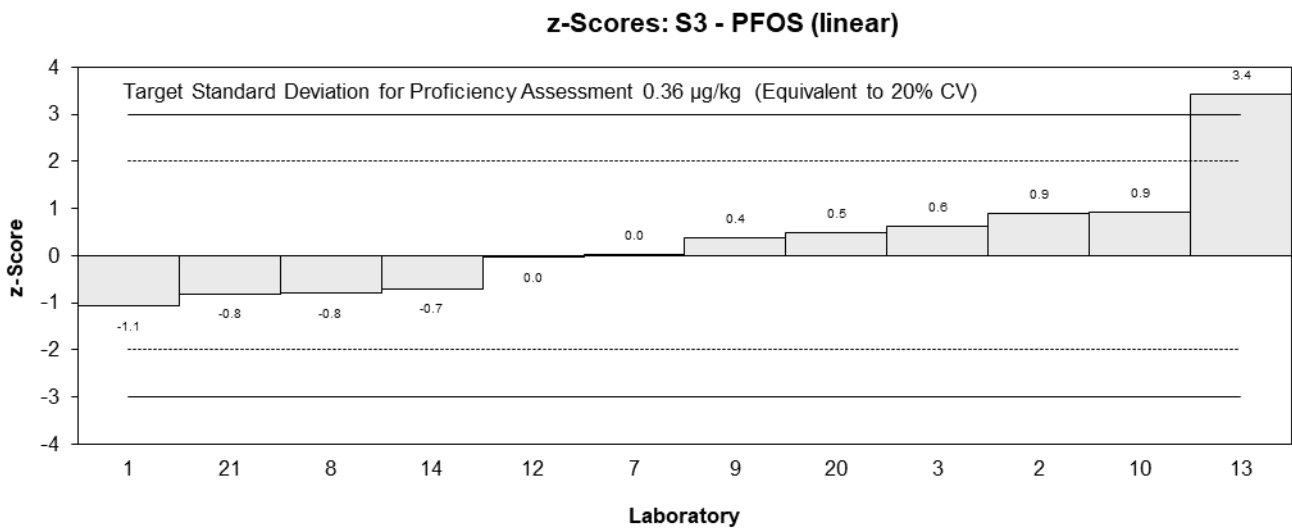
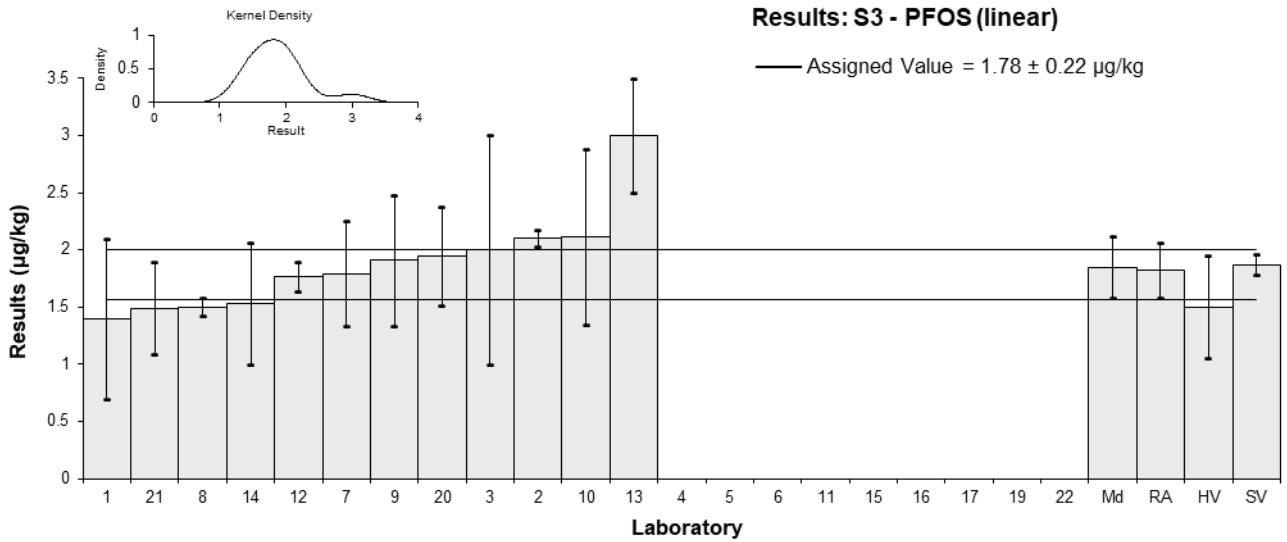


Figure 61

Table 67

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFNS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 2	1	NR		
2	1.9	0.23	130	0.65	0.72
3	2	1	102	0.95	0.31
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	1.71	0.09	97	0.09	0.14
8	1.54	0.062	NR	-0.42	-0.67
9	1.53	0.46	103	-0.45	-0.30
10	NT	NT	NT		
11	NS	NS	NS		
12	1.55	0.11	NR	-0.39	-0.57
13	1.4	0.30	NR	-0.83	-0.78
14	NT	NT	NT		
15	NS	NS	NS		
16	NS	NS	NS		
17	1.825	0.093	76	0.43	0.66
19	1.14	0.189	67	-1.61	-1.96
20	1.92	1.05	89.4	0.71	0.22
21	1.78	0.5	NR	0.30	0.19
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	1.68	0.20
<b>Spike Value</b>	1.88	0.09
<b>Homogeneity Value</b>	1.73	0.52
<b>Robust Average</b>	1.68	0.20
<b>Median</b>	1.71	0.20
<b>Mean</b>	1.66	
<b>N</b>	11	
<b>Max</b>	2	
<b>Min</b>	1.14	
<b>Robust SD</b>	0.26	
<b>Robust CV</b>	16%	

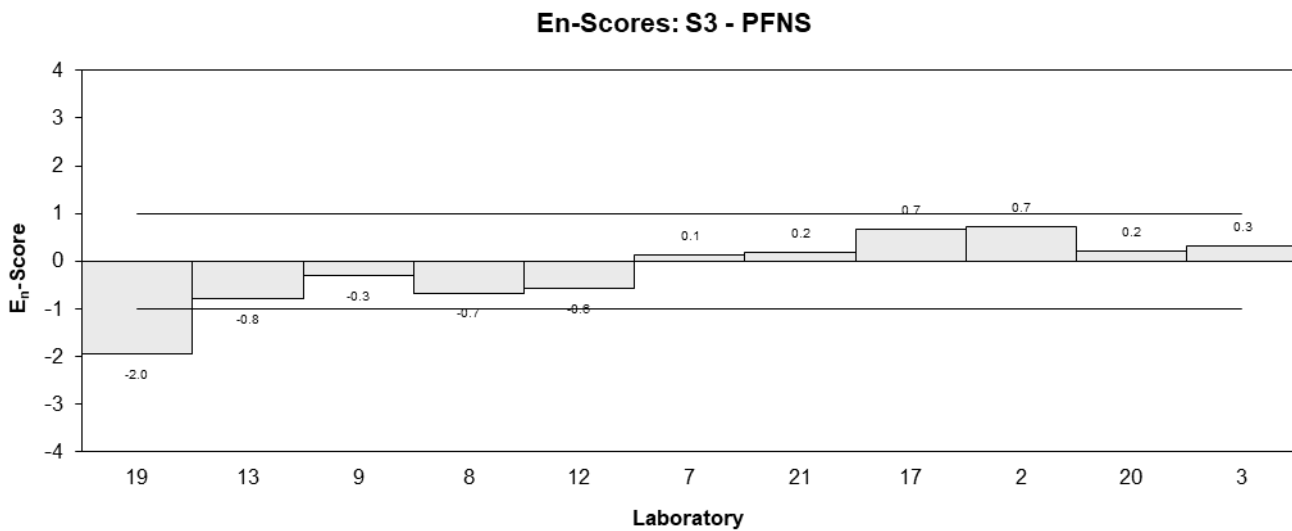
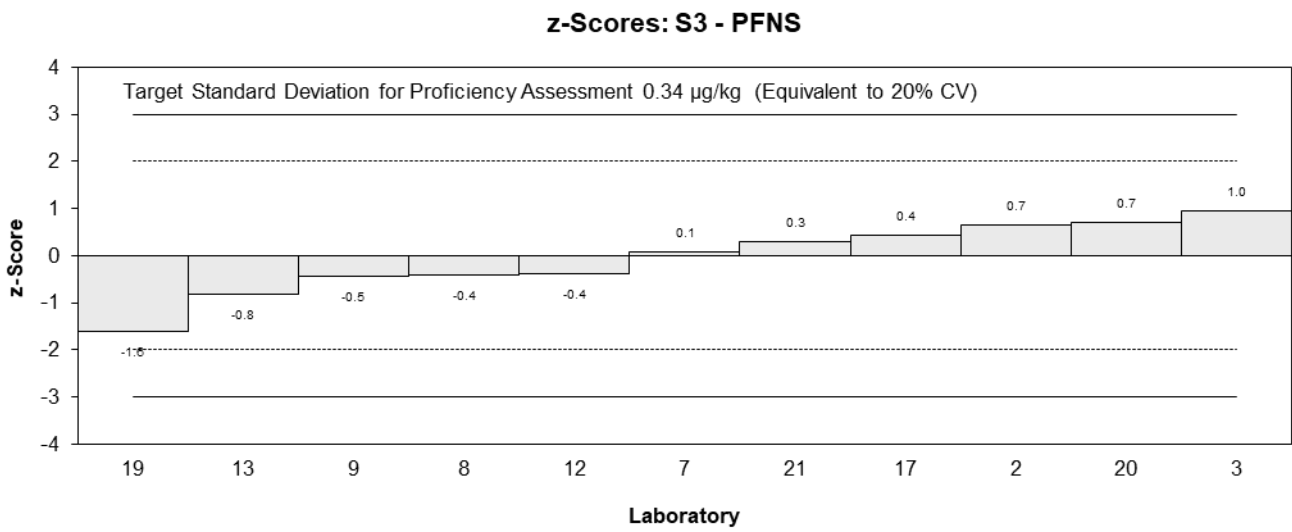
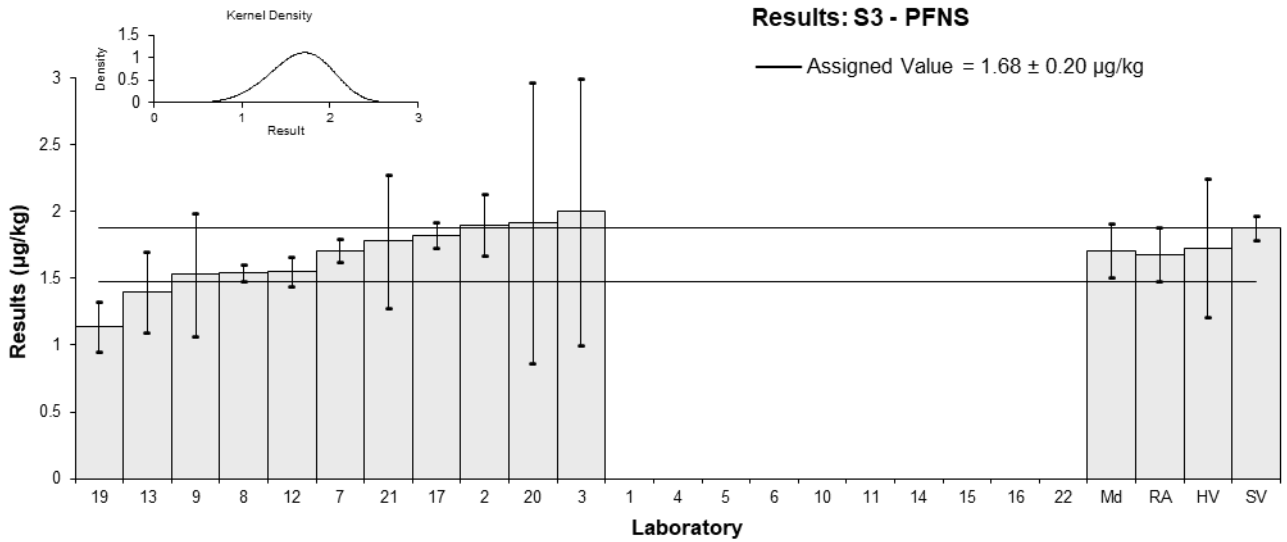


Figure 62

Table 68

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFDS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 2	1	NR		
2	1.6	0.045	145	0.06	0.08
3	2	1	102	1.33	0.41
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	1.33	0.42	97	-0.79	-0.52
8	1.51	0.066	NR	-0.22	-0.28
9	1.3	0.39	85	-0.89	-0.61
10	1.856	0.7	57.04	0.87	0.37
11	NS	NS	NS		
12	1.49	0.11	NR	-0.28	-0.34
13*	2.6	0.50	NR	3.23	1.84
14	1.3273	0.4646	80	-0.80	-0.48
15	NS	NS	NS		
16	NS	NS	NS		
17	1.668	0.109	76	0.28	0.33
19	0.827	0.17	67	-2.38	-2.56
20	2.01	1.18	89.4	1.36	0.36
21	1.8	0.7	NR	0.70	0.30
22	NS	NS	NS		

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	1.58	0.24
<b>Spike Value</b>	1.89	0.09
<b>Homogeneity Value</b>	1.63	0.65
<b>Robust Average</b>	1.63	0.26
<b>Median</b>	1.60	0.28
<b>Mean</b>	1.64	
<b>N</b>	13	
<b>Max</b>	2.6	
<b>Min</b>	0.827	
<b>Robust SD</b>	0.38	
<b>Robust CV</b>	23%	

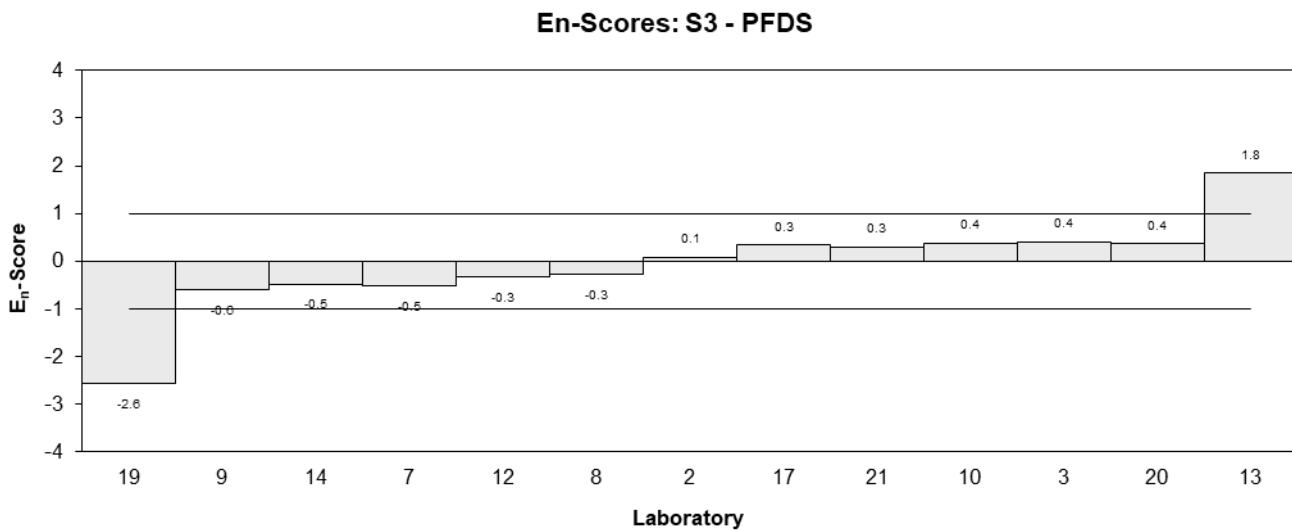
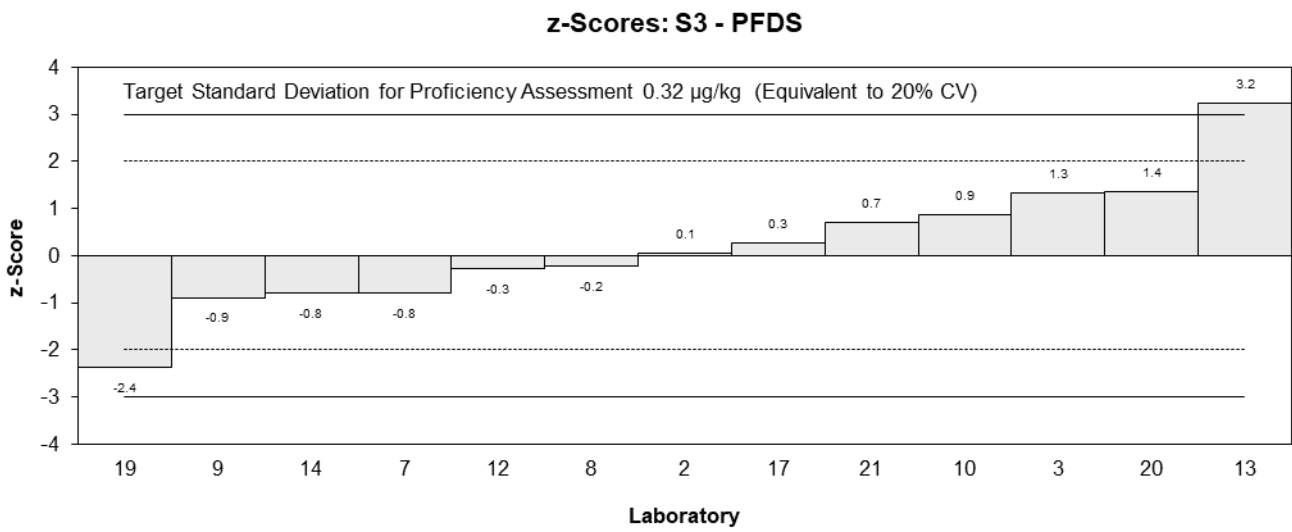
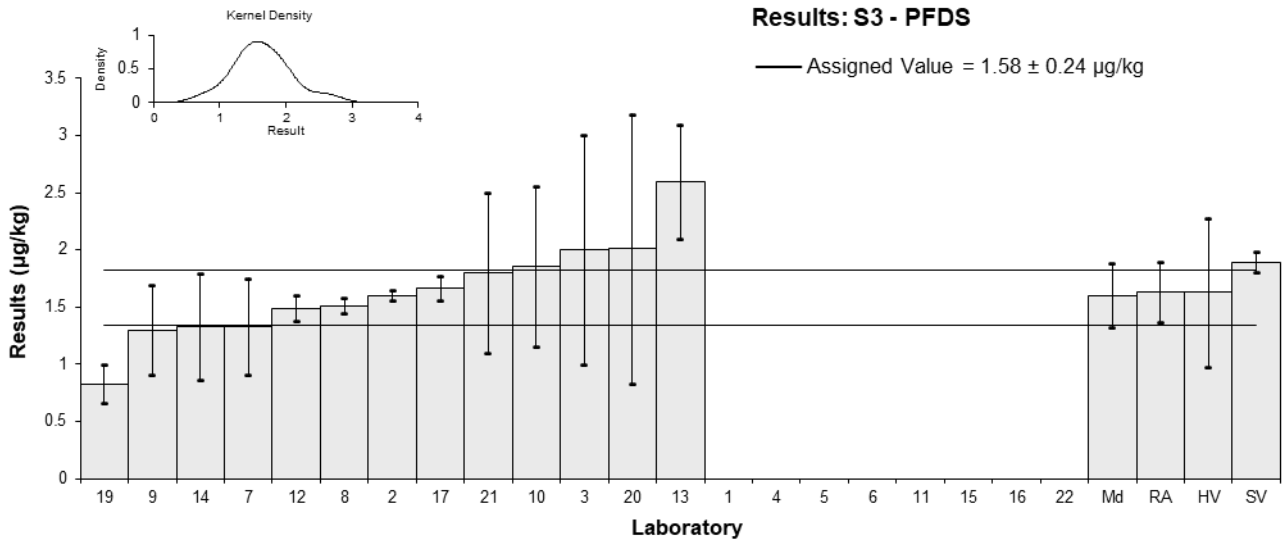


Figure 63

Table 69

**Sample Details**

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFBA
<b>Unit</b>	µg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>Uncertainty</b>	<b>Rec</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	5.4	2.7	101	-0.83	-0.38
2	6.8	0.33	105	0.26	0.39
3	7	3	118	0.41	0.17
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	7.01	1.06	73	0.42	0.41
8	6.21	0.068	70.9	-0.20	-0.33
9	5.12	1.54	86	-1.04	-0.78
10	7.434	3.2	18.99	0.74	0.29
11	NS	NS	NS		
12	8.57	0.61	NR	1.62	2.12
13	NT	NT	NT		
14	NT	NT	20		
15	NS	NS	NS		
16	NS	NS	NS		
17*	2.839	0.311	102	-2.81	-4.32
19	5.96	1.07	77	-0.39	-0.39
20	6.70	1.53	69.1	0.18	0.13
21	5.51	2	73	-0.74	-0.45
22	NS	NS	NS		

\* Outlier, see Section 4.2

**Statistics**

<b>Assigned Value</b>	6.47	0.78
<b>Spike Value</b>	6.77	0.34
<b>Homogeneity Value</b>	5.6	2.2
<b>Robust Average</b>	6.31	0.85
<b>Median</b>	6.46	0.80
<b>Mean</b>	6.21	
<b>N</b>	12	
<b>Max</b>	8.57	
<b>Min</b>	2.839	
<b>Robust SD</b>	1.2	
<b>Robust CV</b>	19%	



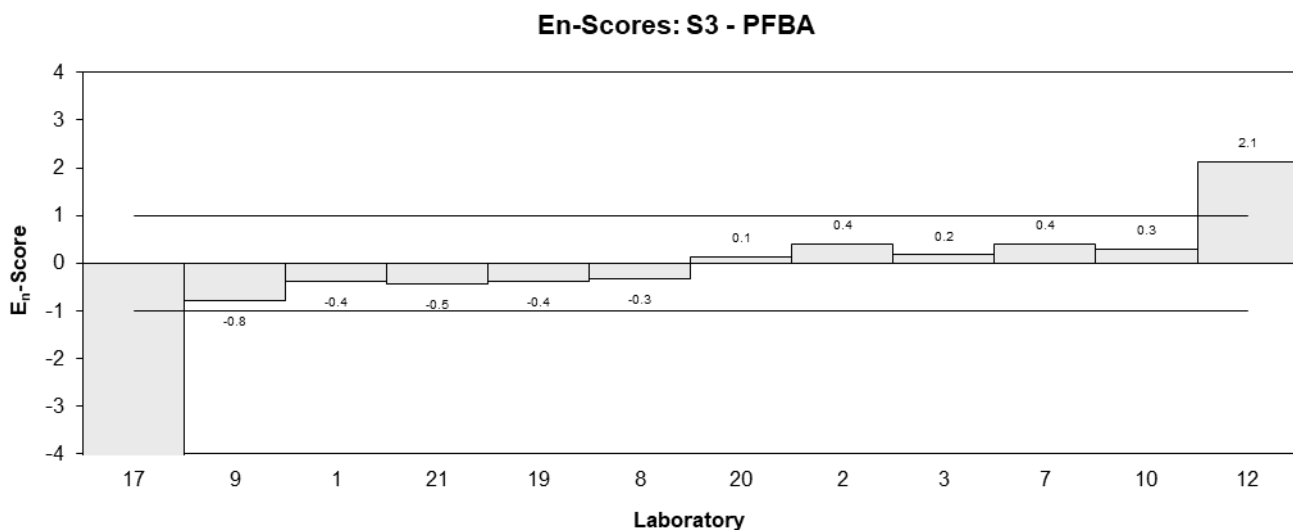
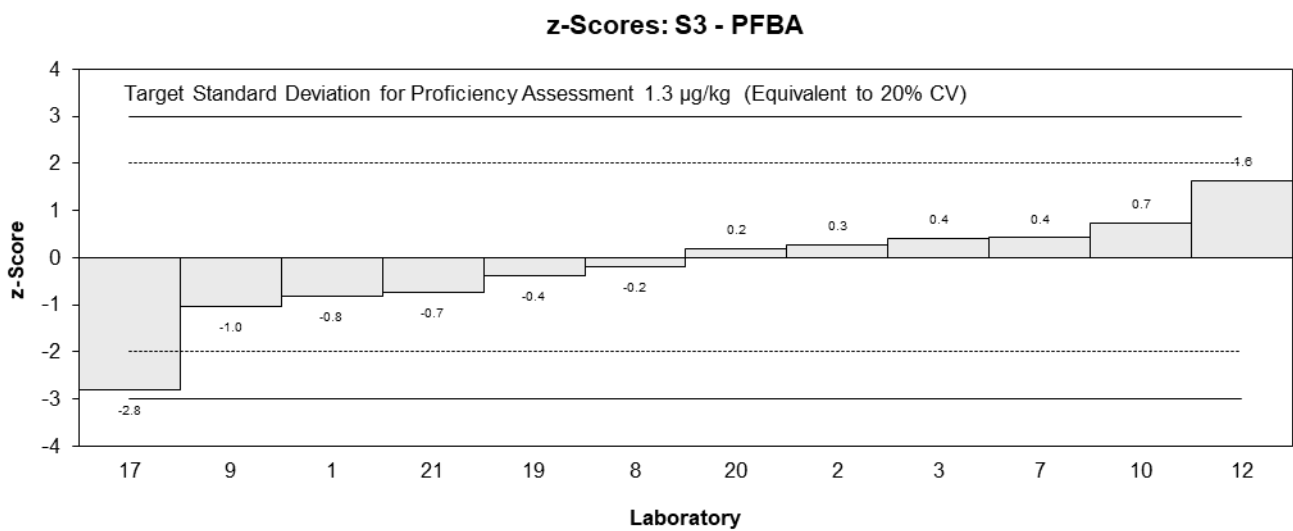
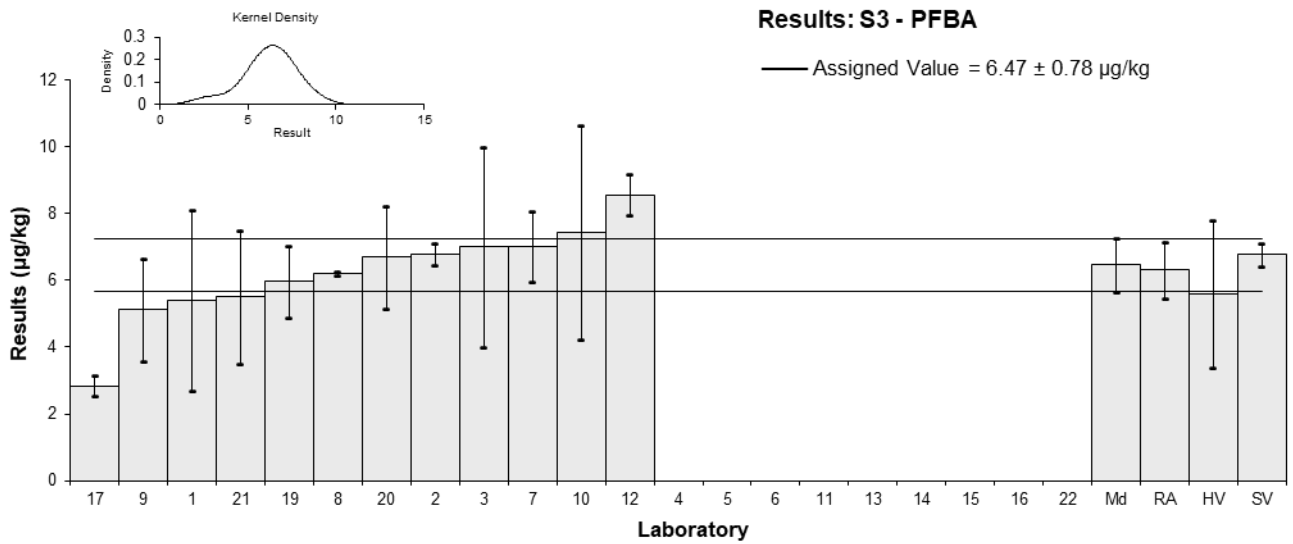


Figure 64

Table 70

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFPeA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	2	1	96	-0.59	-0.26
2	2.4	0.27	105	0.29	0.37
3	3	1	123	1.61	0.71
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	2.61	0.31	86	0.75	0.89
8	2.15	0.075	79.7	-0.26	-0.52
9	1.85	0.55	84	-0.93	-0.71
10	2.124	0.76	50.49	-0.32	-0.18
11	NS	NS	NS		
12	2.32	0.17	NR	0.11	0.18
13*	6.3	1.00	NR	8.88	3.94
14	2.1592	0.7557	37	-0.24	-0.14
15	NS	NS	NS		
16	NS	NS	NS		
17	2.283	0.029	60	0.03	0.06
19	2.32	0.578	91	0.11	0.08
20	2.65	2.53	83.6	0.84	0.15
21	1.95	0.6	74	-0.70	-0.50
22	NS	NS	NS		

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	2.27	0.22
<b>Spike Value</b>	2.44	0.12
<b>Homogeneity Value</b>	2.12	0.64
<b>Robust Average</b>	2.33	0.25
<b>Median</b>	2.30	0.24
<b>Mean</b>	2.58	
<b>N</b>	14	
<b>Max</b>	6.3	
<b>Min</b>	1.85	
<b>Robust SD</b>	0.37	
<b>Robust CV</b>	16%	

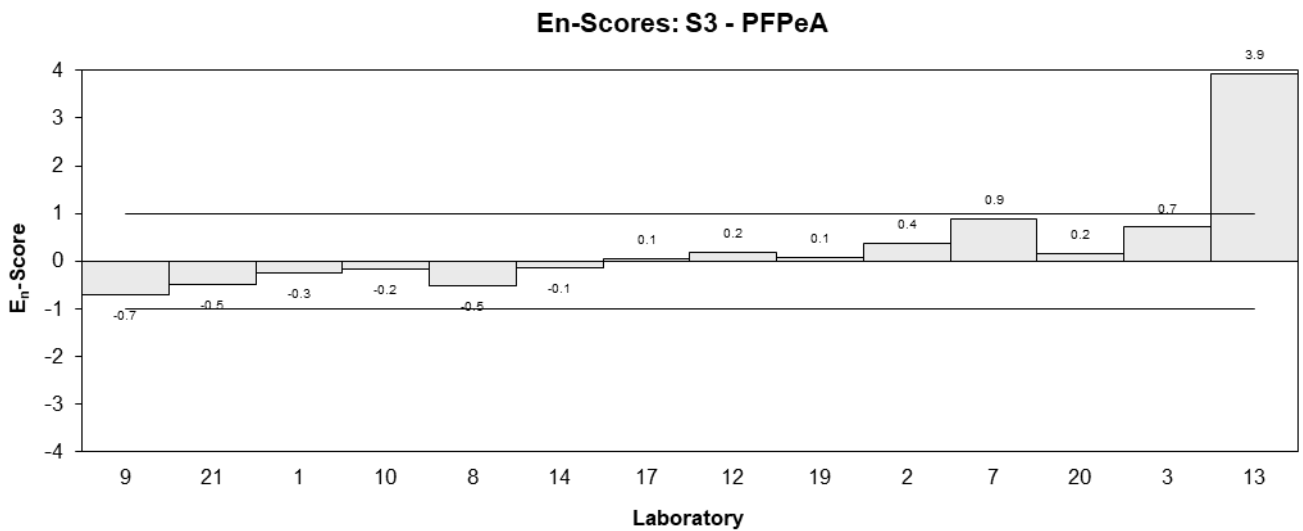
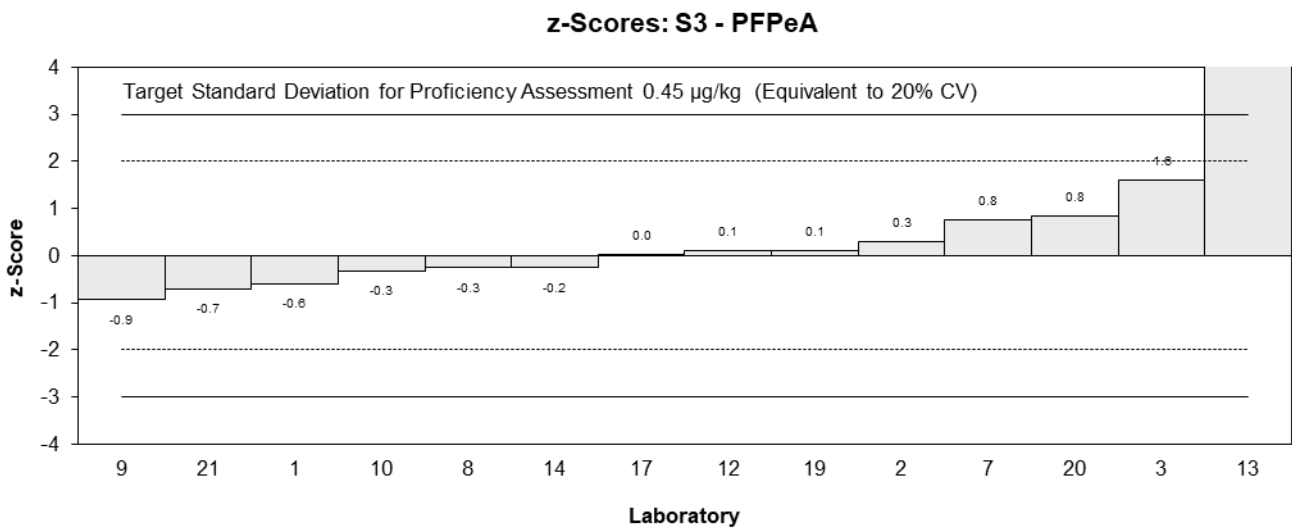
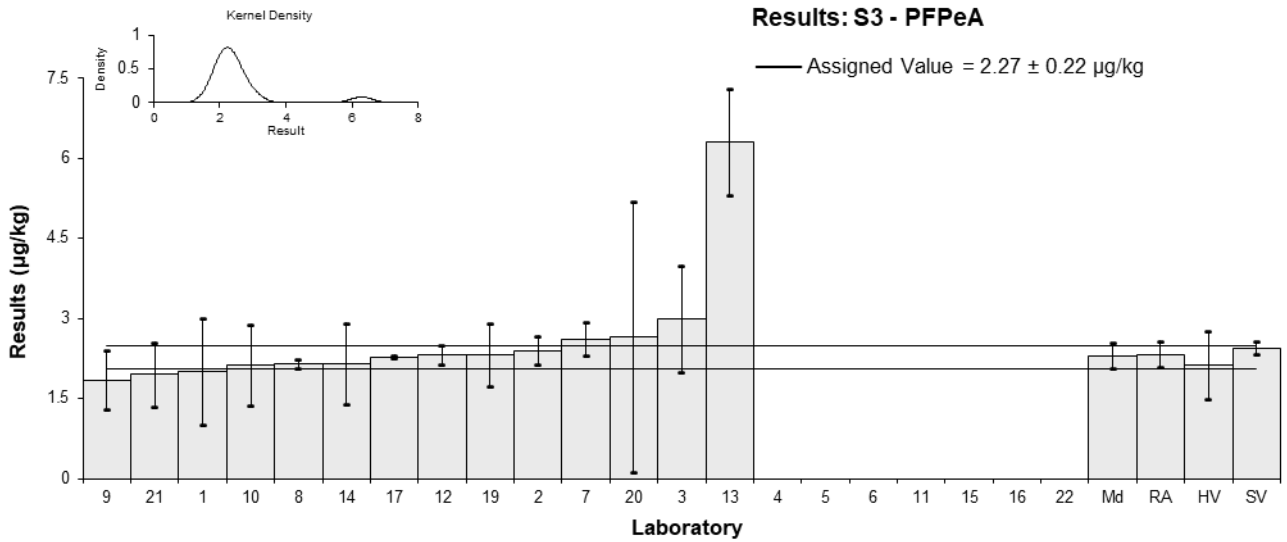


Figure 65

Table 71

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFHxA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	6	3	102	-0.63	-0.28
2	7.2	0.035	107	0.25	0.64
3	8	3	105	0.83	0.37
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	7.69	0.45	86	0.60	1.19
8	6.28	0.074	82.1	-0.42	-1.08
9	6.39	1.91	87	-0.34	-0.24
10	6.25	2.43	50.12	-0.44	-0.25
11	NS	NS	NS		
12	6.84	0.48	NR	-0.01	-0.03
13	7.8	1.00	NR	0.69	0.83
14	6.5115	2.2790	36	-0.25	-0.15
15	NS	NS	NS		
16	NS	NS	NS		
17	6.831	0.162	63	-0.02	-0.05
19	6.58	1.16	93	-0.20	-0.22
20	7.69	1.80	78.0	0.60	0.44
21	5.94	2	75	-0.67	-0.44
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	6.86	0.53
<b>Spike Value</b>	6.89	0.34
<b>Homogeneity Value</b>	6.6	2.0
<b>Robust Average</b>	6.86	0.53
<b>Median</b>	6.71	0.47
<b>Mean</b>	6.86	
<b>N</b>	14	
<b>Max</b>	8	
<b>Min</b>	5.94	
<b>Robust SD</b>	0.80	
<b>Robust CV</b>	12%	

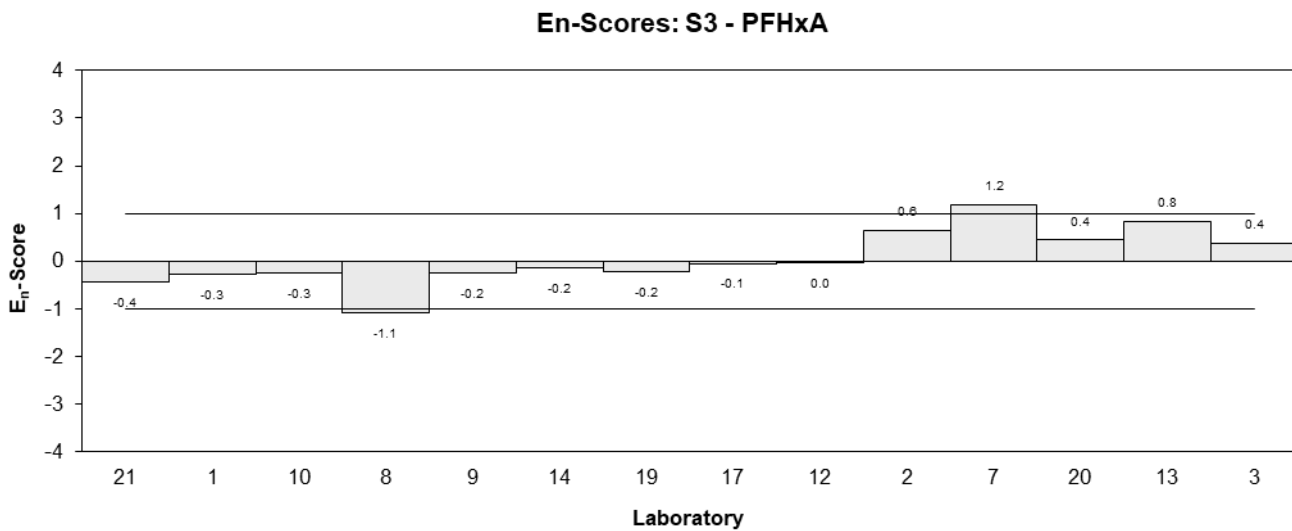
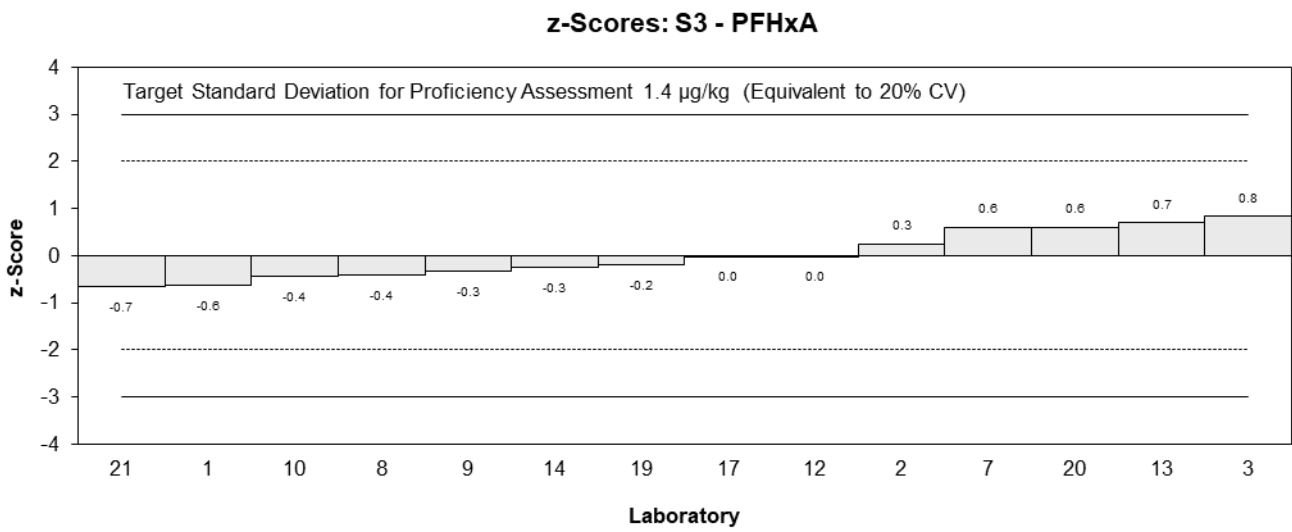
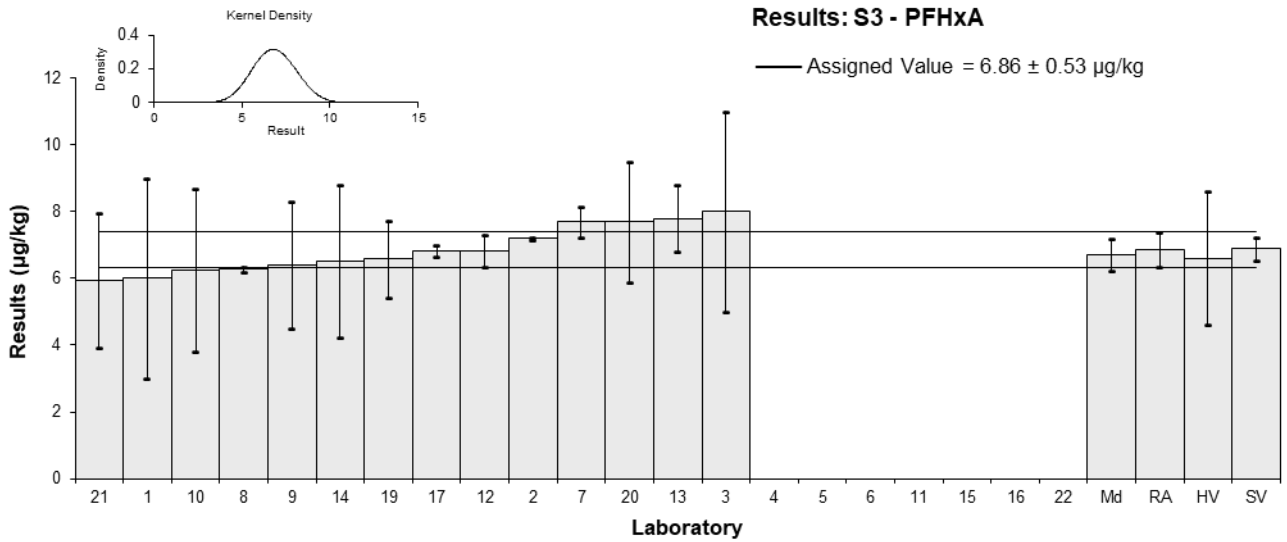


Figure 66

Table 72

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFHpA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	6.6	3.3	97	-0.50	-0.22
2	7.8	0.58	110	0.31	0.51
3	8	3	96	0.45	0.21
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	9.22	0.97	75	1.28	1.57
8	6.64	0.089	NR	-0.48	-0.99
9	6.5	1.95	84	-0.57	-0.41
10	7.126	2.65	54.66	-0.15	-0.08
11	NS	NS	NS		
12	7.61	0.54	NR	0.18	0.31
13	5.0	0.80	NR	-1.59	-2.20
14	6.5431	2.2901	46	-0.54	-0.33
15	NS	NS	NS		
16	NS	NS	NS		
17	8.619	0.412	63	0.87	1.57
19	7.29	1.2	89	-0.03	-0.04
20	8.44	2.34	56.1	0.75	0.45
21	6.91	2	74	-0.29	-0.20
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	7.34	0.70
<b>Spike Value</b>	7.84	0.39
<b>Homogeneity Value</b>	7.1	2.1
<b>Robust Average</b>	7.34	0.70
<b>Median</b>	7.21	0.63
<b>Mean</b>	7.31	
<b>N</b>	14	
<b>Max</b>	9.22	
<b>Min</b>	5	
<b>Robust SD</b>	1.0	
<b>Robust CV</b>	14%	

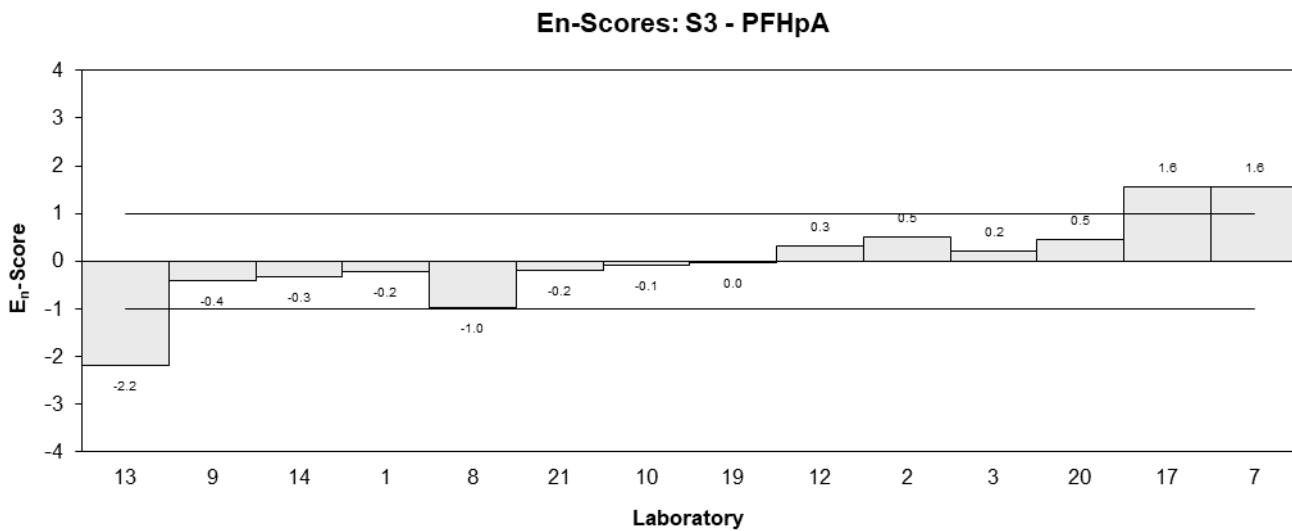
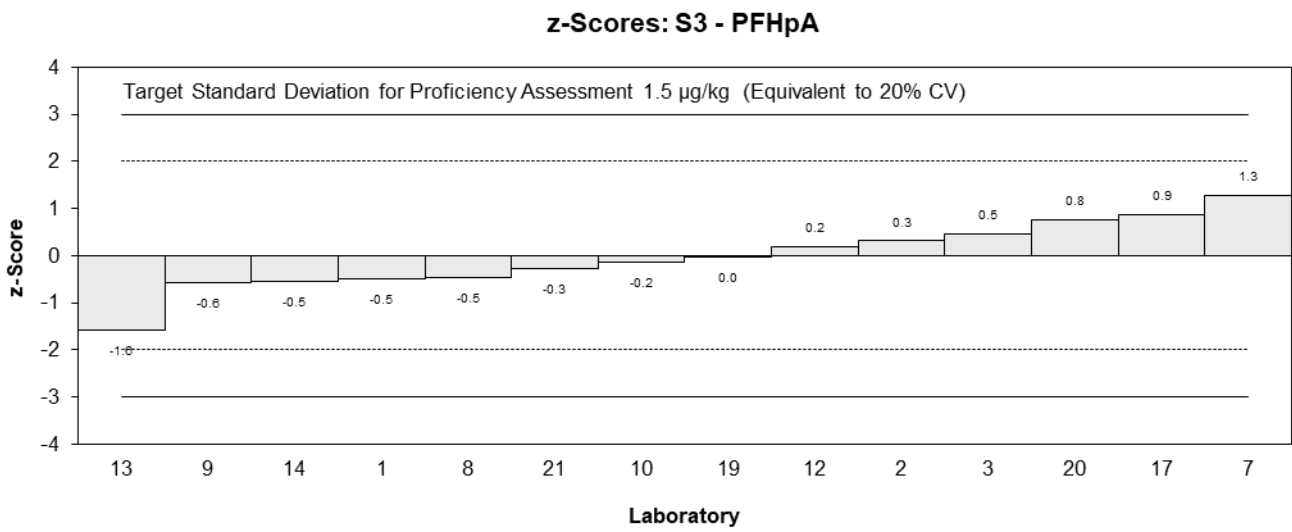
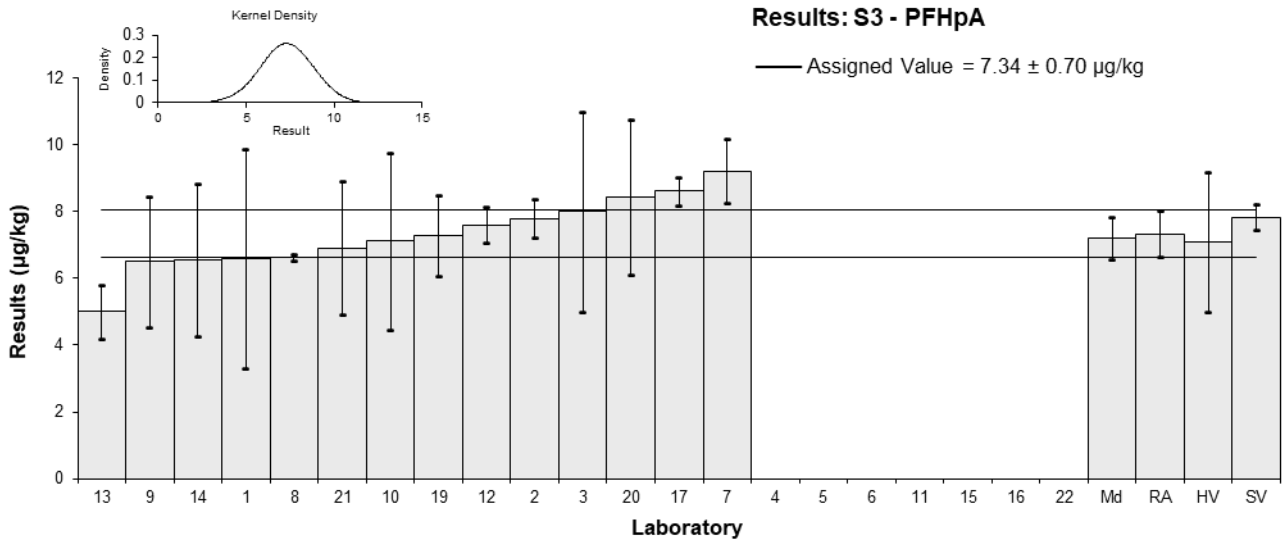


Figure 67

Table 73

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFOA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 5	2.5	106		
2	1.1	0.016	106	0.50	1.23
3	1	1	95	0.00	0.00
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	1.00	0.25	86	0.00	0.00
8	0.916	0.074	95.5	-0.42	-0.77
9	0.87	0.26	89	-0.65	-0.48
10	1.102	0.4	57.4	0.51	0.25
11	NS	NS	NS		
12	0.95	0.07	NR	-0.25	-0.47
13	1.3	0.30	NR	1.50	0.97
14	0.8711	0.3049	48	-0.64	-0.41
15	NS	NS	NS		
16	NS	NS	NS		
17	1.07	0.072	67	0.35	0.65
19	1.00	0.185	85	0.00	0.00
20	1.09	0.203	99.9	0.45	0.41
21	0.91	0.3	79	-0.45	-0.29
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	1.00	0.08
<b>Spike Value</b>	0.951	0.048
<b>Homogeneity Value</b>	1.01	0.30
<b>Robust Average</b>	1.00	0.08
<b>Median</b>	1.00	0.09
<b>Mean</b>	1.01	
<b>N</b>	13	
<b>Max</b>	1.3	
<b>Min</b>	0.87	
<b>Robust SD</b>	0.11	
<b>Robust CV</b>	11%	



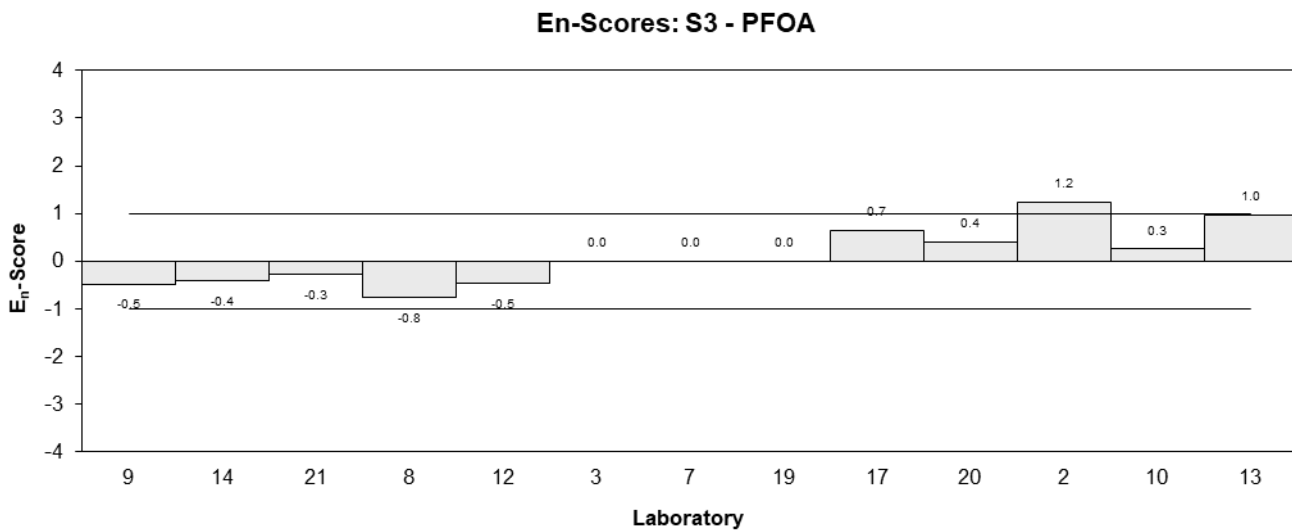
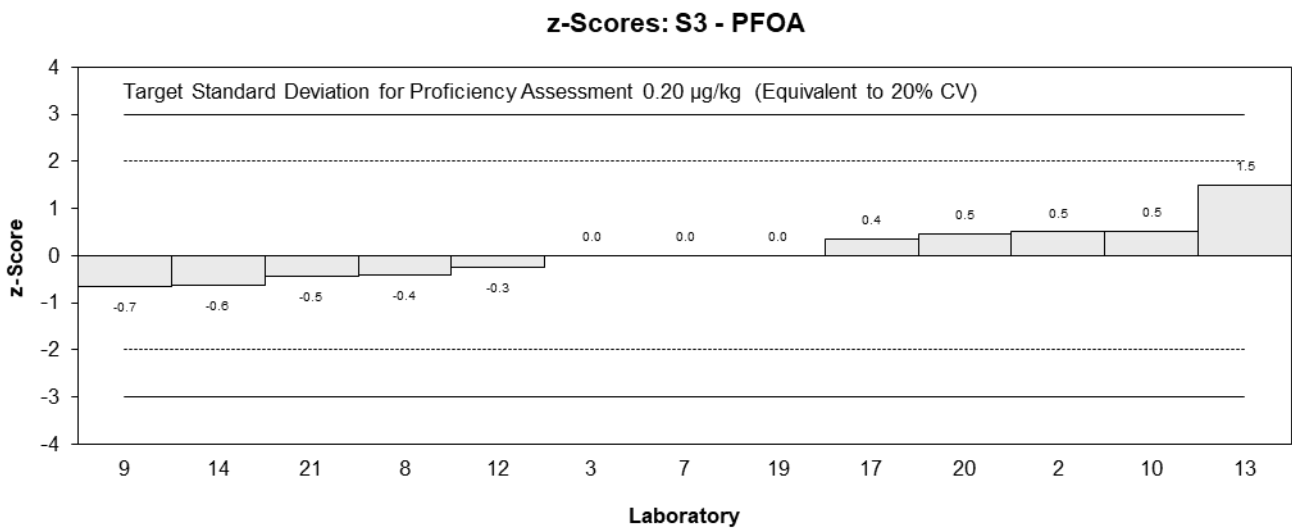
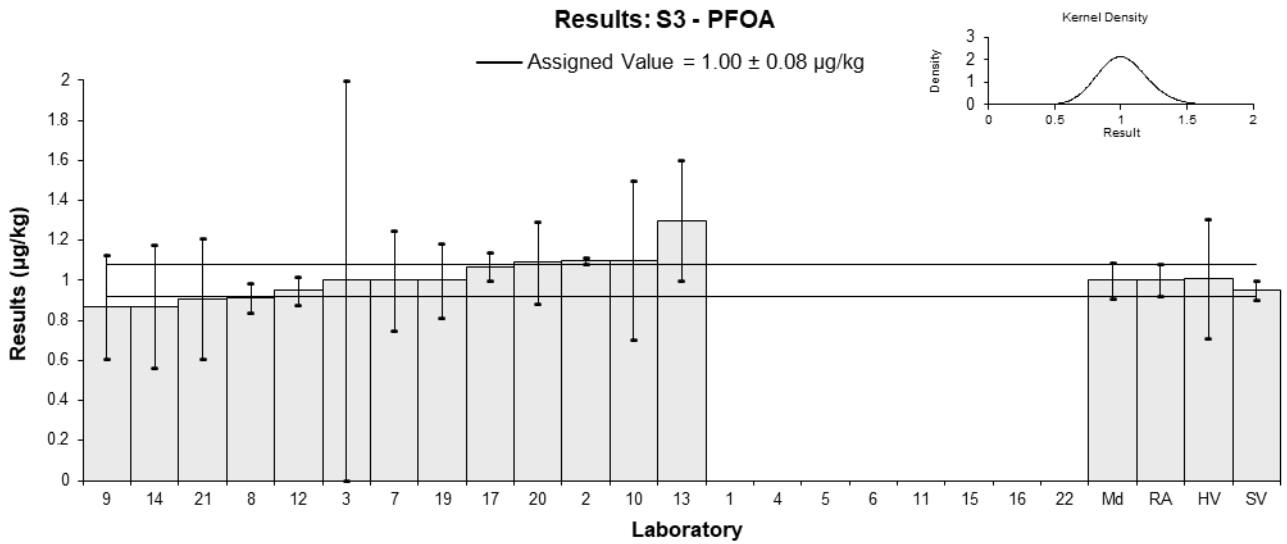


Figure 68

Table 74

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFNA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 2	1	100		
2	2.1	0.40	116	0.71	0.61
3	2	1	98	0.43	0.16
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	2.09	0.50	80	0.68	0.48
8	1.87	0.070	NR	0.08	0.18
9	1.49	0.45	78	-0.95	-0.74
10	1.63	0.57	62.59	-0.57	-0.36
11	NS	NS	NS		
12	1.91	0.14	NR	0.19	0.34
13	1.6	0.30	NR	-0.65	-0.72
14	1.8586	0.6505	16	0.05	0.03
15	NS	NS	NS		
16	NS	NS	NS		
17	1.781	0.14	67	-0.16	-0.29
19	1.71	0.258	79	-0.35	-0.44
20	2.06	0.586	93.3	0.60	0.36
21	1.83	0.5	74	-0.03	-0.02
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	1.84	0.15
<b>Spike Value</b>	1.96	0.10
<b>Homogeneity Value</b>	1.99	0.60
<b>Robust Average</b>	1.84	0.15
<b>Median</b>	1.86	0.15
<b>Mean</b>	1.84	
<b>N</b>	13	
<b>Max</b>	2.1	
<b>Min</b>	1.49	
<b>Robust SD</b>	0.22	
<b>Robust CV</b>	12%	

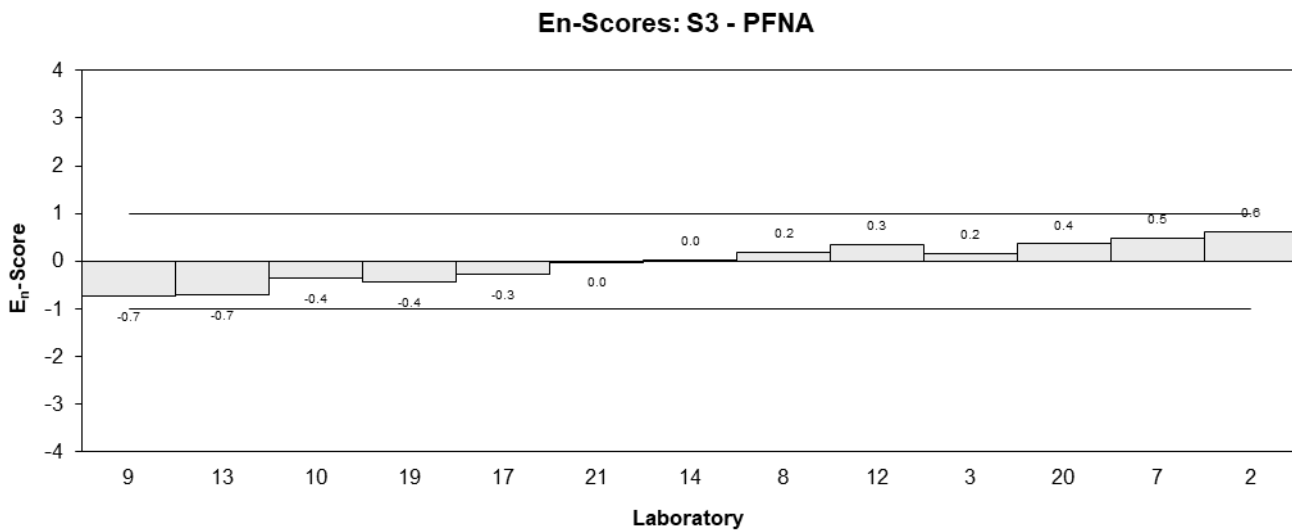
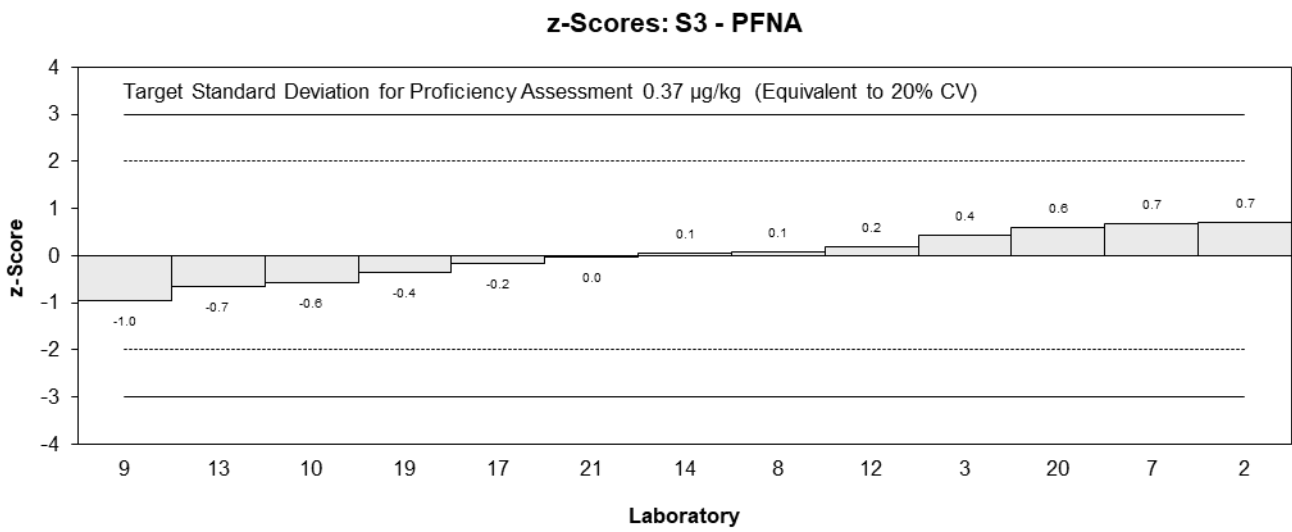
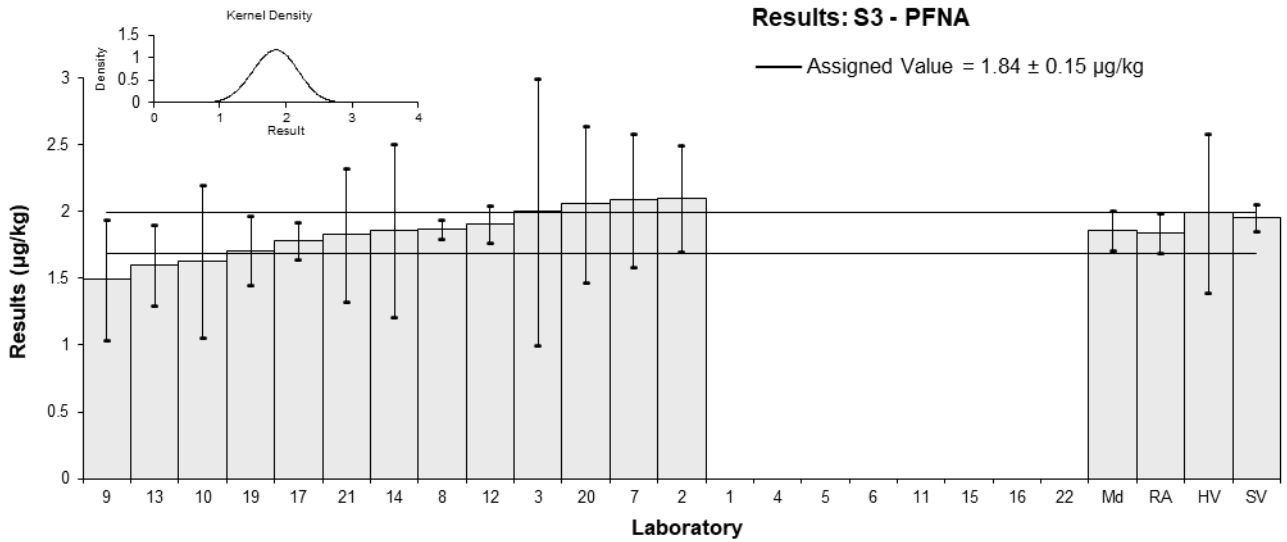


Figure 69

Table 75

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFDA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 5	2.5	105		
2	1.7	0.25	130	0.31	0.36
3	2	1	70	1.25	0.40
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	1.67	0.34	75	0.22	0.19
8	1.44	0.042	NR	-0.50	-1.26
9	1.42	0.43	74	-0.56	-0.40
10	1.728	0.6	61.13	0.40	0.21
11	NS	NS	NS		
12	1.69	0.13	NR	0.28	0.51
13	1.3	0.30	NR	-0.94	-0.93
14	1.4458	0.5060	71	-0.48	-0.30
15	NS	NS	NS		
16	NS	NS	NS		
17	1.665	0.118	67	0.20	0.39
19	1.54	0.251	64	-0.19	-0.22
20	1.67	0.444	86.2	0.22	0.15
21	1.66	0.5	75	0.19	0.12
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	1.60	0.12
<b>Spike Value</b>	1.64	0.08
<b>Homogeneity Value</b>	1.66	0.50
<b>Robust Average</b>	1.60	0.12
<b>Median</b>	1.67	0.06
<b>Mean</b>	1.61	
<b>N</b>	13	
<b>Max</b>	2	
<b>Min</b>	1.3	
<b>Robust SD</b>	0.17	
<b>Robust CV</b>	10%	

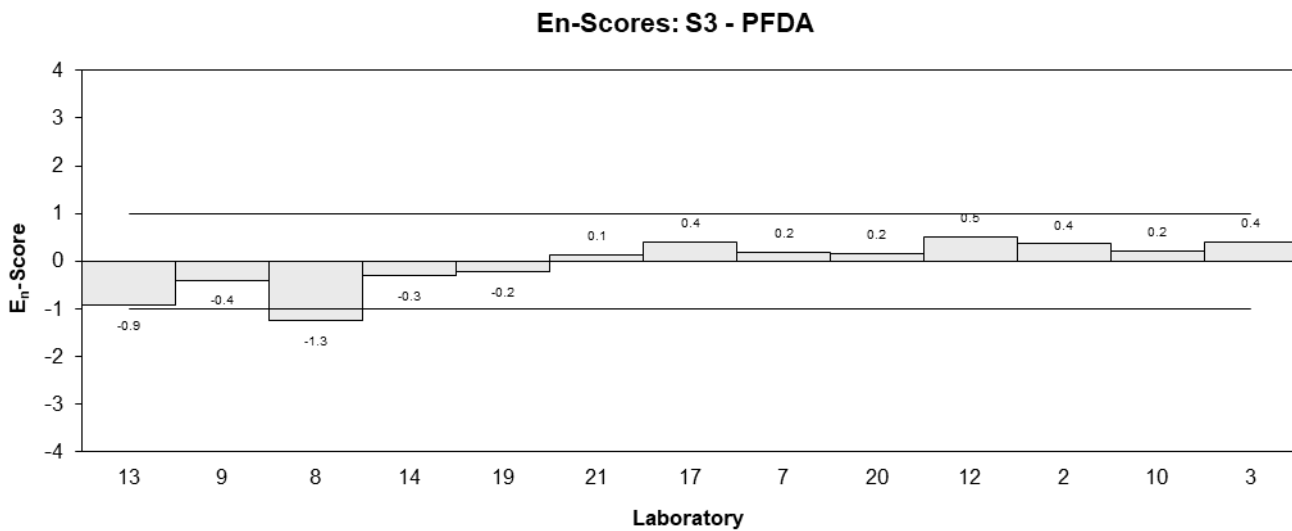
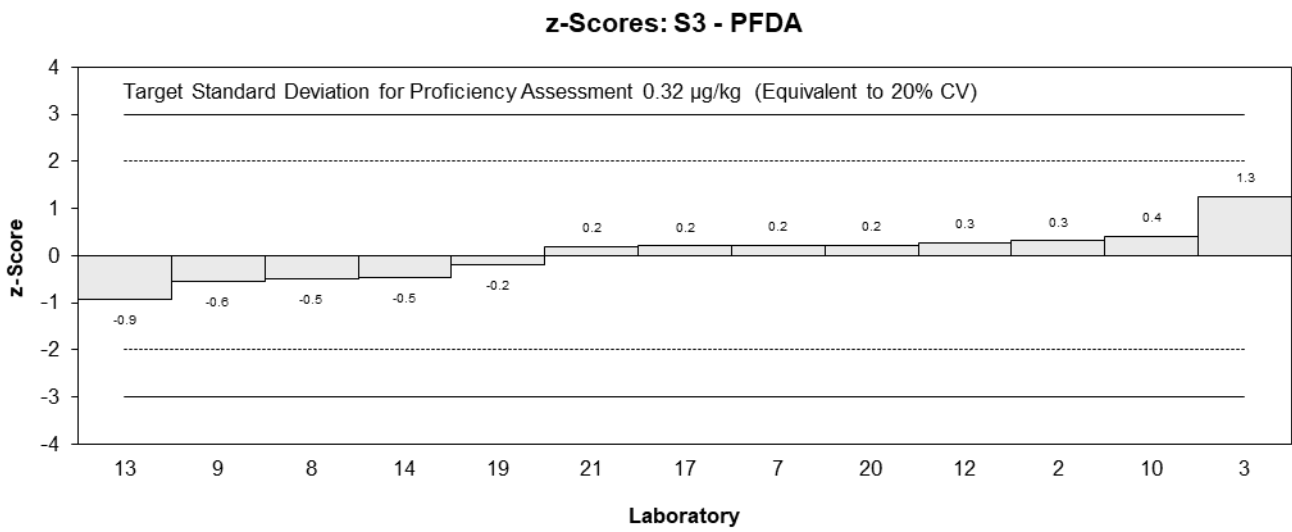
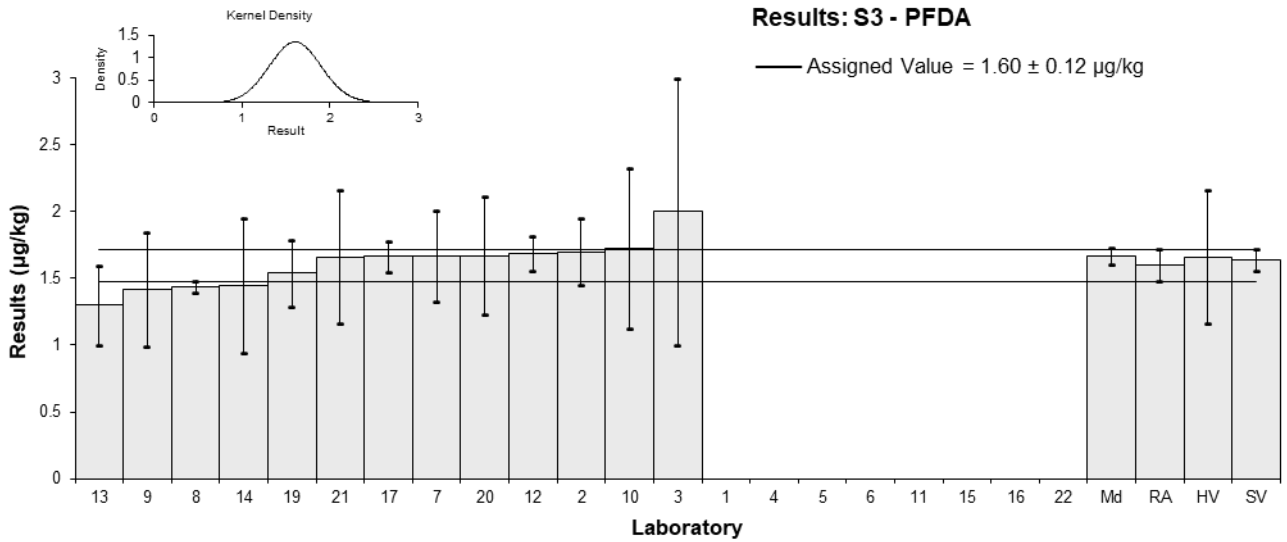


Figure 70

Table 76

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFUdA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 2	1	133		
2	0.77	0.068	145	0.05	0.09
3	< 2	NR	117		
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	<0.1	NR	75		
8	0.712	0.039	93.9	-0.33	-0.85
9	0.7	0.21	76	-0.41	-0.29
10	<1	NR	67.29		
11	NS	NS	NS		
12	0.74	0.06	NR	-0.15	-0.30
13	0.8	0.20	NR	0.24	0.18
14	0.6982	0.2444	82	-0.42	-0.26
15	NS	NS	NS		
16	NS	NS	NS		
17	0.821	0.029	69	0.38	1.07
19	0.751	0.116	41	-0.08	-0.10
20	0.807	0.319	109.6	0.29	0.14
21	0.834	0.3	67	0.47	0.23
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	0.763	0.046
<b>Spike Value</b>	0.781	0.039
<b>Homogeneity Value</b>	0.82	0.25
<b>Robust Average</b>	0.763	0.046
<b>Median</b>	0.761	0.056
<b>Mean</b>	0.763	
<b>N</b>	10	
<b>Max</b>	0.834	
<b>Min</b>	0.6982	
<b>Robust SD</b>	0.058	
<b>Robust CV</b>	7.5%	

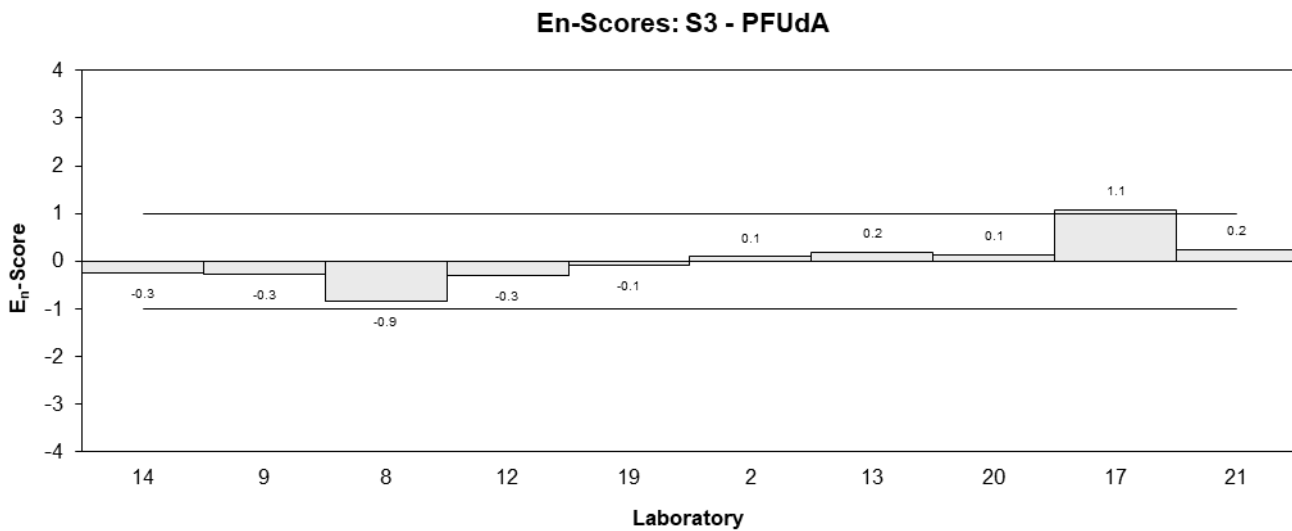
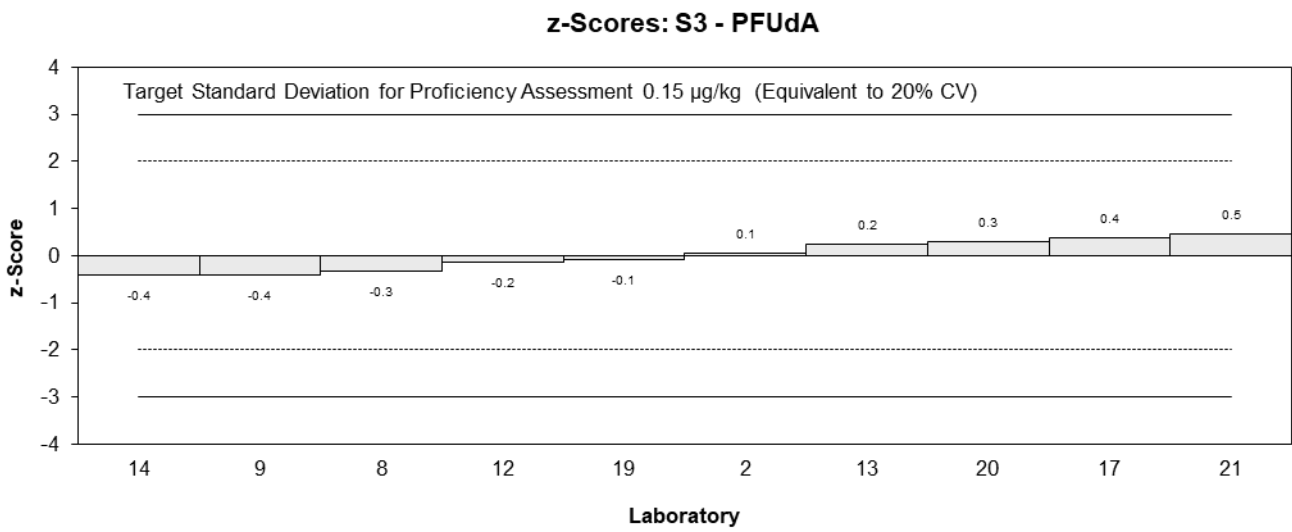
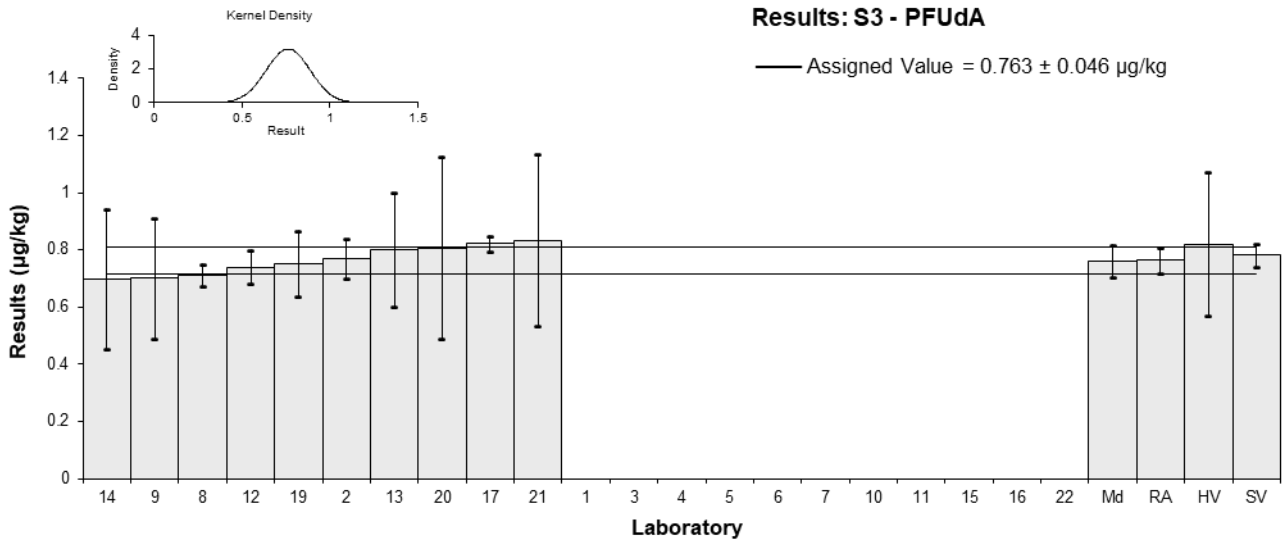


Figure 71

Table 77

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFDoA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 5	2.5	114		
2	3.9	0.42	214	0.49	0.77
3	4	2	103	0.63	0.22
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	3.45	1.05	54	-0.14	-0.09
8	3.40	0.057	91.6	-0.21	-0.79
9	3.51	1.05	62	-0.06	-0.04
10	3.47	1	66.55	-0.11	-0.08
11	NS	NS	NS		
12	3.63	0.26	NR	0.11	0.25
13	NT	NT	NT		
14	3.3080	1.1578	61	-0.34	-0.21
15	NS	NS	NS		
16	NS	NS	NS		
17	3.695	0.348	59	0.20	0.37
19	3.62	0.453	31	0.10	0.14
20	3.51	0.805	110.7	-0.06	-0.05
21	3.15	0.9	68	-0.56	-0.44
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	3.55	0.18
<b>Spike Value</b>	3.92	0.20
<b>Homogeneity Value</b>	3.5	1.1
<b>Robust Average</b>	3.55	0.18
<b>Median</b>	3.51	0.12
<b>Mean</b>	3.55	
<b>N</b>	12	
<b>Max</b>	4	
<b>Min</b>	3.15	
<b>Robust SD</b>	0.25	
<b>Robust CV</b>	7.1%	



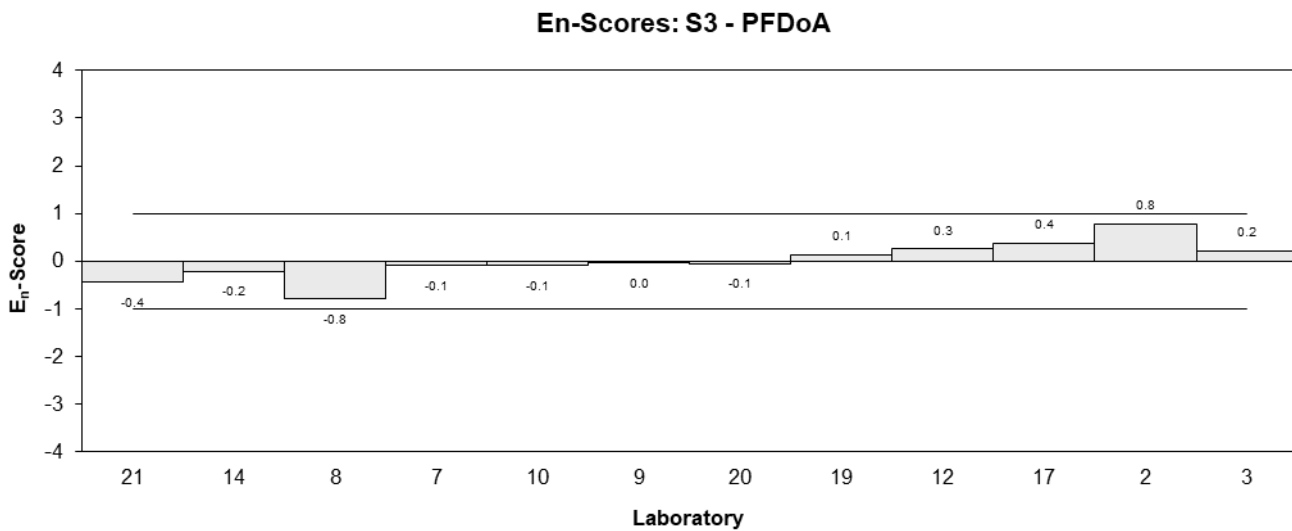
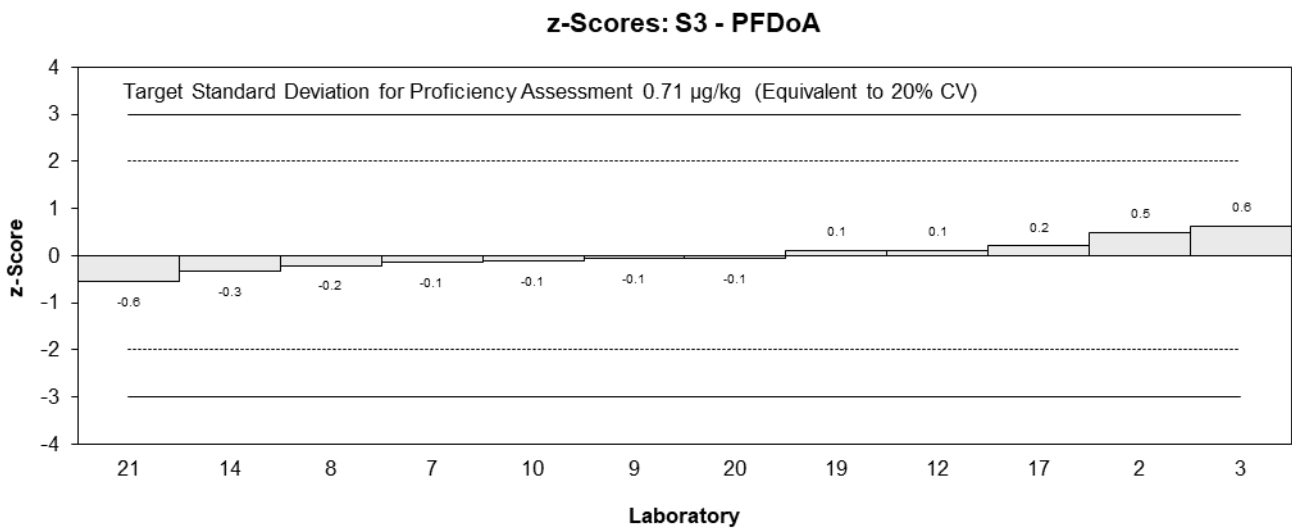
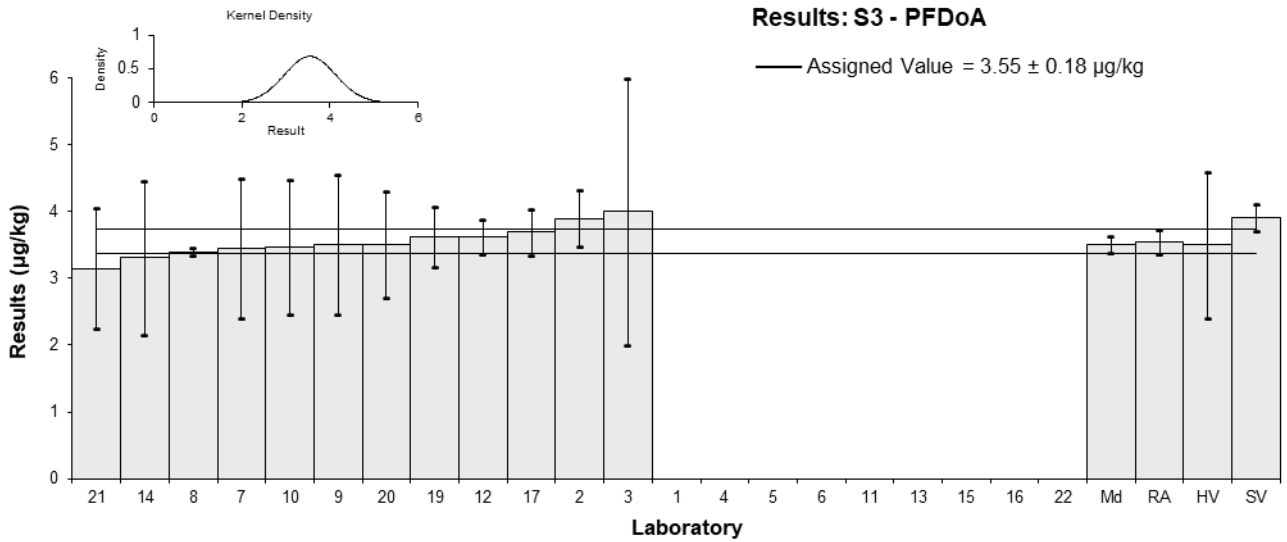


Figure 72

Table 78

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFTeDA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 5	2.5	169		
2	< 0.50	NR	118		
3	< 5	NR	122		
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	1.50	0.44	15	-0.07	-0.04
8	1.44	0.061	75.8	-0.26	-0.47
9	1.41	0.43	59	-0.36	-0.24
10	<2	NR	69.65		
11	NS	NS	NS		
12	1.86	0.13	NR	1.12	1.65
13	0.8	0.20	NR	-2.37	-2.81
14	1.4011	0.4904	62	-0.39	-0.23
15	NS	NS	NS		
16	NS	NS	NS		
17	1.504	0.1	43	-0.05	-0.08
19	1.51	0.23	24	-0.03	-0.04
20	1.76	0.651	82.2	0.79	0.36
21	1.64	0.7	41	0.39	0.17
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	1.52	0.16
<b>Spike Value</b>	1.76	0.09
<b>Homogeneity Value</b>	1.70	0.68
<b>Robust Average</b>	1.52	0.16
<b>Median</b>	1.50	0.11
<b>Mean</b>	1.48	
<b>N</b>	10	
<b>Max</b>	1.86	
<b>Min</b>	0.8	
<b>Robust SD</b>	0.20	
<b>Robust CV</b>	13%	

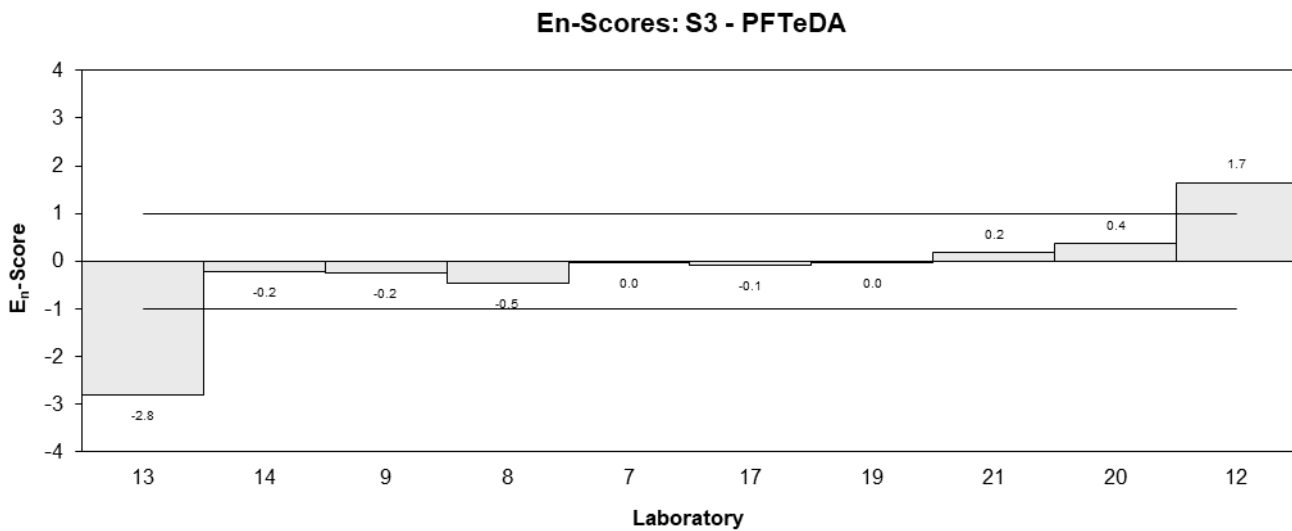
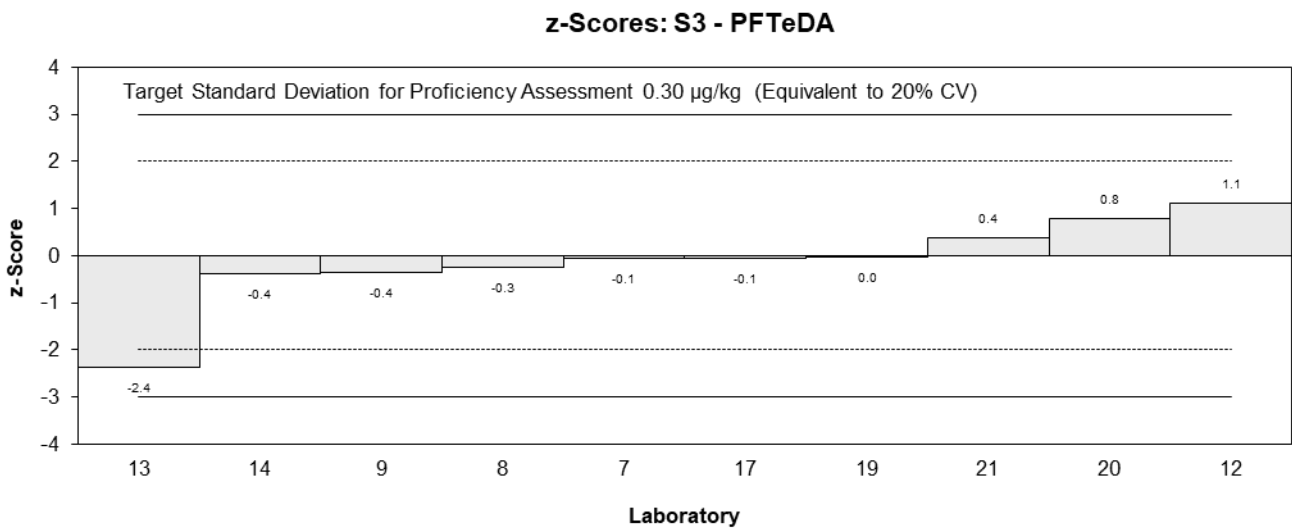
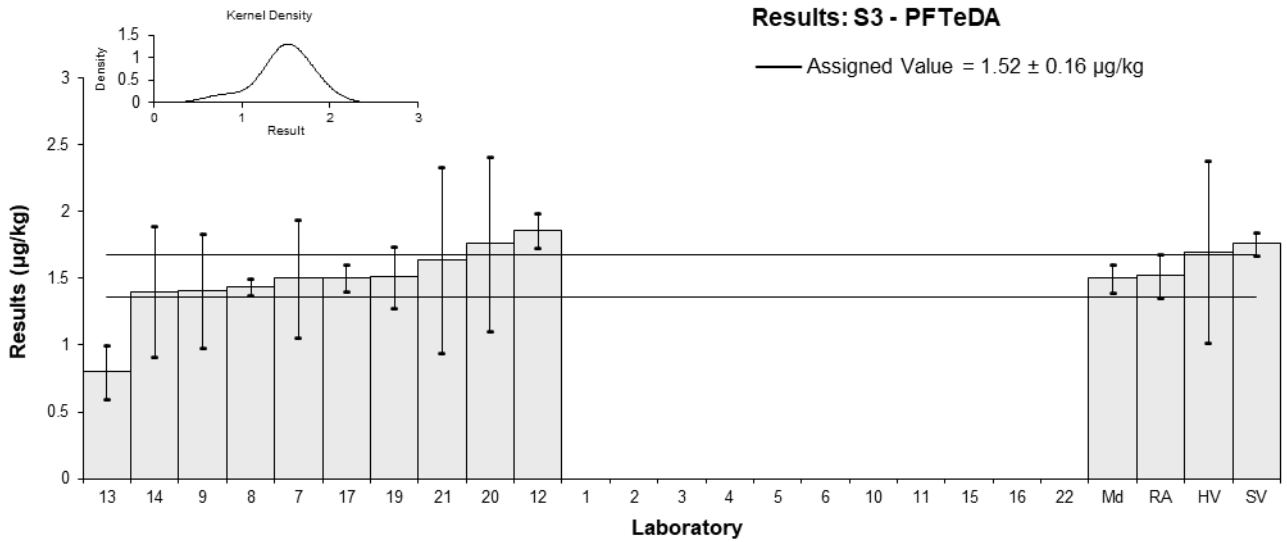


Figure 73

Table 79

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	PFOSA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	4.9	2.45	119	-0.28	-0.11
2	5.6	0.034	94	0.39	0.68
3	7	5	102	1.74	0.36
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	5.06	0.79	71	-0.13	-0.13
8	4.80	0.065	102.8	-0.38	-0.65
9	4.16	1.25	12	-0.99	-0.74
10	5.946	2.35	77.81	0.73	0.31
11	NS	NS	NS		
12	NT	NT	NT		
13	4.1	0.30	NR	-1.05	-1.62
14	NT	NT	NT		
15	NS	NS	NS		
16	NS	NS	NS		
17	6.197	0.289	59	0.97	1.51
19	5.06	1.5	36	-0.13	-0.08
20	5.08	0.964	50.6	-0.11	-0.10
21	4.89	1	70	-0.29	-0.26
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	5.19	0.60
<b>Spike Value</b>	5.58	0.28
<b>Homogeneity Value</b>	4.7	1.4
<b>Robust Average</b>	5.19	0.60
<b>Median</b>	5.06	0.43
<b>Mean</b>	5.23	
<b>N</b>	12	
<b>Max</b>	7	
<b>Min</b>	4.1	
<b>Robust SD</b>	0.83	
<b>Robust CV</b>	16%	

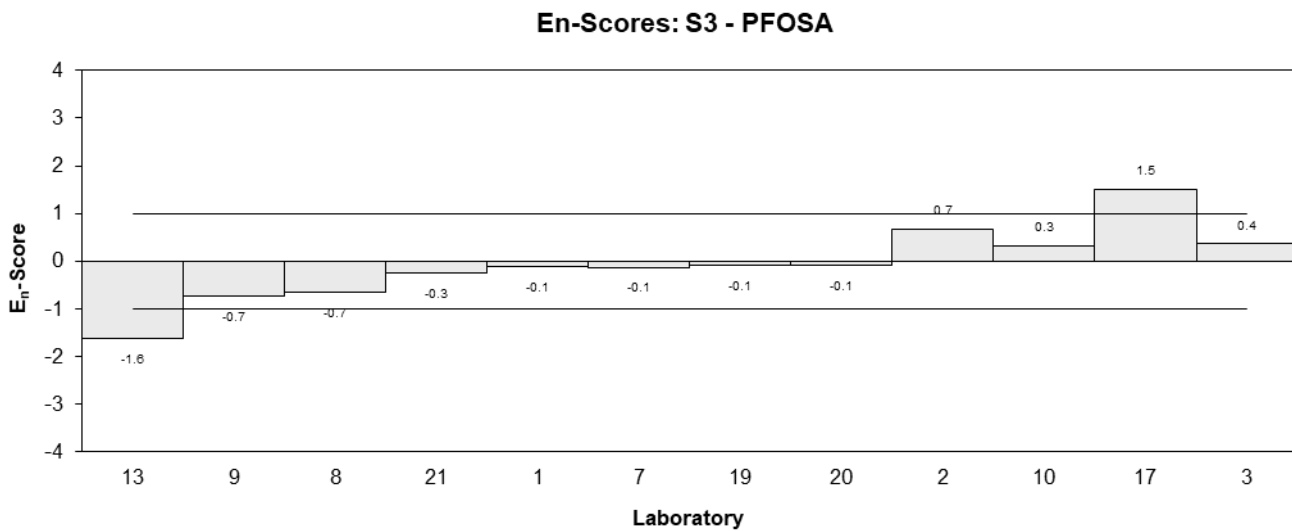
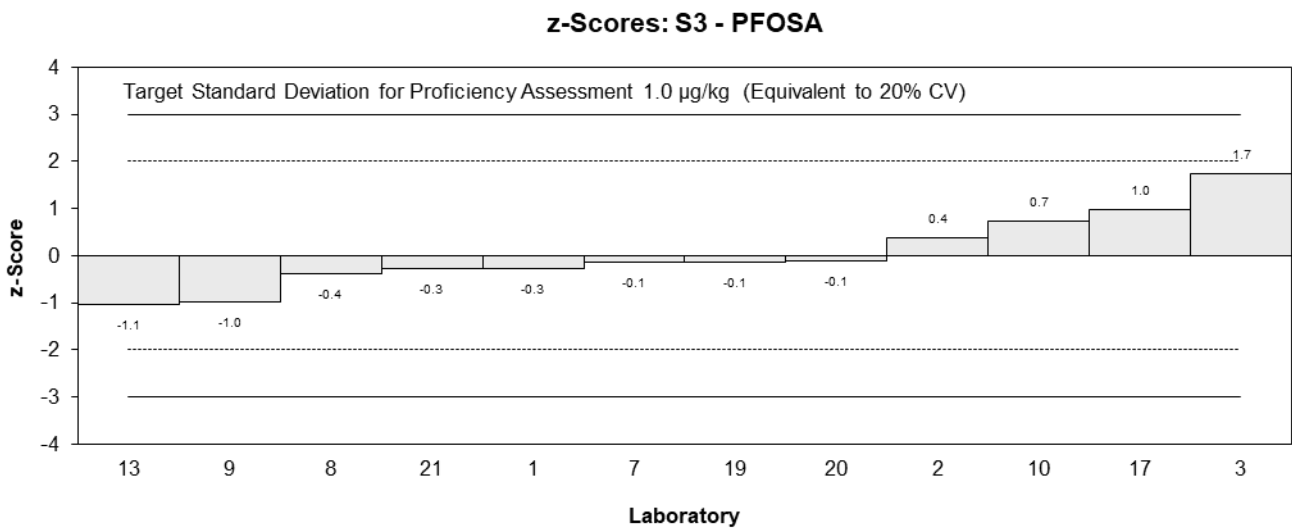
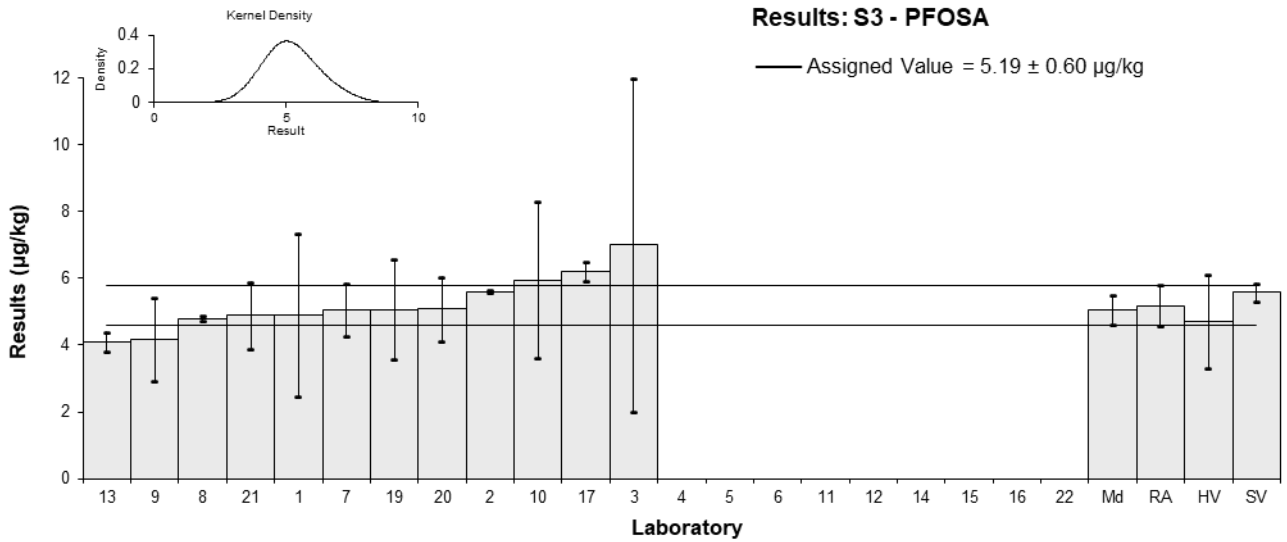


Figure 74

Table 80

**Sample Details**

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	EtFOSA
<b>Unit</b>	µg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>Uncertainty</b>	<b>Rec</b>
1	< 5	2.5	50
2	NR	NR	65
3	5	5	68
4	NS	NS	NS
5	NS	NS	NS
6	NS	NS	NS
7	NT	NT	NT
8	NT	NT	NT
9	4.08	1.2	54
10	4.946	1.96	65.26
11	NS	NS	NS
12	NT	NT	NT
13	1.9	0.58	NR
14	NT	NT	NT
15	NS	NS	NS
16	NS	NS	NS
17	NT	NT	NT
19	5.84	1.41	22
20	1.50	0.116	50.6
21	4.47	2	39
22	NS	NS	NS

**Statistics**

<b>Assigned Value</b>	Not Set	
<b>Spike Value</b>	4.89	0.24
<b>Homogeneity Value</b>	4.5	1.8
<b>Robust Average</b>	4.0	1.8
<b>Median</b>	4.47	0.74
<b>Mean</b>	4.0	
<b>N</b>	7	
<b>Max</b>	5.84	
<b>Min</b>	1.5	
<b>Robust SD</b>	1.9	
<b>Robust CV</b>	47%	

Results: S3 - EtFOSA

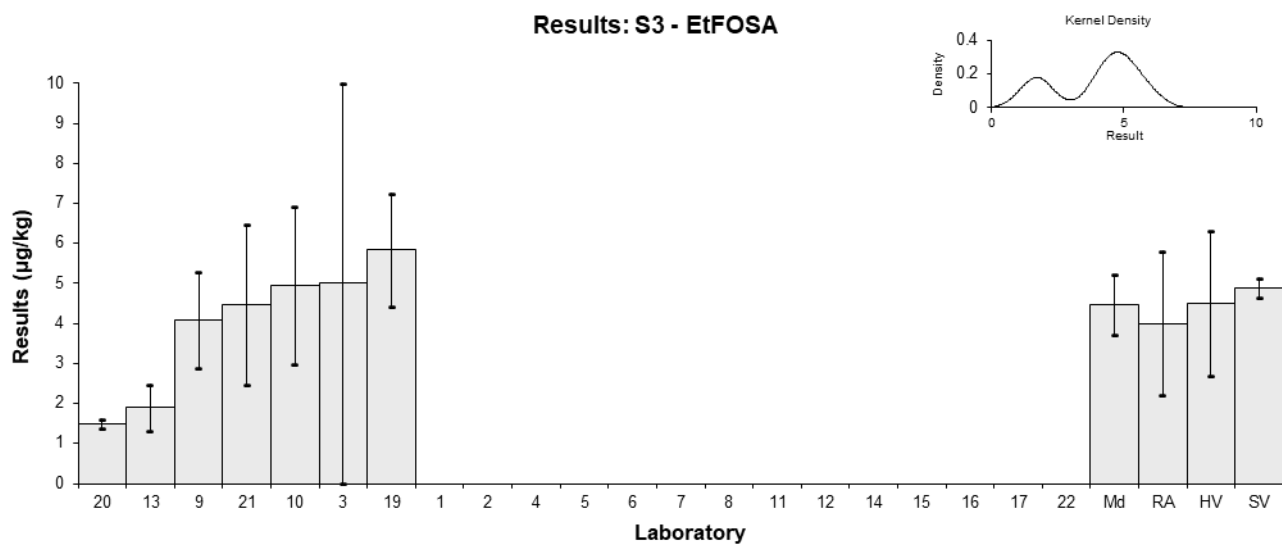


Figure 75

Table 81

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	EtFOSAA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 5	2.5	138		
2	NR	NR	269		
3	5	3	98	0.53	0.16
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	3.97	0.57	35	-0.61	-0.60
8	NT	NT	NT		
9	3.82	1.15	92	-0.77	-0.52
10	4.384	1.51	108.1	-0.15	-0.08
11	NS	NS	NS		
12	NT	NT	NT		
13	6.5	0.80	NR	2.19	1.85
14	NT	NT	NT		
15	NS	NS	NS		
16	NS	NS	NS		
17	NT	NT	NT		
19	4.79	1.14	61	0.30	0.20
20	NT	NT	NT		
21	4.02	1	87	-0.55	-0.41
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	4.52	0.71
<b>Spike Value</b>	4.89	0.24
<b>Homogeneity Value</b>	3.9	1.2
<b>Robust Average</b>	4.52	0.71
<b>Median</b>	4.38	0.58
<b>Mean</b>	4.64	
<b>N</b>	7	
<b>Max</b>	6.5	
<b>Min</b>	3.82	
<b>Robust SD</b>	0.75	
<b>Robust CV</b>	17%	



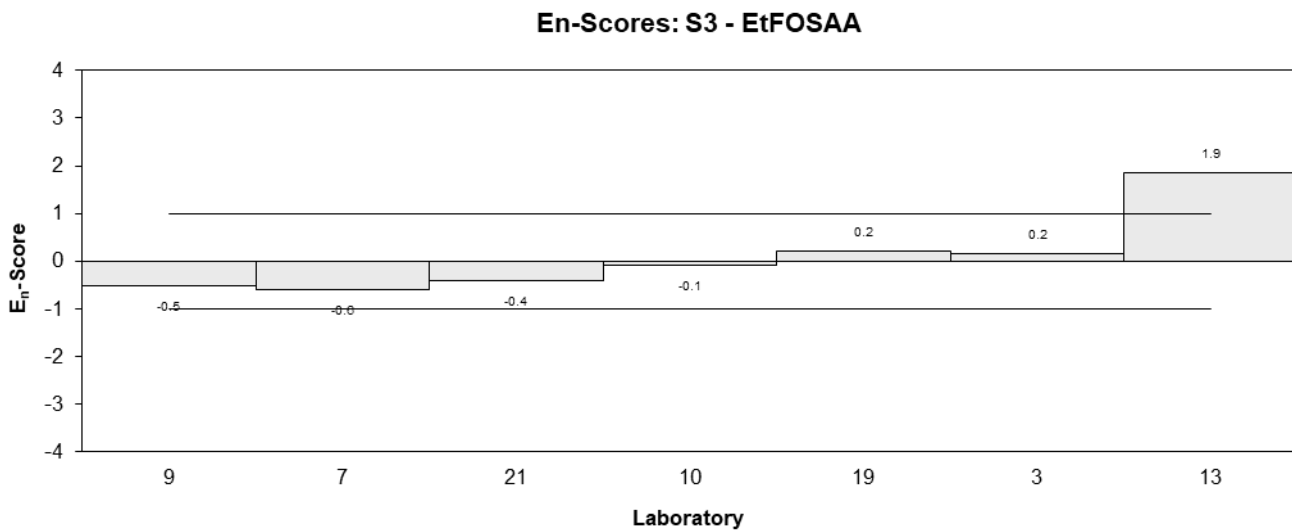
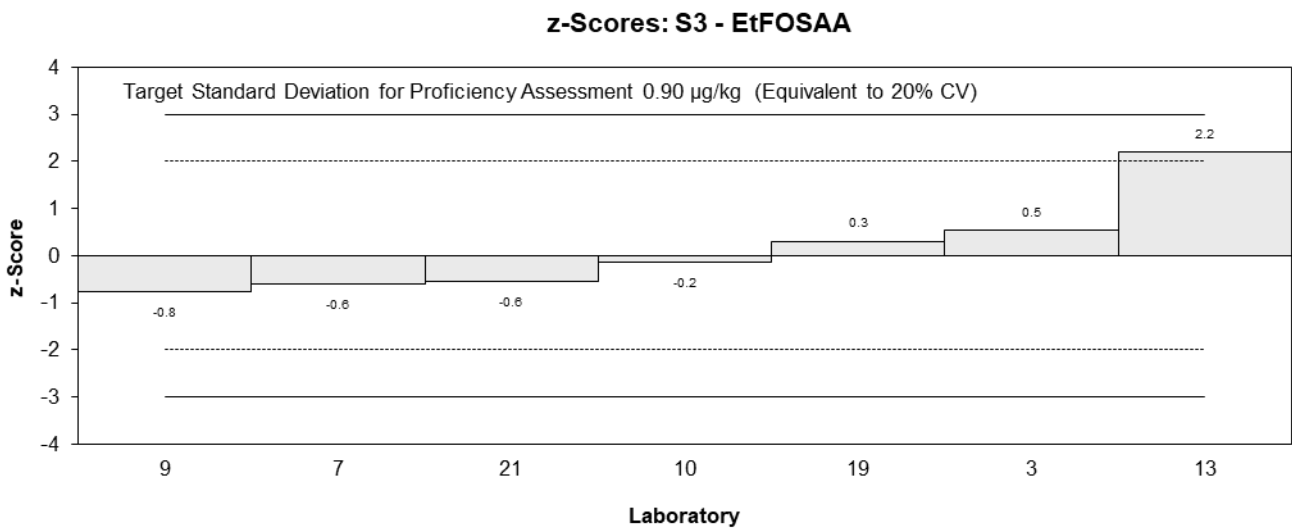
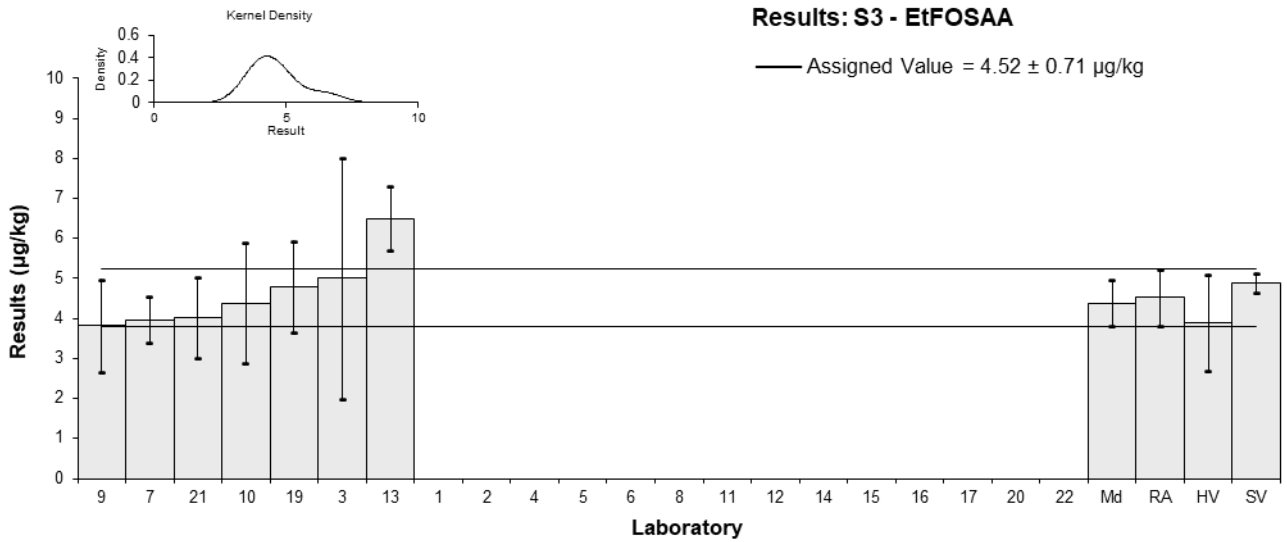


Figure 76

Table 82

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	8:2FTS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	< 1	0.5	104		
2	11	1.1	179	1.08	1.43
3	10	4	100	0.52	0.23
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	9.03	1.82	144	-0.01	-0.01
8	8.29	0.15	127.2	-0.42	-0.92
9	7.76	2.33	57	-0.71	-0.52
10	9.582	3.43	116.33	0.29	0.15
11	NS	NS	NS		
12	NT	NT	NT		
13	8.3	1.00	NR	-0.41	-0.58
14	NT	NT	NT		
15	NS	NS	NS		
16	NS	NS	NS		
17	9.359	0.237	61	0.17	0.37
19	8.21	1.15	99	-0.46	-0.60
20	NT	NT	NT		
21	9.4	3	68	0.19	0.11
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	9.05	0.81
<b>Spike Value</b>	9.39	0.47
<b>Homogeneity Value</b>	10.4	3.1
<b>Robust Average</b>	9.05	0.81
<b>Median</b>	9.2	1.0
<b>Mean</b>	9.09	
<b>N</b>	10	
<b>Max</b>	11	
<b>Min</b>	7.76	
<b>Robust SD</b>	1.0	
<b>Robust CV</b>	11%	

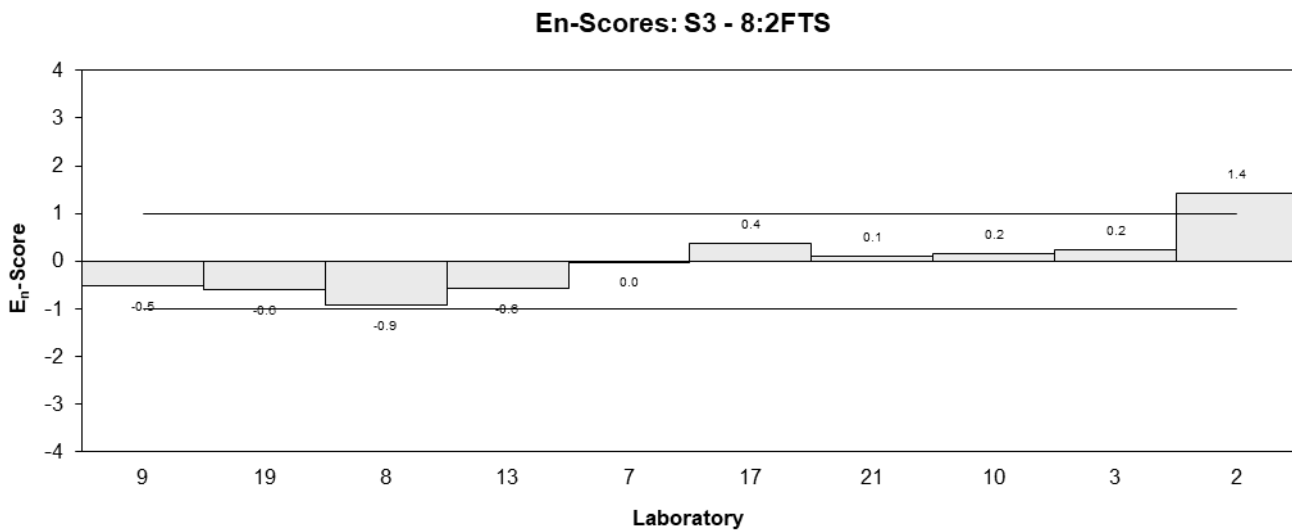
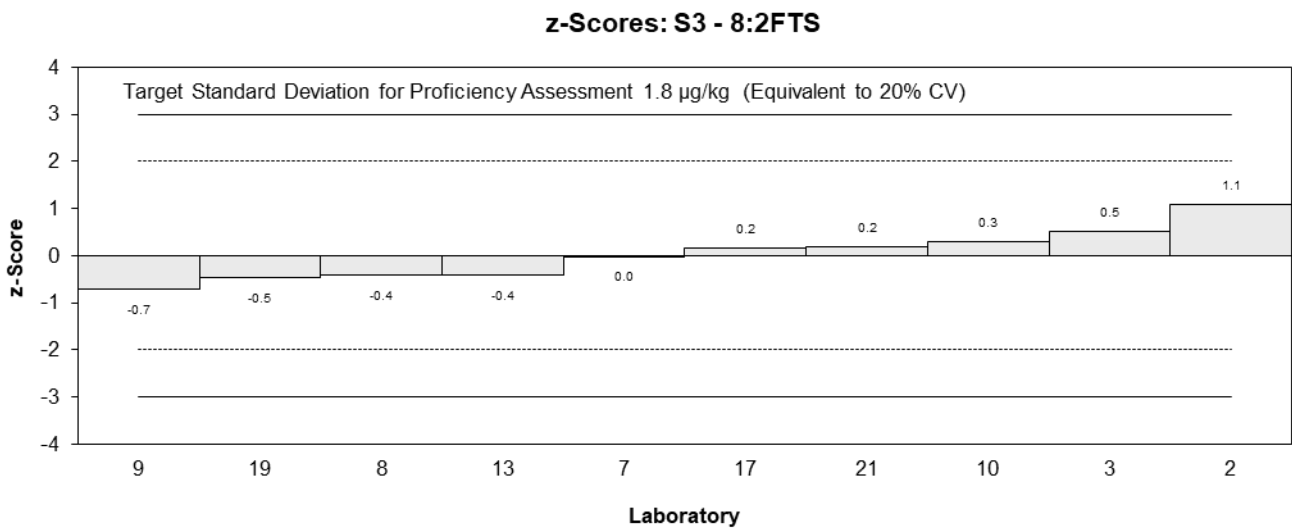
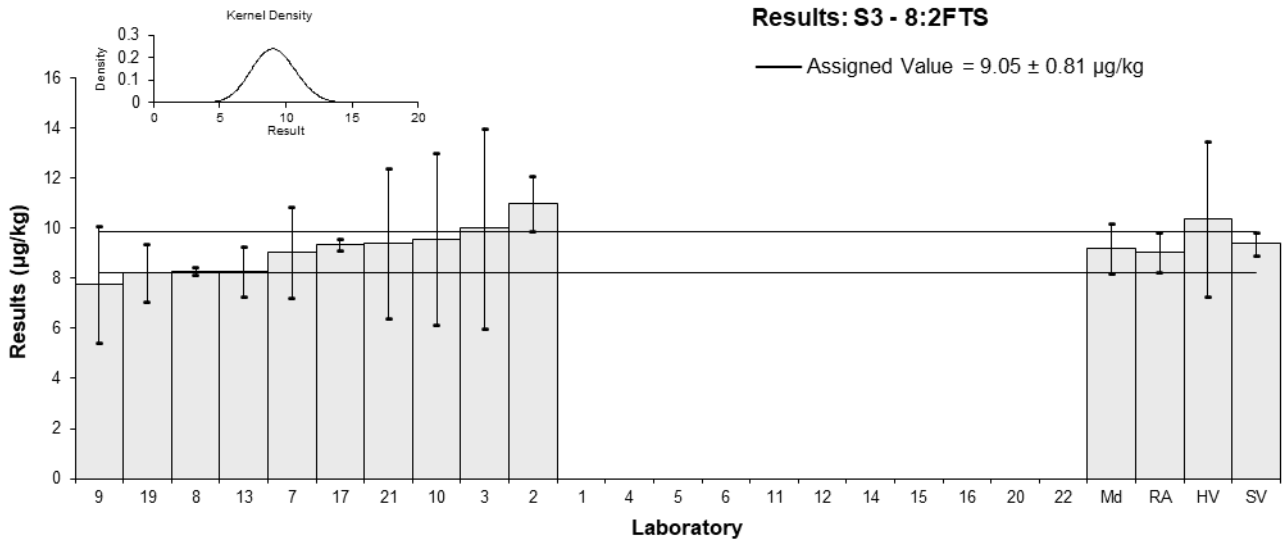


Figure 77

Table 83

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	GenX
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	NT	NT	NT		
2	15	1.6	89	0.51	0.72
3	< 50	NR	95		
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	NT	NT	NT		
8	12.9	0.057	NR	-0.26	-0.64
9	12.36	3.7	38	-0.46	-0.32
10	NT	NT	NT		
11	NS	NS	NS		
12	14.28	1.01	NR	0.25	0.46
13*	21	2.00	NR	2.72	3.24
14	NT	NT	NT		
15	NS	NS	NS		
16	NS	NS	NS		
17	14.653	0.341	65	0.39	0.91
19	13.2	3.52	72	-0.15	-0.11
20	NT	NT	NT		
21	13	4	69	-0.22	-0.14
22	NS	NS	NS		

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	13.6	1.1
<b>Spike Value</b>	14.7	0.7
<b>Homogeneity Value</b>	13.6	4.1
<b>Robust Average</b>	13.9	1.3
<b>Median</b>	13.7	1.1
<b>Mean</b>	14.5	
<b>N</b>	8	
<b>Max</b>	21	
<b>Min</b>	12.36	
<b>Robust SD</b>	1.4	
<b>Robust CV</b>	10%	

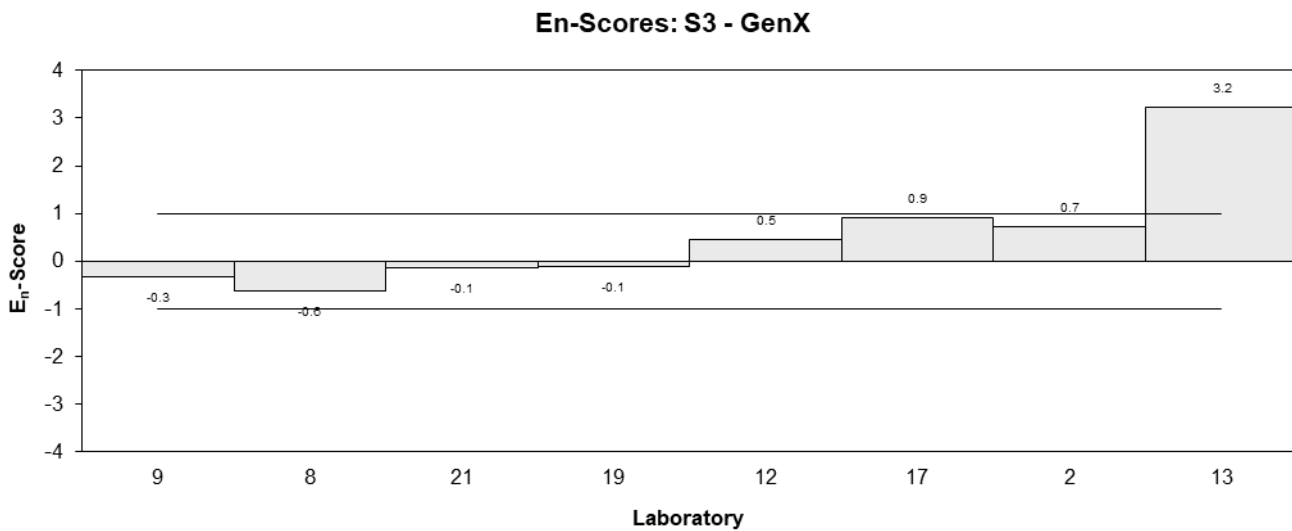
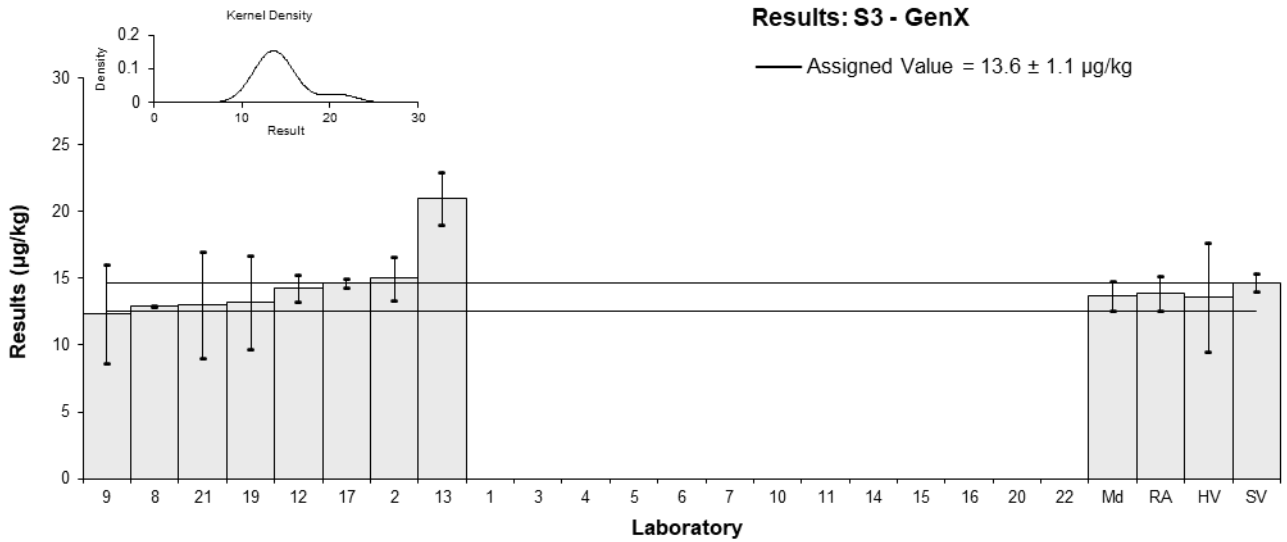


Figure 78

Table 84

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	ADONA
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	NT	NT	NT		
2	18	1.3	110	0.42	0.53
3	16	6	96	-0.18	-0.09
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	15.67	2.09	86	-0.28	-0.30
8	16.5	0.052	NR	-0.03	-0.04
9*	4.96	1.49	103	-3.51	-4.25
10	NT	NT	NT		
11	NS	NS	NS		
12	14.18	1.07	NR	-0.73	-0.95
13	11	1.00	NR	-1.69	-2.23
14	NT	NT	NT		
15	NS	NS	NS		
16	NS	NS	NS		
17	18.686	0.686	67	0.63	0.87
19	22.6	6.46	67	1.81	0.87
20	NT	NT	NT		
21	17.1	5	NR	0.15	0.09
22	NS	NS	NS		

\* Outlier, see Section 4.2

## Statistics

<b>Assigned Value</b>	16.6	2.3
<b>Spike Value</b>	18.4	0.9
<b>Homogeneity Value</b>	18.1	5.4
<b>Robust Average</b>	15.9	3.2
<b>Median</b>	16.3	2.2
<b>Mean</b>	15.5	
<b>N</b>	10	
<b>Max</b>	22.6	
<b>Min</b>	4.96	
<b>Robust SD</b>	4.0	
<b>Robust CV</b>	25%	

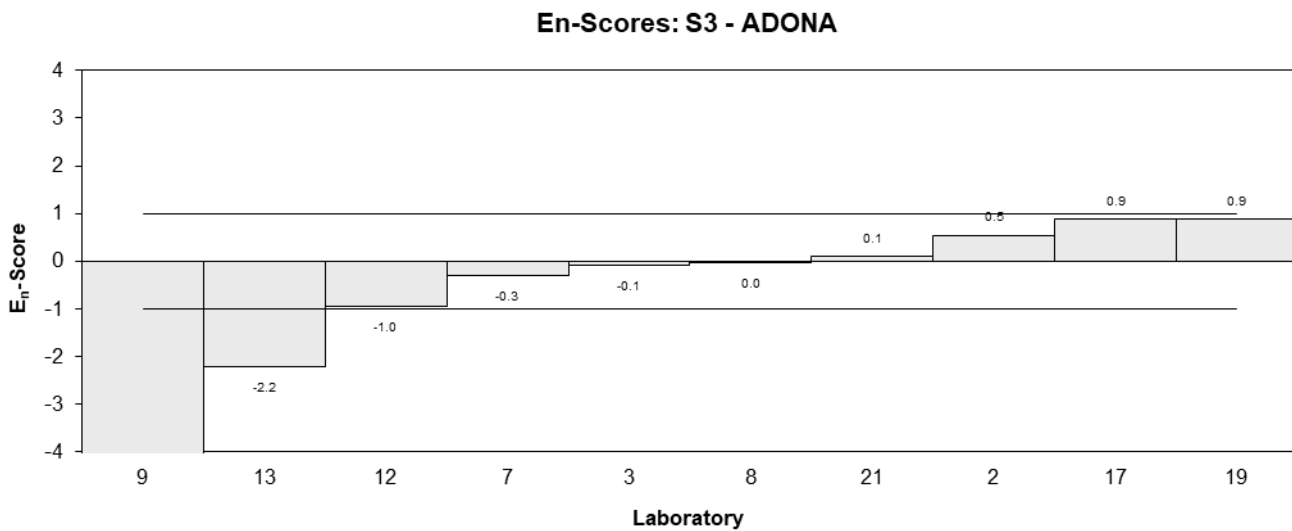
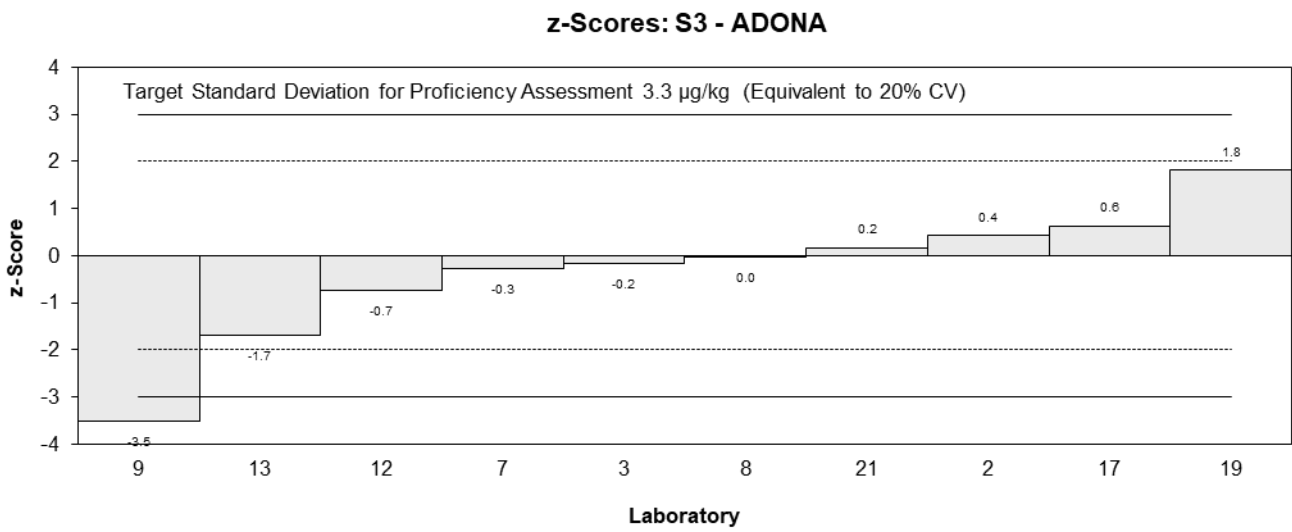
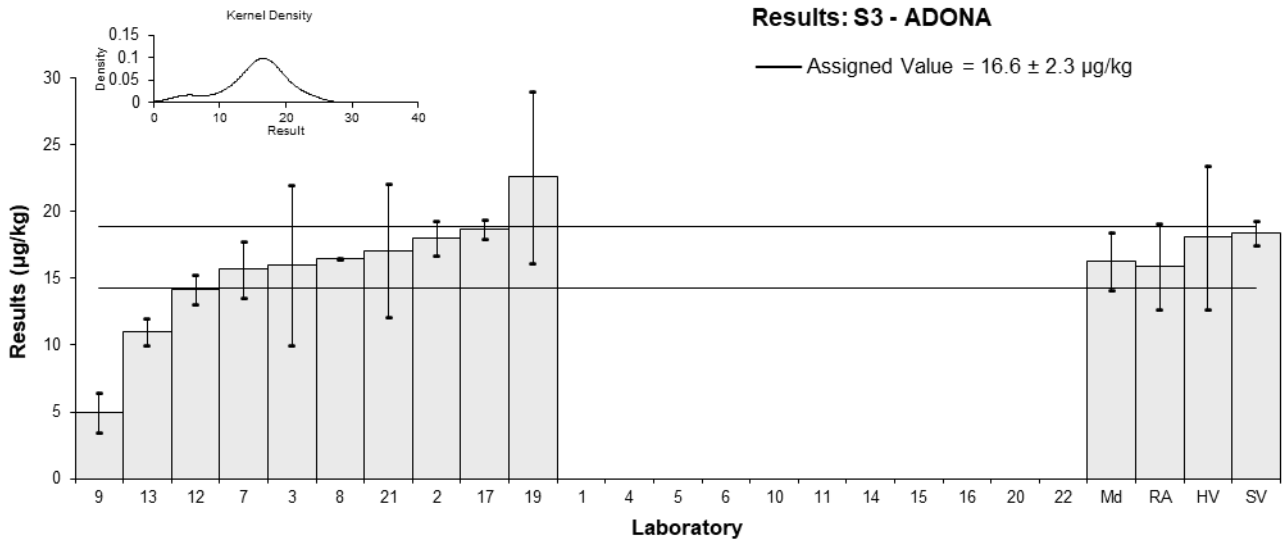


Figure 79

Table 85

## Sample Details

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	9CI-PF3ONS
<b>Unit</b>	µg/kg

## Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	NT	NT	NT		
2	23	1.0	97	0.90	1.22
3	19	6	102	-0.13	-0.08
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	21.43	1.84	97	0.49	0.59
8	18.7	0.080	NR	-0.21	-0.30
9	15.53	4.66	87	-1.02	-0.74
10	NT	NT	NT		
11	NS	NS	NS		
12	19.77	1.41	NR	0.07	0.09
13	15	2.53	NR	-1.15	-1.22
14	NT	NT	NT		
15	NS	NS	NS		
16	NS	NS	NS		
17	21.876	1.194	78	0.61	0.80
19	16.9	3.93	67	-0.67	-0.55
20	NT	NT	NT		
21	23.3	9	NR	0.97	0.40
22	NS	NS	NS		

## Statistics

<b>Assigned Value</b>	19.5	2.7
<b>Spike Value</b>	22.8	1.1
<b>Homogeneity Value</b>	23.4	9.4
<b>Robust Average</b>	19.5	2.7
<b>Median</b>	19.4	2.9
<b>Mean</b>	19.5	
<b>N</b>	10	
<b>Max</b>	23.3	
<b>Min</b>	15	
<b>Robust SD</b>	3.4	
<b>Robust CV</b>	17%	



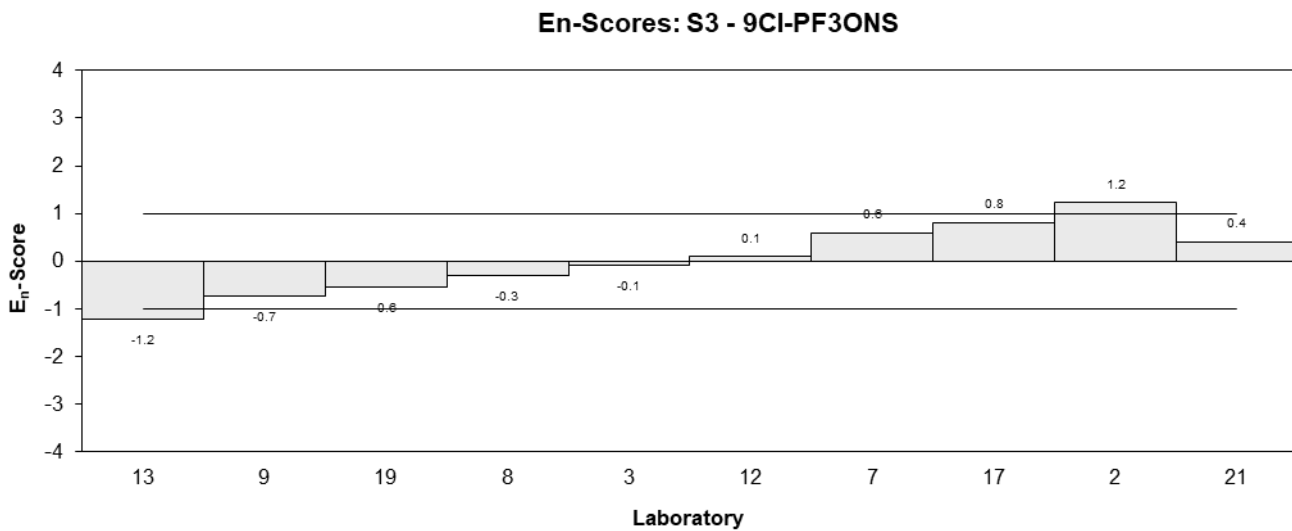
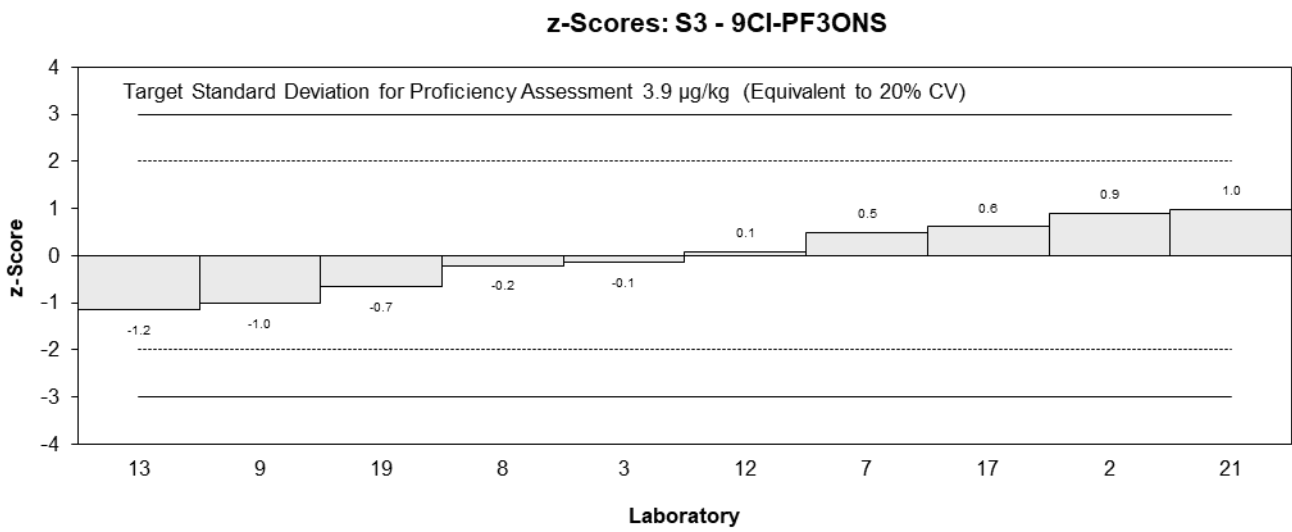
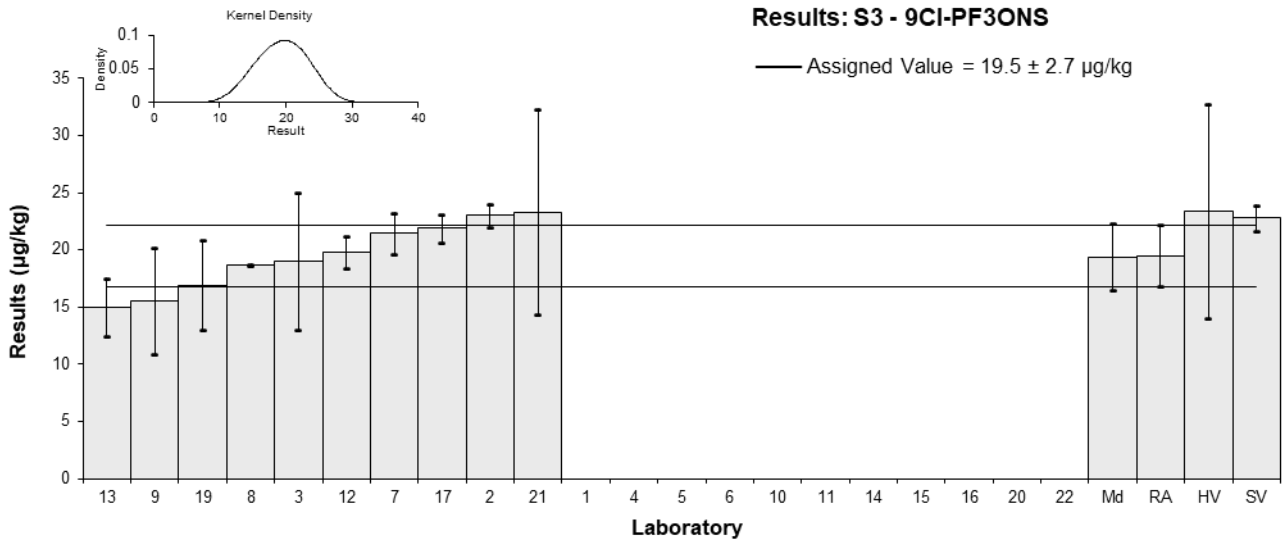


Figure 80

Table 86

**Sample Details**

<b>Sample No.</b>	S3
<b>Matrix</b>	Bovine Liver
<b>Analyte</b>	11Cl-PF3OUdS
<b>Unit</b>	µg/kg

**Participant Results**

Lab. Code	Result	Uncertainty	Rec	z	E <sub>n</sub>
1	NT	NT	NT		
2	28	1.1	145	1.73	2.64
3	23	8	102	0.53	0.26
4	NS	NS	NS		
5	NS	NS	NS		
6	NS	NS	NS		
7	19.39	3.75	97	-0.34	-0.31
8	21.1	0.091	NR	0.07	0.12
9	16.6	4.98	87	-1.01	-0.75
10	NT	NT	NT		
11	NS	NS	NS		
12	18.6	1.33	NR	-0.53	-0.78
13	19	2.50	NR	-0.43	-0.51
14	NT	NT	NT		
15	NS	NS	NS		
16	NS	NS	NS		
17	21.34	0.899	78	0.13	0.20
19*	10.0	3.79	67	-2.60	-2.38
20	NT	NT	NT		
21	22.5	9	NR	0.41	0.18
22	NS	NS	NS		

\* Outlier, see Section 4.2

**Statistics**

<b>Assigned Value</b>	20.8	2.5
<b>Spike Value</b>	27.7	1.4
<b>Homogeneity Value</b>	22.9	9.2
<b>Robust Average</b>	20.2	2.9
<b>Median</b>	20.2	2.3
<b>Mean</b>	20.0	
<b>N</b>	10	
<b>Max</b>	28	
<b>Min</b>	10	
<b>Robust SD</b>	3.6	
<b>Robust CV</b>	18%	

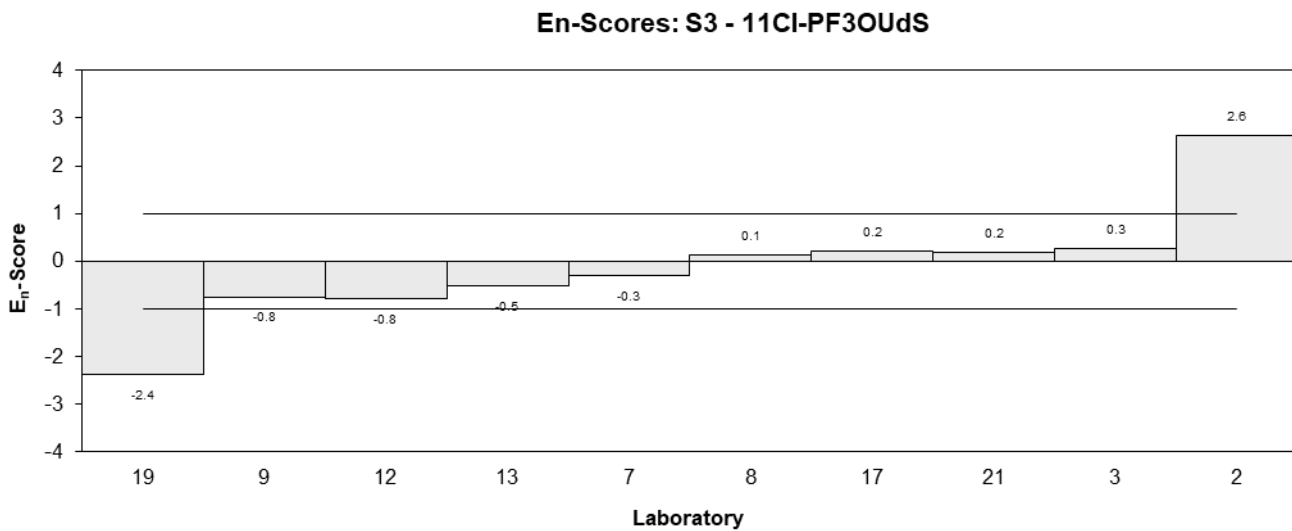
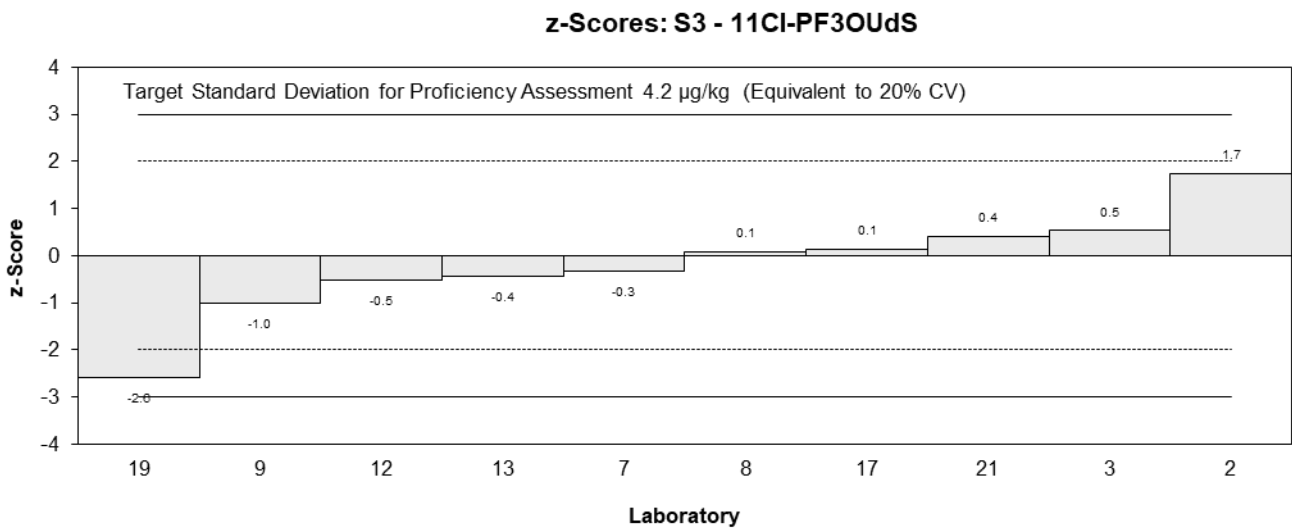
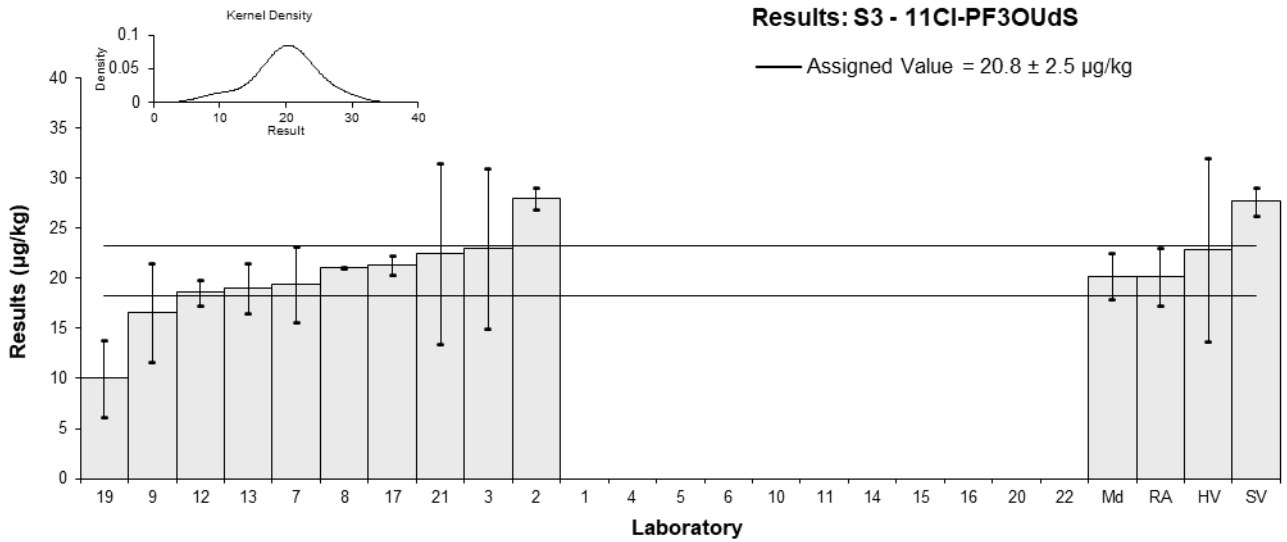


Figure 81

## 6 DISCUSSION OF RESULTS

### 6.1 Assigned Value

The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528.<sup>6</sup> The assigned values for all scored analytes were the robust averages of participants' results, after results less than 50% and greater than 150% of the robust average had been removed.<sup>3,4</sup> The calculation of the expanded uncertainty for the robust average is presented in Appendix 3, using Sample S1 PFHxS as an example.

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

No assigned values were set for Sample S3 EtFOSA as reported results were too variable. The variability may have been due to difficulties in the analysis caused by the matrix, analyte mass fraction level, properties of the analyte itself, or a combination of these factors. For this analyte without an assigned value, participants may still compare their results with the descriptive statistics and spiked value as presented in Section 5.

A comparison of the assigned values (or robust average if the analyte was not scored) and spiked values is presented in Table 87. For this study, the assigned values for scored analytes were within 61% to 100%, 87% to 115% and 75% to 105% of the spiked values for Samples S1, S2 and S3 respectively. These recoveries are similar to previous NMI PFAS in biota and food PT studies and provides good support for the assigned values.

Table 87 Comparison of Assigned Values (*Robust Averages*) and Spiked Values

Sample	Analyte	Assigned Value ( <i>Robust Average</i> ) (µg/kg)	Spiked Value (µg/kg)	Assigned Value ( <i>Robust Average</i> ) / Spiked Value (%)
S1 (Fish)	PFBS	1.13	1.41	80
	PFPeS	2.87	3.08	93
	PFHxS	5.65	6.24	91
	PFHxS (linear)	5.97	6.24	96
	PFHpS	1.28	1.34	96
	PFOS	1.60	1.79	89
	PFOS (linear)	1.61	1.79	90
	PFNS	1.59	1.80	88
	PFDS	1.60	1.80	89
	PFBA	6.20	6.57	94
	PFPeA	2.28	2.39	95
	PFHxA	6.21	6.54	95
	PFHpA	6.68	7.48	89
	PFOA	0.940	0.943	100
	PFNA	1.79	1.89	95
	PFDA	1.58	1.59	99
	PFUdA	0.722	0.756	96
PFDoA	3.10	3.70	84	

Sample	Analyte	Assigned Value ( <i>Robust Average</i> ) (µg/kg)	Spiked Value (µg/kg)	Assigned Value ( <i>Robust Average</i> ) / Spiked Value (%)
	PFTeDA	1.32	1.68	79
	PFOSA	4.69	5.39	87
	EtFOSA	2.85	4.67	61
	EtFOSAA	4.27	4.67	91
	8:2FTS	8.26	9.00	92
	GenX	12.7	14.1	90
	ADONA	15.6	17.6	89
	9Cl-PF3ONS	19.5	21.8	89
	11Cl-PF3OUdS	23.3	26.4	88
S2 (Spinach)	PFBS	4.02	4.62	87
	PFPeS	3.38	3.47	97
	PFHxS	2.26	2.20	103
	PFHxS (linear)	2.19	2.20	100
	PFHpS	1.70	1.76	97
	PFOS	2.56	2.50	102
	PFOS (linear)	2.54	2.50	102
	PFNS	3.42	3.55	96
	PFDS	2.52	2.68	94
	PFBA	12.1	10.7	113
	PFPeA	4.66	4.60	101
	PFHxA	5.28	4.61	115
	PFHpA	2.90	2.77	105
	PFOA	6.07	5.57	109
	PFNA	4.90	4.64	106
	PFDA	2.36	2.09	113
	PFUdA	4.06	3.73	109
	PFTTrDA	2.90	2.78	104
	PFOSA	12.1	12.3	98
	EtFOSA	6.7	7.46	90
	EtFOSAA	8.9	9.26	96
	6:2FTS	4.58	4.39	104
	GenX	8.51	8.40	101
	ADONA	13.9	15.8	88
9Cl-PF3ONS	18.5	19.0	97	
11Cl-PF3OUdS	20.7	22.6	92	
	PFBS	1.19	1.46	82

Sample	Analyte	Assigned Value ( <i>Robust Average</i> ) ( $\mu\text{g}/\text{kg}$ )	Spiked Value ( $\mu\text{g}/\text{kg}$ )	Assigned Value ( <i>Robust Average</i> ) / Spiked Value (%)
S3 (Bovine Liver)	PFPeS	2.89	3.22	90
	PFHxS	5.94	6.50	91
	PFHxS (linear)	5.96	6.50	92
	PFHpS	1.29	1.41	91
	PFOS	1.80	1.87	96
	PFOS (linear)	1.78	1.87	95
	PFNS	1.68	1.88	89
	PFDS	1.58	1.89	84
	PFBA	6.47	6.77	96
	PFPeA	2.27	2.44	93
	PFHxA	6.86	6.89	100
	PFHpA	7.34	7.84	94
	PFOA	1.00	0.951	105
	PFNA	1.84	1.96	94
	PFDA	1.60	1.64	98
	PFUdA	0.763	0.781	98
	PFDoA	3.55	3.92	91
	PFTeDA	1.52	1.76	86
	PFOSA	5.19	5.58	93
	EtFOSA	(4.0)	4.89	(82)
	EtFOSAA	4.52	4.89	92
	8:2FTS	9.05	9.39	96
	GenX	13.6	14.7	93
	ADONA	16.6	18.4	90
9CI-PF3ONS	19.5	22.8	86	
11CI-PF3OUdS	20.8	27.7	75	

## 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 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>8</sup>

Of 1168 numeric results reported for spiked analytes in this study, 1153 (99%) were reported with a measurement uncertainty. Laboratory 4 did not report any uncertainties; this participant reported that they were not accredited.

Laboratory 1 attached estimates of MU to all their reported 'less-than' results ( $< x$ ). An uncertainty expressed as a value should not be attached to a non-value result.<sup>9</sup>

Participants' procedures for estimating their uncertainty are presented in Table 4, with additional comments presented in Table 5. Two participants reported using the NATA GAG Estimating and Reporting MU as their guide; NATA no longer publishes this document.<sup>10</sup>

Laboratory 2 reported an uncertainty of '0.00' for Sample S2 PFOSA. Other than this result, the magnitudes of the MUs for analytes in this study were within the range 0.06% to 100% of the reported value. In general, an expanded uncertainty of less than 10% relative is likely to be unrealistically small for the routine analysis of PFAS, while over 50% is likely too large and not fit for purpose. Of the 1153 MUs, 800 (69%) were between 10% and 50% relative, 313 were less than 10% relative and 40 were greater than 50% relative.

Uncertainties associated with results returning an acceptable  $z$ -score but an unacceptable  $E_n$ -score may have been underestimated.

In some cases, results and/or uncertainties were reported with an inappropriate number of significant figures. Including too many significant figures may inaccurately reflect the precision of measurements. The recommended format is to write the uncertainty to no more than two significant figures and then to write the result with the corresponding number of decimal places. For example, instead of  $3.7318 \pm 1.3061 \mu\text{g}/\text{kg}$ , it is better to report this as  $3.7 \pm 1.3 \mu\text{g}/\text{kg}$ .<sup>9</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>7</sup> the between-laboratory CVs obtained in this study, and the target SDs (as PCVs) are presented for comparison in Table 88.

Table 88 Comparison of Thompson-Horwitz CVs, Between-Laboratory CVs, and Target SDs

Sample	Analyte	Assigned Value (Robust Average) ( $\mu\text{g}/\text{kg}$ )	Thompson-Horwitz CV <sup>a</sup> (%)	Between-Laboratory CV <sup>b</sup> (%)	Target SD (as PCV) (%)
S1 (Fish)	PFBS	1.13	22	17	20
	PFPeS	2.87	22	19	20
	PFHxS	5.65	22	17	20
	PFHxS (linear)	5.97	22	19	20
	PFHpS	1.28	22	14	20
	PFOS	1.60	22	15	20
	PFOS (linear)	1.61	22	13	20
	PFNS	1.59	22	12	20
	PFDS	1.60	22	20	20
	PFBA	6.20	22	18	20
	PFPeA	2.28	22	15	20
	PFHxA	6.21	22	16	20
	PFHpA	6.68	22	19	20
	PFOA	0.940	22	14	20
	PFNA	1.79	22	19	20
	PFDA	1.58	22	16	20
	PFUdA	0.722	22	13	20
PFDoA	3.10	22	18	20	

Sample	Analyte	Assigned Value (Robust Average) (µg/kg)	Thompson-Horwitz CV <sup>a</sup> (%)	Between-Laboratory CV <sup>b</sup> (%)	Target SD (as PCV) (%)
	PFTeDA	1.32	22	24	20
	PFOSA	4.69	22	15	20
	EtFOSA	2.85	22	21	20
	EtFOSAA	4.27	22	26	20
	8:2FTS	8.26	22	15	20
	GenX	12.7	22	25	20
	ADONA	15.6	22	26	20
	9Cl-PF3ONS	19.5	22	19	20
	11Cl-PF3OUdS	23.3	22	27	20
S2 (Spinach)	PFBS	4.02	22	14	20
	PFPeS	3.38	22	17	20
	PFHxS	2.26	22	21	20
	PFHxS (linear)	2.19	22	18	20
	PFHpS	1.70	22	17	20
	PFOS	2.56	22	17	20
	PFOS (linear)	2.54	22	16	20
	PFNS	3.42	22	18	20
	PFDS	2.52	22	18	20
	PFBA	12.1	22	17	20
	PFPeA	4.66	22	17	20
	PFHxA	5.28	22	13	20
	PFHpA	2.90	22	16	20
	PFOA	6.07	22	15	20
	PFNA	4.90	22	15	20
	PFDA	2.36	22	18	20
	PFUdA	4.06	22	14	20
	PFTrDA	2.90	22	24	20
	PFOSA	12.1	22	14	20
	EtFOSA	6.7	22	31	20
	EtFOSAA	8.9	22	14	20
	6:2FTS	4.58	22	13	20
	GenX	8.51	22	6.6	20
ADONA	13.9	22	23	20	
9Cl-PF3ONS	18.5	22	20	20	
11Cl-PF3OUdS	20.7	22	21	20	
	PFBS	1.19	22	14	20



Sample	Analyte	Assigned Value (Robust Average) (µg/kg)	Thompson-Horwitz CV <sup>a</sup> (%)	Between-Laboratory CV <sup>b</sup> (%)	Target SD (as PCV) (%)
S3 (Bovine Liver)	PFPeS	2.89	22	18	20
	PFHxS	5.94	22	11	20
	PFHxS (linear)	5.96	22	12	20
	PFHpS	1.29	22	18	20
	PFOS	1.80	22	16	20
	PFOS (linear)	1.78	22	17	20
	PFNS	1.68	22	16	20
	PFDS	1.58	22	21	20
	PFBA	6.47	22	16	20
	PFPeA	2.27	22	14	20
	PFHxA	6.86	22	12	20
	PFHpA	7.34	22	14	20
	PFOA	1.00	22	11	20
	PFNA	1.84	22	12	20
	PFDA	1.60	22	10	20
	PFUdA	0.763	22	7.5	20
	PFDoA	3.55	22	7.1	20
	PFTeDA	1.52	22	13	20
	PFOSA	5.19	22	16	20
	EtFOSA	(4.0)	22	47	Not Set
	EtFOSAA	4.52	22	17	20
	8:2FTS	9.05	22	11	20
	GenX	13.6	22	8.4	20
	ADONA	16.6	22	17	20
9Cl-PF3ONS	19.5	22	17	20	
11Cl-PF3OUdS	20.8	22	14	20	

<sup>a</sup> Calculated from the assigned value (*robust average*).

<sup>b</sup> Robust between-laboratory CV with outliers removed, if applicable. Shaded cells are between-laboratory CVs for analytes which were higher than both the target SD and the Thompson-Horwitz CV.

To account for possible low bias in the consensus value due to laboratories using inefficient analytical or extraction techniques, two  $z$ -scores were adjusted across Sample S1 PFBS and EtFOSA. A maximum acceptable result was set as the spiked value plus two target SDs of the spiked value. Results lower than the maximum acceptable result but with a  $z$ -score greater than 2.0 had their  $z$ -score adjusted to 2.0. This ensured that any participants reporting results close to the spiked value were not penalised.  $z$ -Scores for results greater than the maximum acceptable result, and  $z$ -scores less than 2.0, were left unaltered.

Of 1161 results for which  $z$ -scores were calculated, 1091 (94%) returned  $|z| \leq 2.0$ , indicating an acceptable performance.

Fourteen participants analysed all three matrices. Laboratory **21** reported numeric results for all 79 scored analytes and returned acceptable z-scores across all analytes. Three other participants analysing all samples received acceptable z-scores for all reported results that were scored: **2** (75), **8** (74) and **1** (44).

Six participants analysed Sample S1 fish and Sample S2 spinach only, and one participant analysed Sample S1 fish only. Of these participants, Laboratory **11** received acceptable z-scores for all reported results that were scored (34).

The dispersal of participants' z-scores is presented graphically by laboratory in Figure 82 and by analyte in Figures 83 to 85.

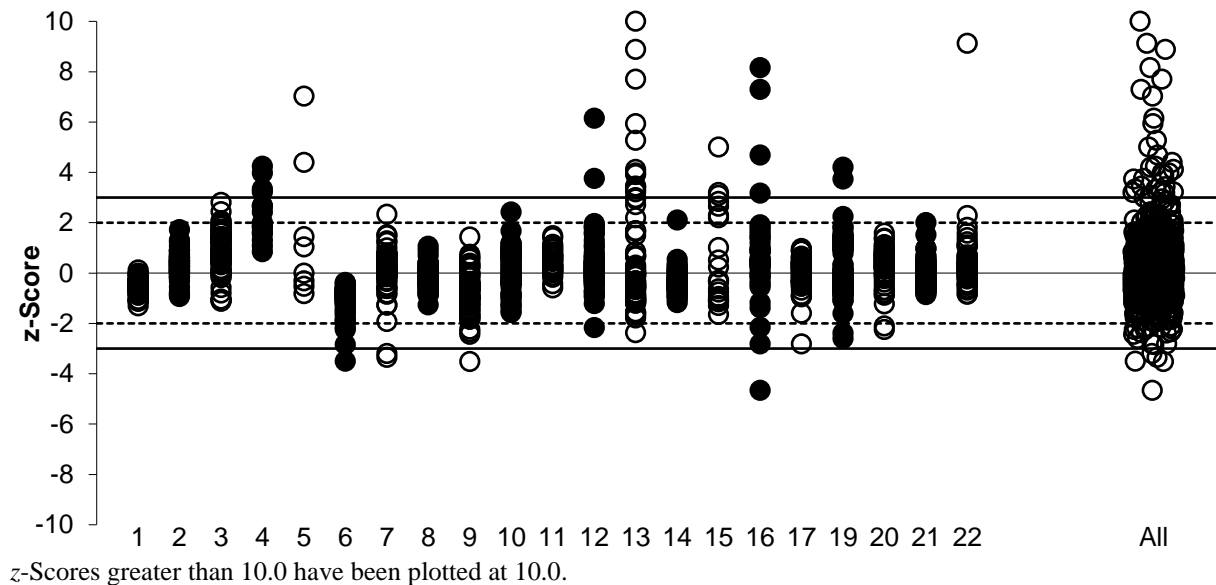


Figure 82 z-Score Dispersal by Laboratory

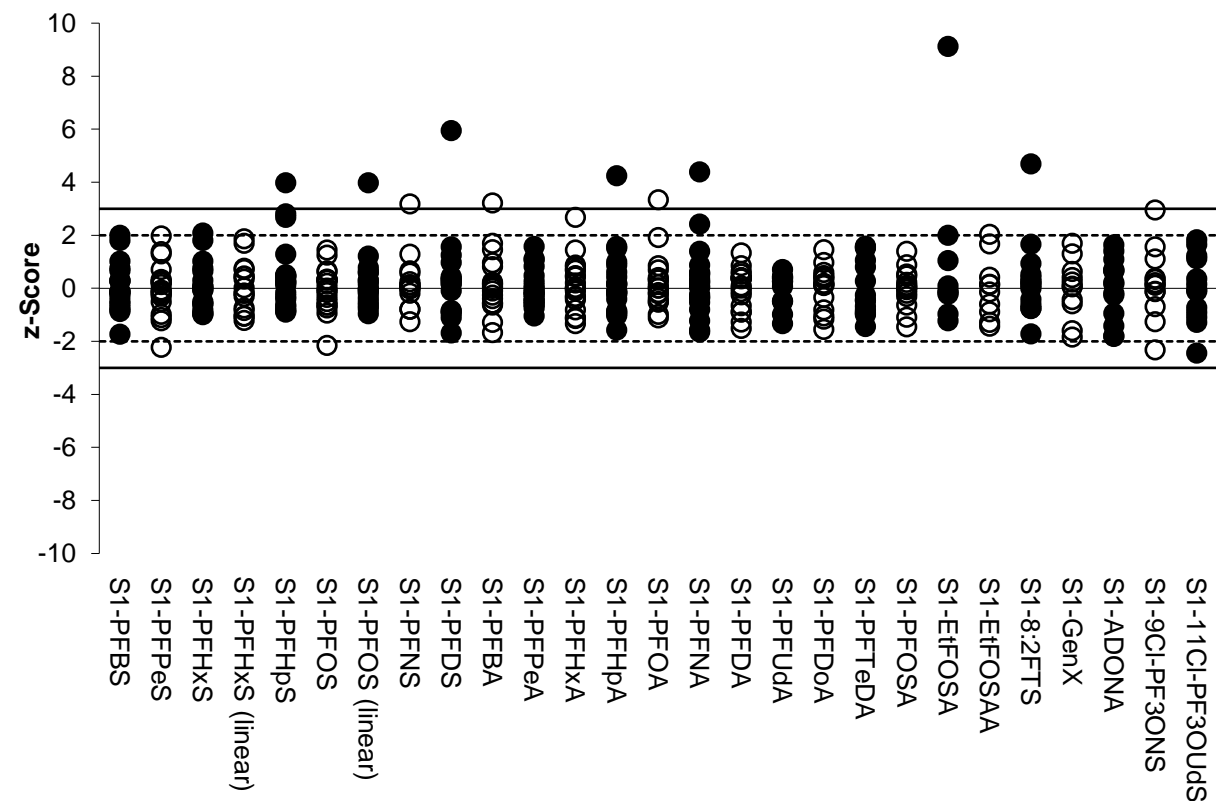
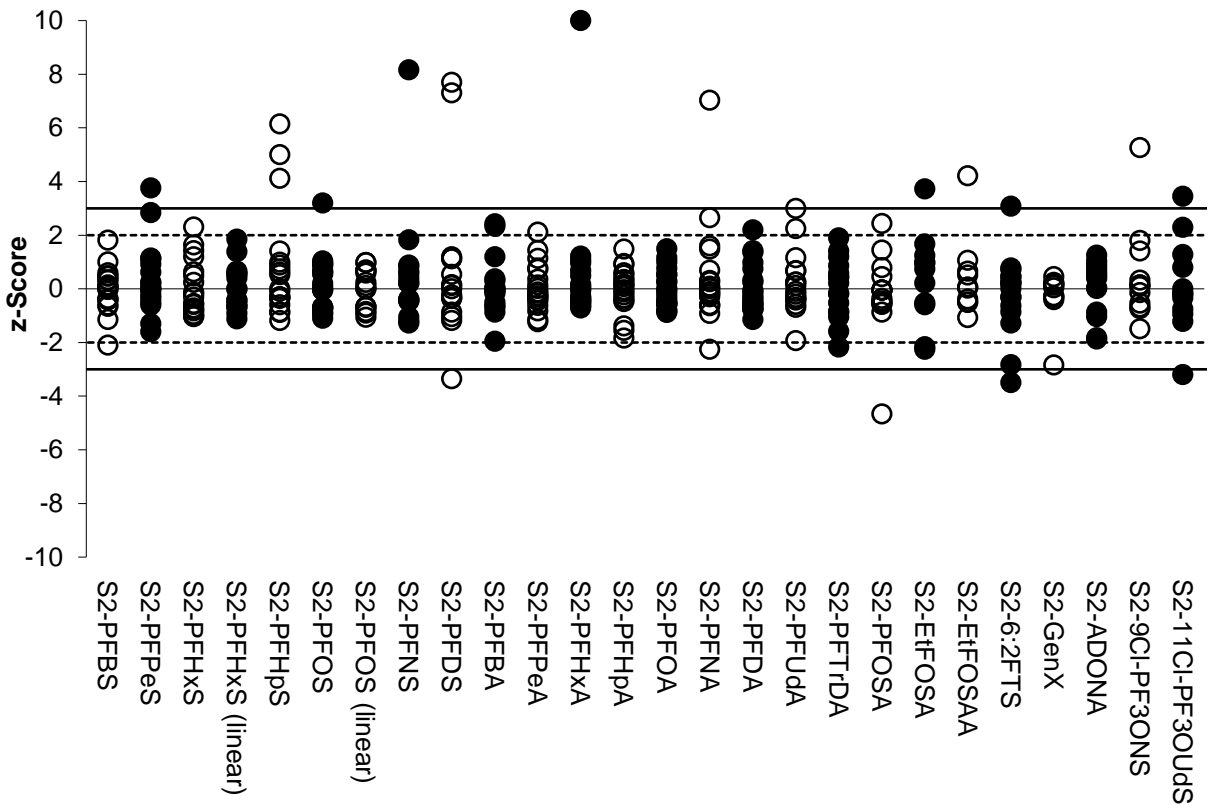


Figure 83 z-Score Dispersal by Analyte for Sample S1 Fish



z-Scores greater than 10.0 have been plotted at 10.0.

Figure 84 z-Score Dispersal by Analyte for Sample S2 Spinach

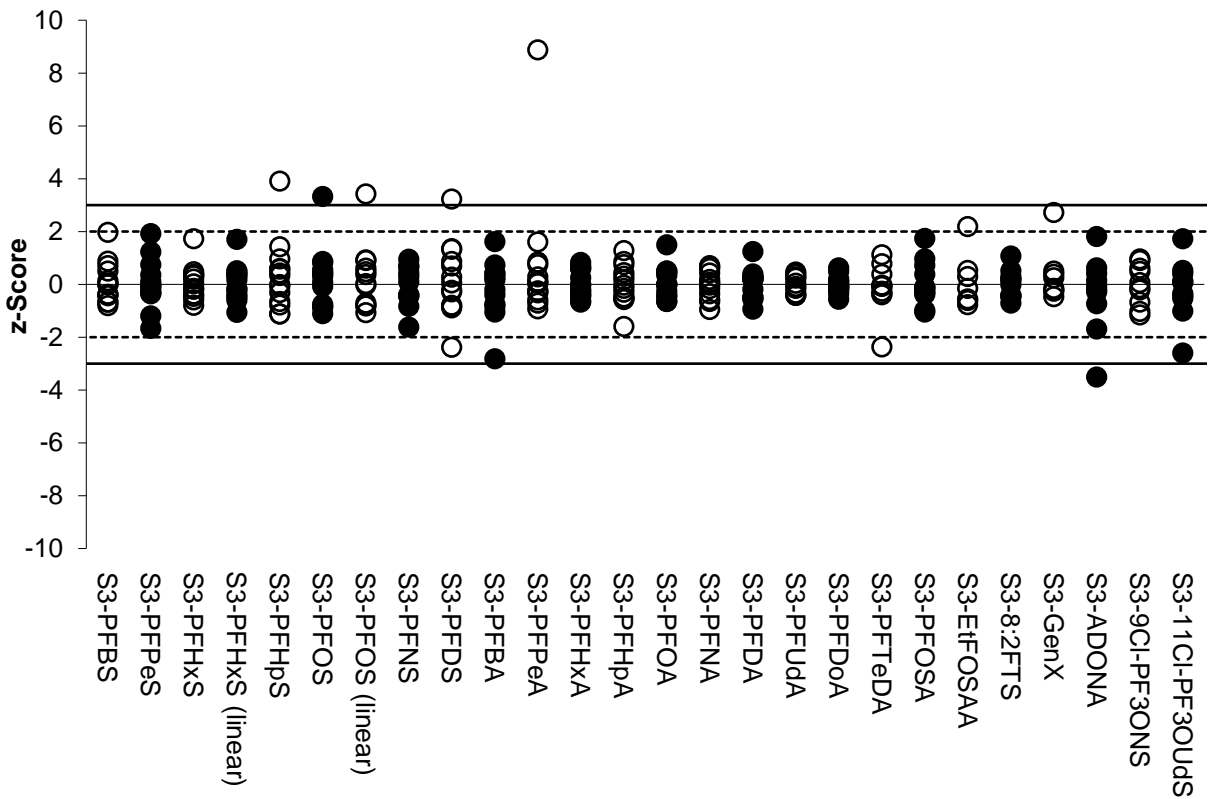


Figure 85 z-Score Dispersal by Analyte for Sample S3 Bovine Liver

For this study, analytes in Samples S1 fish and S3 bovine liver were spiked at similar levels. Scatter plots of z-scores for analytes scored in both Samples S1 and S3 are presented in

Figures 86 to 111. Scores are predominantly in the upper right and lower left quadrants, indicating that laboratory bias was the major contributor to the variability of results, and that participants were generally biased in a similar manner for both fish and bovine liver analysis. Points close to the diagonal axis demonstrate excellent repeatability, while points close to the zero demonstrate excellent repeatability and accuracy.

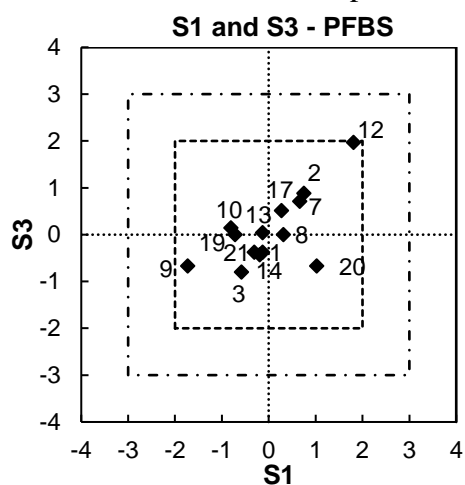


Figure 86 z-Score Scatter Plot – PFBS

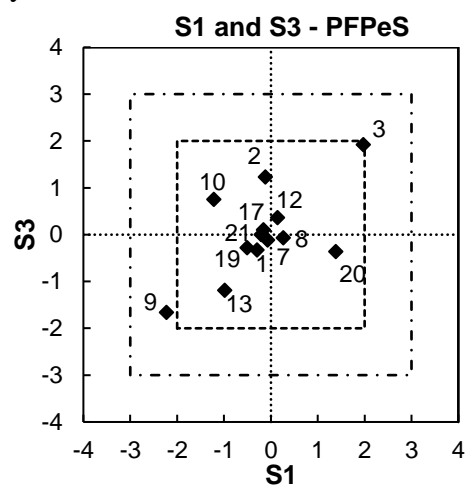


Figure 87 z-Score Scatter Plot – PFPeS

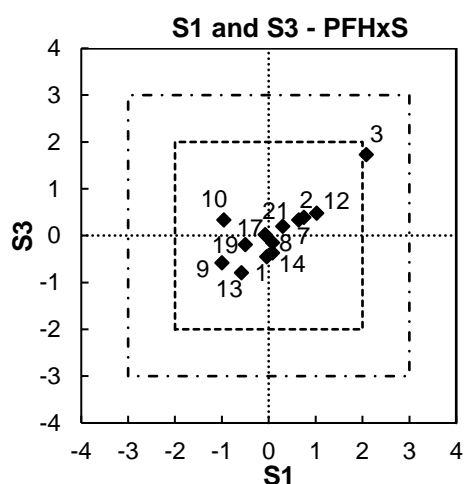


Figure 88 z-Score Scatter Plot – PFHxS

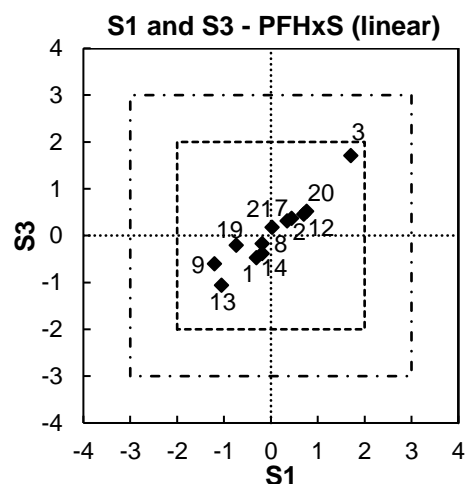


Figure 89 z-Score Scatter Plot – PFHxS (linear)

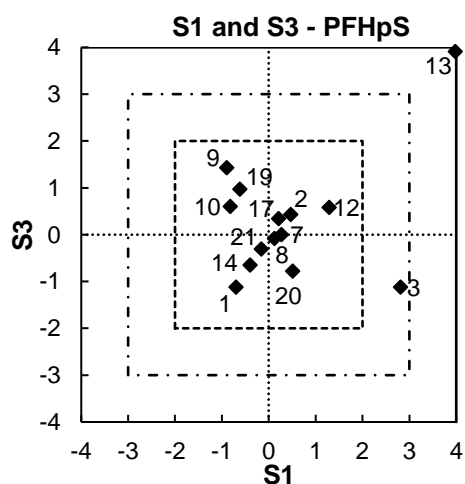


Figure 90 z-Score Scatter Plot – PFHpS

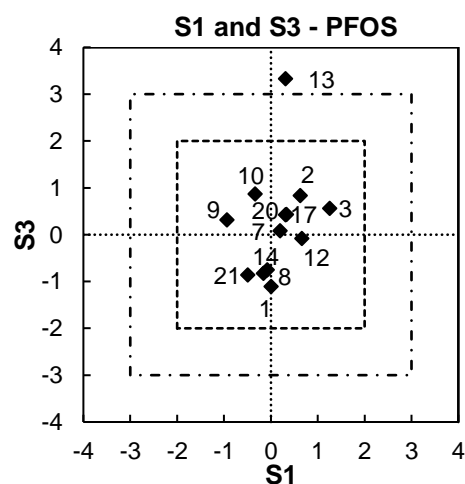


Figure 91 z-Score Scatter Plot – PFOS

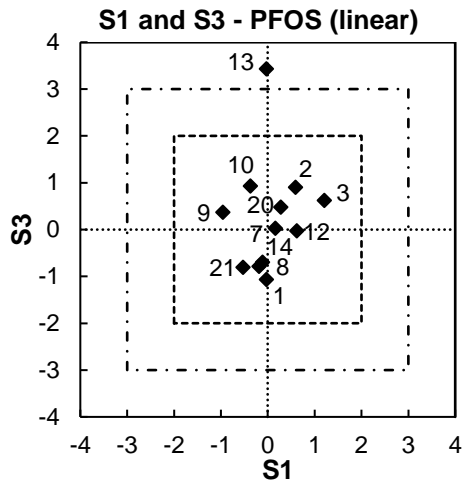


Figure 92 z-Score Scatter Plot – PFOS (linear)

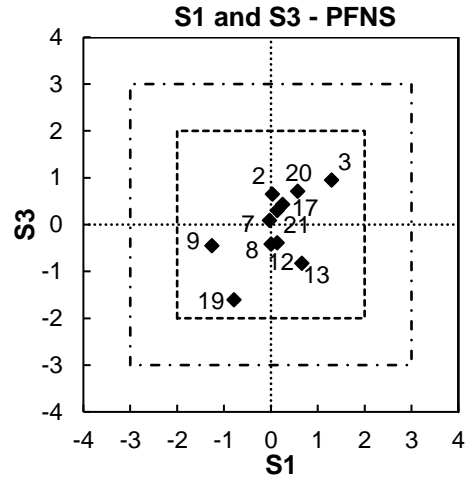
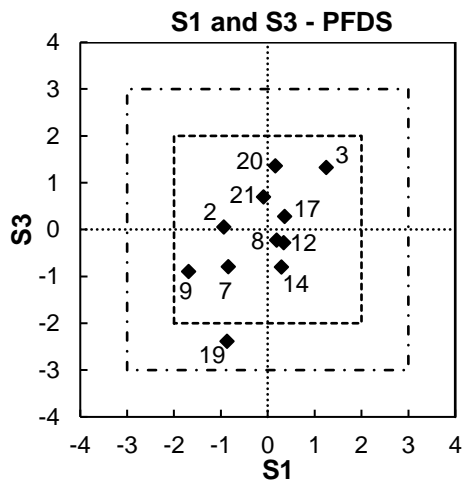


Figure 93 z-Score Scatter Plot – PFNS



Laboratory 13 is off-scale.

Figure 94 z-Score Scatter Plot – PFDS

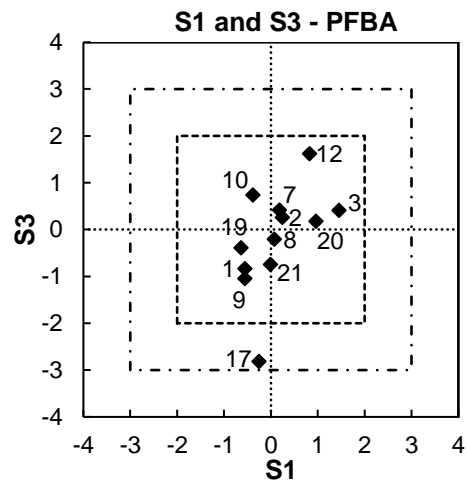
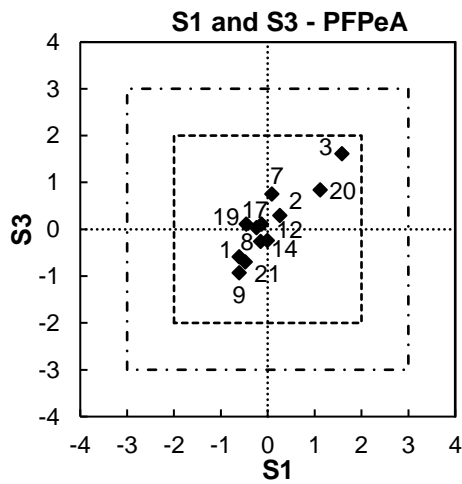


Figure 95 z-Score Scatter Plot – PFBA



Laboratory 13 is off-scale.

Figure 96 z-Score Scatter Plot – PFPeA

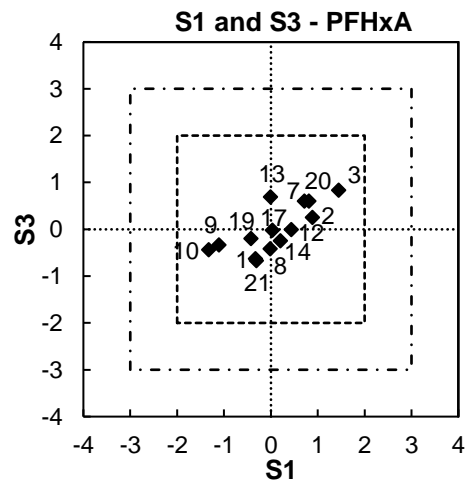


Figure 97 z-Score Scatter Plot – PFHxA

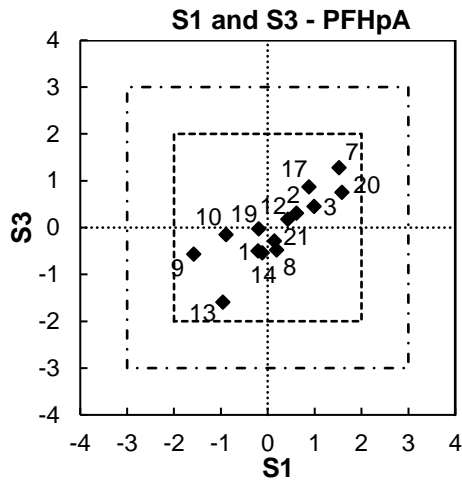


Figure 98 z-Score Scatter Plot – PFHpA

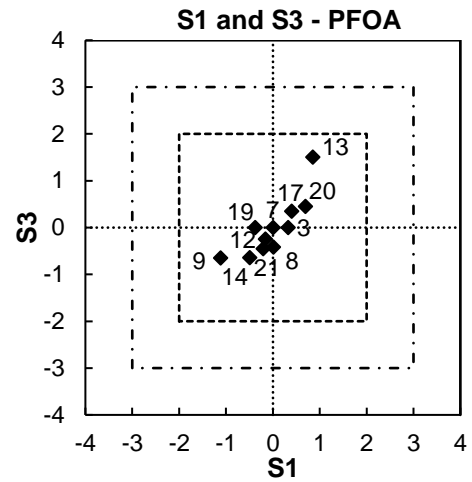


Figure 99 z-Score Scatter Plot – PFOA

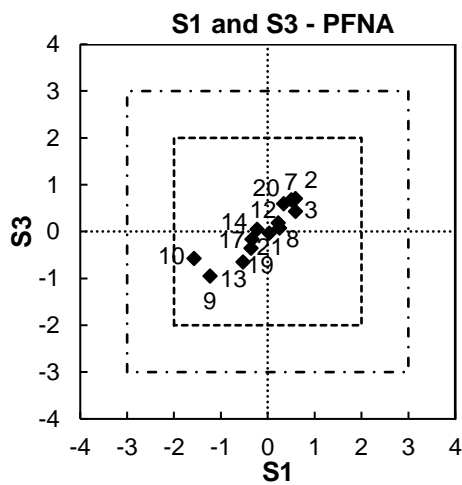


Figure 100 z-Score Scatter Plot – PFNA

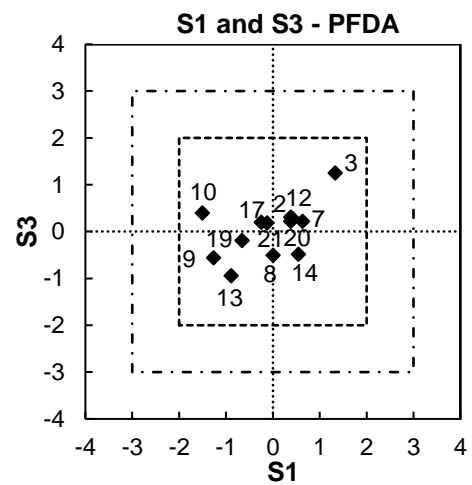


Figure 101 z-Score Scatter Plot – PFDA

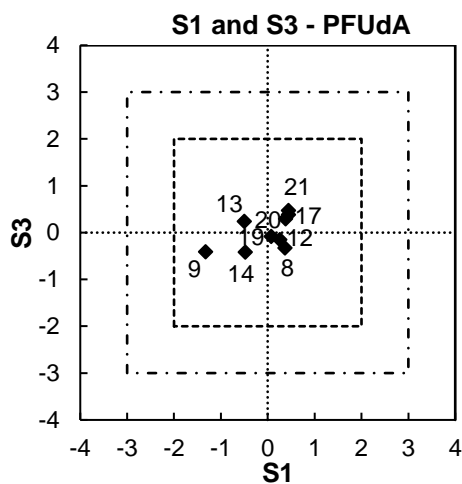


Figure 102 z-Score Scatter Plot – PFUdA

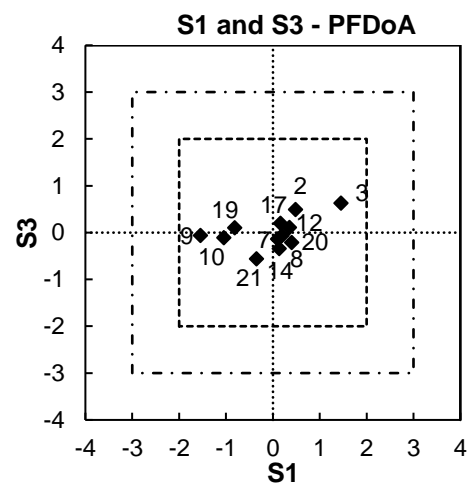


Figure 103 z-Score Scatter Plot – PFDoA

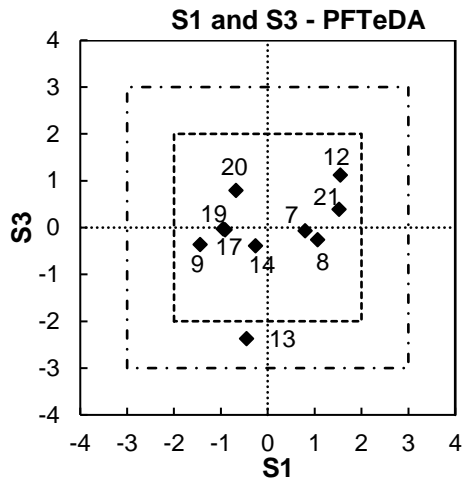


Figure 104 z-Score Scatter Plot – PFTeDA

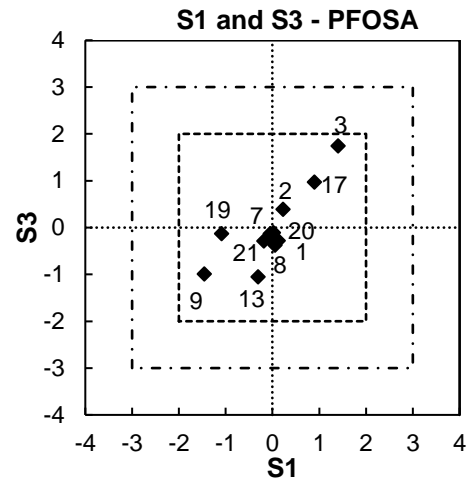


Figure 105 z-Score Scatter Plot – PFOSA

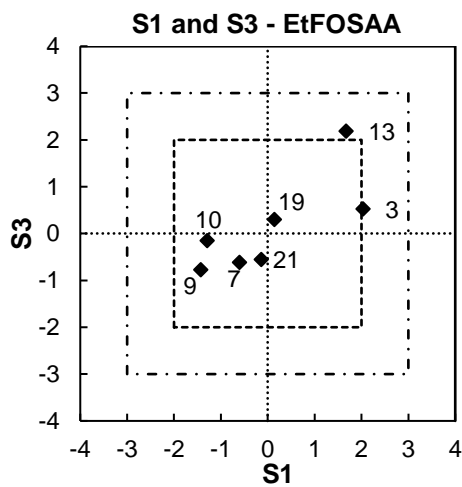


Figure 106 z-Score Scatter Plot – EtFOSAA

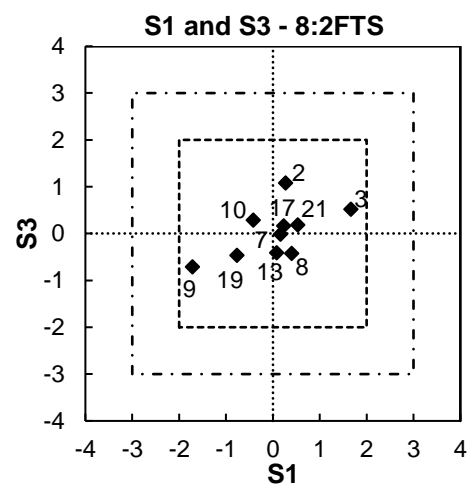


Figure 107 z-Score Scatter Plot – 8:2FTS

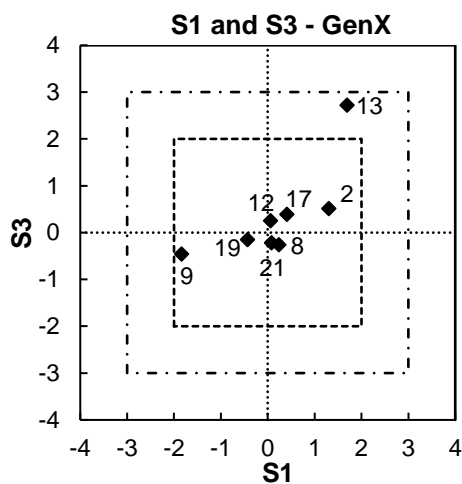


Figure 108 z-Score Scatter Plot – GenX

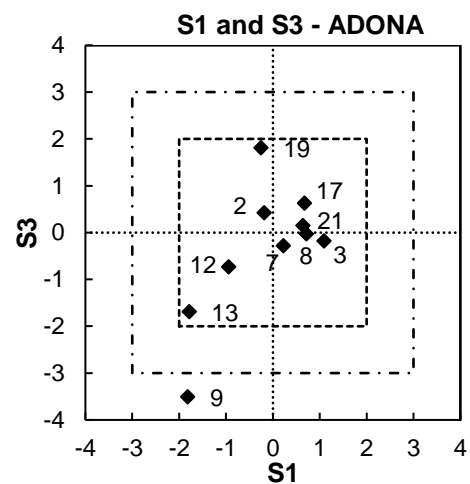


Figure 109 z-Score Scatter Plot – ADONA

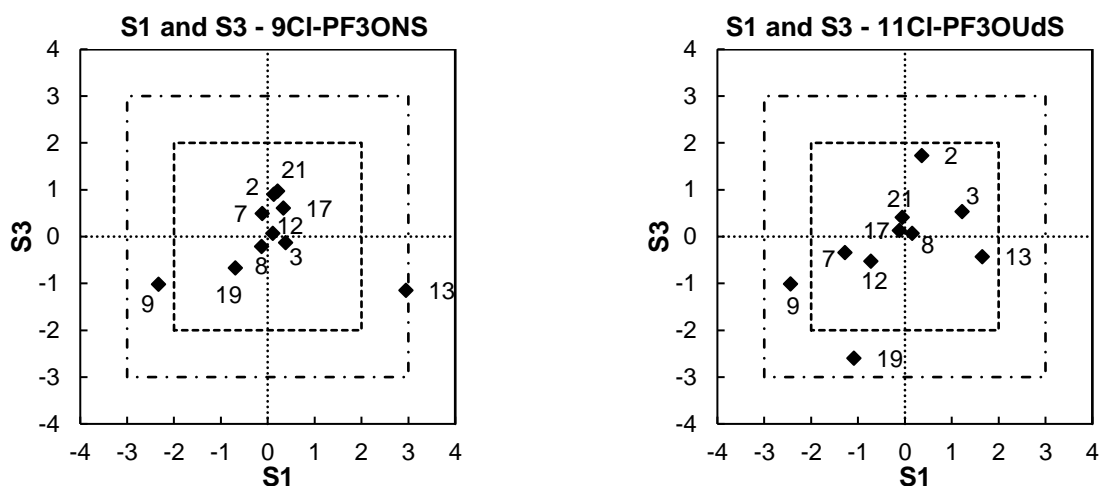


Figure 110  $z$ -Score Scatter Plot – 9CI-PF3ONS Figure 111  $z$ -Score Scatter Plot – 11CI-PF3OUdS

#### 6.4 $E_n$ -Score

$E_n$ -Scores can be interpreted in conjunction with  $z$ -scores, as an unacceptable  $E_n$ -score can either be caused by issues with measurement, or uncertainty, or both. If a participant did not report any uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the  $E_n$ -score. For results whose  $z$ -scores were adjusted as discussed in Section 6.3  $z$ -Score, no  $E_n$ -score has been calculated.

Of 1159 results for which  $E_n$ -scores were calculated, 959 (83%) 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 acceptable  $E_n$ -scores for all 79 analytes of interest in this study. Laboratory **21** reported numeric results for all analytes, however as one of their result's  $z$ -score was adjusted as described above,  $E_n$ -scores were only calculated for 78 results; this participant returned acceptable  $E_n$ -scores for all 78 results.

Three other participants analysing all samples received acceptable  $E_n$ -scores for all reported results that were scored: **3** (71), **14** (46) and **1** (44).

Of participants analysing a subset of samples, Laboratory **11** received acceptable  $E_n$ -scores for all results that were scored (34).

Laboratory **4** returned unacceptable  $E_n$ -scores across all results that were scored (14).

The dispersal of participants'  $E_n$ -scores is presented graphically in Figure 112.



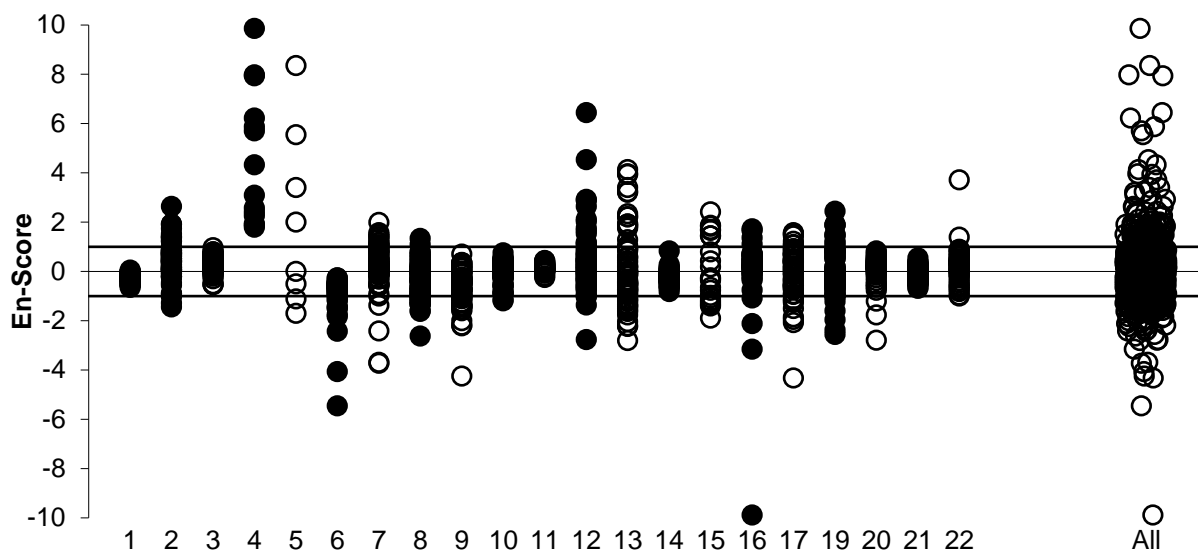


Figure 112  $E_n$ -Score Dispersal by Laboratory

### 6.5 Range of PFAS Analysed by Participants

Participants were provided with a list of analytes that may have been spiked into the test samples (Table 1). Of these, 27 different analytes were spiked for this study, with 25 analytes being spiked into Samples S1 and S3, and 24 analytes being spiked into Sample S2. For PFHxS and PFOS, participants were requested to report for both linear isomers only and total value, however both samples were spiked with linear isomers only. Participants were not required to test for all potential analytes and were requested to report 'NT' (for 'Not Tested') for any analyte they did not test the samples for.

A summary of participants' testing of the spiked analytes is presented in Table 89.

Laboratories **2, 3, 6, 9, 19, 21** and **22** analysed all spiked analytes. All participants tested for at least one spiked analyte, with the proportion of PFAS being analysed by each participant ranging from 14% to 100%.

Out of the spiked analytes in this study, PFOA and PFNA were tested for by the highest proportion of participants (100% for both). In general, perfluoroalkyl acids were very well represented by participants, with the overall proportion of analysis by participants being 87% and 90% for perfluoroalkane sulfonates and perfluoroalkyl carboxylic acids respectively. A lower proportion of participants analysed the perfluoroalkane sulfonamide (PFOSA), perfluoroalkane sulfonamido (EtFOSA and EtFOSAA), fluorotelomer (6:2FTS and 8:2FTS) and PFAS replacement compounds (GenX, ADONA, 9Cl-PF3ONS and 11Cl-PF3OUdS), being 71%, 52%, 78% and 62% respectively.

Table 89 Summary of PFAS Analysed by Participants<sup>a</sup>

Lab. Code Analyte	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	19	20	21	22	Proportion of Participants (%)	
PFBS	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	95
PFPeS	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	90
PFHxS	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	NT	✓	✓	86
PFHxS (linear)	✓	✓	✓	✓	NT	✓	✓	✓	✓	NT	✓	✓	✓	✓	NT	NT	✓	✓	✓	✓	✓	✓	81
PFHpS	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	95
PFOS	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	90
PFOS (linear)	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	NT	NT	✓	✓	✓	✓	✓	✓	86
PFNS	✓	✓	✓	NT	NT	✓	✓	✓	✓	NT	✓	✓	✓	NT	NT	✓	✓	✓	✓	✓	✓	✓	76
PFDS	✓ <sup>d</sup>	✓	✓	NT	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	86
PFBA	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	NT	NT	✓ <sup>e</sup>	✓	✓	✓	✓	✓	✓	✓	86
PFPeA	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	95
PFHxA	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	95
PFHpA	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	95
PFOA	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	100
PFNA	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	100
PFDA	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	95
PFUdA	✓	✓	✓	NT	NT	✓	✓	✓	✓	✓	NT	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	81
PFTTrDA <sup>c</sup>	NT	✓	✓		NT	✓	✓	✓	✓	✓	✓	✓	✓	NT	NT	✓	✓	✓	✓	✓	✓	✓	80
PFDoA <sup>b</sup>	✓	✓	✓	NT	NT	✓	✓	✓	✓	✓	NT	✓	NT	✓	NT	✓	✓	✓	✓	✓	✓	✓	76
PFTeDA <sup>b</sup>	✓	✓	✓	NT	NT	✓	✓	✓	✓	✓	✓	✓	✓	✓	NT	✓	✓	✓	✓	✓	✓	✓	86
PFOSA	✓	✓	✓	NT	NT	✓	✓	✓	✓	✓	NT	NT	✓	NT	NT	✓	✓	✓	✓	✓	✓	✓	71

Lab. Code Analyte	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	19	20	21	22	Proportion of Participants (%)	
EtFOSA	✓	✓	✓	NT	NT	✓	NT	NT	✓	✓	NT	NT	✓	NT	NT	NT	NT	✓	✓	✓	✓	52	
EtFOSAA	✓	✓	✓	NT	NT	✓	✓	NT	✓	✓	NT	NT	✓	NT	NT	NT	NT	✓	NT	✓	✓	52	
6:2FTS <sup>c</sup>	✓	✓	✓		NT	✓	✓	✓	✓	✓	✓	NT	✓	NT	✓	✓	✓	✓	✓	NT	✓	✓	80
8:2FTS <sup>b</sup>	✓	✓	✓	NT	NT	✓	✓	✓	✓	✓	✓	NT	✓	NT	✓	✓	✓	✓	✓	NT	✓	✓	76
GenX	NT	✓	✓	NT	NT	✓	NT	✓	✓	NT	✓	✓	✓	NT	NT	NT	✓	✓	NT	✓	✓	57	
ADONA	NT	✓	✓	✓	NT	✓	✓	✓	✓	NT	NT	✓	✓	NT	NT	✓	✓	✓	✓	NT	✓	✓	67
9Cl-PF3ONS	NT	✓	✓	✓	NT	✓	✓	✓	✓	NT	NT	✓	✓	NT	NT	NT	✓	✓	NT	✓	✓	62	
11Cl-PF3OUdS	NT	✓	✓	✓	NT	✓	✓	✓	✓	NT	NT	✓	✓	NT	NT	NT	✓	✓	NT	✓	✓	62	
Proportion of Analytes (%)	83	100	100	56	14	100	93	93	100	79	66	83	93	55	48	76	93	100	72	100	100		

<sup>a</sup> Shaded cells indicate that the participant did not enrol for and was not supplied a sample containing that analyte; proportions have been adjusted accordingly.

<sup>b</sup> Spiked into Samples S1 and S3 only.

<sup>c</sup> Spiked into Sample S2 only.

<sup>d</sup> Laboratory 1 reported analysing for PFDS in Samples S1 and S3, but not Sample S2.

<sup>e</sup> Laboratory 15 reported analysing for PFBA in Sample S1, but not Sample S2.

## 6.6 PFAS in Food Trigger Points

There are currently no maximum regulatory limits in Australia for PFAS contaminants in food. However, Food Standards Australia New Zealand (FSANZ) has proposed non-regulatory ‘trigger points’ in a variety of food products for 3 common PFAS compounds, namely PFHxS, PFOS and PFOA, based on food consumption rates and set tolerable daily intakes for these analytes.<sup>11</sup> Where an analyte is found to be exceeding the corresponding trigger point, this may indicate that further investigation is required.

The assigned values in this study and relevant FSANZ trigger points are given in Table 90.

For Sample S1, PFHxS was spiked to be above the trigger point, and while the assigned value was also above the trigger point, the assigned value’s uncertainty spanned the trigger point. For PFOS and PFOA, both the spiked and assigned values were below the relevant trigger point.

For Sample S2, PFHxS and PFOS were spiked above, and PFOA was spiked below, the relevant trigger points. The assigned values were in agreement with the spiked values relative to the trigger points across all analytes.

With the very high trigger points for mammalian offal, Sample S3 PFHxS, PFOS and PFOA were all well below the relevant trigger points for both the spiked values and assigned values.

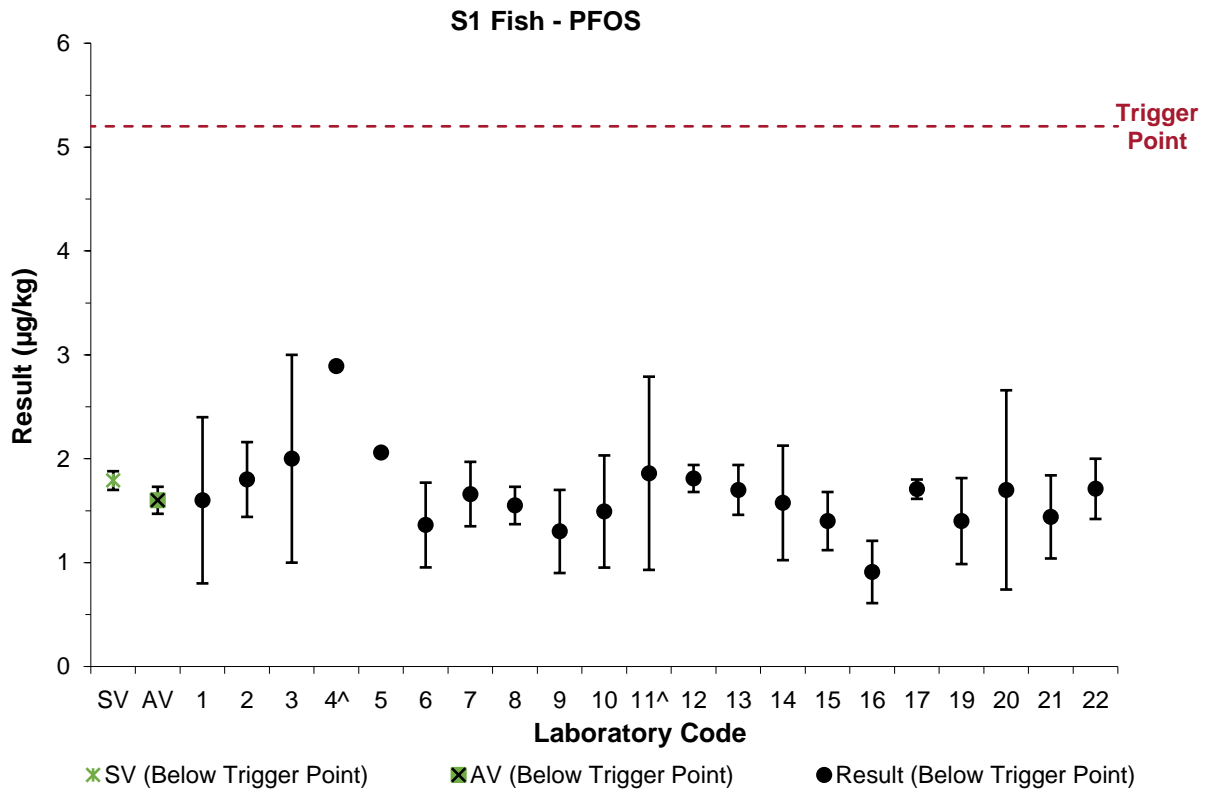
Table 90 Spiked Values, Assigned Values and FSANZ Trigger Points for PFHxS, PFOS and PFOA<sup>11</sup>

Sample	Matrix	Classification	PFHxS (µg/kg)			PFOS (µg/kg)			PFOA (µg/kg)		
			Spiked Value	Assigned Value	Trigger Point	Spiked Value	Assigned Value	Trigger Point	Spiked Value	Assigned Value	Trigger Point
S1	Fish	Finfish	6.24 ± 0.31	5.65 ± 0.58	5.2	1.79 ± 0.09	1.60 ± 0.13	5.2	0.943 ± 0.047	0.940 ± 0.079	41
S2	Spinach	Vegetables	2.20 ± 0.11	2.26 ± 0.28	1.1	2.50 ± 0.12	2.56 ± 0.26	1.1	5.57 ± 0.28	6.07 ± 0.52	8.8
S3	Bovine Liver	Offal mammalian	6.50 ± 0.32	5.94 ± 0.44	96	1.87 ± 0.09	1.80 ± 0.21	96	0.951 ± 0.048	1.00 ± 0.08	765

Figures 113 to 120 show comparisons of the spiked values (SV), assigned values (AV), participants' results, and FSANZ trigger points for these analytes (except for Sample S1 PFHxS). Where no numeric result was reported, or if a 'less-than' value ( $< x$ ) was reported, these results have been excluded from consideration. Where a participant did not report the total value of an analyte, but did report a linear isomers only value, the linear value has been plotted.

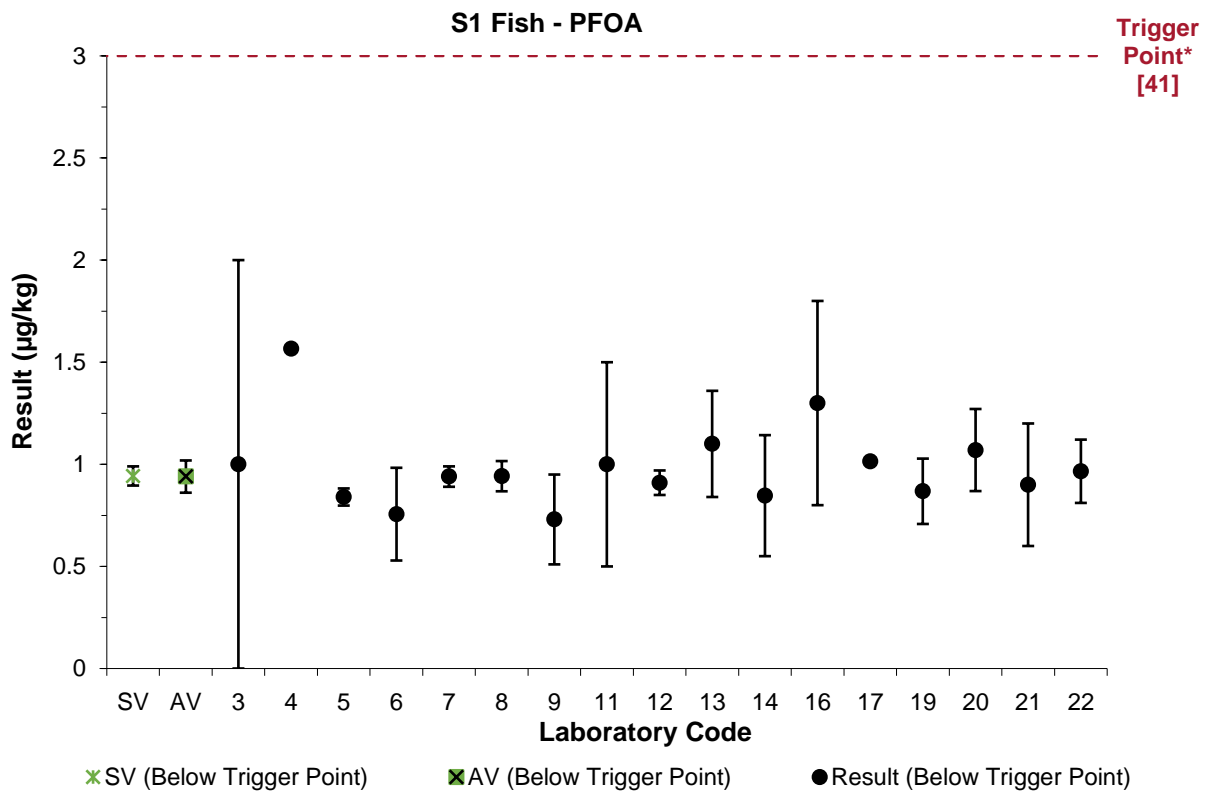
Of the results for the eight analytes where both the spiked and assigned values were greater than or less than the trigger point (inclusive of uncertainty), 131 of a total 138 results (95%) were correctly above or below the trigger point. The remaining seven results were also correctly above or below the trigger point, however with uncertainty spanning the trigger point.

Results from Laboratories **7, 8, 9, 12, 14, 17, 20** and **21** correctly reflected whether the analyte mass fractions (inclusive of uncertainties) were above or below the trigger points for all eight assessed analytes.



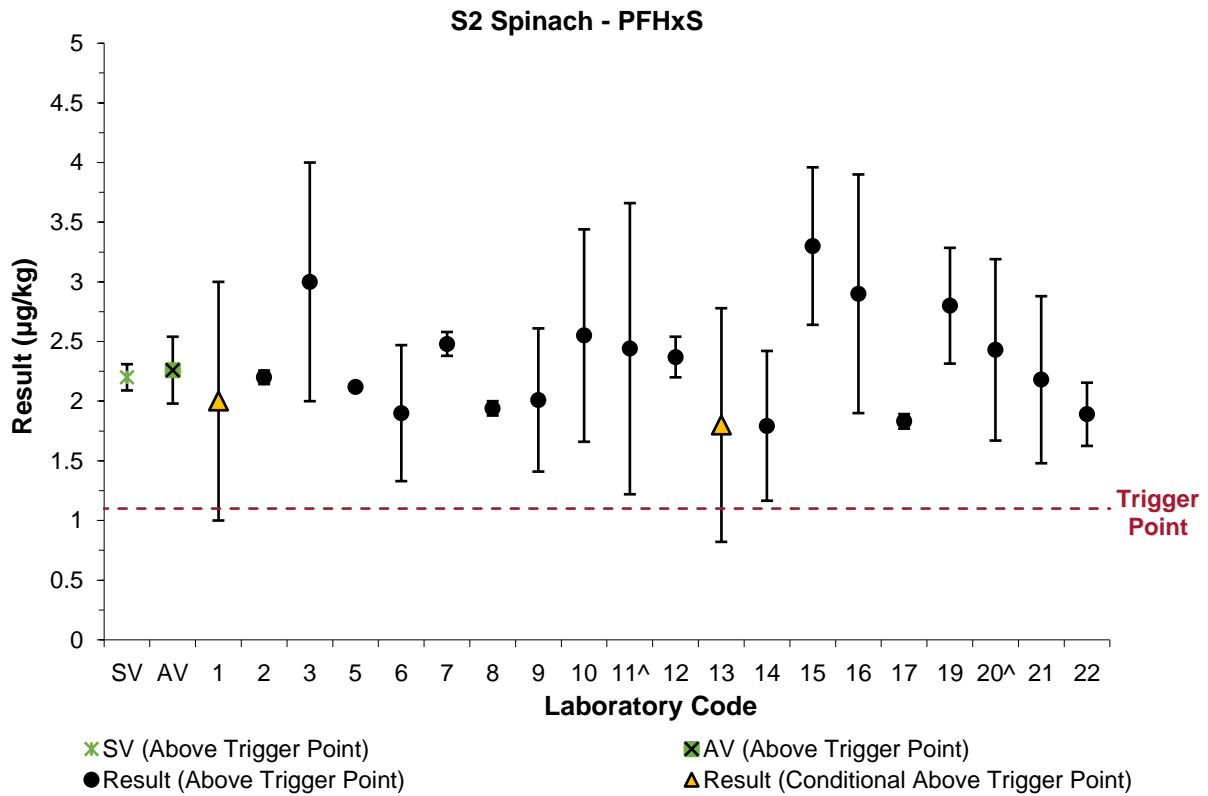
<sup>^</sup> Result for linear isomers only has been plotted.

Figure 113 Sample S1 Fish PFOS Spiked and Assigned Values, Participant Results and Trigger Point



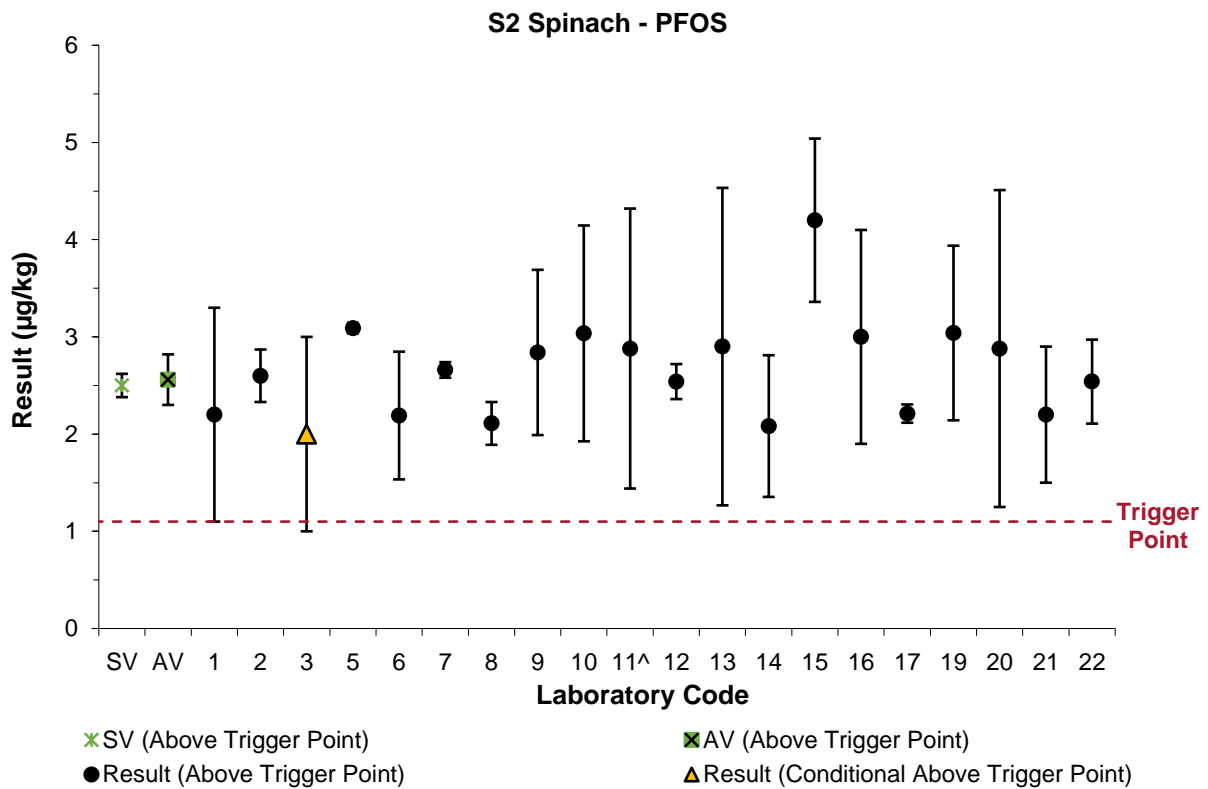
\* The trigger point has been scaled to fit on the chart; actual value in brackets.

Figure 114 Sample S1 Fish PFOA Spiked and Assigned Values, Participant Results and Trigger Point



^ Result for linear isomers only has been plotted.

Figure 115 Sample S2 Spinach PFHxS Spiked and Assigned Values, Participant Results and Trigger Point



^ Result for linear isomers only has been plotted.

Figure 116 Sample S2 Spinach PFOS Spiked and Assigned Values, Participant Results and Trigger Point

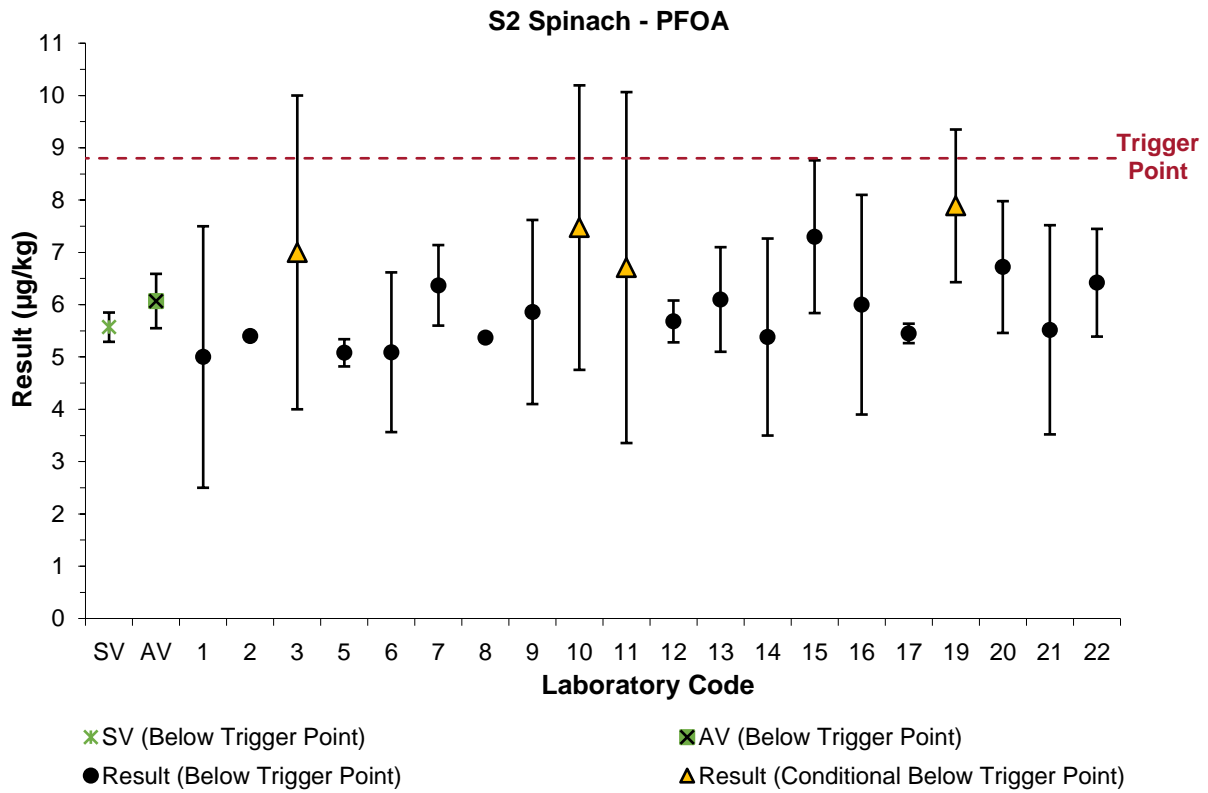
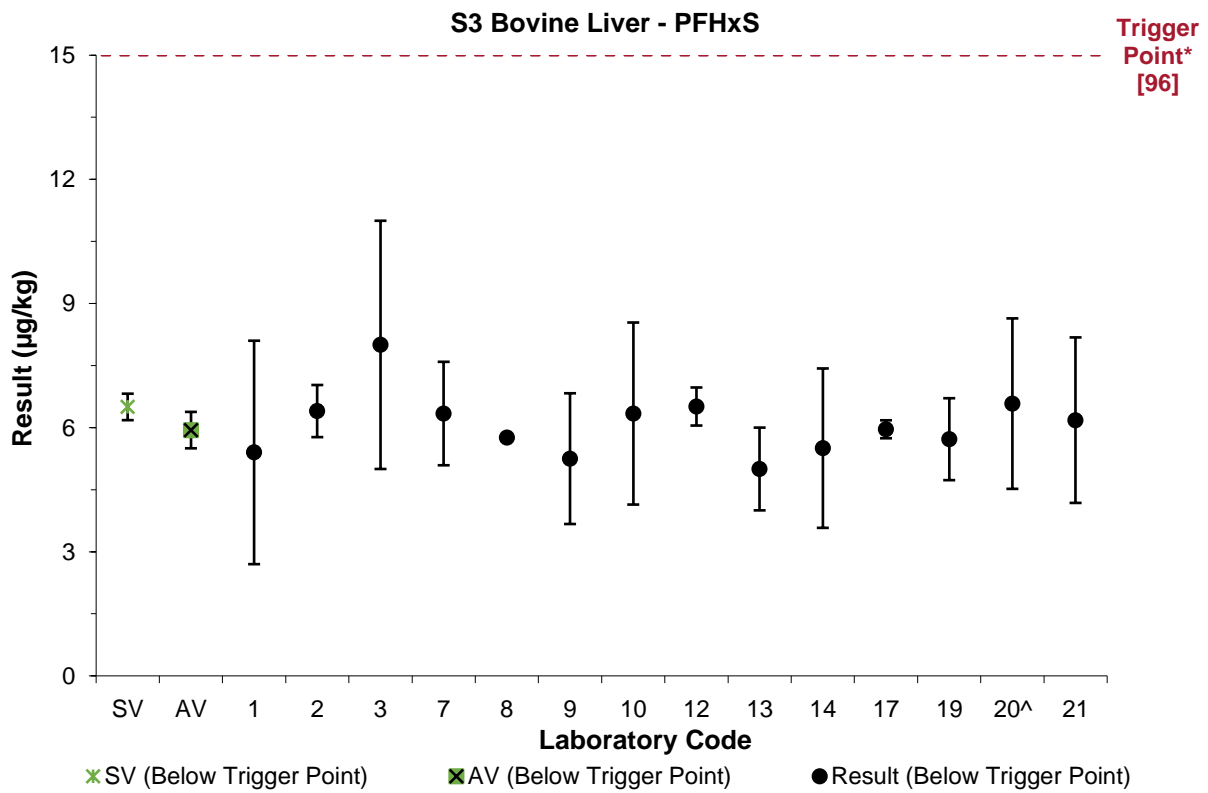
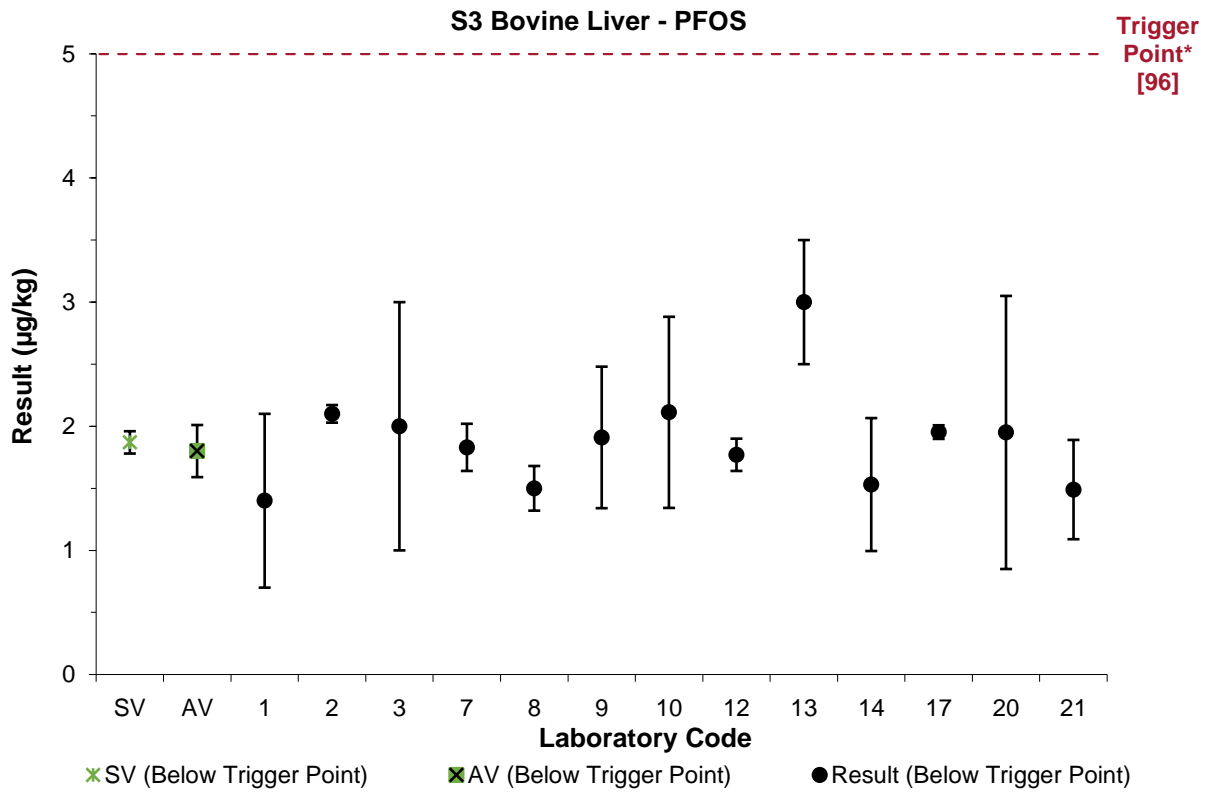


Figure 117 Sample S2 Spinach PFOA Spiked and Assigned Values, Participant Results and Trigger Point



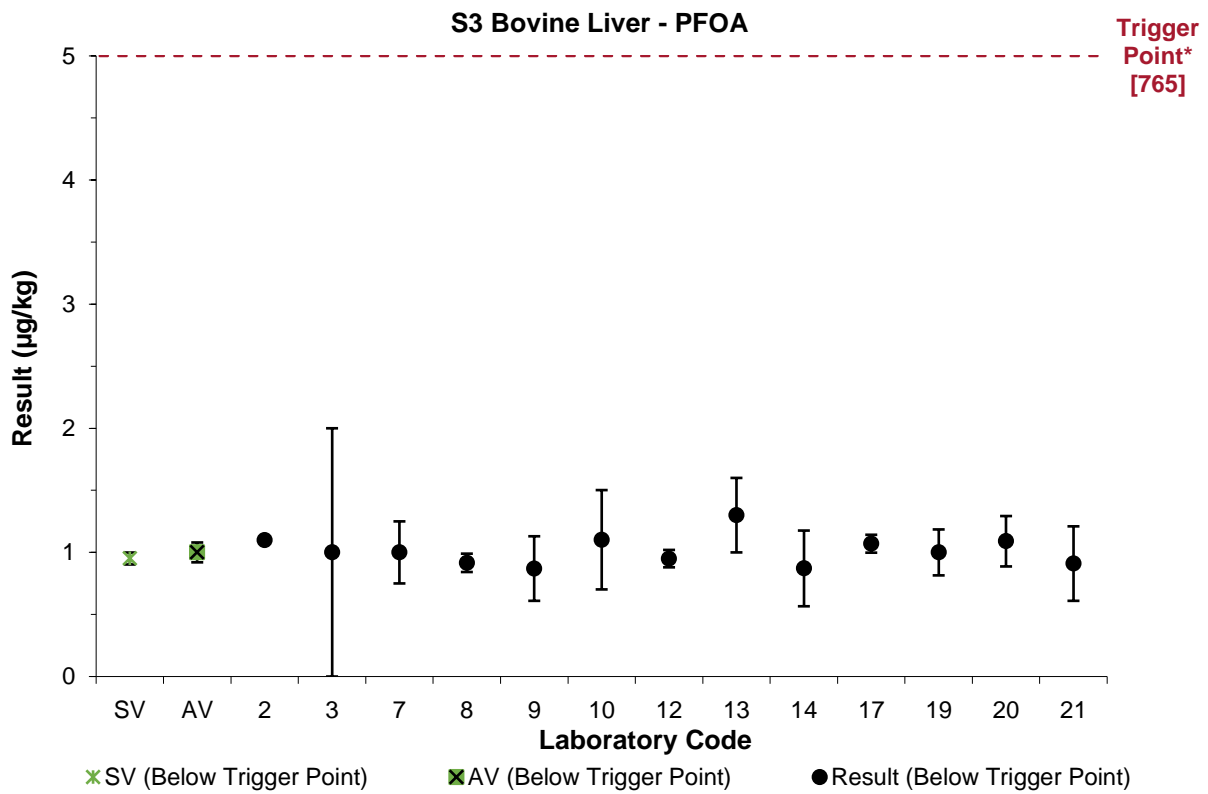
\* The trigger point has been scaled to fit on the chart; actual value in brackets.

Figure 118 Sample S3 Bovine Liver PFHxS Spiked and Assigned Values, Participant Results and Trigger Point



\* The trigger point has been scaled to fit on the chart; actual value in brackets.

Figure 119 Sample S3 Bovine Liver PFOS Spiked and Assigned Values, Participant Results and Trigger Point

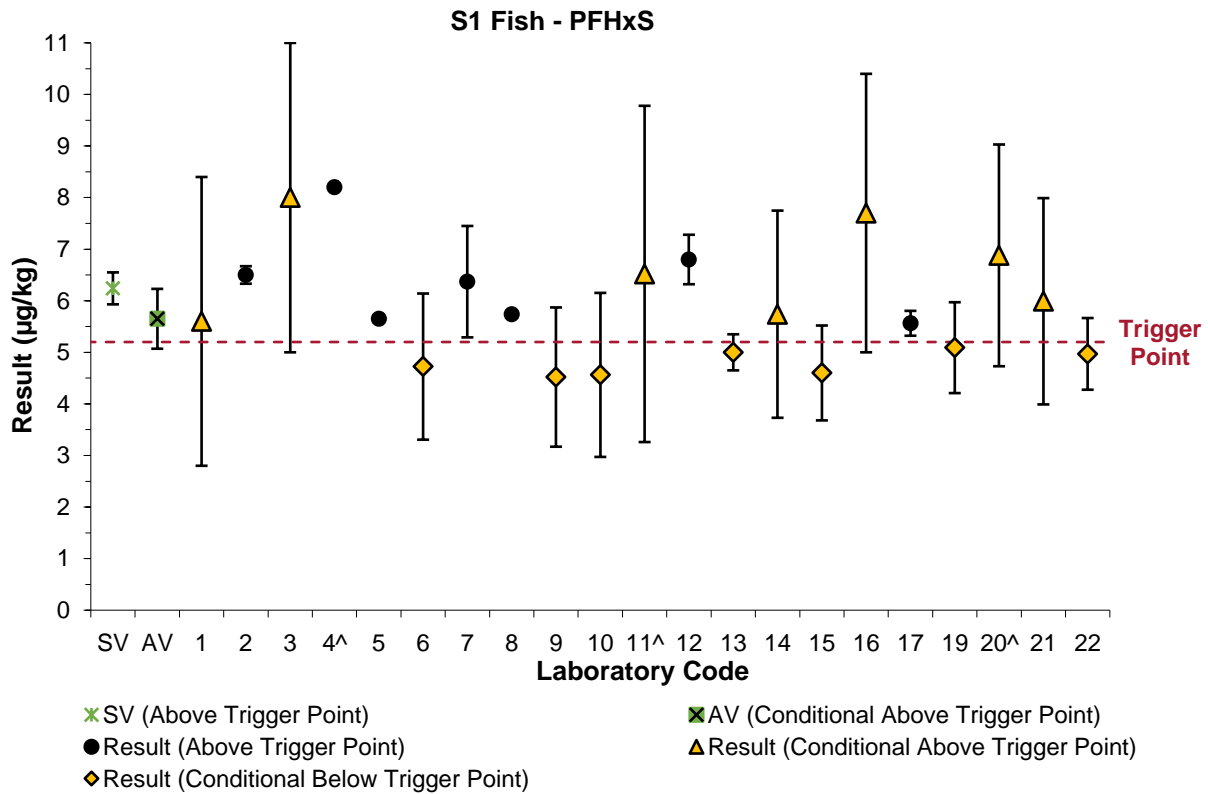


\* The trigger point has been scaled to fit on the chart; actual value in brackets.

Figure 120 Sample S3 Bovine Liver PFOA Spiked and Assigned Values, Participant Results and Trigger Point



As discussed above, while Sample S1 PFHxS was spiked to be above the trigger point and the assigned value was also above the trigger point, the uncertainty spanned the trigger point. Participants' results were all generally close to the trigger point (Figure 121). Laboratories 2, 4, 5, 7, 8, 12 and 17 reported results greater than the trigger point, Laboratories 1, 3, 11, 14, 16, 20 and 21 reported results greater than the trigger point, with uncertainty spanning the trigger point, and Laboratories 6, 9, 10, 13, 15, 19 and 22 reported results lower than the trigger point, with uncertainty spanning the trigger point.



^ Result for linear isomers only has been plotted

Figure 121 Sample S1 Fish PFHxS Spiked and Assigned Values, Participant Results and Trigger Point

## 6.7 False Negatives

Table 91 presents false negative results. These are analytes present in the samples which a participant tested for, but did not report a result; for example, when participants reported a 'less-than' result ( $< x$ ) when the assigned value was higher than their limit of reporting (LOR), or if no value was reported. For analytes where no assigned value was set, results have only been considered to be false negatives where the robust average and spiked value were significantly higher than the participants' LOR (i.e. the robust average minus the expanded uncertainty and the spiked value minus the expanded uncertainty were both greater than the LOR), or if no value was reported.

Table 91 False Negatives

Lab. Code	Sample	Analyte	Assigned Value ( <i>Robust Average</i> ) (µg/kg)	Spiked Value (µg/kg)	Result <sup>a</sup> (µg/kg)
1	S1	8:2FTS	8.26	9.00	< 1
	S3	8:2FTS	9.05	9.39	< 1
2 <sup>b</sup>	S3	PFTeDA	1.52	1.76	< 0.50

Lab. Code	Sample	Analyte	Assigned Value ( <i>Robust Average</i> ) (µg/kg)	Spiked Value (µg/kg)	Result <sup>a</sup> (µg/kg)
		EtFOSA	(4.0)	4.89	NR
		EtFOSAA	4.52	4.89	NR
7	S1	PFUdA	0.722	0.756	<0.1
	S3	PFUdA	0.763	0.781	<0.1
9	S2	PFPeS	3.38	3.47	<0.5
10	S1	PFPeA	2.28	2.39	<2
15	S1	PFBS	1.13	1.41	< 1
	S1	PFDA	1.58	1.59	< 1
17 <sup>c</sup>	S1	PFHxS (linear)	5.97	6.24	NR
		PFOS (linear)	1.61	1.79	NR
	S2	PFHxS (linear)	2.19	2.20	NR
		PFOS (linear)	2.54	2.50	NR
	S3	PFHxS (linear)	5.96	6.50	NR
		PFOS (linear)	1.78	1.87	NR

<sup>a</sup> NR results may or may not be false negatives, depending on the participant's actual LOR.

<sup>b</sup> Laboratory 2 commented that Sample S3 EtFOSA was not reported (NR) because of poor recovery of their QC sample, and Sample S3 EtFOSAA was not reported because of a high internal standard recovery.

<sup>c</sup> Laboratory 17 reported numeric results for the totals of PFHxS and PFOS across all samples. This participant has been included in this table as they did not report results for the linear PFHxS and PFOS across all samples, and they did not mark these analytes as 'NT'.

## 6.8 Reporting of Additional Analytes

Table 92 presents results reported by participants for analytes that were not spiked into the test samples by the study coordinator. Participants should take care to avoid any potential cross-contamination between samples during analysis.

Table 92 Non-Spiked Analytes Reported by Participants

Lab. Code	Sample	Analyte	Result (µg/kg)	Uncertainty (µg/kg)	Recovery (%)
12	S1	PFTTrDA	0.02	0.03	NR
	S2	PFDoA	0.11	0.04	NR
14	S2	PFDoA	2.8966	1.0138	73
16	S1	PFTTrDA	0.08	0.0	NR
		PFUdS	0.02	0.02	NR
	S2	PFDoS	0.15	0.1	NR
		PFTTrDS	0.094	0.05	NR
		PFDoA	0.02	0.02	NR
		PFTeDA	0.01	0.02	NR
19	S1	10:2FTS	0.109	0.026	107
	S2	10:2FTS	0.105	0.025	145
22	S1	6:2FTS	0.704	0.38	88.7

## 6.9 Participants' Methods

Participants were requested to analyse the samples using their normal test method and to report a single result as they would normally report to a client. Methodologies as provided by participants are presented in Appendix 4. A summary of reported methodologies is presented below as response (number of participants):

- Sample Weight for Analysis (g)
  - S1: 0.1 – 0.2 (2), 0.5 – 0.6 (3), 1 – 1.1 (8), 2 – 2.2 (2), 5 (2)
  - S2: 0.1 – 0.2 (2), 0.5 – 0.6 (1), 1 – 1.1 (5), 2 – 2.2 (4), 5 (3), 10 (2)
  - S3: 0.1 – 0.2 (2), 0.5 – 0.6 (4), 1 – 1.1 (5), 5 (2)
- Labelled Standards Added Before Extraction?
  - S1: Yes (16)
  - S2: Yes (16)
  - S3: Yes (12)
- Equilibration Time for Labelled Standard (min)
  - S1: 10 (1), 15 (2), 30 (5), 30 – 60 (1)
  - S2: 10 (1), 15 (2), 30 (5), 30 – 60 (1)
  - S3: 10 (1), 15 (2), 30 (4), 30 – 60 (1)
- Other Sample Pre-Treatment
  - S1: Homogenisation (5), pH adjustment (2)
  - S2: Homogenisation (5), pH adjustment (2)
  - S3: Homogenisation (5), pH adjustment (2)
- Extraction Techniques
  - S1: QuEChERS (6), Alkaline Digestion (7), Solid-Liquid Extraction (5)
  - S2: QuEChERS (6), Alkaline Digestion (7), Solid-Liquid Extraction (5)
  - S3: QuEChERS (3), Alkaline Digestion (7), Solid-Liquid Extraction (4)
- Extraction Solvent(s)
  - S1: Methanol/Base (5), Methanol (1), Acetonitrile/Acid (2), Acetonitrile (8)
  - S2: Methanol/Base (5), Methanol (1), Acetonitrile/Acid (2), Acetonitrile (8)
  - S3: Methanol/Base (4), Methanol (1), Acetonitrile/Acid (2), Acetonitrile (5)
- Total Extraction Time (min)
  - S1: 8 (1), 15 (1), 20 (1), 30 (4), 60 (4), 90 (1), 180 (1), 480 (1)
  - S2: 8 (1), 15 (1), 20 (1), 30 (5), 60 (5), 90 (1), 480 (1)
  - S3: 8 (1), 15 (1), 30 (2), 60 (4), 90 (1), 480 (1)
- Carbon Clean-Up?
  - S1: Yes (11), No (2)
  - S2: Yes (10), No (2)
  - S3: Yes (8), No (2)

- Extract Concentration Temperature (° C)
  - S1: Ambient (6), 35 (1), 40 (1), 45 (2), 50 (1), 55 (1), 60 (1)
  - S2: Ambient (5), 35 (1), 40 (1), 45 (2), 50 (1), 55 (1), 60 (1)
  - S3: Ambient (4), 35 (1), 40 (1), 45 (1), 50 (1), 60 (1)
- Total Extract Concentration Time (min)
  - S1: 30 (1), 40 (1), 40 – 60 (1), 60 (2), 60 – 90 (1), 90 (1), 200 (1), 900 (1), Variable (1)
  - S2: 30 (1), 40 – 60 (1), 60 (2), 60 – 90 (1), 90 (1), 200 (1), 900 (1), Variable (1)
  - S3: 30 (1), 60 (2), 60 – 90 (1), 90 (1), 900 (1), Variable (1)
- Clean-Up Techniques
  - S1: (Dispersive) Solid-Phase Extraction (14), Filtration (2), Liquid-Liquid Extraction (1)
  - S2: (Dispersive) Solid-Phase Extraction (13), Filtration (2), Liquid-Liquid Extraction (1)
  - S3: (Dispersive) Solid-Phase Extraction (10), Filtration (2), Liquid-Liquid Extraction (1)
- Elution Solvent(s)
  - S1: Methanol/Base (8), Methanol (1), Acetonitrile (1), Acetonitrile/Acetone/Base (1)
  - S2: Methanol/Base (8), Methanol (1), Acetonitrile (1), Acetonitrile/Acetone/Base (1)
  - S3: Methanol/Base (6), Methanol (1), Acetonitrile/Acetone/Base (1)
- Final pH Adjustment?
  - S1: Yes (2), No (10)
  - S2: Yes (2), No (9)
  - S3: Yes (2), No (6)
- Instrument
  - S1: LC-Orbitrap (2), LC-MS/MS or LC-QQQ (15)
  - S2: LC-Orbitrap (2), LC-MS/MS or LC-QQQ (15)
  - S3: LC-Orbitrap (1), LC-MS/MS or LC-QQQ (12)
- Dilution Before Analysis?
  - S1: Yes (3), No (9)
  - S2: Yes (3), No (9), Variable (1)
  - S3: Yes (1), No (7)
- Blank Correction?
  - S1: Yes (5), No (11)
  - S2: Yes (5), No (11)
  - S3: Yes (3), No (9)

- Labelled Standard Source
  - S1: Wellington Laboratories (17), Cambridge Isotope Laboratories (4)
  - S2: Wellington Laboratories (15), Cambridge Isotope Laboratories (4)
  - S3: Wellington Laboratories (12), Cambridge Isotope Laboratories (3)

- Recovery Correction

Participants reported a very broad range of recoveries, ranging from 7% to 348%, though around half of the reported recoveries were between 75% and 100%.

- S1: Yes (15), No (2)
- S2: Yes (13), No (2)
- S3: Yes (10), No (2)

Comparisons of  $z$ -scores with various sample preparation and analysis parameters are given in Figures 122 to 135 (for all charts, where the  $z$ -score was greater than 10.0, this has been plotted at 10.0 instead).

Participants used a very wide variety of methodologies for the analysis of the samples in this study. These different methodologies mostly produced compatible results.

In this study, results from participants using 0.1 – 0.2 g for sample analysis returned varied  $z$ -scores, including several that were biased high. Caution should be exercised when a small sample size is taken for analysis, as this may not be a suitable representation of the whole sample.

Two participants reported using an LC-Orbitrap for analysis and while their results were mostly acceptable, their results were biased low. One of these participants was also the only participant in this study to report using acetonitrile only as their elution solvent (the other participant did not report what solvent was used). These participants may need to review if their methodology introduced bias to their measurements.

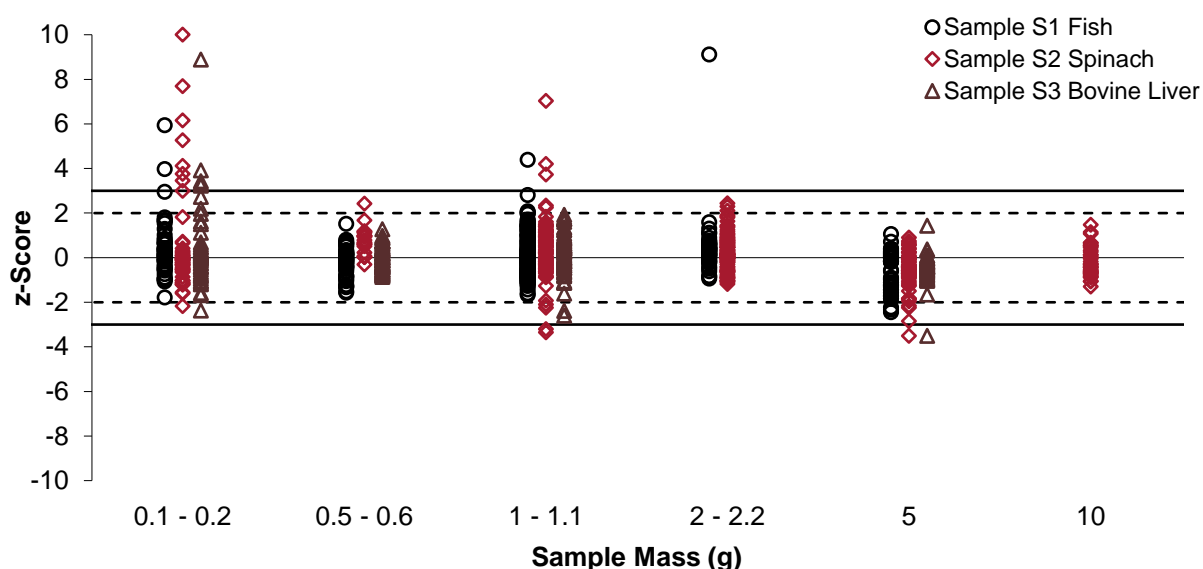


Figure 122  $z$ -Score vs Sample Mass Used for Analysis

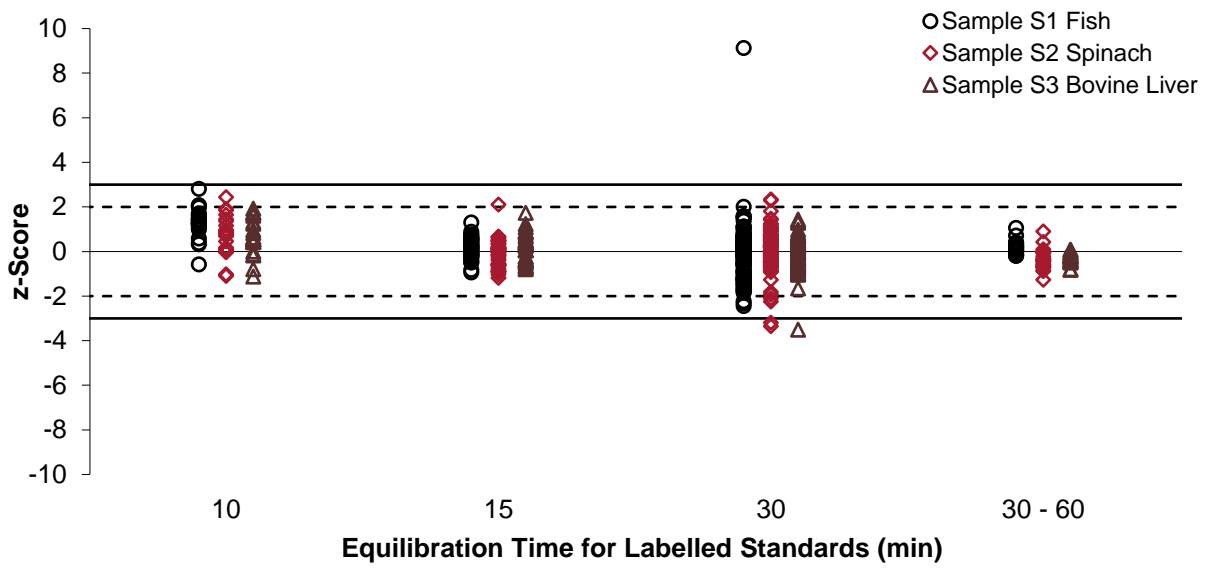


Figure 123 z-Score vs Equilibration Time for Labelled Standards

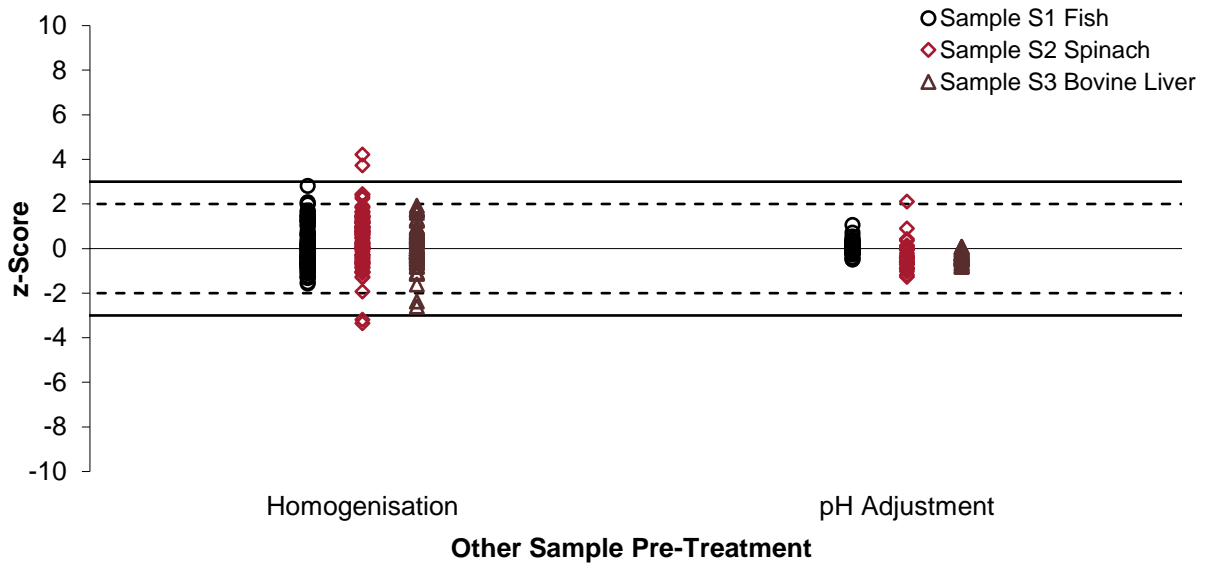


Figure 124 z-Score vs Other Sample Pre-Treatment

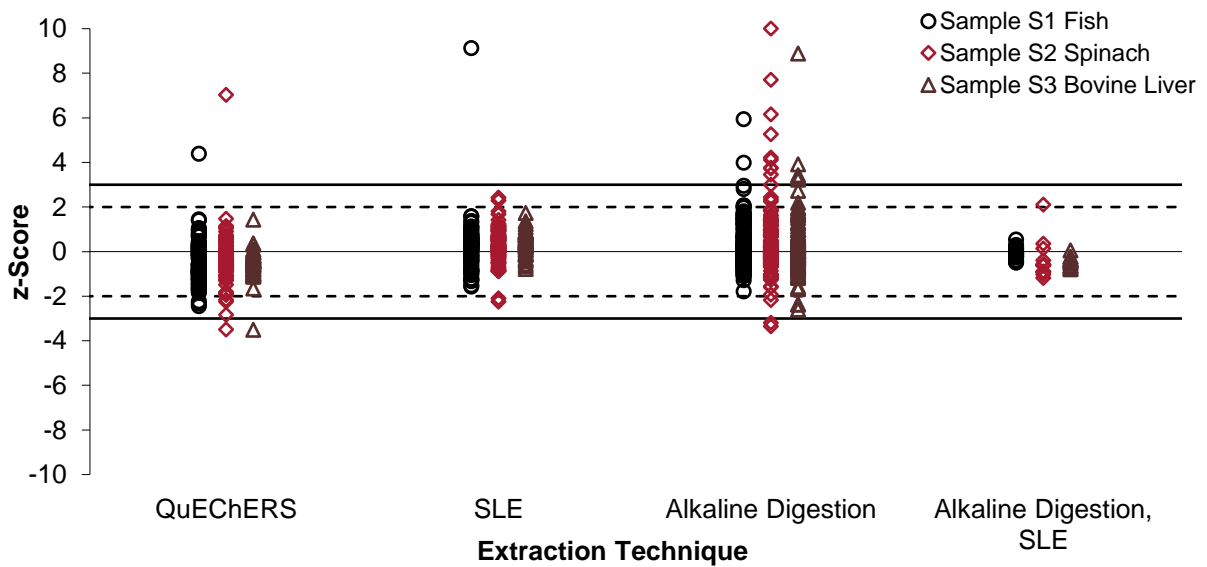


Figure 125 z-Score vs Extraction Technique

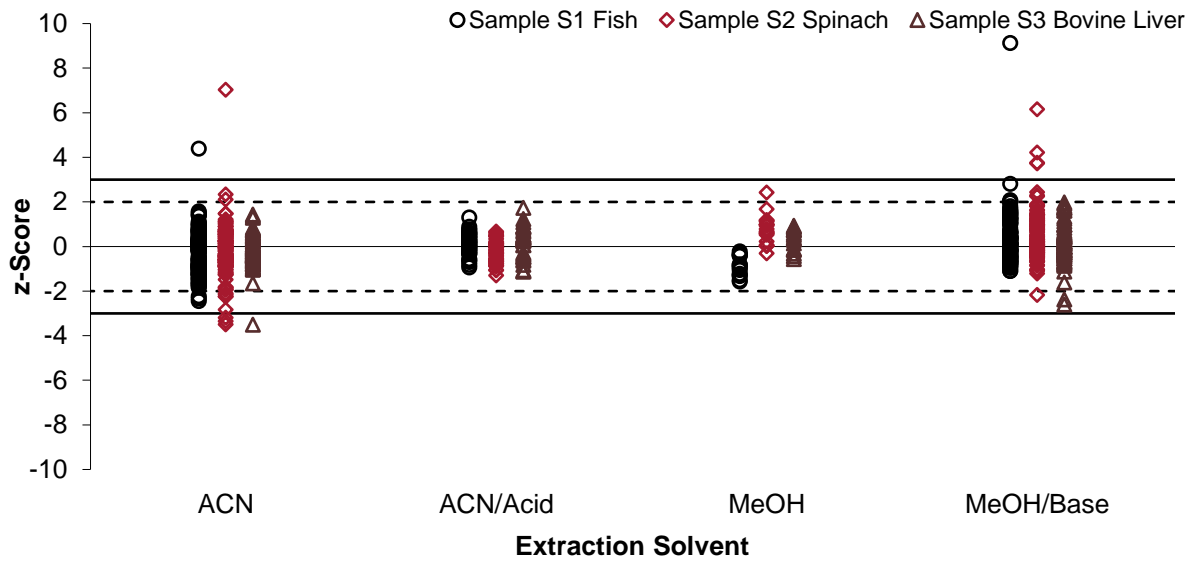


Figure 126 z-Score vs Extraction Solvent

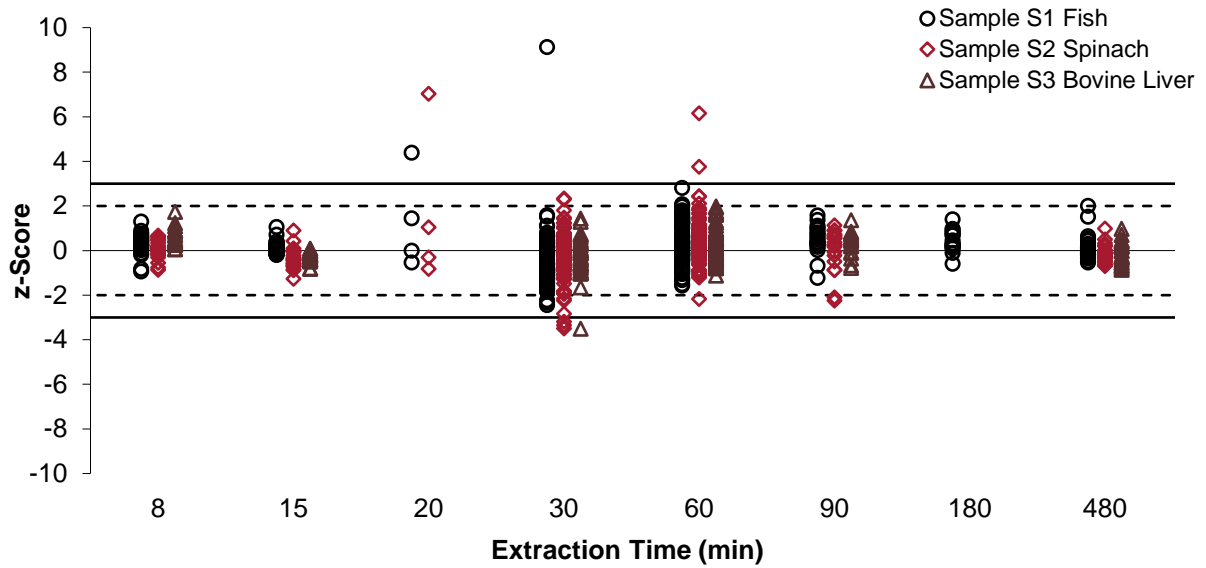


Figure 127 z-Score vs Extraction Time

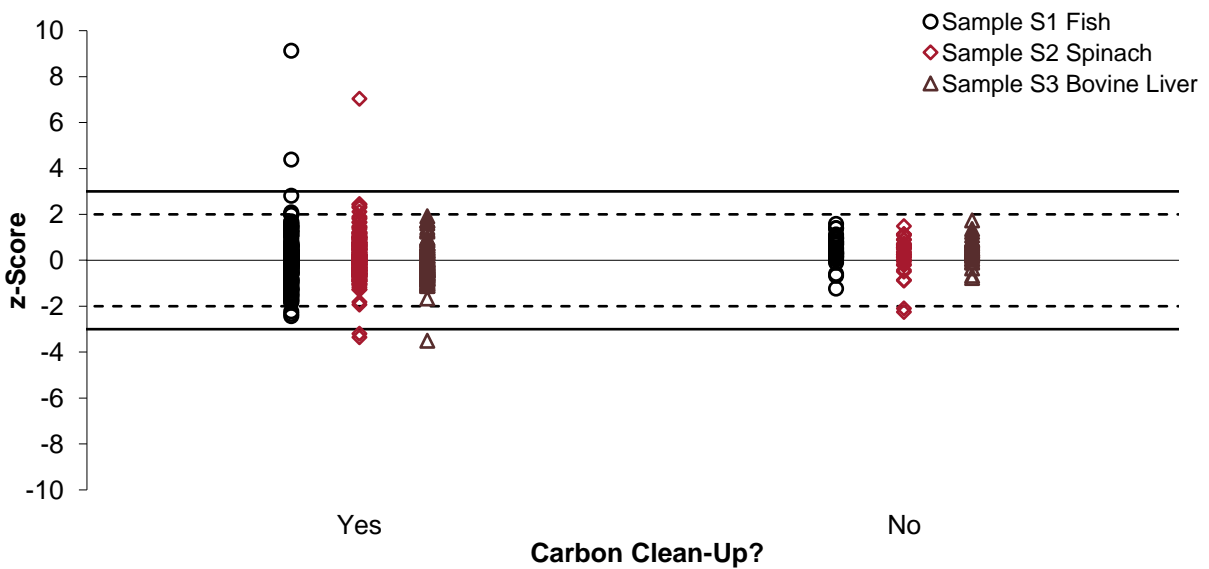


Figure 128 z-Score vs Carbon Clean-Up

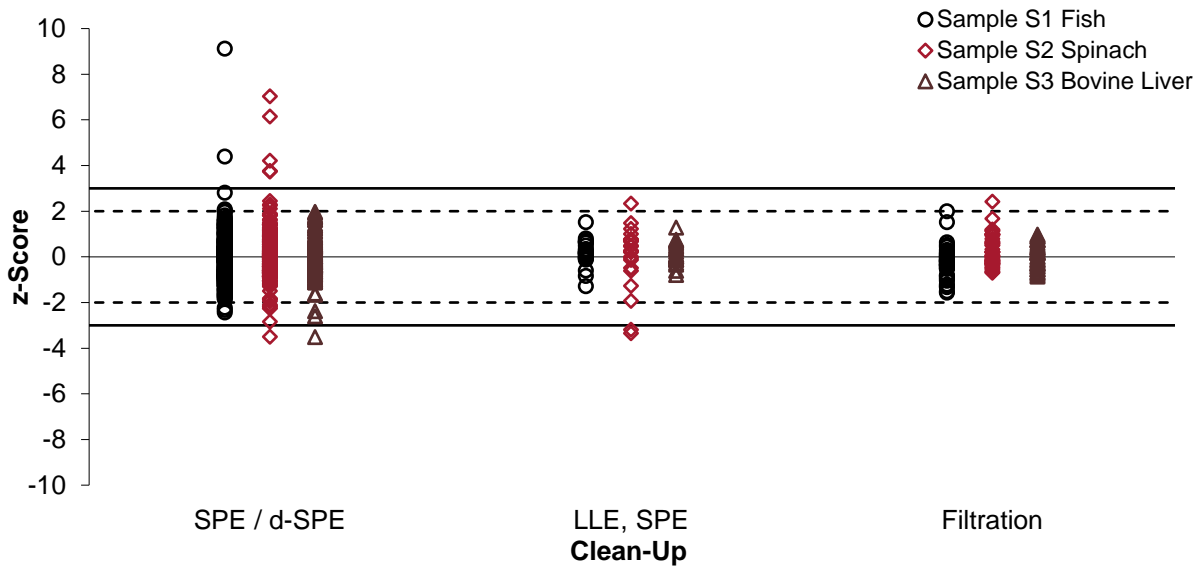


Figure 129 z-Score vs Clean-Up

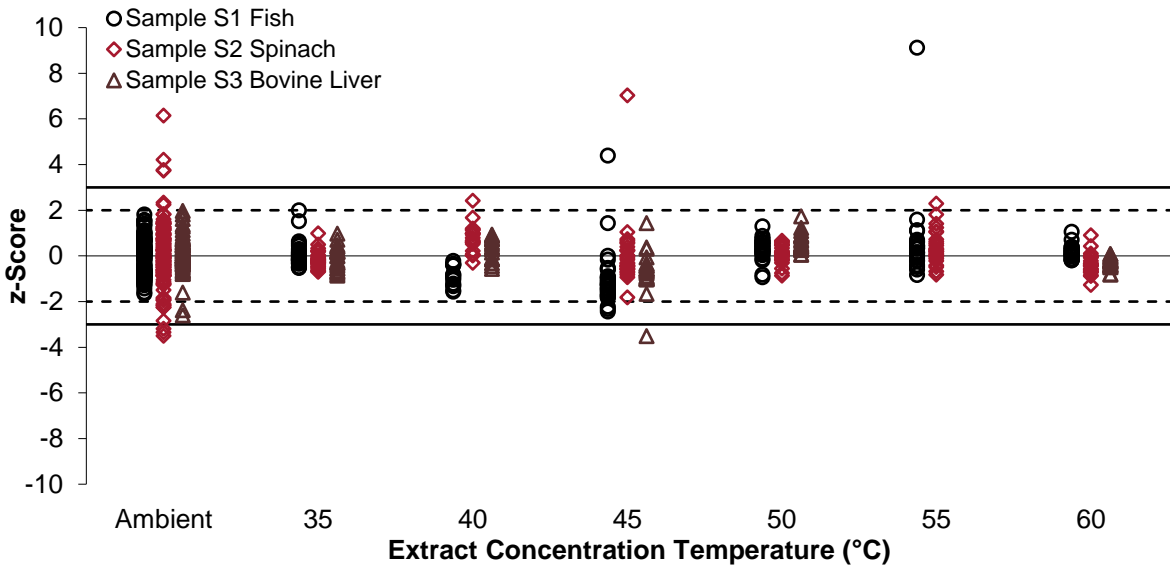


Figure 130 z-Score vs Extract Concentration Temperature

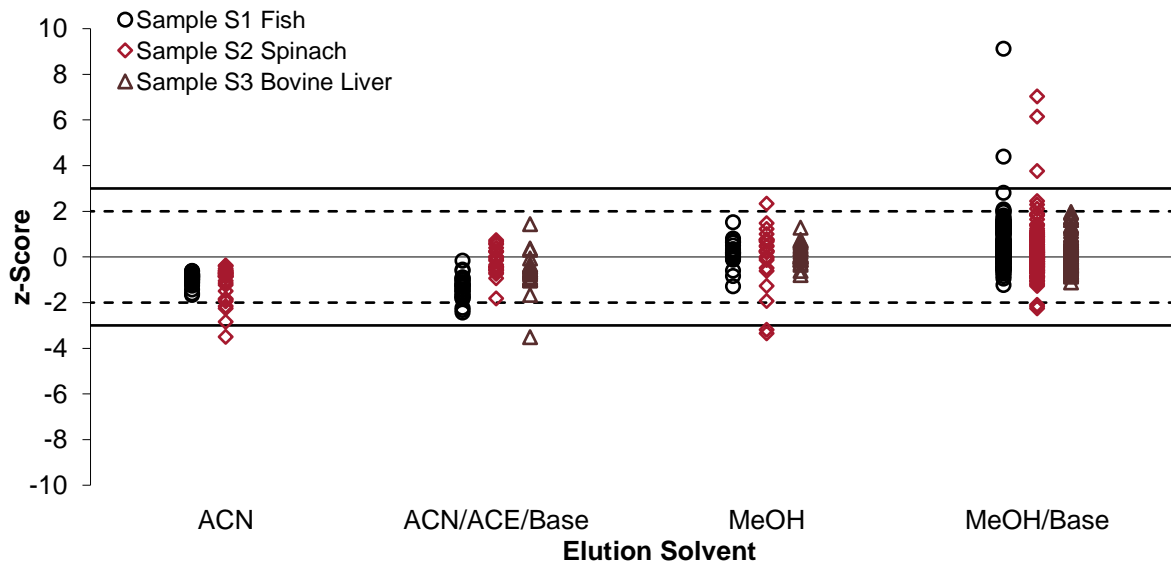


Figure 131 z-Score vs Elution Solvent



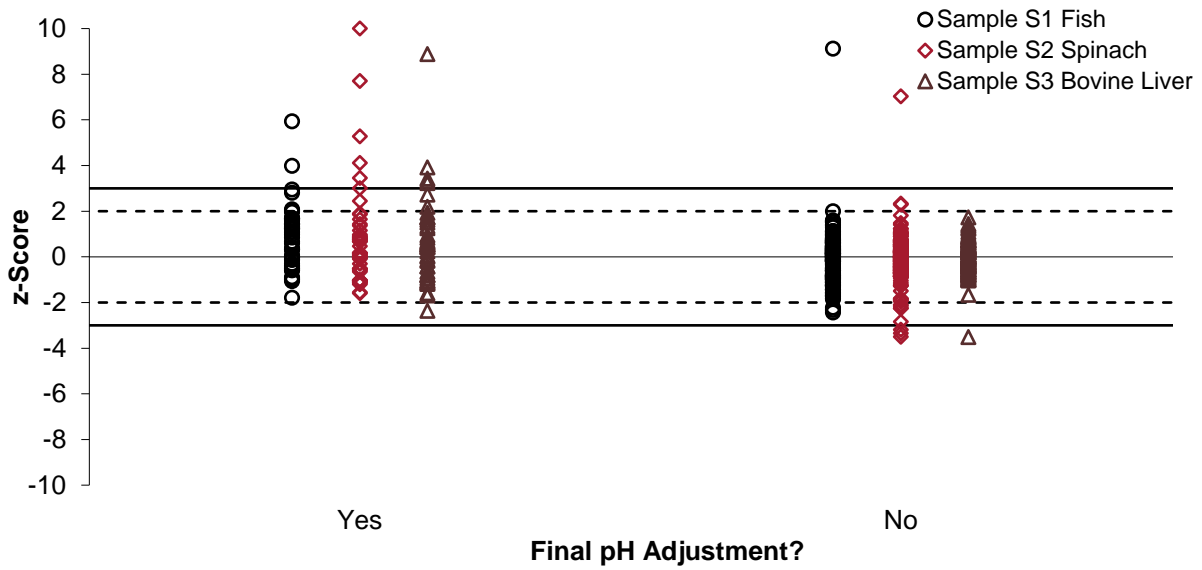


Figure 132 z-Score vs Final pH Adjustment

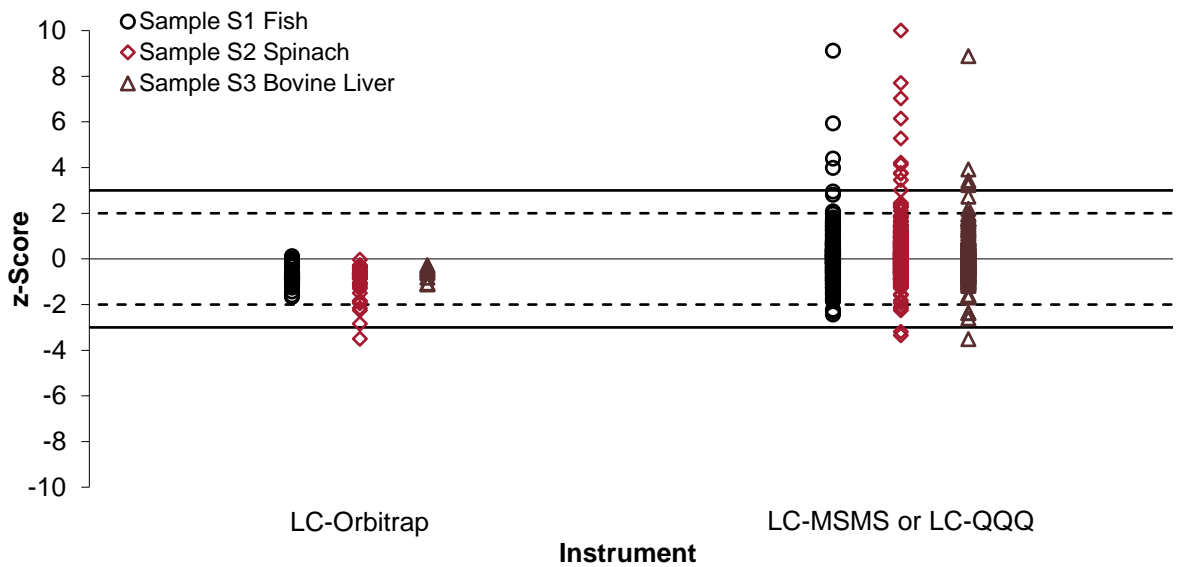


Figure 133 z-Score vs Measurement Instrument

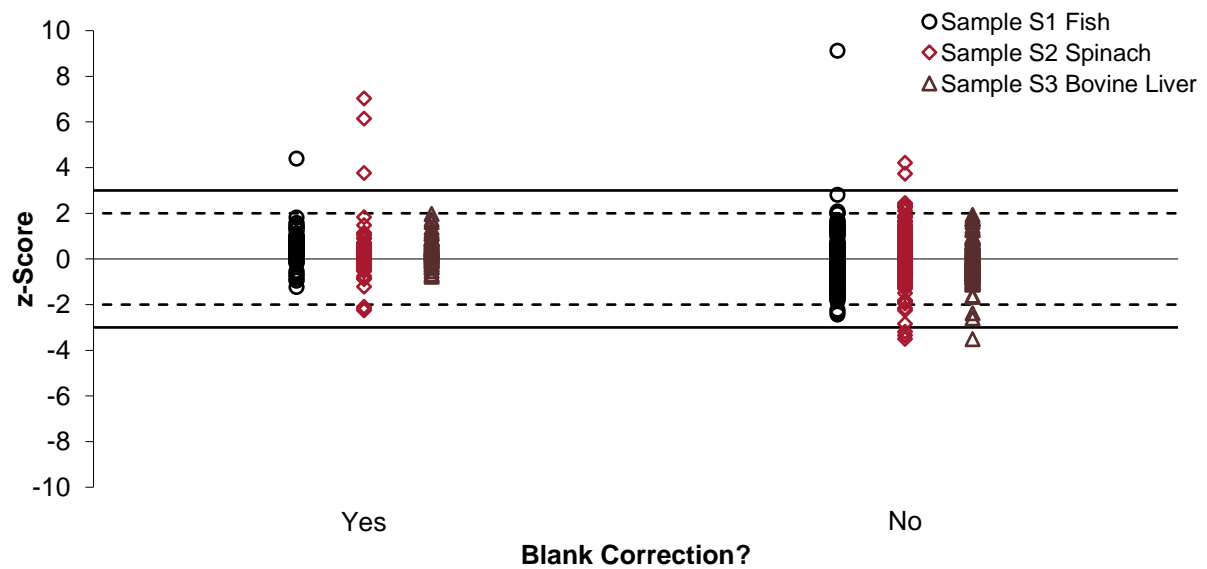


Figure 134 z-Score vs Blank Correction

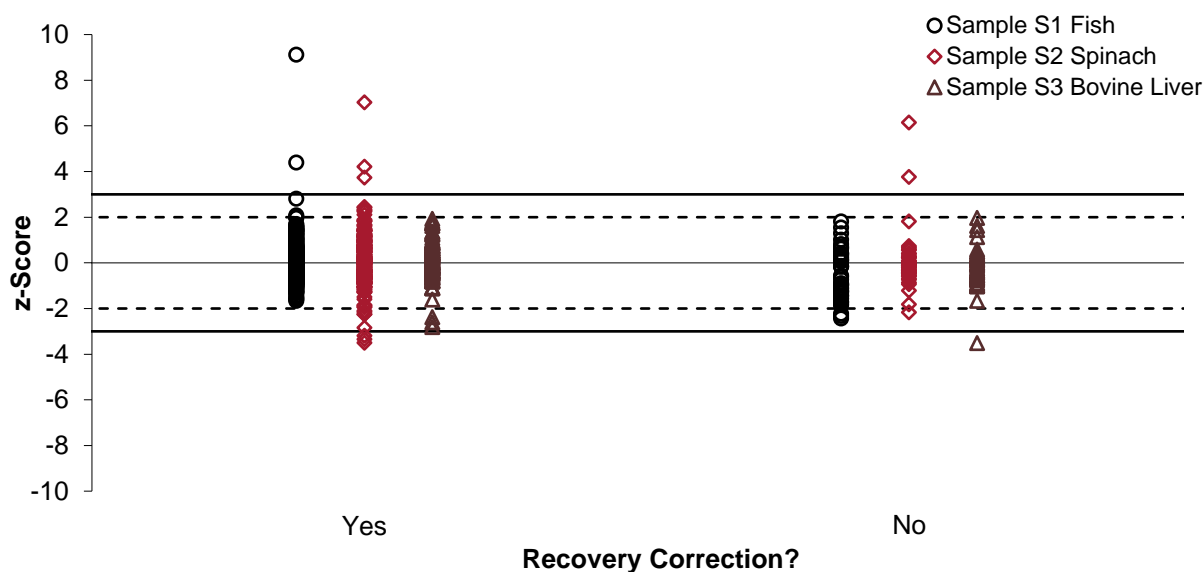


Figure 135 z-Score vs Recovery Correction

### 6.10 Total vs Linear 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 93. Most participants reported numeric results for both linear and total.

Table 93 Number of Participants Reporting Numeric PFHxS and PFOS Results

Sample	PFHxS			PFOS		
	Linear and Total	Linear Only	Total Only	Linear and Total	Linear Only	Total Only
S1	13	3	5	15	2	4
S2	13	2	5	15	1	4
S3	11	1	2	12	0	1

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

#### PFHxS

Summaries of participants' results for linear and total PFHxS in Samples S1, S2 and S3 are presented in Figures 136 to 138.

Of the participants reporting numeric results for both linear and total PFHxS, the majority correctly reported the same result for both. Laboratory **13** reported lower results for PFHxS linear as compared to total (94%) across all three samples, though results were in agreement with each other within their respective uncertainties.

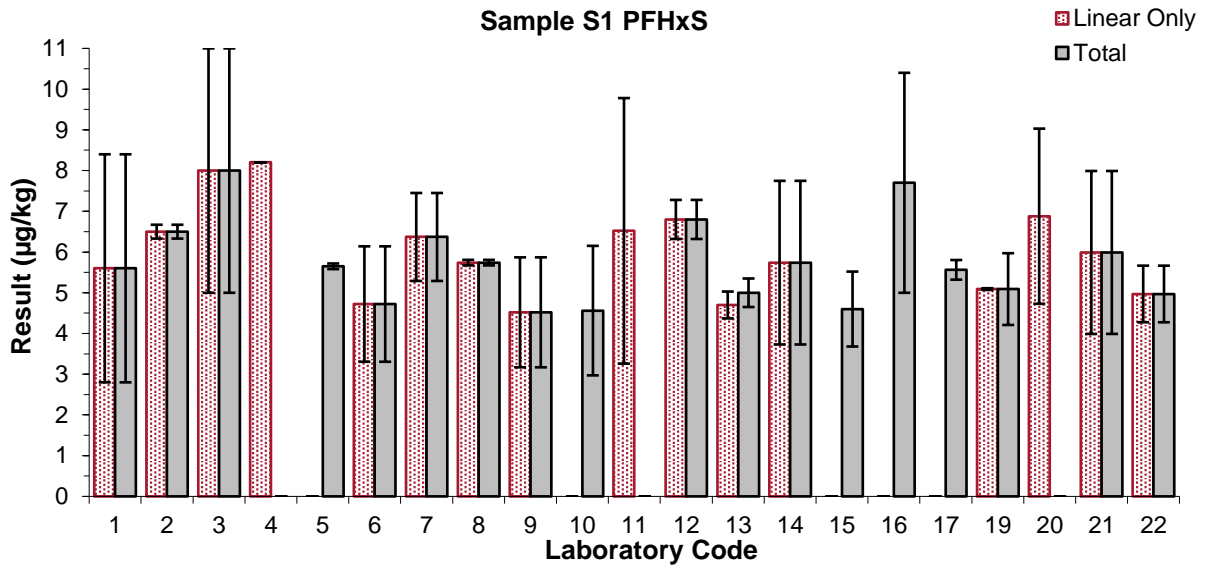


Figure 136 Participant Results for Sample S1 PFHxS (linear and total)

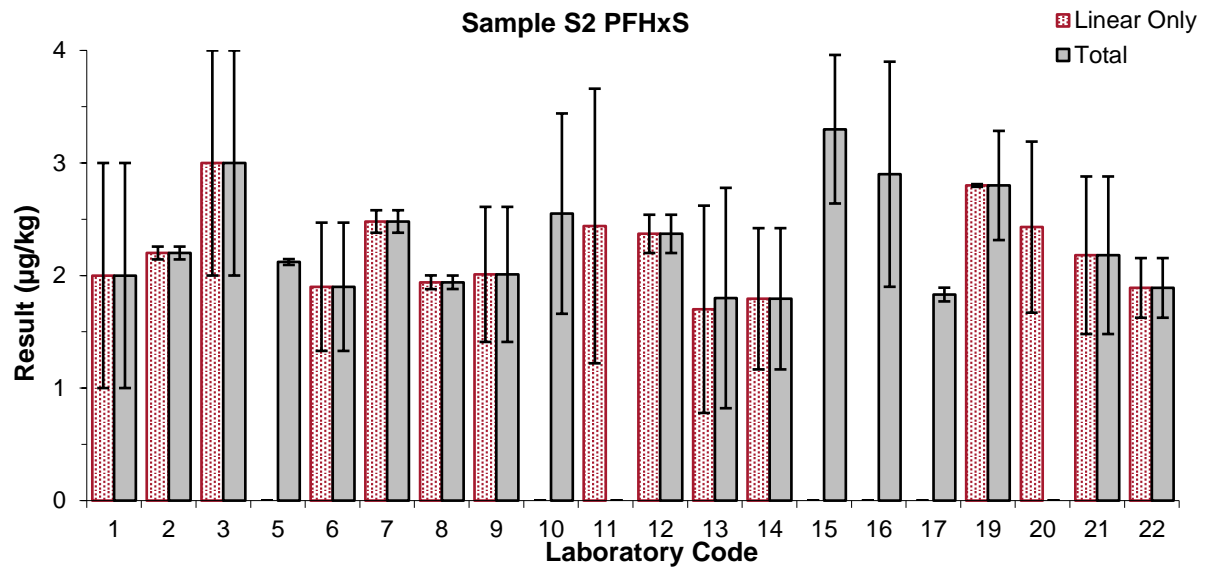


Figure 137 Participant Results for Sample S2 PFHxS (linear and total)

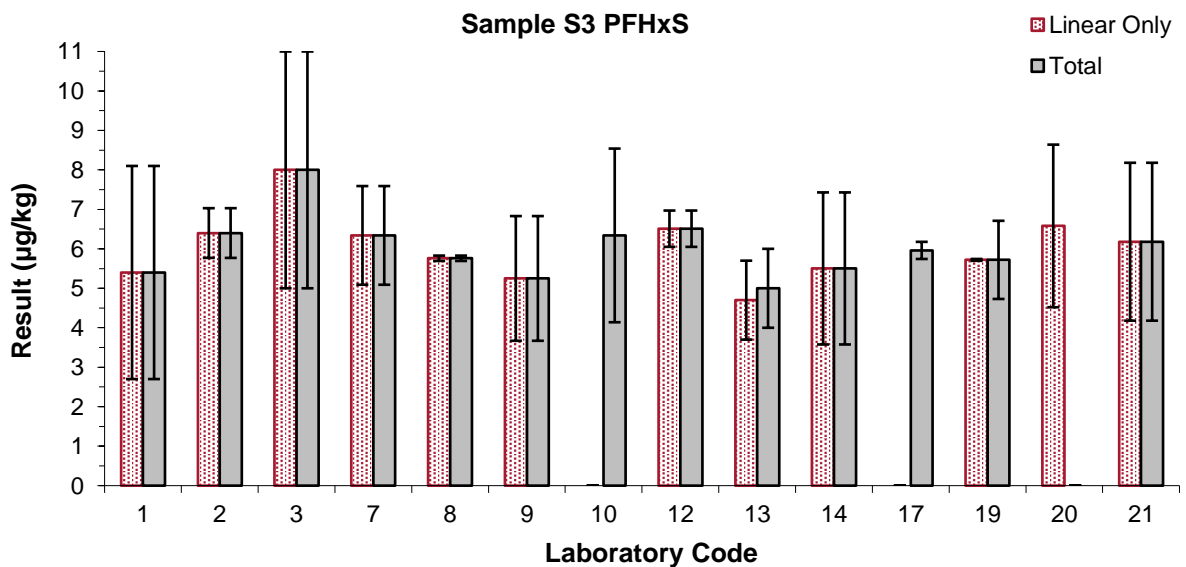


Figure 138 Participant Results for Sample S3 PFHxS (linear and total)

## PFOS

Summaries of participants' results for linear and total PFOS in Samples S1 and S2 are presented in Figures 139 to 141.

Of the participants reporting numeric results for both linear and total PFOS, the majority of participants correctly reported the same result for both. Laboratories **7** and **13** reported lower results for PFOS linear as compared to total for Samples S3 (98%) and S1 (94%) respectively. In both cases, results were in agreement with each other within their respective uncertainties.

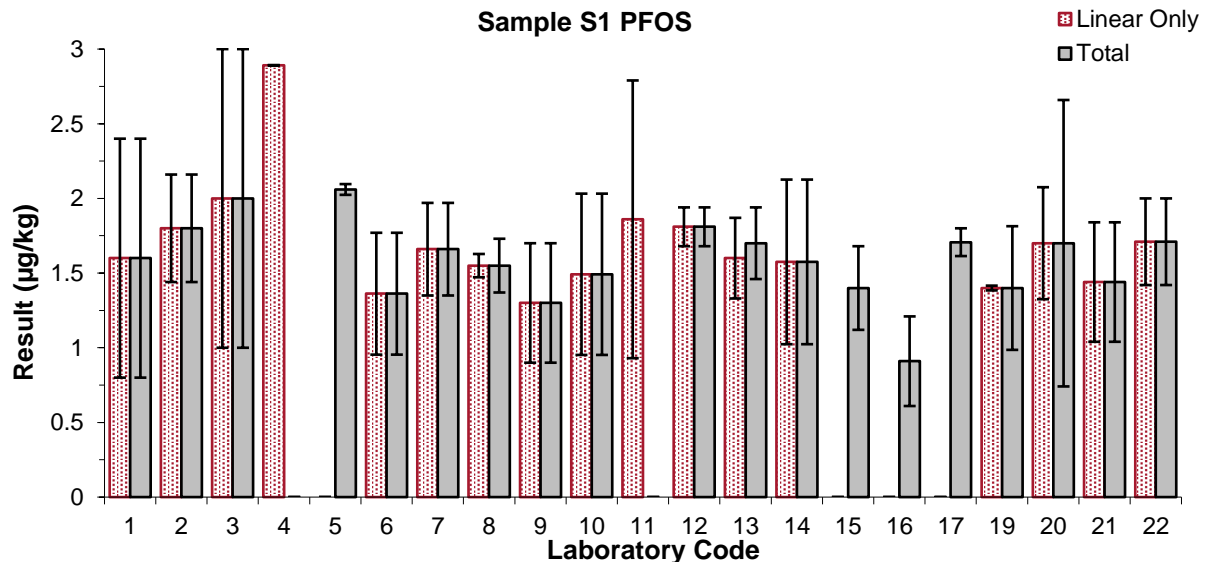


Figure 139 Participant Results for Sample S1 PFOS (linear and total)

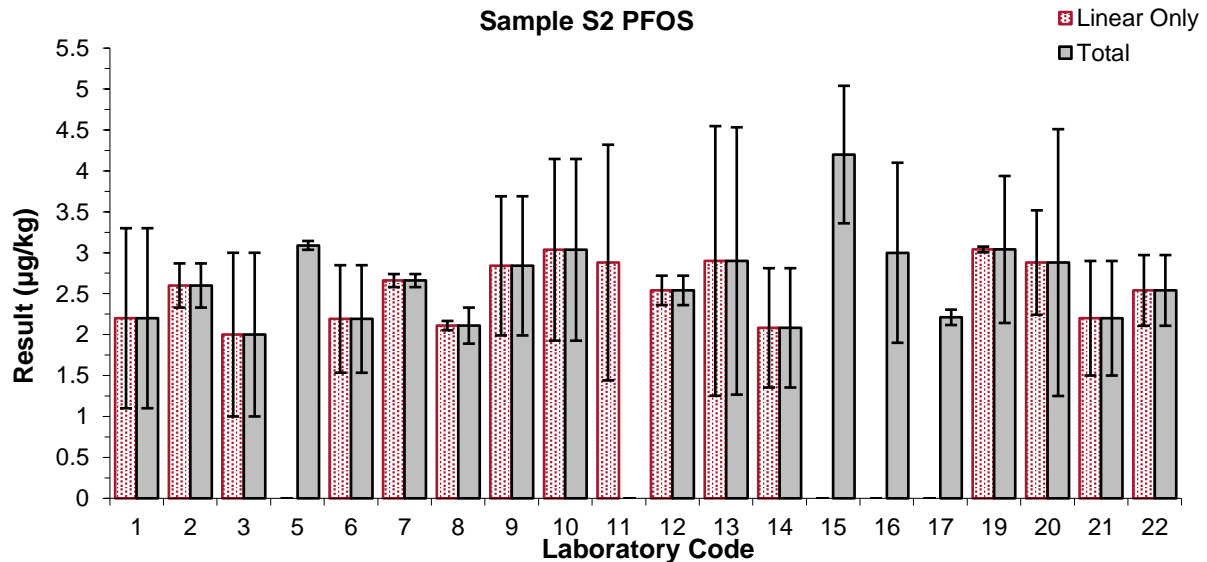


Figure 140 Participant Results for Sample S2 PFOS (linear and total)

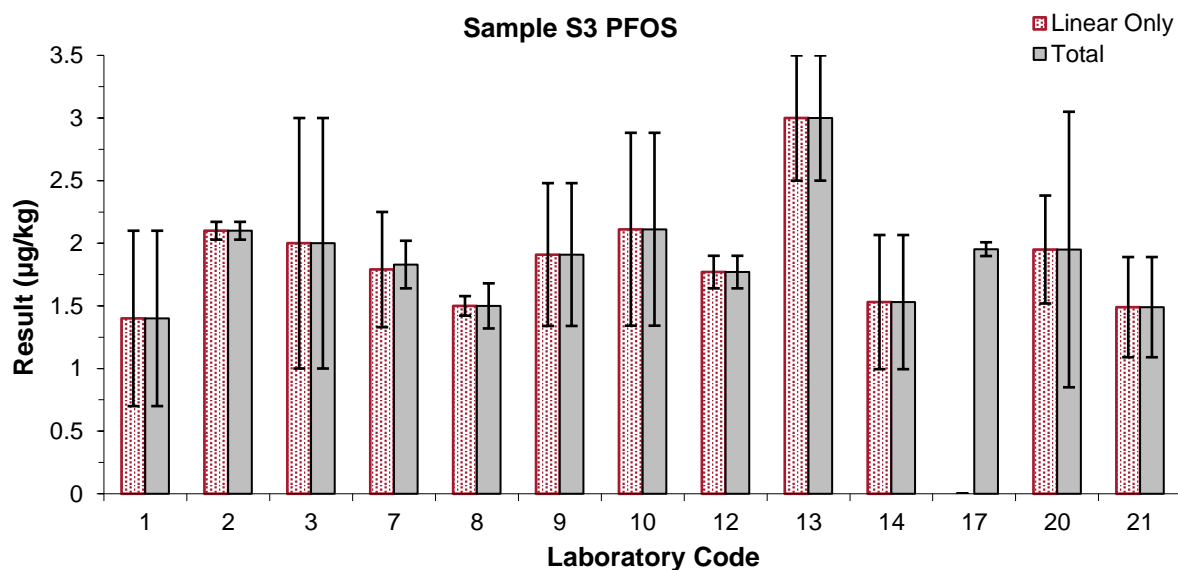


Figure 141 Participant Results for Sample S3 PFOS (linear and total)

### 6.11 Effects of Sample Matrix

The samples in this study were spiked fish (Sample S1), spinach (Sample S2) and bovine liver (Sample S3). A summary of the results reported and  $z$ -scores obtained by matrix is presented in Table 94.

For this study, participants performed slightly better with the fish and bovine liver matrices, achieving a higher proportion of acceptable  $z$ -scores as compared to spinach.

Table 94 Result Comparison by Matrix

Sample	Matrix	Expected Number of Results	Numeric Results Reported	$z$ -Scores Calculated	Acceptable $z$ -Scores
S1	Fish	567	432 (76%)	432	411 (95%)
S2	Spinach	520	422 (81%)	422	385 (91%)
S3	Bovine Liver	378	314 (83%)	307	295 (96%)

## 6.12 Summary of Participants' Results and Performances

Summaries of participants' results and performances for scored analytes in this PT study are presented in Tables 95 to 100, and Figure 142.

Table 95 Summary of Participants' Sample S1 Results (part 1)\*

Lab. Code	PFBS	PFPeS	PFHxS	PFHxS (linear)	PFHpS	PFOS	PFOS (linear)	PFNS	PFDS	PFBA	PFPeA	PFHxA	PFHpA
AV	1.13	2.87	5.65	5.97	1.28	1.60	1.61	1.59	1.60	6.20	2.28	6.21	6.68
HV	1.05	3.11	5.4	5.4	1.17	1.55	1.55	1.51	1.44	5.8	2.21	6.3	7.2
SV	1.41	3.08	6.24	6.24	1.34	1.79	1.79	1.80	1.80	6.57	2.39	6.54	7.48
1	1.1	2.7	5.6	5.6	1.1	1.6	1.6	< 2	< 2	5.5	2	5.8	6.4
2	1.3	2.8	6.5	6.5	1.4	1.8	1.8	1.6	1.3	6.5	2.4	7.3	7.5
3	1	4	8	8	2	2	2	2	2	8	3	8	8
4	1.6804	3.616	NT	8.2013	1.964	NT	2.892	NT	NT	10.192	2.77	9.524	12.348
5	NT	NT	5.65	NT	NT	2.06	NT	NT	NT	NT	NT	NT	NT
6	0.932	2.237	4.723	4.723	1.062	1.362	1.362	1.338	1.247	4.134	1.943	5.207	5.55
7	1.28	2.83	6.37	6.37	1.35	1.66	1.66	1.58	1.33	6.42	2.32	7.09	8.71
8	1.20	3.02	5.74	5.74	1.31	1.55	1.55	1.59	1.66	6.29	2.21	6.18	6.94
9	0.74	1.59	4.52	4.52	1.05	1.3	1.3	1.19	1.06	5.51	2	4.83	4.57
10	0.948	2.172	4.562	NT	1.07	1.492	1.492	NT	<2	5.712	<2	4.552	5.49
11	1.19	3.27	NT	6.52	1.34	NT	1.86	1.63	1.91	6.07	2.66	7.06	6.89
12	1.54	2.95	6.8	6.8	1.61	1.81	1.81	1.63	1.71	7.22	2.22	6.74	7.26
13	1.1	2.3	5.0	4.7	2.3	1.7	1.6	1.8	3.5	NT	2.1	6.2	5.4
14	1.0878	NT	5.7392	5.7392	1.1774	1.5750	1.5750	NT	1.6938	NT	2.2760	6.4614	6.5226
15	< 1	3.0	4.6	NT	1.2	1.40	NT	NT	NT	4.6	1.8	4.8	5.3
16	1.1	3.0	7.7	NT	1.4	0.91	NT	2.6	2.1	8.3	2.5	6.8	6.5
17	1.191	2.778	5.563	NR	1.334	1.707	NR	1.668	1.716	5.873	2.169	6.246	7.854
19	0.968	2.58	5.09	5.09	1.12	1.40	1.40	1.34	1.32	5.41	2.07	5.67	6.43
20	1.36	3.66	NT	6.88	1.41	1.70	1.70	1.77	1.65	7.39	2.79	7.21	8.79
21	1.06	2.75	5.99	5.99	1.24	1.44	1.44	1.63	1.57	6.19	2.06	5.83	6.87
22	1.02	3.06	4.97	4.97	1.39	1.71	1.71	1.53	1.74	6.38	2.17	6.09	6.14

\* AV = Assigned Value; HV = Homogeneity Value; SV = Spiked Value. All values are in µg/kg. Shaded cells are results which returned a questionable or unacceptable z-score.

Table 96 Summary of Participants' Sample S1 Results (part 2)\*

Lab. Code	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFTeDA	PFOSA	EtFOSA	EtFOSAA	8:2FTS	GenX	ADONA	9CI-PF3ONS	11CI-PF3OUdS
AV	0.940	1.79	1.58	0.722	3.10	1.32	4.69	2.85	4.27	8.26	12.7	15.6	19.5	23.3
HV	1.03	1.91	1.66	0.81	3.09	1.91	4.3	4.1	4.5	10.3	12.4	14.5	19.7	22.1
SV	0.943	1.89	1.59	0.756	3.70	1.68	5.39	4.67	4.67	9.00	14.1	17.6	21.8	26.4
1	< 5	< 2	< 5	< 2	< 5	< 5	4.8	< 5	< 5	< 1	NT	NT	NT	NT
2	< 1.0	2.0	1.7	< 1.0	3.4	1.1	4.9	2.9	4.4	8.7	16	15	20	25
3	1	2	2	< 2	4	< 5	6	< 5	6	11	< 50	19	21	29
4	1.567	2.656	1.85	NT	NT	NT	NT	NT	NT	NT	NT	20.679	25.6	31.829
5	0.84	3.36	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
6	0.756	1.505	1.295	0.581	2.379	1.056	4.097	2.297	3.507	7.256	8.637	11.158	14.591	19.045
7	0.94	1.97	1.78	<0.1	3.16	1.53	4.64	NT	3.76	8.52	NT	16.28	19.02	17.35
8	0.942	1.88	1.58	0.776	3.35	1.60	4.74	NT	NT	8.92	13.3	17.8	19.0	24.0
9	0.73	1.35	1.18	0.53	2.14	0.94	3.32	2.76	3.05	5.42	8.03	9.91	10.4	11.94
10	<1	1.228	1.104	<1	2.448	<2	<5	2.724	3.168	7.562	NT	NT	NT	NT
11	1	2.29	1.61	NT	NT	1.39	NT	NT	NT	9.79	11.19	NT	NT	NT
12	0.91	1.87	1.71	0.76	3.32	1.73	NT	NT	NT	NT	12.86	12.65	19.93	19.91
13	1.1	1.6	1.3	0.65	NT	1.2	4.4	2.8	5.7	8.4	17	10	31	31
14	0.8466	1.7093	1.7502	0.6533	3.1792	1.2525	NT	NT	NT	NT	NT	NT	NT	NT
15	< 1	1.2	< 1	NT	NT	NT	NT	NT	NT	7.0	NT	NT	NT	NT
16	1.3	2.1	1.7	0.76	3.7	1.2	5.2	NT	NT	16	NT	20	NT	NT
17	1.015	1.667	1.501	0.785	3.197	1.079	5.521	NT	NT	8.637	13.753	17.693	20.789	22.731
19	0.868	1.66	1.37	0.732	2.59	1.07	3.67	3.44	4.39	6.99	11.6	14.8	16.8	18.2
20	1.07	1.91	1.70	0.777	3.22	1.14	4.71	2.15	NT	NT	NT	NT	NT	NT
21	0.90	1.8	1.54	0.785	2.88	1.72	4.51	4.59	4.15	9.13	12.9	17.6	20.3	23
22	0.966	1.82	1.69	0.823	3.47	1.74	5.12	8.05	4.63	8.25	14.3	16.1	23.8	28.5

\* AV = Assigned Value; HV = Homogeneity Value; SV = Spiked Value. All values are in µg/kg. Shaded cells are results which returned a questionable or unacceptable z-score.

Table 97 Summary of Participants' Sample S2 Results (part 1)\*

Lab. Code	PFBS	PFPeS	PFHxS	PFHxS (linear)	PFHpS	PFOS	PFOS (linear)	PFNS	PFDS	PFBA	PFPeA	PFHxA	PFHpA
AV	4.02	3.38	2.26	2.19	1.7	2.56	2.54	3.42	2.52	12.1	4.66	5.28	2.90
HV	3.7	3.5	2.19	2.19	1.66	2.31	2.31	3.6	2.5	11.4	4.5	5.0	3.03
SV	4.62	3.47	2.20	2.20	1.76	2.50	2.50	3.55	2.68	10.7	4.60	4.61	2.77
1	3.7	2.5	2	2	1.6	2.2	2.2	2.7	NT	10	3.9	5	2.7
2	4.4	3.8	2.2	2.2	1.7	2.6	2.6	3.8	2.6	12	4.8	5.8	3.1
3	4	4	3	3	2	2	2	4	2	12	6	6	3
4	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
5	NT	NT	2.12	NT	NT	3.09	NT	NT	NT	NT	NT	NT	NT
6	3.527	3.034	1.9	1.9	1.407	2.191	2.191	3.145	2.084	7.393	3.518	4.529	1.831
7	4.82	2.97	2.48	2.48	1.95	2.66	2.66	2.55	0.83	17.75	5.39	6.57	3.76
8	4.10	3.35	1.94	1.94	1.58	2.11	2.11	3.11	2.41	11.9	4.30	4.61	2.88
9	4.01	<0.5	2.01	2.01	1.67	2.84	2.84	3.69	2.52	10.44	4.25	4.81	2.62
10	4.08	4.136	2.55	NT	2.038	3.036	3.036	NT	3.122	17.96	4.384	5.298	3.246
11	4.28	3.39	NT	2.44	1.88	NT	2.88	3.87	2.58	12.17	5.72	6.42	2.65
12	5.48	5.92	2.37	2.37	3.79	2.54	2.54	3.11	2.34	12.82	4.55	5.22	2.95
13	3.5	2.3	1.8	1.7	3.1	2.9	2.9	2.6	6.4	NT	3.6	26	2.0
14	4.1321	NT	1.7933	1.7933	1.4971	2.0827	2.0827	NT	1.9229	NT	6.6309	4.6530	3.10285
15	3.1	5.3	3.3	NT	3.4	4.2	NT	NT	NT	NT	4.4	4.9	3.2
16	4.5	3.4	2.9	NT	1.3	3	NT	9	6.2	15	5.0	6.3	2.1
17	4.417	3.144	1.831	NR	1.589	2.211	NR	3.161	2.357	10.892	4.499	4.889	3.005
19	4.29	4.16	2.80	2.80	2.18	3.04	3.04	4.67	3.09	10.6	4.42	5.35	3.45
20	2.33	3.23	NT	2.43	1.40	2.88	2.88	4.03	3.09	13.0	5.34	5.46	3.43
21	3.73	3.52	2.18	2.18	1.6	2.2	2.2	3.57	2.35	11.7	4.08	4.77	2.83
22	3.68	3.55	1.89	1.89	1.91	2.54	2.54	3.56	2.79	12.2	4.68	4.76	2.77

\* AV = Assigned Value; HV = Homogeneity Value; SV = Spiked Value. All values are in µg/kg. Shaded cells are results which returned a questionable or unacceptable z-score.



Table 98 Summary of Participants' Sample S2 Results (part 2)\*

Lab. Code	PFOA	PFNA	PFDA	PFUdA	PFTTrDA	PFOSA	EtFOSA	EtFOSAA	6:2FTS	GenX	ADONA	9Cl-PF3ONS	11Cl-PF3OUdS
AV	6.07	4.90	2.36	4.06	2.90	12.1	6.7	8.9	4.58	8.51	13.9	18.5	20.7
HV	6.0	4.9	2.24	4.2	2.69	11.7	9.7	8.2	5.0	8.0	17.9	20.4	24.5
SV	5.57	4.64	2.09	3.73	2.78	12.3	7.46	9.26	4.39	8.40	15.8	19.0	22.6
1	5	4.3	2	3.5	NT	12	6	7	4	NT	NT	NT	NT
2	5.4	4.7	2.0	4.6	2.4	12	7.0	9.0	4.3	9.3	14	19	20
3	7	5	3	5	4	18	8	9	5	< 50	16	19	24
4	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
5	5.08	11.79	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
6	5.091	2.691	1.821	3.587	2.292	10.006	3.802	8.22	1.371	3.684	8.693	12.976	17.588
7	6.37	5.59	2.72	2.49	3.16	11.99	NT	8.02	4.83	NT	13.94	18.00	7.44
8	5.37	4.91	2.02	3.73	3.15	11.0	NT	NT	3.42	7.97	16.4	16.0	20.7
9	5.86	4.32	2.2	4.26	3.03	10.69	7.69	10.07	4.6	8.91	8.83	15.78	16.84
10	7.474	4.93	2.824	4.616	3.242	14.01	8.942	9.832	4.78	NT	NT	NT	NT
11	6.71	6.35	2.48	NT	2.8	NT	NT	NT	5	8.73	NT	NT	NT
12	5.68	4.7	2.31	3.9	1.64	NT	NT	NT	NT	8.04	11.48	18.85	15.71
13	6.1	4.8	2.1	6.5	2.6	12	5.9	8.0	4.3	8.8	11	38	35
14	5.3815	4.283	2.0614	3.7318	NT	NT	NT	NT	NT	NT	NT	NT	NT
15	7.3	7.5	3.4	NT	NT	NT	NT	NT	7.4	NT	NT	NT	NT
16	6.0	5.2	2.1	3.8	3.4	0.82	NT	NT	2.0	NT	15	NT	NT
17	5.451	4.001	2.157	3.785	1.979	11.258	NT	NT	3.789	8.571	15.601	16.547	19.315
19	7.89	6.47	3.03	5.89	3.59	15.6	11.7	16.4	5.29	7.83	17.4	23.7	26.0
20	6.72	5.15	2.33	4.26	2.40	10.9	3.69	NT	NT	NT	NT	NT	NT
21	5.52	4.62	2.24	3.99	2.79	10.8	8.01	8.96	4.75	8.07	15.3	19.7	19.9
22	6.42	4.78	2.51	4.16	3.72	13.2	8.36	10.8	5.24	8.84	16.8	25.2	30.2

\* AV = Assigned Value; HV = Homogeneity Value; SV = Spiked Value. All values are in µg/kg. Shaded cells are results which returned a questionable or unacceptable z-score.

Table 99 Summary of Participants' Sample S3 Results (part 1)\*

Lab. Code	PFBS	PFPeS	PFHxS	PFHxS (linear)	PFHpS	PFOS	PFOS (linear)	PFNS	PFDS	PFBA	PFPeA	PFHxA	PFHpA
AV	1.19	2.89	5.94	5.96	1.29	1.80	1.78	1.68	1.58	6.47	2.27	6.86	7.34
HV	1.06	2.87	5.6	5.6	1.18	1.50	1.50	1.73	1.63	5.6	2.12	6.6	7.1
SV	1.46	3.22	6.5	6.50	1.41	1.87	1.87	1.88	1.89	6.77	2.44	6.89	7.84
1	1.1	2.7	5.4	5.4	1	1.4	1.4	< 2	< 2	5.4	2	6	6.6
2	1.4	3.6	6.4	6.4	1.4	2.1	2.1	1.9	1.6	6.8	2.4	7.2	7.8
3	1	4	8	8	1	2	2	2	2	7	3	8	8
4	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
5	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
6	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
7	1.36	2.82	6.34	6.34	1.29	1.83	1.79	1.71	1.33	7.01	2.61	7.69	9.22
8	1.19	2.85	5.76	5.76	1.27	1.50	1.50	1.54	1.51	6.21	2.15	6.28	6.64
9	1.03	1.93	5.25	5.25	1.66	1.91	1.91	1.53	1.3	5.12	1.85	6.39	6.5
10	1.224	3.324	6.34	NT	1.446	2.112	2.112	NT	1.856	7.434	2.124	6.25	7.126
11	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
12	1.66	3.1	6.51	6.51	1.44	1.77	1.77	1.55	1.49	8.57	2.32	6.84	7.61
13	1.2	2.2	5.0	4.7	2.3	3.0	3.0	1.4	2.6	NT	6.3	7.8	5.0
14	1.0868	NT	5.5033	5.5033	1.1228	1.5304	1.5304	NT	1.3273	NT	2.1592	6.5115	6.5431
15	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
16	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
17	1.312	2.949	5.96	NR	1.377	1.953	NR	1.825	1.668	2.839	2.283	6.831	8.619
19	1.19	2.73	5.72	5.72	1.54	<2.38	<2.38	1.14	0.827	5.96	2.32	6.58	7.29
20	1.03	2.68	NT	6.58	1.09	1.95	1.95	1.92	2.01	6.70	2.65	7.69	8.44
21	1.1	2.89	6.18	6.18	1.21	1.49	1.49	1.78	1.8	5.51	1.95	5.94	6.91
22	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

\* AV = Assigned Value; HV = Homogeneity Value; SV = Spiked Value. All values are in µg/kg. Shaded cells are results which returned a questionable or unacceptable z-score.

Table 100 Summary of Participants' Sample S3 Results (part 2)\*

Lab. Code	PFOA	PFNA	PFDA	PFUdA	PFDoA	PFTeDA	PFOSA	EtFOSAA	8:2FTS	GenX	ADONA	9Cl-PF3ONS	11Cl-PF3OUdS
AV	1.00	1.84	1.60	0.763	3.55	1.52	5.19	4.52	9.05	13.6	16.6	19.5	20.8
HV	1.01	1.99	1.66	0.82	3.5	1.70	4.7	3.9	10.4	13.6	18.1	23.4	22.9
SV	0.951	1.96	1.64	0.781	3.92	1.76	5.58	4.89	9.39	14.7	18.4	22.8	27.7
1	< 5	< 2	< 5	< 2	< 5	< 5	4.9	< 5	< 1	NT	NT	NT	NT
2	1.1	2.1	1.7	0.77	3.9	< 0.50	5.6	NR	11	15	18	23	28
3	1	2	2	< 2	4	< 5	7	5	10	< 50	16	19	23
4	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
5	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
6	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
7	1.00	2.09	1.67	<0.1	3.45	1.50	5.06	3.97	9.03	NT	15.67	21.43	19.39
8	0.916	1.87	1.44	0.712	3.40	1.44	4.80	NT	8.29	12.9	16.5	18.7	21.1
9	0.87	1.49	1.42	0.7	3.51	1.41	4.16	3.82	7.76	12.36	4.96	15.53	16.6
10	1.102	1.63	1.728	<1	3.47	<2	5.946	4.384	9.582	NT	NT	NT	NT
11	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
12	0.95	1.91	1.69	0.74	3.63	1.86	NT	NT	NT	14.28	14.18	19.77	18.6
13	1.3	1.6	1.3	0.8	NT	0.8	4.1	6.5	8.3	21	11	15	19
14	0.8711	1.8586	1.4458	0.6982	3.3080	1.4011	NT	NT	NT	NT	NT	NT	NT
15	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
16	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
17	1.07	1.781	1.665	0.821	3.695	1.504	6.197	NT	9.359	14.653	18.686	21.876	21.34
19	1.00	1.71	1.54	0.751	3.62	1.51	5.06	4.79	8.21	13.2	22.6	16.9	10.0
20	1.09	2.06	1.67	0.807	3.51	1.76	5.08	NT	NT	NT	NT	NT	NT
21	0.91	1.83	1.66	0.834	3.15	1.64	4.89	4.02	9.4	13	17.1	23.3	22.5
22	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

\* AV = Assigned Value; HV = Homogeneity Value; SV = Spiked Value. All values are in µg/kg. Shaded cells are results which returned a questionable or unacceptable z-score.

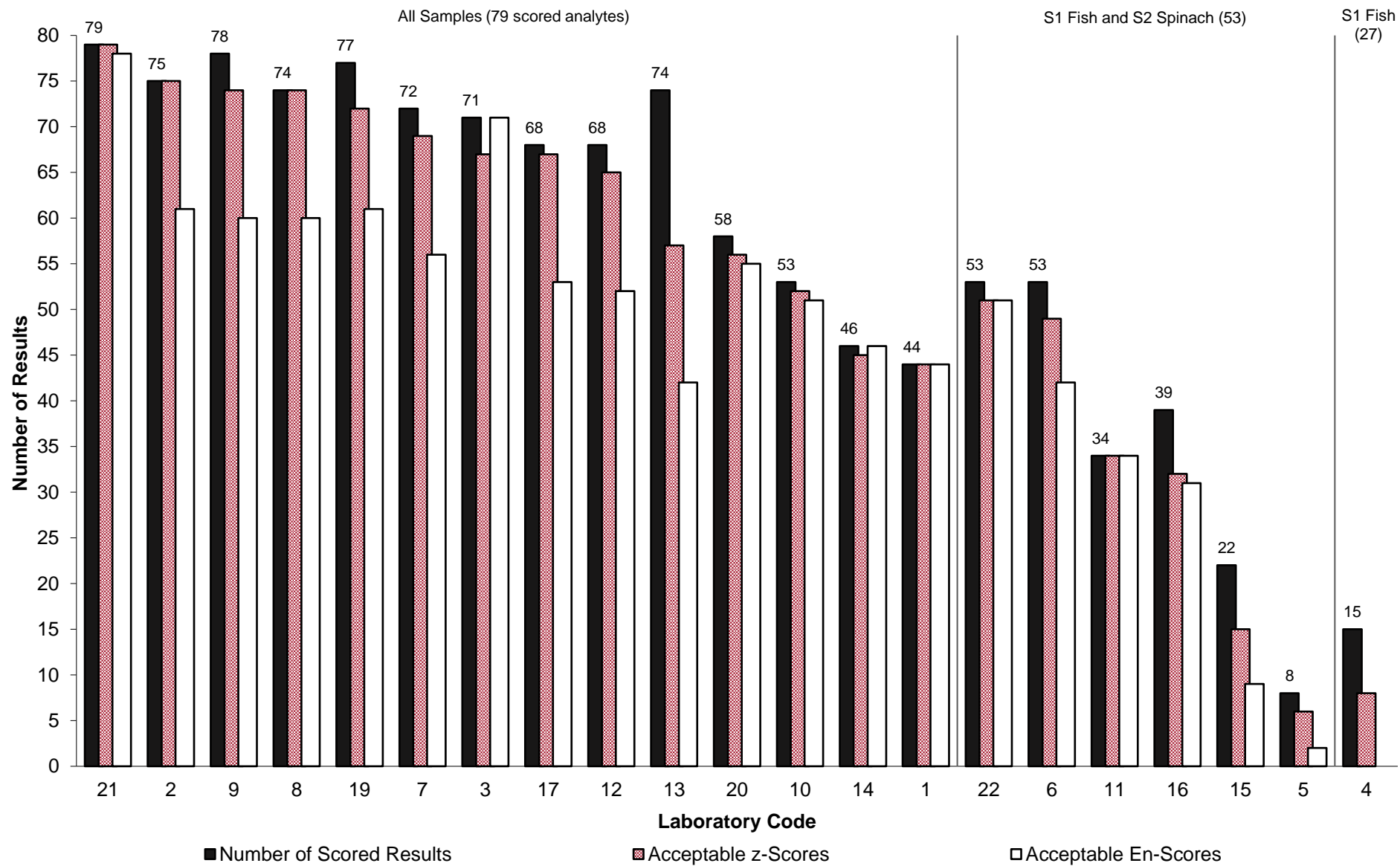


Figure 142 Summary of Participants' Performance

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

NMI has run PFAS in Biota and Food PT studies since 2016. A summary of participation and reported results rates over the last eight studies (2016 to 2023) is presented in Figure 143. Proportions of PFAS analysed and numeric results reported have remained steady over this period, despite the increased number of spiked analytes as compared to the original studies.

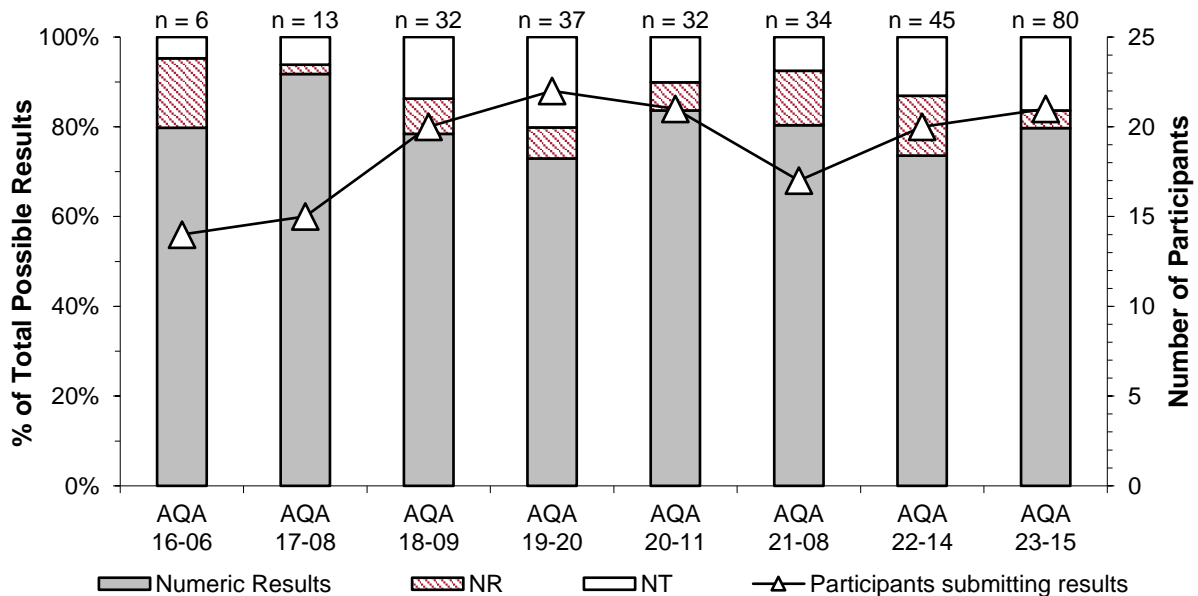


Figure 143 Summary of Participation and Reported Results in PFAS in Biota and Food PT Studies (n = number of spiked analytes)

A summary of the acceptable 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 eight studies (2016 to 2023) is presented in Figure 144. The target SD used to calculate z-scores has been kept constant at 20% PCV, which enables comparison between different studies. Proportions of acceptable scores has remained relatively consistent, though this study had the highest proportion of acceptable z-scores so far at 94%. The average proportion of acceptable scores over this period being 89% for z-scores and 77% for  $E_n$ -scores.

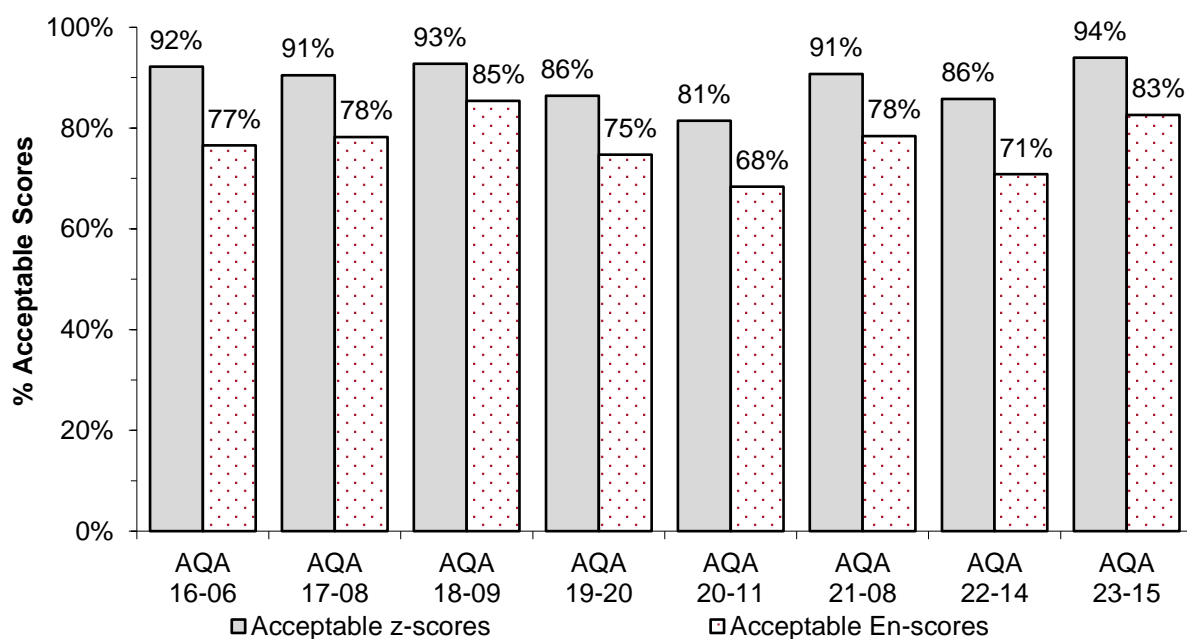


Figure 144 Summary of Participants' Performance for PFAS in Biota and Food PT Studies

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 acceptable scores for these analytes over the last eight studies is presented in Figure 145.

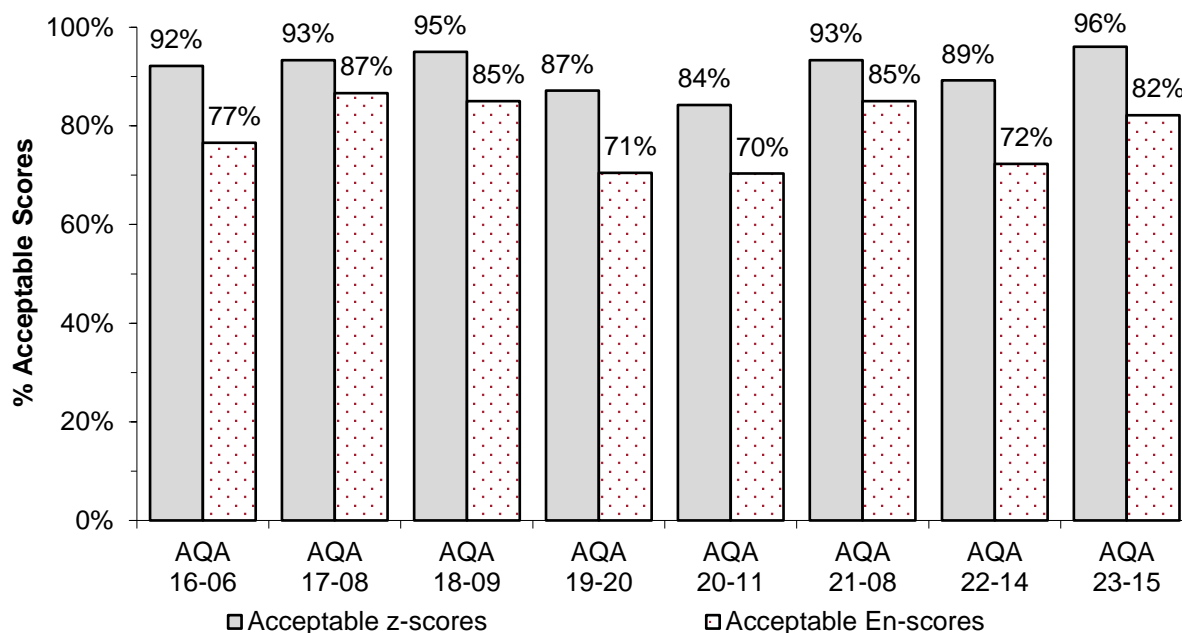


Figure 145 Summary of Participants' Performance for PFOS and PFOA in Biota and Food PT Studies

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

As discussed in Section 6.2, it is a requirement of ISO/IEC 17025 that laboratories report their uncertainty. Figure 146 presents a summary of relative uncertainties as reported by participants over the last eight studies (2016 to 2023). Over this period, most numeric results were reported with uncertainties (97%), despite only 59% of participants reporting that they were accredited to ISO/IEC 17025. A few participants are still reporting non-numeric results with numeric uncertainties.

Over the last four studies specifically, there has been an increased number of participants reporting potentially unrealistically small or large relative uncertainties for routine PFAS measurements (i.e. less than 10% or larger than 50% relative). Participants reporting results with acceptable z-scores, but with smaller relative uncertainties and unacceptable  $E_n$ -scores, may need to assess whether their uncertainties have been underestimated.

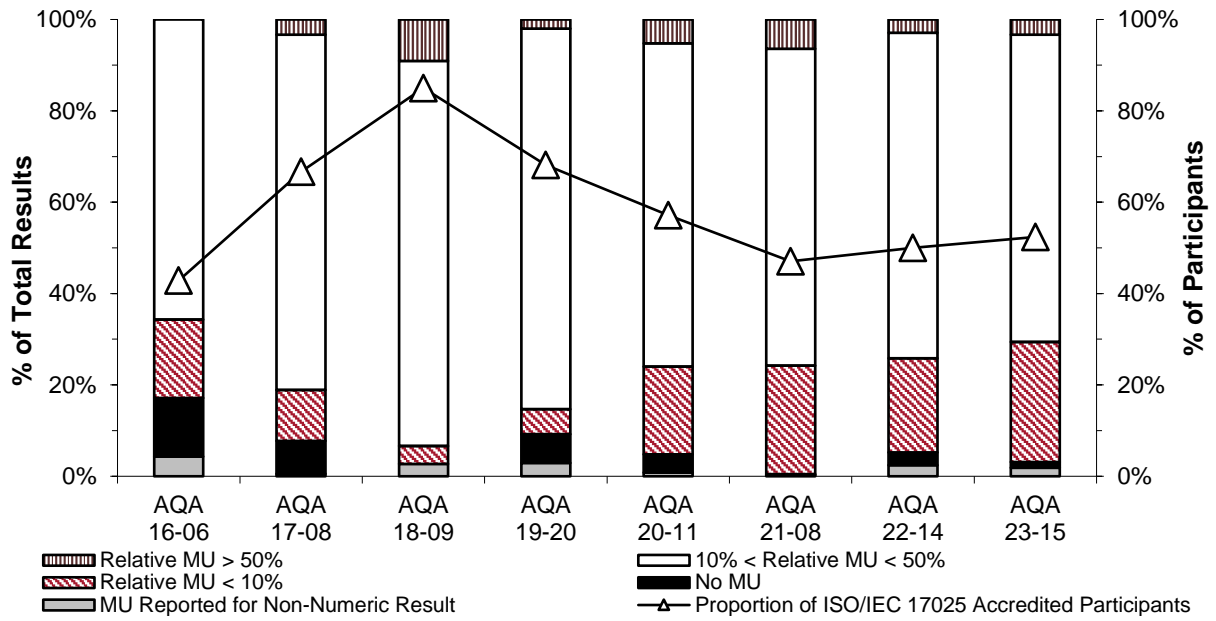


Figure 146 Summary of Participants' Relative Uncertainties for PFAS in Biota and Food PT Studies

## 7 REFERENCES

Please note that for all undated references, the latest edition of the referenced document (including any amendments) applies.

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- [12] Thompson, M. and Fearn, T., 2001, ‘A new test for ‘sufficient homogeneity’’, *Analyst*, vol. 126, pp. 1414-1417.



## APPENDIX 1 SAMPLE PREPARATION

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

**Sample S2:** Organic spinach bought from a local organic fruit and vegetable wholesaler was blended to yield a puree. The puree was passed through an 850 µm sieve. This was then spiked with PFAS analytes and stirred for at least two hours. Portions of 30 mL were then dispensed into sample tubes. The tubes were labelled, shrink-wrapped, and then stored at -20 °C prior to dispatch.

**Sample S3:** Bovine liver purchased from a local butcher was blended to yield a puree. The puree was spiked with PFAS analytes, before being further blended and mixed. Portions of 5 g were then packed into sample tubes. The tubes were labelled, shrink-wrapped, and then stored at -80 °C prior to dispatch.

## APPENDIX 2 HOMOGENEITY AND STABILITY ASSESSMENT

### A2.1 Sample S1 Fish Homogeneity and Stability Assessment

Samples were analysed by the NMI Australian Ultra Trace Laboratory. For homogeneity testing, measurements were made under repeatability conditions in a random order. Samples were prepared in duplicate by accurately weighing 1 g of the sample then spiking with 100 µL of labelled internal standard in methanol. The samples were extracted by overnight tumbling in alkaline methanol (0.01 N potassium hydroxide), then centrifuged and a portion was purified by passing through activated carbon (SUPLCLEAN ENVI-CARB, 500 mg, 120-400 Mesh) eluted using methanol. After evaporation under nitrogen, the extract was reconstituted to 600 µL in mobile phase and spiked with 20 µL labelled recovery standard in methanol. All chemicals were analytical reagents or LCMS grade solvents. Instrument analysis was performed using an Ultra Performance Liquid Chromatography (UPLC) coupled with a Liquid Chromatography Qtrap Mass Spectrometer (ABSciex 6500+), operating in multiple reaction monitoring mode. 2 µL of extract was injected onto a Waters Acquity BEH C18 column (2.1 mm x 100 mm x 1.7 µm, 130 Å) with a mobile phase gradient consisting of water:methanol (2 mM ammonium acetate). Two mass transitions were monitored for each target analyte and labelled internal standard, and abundance ratios checked. The instrument mass accuracy was calibrated annually during preventative maintenance, and the six point calibration curve established for each analytical batch. A solvent batch blank was extracted and analysed with each batch, and sample results were reported if results were at least three times the level of any analyte detected in the batch blank. Quantification was based on the use of the labelled internal standards using relative retention factors from the multipoint calibration, and was corrected for internal standard recoveries. The analysis was based on USEPA Draft Method 1633 and used calibration, internal and recovery standards supplied by Wellington Laboratories.

#### Homogeneity Testing

Sample S1 was prepared using a process previously demonstrated to produce sufficiently homogeneous PT samples.<sup>5</sup> Partial homogeneity testing was also conducted for this sample; three containers were analysed in duplicate, and the average of the results was reported as the homogeneity value in Section 5. The results of this testing are presented in Tables 101 to 127.

Table 101 Sample S1 PFBS Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	1.01	1.03
26	1.02	1.06
47	1.13	1.07
Mean	1.05	
CV	4.2%	

Table 102 Sample S1 PFPeS Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	2.64	3.02
26	3.45	3.15
47	3.25	3.13
Mean	3.11	
CV	8.7%	

Table 103 Sample S1 PFHxS Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	4.7	5.4
26	5.3	5.3
47	5.2	6.3
Mean	5.4	
CV	9.7%	

Table 104 Sample S1 PFHxS (linear) Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	4.7	5.4
26	5.3	5.3
47	5.2	6.3
Mean	5.4	
CV	9.7%	

Table 105 Sample S1 PFHpS Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	1.15	1.23
26	1.12	1.23
47	1.09	1.21
Mean	1.17	
CV	5.1%	

Table 106 Sample S1 PFOS Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	1.31	1.48
26	1.42	1.51
47	1.97	1.58
Mean	1.55	
CV	15%	

Table 107 Sample S1 PFOS (linear) Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	1.31	1.48
26	1.42	1.51
47	1.97	1.58
Mean	1.55	
CV	15%	

Table 108 Sample S1 PFNS Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	1.51	1.40
26	1.43	1.44
47	1.78	1.51
Mean	1.51	
CV	9.2%	

Table 109 Sample S1 PFDS Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	1.17	1.18
26	1.31	1.40
47	2.01	1.58
Mean	1.44	
CV	22%	

Table 110 Sample S1 PFBA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	5.6	5.5
26	5.4	5.6
47	6.5	6.2
Mean	5.8	
CV	7.6%	

Table 111 Sample S1 PFPeA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	2.08	2.17
26	2.02	2.01
47	2.37	2.62
Mean	2.21	
CV	11%	

Table 112 Sample S1 PFHxA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	6.4	6.3
26	6.0	5.5
47	6.8	7.1
Mean	6.3	
CV	9.0%	

Table 113 Sample S1 PFHpA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	6.8	7.0
26	6.9	7.3
47	7.5	7.5
Mean	7.2	
CV	4.6%	

Table 114 Sample S1 PFOA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	0.98	0.99
26	1.04	1.00
47	1.13	1.01
Mean	1.03	
CV	5.4%	

Table 115 Sample S1 PFNA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	1.95	1.73
26	1.85	1.89
47	1.96	2.05
Mean	1.91	
CV	5.7%	

Table 116 Sample S1 PFDA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	1.54	1.56
26	1.61	1.66
47	1.89	1.69
Mean	1.66	
CV	7.7%	

Table 117 Sample S1 PFUdA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	0.75	0.73
26	0.81	0.81
47	0.83	0.94
Mean	0.81	
CV	9.1%	

Table 118 Sample S1 PFDoA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	3.02	3.10
26	2.86	3.21
47	3.14	3.21
Mean	3.09	
CV	4.3%	

Table 119 Sample S1 PFTeDA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	1.70	1.74
26	1.97	1.83
47	2.11	2.11
Mean	1.91	
CV	9.5%	

Table 120 Sample S1 PFOSA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	4.2	4.3
26	4.6	3.8
47	4.2	5.1
Mean	4.3	
CV	9.6%	

Table 121 Sample S1 EtFOSA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	4.1	3.4
26	4.2	3.9
47	4.4	4.7
Mean	4.1	
CV	10%	

Table 122 Sample S1 EtFOSAA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	4.3	3.8
26	4.5	4.2
47	5.1	5.0
Mean	4.5	
CV	11%	

Table 123 Sample S1 8:2FTS Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	9.6	10.1
26	10.3	10.0
47	11.0	11.0
Mean	10.3	
CV	5.4%	

Table 124 Sample S1 GenX Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	12.4	11.7
26	12.2	12.7
47	12.7	13.0
Mean	12.4	
CV	3.9%	

Table 125 Sample S1 ADONA Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	13.5	14.8
26	14.1	15.9
47	14.3	14.5
Mean	14.5	
CV	5.4%	



Table 126 Sample S1 9CI-PF3ONS Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	19.9	20.4
26	18.2	22.9
47	18.8	18.0
Mean	19.7	
CV	9.3%	

Table 127 Sample S1 11CI-PF3OUdS Partial Homogeneity Results

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
5	20.7	22.0
26	22.8	23.3
47	21.3	22.6
Mean	22.1	
CV	4.4%	

### Comparison of Results and Container Numbers

A comparison of *z*-scores obtained to the container number analysed by participants for all scored analytes is presented for information in Figure 147 (results have only been included if the participant was sent one sample set). Participants' results in this study gave no reason to question the samples' homogeneity.

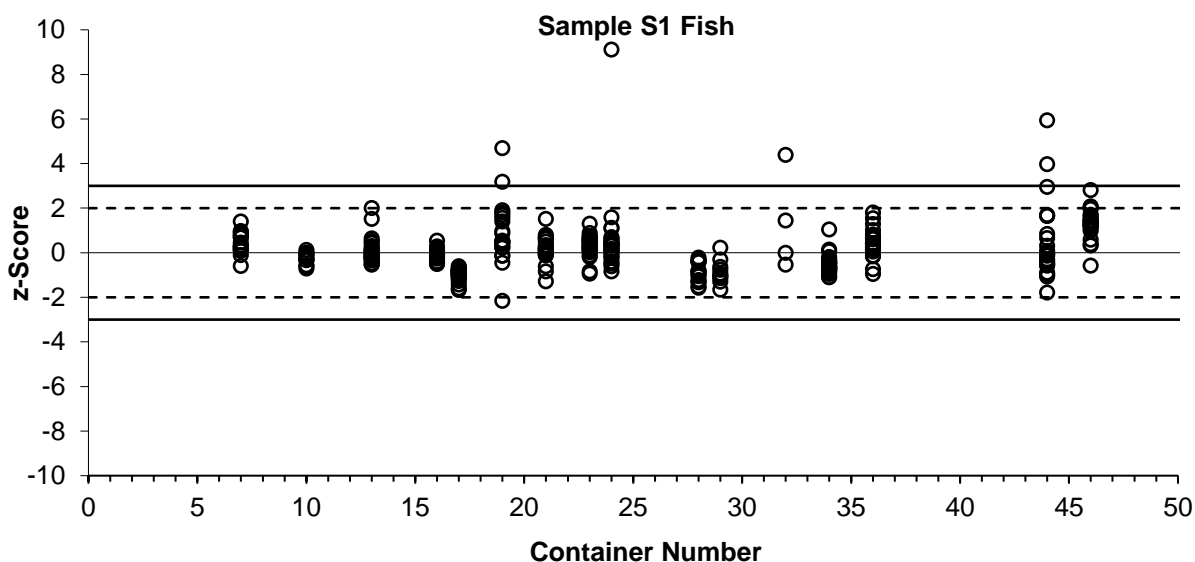


Figure 147 Sample S1 Fish *z*-Score vs Container Number

### Stability Testing

Sample S1 was packaged, stored and dispatched using a process previously demonstrated to produce sufficiently stable PT samples.<sup>5</sup> No additional stability testing was performed for this study.

## Comparison of Results and Transit Time

A comparison of  $z$ -scores obtained to the number of days the samples spent in transit for all scored analytes is presented in Figure 148.

For one international participant, there were substantial customs delays with their sample delivery, resulting in a significantly longer time in transit. Nevertheless, participants' results in this study gave no reason to question the samples' transportation stability.

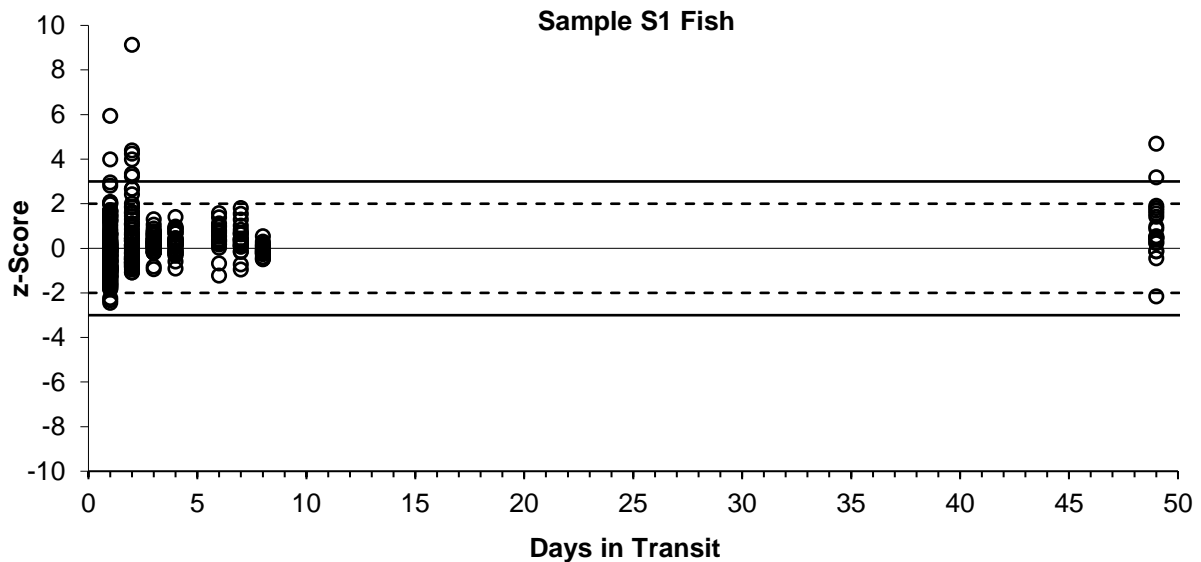


Figure 148 Sample S1 Fish  $z$ -Score vs Days in Transit

### A2.2 Sample S2 Spinach Homogeneity and Stability Assessment

Samples were analysed by the NMI Australian Ultra Trace Laboratory using the same process as described in Section A2.1.

#### Homogeneity Testing

Sample S2 was prepared using a process previously demonstrated to produce sufficiently homogeneous PT samples of similar analytes and matrices. Homogeneity testing was also conducted for this sample.

Homogeneity checks were based on that described by Thompson and Fearn,<sup>12</sup> which is also the procedure as described in the International Harmonized Protocol.<sup>4</sup> The results are presented in Tables 128 to 153. Samples were found to be sufficiently homogeneous for use in a PT study with a target SD (as PCV) of 20%.

Table 128 Sample S2 PFBS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	3.6	3.6
21	3.7	3.6
30	3.6	3.5
36	3.6	3.8
46	3.6	3.6
62	3.7	3.8
71	3.8	3.7
Mean	3.7	
CV	2.3%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.569	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.107	0.500	<b>Pass</b>
$s^2_{sam}$	0.001	0.110	<b>Pass</b>

Table 129 Sample S2 PFPeS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	3.6	3.3
21	3.6	3.3
30	3.4	3.3
36	3.3	3.5
46	3.3	3.5
62	3.7	3.5
71	3.4	3.8
Mean	3.5	
CV	4.7%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.376	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.251	0.500	<b>Pass</b>
$s^2_{sam}$	0.000	0.135	<b>Pass</b>

Table 130 Sample S2 PFHxS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	2.20	2.33
21	2.07	1.95
30	2.26	2.28
36	2.19	2.17
46	2.30	2.28
62	2.01	2.22
71	2.21	2.22
Mean	2.19	
CV	5.1%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.575	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.169	0.500	<b>Pass</b>
$s^2_{sam}$	0.007	0.044	<b>Pass</b>

Table 131 Sample S2 PFHxS (linear) Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	2.20	2.33
21	2.07	1.95
30	2.26	2.28
36	2.19	2.17
46	2.30	2.28
62	2.01	2.22
71	2.21	2.22
Mean	2.19	
CV	5.1%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.575	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.169	0.500	<b>Pass</b>
$s^2_{sam}$	0.007	0.044	<b>Pass</b>

Table 132 Sample S2 PFHpS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	1.62	1.70
21	1.59	1.59
30	1.71	1.81
36	1.55	1.47
46	1.72	1.60
62	1.64	1.69
71	1.78	1.71
Mean	1.66	
CV	5.6%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.323	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.170	0.500	<b>Pass</b>
$s^2_{sam}$	0.006	0.025	<b>Pass</b>

Table 133 Sample S2 PFOS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	2.53	2.38
21	2.23	2.29
30	2.13	2.18
36	2.36	2.26
46	2.20	2.23
62	2.25	2.46
71	2.48	2.36
Mean	2.31	
CV	5.2%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.450	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.181	0.500	<b>Pass</b>
$s^2_{sam}$	0.008	0.050	<b>Pass</b>

Table 134 Sample S2 PFOS (linear) Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	2.53	2.38
21	2.23	2.29
30	2.13	2.18
36	2.36	2.26
46	2.20	2.23
62	2.25	2.46
71	2.48	2.36
Mean	2.31	
CV	5.2%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.450	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.181	0.500	<b>Pass</b>
$s^2_{sam}$	0.008	0.050	<b>Pass</b>

Table 135 Sample S2 PFNS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	4.0	3.5
21	3.8	3.8
30	3.4	3.3
36	3.6	3.4
46	3.3	3.3
62	3.4	3.4
71	4.1	3.8
Mean	3.6	
CV	7.6%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.526	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.241	0.500	<b>Pass</b>
$s^2_{sam}$	0.048	0.140	<b>Pass</b>

Table 136 Sample S2 PFDS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	2.8	2.5
21	2.5	2.5
30	2.3	2.4
36	2.6	2.3
46	2.5	2.6
62	2.4	2.7
71	2.9	2.5
Mean	2.5	
CV	7.2%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.286	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.365	0.500	<b>Pass</b>
$s^2_{sam}$	0.000	0.097	<b>Pass</b>

Table 137 Sample S2 PFBA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	11.2	12.0
21	11.3	10.8
30	11.3	10.9
36	11.4	11.6
46	11.9	11.8
62	11.5	11.9
71	11.3	11.3
Mean	11.4	
CV	3.3%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.554	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.138	0.500	<b>Pass</b>
$s^2_{sam}$	0.045	1.132	<b>Pass</b>

Table 138 Sample S2 PFPeA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7*	4.6	4.1
21	4.6	4.5
30	4.5	4.5
36	4.5	4.3
46	4.4	4.4
62	4.6	4.8
71	4.7	4.6
Mean	4.5	
CV	3.8%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.418	0.781	<b>Pass</b>
$s_{an}/\sigma$	0.093	0.500	<b>Pass</b>
$s^2_{sam}$	0.013	0.177	<b>Pass</b>

\* Results from container 7 were not included in the test for homogeneity, being identified as Cochran outliers due to the difference between replicates.<sup>12</sup>

Table 139 Sample S2 PFHxA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	5.0	5.0
21	5.2	5.0
30	4.7	4.9
36	5.0	4.9
46	4.4	4.8
62	5.1	5.0
71	5.1	5.4
Mean	5.0	
CV	4.9%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.513	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.177	0.500	<b>Pass</b>
$s^2_{sam}$	0.030	0.230	<b>Pass</b>

Table 140 Sample S2 PFHpA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	3.13	3.00
21	3.25	2.89
30	3.15	3.15
36	3.15	2.96
46	2.75	2.86
62	3.04	2.92
71	2.96	3.21
Mean	3.03	
CV	4.9%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.477	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.230	0.500	<b>Pass</b>
$s^2_{sam}$	0.003	0.097	<b>Pass</b>

Table 141 Sample S2 PFOA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	6.1	5.9
21	6.0	5.5
30	5.9	5.9
36	5.9	5.7
46	5.5	5.6
62	6.5	6.6
71	5.9	6.4
Mean	6.0	
CV	5.6%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.463	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.175	0.500	<b>Pass</b>
$s^2_{sam}$	0.072	0.330	<b>Pass</b>

Table 142 Sample S2 PFNA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	4.9	5.1
21	4.7	5.2
30	4.4	4.8
36	4.8	4.3
46	4.9	4.6
62	5.1	5.6
71	4.8	4.9
Mean	4.9	
CV	6.4%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.250	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.252	0.500	<b>Pass</b>
$s^2_{sam}$	0.039	0.266	<b>Pass</b>

Table 143 Sample S2 PFDA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	2.16	2.04
21	2.21	2.20
30	2.16	2.28
36	2.51	2.04
46	2.16	2.29
62	2.14	2.49
71	2.34	2.38
Mean	2.24	
CV	6.6%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.565	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.372	0.500	<b>Pass</b>
$s^2_{sam}$	0.000	0.078	<b>Pass</b>

Table 144 Sample S2 PFUdA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	4.2	4.1
21	4.2	4.0
30	4.5	4.3
36	4.3	4.3
46	4.2	4.2
62	4.0	4.4
71	4.2	4.1
Mean	4.2	
CV	3.3%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.619	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.137	0.500	<b>Pass</b>
$s^2_{sam}$	0.006	0.153	<b>Pass</b>

Table 145 Sample S2 PFTrDA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	2.28	2.48
21	2.84	2.41
30	2.68	2.96
36	2.67	2.66
46	2.76	2.94
62	2.79	2.81
71	2.70	2.73
Mean	2.69	
CV	7.1%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.549	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.288	0.500	<b>Pass</b>
$s^2_{sam}$	0.014	0.089	<b>Pass</b>



Table 146 Sample S2 PFOSA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	11.3	10.9
21	12.6	10.9
30	12.5	12.2
36	12.0	11.7
46	11.3	11.5
62	10.7	12.3
71	11.8	11.5
Mean	11.7	
CV	5.3%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.470	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.281	0.500	<b>Pass</b>
$s^2_{sam}$	0.000	1.643	<b>Pass</b>

Table 147 Sample S2 EtFOSA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7*	7.3	9.9
21	10.1	11.0
30	9.9	9.6
36	9.5	10.2
46	9.1	9.8
62	10.2	10.3
71	9.4	10.0
Mean	9.7	
CV	8.6%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.402	0.781	<b>Pass</b>
$s_{an}/\sigma$	0.223	0.500	<b>Pass</b>
$s^2_{sam}$	0.062	1.116	<b>Pass</b>

\* Results from container 7 were not included in the test for homogeneity, being identified as Cochran outliers due to the difference between replicates.<sup>12</sup>

Table 148 Sample S2 EtFOSAA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	8.9	7.6
21	8.4	7.9
30	8.5	8.0
36	8.6	8.1
46	7.4	8.0
62	8.1	8.1
71	8.4	8.9
Mean	8.2	
CV	5.4%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.531	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.288	0.500	<b>Pass</b>
$s^2_{sam}$	0.000	0.830	<b>Pass</b>

Table 149 Sample S2 6:2FTS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	5.3	4.5
21	4.4	4.9
30	5.2	4.8
36	5.1	5.0
46	4.7	5.2
62	4.8	5.2
71	5.1	5.2
Mean	5.0	
CV	5.9%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.357	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.330	0.500	<b>Pass</b>
$s^2_{sam}$	0.000	0.338	<b>Pass</b>

Table 150 Sample S2 GenX Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	8.0	7.7
21	8.0	7.9
30	8.0	7.9
36	8.0	7.9
46	7.9	8.1
62	8.1	8.4
71	8.2	8.3
Mean	8.0	
CV	2.2%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.368	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.085	0.500	<b>Pass</b>
$s^2_{sam}$	0.014	0.512	<b>Pass</b>

Table 151 Sample S2 ADONA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	17.4	18.2
21	18.3	18.0
30	18.2	18.2
36	17.1	16.9
46	17.1	17.4
62	18.1	19.6
71	17.2	18.9
Mean	17.9	
CV	4.2%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.490	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.179	0.500	<b>Pass</b>
$s^2_{sam}$	0.177	3.009	<b>Pass</b>

Table 152 Sample S2 9Cl-PF3ONS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	21.0	21.2
21	19.7	20.2
30	19.8	20.4
36	21.0	19.5
46	18.4	21.2
62	19.9	22.7
71	18.3	21.8
Mean	20.4	
CV	6.0%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.403	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.362	0.500	<b>Pass</b>
$s^2_{sam}$	0.000	6.261	<b>Pass</b>

Table 153 Sample S2 11Cl-PF3OUdS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
7	21.9	25.1
21	26.1	25.4
30	24.7	26.7
36	23.7	23.1
46	22.6	24.5
62	25.2	25.2
71	23.6	25.5
Mean	24.5	
CV	5.6%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.460	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.260	0.500	<b>Pass</b>
$s^2_{sam}$	0.291	6.862	<b>Pass</b>

### Comparison of Results and Container Numbers

A comparison of z-scores obtained to the container number analysed by participants for all scored analytes is presented for information in Figure 149 (results have only been included if the participant was sent one sample set). Participants' results in this study gave no reason to question the samples' homogeneity.

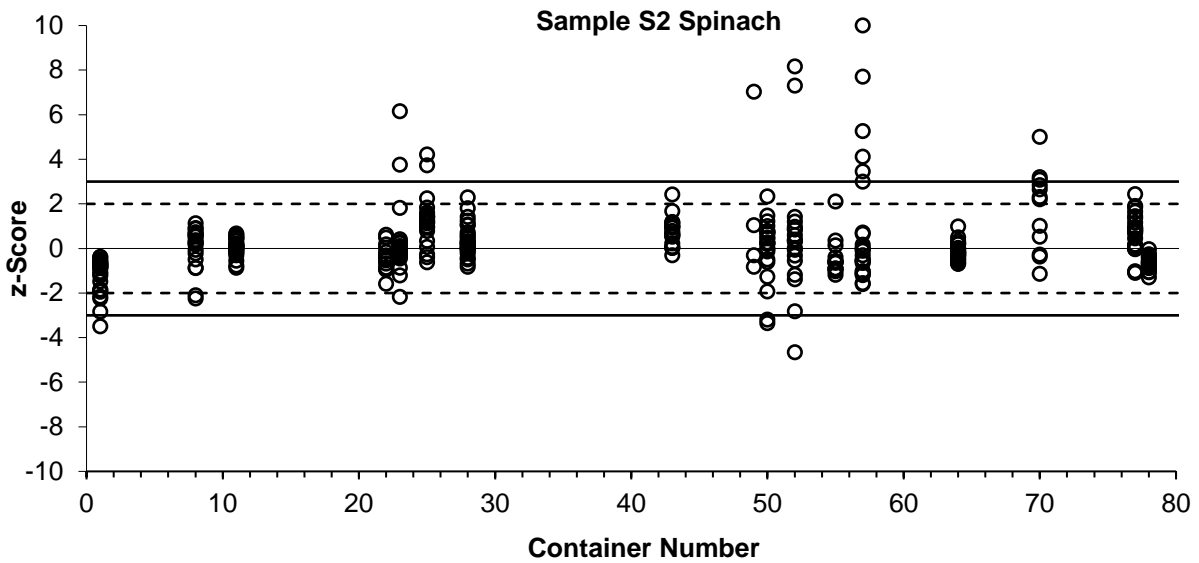


Figure 149 Sample S2 Spinach z-Score vs Container Number

### Stability Testing

Sample S2 was packaged, stored and dispatched using a process previously demonstrated to produce sufficiently stable PT samples of similar analytes and matrices. Stability testing was also conducted for this sample.

The spinach samples were analysed at an initial time point in August 2023. At sample dispatch (September 2023), samples were set aside and packaged in the same way as the samples dispatched to participants. This was stored at ambient conditions until all samples had been delivered to participants (October 2023), to reflect transportation stability. Additional samples were stored at freezer temperature, to reflect long-term storage temperature at a participant’s laboratory; samples were taken for analysis at the conclusion of the PT study (November 2023).

Results were in good agreement with each other and the assigned value within their respective uncertainties (Figures 150 to 175; T = Transportation Stability, F = Freezer Stability, AV = Assigned Value). The samples were also shown to be sufficiently stable when assessed against the criteria specified in ISO 13528.<sup>6</sup>

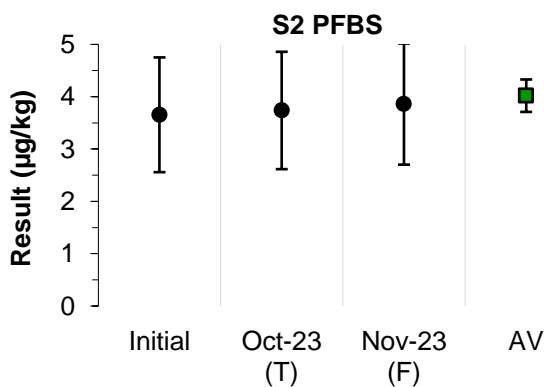


Figure 150 S2 PFBS Stability Testing

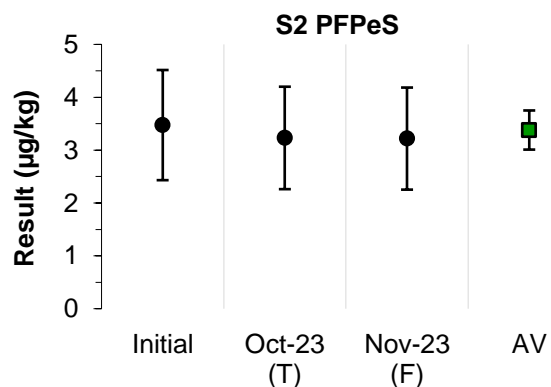


Figure 151 S2 PFPeS Stability Testing

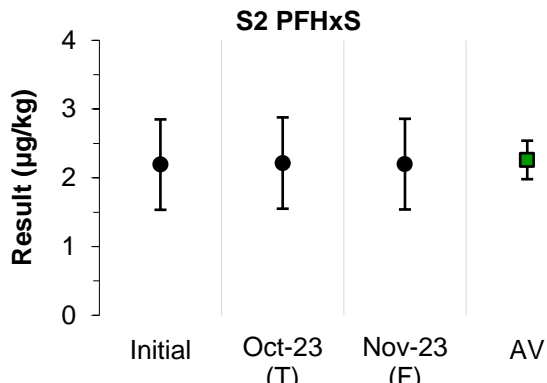


Figure 152 S2 PFHxS Stability Testing

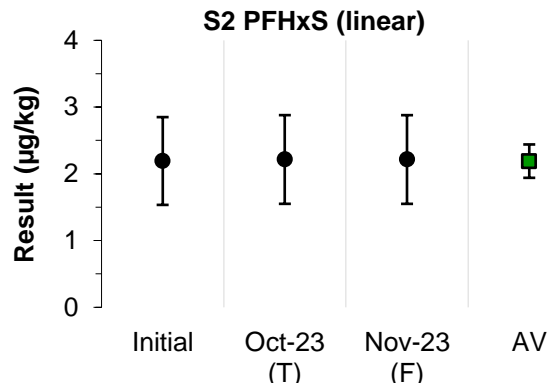


Figure 153 S2 PFHxS (linear) Stability Testing

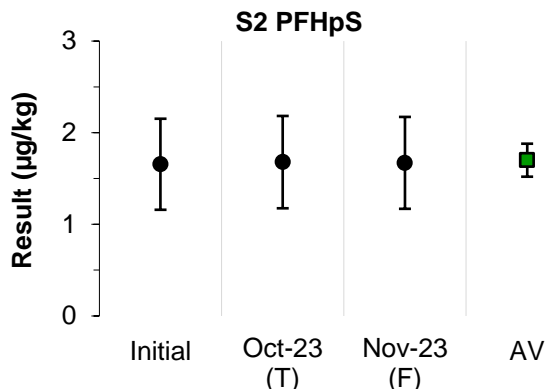


Figure 154 S2 PFHpS Stability Testing

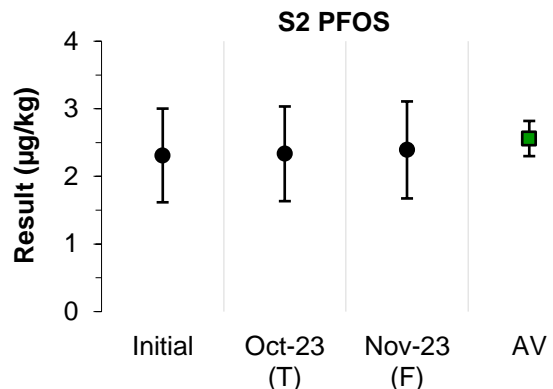


Figure 155 S2 PFOS Stability Testing

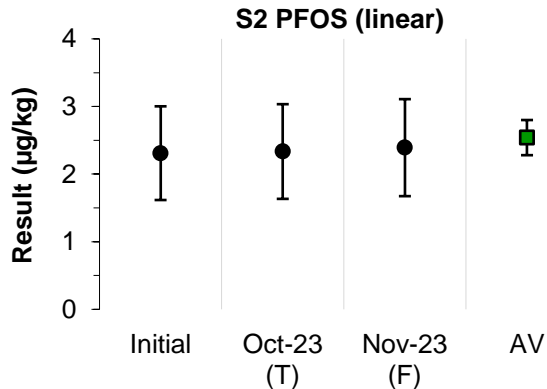


Figure 156 S2 PFOS (linear) Stability Testing

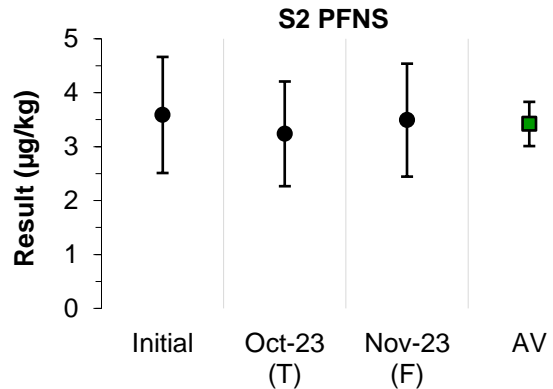


Figure 157 S2 PFNS Stability Testing

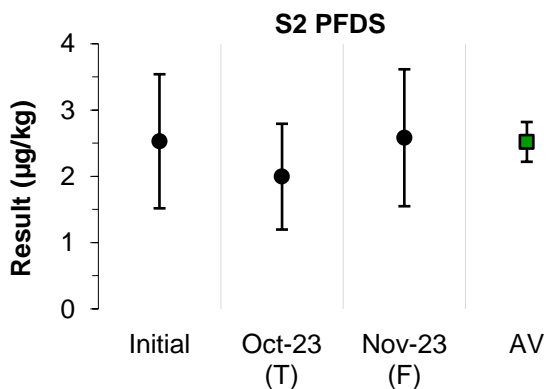


Figure 158 S2 PFDS Stability Testing

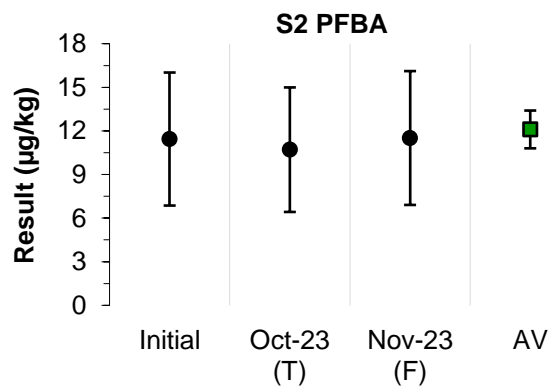


Figure 159 S2 PFBA Stability Testing

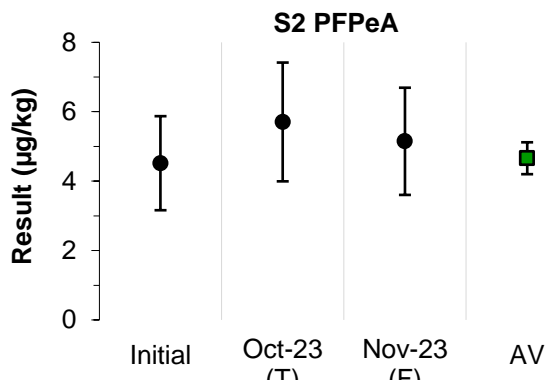


Figure 160 S2 PFPeA Stability Testing

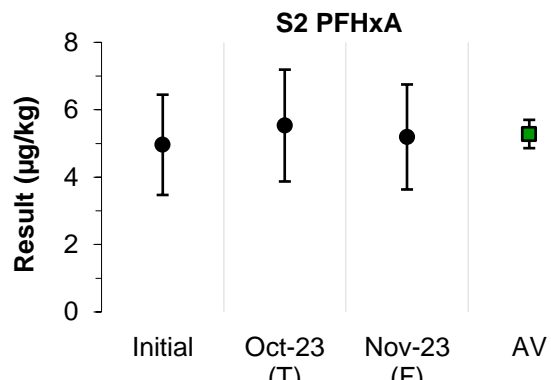


Figure 161 S2 PFHxA Stability Testing

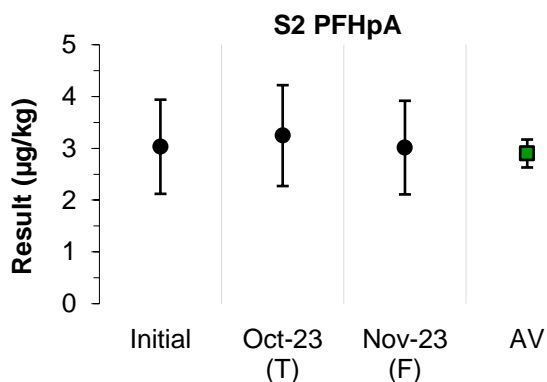


Figure 162 S2 PFHpA Stability Testing

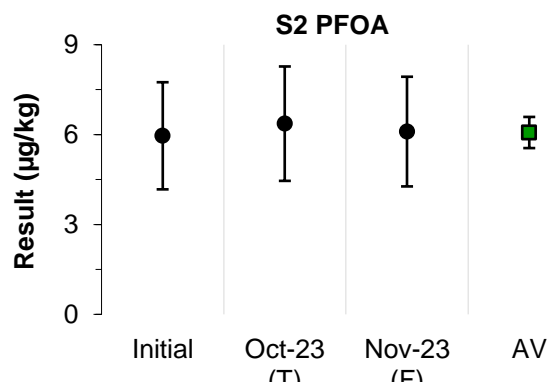


Figure 163 S2 PFOA Stability Testing

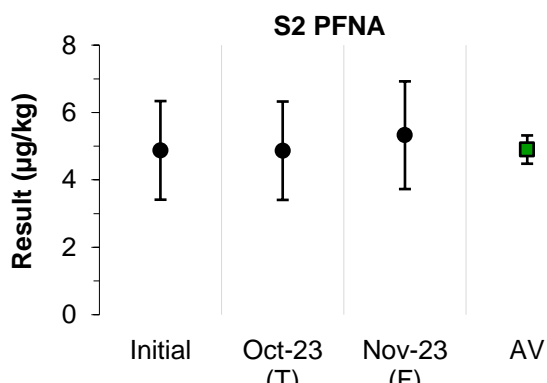


Figure 164 S2 PFNA Stability Testing

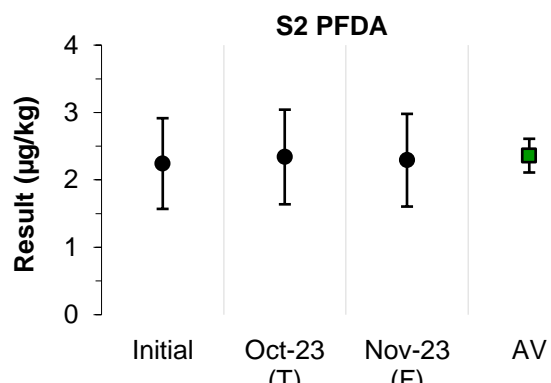


Figure 165 S2 PFDA Stability Testing

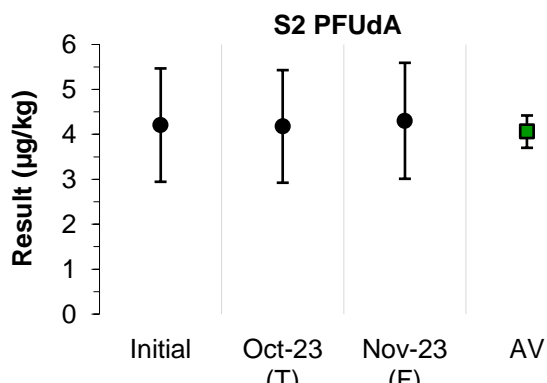


Figure 166 S2 PFUdA Stability Testing

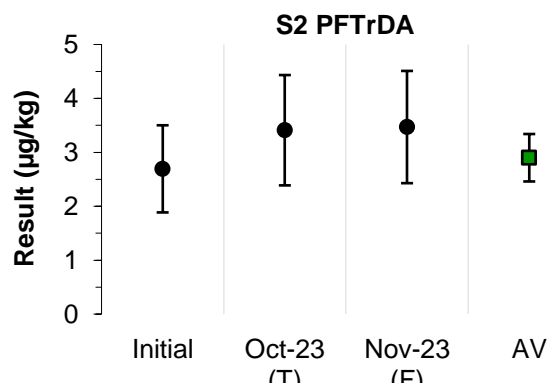


Figure 167 S2 PFTrDA Stability Testing

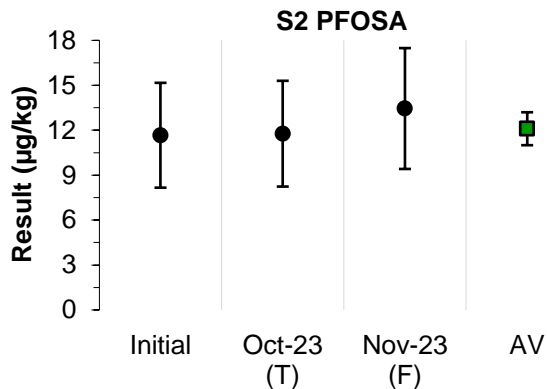


Figure 168 S2 PFOSA Stability Testing

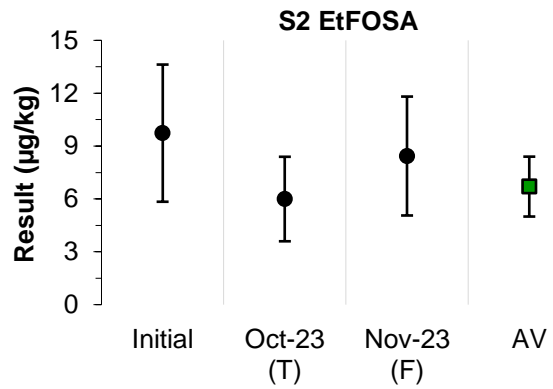


Figure 169 S2 EtFOSA Stability Testing

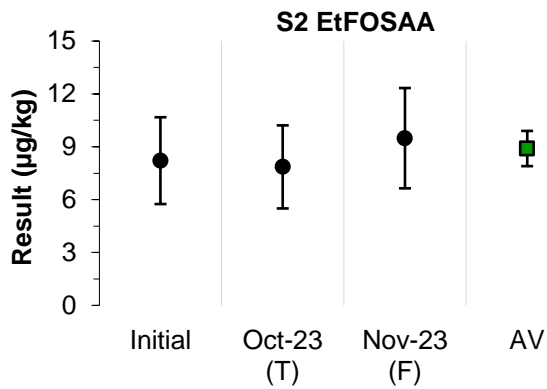


Figure 170 S2 EtFOSAA Stability Testing

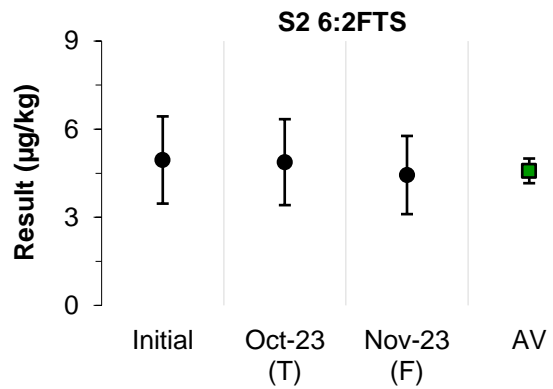


Figure 171 S2 6:2FTS Stability Testing

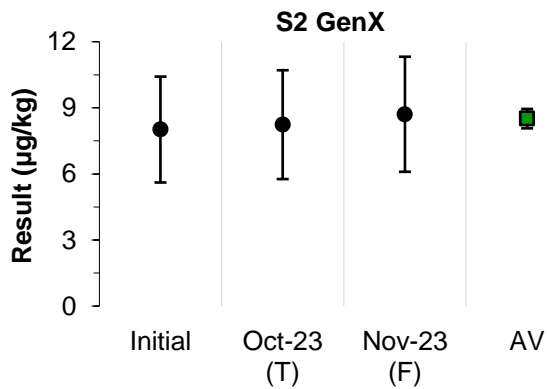


Figure 172 S2 GenX Stability Testing

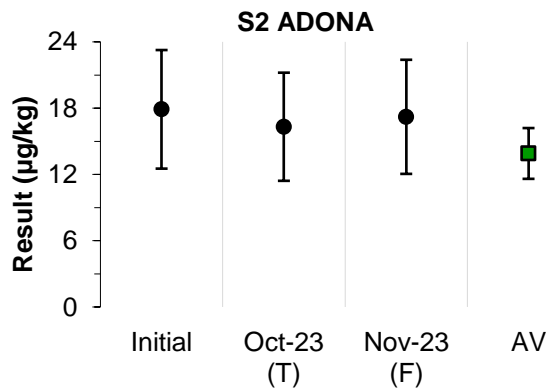


Figure 173 S2 ADONA Stability Testing

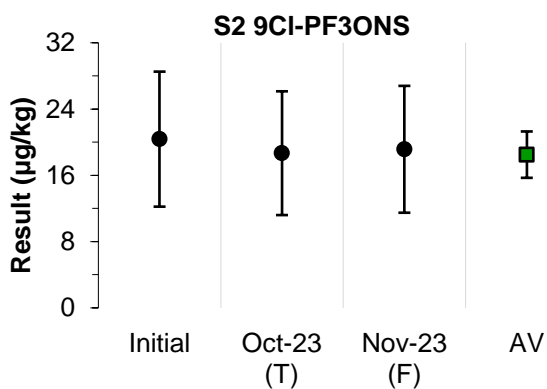


Figure 174 S2 9Cl-PF3ONS Stability Testing

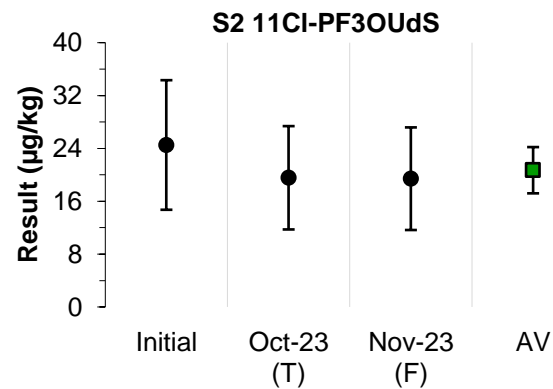


Figure 175 S2 11Cl-PF3OUdS Stability Testing

## Comparison of Results and Transit Time

A comparison of  $z$ -scores obtained to the number of days the samples spent in transit for all scored analytes is presented in Figure 176.

For one international participant, there were substantial customs delays with their sample delivery, resulting in a significantly longer time in transit. Nevertheless, participants' results in this study gave no reason to question the samples' transportation stability.

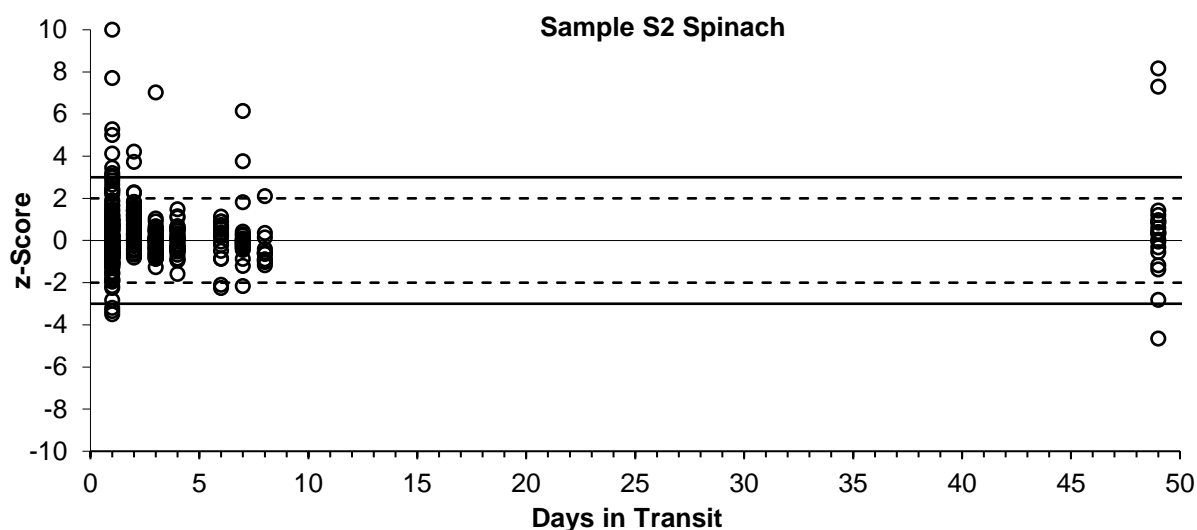


Figure 176 Sample S2 Spinach  $z$ -Score vs Days in Transit

### A2.3 Sample S3 Bovine Liver Homogeneity and Stability Assessment

Samples were analysed by the NMI Australian Ultra Trace Laboratory using the same process as described in Section A2.1.

#### Homogeneity Testing

Homogeneity testing was conducted for Sample S3.

Homogeneity checks were based on that described by Thompson and Fearn,<sup>12</sup> which is also the procedure as described in the International Harmonized Protocol.<sup>4</sup> The results are presented in Tables 154 to 180. Samples were found to be sufficiently homogeneous for use in a PT study with a target SD (as PCV) of 20%.

Table 154 Sample S3 PFBS Homogeneity Testing

Container Number	Result ( $\mu\text{g}/\text{kg}$ )	
	Replicate 1	Replicate 2
10	1.01	1.01
29	1.00	1.01
38*	1.16	1.02
43	1.02	1.05
63	1.10	1.07
64	1.13	1.14
92	1.06	1.10
Mean	1.06	
CV	5.1%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.353	0.781	<b>Pass</b>
$s_{\text{an}}/\sigma$	0.081	0.500	<b>Pass</b>
$s^2_{\text{sam}}$	0.002	0.009	<b>Pass</b>

\* Results from container 38 were not included in the test for homogeneity, being identified as Cochran outliers due to the difference between replicates.<sup>12</sup>



Table 155 Sample S3 PFPeS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	2.84	2.65
29	2.63	2.91
38	3.10	2.85
43	2.95	2.83
63	2.71	2.75
64	2.76	2.96
92	3.14	3.07
Mean	2.87	
CV	5.7%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.323	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.229	0.500	<b>Pass</b>
$s^2_{sam}$	0.010	0.087	<b>Pass</b>

Table 156 Sample S3 PFHxS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	5.1	5.0
29	5.3	5.5
38	6.3	5.6
43	5.3	5.2
63	5.8	5.3
64	5.9	5.9
92	5.8	6.3
Mean	5.6	
CV	7.6%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.543	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.242	0.500	<b>Pass</b>
$s^2_{sam}$	0.117	0.342	<b>Pass</b>

Table 157 Sample S3 PFHxS (linear) Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	5.1	5.0
29	5.3	5.5
38	6.3	5.6
43	5.3	5.2
63	5.8	5.3
64	5.9	5.9
92	5.8	6.3
Mean	5.6	
CV	7.6%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.543	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.242	0.500	<b>Pass</b>
$s^2_{sam}$	0.117	0.342	<b>Pass</b>

Table 158 Sample S3 PFHpS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	1.07	1.13
29	1.07	1.20
38	1.21	1.16
43	1.20	1.17
63	1.11	1.12
64	1.14	1.18
92	1.31	1.38
Mean	1.18	
CV	7.2%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.575	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.201	0.500	<b>Pass</b>
$s^2_{sam}$	0.005	0.014	<b>Pass</b>

Table 159 Sample S3 PFOS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	1.45	1.61
29	1.39	1.36
38	1.71	1.43
43	1.37	1.55
63	1.48	1.54
64	1.47	1.48
92	1.60	1.64
Mean	1.50	
CV	7.1%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.557	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.337	0.500	<b>Pass</b>
$s^2_{sam}$	0.001	0.032	<b>Pass</b>

Table 160 Sample S3 PFOS (linear) Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	1.45	1.61
29	1.39	1.36
38	1.71	1.43
43	1.37	1.55
63	1.48	1.54
64	1.47	1.48
92	1.60	1.64
Mean	1.50	
CV	7.1%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.557	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.337	0.500	<b>Pass</b>
$s^2_{sam}$	0.001	0.032	<b>Pass</b>

Table 161 Sample S3 PFNS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	1.66	1.66
29	1.58	1.73
38	1.95	1.74
43	1.56	1.80
63	1.68	1.72
64	1.73	1.80
92	1.76	1.85
Mean	1.73	
CV	6.0%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.416	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.290	0.500	<b>Pass</b>
$s^2_{sam}$	0.001	0.037	<b>Pass</b>

Table 162 Sample S3 PFDS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	1.54	1.54
29	1.39	1.54
38	1.72	1.48
43	1.76	1.69
63	1.41	1.82
64	1.49	1.71
92	1.72	1.93
Mean	1.63	
CV	10%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.480	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.484	0.500	<b>Pass</b>
$s^2_{sam}$	0.002	0.055	<b>Pass</b>

Table 163 Sample S3 PFBA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	5.5	5.3
29	5.5	5.5
38	6.1	5.5
43	5.5	5.3
63	5.5	5.5
64	5.3	5.7
92	6.0	5.9
Mean	5.6	
CV	4.5%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.645	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.181	0.500	<b>Pass</b>
$s^2_{sam}$	0.024	0.293	<b>Pass</b>

Table 164 Sample S3 PFPeA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	2.07	2.10
29	1.99	2.00
38	2.29	2.14
43	2.17	2.13
63	2.09	2.21
64	1.97	2.08
92	2.19	2.25
Mean	2.12	
CV	4.6%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.424	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.151	0.500	<b>Pass</b>
$s^2_{sam}$	0.006	0.040	<b>Pass</b>

Table 165 Sample S3 PFHxA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	6.4	6.3
29	6.5	6.8
38	7.0	6.2
43	6.1	6.7
63	6.6	6.6
64	6.3	6.6
92	7.1	7.2
Mean	6.6	
CV	5.1%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.515	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.226	0.500	<b>Pass</b>
$s^2_{sam}$	0.027	0.457	<b>Pass</b>

Table 166 Sample S3 PFHpA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	6.8	7.0
29	6.4	7.0
38	7.3	7.2
43	7.1	7.1
63	7.2	7.4
64	7.4	7.0
92	7.0	7.5
Mean	7.1	
CV	4.0%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.432	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.181	0.500	<b>Pass</b>
$s^2_{sam}$	0.017	0.476	<b>Pass</b>

Table 167 Sample S3 PFOA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	0.98	1.11
29	0.94	1.01
38	1.13	0.95
43	0.98	0.95
63	1.04	1.00
64	0.99	1.03
92	1.03	1.05
Mean	1.01	
CV	5.6%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.532	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.320	0.500	<b>Pass</b>
$s^2_{sam}$	0.000	0.014	<b>Pass</b>

Table 168 Sample S3 PFNA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	1.86	1.98
29	1.86	1.99
38	2.11	1.98
43	1.88	1.92
63	2.02	2.04
64	1.91	2.05
92	2.16	2.05
Mean	1.99	
CV	4.6%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.222	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.189	0.500	<b>Pass</b>
$s^2_{sam}$	0.003	0.038	<b>Pass</b>

Table 169 Sample S3 PFDA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	1.53	1.69
29	1.45	1.61
38	1.68	1.67
43	1.70	1.57
63	1.71	1.65
64	1.63	1.75
92	1.72	1.82
Mean	1.66	
CV	5.7%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.257	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.249	0.500	<b>Pass</b>
$s^2_{sam}$	0.002	0.030	<b>Pass</b>

Table 170 Sample S3 PFUdA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	0.78	0.77
29	0.73	0.83
38	0.90	0.76
43	0.87	0.78
63	0.86	0.81
64	0.79	0.83
92	0.86	0.89
Mean	0.82	
CV	6.4%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.460	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.334	0.500	<b>Pass</b>
$s^2_{sam}$	0.000	0.009	<b>Pass</b>

Table 171 Sample S3 PFDoA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	3.3	3.4
29	3.4	3.5
38	3.6	3.3
43	3.5	3.6
63	3.7	3.4
64	3.6	3.5
92	3.9	3.8
Mean	3.5	
CV	4.8%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.315	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.148	0.500	<b>Pass</b>
$s^2_{sam}$	0.019	0.109	<b>Pass</b>

Table 172 Sample S3 PFTeDA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	1.68	1.51
29	1.66	1.61
38	1.82	1.64
43	1.65	1.64
63	1.77	1.90
64	1.74	1.68
92	1.74	1.80
Mean	1.70	
CV	5.9%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.366	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.240	0.500	<b>Pass</b>
$s^2_{sam}$	0.004	0.031	<b>Pass</b>

Table 173 Sample S3 PFOSA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	4.5	4.6
29	4.2	4.6
38	5.2	4.8
43	4.5	4.5
63	4.7	4.5
64	4.6	4.8
92	4.9	5.1
Mean	4.7	
CV	5.6%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.336	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.177	0.500	<b>Pass</b>
$s^2_{sam}$	0.044	0.205	<b>Pass</b>

Table 174 Sample S3 EtFOSA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	4.5	4.3
29	4.5	4.4
38	5.0	4.6
43	4.0	4.5
63	4.8	4.1
64	4.5	4.7
92	4.9	4.7
Mean	4.5	
CV	6.5%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.410	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.316	0.500	<b>Pass</b>
$s^2_{sam}$	0.005	0.273	<b>Pass</b>

Table 175 Sample S3 EtFOSAA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	3.8	3.6
29	3.2	4.1
38	3.9	3.8
43	3.5	4.3
63	4.0	4.3
64	3.9	4.1
92	4.2	4.3
Mean	3.9	
CV	8.1%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.469	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.410	0.500	<b>Pass</b>
$s^2_{sam}$	0.000	0.266	<b>Pass</b>

Table 176 Sample S3 8:2FTS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	10.7	10.9
29	9.8	10.6
38	10.5	9.8
43	10.3	11.0
63	9.2	9.1
64	11.0	10.6
92	11.0	10.6
Mean	10.4	
CV	6.2%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.293	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.178	0.500	<b>Pass</b>
$s^2_{sam}$	0.304	1.007	<b>Pass</b>

Table 177 Sample S3 GenX Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	13.0	13.0
29	12.6	13.7
38	14.0	13.1
43	13.5	13.7
63	13.6	13.4
64	13.5	13.9
92	14.2	14.7
Mean	13.6	
CV	4.0%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.513	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.147	0.500	<b>Pass</b>
$s^2_{sam}$	0.141	1.618	<b>Pass</b>

Table 178 Sample S3 ADONA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	17.5	17.1
29	16.6	18.8
38	19.1	18.2
43	17.1	17.8
63	18.2	17.8
64	17.8	19.2
92	19.2	18.9
Mean	18.1	
CV	4.7%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.575	0.727	<b>Pass</b>
$s_{an}/\sigma$	0.212	0.500	<b>Pass</b>
$s^2_{sam}$	0.145	3.316	<b>Pass</b>



Table 179 Sample S3 9Cl-PF3ONS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	21.9	22.9
29	21.3	23.5
38	24.2	24.2
43	23.1	23.9
63	24.1	22.8
64	23.9	22.7
92	23.1	26.3
Mean	23.4	
CV	5.1%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.514	0.727	<b>Pass</b>
$s_{\text{an}}/\sigma$	0.255	0.500	<b>Pass</b>
$s^2_{\text{sam}}$	0.000	6.187	<b>Pass</b>

Table 180 Sample S3 11Cl-PF3OUdS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
10	22.7	22.1
29	20.3	23.8
38	23.8	21.7
43	22.6	22.5
63	23.4	22.9
64	22.6	23.2
92	24.3	24.5
Mean	22.9	
CV	4.8%	

Thompson and Fearn Homogeneity Tests<sup>12</sup>

Test	Value	Critical	Result
Cochran	0.683	0.727	<b>Pass</b>
$s_{\text{an}}/\sigma$	0.244	0.500	<b>Pass</b>
$s^2_{\text{sam}}$	0.000	5.745	<b>Pass</b>

### Comparison of Results and Container Numbers

A comparison of z-scores obtained to the container number analysed by participants for all scored analytes is presented for information in Figure 177 (results have only been included where the participant was sent one sample set only). Participants' results in this study gave no reason to question the samples' homogeneity.

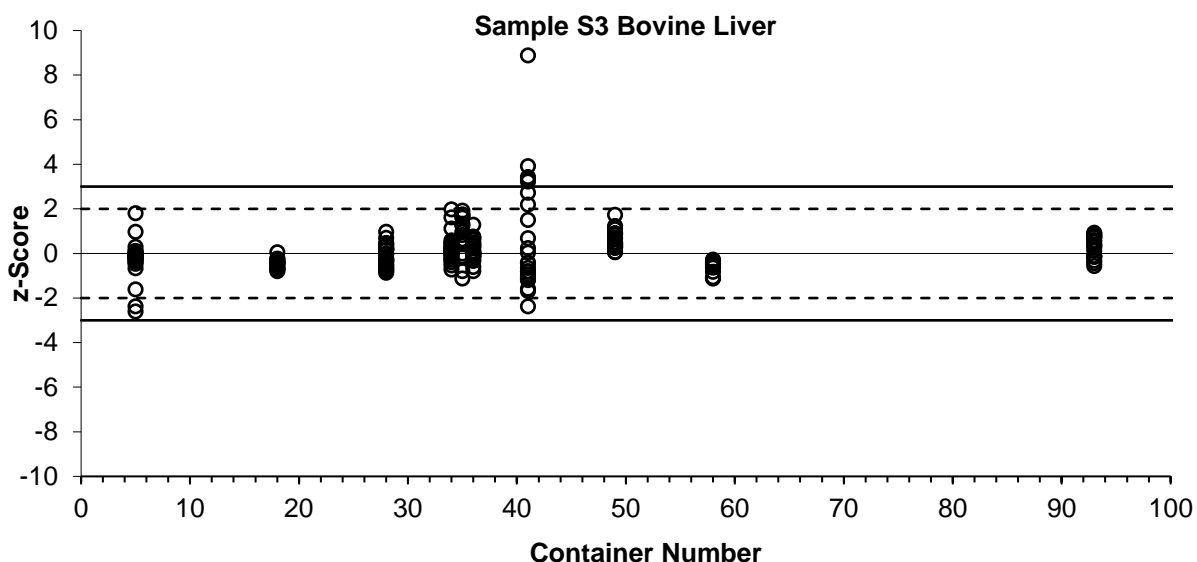


Figure 177 Sample S3 Bovine Liver z-Score vs Container Number

### Stability Testing

Sample S3 was stored and dispatched using a process previously demonstrated to produce sufficiently stable PT samples of similar analytes and matrices. Stability testing was also conducted for this sample.

The bovine liver samples were analysed at an initial time point in August 2023. At sample dispatch (September 2023), samples were set aside and packaged in the same way as the samples dispatched to participants. This was stored at ambient conditions until all samples had been delivered to participants (October 2023), to reflect transportation stability. Additional samples were stored at freezer temperature, to reflect long-term storage temperature at a participant’s laboratory; samples were taken for analysis at the conclusion of the PT study (November 2023).

Results were in good agreement with each other and the assigned value within their respective uncertainties (Figures 178 to 204; T = Transportation Stability, F = Freezer Stability, AV = Assigned Value). The samples were also shown to be sufficiently stable when assessed against the criteria specified in ISO 13528.<sup>6</sup>

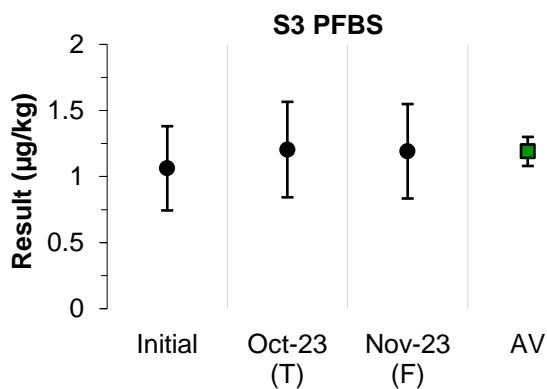


Figure 178 S3 PFBS Stability Testing

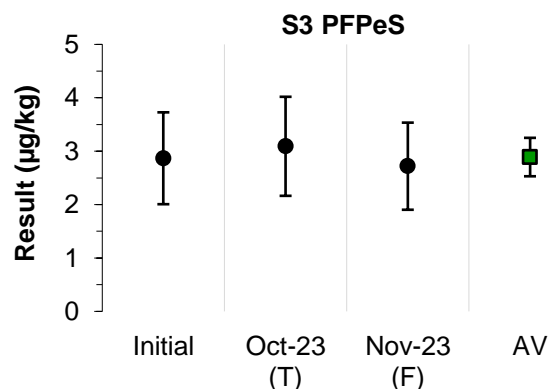


Figure 179 S3 PFPeS Stability Testing

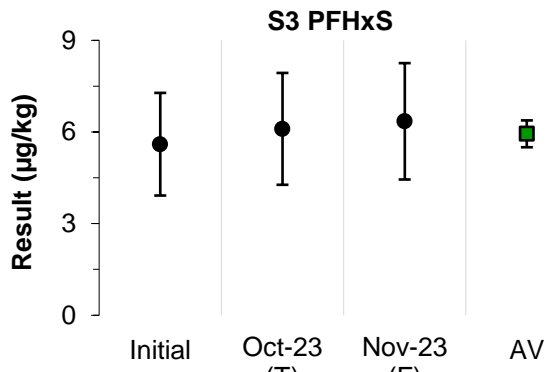


Figure 180 S3 PFHxS Stability Testing

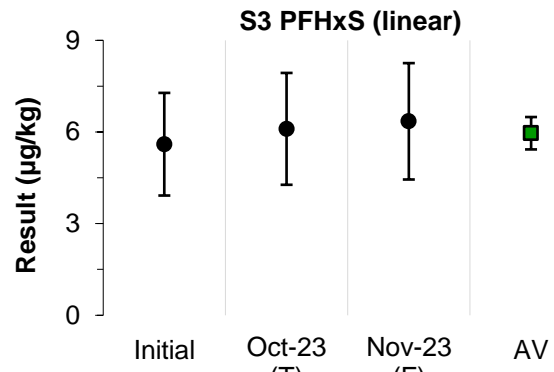


Figure 181 S3 PFHxS (linear) Stability Testing

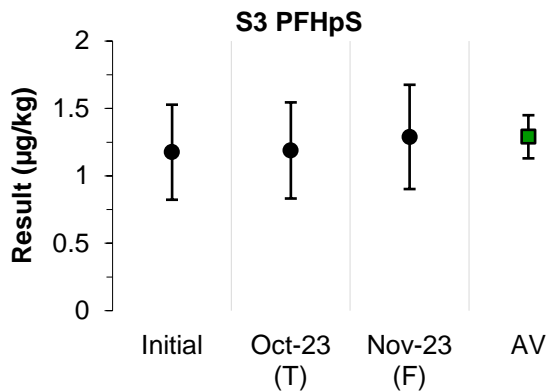


Figure 182 S3 PFHpS Stability Testing

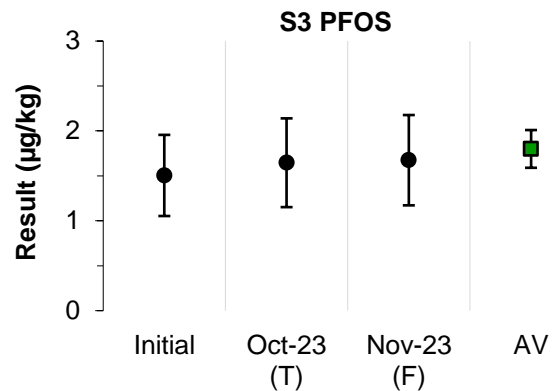


Figure 183 S3 PFOS Stability Testing

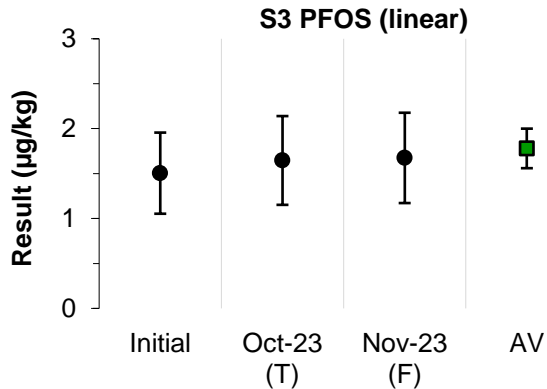


Figure 184 S3 PFOS (linear) Stability Testing

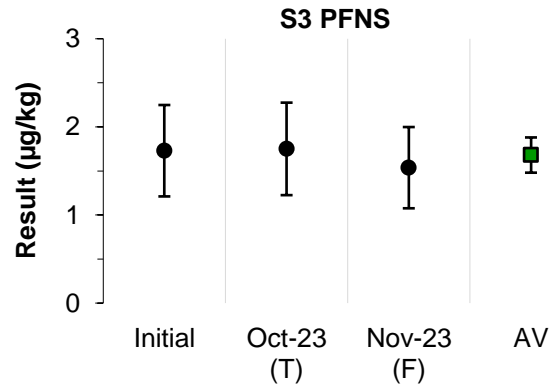


Figure 185 S3 PFNS Stability Testing

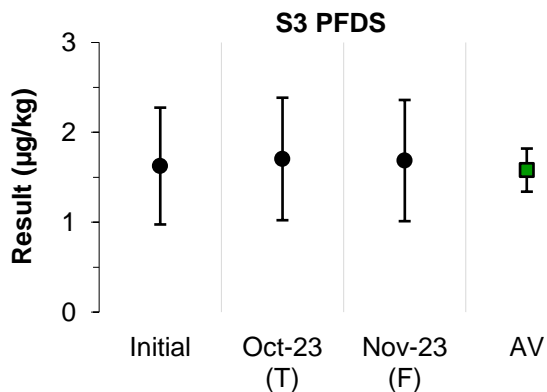


Figure 186 S3 PFDS Stability Testing

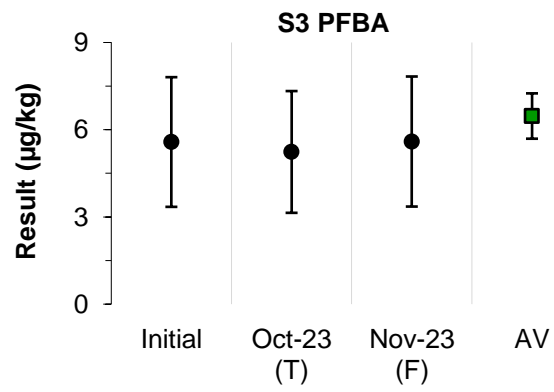


Figure 187 S3 PFBA Stability Testing

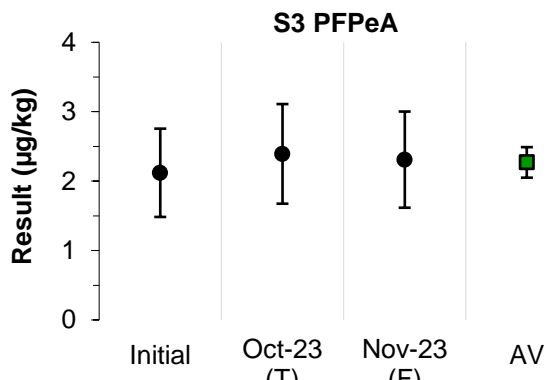


Figure 188 S3 PFPeA Stability Testing

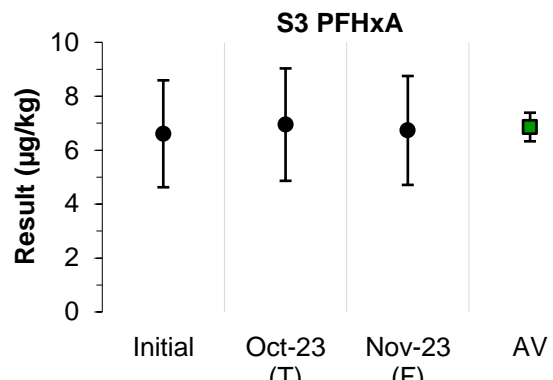


Figure 189 S3 PFHxA Stability Testing

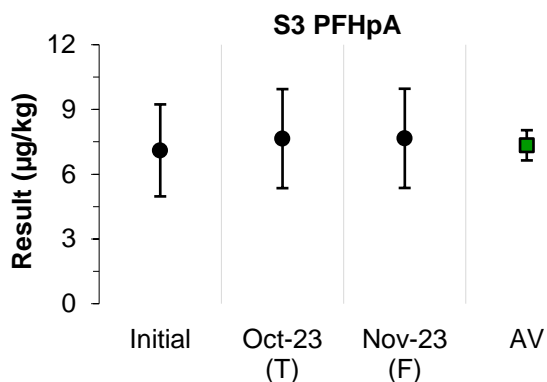


Figure 190 S3 PFHpA Stability Testing

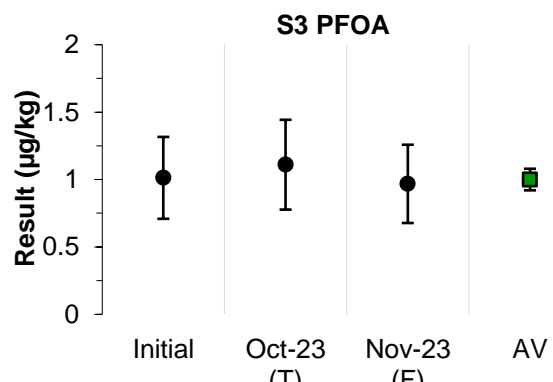


Figure 191 S3 PFOA Stability Testing

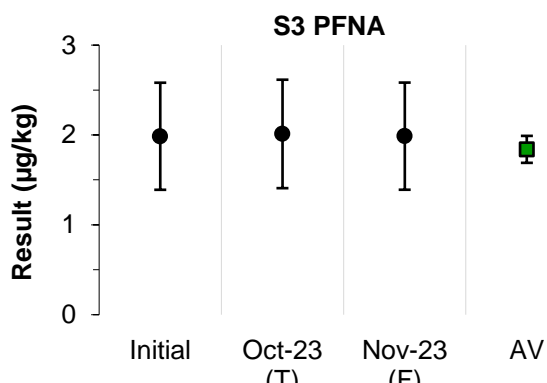


Figure 192 S3 PFNA Stability Testing

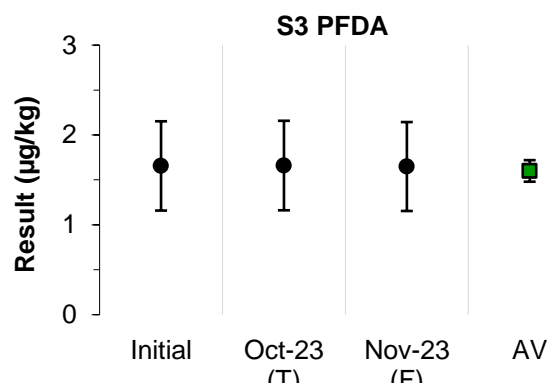


Figure 193 S3 PFDA Stability Testing

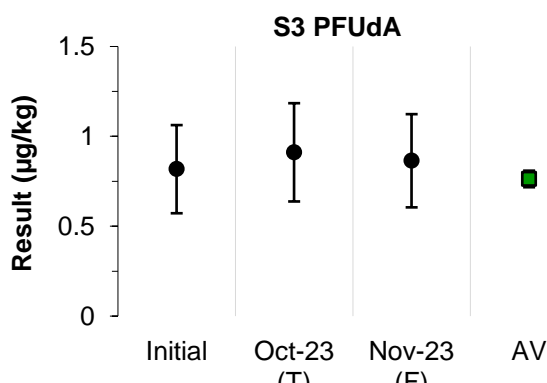


Figure 194 S3 PFUdA Stability Testing

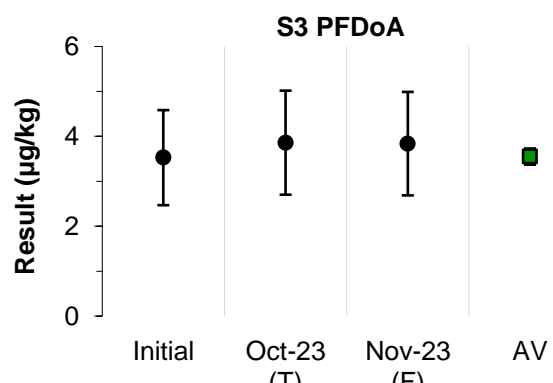


Figure 195 S3 PFDoA Stability Testing

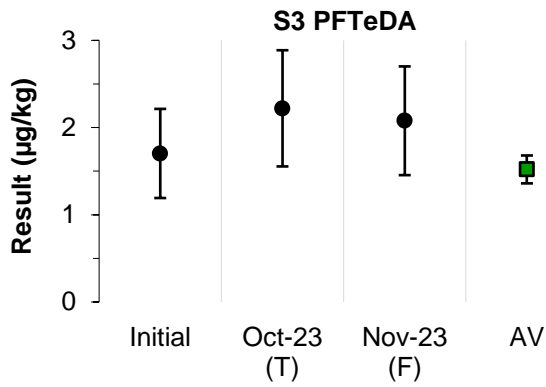


Figure 196 S3 PFTeDA Stability Testing

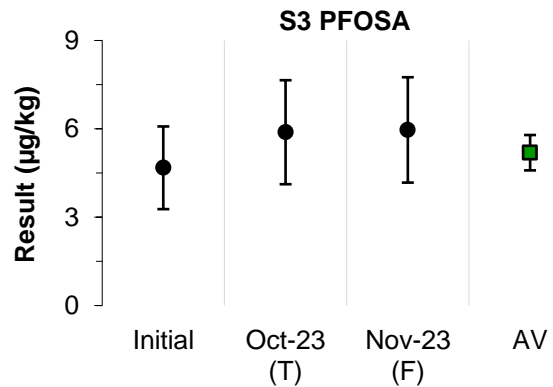
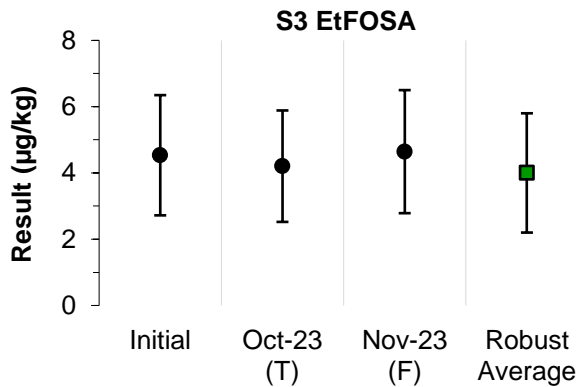


Figure 197 S3 PFOSA Stability Testing



No assigned value was set for S3 EtFOSA, so the robust average is presented here instead.

Figure 198 S3 EtFOSA Stability Testing

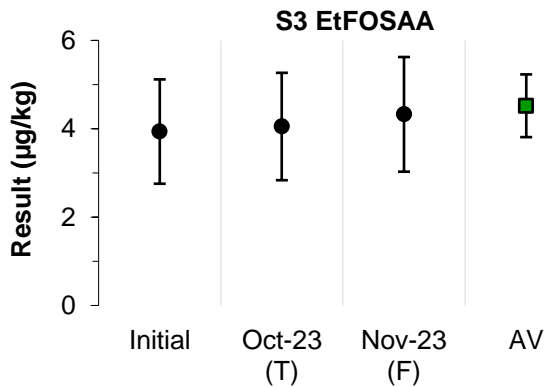


Figure 199 S3 EtFOSAA Stability Testing

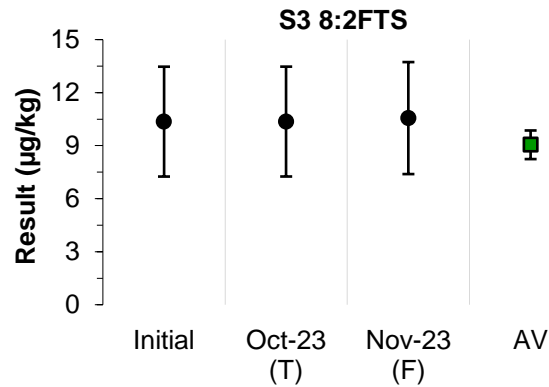


Figure 200 S3 8:2FTS Stability Testing

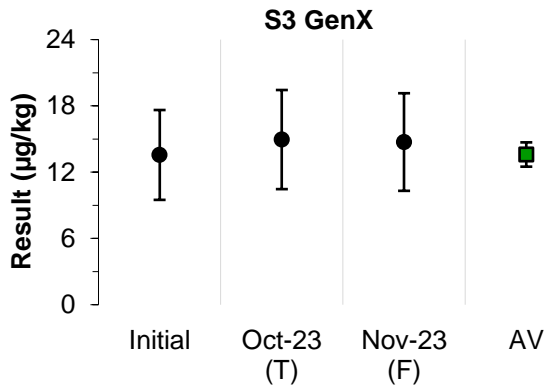


Figure 201 S3 GenX Stability Testing

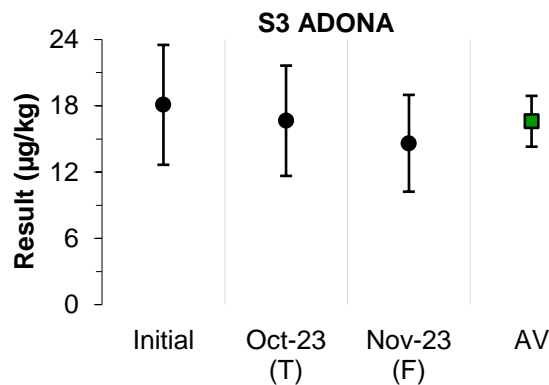


Figure 202 S3 ADONA Stability Testing

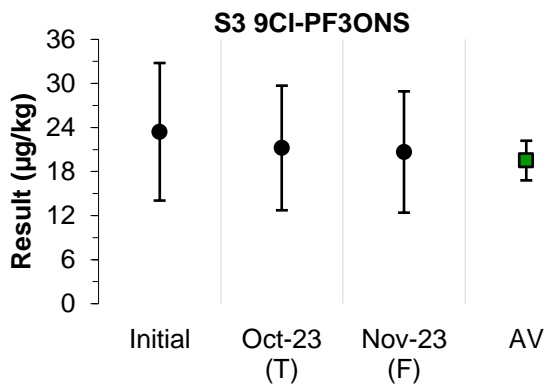


Figure 203 S3 9CI-PF3ONS Stability Testing

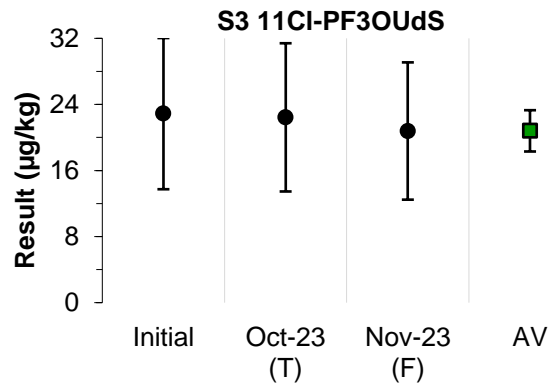


Figure 204 S2 11Cl-PF3OUdS Stability Testing

### Comparison of Results and Transit Time

A comparison of  $z$ -scores obtained to the number of days the samples spent in transit for all scored analytes is presented in Figure 205. Participants' results in this study gave no reason to question the samples' transportation stability.

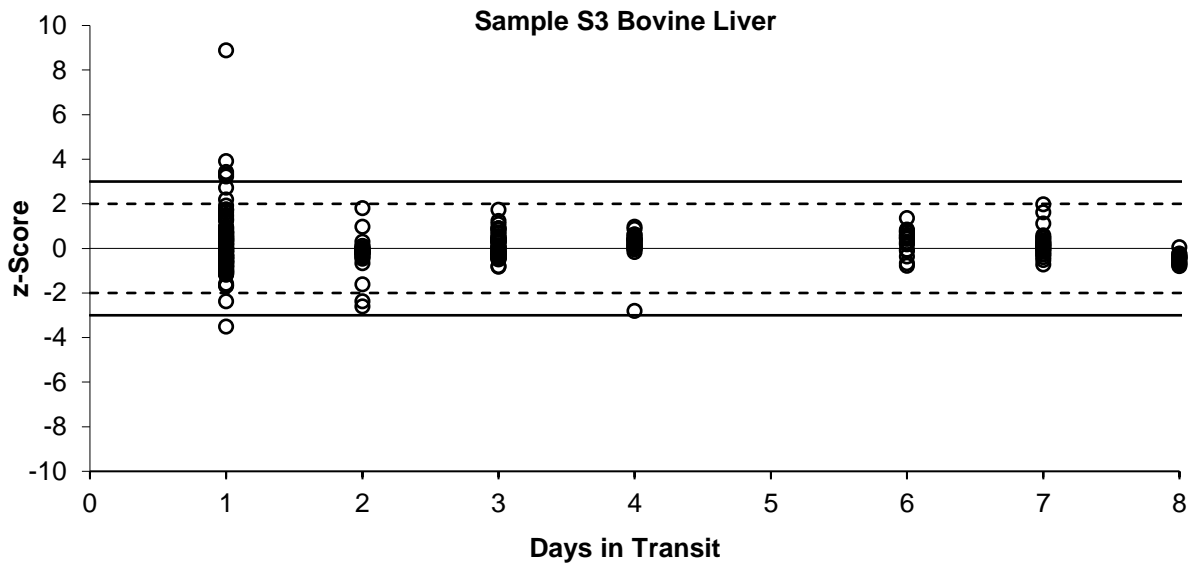


Figure 205 Sample S3 Bovine Liver  $z$ -Score vs Days in Transit

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

### A3.1 Robust Average and Associated Uncertainty

Robust averages were calculated using the procedure described in ISO 13528.<sup>6</sup> The associated uncertainties were estimated as according to Equation 4.

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

where:

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

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

$p$  is the number of results

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

A worked example is set out below in Table 181.

Table 181 Uncertainty Estimate for Robust Average of Sample S1 PFHxS

Number of Results (p)	18
Robust Average	5.65 µg/kg
$S_{rob\ av}$	0.98 µg/kg
$u_{rob\ av}$	0.29 µg/kg
$k$	2
$U_{rob\ av}$	0.58 µg/kg

Therefore, the robust average for Sample S1 PFHxS is  $5.65 \pm 0.58$  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 (Section 4).

A worked example is set out below in Table 182.

Table 182 z-Score and E<sub>n</sub>-Score for Sample S1 PFBS Result Reported by Laboratory 1

Participant Result (µg/kg)	Assigned Value (µg/kg)	Target Standard Deviation	z-Score	E <sub>n</sub> -Score
1.1 ± 0.55	1.13 ± 0.11	20% as PCV, or: 0.2 × 1.13 = 0.226 µg/kg	$z = \frac{1.1 - 1.13}{0.226}$ = -0.13	$E_n = \frac{1.1 - 1.13}{\sqrt{0.55^2 + 0.11^2}}$ = -0.05

#### APPENDIX 4 PARTICIPANTS' TEST METHODS

Participants' responses to the methodology questionnaire are presented in Tables 183 to 274. Some responses may have been modified so that the participant cannot be identified.

Table 183 Participant Methodology – Sample S1 Fish Sample Preparation and Extraction

Lab. Code	Sample Weight (g)	Labelled Standard(s) Added Before Extraction?	Equilibration Time for Labelled Standard (min)	Sample Pre-treatment, if other	Extraction Technique	Number of Steps (if staggered extraction)	Extraction Solvent(s)	Total Extraction Time (min)	Additional Information
1	1	Yes		Homogenisation	QuEChERS - modified AOAC		Acetonitrile with 1% Acetic Acid		Geno/Grinder 14min & Centrifuge 10min. In this method the linear standards are used to quantify both the linear as well as the branched isomers.
2	2.020 and 2.016 (duplicate)	Yes	15 min	NA	Solid-Liquid Extraction (vortexed and centrifuged)	NA	2% formic acid in acetonitrile	8 min	Extraction using Merris-Minimix shaker
3	1g	Yes	10	Homogenisation	Alkaline Digestion	N/A	Basic MeOH	60	
4									
5	1	Yes	NA	NA	QuEChERS	NA	ACN	20	NA
6	1g	Yes			QuEChERS		ACN	30	
7	0.5	Yes	30	Homogenising	Multiple, see additional info		ACN	2 x 15 min	Digestion with 200mM NaOH in methanol, then extraction with acetonitrile.
8	5	Yes	30-60	15 mL reagent water acidified with 150µL formic acid	QuEChERS		ACN	15	



Lab. Code	Sample Weight (g)	Labelled Standard(s) Added Before Extraction?	Equilibration Time for Labelled Standard (min)	Sample Pre-treatment, if other	Extraction Technique	Number of Steps (if staggered extraction)	Extraction Solvent(s)	Total Extraction Time (min)	Additional Information
				added prior to extraction					
9	5g	Yes	30		QuEChERS		ACN	30	
10	0.5	Yes		Homogenisation	Solid-Liquid Extraction (vortexed and centrifuged)		MeOH	60	
11	1	Yes		no	QuEChERS		ACN	180	
12	0.2	Yes			Alkaline Digestion		NaOH/MeOH	60	
13	0.1	Yes			Alkaline Digestion				
14	0.5302	Yes	15	pH Adjustment	Alkaline Digestion	2	ACN	30 mins x2	Alkaline digestion, followed by solid/liquid extraction.
15									
16									
17									
19	1.03			Homogenisation	Alkaline Digestion		KOH/MeOH		Combination of Shaker/Sonication for extraction
20	1.0322	Yes	30		Solid-Liquid Extraction (vortexed and centrifuged)	3 (sonicate, vortex, centrifuge)	ACN	90	
21	1	Yes	30		Alkaline Digestion		KOH/MeOH	480	

Lab. Code	Sample Weight (g)	Labelled Standard(s) Added Before Extraction?	Equilibration Time for Labelled Standard (min)	Sample Pre-treatment, if other	Extraction Technique	Number of Steps (if staggered extraction)	Extraction Solvent(s)	Total Extraction Time (min)	Additional Information
22	2.04 g	Yes	30 min		Solid-Liquid Extraction (vortexed and centrifuged)	3	KOH/MeOH	30	

Table 184 Participant Methodology – Sample S1 Fish Sample Clean-Up and Concentration

Lab. Code	Carbon Clean-Up?	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Elution Solvent	Final pH Adjustment	Additional Information
1	Yes			envicarb			
2	Yes	50°C	Variable	None	Not Applicable	No	Carbon clean up using dSPE (C18, Envicarb, MgSO4)
3	Yes	N/A	N/A	Solid-Phase Extraction	Basic MeOH	Yes	
4							
5	Yes	45	40-60	Solid-Phase Extraction	Basic MeOH	NA	
6		Ambient	40	Solid-Phase Extraction	ACN	No	
7	Yes	Room temperature		Liquid-liquid extraction	MeOH	No	Clean up: liquid-liquid extraction with n-hexane, then Bond Elut Carbon SPE
8	Yes	60	60-90	Solid-Phase Extraction	MeOH, 0.3% NH3	No	
9	Yes	45	30	Solid-Phase Extraction	Basic ACN and Acetone	No	
10	Yes	40	60	Filtration			
11	No	Ambient		D-SPE		No	

Lab. Code	Carbon Clean-Up?	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Elution Solvent	Final pH Adjustment	Additional Information
12		RT		Solid-Phase Extraction	NH4OH/MeOH		
13						Yes	
14	Yes			Solid-Phase Extraction	2% NH4OH		2D-SPE (Waters Oasis WAX SPE + Strata GBC cartridge)
15							
16							
17							
19		Room temperature	3 hour shake, 12 hour sonication bath	SPE (WAX 150mg/6cc)			
20	No	Room Temp.	60	Solid-Phase Extraction	MeOH, 1% NH4OH in MeOH	No	
21	Yes	35	90	Filtration	NH4OH/MeOH	No	
22	Yes	55C	200 min	Solid-Phase Extraction	NH4OH/MeOH	No	

Table 185 Participant Methodology – Sample S1 Fish Instrumental Technique

Lab. Code	Instrument	Dilution Before Analysis and Dilution Factor (if applicable)	Blank Correction?	Additional Information
1	LC-Orbitrap		No	
2	LC-MSMS or LC-QQQ	No	Yes	NA
3	LC-MSMS or LC-QQQ	5	No	
4				
5	LC-MSMS or LC-QQQ	NA	Yes	NA
6	LC-Orbitrap	DF1, DF10, DF100	No	
7	LC-MSMS or LC-QQQ	No	No	
8	Sciex 6500+ Triple		No	C-18 LC column (3µm, 150mm x 2mm)

Lab. Code	Instrument	Dilution Before Analysis and Dilution Factor (if applicable)	Blank Correction?	Additional Information
9	LC-MSMS or LC-QQQ	No	No	
10	LC-MSMS or LC-QQQ	No	No	
11	LC-MSMS or LC-QQQ	yes, 10x	Yes	
12	LC-MSMS or LC-QQQ		Yes	
13	LC-MSMS or LC-QQQ			
14	LC-MSMS or LC-QQQ	No	No	N/A
15				
16				
17				
19	LC-MSMS or LC-QQQ	No	No	
20	LC-MSMS or LC-QQQ		Yes	
21	LC-MSMS or LC-QQQ	No	No	
22	LC-MSMS or LC-QQQ	No	No	

Table 186 Participant Methodology – Sample S1 Fish Labelled Standards

Lab. Code	Labelled Standard Source	Recovery Correction?	Standard Method Used?	Additional Information
1	Wellington	Yes	In house	
2	Wellington Laboratory	Yes	No	NA
3	Wellington Labs	Yes	No. In-house	
4				
5	Wellington, CIL	Yes	NA	NA
6	Wellington	Yes		Results corrected by ISTD added before instrumentation
7	Wellington	Yes	Isotopic Dilution	

Lab. Code	Labelled Standard Source	Recovery Correction?	Standard Method Used?	Additional Information
8	Cambridge (FTS compounds); Wellington (remainder)	Yes		
9	Wellington, Cambridge Isotope laboratories	No	No	
10	Wellington Laboratories	Yes		
11	Wellington	Yes		
12	Wellington	No		
13				
14	Wellington (Greyhound)	Yes	No - method developed in house	N/A
15				
16				
17	Wellington, Cambridge Isotopes	Yes	US FDA Foods Program Compendium of Analytical Laboratory Methods; method C-010.02	d5NN EtFOSAA added before instrument analysis
19	Wellington	Yes	No	
20	Wellington	Yes	No	
21	Wellington	Yes	No	
22	Wellington	Yes	No	

Table 187 Labelled Standards for Sample S1 Fish PFBS

Lab. Code	Before Extraction	Before Instrument Analysis
1	Sodium perfluoro-1-[2,3,4 <sup>13</sup> C <sub>3</sub> ] butanesulfonate M3PFBS	
2	M3PFBS	NA
3	<sup>13</sup> C <sub>3</sub> -PFBS	N/A
4		
5	NA	NA
6	PFOS-13C8	PFBS-13C3
7	<sup>13</sup> C <sub>3</sub> -PFBS	<sup>13</sup> C <sub>3</sub> -PFHxS
8	yes	
9	<sup>13</sup> C <sub>3</sub> -PFBS	
10	<sup>13</sup> C <sub>3</sub> -PFBS	
11		
12	<sup>13</sup> C <sub>3</sub> -PFBS	
13		
14	<sup>13</sup> C-PFBS	MPFOS
15		
16		
17	M3 PFBS	
19	<sup>13</sup> C <sub>3</sub> PFBS	
20	18O <sub>2</sub> -PFHxS	18O <sub>2</sub> -PFOS
21	<sup>13</sup> C <sub>3</sub> PFBS	
22	<sup>13</sup> C <sub>3</sub> -PFBS	18O <sub>2</sub> -PFHxS

Table 188 Labelled Standards for Sample S1 Fish PFPeS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M5PFHxA	NA
3	18O <sub>2</sub> -PFHxS	N/A
4		
5	NA	NA
6	PFOS-13C8	PFOS-13C4
7	18O <sub>2</sub> -PFHxS	<sup>13</sup> C <sub>3</sub> -PFHxS
8		
9	N/A	
10	16O <sub>2</sub> -PFHxS	
11		
12	<sup>13</sup> C <sub>3</sub> -PFBS	
13		
14		
15		
16		
17	M3 PFHxS	
19	<sup>13</sup> C <sub>3</sub> PFBS	
20	18O <sub>2</sub> -PFHxS	18O <sub>2</sub> -PFOS
21	<sup>13</sup> C <sub>3</sub> PFBS	
22	<sup>13</sup> C <sub>3</sub> -PFHxS	18O <sub>2</sub> -PFHxS

Table 189 Labelled Standards for Sample S1 Fish PFHxS

Lab. Code	Before Extraction	Before Instrument Analysis
1	Sodium perfluoro-1-[1,2,3 13C3] hexanesulfonate M3PFHxS	
2	M3PFHxS	NA
3	18O2-PFHxS	N/A
4		
5	NA	NA
6	PFOS-13C8	PFHxS-18O2
7	18O2-PFHxS	13C3-PFHxS
8		
9	18O2-PFHxS	
10	16O2-PFHxS	
11		
12	13C3-PFHxS	
13		
14	13C-PFHxS	MPFOS
15		
16		
17	M3 PFHxS	
19	18O2 PFHxS	
20		
21	13C3 PFHxS	PFHxS18O2
22	13C3-PFHxS	18O2-PFHxS

Table 190 Labelled Standards for Sample S1 Fish PFHxS (linear)

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M3PFHxS	NA
3	18O2-PFHxS	N/A
4		
5	Yes	NA
6	PFOS-13C8	PFHxS-18O2
7	18O2-PFHxS	13C3-PFHxS
8	yes	
9	18O2-PFHxS	
10	NT	
11		
12	13C3-PFHxS	
13		
14	13C-PFHxS	MPFOS
15		
16		
17		
19	18O2 PFHxS	
20	18O2-PFHxS	18O2-PFOS
21	13C3 PFHxS	
22	13C3-PFHxS	18O2-PFHxS

Table 191 Labelled Standards for Sample S1 Fish PFHpS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M3PFHxS	NA
3	13C4-PFOS	N/A
4		
5	NA	NA
6	PFOS-13C8	PFOS-C4
7	18O2-PFHxS	13C3-PFHxS
8		
9	N/A	
10	13C8-PFOS	
11		
12	13C3-PFHxS	
13		
14	13C-PFOS	MPFOS
15		
16		
17	M3 PFHxS	
19	18O2 PFHxS	
20	18O2-PFHxS	18O2-PFOS
21	13C3 PFHxS	
22	13C8-PFOS	13C4-PFOS

Table 192 Labelled Standards for Sample S1 Fish PFOS

Lab. Code	Before Extraction	Before Instrument Analysis
1	Sodium perfluoro-1-[ 13C8] octanesulfonate M8PFOS	
2	M8PFOS	NA
3	13C4-PFOS	N/A
4		
5	NA	NA
6	PFOS-13C8	PFOS-C4
7	13C4-PFOS	13C8-PFOS
8	yes	
9	13C8-PFOS	
10	13C4-PFOS	
11		
12	13C8-PFOS	
13		
14	13C-PFOS	MPFOS
15		
16		
17	13C PFOS	
19	13C4 PFOS	
20	13C4-PFOS	18O2-PFOS
21	13C8 PFOS	13C4 PFOS
22	13C8-PFOS	13C4-PFOS



Table 193 Labelled Standards for Sample S1 Fish PFOS (linear)

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M8PFOS	NA
3	13C4-PFOS	N/A
4		
5	Yes	NA
6	PFOS-13C8	PFOS-C4
7	13C4-PFOS	13C8-PFOS
8		yes
9	13C8-PFOS	
10	13C8-PFOS	
11	Yes	
12	13C8-PFOS	
13		
14	13C-PFOS	MPFOS
15		
16		
17		
19	13C4 PFOS	
20	13C4-PFOS	18O2-PFOS
21	13C8 PFOS	
22	13C8-PFOS	13C4-PFOS

Table 194 Labelled Standards for Sample S1 Fish PFNS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M8PFOS	NA
3	13C4-PFOS	N/A
4		
5	NA	NA
6	PFOS-13C8	PFBS-13C3
7	13C4-PFOS	13C8-PFOS
8		
9	N/A	
10	NT	
11		
12	13C8-PFOS	
13		
14		
15		
16		
17	13C PFOS	
19	13C4 PFOS	
20	13C4-PFOS	18O2-PFOS
21	13C8 PFOS	
22	13C8-PFOS	13C4-PFOS

Table 195 Labelled Standards for Sample S1 Fish PFDS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M8PFOS	NA
3	13C4-PFOS	N/A
4		
5	NA	NA
6	PFOS-13C8	PFBA-13C4
7	13C4-PFOS	13C8-PFOS
8		
9	N/A	
10	13C8-PFOS	
11		
12	13C8-PFOS	
13		
14	13C-PFOS	MPFOS
15		
16		
17	13C PFOS	
19	13C4 PFOS	
20	13C4-PFOS	18O2-PFOS
21	13C8 PFOS	
22	13C8-PFOS	13C4-PFOS

Table 196 Labelled Standards for Sample S1 Fish PFBA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[13C4]butanoic acid MPFBA	
2	M4PFBA	NA
3	13C4-PFBA	N/A
4		
5	NA	NA
6	PFOS-13C8	PFBA-13C4
7	13C4-PFBA	13C3-PFBA
8	yes	
9	13C4-PFBA	
10	13C4-PFBA	
11	Yes	
12	13C4-PFBA	
13		
14	13C-PFBA	M3PFBA
15		
16		
17	M3 PFBA	
19	13C2 PFHxA	
20	13C4-PFBA	13C8-PFOA
21	13C4 PFBA	13C3 PFBA
22	13C4-PFBA	13C3-PFBA

Table 197 Labelled Standards for Sample S1 Fish PFPeA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[13C5]pentanoic acid M5PFPeA	
2	M5PFPeA	NA
3	13C3-PFPeA	N/A
4		
5	NA	NA
6	PFOS-13C8	PFPeA-13C3
7	13C4-PFPeA	13C5 -PFPeA
8	yes	
9	13C5-PFPeA	
10	13C5-PFPeA	
11		
12	13C5-PFPeA	
13		
14	13C-PFPeA	M3PFBA
15		
16		
17	M3 PFPeA	
19	13C4 PFHpA	
20	13C5-PFPeA	13C8-PFOA
21	13C5 PFPeA	
22	13C5-PFPeA	13C2-PFHxA

Table 198 Labelled Standards for Sample S1 Fish PFHxA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2,3,4,6- 13C5]hexanoic acid M5PFHxA	
2	M5PFHxA	NA
3	13C2-PFHxA	N/A
4		
5	NA	NA
6	PFOS-13C8	PFHxA-13C2
7	13C2-PFHxA	13C5 -PFPeA
8	yes	
9	13C2-PFHxA	
10	13C5-PFHxA	
11		
12	13C5-PFHxA	
13		
14	13C-PFHxA	M2PFOA
15		
16		
17	M5 PFHxA	
19	13C2 PFHxA	
20	13C5-PFHxA	13C8-PFOA
21	13C5 PFHxA	13C2 PFHxA
22	13C5-PFHxA	13C2-PFHxA

Table 199 Labelled Standards for Sample S1 Fish PFHpA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]heptanoic acid M4PFHpA	
2	MPFHpA	NA
3	<sup>13</sup> C <sub>4</sub> -PFHpA	N/A
4		
5	NA	NA
6	PFOS- <sup>13</sup> C <sub>8</sub>	PFHpA- <sup>13</sup> C <sub>4</sub>
7	<sup>13</sup> C <sub>3</sub> -PFHpA	<sup>13</sup> C <sub>8</sub> -PFOA
8		
9	<sup>13</sup> C <sub>4</sub> -PFHpA	
10	<sup>13</sup> C <sub>4</sub> -PFHpA	
11		
12	<sup>13</sup> C <sub>4</sub> -PFHpA	
13		
14	<sup>13</sup> C-PFHpA	M2PFOA
15		
16		
17	M5 PFHxA	
19	<sup>13</sup> C <sub>4</sub> PFHpA	
20	<sup>13</sup> C <sub>4</sub> -PFHpA	<sup>13</sup> C <sub>8</sub> -PFOA
21	<sup>13</sup> C <sub>4</sub> PFHpA	
22	<sup>13</sup> C <sub>4</sub> -PFHpA	<sup>13</sup> C <sub>4</sub> -PFOA

Table 200 Labelled Standards for Sample S1 Fish PFOA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[ <sup>13</sup> C <sub>8</sub> ]octanoic acid M8PFOA	
2	M8PFOA	NA
3	<sup>13</sup> C <sub>4</sub> -PFOA	N/A
4		
5	Yes	NA
6	PFOS- <sup>13</sup> C <sub>8</sub>	PFOA- <sup>13</sup> C <sub>4</sub>
7	<sup>13</sup> C <sub>4</sub> -PFOA	<sup>13</sup> C <sub>8</sub> -PFOA
8	yes	yes
9	<sup>13</sup> C <sub>8</sub> -PFOA	
10	<sup>13</sup> C <sub>8</sub> -PFOA	
11	Yes	
12	<sup>13</sup> C <sub>8</sub> -PFOA	
13		
14	<sup>13</sup> C-PFOA	M2PFOA
15		
16		
17	<sup>13</sup> C PFOA	
19	<sup>13</sup> C <sub>4</sub> PFOA	
20	<sup>13</sup> C <sub>4</sub> -PFOA	<sup>13</sup> C <sub>8</sub> -PFOA
21	<sup>13</sup> C <sub>8</sub> PFOA	<sup>13</sup> C <sub>4</sub> PFOA
22	<sup>13</sup> C <sub>8</sub> -PFOA	<sup>13</sup> C <sub>4</sub> -PFOA

Table 201 Labelled Standards for Sample S1 Fish PFNA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[13C9]nonanoic acid M9PFNA	
2	M9PFNA	NA
3	13C5-PFNA	N/A
4		
5	Yes	NA
6	PFOS-13C8	PFNA-13C5
7	13C5-PFNA	13C8-PFOA
8		
9	13C5-PFNA	
10	13C5-PFNA	
11		
12	13C9-PFNA	
13		
14	13C-PFNA	M2PFOA
15		
16		
17	13C PFOA	
19	13C5 PFNA	
20	13C9-PFNA	13C5-PFNA
21	13C9 PFNA	13C5 PFNA
22	13C9-PFNA	13C5-PFNA

Table 202 Labelled Standards for Sample S1 Fish PFDA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2,3,4,6- 13C6]decanoic acid M6PFDA	
2	M6PFDA	NA
3	13C2-PFDA	N/A
4		
5	NA	NA
6	PFOS-13C8	PFDA-13C2
7	13C2-PFDA	13C8-PFOA
8		
9	13C6-PFDA	
10	13C6-PFDA	
11	Yes	
12	13C6-PFDA	
13		
14	13C-PFDA	MPFDA
15		
16		
17	13C PFOA	
19	13C2 PFDA	
20	13C2-PFDA	13C5-PFNA
21	13C6 PFDA	13C2 PFDA
22	13C6-PFDA	13C2-PFDA

Table 203 Labelled Standards for Sample S1 Fish PFUdA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2,3,4,6,7- <sup>13</sup> C7]undecanoic acid M7PFUdA	
2	M7PFUnDA	NA
3	<sup>13</sup> C2-PFUdA	N/A
4		
5	NA	NA
6	PFOS- <sup>13</sup> C8	PFUNDA- <sup>13</sup> C2
7	<sup>13</sup> C2-PFUdA	<sup>13</sup> C8-PFOA
8	yes	yes
9	<sup>13</sup> C2-PFUnA	
10	<sup>13</sup> C2-PFUnDA	
11		
12	<sup>13</sup> C7-PFUnA	
13		
14	<sup>13</sup> C-PFUdA	MPFDA
15		
16		
17	MPFUdA	
19	<sup>13</sup> C2 PFUnA	
20	<sup>13</sup> C2-PFUdA	<sup>13</sup> C5-PFNA
21	<sup>13</sup> C7 PFUnA	
22	<sup>13</sup> C7-PFUNA	<sup>13</sup> C2-PFDA

Table 204 Labelled Standards for Sample S1 Fish PFDoA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2- <sup>13</sup> C2]dodecanoic acid MPFDoA	
2	MPFDoDA	NA
3	<sup>13</sup> C2-PFDoDA	N/A
4		
5	NA	NA
6	PFOS- <sup>13</sup> C8	PFDoDA- <sup>13</sup> C2
7	<sup>13</sup> C2-PFDoA	<sup>13</sup> C8-PFOA
8	yes	
9	<sup>13</sup> C2-PFDoA	
10	<sup>13</sup> C2-PFDoDA	
11		
12	<sup>13</sup> C2-PFDoA	
13		
14	<sup>13</sup> C-PFDoA	MPFDA
15		
16		
17	MPFDoA	
19	<sup>13</sup> C2 PFDoA	
20	<sup>13</sup> C2-PFDoA	<sup>13</sup> C5-PFNA
21	<sup>13</sup> C2 PFDoA	
22	<sup>13</sup> C2-PFDoA	<sup>13</sup> C2-PFDA

Table 205 Labelled Standards for Sample S1 Fish PFTeDA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2- <sup>13</sup> C <sub>2</sub> ]tetradecanoic acid M2PFTeDA	
2	MPFTeDA	NA
3	<sup>13</sup> C <sub>2</sub> -PFTeDA	N/A
4		
5	NA	NA
6	PFOS- <sup>13</sup> C <sub>8</sub>	PFTeDA- <sup>13</sup> C <sub>2</sub>
7	<sup>13</sup> C <sub>2</sub> -PFTeDA	<sup>13</sup> C <sub>8</sub> -PFOA
8	yes	
9	<sup>13</sup> C <sub>2</sub> -PFTeDA	
10	<sup>13</sup> C <sub>2</sub> -PFTeDA	
11		
12	<sup>13</sup> C <sub>2</sub> -PFTeDA	
13		
14	<sup>13</sup> C-PFTeDA	MPFDA
15		
16		
17	MPFTeDA	
19	<sup>13</sup> C <sub>2</sub> PFTeDA	
20	<sup>13</sup> C <sub>2</sub> -PFH <sub>x</sub> DA	<sup>13</sup> C <sub>2</sub> -PFTeDA
21	<sup>13</sup> C <sub>2</sub> PFTeDA	
22	<sup>13</sup> C <sub>2</sub> -PFTeDA	<sup>13</sup> C <sub>2</sub> -PFDA

Table 206 Labelled Standards for Sample S1 Fish PFOSA

Lab. Code	Before Extraction	Before Instrument Analysis
1	N-methyl-d <sub>3</sub> -perfluoro-1-octanesulfonamide	
2	MPFOSA	NA
3	D <sub>3</sub> -M PFOSA	N/A
4		
5	NA	NA
6	PFOS- <sup>13</sup> C <sub>8</sub>	FOSA- <sup>13</sup> C <sub>8</sub>
7	<sup>13</sup> C <sub>8</sub> -FOSA	
8	yes	
9	<sup>13</sup> C <sub>8</sub> -FOSA	
10	<sup>13</sup> C <sub>8</sub> -FOSA	
11		
12		
13		
14		
15		
16		
17	M <sub>8</sub> FOSA	
19	<sup>13</sup> C <sub>8</sub> PFOSA	
20	<sup>13</sup> C <sub>8</sub> -PFOSA	<sup>13</sup> C <sub>2</sub> -PFTeDA
21	<sup>13</sup> C <sub>8</sub> PFOSA	
22	<sup>13</sup> C <sub>8</sub> -PFOSA	<sup>13</sup> C <sub>4</sub> -PFOS

Table 207 Labelled Standards for Sample S1 Fish EtFOSA

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	d-NEtFOSA-M	NA
3	D3-Me-FOSAA	N/A
4		
5	NA	NA
6	PFOS-13C8	EtFOSA-D5
7	D5-N-Et FOSA	
8		
9	d5-N-EtFOSA	
10	d5-EtFOSA	
11		
12		
13		
14		
15		
16		
17		
19	d-N-EtFOSA-M	
20	13C8-PFOSA	13C2-PFTeDA
21	d5-N-EtFOSA	
22	D5-N-EtFOSA	13C4-PFOS

Table 208 Labelled Standards for Sample S1 Fish EtFOSAA

Lab. Code	Before Extraction	Before Instrument Analysis
1	d7-N-MeFOSE-M 2-(N-methyl-d3-perfluoro-1-octanesulfonamido) ethand4-ol	
2	d5-NEtFOSAA	NA
3	D7-Me-FOSE	N/A
4		
5	NA	NA
6	PFOS-13C8	EtFOSAA-D5
7	D5-N-Et FOSAA	
8		yes
9	d5-NEtFOSAA	
10	d5-EtFOSAA	
11		
12		
13		
14		
15		
16		
17		
19	d7-NEtFOSAA	
20	NT	NT
21	d5-N-EtFOSAA	
22	D5-N-EtFOSAA	13C2-D4-6:2 FTS



Table 209 Labelled Standards for Sample S1 Fish 8:2FTS

Lab. Code	Before Extraction	Before Instrument Analysis
1	M2-8:2FTS -1H,1H,2H,2H-perfluoro1-[1,2-13C2]-decane sulfonate (8:2)	
2	M8:2 FTS	NA
3	13C2 8:2-FTS	N/A
4		
5	NA	NA
6	PFOS-13C8	8:2 FTS-13C2
7	13C2-8:2 FTS	
8	yes	
9	13C2-8-2 FTS	
10	13C2-8:2 FTS	
11	Yes	
12		
13		
14		
15		
16		
17	13C2D4 8:2 FTS	
19	M2-8:2 FTS	
20	NT	NT
21	13C2 6:2 FTS	
22	13C2-8:2 FTS	13C2-D4-6:2 FTS

Table 210 Labelled Standards for Sample S1 Fish GenX

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M3HFPO-DA	NA
3	13C312C3HF11O3	N/A
4		
5	NA	NA
6	PFOS-13C8	
7	13C4-PFOA	
8	yes	
9	13C3-GenX	
10	NT	
11		
12	13C8-PFOA	
13		
14		
15		
16		
17	M3 HFPO	
19	13C3 HFPO-DA	
20	NT	NT
21	13C3 HFPO-DA	
22	13C3-HFPO-DA	13C2-PFHxA

Table 211 Labelled Standards for Sample S1 Fish ADONA

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	MPFHpA	NA
3	13C4-PFHpA	N/A
4		
5	NA	NA
6	PFOS-13C8	
7	13C4-PFOA	
8		
9	N/A	
10	NT	
11		
12	13C8-PFOA	
13		
14		
15		
16		
17	13C PFOA	
19	13C4 PFOS	
20	NT	NT
21	13C3 HFPO-DA	
22	13C3-HFPO-DA	13C2-PFHxA

Table 212 Labelled Standards for Sample S1 Fish 9C1-PF3ONS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M8PFOS	NA
3	13C4-PFOS	N/A
4		
5	NA	NA
6	PFOS-13C8	PFPeA-13C3
7	13C4-PFOS	13C8-PFOS
8		
9	N/A	
10	NT	
11		
12	13C8-PFOS	
13		
14		
15		
16		
17	M3 PFHxS	
19	13C4 PFOS	
20	NT	NT
21	13C3 HFPO-DA	
22	13C3-HFPO-DA	13C2-PFHxA

Table 213 Labelled Standards for Sample S1 Fish 11Cl-PF3OUdS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M7PFUnDA	NA
3	13C4-PFOS	N/A
4		
5	NA	NA
6	PFOS-13C8	FOSA-13C8
7	13C4-PFOS	13C8-PFOS
8		
9	N/A	
10	NT	
11		
12	13C8-PFOS	
13		
14		
15		
16		
17	M3 PFHxS	
19		
20	NT	NT
21	13C3 HFPO-DA	
22	13C3-HFPO-DA	13C2-PFHxA

Table 214 Participant Methodology – Sample S2 Spinach Sample Preparation and Extraction

Lab. Code	Sample Weight (g)	Labelled Standard(s) Added Before Extraction?	Equilibration Time for Labelled Standard (min)	Sample Pre-treatment, if other	Extraction Technique	Number of Steps (if staggered extraction)	Extraction Solvent(s)	Total Extraction Time (min)	Additional Information
1	10	Yes		Homogenisation	QuEChERS - modified AOAC		Acetonitrile with 1% Acetic Acid	Sonicate 30 min at 30-35 degrees	Geno/Grinder 14min & Centrifuge 10min. In this method the linear standards are used to quantify both the linear as well as the branched isomers.
2	2.018 and 2.005 (duplicate)	Yes	15 min	NA	Solid-Liquid Extraction (vortexed and centrifuged)	NA	2% formic acid in acetonitrile	8 min	Extraction using Merris-Minimix shaker
3	2g	Yes	10	Homogenisation	Alkaline Digestion	N/A	Basic MeOH	60	
4	NA	NA	NA	NA	NA	NA	NA	NA	NA
5	1	Yes	NA	NA	QuEChERS	NA	ACN	20	
6	5	Yes			QuEChERS		ACN	30	
7	1	Yes	30	Homogenising	Multiple, see additional info		ACN	2 x 15 min	Digestion with 200mM NaOH in methanol, then extraction with acetonitrile.
8	5	Yes	30-60	5 mL reagent water acidified with 150µL formic acid added prior to extraction	QuEChERS		ACN	15	
9	5g	Yes	30		QuEChERS		ACN	30	

Lab. Code	Sample Weight (g)	Labelled Standard(s) Added Before Extraction?	Equilibration Time for Labelled Standard (min)	Sample Pre-treatment, if other	Extraction Technique	Number of Steps (if staggered extraction)	Extraction Solvent(s)	Total Extraction Time (min)	Additional Information
10	0.5	Yes		Homogenisation	Solid-Liquid Extraction (vortexed and centrifuged)		MeOH	60	
11	10	Yes	no		QuEChERS		ACN	60	
12	0.2	Yes			Alkaline Digestion		NaOH/MeOH	60	
13	0.1	Yes			Alkaline Digestion				
14	1.9904	Yes	15	pH Adjustment	Alkaline Digestion		ACN	30 mins x2	Alkaline digestion, followed by solid/liquid extraction.
15									
16									
17									
19	1			Homogenisation	Alkaline Digestion		KOH/MeOH		Combination of Shaker/Sonication for extraction
20	1.0259	Yes	30		Solid-Liquid Extraction (vortexed and centrifuged)	3 (sonicate, vortex, centrifuge)	ACN	90	
21	1	Yes	30		Alkaline Digestion		KOH/MeOH	480	
22	2.12 g	Yes	30 min		Solid-Liquid Extraction (vortexed and centrifuged)	3	KOH/MeOH	30	

Table 215 Participant Methodology – Sample S2 Spinach Sample Clean-Up and Concentration

Lab. Code	Carbon Clean-Up?	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Elution Solvent	Final pH Adjustment	Additional Information
1				envicarb			
2	Yes	50°C	Variable	None	Not Applicable	No	Carbon clean up using dSPE (C18, Envicarb, MgSO4)
3	Yes	N/A	N/A	Solid-Phase Extraction	Basic MeOH	Yes	
4	NA	NA	NA	NA	NA	NA	NA
5	Yes	45	40-60	Solid-Phase Extraction	Basic MeOH	No	
6		Ambient		Solid-Phase Extraction	ACN	No	
7	Yes	Room temperature		Liquid-liquid extraction	MeOH	No	Clean up: liquid-liquid extraction with n-hexane, then Bond Elut Carbon SPE
8	Yes	60	60-90	Solid-Phase Extraction	MeOH, 0.3% NH3	No	
9	Yes	45	30	Solid-Phase Extraction	Basic ACN and Acetone	No	
10	Yes	40	60	Filtration			
11	No			None			
12		RT		Solid-Phase Extraction	NH4OH/MeOH		
13						Yes	
14	Yes			Solid-Phase Extraction	2% NH4OH		2D-SPE (Waters Oasis WAX SPE + Strata GBC cartridge)
15							
16							

Lab. Code	Carbon Clean-Up?	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Elution Solvent	Final pH Adjustment	Additional Information
17							
19		Room temperature	3 hour shake, 12 hour sonication bath	SPE (WAX 150mg/6cc)			
20	No	Room Temp.	60	Solid-Phase Extraction	MeOH, 1% NH4OH in MeOH	No	
21	Yes	35	90	Filtration	NH4OH/MeOH	No	
22	Yes	55C	200 min	Solid-Phase Extraction	NH4OH/MeOH	No	

Table 216 Participant Methodology – Sample S2 Spinach Instrumental Technique

Lab. Code	Instrument	Dilution Before Analysis and Dilution Factor (if applicable)	Blank Correction?	Additional Information
1	LC-Orbitrap	x10 diluted sample run for EtFOSE only	No	
2	LC-MSMS or LC-QQQ	No	Yes	NA
3	LC-MSMS or LC-QQQ	5	No	
4	NA	NA	NA	NA
5	LC-MSMS or LC-QQQ	NA	Yes	NA
6	LC-Orbitrap	DF10, DF100	No	
7	LC-MSMS or LC-QQQ	No	No	
8	Sciex 6500+ Triple		No	C-18 LC column (3µm, 150mm x 2mm)
9	LC-MSMS or LC-QQQ	No	No	
10	LC-MSMS or LC-QQQ	No	No	
11	LC-MSMS or LC-QQQ	yes, 10x	Yes	
12	LC-MSMS or LC-QQQ		Yes	
13	LC-MSMS or LC-QQQ			
14	LC-MSMS or LC-QQQ	No	No	N/A
15				

Lab. Code	Instrument	Dilution Before Analysis and Dilution Factor (if applicable)	Blank Correction?	Additional Information
16				
17				
19	LC-MSMS or LC-QQQ	No	No	
20	LC-MSMS or LC-QQQ		Yes	
21	LC-MSMS or LC-QQQ	No	No	
22	LC-MSMS or LC-QQQ	No	No	

Table 217 Participant Methodology – Sample S2 Spinach Labelled Standards

Lab. Code	Labelled Standard Source	Recovery Correction?	Standard Method Used?	Additional Information
1	Wellington	Yes	In house	
2	Wellington Laboratory	Yes	No	NA
3	Wellington Labs	Yes	No. In-house	
4	NA	NA	NA	NA
5	Wellington, CIL	Yes	NA	NA
6	Wellington	Yes		Results corrected by ISTD added before instrumentation
7	Wellington	Yes	Isotopic Dilution	
8	Cambridge (FTS compounds); Wellington (remainder)	Yes		
9	Wellington, Cambridge Isotope laboratories	No	No	
10	Wellington Laboratories	Yes		
11				
12	Wellington	No		
13				
14	Wellington (Greyhound)	Yes	No - method developed in house	N/A



Lab. Code	Labelled Standard Source	Recovery Correction?	Standard Method Used?	Additional Information
15				
16				
17	Wellington, Cambridge Isotopes	Yes	US FDA Foods Program Compendium of Analytical Laboratory Methods; method C-010.02	d5NN EtFOSAA added before instrument analysis
19	Wellington	Yes	No	
20	Wellington	Yes	No	
21				
22	Wellington	Yes	No	

Table 218 Labelled Standards for Sample S2 Spinach PFBS

Lab. Code	Before Extraction	Before Instrument Analysis
1	Sodium perfluoro-1-[2,3,4 <sup>13</sup> C <sub>3</sub> ] butanesulfonate M3PFBS	
2	M3PFBS	NA
3	<sup>13</sup> C <sub>3</sub> -PFBS	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	PFBS-13C3
7	<sup>13</sup> C <sub>3</sub> -PFBS	<sup>13</sup> C <sub>3</sub> -PFHxS
8	yes	
9	<sup>13</sup> C <sub>3</sub> -PFBS	
10	<sup>13</sup> C <sub>3</sub> -PFBS	
11		
12	<sup>13</sup> C <sub>3</sub> -PFBS	
13		
14	<sup>13</sup> C-PFBS	MPFOS
15		
16		
17	M3 PFBS	
19	<sup>13</sup> C <sub>3</sub> PFBS	
20	18O <sub>2</sub> -PFHxS	18O <sub>2</sub> -PFOS
21	<sup>13</sup> C <sub>3</sub> PFBS	
22	<sup>13</sup> C <sub>3</sub> -PFBS	18O <sub>2</sub> -PFHxS

Table 219 Labelled Standards for Sample S2 Spinach PFPeS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M5PFHxA	NA
3	18O <sub>2</sub> -PFHxS	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	PFOS-13C4
7	18O <sub>2</sub> -PFHxS	<sup>13</sup> C <sub>3</sub> -PFHxS
8		
9	N/A	
10	16O <sub>2</sub> -PFHxS	
11		
12	<sup>13</sup> C <sub>3</sub> -PFBS	
13		
14		
15		
16		
17	M3 PFHxS	
19	<sup>13</sup> C <sub>3</sub> PFBS	
20	18O <sub>2</sub> -PFHxS	18O <sub>2</sub> -PFOS
21	<sup>13</sup> C <sub>3</sub> PFBS	
22	<sup>13</sup> C <sub>3</sub> -PFHxS	18O <sub>2</sub> -PFHxS

Table 220 Labelled Standards for Sample S2 Spinach PFHxS

Lab. Code	Before Extraction	Before Instrument Analysis
1	Sodium perfluoro-1-[1,2,3 13C3] hexanesulfonate M3PFHxS	
2	M3PFHxS	NA
3	18O2-PFHxS	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	PFHxS-18O2
7	18O2-PFHxS	13C3-PFHxS
8		
9	18O2-PFHxS	
10	16O2-PFHxS	
11		
12	13C3-PFHxS	
13		
14	13C-PFHxS	MPFOS
15		
16		
17	M3 PFHxS	
19	18O2 PFHxS	
20		
21	13C3 PFHxS	PFHxS18O2
22	13C3-PFHxS	18O2-PFHxS

Table 221 Labelled Standards for Sample S2 Spinach PFHxS (linear)

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M3PFHxS	NA
3	18O2-PFHxS	N/A
4	NA	NA
5	Yes	NA
6	PFOS-13C8	PFHxS-18O2
7	18O2-PFHxS	13C3-PFHxS
8	yes	
9	18O2-PFHxS	
10	NT	
11		
12	13C3-PFHxS	
13		
14	13C-PFHxS	MPFOS
15		
16		
17		
19	18O2 PFHxS	
20	18O2-PFHxS	18O2-PFOS
21	13C3 PFHxS	
22	13C3-PFHxS	18O2-PFHxS

Table 222 Labelled Standards for Sample S2 Spinach PFHpS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M3PFHxS	NA
3	13C4-PFOS	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	PFOS-C4
7	18O2-PFHxS	13C3-PFHxS
8		
9	N/A	
10	13C8-PFOS	
11		
12	13C3-PFHxS	
13		
14	13C-PFOS	MPFOS
15		
16		
17	M3 PFHxS	
19	18O2 PFHxS	
20	18O2-PFHxS	18O2-PFOS
21	13C3 PFHxS	
22	13C8-PFOS	13C4-PFOS

Table 223 Labelled Standards for Sample S2 Spinach PFOS

Lab. Code	Before Extraction	Before Instrument Analysis
1	Sodium perfluoro-1-[ 13C8] octanesulfonate M8PFOS	
2	M8PFOS	NA
3	13C4-PFOS	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	PFOS-C4
7	13C4-PFOS	13C8-PFOS
8	yes	
9	13C8-PFOS	
10	13C4-PFOS	
11		
12	13C8-PFOS	
13		
14	13C-PFOS	MPFOS
15		
16		
17	13C PFOS	
19	13C4 PFOS	
20	13C4-PFOS	18O2-PFOS
21	13C8 PFOS	13C4 PFOS
22	13C8-PFOS	13C4-PFOS

Table 224 Labelled Standards for Sample S2 Spinach PFOS (linear)

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M8PFOS	NA
3	13C4-PFOS	N/A
4	NA	NA
5	Yes	NA
6	PFOS-13C8	PFOS-C4
7	13C4-PFOS	13C8-PFOS
8		yes
9	13C8-PFOS	
10	13C8-PFOS	
11	Yes	
12	13C8-PFOS	
13		
14	13C-PFOS	MPFOS
15		
16		
17		
19	13C4 PFOS	
20	13C4-PFOS	18O2-PFOS
21	13C8 PFOS	
22	13C8-PFOS	13C4-PFOS

Table 225 Labelled Standards for Sample S2 Spinach PFNS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M8PFOS	NA
3	13C4-PFOS	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	PFBS-13C3
7	13C4-PFOS	13C8-PFOS
8		
9	N/A	
10	NT	
11		
12	13C8-PFOS	
13		
14		
15		
16		
17	13C PFOS	
19	13C4 PFOS	
20	13C4-PFOS	18O2-PFOS
21	13C8 PFOS	
22	13C8-PFOS	13C4-PFOS

Table 226 Labelled Standards for Sample S2 Spinach PFDS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M8PFOS	NA
3	13C4-PFOS	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	PFBA-13C4
7	13C4-PFOS	13C8-PFOS
8		
9	N/A	
10	13C8-PFOS	
11		
12	13C8-PFOS	
13		
14	13C-PFOS	MPFOS
15		
16		
17	13C PFOS	
19	13C4 PFOS	
20	13C4-PFOS	18O2-PFOS
21	13C8 PFOS	
22	13C8-PFOS	13C4-PFOS

Table 227 Labelled Standards for Sample S2 Spinach PFBA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[13C4]butanoic acid MPFBA	
2	M4PFBA	NA
3	13C4-PFBA	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	PFBA-13C4
7	13C4-PFBA	13C3-PFBA
8	yes	
9	13C4-PFBA	
10	13C4-PFBA	
11	Yes	
12	13C4-PFBA	
13		
14	13C-PFBA	M3PFBA
15		
16		
17	M3 PFBA	
19	13C2 PFHxA	
20	13C4-PFBA	13C8-PFOA
21	13C4 PFBA	13C3 PFBA
22	13C4-PFBA	13C3-PFBA

Table 228 Labelled Standards for Sample S2 Spinach PFPeA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[13C5]pentanoic acid M5PFPeA	
2	M5PFPeA	NA
3	13C3-PFPeA	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	PFPeA-13C3
7	13C4-PFPeA	13C5 -PFPeA
8	yes	
9	13C5-PFPeA	
10	13C5-PFPeA	
11		
12	13C5-PFPeA	
13		
14	13C-PFPeA	M3PFBA
15		
16		
17	M3 PFPeA	
19	13C4 PFHpA	
20	13C5-PFPeA	13C8-PFOA
21	13C5 PFPeA	
22	13C5-PFPeA	13C2-PFHxA

Table 229 Labelled Standards for Sample S2 Spinach PFHxA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2,3,4,6- 13C5]hexanoic acid M5PFHxA	
2	M5PFHxA	NA
3	13C2-PFHxA	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	PFHxA-13C2
7	13C2-PFHxA	13C5 -PFPeA
8	yes	
9	13C2-PFHxA	
10	13C5-PFHxA	
11		
12	13C5-PFHxA	
13		
14	13C-PFHxA	M2PFOA
15		
16		
17	M5 PFHxA	
19	13C2 PFHxA	
20	13C5-PFHxA	13C8-PFOA
21	13C5 PFHxA	13C2 PFHxA
22	13C5-PFHxA	13C2-PFHxA

Table 230 Labelled Standards for Sample S2 Spinach PFHpA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]heptanoic acid M4PFHpA	
2	MPFHpA	NA
3	<sup>13</sup> C <sub>4</sub> -PFHpA	N/A
4	NA	NA
5	NA	NA
6	PFOS- <sup>13</sup> C <sub>8</sub>	PFHpA- <sup>13</sup> C <sub>4</sub>
7	<sup>13</sup> C <sub>3</sub> -PFHpA	<sup>13</sup> C <sub>8</sub> -PFOA
8		
9	<sup>13</sup> C <sub>4</sub> -PFHpA	
10	<sup>13</sup> C <sub>4</sub> -PFHpA	
11		
12	<sup>13</sup> C <sub>4</sub> -PFHpA	
13		
14	<sup>13</sup> C-PFHpA	M2PFOA
15		
16		
17	M5 PFHxA	
19	<sup>13</sup> C <sub>4</sub> PFHpA	
20	<sup>13</sup> C <sub>4</sub> -PFHpA	<sup>13</sup> C <sub>8</sub> -PFOA
21	<sup>13</sup> C <sub>4</sub> PFHpA	
22	<sup>13</sup> C <sub>4</sub> -PFHpA	<sup>13</sup> C <sub>4</sub> -PFOA

Table 231 Labelled Standards for Sample S2 Spinach PFOA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[ <sup>13</sup> C <sub>8</sub> ]octanoic acid M8PFOA	
2	M8PFOA	NA
3	<sup>13</sup> C <sub>4</sub> -PFOA	N/A
4	NA	NA
5	Yes	NA
6	PFOS- <sup>13</sup> C <sub>8</sub>	PFOA- <sup>13</sup> C <sub>4</sub>
7	<sup>13</sup> C <sub>4</sub> -PFOA	<sup>13</sup> C <sub>8</sub> -PFOA
8	yes	yes
9	<sup>13</sup> C <sub>8</sub> -PFOA	
10	<sup>13</sup> C <sub>8</sub> -PFOA	
11	Yes	
12	<sup>13</sup> C <sub>8</sub> -PFOA	
13		
14	<sup>13</sup> C-PFOA	M2PFOA
15		
16		
17	<sup>13</sup> C PFOA	
19	<sup>13</sup> C <sub>4</sub> PFOA	
20	<sup>13</sup> C <sub>4</sub> -PFOA	<sup>13</sup> C <sub>8</sub> -PFOA
21	<sup>13</sup> C <sub>8</sub> PFOA	<sup>13</sup> C <sub>4</sub> PFOA
22	<sup>13</sup> C <sub>8</sub> -PFOA	<sup>13</sup> C <sub>4</sub> -PFOA



Table 232 Labelled Standards for Sample S2 Spinach PFNA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[13C9]nonanoic acid M9PFNA	
2	M9PFNA	NA
3	13C5-PFNA	N/A
4	NA	NA
5	Yes	NA
6	PFOS-13C8	PFNA-13C5
7	13C5-PFNA	13C8-PFOA
8		
9	13C5-PFNA	
10	13C5-PFNA	
11		
12	13C9-PFNA	
13		
14	13C-PFNA	M2PFOA
15		
16		
17	13C PFOA	
19	13C5 PFNA	
20	13C9-PFNA	13C5-PFNA
21	13C9 PFNA	13C5 PFNA
22	13C9-PFNA	13C5-PFNA

Table 233 Labelled Standards for Sample S2 Spinach PFDA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2,3,4,6- 13C6]decanoic acid M6PFDA	
2	M6PFDA	NA
3	13C2-PFDA	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	PFDA-13C2
7	13C2-PFDA	13C8-PFOA
8		
9	13C6-PFDA	
10	13C6-PFDA	
11	Yes	
12	13C6-PFDA	
13		
14	13C-PFDA	MPFDA
15		
16		
17	13C PFOA	
19	13C2 PFDA	
20	13C2-PFDA	13C5-PFNA
21	13C6 PFDA	13C2 PFDA
22	13C6-PFDA	13C2-PFDA

Table 234 Labelled Standards for Sample S2 Spinach PFUdA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2,3,4,6,7- <sup>13</sup> C7]undecanoic acid M7PFUdA	
2	M7PFUnDA	NA
3	<sup>13</sup> C2-PFUdA	N/A
4	NA	NA
5	NA	NA
6	PFOS- <sup>13</sup> C8	PFUNDA- <sup>13</sup> C2
7	<sup>13</sup> C2-PFUdA	<sup>13</sup> C8-PFOA
8	yes	yes
9	<sup>13</sup> C2-PFUnA	
10	<sup>13</sup> C2-PFUnDA	
11		
12	<sup>13</sup> C7-PFUnA	
13		
14	<sup>13</sup> C-PFUdA	MPFDA
15		
16		
17	MPFUdA	
19	<sup>13</sup> C2 PFUnA	
20	<sup>13</sup> C2-PFUdA	<sup>13</sup> C5-PFNA
21	<sup>13</sup> C7 PFUnA	
22	<sup>13</sup> C7-PFUNA	<sup>13</sup> C2-PFDA

Table 235 Labelled Standards for Sample S2 Spinach PFTrDA

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	MPFDoDA	NA
3	<sup>13</sup> C2-PFTeDA	N/A
4	NA	NA
5	NA	NA
6	PFOS- <sup>13</sup> C8	PFTeDA- <sup>13</sup> C2
7	<sup>13</sup> C2-PFTeDA	<sup>13</sup> C8-PFOA
8		
9	N/A	
10	<sup>13</sup> C2-PFTeDA	
11		
12	<sup>13</sup> C2-PFDoA	
13		
14	<sup>13</sup> C-PFTeDA	MPFDA
15		
16		
17	MPFDoA	
19	<sup>13</sup> C2 PFDoA	
20	<sup>13</sup> C2-PFHxDA	<sup>13</sup> C2-PFTeDA
21	<sup>13</sup> C2 PFDoA	
22	<sup>13</sup> C2-PFDoA/ <sup>13</sup> C2-PFTeDA	<sup>13</sup> C2-PFDA

Table 236 Labelled Standards for Sample S2 Spinach PFOSA

Lab. Code	Before Extraction	Before Instrument Analysis
1	N-methyl-d3-perfluoro-1-octanesulfonamide	
2	MPFOSA	NA
3	D3-M PFOSA	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	FOSA-13C8
7	13C8-FOSA	
8	yes	
9	13C8-FOSA	
10	13C8-FOSA	
11		
12		
13		
14		
15		
16		
17	M8 FOSA	
19	13C8 PFOSA	
20	13C8-PFOSA	13C2-PFTeDA
21	13C8 PFOSA	
22	13C8-PFOSA	13C4-PFOS

Table 237 Labelled Standards for Sample S2 Spinach EtFOSA

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	d-NEtFOSA-M	NA
3	D3-Me-FOSAA	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	EtFOSA-D5
7	D5-N-Et FOSA	
8		
9	d5-N-EtFOSA	
10	d5-EtFOSA	
11		
12		
13		
14		
15		
16		
17		
19	d-N-EtFOSA-M	
20	13C8-PFOSA	13C2-PFTeDA
21	d5-N-EtFOSA	
22	D5-N-EtFOSA	13C4-PFOS

Table 238 Labelled Standards for Sample S2 Spinach EtFOSAA

Lab. Code	Before Extraction	Before Instrument Analysis
1	d7-N-MeFOSE-M 2-(N-methyl-d3-perfluoro-1-octanesulfonamido) ethand4-ol	
2	d5-NEtFOSAA	NA
3	D7-Me-FOSE	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	EtFOSAA-D5
7	D5-N-Et FOSAA	
8		yes
9	d5-NEtFOSAA	
10	d5-EtFOSAA	
11		
12		
13		
14		
15		
16		
17		
19	d7-NEtFOSAA	
20	NT	NT
21	d5-N-EtFOSAA	
22	D5-N-EtFOSAA	13C2-D4-6:2 FTS

Table 239 Labelled Standards for Sample S2 Spinach 6:2FTS

Lab. Code	Before Extraction	Before Instrument Analysis
1	M2-6:2FTS -1H,1H,2H,2H-perfluoro1-[1,2-13C2]-octane sulfonate (6:2)	
2	M6:2 FTS	NA
3	13C2,12C6 6:2-FTS	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	6:2 FTS-13C2
7	13C2-6:2 FTS	
8	yes	
9	13C2-6-2 FTS	
10	13C2-6:2 FTS	
11		
12		
13		
14	13C-6:2 FTS	M2PFOA
15		
16		
17	13C2D4 6:2 FTS	
19	M2-6:2 FTS	
20	NT	NT
21	13C2 4:2 FTS	
22	13C2-6:2 FTS	13C2-D4-6:2 FTS

Table 240 Labelled Standards for Sample S2 Spinach GenX

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M3HFPO-DA	NA
3	13C312C3HF11O3	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	
7	13C4-PFOA	
8	yes	
9	13C3-GenX	
10	NT	
11		
12	13C8-PFOA	
13		
14		
15		
16		
17	M3 HFPO	
19	13C3 HFPO-DA	
20	NT	NT
21	13C3 HFPO-DA	
22	13C3-HFPO-DA	13C2-PFHxA

Table 241 Labelled Standards for Sample S2 Spinach ADONA

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	MPFHpA	NA
3	13C4-PFHpA	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	
7	13C4-PFOA	
8		
9	N/A	
10	NT	
11		
12	13C8-PFOA	
13		
14		
15		
16		
17	13C PFOA	
19	13C4 PFOS	
20	NT	NT
21	13C3 HFPO-DA	
22	13C3-HFPO-DA	13C2-PFHxA

Table 242 Labelled Standards for Sample S2 Spinach 9CI-PF3ONS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M8PFOS	NA
3	13C4-PFOS	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	PFPeA-13C3
7	13C4-PFOS	13C8-PFOS
8		
9	N/A	
10	NT	
11		
12	13C8-PFOS	
13		
14		
15		
16		
17	M3 PFHxS	
19	13C4 PFOS	
20	NT	NT
21	13C3 HFPO-DA	
22	13C3-HFPO-DA	13C2-PFHxA

Table 243 Labelled Standards for Sample S2 Spinach 11CI-PF3OUdS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	MPFDoDA	NA
3	13C4-PFOS	N/A
4	NA	NA
5	NA	NA
6	PFOS-13C8	FOSA-13C8
7	13C4-PFOS	13C8-PFOS
8		
9	N/A	
10	NT	
11		
12	13C8-PFOS	
13		
14		
15		
16		
17	M3 PFHxS	
19		
20	NT	NT
21	13C3 HFPO-DA	
22	13C3-HFPO-DA	13C2-PFHxA

Table 244 Participant Methodology – Sample S3 Bovine Liver Sample Preparation and Extraction

Lab. Code	Sample Weight (g)	Labelled Standard(s) Added Before Extraction?	Equilibration Time for Labelled Standard (min)	Sample Pre-treatment, if other	Extraction Technique	Number of Steps (if staggered extraction)	Extraction Solvent(s)	Total Extraction Time (min)	Additional Information
1	1	Yes		Homogenisation	QuEChERS - modified AOAC		Acetonitrile with 1% Acetic Acid		Geno/Grinder 14min & Centrifuge 10min. In this method the linear standards are used to quantify both the linear as well as the branched isomers.
2	1.053 and 1.062 (duplicate)	Yes	15 min	NA	Solid-Liquid Extraction (vortexed and centrifuged)	NA	2% formic acid in acetonitrile	8 min	Extraction using Merris-Minimix shaker
3	1g	Yes	10	Homogenisation	Alkaline Digestion	N/A	Basic MeOH	60	
4	NA	NA	NA	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA	NA	NA	NA
7	0.5	Yes	30	Homogenising	Multiple, see additional info		ACN	2 x 15 min	Digestion with 200mM NaOH in methanol, then extraction with acetonitrile.
8	5	Yes	30-60	15 mL reagent water acidified with 150µL formic acid added prior to extraction	QuEChERS		ACN	15	
9	5g	Yes	30		QuEChERS		ACN	30	

Lab. Code	Sample Weight (g)	Labelled Standard(s) Added Before Extraction?	Equilibration Time for Labelled Standard (min)	Sample Pre-treatment, if other	Extraction Technique	Number of Steps (if staggered extraction)	Extraction Solvent(s)	Total Extraction Time (min)	Additional Information
10	0.5	Yes		Homogenisation	Solid-Liquid Extraction (vortexed and centrifuged)		MeOH	60	
11	NA	NA	NA	NA	NA	NA	NA	NA	NA
12	0.2	Yes			Alkaline Digestion		NaOH/MeOH	60	
13	0.1	Yes			Alkaline Digestion				
14	0.5756	Yes	15	pH Adjustment	Alkaline Digestion		ACN	30 mins x2	Alkaline digestion, followed by solid/liquid extraction.
15	NA	NA	NA	NA	NA	NA	NA	NA	NA
16	NA	NA	NA	NA	NA	NA	NA	NA	NA
17									
19	1.05			Homogenisation	Alkaline Digestion		KOH/MeOH		Combination of Shaker/Sonication for extraction
20	1.0383	Yes	30		Solid-Liquid Extraction (vortexed and centrifuged)	3 (sonicate, vortex, centrifuge)	ACN	90	
21	0.5	Yes	30		Alkaline Digestion		KOH/MeOH	480	
22	NA	NA	NA	NA	NA	NA	NA	NA	NA



Table 245 Participant Methodology – Sample S3 Bovine Liver Sample Clean-Up and Concentration

Lab. Code	Carbon Clean-Up?	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Elution Solvent	Final pH Adjustment	Additional Information
1	Yes			envicarb			
2	No	50°C	Variable	None	Not Applicable	No	Clean up using dSPE (Z-Sep sorbent)
3	Yes	N/A	N/A	Solid-Phase Extraction	Basic MeOH	Yes	
4	NA	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA	NA
7	Yes	Room temperature		Liquid-liquid extraction	MeOH	No	Clean up: liquid-liquid extraction with n-hexane, then Bond Elut Carbon SPE
8	Yes	60	60-90	Solid-Phase Extraction	MeOH, 0.3% NH3	No	
9	Yes	45	30	Solid-Phase Extraction	Basic ACN and Acetone	No	
10	Yes	40	60	Filtration			
11	NA	NA	NA	NA	NA	NA	NA
12		RT		Solid-Phase Extraction	NH4OH/MeOH		
13						Yes	
14	Yes			Solid-Phase Extraction	2% NH4OH		2D-SPE (Waters Oasis WAX SPE + Strata GBC cartridge)
15	NA	NA	NA	NA	NA	NA	NA
16	NA	NA	NA	NA	NA	NA	NA
17							

Lab. Code	Carbon Clean-Up?	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Clean-Up	Elution Solvent	Final pH Adjustment	Additional Information
19		Room temperature	3 hour shake, 12 hour sonication bath	SPE (WAX 150mg/6cc)			Combination of Shaker/Sonication for extraction
20	No	Room Temp.	60	Solid-Phase Extraction	MeOH, 1% NH4OH in MeOH	No	
21	Yes	35	90	Filtration	NH4OH/MeOH	No	
22	NA	NA	NA	NA	NA	NA	NA

Table 246 Participant Methodology – Sample S3 Bovine Liver Instrumental Technique

Lab. Code	Instrument	Dilution Before Analysis and Dilution Factor (if applicable)	Blank Correction?	Additional Information
1	LC-Orbitrap		No	
2	LC-MSMS or LC-QQQ	No	Yes	NA
3	LC-MSMS or LC-QQQ	5	No	
4	NA	NA	NA	NA
5	NA	NA	NA	NA
6	NA	NA	NA	NA
7	LC-MSMS or LC-QQQ	No	No	
8	Sciex 6500+ Triple		No	C-18 LC column (3µm, 150mm x 2mm)
9	LC-MSMS or LC-QQQ	No	No	
10	LC-MSMS or LC-QQQ	no	No	
11	NA	NA	NA	NA
12	LC-MSMS or LC-QQQ		Yes	
13	LC-MSMS or LC-QQQ			
14	LC-MSMS or LC-QQQ	No	No	N/A
15	NA	NA	NA	NA

Lab. Code	Instrument	Dilution Before Analysis and Dilution Factor (if applicable)	Blank Correction?	Additional Information
16	NA	NA	NA	NA
17				
19	LC-MSMS or LC-QQQ	No	No	
20	LC-MSMS or LC-QQQ		Yes	
21	LC-MSMS or LC-QQQ	No	No	
22	NA	NA	NA	NA

Table 247 Participant Methodology – Sample S3 Bovine Liver Labelled Standards

Lab. Code	Labelled Standard Source	Recovery Correction?	Standard Method Used?	Additional Information
1	Wellington	Yes	In house	
2	Wellington Laboratory	Yes	No	NA
3	Wellington Labs	Yes	No. In-house	
4	NA	NA	NA	NA
5	NA	NA	NA	NA
6	NA	NA	NA	NA
7	Wellington	Yes	Isotopic Dilution	
8	Cambridge (FTS compounds); Wellington (remainder)	Yes		
9	Wellington, Cambridge Isotope laboratories	No	No	
10	Wellington Laboratories	Yes		
11	NA	NA	NA	NA
12	Wellington	No		
13				
14	Wellington (Greyhound)	Yes	No - method developed in house	N/A
15	NA	NA	NA	NA

Lab. Code	Labelled Standard Source	Recovery Correction?	Standard Method Used?	Additional Information
16	NA	NA	NA	NA
17	Wellington, Cambridge Isotopes	Yes	US FDA Foods Program Compendium of Analytical Laboratory Methods; method C-010.02	d5NN EtFOSAA added before instrument analysis
19	Wellington	Yes	No	
20	Wellington	Yes	No	
21				
22	NA	NA	NA	NA

Table 248 Labelled Standards for Sample S3 Bovine Liver PFBS

Lab. Code	Before Extraction	Before Instrument Analysis
1	Sodium perfluoro-1-[2,3,4 <sup>13</sup> C <sub>3</sub> ] butanesulfonate M3PFBS	
2	M3PFBS	NA
3	<sup>13</sup> C <sub>3</sub> -PFBS	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	<sup>13</sup> C <sub>3</sub> -PFBS	<sup>13</sup> C <sub>3</sub> -PFHxS
8	yes	
9	<sup>13</sup> C <sub>3</sub> -PFBS	
10	<sup>13</sup> C <sub>3</sub> -PFBS	
11	NA	NA
12	<sup>13</sup> C <sub>3</sub> -PFBS	
13		
14	<sup>13</sup> C-PFBS	MPFOS
15	NA	NA
16	NA	NA
17	M3 PFBS	
19	<sup>13</sup> C <sub>3</sub> PFBS	
20	18O <sub>2</sub> -PFHxS	18O <sub>2</sub> -PFOS
21	<sup>13</sup> C <sub>3</sub> PFBS	
22	NA	NA

Table 249 Labelled Standards for Sample S3 Bovine Liver PFPeS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M5PFHxA	NA
3	18O <sub>2</sub> -PFHxS	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	18O <sub>2</sub> -PFHxS	<sup>13</sup> C <sub>3</sub> -PFHxS
8		
9	N/A	
10	16O <sub>2</sub> -PFHxS	
11	NA	NA
12	<sup>13</sup> C <sub>3</sub> -PFBS	
13		
14		
15	NA	NA
16	NA	NA
17	M3 PFHxS	
19	<sup>13</sup> C <sub>3</sub> PFBS	
20	18O <sub>2</sub> -PFHxS	18O <sub>2</sub> -PFOS
21	<sup>13</sup> C <sub>3</sub> PFBS	
22	NA	NA

Table 250 Labelled Standards for Sample S3 Bovine Liver PFHxS

Lab. Code	Before Extraction	Before Instrument Analysis
1	Sodium perfluoro-1-[1,2,3 13C3] hexanesulfonate M3PFHxS	
2	M3PFHxS	NA
3	18O2-PFHxS	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	18O2-PFHxS	13C3-PFHxS
8		
9	18O2-PFHxS	
10	16O2-PFHxS	
11	NA	NA
12	13C3-PFHxS	
13		
14	13C-PFHxS	MPFOS
15	NA	NA
16	NA	NA
17	M3 PFHxS	
19	18O2 PFHxS	
20		
21	13C3 PFHxS	PFHxS18O2
22	NA	NA

Table 251 Labelled Standards for Sample S3 Bovine Liver PFHxS (linear)

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M3PFHxS	NA
3	18O2-PFHxS	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	18O2-PFHxS	13C3-PFHxS
8	yes	
9	18O2-PFHxS	
10	NT	
11	NA	NA
12	13C3-PFHxS	
13		
14	13C-PFHxS	MPFOS
15	NA	NA
16	NA	NA
17		
19	18O2 PFHxS	
20	18O2-PFHxS	18O2-PFOS
21	13C3 PFHxS	
22	NA	NA

Table 252 Labelled Standards for Sample S3 Bovine Liver PFHxS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M3PFHxS	NA
3	13C4-PFOS	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	18O2-PFHxS	13C3-PFHxS
8		
9	N/A	
10	13C8-PFOS	
11	NA	NA
12	13C3-PFHxS	
13		
14	13C-PFOS	MPFOS
15	NA	NA
16	NA	NA
17	M3 PFHxS	
19	18O2 PFHxS	
20	18O2-PFHxS	18O2-PFOS
21	13C3 PFHxS	
22	NA	NA

Table 253 Labelled Standards for Sample S3 Bovine Liver PFOS

Lab. Code	Before Extraction	Before Instrument Analysis
1	Sodium perfluoro-1-[ 13C8] octanesulfonate M8PFOS	
2	M8PFOS	NA
3	13C4-PFOS	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C4-PFOS	13C8-PFOS
8	yes	
9	13C8-PFOS	
10	13C4-PFOS	
11	NA	NA
12	13C8-PFOS	
13		
14	13C-PFOS	MPFOS
15	NA	NA
16	NA	NA
17	13C PFOS	
19	13C4 PFOS	
20	13C4-PFOS	18O2-PFOS
21	13C8 PFOS	13C4 PFOS
22	NA	NA

Table 254 Labelled Standards for Sample S3 Bovine Liver PFOS  
(linear)

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

Table 255 Labelled Standards for Sample S3 Bovine Liver PFNS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M6PFDA	NA
3	13C4-PFOS	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C4-PFOS	13C8-PFOS
8		
9	N/A	
10	NT	
11	NA	NA
12	13C8-PFOS	
13		
14		
15	NA	NA
16	NA	NA
17	13C PFOS	
19	13C4 PFOS	
20	13C4-PFOS	18O2-PFOS
21	13C8 PFOS	
22	NA	NA



Table 256 Labelled Standards for Sample S3 Bovine Liver PFDS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M7PFUnDA	NA
3	13C4-PFOS	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C4-PFOS	13C8-PFOS
8		
9	N/A	
10	13C8-PFOS	
11	NA	NA
12	13C8-PFOS	
13		
14	13C-PFOS	MPFOS
15	NA	NA
16	NA	NA
17	13C PFOS	
19	13C4 PFOS	
20	13C4-PFOS	18O2-PFOS
21	13C8 PFOS	
22	NA	NA

Table 257 Labelled Standards for Sample S3 Bovine Liver PFBA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[13C4]butanoic acid MPFBA	
2	M4PFBA	NA
3	13C4-PFBA	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C4-PFBA	13C3-PFBA
8	yes	
9	13C4-PFBA	
10	13C4-PFBA	
11	NA	NA
12	13C4-PFBA	
13		
14	13C-PFBA	M3PFBA
15	NA	NA
16	NA	NA
17	M3 PFBA	
19	13C2 PFHxA	
20	13C4-PFBA	13C8-PFOA
21	13C4 PFBA	13C3 PFBA
22	NA	NA

Table 258 Labelled Standards for Sample S3 Bovine Liver PFPeA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[13C5]pentanoic acid M5PFPeA	
2	M5PFPeA	NA
3	13C3-PFPeA	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C4-PFPeA	13C5 -PFPeA
8	yes	
9	13C5-PFPeA	
10	13C5-PFPeA	
11	NA	NA
12	13C5-PFPeA	
13		
14	13C-PFPeA	M3PFBA
15	NA	NA
16	NA	NA
17	M3 PFPeA	
19	13C4 PFHpA	
20	13C5-PFPeA	13C8-PFOA
21	13C5 PFPeA	
22	NA	NA

Table 259 Labelled Standards for Sample S3 Bovine Liver PFHxA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2,3,4,6- 13C5]hexanoic acid M5PFHxA	
2	M5PFHxA	NA
3	13C2-PFHxA	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C2-PFHxA	13C5 -PFPeA
8	yes	
9	13C2-PFHxA	
10	13C5-PFHxA	
11	NA	NA
12	13C5-PFHxA	
13		
14	13C-PFHxA	M2PFOA
15	NA	NA
16	NA	NA
17	M5 PFHxA	
19	13C2 PFHxA	
20	13C5-PFHxA	13C8-PFOA
21	13C5 PFHxA	13C2 PFHxA
22	NA	NA

Table 260 Labelled Standards for Sample S3 Bovine Liver PFHpA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2,3,4- <sup>13</sup> C <sub>4</sub> ]heptanoic acid M4PFHpA	
2	MPFHpA	NA
3	<sup>13</sup> C <sub>4</sub> -PFHpA	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	<sup>13</sup> C <sub>3</sub> -PFHpA	<sup>13</sup> C <sub>8</sub> -PFOA
8		
9	<sup>13</sup> C <sub>4</sub> -PFHpA	
10	<sup>13</sup> C <sub>4</sub> -PFHpA	
11	NA	NA
12	<sup>13</sup> C <sub>4</sub> -PFHpA	
13		
14	<sup>13</sup> C-PFHpA	M2PFOA
15	NA	NA
16	NA	NA
17	M5 PFHxA	
19	<sup>13</sup> C <sub>4</sub> PFHpA	
20	<sup>13</sup> C <sub>4</sub> -PFHpA	<sup>13</sup> C <sub>8</sub> -PFOA
21	<sup>13</sup> C <sub>4</sub> PFHpA	
22	NA	NA

Table 261 Labelled Standards for Sample S3 Bovine Liver PFOA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[ <sup>13</sup> C <sub>8</sub> ]octanoic acid M8PFOA	
2	M8PFOA	NA
3	<sup>13</sup> C <sub>4</sub> -PFOA	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	<sup>13</sup> C <sub>4</sub> -PFOA	<sup>13</sup> C <sub>8</sub> -PFOA
8	yes	yes
9	<sup>13</sup> C <sub>8</sub> -PFOA	
10	<sup>13</sup> C <sub>8</sub> -PFOA	
11	NA	NA
12	<sup>13</sup> C <sub>8</sub> -PFOA	
13		
14	<sup>13</sup> C-PFOA	M2PFOA
15	NA	NA
16	NA	NA
17	<sup>13</sup> C PFOA	
19	<sup>13</sup> C <sub>4</sub> PFOA	
20	<sup>13</sup> C <sub>4</sub> -PFOA	<sup>13</sup> C <sub>8</sub> -PFOA
21	<sup>13</sup> C <sub>8</sub> PFOA	<sup>13</sup> C <sub>4</sub> PFOA
22	NA	NA

Table 262 Labelled Standards for Sample S3 Bovine Liver PFNA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[13C9]nonanoic acid M9PFNA	
2	M9PFNA	NA
3	13C5-PFNA	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C5-PFNA	13C8-PFOA
8		
9	13C5-PFNA	
10	13C5-PFNA	
11	NA	NA
12	13C9-PFNA	
13		
14	13C-PFNA	M2PFOA
15	NA	NA
16	NA	NA
17	13C PFOA	
19	13C5 PFNA	
20	13C9-PFNA	13C5-PFNA
21	13C9 PFNA	13C5 PFNA
22	NA	NA

Table 263 Labelled Standards for Sample S3 Bovine Liver PFDA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2,3,4,6- 13C6]decanoic acid M6PFDA	
2	M6PFDA	NA
3	13C2-PFDA	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C2-PFDA	13C8-PFOA
8		
9	13C6-PFDA	
10	13C6-PFDA	
11	NA	NA
12	13C6-PFDA	
13		
14	13C-PFDA	MPFDA
15	NA	NA
16	NA	NA
17	13C PFOA	
19	13C2 PFDA	
20	13C2-PFDA	13C5-PFNA
21	13C6 PFDA	13C2 PFDA
22	NA	NA

Table 264 Labelled Standards for Sample S3 Bovine Liver PFUdA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2,3,4,6,7- <sup>13</sup> C7]undecanoic acid M7PFUdA	
2	M7PFUnDA	NA
3	<sup>13</sup> C2-PFUdA	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	<sup>13</sup> C2-PFUdA	<sup>13</sup> C8-PFOA
8	yes	yes
9	<sup>13</sup> C2-PFUnA	
10	<sup>13</sup> C2-PFUnDA	
11	NA	NA
12	<sup>13</sup> C7-PFUnA	
13		
14	<sup>13</sup> C-PFUdA	MPFDA
15	NA	NA
16	NA	NA
17	MPFUdA	
19	<sup>13</sup> C2 PFUnA	
20	<sup>13</sup> C2-PFUdA	<sup>13</sup> C5-PFNA
21	<sup>13</sup> C7 PFUnA	
22	NA	NA

Table 265 Labelled Standards for Sample S3 Bovine Liver PFDoA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2- <sup>13</sup> C2]dodecanoic acid MPFDoA	
2	MPFDoDA	NA
3	<sup>13</sup> C2-PFDoDA	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	<sup>13</sup> C2-PFDoA	<sup>13</sup> C8-PFOA
8	yes	
9	<sup>13</sup> C2-PFDoA	
10	<sup>13</sup> C2-PFDoDA	
11	NA	NA
12	<sup>13</sup> C2-PFDoA	
13		
14	<sup>13</sup> C-PFDoA	MPFDA
15	NA	NA
16	NA	NA
17	MPFDoA	
19	<sup>13</sup> C2 PFDoA	
20	<sup>13</sup> C2-PFDoA	<sup>13</sup> C5-PFNA
21	<sup>13</sup> C2 PFDoA	
22	NA	NA

Table 266 Labelled Standards for Sample S3 Bovine Liver PFTeDA

Lab. Code	Before Extraction	Before Instrument Analysis
1	Perfluoro-n-[1,2 13C2]tetradecanoic acid M2PFTeDA	
2	MPFTeDA	NA
3	13C2-PFTeDA	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C2-PFTeDA	13C8-PFOA
8	yes	
9	13C2-PFTeDA	
10	13C2-PFTeDA	
11	NA	NA
12	13C2-PFTeDA	
13		
14	13C-PFTeDA	MPFDA
15	NA	NA
16	NA	NA
17	MPFTeDA	
19	13C2 PFTeDA	
20	13C2-PFHxDA	13C2-PFTeDA
21	13C2 PFTeDA	
22	NA	NA

Table 267 Labelled Standards for Sample S3 Bovine Liver PFOSA

Lab. Code	Before Extraction	Before Instrument Analysis
1	N-methyl-d3-perfluoro-1- octancesulfonamide	
2	MPFOSA	NA
3	D3-M PFOSA	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C8-FOSA	
8	yes	
9	13C8-FOSA	
10	13C8-FOSA	
11	NA	NA
12		
13		
14		
15	NA	NA
16	NA	NA
17	M8 FOSA	
19	13C8 PFOSA	
20	13C8-PFOSA	13C2-PFTeDA
21	13C8 PFOSA	
22	NA	NA

Table 268 Labelled Standards for Sample S3 Bovine Liver EtFOSA

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	d-NEtFOSA-M	NA
3	D3-Me-FOSAA	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	D5-N-Et FOSA	
8		
9	d5-N-EtFOSA	
10	d5-EtFOSA	
11	NA	NA
12		
13		
14		
15	NA	NA
16	NA	NA
17		
19	d-N-EtFOSA-M	
20	13C8-PFOSA	13C2-PFTeDA
21	d5-N-EtFOSA	
22	NA	NA

Table 269 Labelled Standards for Sample S3 Bovine Liver EtFOSAA

Lab. Code	Before Extraction	Before Instrument Analysis
1	d7-N-MeFOSE-M 2-(N-methyl-d3-perfluoro-1-octanesulfonamido) ethand4-ol	
2	d5-NEtFOSAA	NA
3	D7-Me-FOSE	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	D5-N-Et FOSAA	
8		yes
9	d5-NEtFOSAA	
10	d5-EtFOSAA	
11	NA	NA
12		
13		
14		
15	NA	NA
16	NA	NA
17		
19	d7-NEtFOSAA	
20	NT	NT
21	d5-N-EtFOSAA	
22	NA	NA

Table 270 Labelled Standards for Sample S3 Bovine Liver 8:2FTS

Lab. Code	Before Extraction	Before Instrument Analysis
1	M2-8:2FTS -1H,1H,2H,2H-perfluoro1-[1,2-13C2]-decane sulfonate (8:2)	
2	M8:2 FTS	NA
3	13C2 8:2-FTS	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C2-8:2 FTS	
8	yes	
9	13C2-8-2 FTS	
10	13C2-8:2 FTS	
11	NA	NA
12		
13		
14		
15	NA	NA
16	NA	NA
17	13C2D4 8:2 FTS	
19	M2-8:2 FTS	
20	NT	NT
21	13C2 6:2 FTS	
22	NA	NA

Table 271 Labelled Standards for Sample S3 Bovine Liver GenX

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M3HFPO-DA	NA
3	13C312C3HF11O3	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C4-PFOA	
8	yes	
9	13C3-GenX	
10	NT	
11	NA	NA
12	13C8-PFOA	
13		
14		
15	NA	NA
16	NA	NA
17	M3 HFPO	
19	13C3 HFPO-DA	
20	NT	NT
21	13C3 HFPO-DA	
22	NA	NA



Table 272 Labelled Standards for Sample S3 Bovine Liver ADONA

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	MPFHpA	NA
3	13C4-PFHpA	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C4-PFOA	
8		
9	N/A	
10	NT	
11	NA	NA
12	13C8-PFOA	
13		
14		
15	NA	NA
16	NA	NA
17	13C PFOA	
19	13C4 PFOS	
20	NT	NT
21	13C3 HFPO-DA	
22	NA	NA

Table 273 Labelled Standards for Sample S3 Bovine Liver  
9CI-PF3ONS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M8PFOS	NA
3	13C4-PFOS	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C4-PFOS	13C8-PFOS
8		
9	N/A	
10	NT	
11	NA	NA
12	13C8-PFOS	
13		
14		
15	NA	NA
16	NA	NA
17	M3 PFHxS	
19	13C4 PFOS	
20	NT	NT
21	13C3 HFPO-DA	
22	NA	NA

Table 274 Labelled Standards for Sample S3 Bovine Liver  
11Cl-PF3OUdS

Lab. Code	Before Extraction	Before Instrument Analysis
1		
2	M7PFUnDA	NA
3	13C4-PFOS	N/A
4	NA	NA
5	NA	NA
6	NA	NA
7	13C4-PFOS	13C8-PFOS
8		
9	N/A	
10	NT	
11	NA	NA
12	13C8-PFOS	
13		
14		
15	NA	NA
16	NA	NA
17	M3 PFHxS	
19		
20	NT	NT
21	13C3 HFPO-DA	
22	NA	NA

## APPENDIX 5 ACRONYMS AND ABBREVIATIONS

3:3FTCA	3-Perfluoropropyl propanoic acid
5:3FTCA	2H,2H,3H,3H-Perfluorooctanoic acid
6:2FTS	6:2 Fluorotelomer sulfonate
7:3FTCA	3-Perfluoroheptyl propanoic acid
8:2diPAP	Bis[2-(perfluorooctyl)ethyl] phosphate
8:2FTS	8:2 Fluorotelomer sulfonate
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonate
10:2FTS	10:2 Fluorotelomer sulfonate
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonate
ACE	Acetone
ACN	Acetonitrile
ADONA	4,8-dioxa-3H-perfluorononanoate
AV	Assigned Value
CITAC	Cooperation on International Traceability in Analytical Chemistry
CRM	Certified Reference Material
CV	Coefficient of Variation
dSPE	Dispersive SPE
EtFOSA	N-Ethyl perfluorooctane sulfonamide
EtFOSAA	N-Ethyl perfluorooctane sulfonamido acetic acid
EtFOSE	N-Ethyl perfluorooctane sulfonamido ethanol
FSANZ	Food Standards Australia New Zealand
GAG	General Accreditation Guidance (NATA)
GenX	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)-propanoic acid
GUM	Guide to the Expression of Uncertainty in Measurement
HV	Homogeneity Value
IEC	International Electrotechnical Commission
ISO	International Organization for Standardization
LC	Liquid Chromatography
LLE	Liquid-Liquid Extraction
LOR	Limit of Reporting
Max	Maximum
Md	Median
MeFOSA	N-Methyl perfluorooctane sulfonamide
MeFOSAA	N-Methyl perfluorooctane sulfonamido acetic acid

MeFOSE	N-Methyl perfluorooctane sulfonamido ethanol
MeOH	Methanol
Min	Minimum
MS	Mass Spectrometry
MS/MS	Tandem Mass Spectrometry
MU	Measurement Uncertainty
N	Number of numeric results
NA	Not Applicable
NATA	National Association of Testing Authorities, Australia
NMI	National Measurement Institute, Australia
NR	Not Reported
NS	Not Supplied
NT	Not Tested
PCV	Performance Coefficient of Variation
PFAS	Per- and Polyfluoroalkyl Substances
PFBA	Perfluorobutanoic acid
PFBS	Perfluorobutane sulfonate
PFDA	Perfluorodecanoic acid
PFDoA	Perfluorododecanoic acid
PFDoS	Perfluorododecane sulfonate
PFDS	Perfluorodecane sulfonate
PFHpA	Perfluoroheptanoic acid
PFHpS	Perfluoroheptane sulfonate
PFHxA	Perfluorohexanoic acid
PFHxS	Perfluorohexane sulfonate
PFNA	Perfluorononanoic acid
PFNS	Perfluorononane sulfonate
PFOA	Perfluorooctanoic acid
PFODA	Perfluorooctadecanoic acid
PFOS	Perfluorooctane sulfonate
PFOSA	Perfluorooctane sulfonamide
PFPeA	Perfluoropentanoic acid
PFPeS	Perfluoropentane sulfonate
PFTeDA	Perfluorotetradecanoic acid
PFTTrDA	Perfluorotridecanoic acid

PFTTrDS	Perfluorotridecane sulfonate
PFUdA	Perfluoroundecanoic acid
PFUdS	Perfluoroundecane sulfonate
PT	Proficiency Testing
QQQ	Triple Quadrupole Mass Spectrometry
QuEChERS	Quick, Easy, Cheap, Effective, Rugged and Safe extraction method
RA	Robust Average
Rec	Recovery
RM	Reference Material
$s_{an}$	Analytical standard deviation
SD	Standard Deviation
SI	International System of Units
SLE	Solid-Liquid Extraction
SPE	Solid-Phase Extraction
SS	Spiked Samples
$s_{sam}$	Between-sample standard deviation
SV	Spiked Value (or formulated concentration of a PT sample)
UPLC	Ultra Performance Liquid Chromatography
USEPA	United States Environmental Protection Agency
WAX	Weak Anion Exchange

**END OF REPORT**