



Australian Government  
Department of Industry,  
Science and Resources

National  
Measurement  
Institute

# Proficiency Test Final Report AQA 22-14 PFAS in Biota and Food

January 2023



## ACKNOWLEDGMENTS

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

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

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

Jenny Xu

Mark Lewin

Luminita Antin

Geoff Morschel

Hamish Lenton

Isaac Schipp

Gavin Stevenson

Jesuina De Araujo

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

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## SUMMARY

AQA 22-14 PFAS in Biota and Food commenced in August 2022. Twenty laboratories registered to participate, with one laboratory requesting two sets of samples to be analysed independently. Twenty participants submitted results by the due date.

The sample set consisted of one spiked prawn sample (Sample S1) and one spiked carrot sample (Sample S2). 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, PFTrDA, PFOSA, MeFOSA, EtFOSA, 6:2 FTS, GenX, ADONA, 9Cl-PF3ONS and 11Cl-PF3OUdS.

Of 811 possible results, 597 (74%) were numeric results. One hundred and eight results were a 'less than' value ( $< x$ ) or Not Reported (NR), and 106 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. The results from Laboratories **3, 8, 10, 16** and **20** in Sample S1, and from Laboratory **16** in Sample S2, were consistently lower than the spiked value by around the same factor for all analytes, an indication of laboratory bias. To avoid unfair scoring, these results were excluded from robust average calculations as it will bias low the assigned value; they were also excluded from the calculation of all summary statistics.

**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, 16 participants were sent both matrices, three participants were sent prawn only, and one participant was sent carrot only. Laboratories **7, 12, 13** and **17** reported numeric results for all 39 scored analytes across both matrices.

Ten participants did not report results for analytes that they tested for and were spiked into the samples (total of 53 results), while seven participants reported analytes that were not spiked into the samples (total of 11 results).

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

Of 528  $z$ -scores, 453 (86%) returned  $|z| \leq 2.0$ , indicating a satisfactory performance.

Of 528  $E_n$ -scores, 374 (71%) returned  $|E_n| \leq 1.0$ , indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Laboratories **7** and **12** returned satisfactory  $z$ -scores for all scored analytes (39). No participant returned satisfactory  $E_n$ -scores for all analytes of interest in this study.

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

Participants used a variety of methods for extraction and analysis. The most popular for this study was homogenisation as pretreatment, followed by alkaline digestion with basified methanol and solid-phase extraction clean-up, and then analysis using LC-MS/MS or LC-QQQ.

In this study, extraction with basified methanol, and long extraction times ( $> 8$  h) were more effective, giving results with higher recoveries with respect to the spiked values.

Participants should take care to avoid any potential dilution, standard preparation, or similar errors in their analyses.

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

Of 597 numeric results for spiked analytes, 580 (97%) were reported with an associated expanded measurement uncertainty.

Although it is a requirement of ISO/IEC 17025 that laboratories have procedures to estimate the uncertainty since 2017, 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 1.8% to 100% of the reported value. Additionally, some laboratories are still reporting numeric estimates of uncertainties for non-numeric results.

- *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 at relevant mass fractions.

Proportions of satisfactory  $z$ -scores and  $E_n$ -scores have also remained relatively consistent over this period, though for this study there was a slight decrease as compared to the previous year's study.

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

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

## **1 INTRODUCTION**

### **1.1 NMI Proficiency Testing Program**

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

Proficiency testing (PT) is the 'evaluation of participant performance against pre-established criteria by means of interlaboratory comparison'.<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 and water, fruit, vegetables and herbs;
- petroleum hydrocarbons in soil and water;
- per- and polyfluoroalkyl substances (PFAS) in soil, water, biota and food;
- inorganic analytes in soil, water, filters, food and pharmaceuticals;
- controlled drug assay, drugs in wipes and clandestine laboratory; and
- allergens in food.

### **1.2 Study 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 17043:2010,<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 | 8/08/2022  |
| Samples sent     | 30/08/2022 |
| Results due      | 26/10/2022 |
| Interim report   | 1/11/2022  |

The results due date was extended to accommodate sample delivery delays to some international participants.

### 2.2 Participation and Laboratory Code

Twenty laboratories registered to participant, with one laboratory requesting two sets of samples to be analysed independently. All participants were assigned a confidential laboratory code number for this study. Twenty participants submitted results by the 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  | PFTrDS | PFDoA   | N-EtFOSE     |
| PFPeS | PFBA   | PFTrDA  | 4:2 FTS      |
| PFHxS | PFPeA  | PFTeDA  | 6:2 FTS      |
| PFHpS | PFHxA  | PFOSA   | 8:2 FTS      |
| PFOS  | PFHpA  | MeFOSA  | 10:2 FTS     |
| PFNS  | PFOA   | EtFOSA  | GenX         |
| PFDS  | PFNA   | MeFOSAA | ADONA        |
| PFUdS | PFDA   | EtFOSAA | 9Cl-PF3ONS   |
| PFDoS | PFUdA  | MeFOSE  | 11Cl-PF3OUdS |

The two samples were prepared in August 2022. Care was taken to avoid any PFAS contamination during sample preparation. The prepared samples were:

- Sample S1: Prawn (5 g portions) spiked with 21 different PFAS analytes.
- Sample S2: Carrot (30 g portions) spiked with 20 different PFAS analytes.

Details of spiked analytes and values are presented in Table 2.

Table 2 Spiked Values of Test Samples

| Analyte | Sample S1 Prawn ( $\mu\text{g}/\text{kg}$ ) | Sample S2 Carrot ( $\mu\text{g}/\text{kg}$ ) |
|---------|---|--|
| PFBS    | 0.399                                       | 0.891  |
| PFPeS   | 4.65  | 7.47   |
| PFHxS*  | 1.89  | 6.61   |
| PFHpS   | 2.00  | 3.00   |
| PFOS*   | 4.77  | 2.12   |

| Analyte      | Sample S1 Prawn ( $\mu\text{g}/\text{kg}$ ) | Sample S2 Carrot ( $\mu\text{g}/\text{kg}$ ) |
|--------------|---|--|
| PFNS         | 11.5  | 1.72   |
| PFDS         | Not Spiked                                  | 6.80   |
| PFBA         | 2.96  | 1.19   |
| PFPeA        | 1.13  | 2.20   |
| PFHxA        | 5.31  | 7.45   |
| PFHpA        | 7.54  | 1.50   |
| PFOA         | 7.92  | 1.20   |
| PFNA         | 0.503                                       | 2.31   |
| PFDA         | 0.902                                       | 9.47   |
| PFUdA        | 1.21  | Not Spiked                                   |
| PFTTrDA      | 8.17  | Not Spiked                                   |
| PFOSA        | 4.46  | 4.95   |
| MeFOSA       | 4.99  | 4.99   |
| EtFOSA       | 3.99  | 3.99   |
| 6:2 FTS      | Not Spiked                                  | 1.89   |
| GenX         | Not Spiked                                  | 11.1   |
| ADONA        | 5.64  | 14.0   |
| 9Cl-PF3ONS   | 14.4  | Not Spiked                                   |
| 11Cl-PF3OUdS | 4.70  | Not Spiked                                   |

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

Further sample preparation details can be found in Appendix 1.

## 2.4 Homogeneity and Stability of Test Materials

The process used to prepare, store and dispatch the test samples has been demonstrated to produce sufficiently homogeneous and stable samples for previous NMI PFAS in biota and food PT studies, for timeframes similar to that of this study.

To consider possible instability of the samples, the results returned by participants were compared to the spiked values. Robust averages for scored analytes were within 69% to 93% and 76% to 111% of the spiked values for Samples S1 and S2 respectively, which were similar to values observed in previous NMI PFAS in biota and food PT studies and provides support for the stability of these analytes.

Additionally, homogeneity and stability testing was conducted on Sample S2 carrot. The samples were demonstrated to be sufficiently homogeneous and stable for the evaluation of participants' performance in this study.

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

## 2.5 Test Material Storage and Dispatch

After preparation, the test material were dispensed into sample tubes, labelled and shrink-wrapped. Prior to sample dispatch, all samples were stored frozen at  $-80\text{ }^{\circ}\text{C}$ .

Samples were packed into insulated polystyrene foam boxes with cooler bricks and sent by courier on 30 August 2022.

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 and report results in units of  $\mu\text{g}/\text{kg}$  on an as received basis.
  - For PFAS analytes that contain linear and branched isomers, report total (the sum of linear and branched isomers).
  - For PFOS and PFHxS you are asked to report total (the sum of linear and branched isomers) and linear (the linear isomers only).
- Report results using the electronic results sheet emailed to you.
- For each analyte report a single result expressed as if reporting to a client (i.e. corrected for recovery or not, according to your standard procedure, but state if results are corrected on the result sheet). This figure will be used in all statistical analysis in the study report.
- For each analyte report the associated expanded measurement uncertainty as  $\mu\text{g}/\text{kg}$  (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 7 October 2022.

Due to sample delivery delays to some international participants caused by customs clearance issues, the results due date was extended to 26 October 2022 for all participants.

## **2.7 Interim Report**

An interim report was emailed to all participants on 1 November 2022.

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

Table 3 Basis of Participants' Uncertainty Estimate

| Lab. Code | Approach to Estimating MU  | Information Sources for MU Estimation*                                |   | Guide Document for Estimating MU                                     |
|-----------|--|---|---|--|
|           |  | Precision   | Method Bias   |  |
| 1         | Standard deviation of replicate analyses multiplied by 2 or 3        | Control samples - SS<br>Duplicate analysis                            | CRM<br>Instrument calibration<br>Recoveries of SS<br>Standard purity                                    | NATA GAG<br>Estimating and Reporting MU                              |
| 2         | standard deviation of triplicate measurements                        | Standard deviation from PT studies only                               |   |  |
| 3         | Professional judgment  | Standard deviation from PT studies only                               |   |  |
| 4         | Top Down - precision and estimates of the method and laboratory bias | Control samples - CRM<br>Duplicate analysis<br>Instrument calibration | CRM<br>Laboratory bias from PT studies<br>Recoveries of SS  | NMI Uncertainty Course   |
| 5         | Standard deviation of replicate analyses multiplied by 2 or 3        | Control samples - SS  | Recoveries of SS  | ISO/GUM  |
| 6         | Bottom Up (ISO/GUM, fish bone/cause and effect diagram)              | Duplicate analysis  | CRM   | ISO/GUM  |
| 7         | Top Down - precision and estimates of the method and laboratory bias | Control samples<br>Duplicate analysis<br>Instrument calibration       | CRM<br>Instrument calibration<br>Laboratory bias from PT studies<br>Recoveries of SS<br>Standard purity | NATA GAG<br>Estimating and Reporting MU (replaced Technical Note 33) |
| 8         | Top Down - precision and estimates of the method and laboratory bias | Control samples - SS<br>Duplicate analysis<br>Instrument calibration  | CRM<br>Instrument calibration<br>Recoveries of SS   | ISO/GUM  |
| 9         | Top Down - precision and estimates of the method and laboratory bias | Control samples - SS<br>Duplicate analysis<br>Instrument calibration  | Instrument calibration<br>Recoveries of SS<br>Standard purity   | Eurachem/CITAC Guide   |
| 10        | Standard deviation of replicate analyses multiplied by 2 or 3        | Control samples - SS  | CRM   | NMI Uncertainty Course   |
| 11        | Top Down - precision and estimates of the method and laboratory bias | Control samples - RM<br>Duplicate analysis                            | CRM<br>Laboratory bias from PT studies<br>Recoveries of SS  | Nordtest Report TR537  |

| Lab. Code | Approach to Estimating MU  | Information Sources for MU Estimation*                               |  | Guide Document for Estimating MU   |
|-----------|--|--|--|--|
|           |  | Precision  | Method Bias  |  |
| 12        | Standard deviation of replicate analyses multiplied by 2 or 3        | Control samples  |  | ISO/GUM  |
| 13        | Standard deviation of replicate analyses multiplied by 2 or 3        | Standard deviation from PT studies only                              |  | Eurachem/CITAC Guide   |
|           |  | Control samples - SS<br>Duplicate analysis<br>Instrument calibration | Instrument calibration<br>Recoveries of SS   |  |
| 15        | 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.                         |
| 16        | Top Down - precision and estimates of the method and laboratory bias | Control samples - SS<br>Duplicate analysis<br>Instrument calibration | Instrument calibration<br>Laboratory bias from PT studies<br>Recoveries of SS        | AQS Baden-Württemberg  |
| 17        | Top Down - precision and estimates of the method and laboratory bias | Control samples - SS<br>Duplicate analysis<br>Instrument calibration | Laboratory bias from PT studies<br>Recoveries of SS                                  | ISO/GUM  |
| 18        | Top Down - precision and estimates of the method and laboratory bias | Control samples - RM<br>Duplicate analysis                           |  | Eurachem/CITAC Guide   |
| 19        | Standard deviation of replicate analyses multiplied by 2 or 3        | Control samples - SS   |  | Statistics and Chemometrics for Analytical Chemistry, Miller and Miller, 5th Edition |
| 20        | Professional judgment  | Duplicate analysis<br>Instrument calibration                         | CRM<br>Instrument calibration<br>Laboratory bias from PT studies<br>Recoveries of SS | Internal document  |
| 21        | Bottom Up (ISO/GUM, fish bone/cause and effect diagram)              | Duplicate analysis   | CRM  |  |

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

Table 4 Uncertainty Estimate Additional Comments

| Lab. Code | Uncertainty Estimate Comments  |
|-----------|--|
| 1         | Recovery and uncertainty data given for analytes at method limit of reporting.   |
| 12        | The expanded measurement uncertainty values were calculated using a coverage factor (K) value of 2.00 and at the 95% confidence limit.   |
| 13        | Uncertainty calculated as 3xSD of replicate analysis.  |
| 19        | 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 |

### 3.3 Participants' Comments

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

Table 5 Participants' Comments

| Lab. Code | Sample | Participant's Comments   | Study Coordinator's Response   |
|-----------|--------|--|--|
| 1         | S1     | Extra Compounds Detected < LOR : PFBA, PFPeA, PFOA, PFUdA, N-EtFOSA, N-MeFOSA  |  |
|           | S2     | Extra Compounds Detected < LOR : PFBA<br>Compounds not in the method scope that were detected: PFDS ca. 6 ug/kg  |  |
|           | All    | Methodology: In this method the linear standards are used to quantify both the linear as well as the branched isomers.   |  |
| 2         | S1     | shrimp had gone through significant decomposition due to being in thawed state for prolonged period of time  | Stability testing conducted in previous PT studies for PFAS in prawn showed that analytes were sufficiently stable for at least two months at room temperature. <sup>5</sup>   |
|           | All    | We use a technical mixture for PFOS as an analytical standard. It appears there is only linear PFOS in this sample, this may result in some bias compared to using just a linear isomer standard   |  |
| 3         | S1     | Linear isomers reported only.  |  |
| 12        | All    | The sample was received at a temperature of 20.4°C; which was above the laboratory method recommended sample storage temperature (less than or equal to 6°C).<br>Methodology: Isotopically labelled surrogate standards were spiked into the sample prior to extraction      | Please see response to Laboratory 2. Additionally, in this study, stability testing was conducted for Sample S2 carrot, and analytes were found to be sufficiently stable (see Appendix 2).  |
| 13        | All    | Please send more of the biota sample next time if possible. 5g does not leave much room for re-doing the experiment if you use 1 g for each replicate  | The amount per sample has been selected to balance the preparation method requirements while allowing participants to perform some replicates, with most participants using around 1 g per analysis.<br>Participants can also order additional samples if required for their analyses. |
| 15        | All    | All linear and branched present have been reported although some branched peaks are not confirmed by traceable standards.  |  |
| 20        | All    | A recovery standard was used to calculate the recovery of the internal standard.<br>The samples were at customs for 4 weeks. There was no information about the storage conditions of the sample. The samples smelled slightly spoiled and also showed visual conspicuities. | Please see responses to Laboratories 2 and 12.   |

## 4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

### 4.1 Results Summary

Participant results are presented in Tables 6 to 50 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 46. An example chart with interpretation guide is shown in Figure 1.

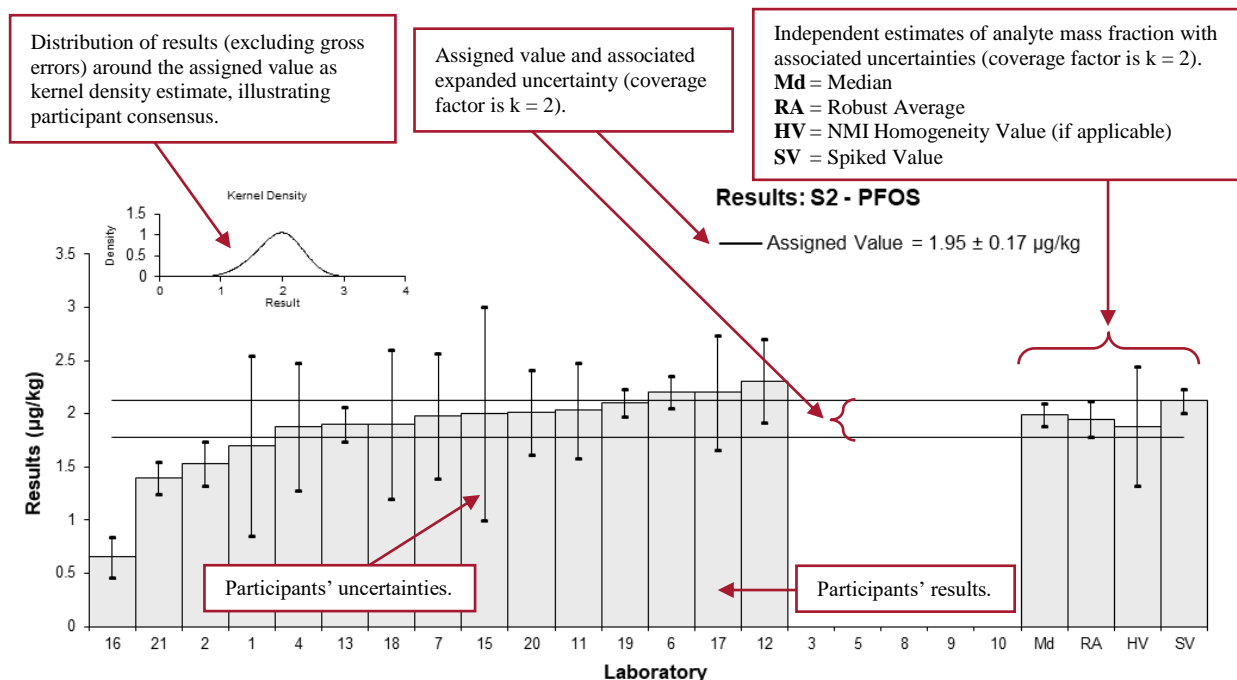


Figure 1 Guide to Presentation of Results

### 4.2 Outliers, Gross Errors and Results Excluded from Robust Average Calculations

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.<sup>3,4</sup> Gross errors were extreme outliers or obvious blunders e.g. results reported with incorrect units or basis, or for a different analyte or sample, and such results were removed for the calculation of all summary statistics.<sup>3,4</sup>

The results from Laboratories **3, 8, 10, 16** and **20** in Sample S1, and from Laboratory **16** in Sample S2, were consistently lower than the spiked value by around the same factor for all analytes. This is an indication of laboratory or method bias. To avoid unfair scoring, these results were excluded from robust average calculations as it will bias low the assigned value; they were also excluded from the calculation of all summary statistics.

### 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 in this study 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 associated expanded MUs, and robust CVs (a measure of the variability of participants' results) were calculated using the procedure described in ISO 13528:2022.<sup>6</sup>

#### 4.5 Performance Coefficient of Variation (PCV)

The performance coefficient of variation (PCV) is a fixed measure of the between-laboratory variation that in the judgement of the study coordinator would be expected from participants given the levels of analytes present. The PCV is not the CV of participants' results; it is set by the study coordinator and is based on the mass fraction of the analytes and experience from previous studies, and is supported by mathematical models such as the Thompson-Horwitz equation.<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 from Equation 1

For the absolute value of a z-score:

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

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

#### 4.8 $E_n$ -Score

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

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

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

#### **4.9 Traceability and Measurement Uncertainty**

Laboratories accredited to ISO/IEC 17025 must establish and demonstrate the traceability and measurement uncertainty associated with their test results.<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 6

### Sample Details

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | PFBS  |
| <b>Unit</b>       | µg/kg |

### Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | < 2    | 1           | 106  |       |                |
| 2         | 0.244  | 0.011       | 94   | -0.96 | -1.18          |
| 3**       | 0.216  | NR          | NR   | -1.42 | -1.79          |
| 4         | <0.5   | NR          | 76   |       |                |
| 5         | NS     | NS          | NS   |       |                |
| 6         | <1     | NR          | NT   |       |                |
| 7         | 0.311  | 0.093       | 88   | 0.15  | 0.09           |
| 8         | <0.5   | NR          | 99   |       |                |
| 9         | <0.5   | NR          | 98   |       |                |
| 10        | <1     | NR          | NR   |       |                |
| 11        | 0.315  | 0.08        | 94.0 | 0.22  | 0.14           |
| 12        | 0.316  | 0.0442      | 90.3 | 0.23  | 0.21           |
| 13        | 0.265  | 0.005       | 105  | -0.61 | -0.77          |
| 15        | <1     | NR          | 82   |       |                |
| 16        | < 0.1  | NR          | 138  |       |                |
| 17        | 0.36   | 0.18        | 89   | 0.96  | 0.31           |
| 18        | <1     | NR          | 135  |       |                |
| 19        | < 1.0  | NR          | 104  |       |                |
| 20**      | 0.220  | 0.044       | 73   | -1.36 | -1.26          |
| 21        | <1     | NR          | NT   |       |                |

\*\* Not included in robust average calculations, see Section 4.2

### Statistics

|                       |       |       |
|-----------------------|-------|-------|
| <b>Assigned Value</b> | 0.302 | 0.048 |
| <b>Spike Value</b>    | 0.399 | 0.020 |
| <b>Robust Average</b> | 0.302 | 0.048 |
| <b>Median</b>         | 0.313 | 0.038 |
| <b>Mean</b>           | 0.302 |       |
| <b>N</b>              | 6     |       |
| <b>Max</b>            | 0.36  |       |
| <b>Min</b>            | 0.244 |       |
| <b>Robust SD</b>      | 0.047 |       |
| <b>Robust CV</b>      | 16%   |       |

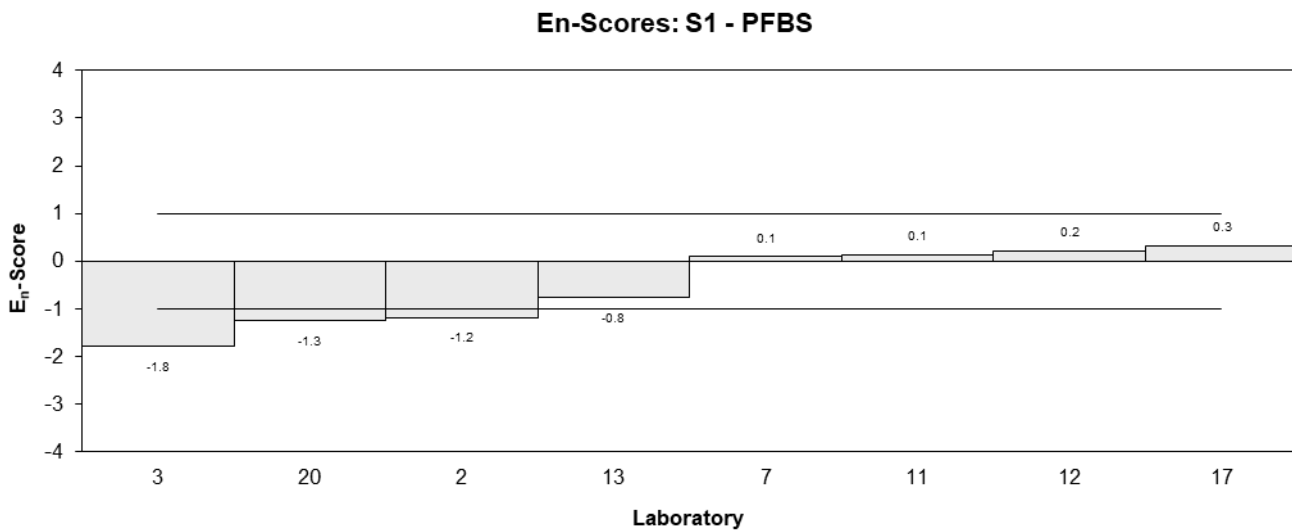
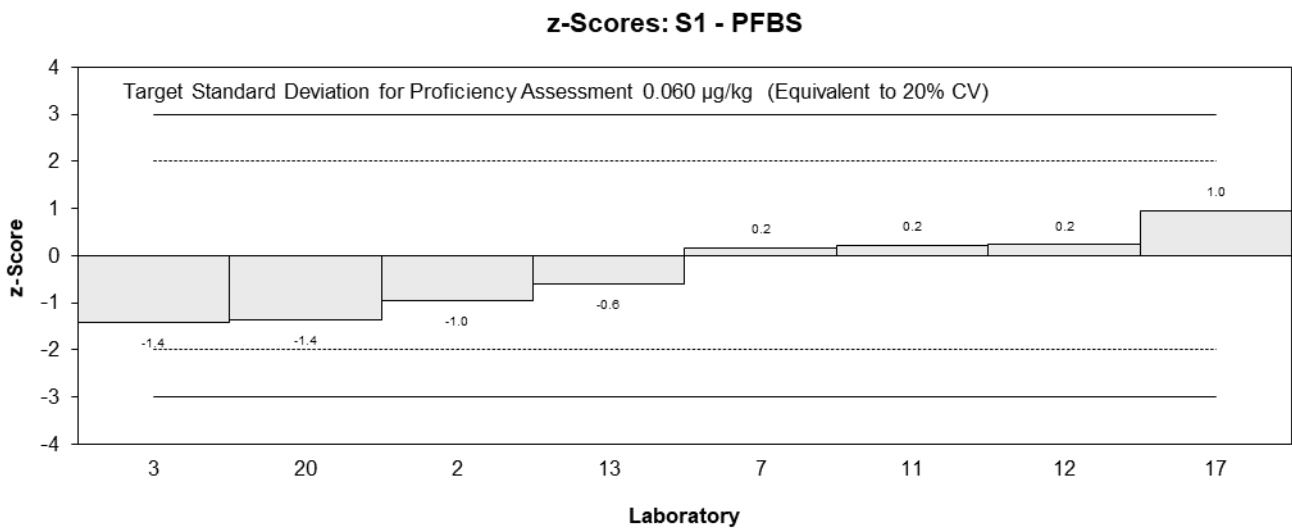
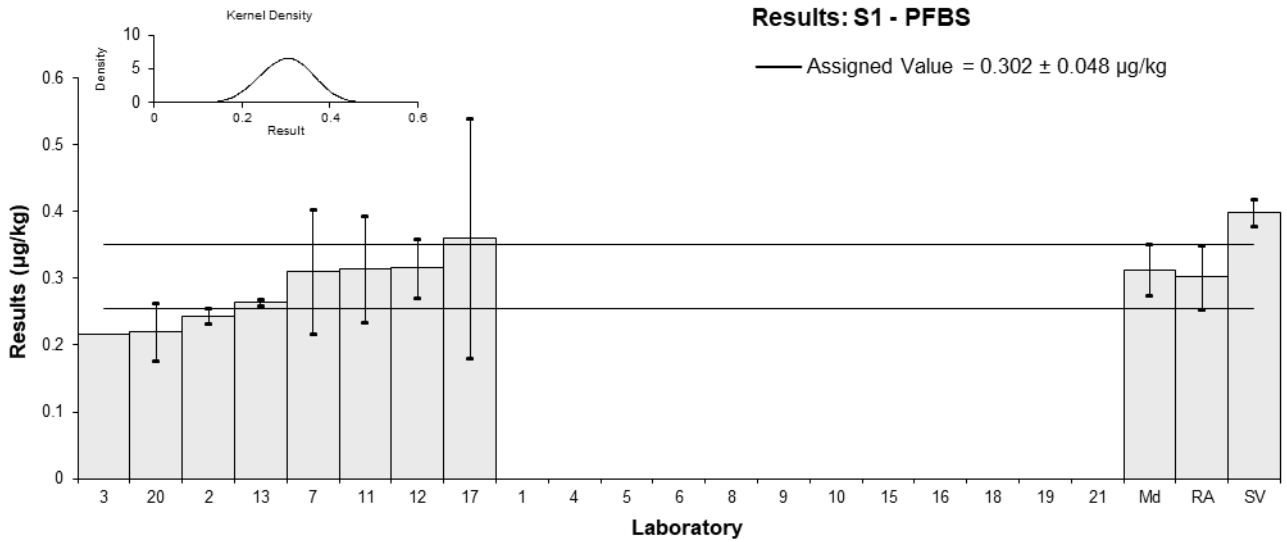


Figure 2

Table 7

## Sample Details

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | PFPeS |
| <b>Unit</b>       | µg/kg |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 3      | 1.5         | NR   | -1.42 | -0.73          |
| 2         | 3.29   | 0.232       | 97   | -1.07 | -1.36          |
| 3**       | 2.662  | NR          | NR   | -1.82 | -2.46          |
| 4         | 4.68   | 1           | NR   | 0.58  | 0.42           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | 4.1    | 0.25        | NT   | -0.11 | -0.13          |
| 7         | 4.96   | 1.5         | 79   | 0.92  | 0.47           |
| 8**       | 2.57   | 0.64        | NR   | -1.93 | -1.82          |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NR     | NR          | NR   |       |                |
| 11        | 4.51   | 1.2822      | 94.0 | 0.38  | 0.22           |
| 12        | 5.15   | 0.566       | 88.9 | 1.15  | 1.14           |
| 13        | 3.55   | 0.108       | 106  | -0.76 | -1.02          |
| 15        | 5      | 2           | 89   | 0.97  | 0.39           |
| 16**      | 1.97   | 0.591       | 138  | -2.65 | -2.59          |
| 17        | 4.9    | 1.2         | 89   | 0.85  | 0.53           |
| 18        | 2.46   | 0.83        | 135  | -2.06 | -1.67          |
| 19        | 4.1    | 0.47        | 113  | -0.11 | -0.12          |
| 20**      | 2.33   | 0.466       | 73   | -2.22 | -2.40          |
| 21        | 4.4    | 0.25        | NT   | 0.25  | 0.31           |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |      |      |
|-----------------------|------|------|
| <b>Assigned Value</b> | 4.19 | 0.62 |
| <b>Spike Value</b>    | 4.65 | 0.23 |
| <b>Robust Average</b> | 4.19 | 0.62 |
| <b>Median</b>         | 4.40 | 0.58 |
| <b>Mean</b>           | 4.16 |      |
| <b>N</b>              | 13   |      |
| <b>Max</b>            | 5.15 |      |
| <b>Min</b>            | 2.46 |      |
| <b>Robust SD</b>      | 0.90 |      |
| <b>Robust CV</b>      | 21%  |      |

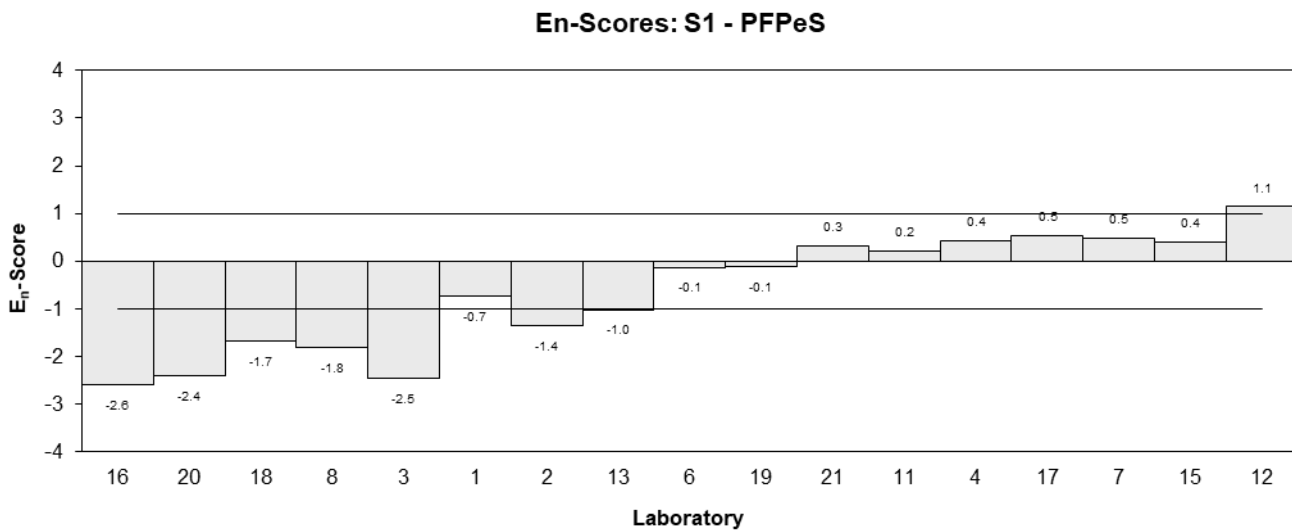
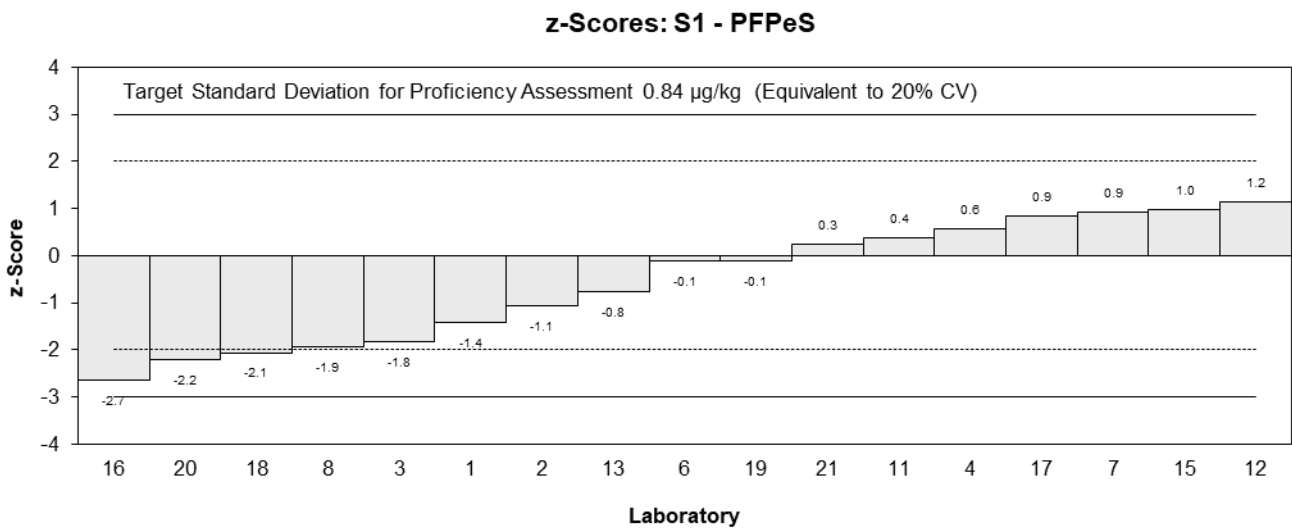
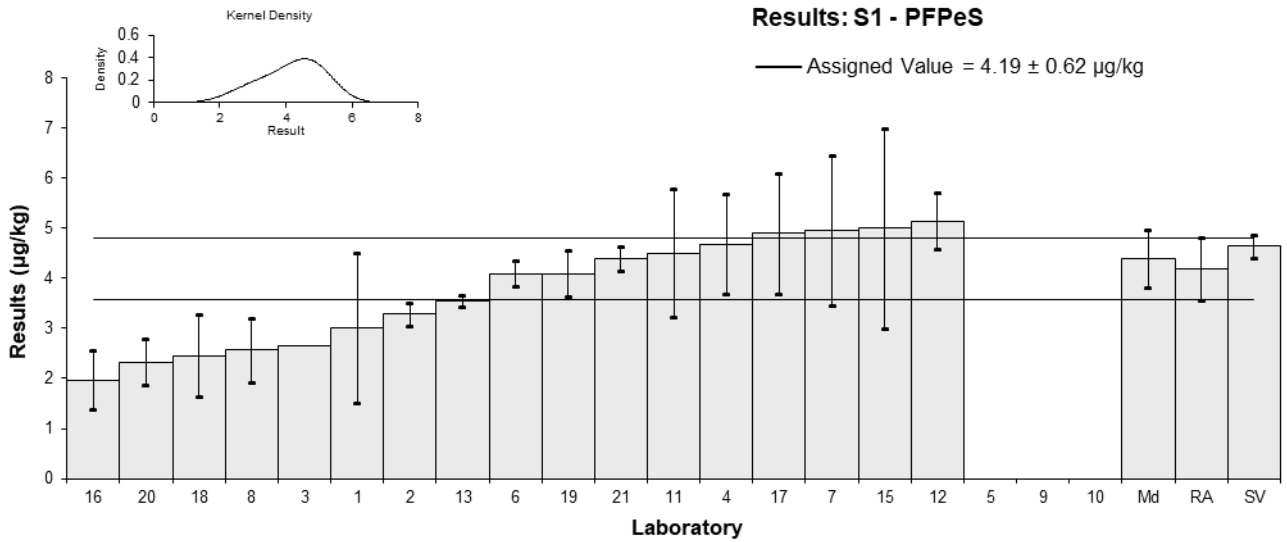


Figure 3

Table 8

## Sample Details

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | PFHxS |
| <b>Unit</b>       | µg/kg |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | < 2    | 1           | 107  |       |                |
| 2         | NR     | NR          | NR   |       |                |
| 3         | NT     | NT          | NT   |       |                |
| 4         | 2.06   | 0.6         | 74   | 1.28  | 0.64           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | 1.7    | 0.2         | NT   | 0.18  | 0.18           |
| 7         | 1.40   | 0.42        | 83   | -0.73 | -0.48          |
| 8**       | 0.93   | 0.23        | 114  | -2.16 | -2.00          |
| 9         | 1.4    | 0.7         | 97   | -0.73 | -0.32          |
| 10        | <1     | NR          | NR   |       |                |
| 11        | NT     | NT          | NT   |       |                |
| 12        | 1.61   | 0.226       | 88.9 | -0.09 | -0.09          |
| 13        | 1.59   | 0.068       | 106  | -0.15 | -0.18          |
| 15        | 2      | 1           | 89   | 1.10  | 0.35           |
| 16**      | 0.418  | 0.1254      | 147  | -3.73 | -4.10          |
| 17        | 1.2    | 0.31        | 89   | -1.34 | -1.07          |
| 18        | <1     | NR          | 135  |       |                |
| 19        | 1.4    | 0.15        | 107  | -0.73 | -0.78          |
| 20**      | 0.950  | 0.189       | 80   | -2.10 | -2.09          |
| 21        | 2.0    | 0.1         | NT   | 1.10  | 1.25           |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |      |      |
|-----------------------|------|------|
| <b>Assigned Value</b> | 1.64 | 0.27 |
| <b>Spike Value</b>    | 1.89 | 0.09 |
| <b>Robust Average</b> | 1.64 | 0.27 |
| <b>Median</b>         | 1.60 | 0.23 |
| <b>Mean</b>           | 1.64 |      |
| <b>N</b>              | 10   |      |
| <b>Max</b>            | 2.06 |      |
| <b>Min</b>            | 1.2  |      |
| <b>Robust SD</b>      | 0.34 |      |
| <b>Robust CV</b>      | 21%  |      |

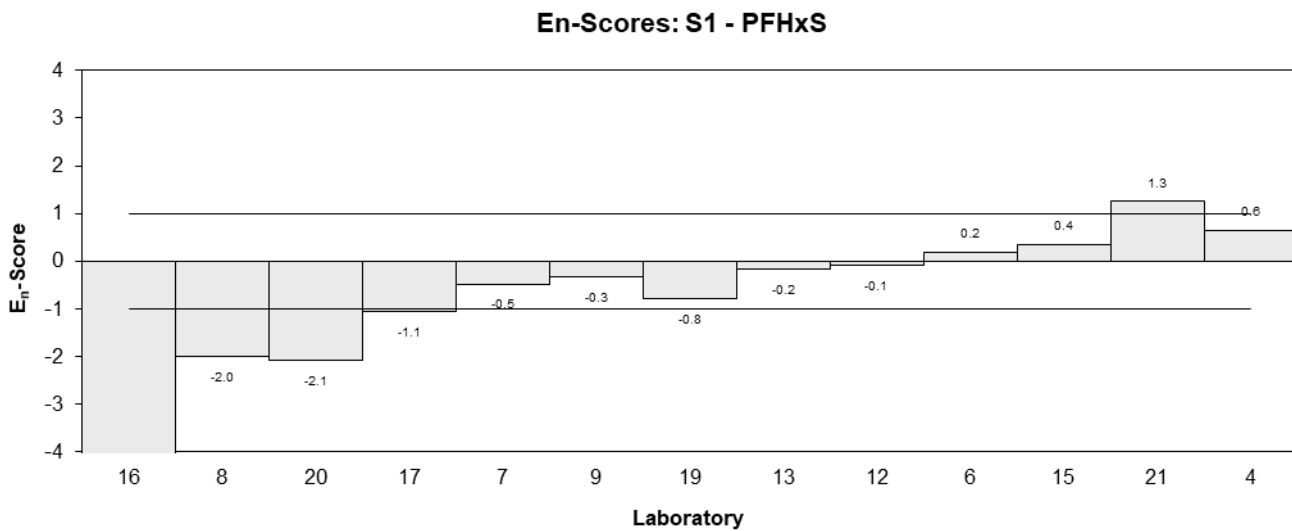
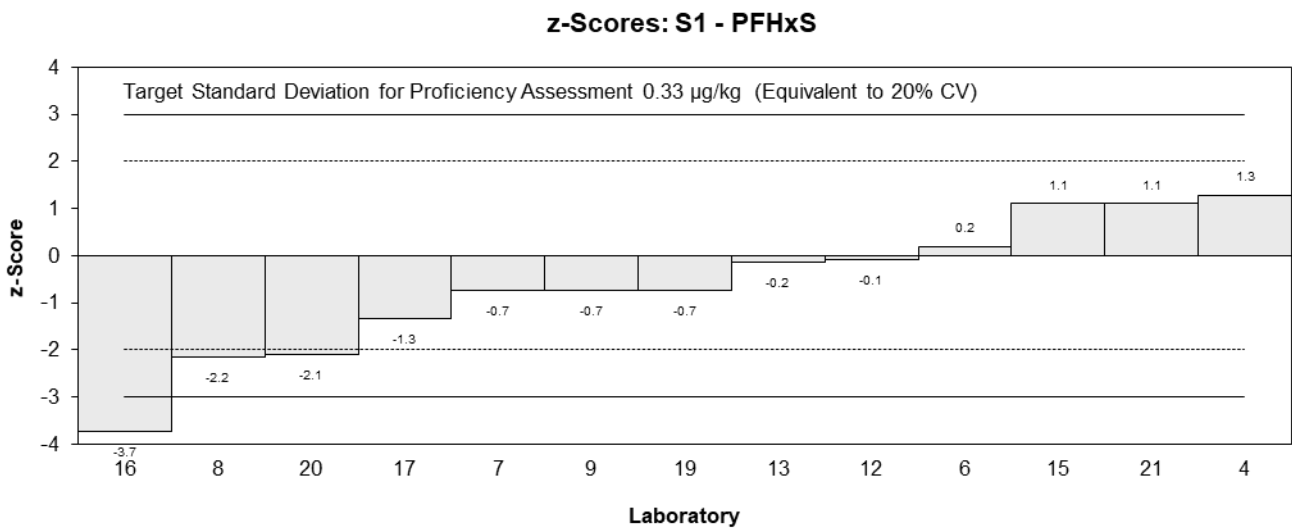
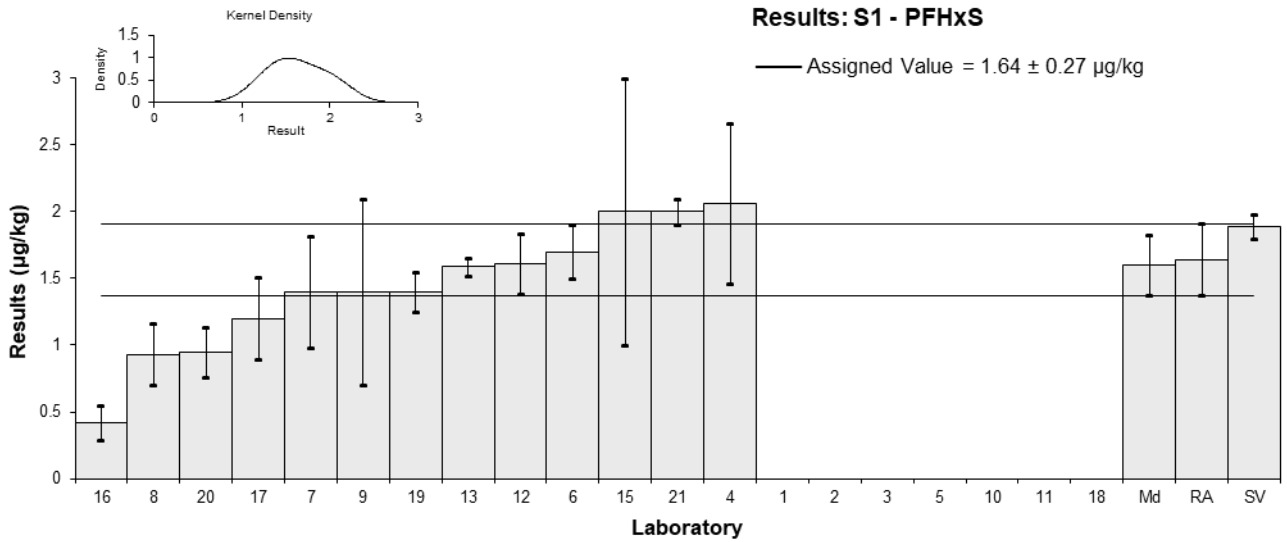


Figure 4

Table 9

## Sample Details

|                   |                |
|-------------------|----------------|
| <b>Sample No.</b> | S1             |
| <b>Matrix</b>     | Prawn          |
| <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   |       |                |
| 2         | 1.28   | 0.066       | 97   | -1.02 | -1.23          |
| 3**       | 0.928  | NR          | NR   | -2.12 | -2.62          |
| 4         | 2.06   | 0.6         | NR   | 1.40  | 0.69           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | 1.7    | 0.2         | NT   | 0.28  | 0.27           |
| 7         | 1.40   | 0.42        | 83   | -0.65 | -0.43          |
| 8**       | 0.93   | 0.23        | 114  | -2.11 | -1.96          |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NR     | NR          | NR   |       |                |
| 11        | 1.61   | 0.521       | 94.0 | 0.00  | 0.00           |
| 12        | 1.6    | 0.226       | 88.9 | -0.03 | -0.03          |
| 13        | 1.45   | 0.067       | 106  | -0.50 | -0.60          |
| 15        | 2      | 1           | 89   | 1.21  | 0.38           |
| 16**      | 0.418  | 0.1254      | 147  | -3.70 | -4.13          |
| 17        | 1.2    | 0.31        | 89   | -1.27 | -1.01          |
| 18        | NT     | NT          | NT   |       |                |
| 19        | 1.4    | 0.15        | 107  | -0.65 | -0.70          |
| 20**      | 0.945  | 0.186       | 80   | -2.07 | -2.08          |
| 21        | 2.0    | 0.1         | NT   | 1.21  | 1.40           |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |      |      |
|-----------------------|------|------|
| <b>Assigned Value</b> | 1.61 | 0.26 |
| <b>Spike Value</b>    | 1.89 | 0.09 |
| <b>Robust Average</b> | 1.61 | 0.26 |
| <b>Median</b>         | 1.60 | 0.22 |
| <b>Mean</b>           | 1.61 |      |
| <b>N</b>              | 11   |      |
| <b>Max</b>            | 2.06 |      |
| <b>Min</b>            | 1.2  |      |
| <b>Robust SD</b>      | 0.34 |      |
| <b>Robust CV</b>      | 21%  |      |



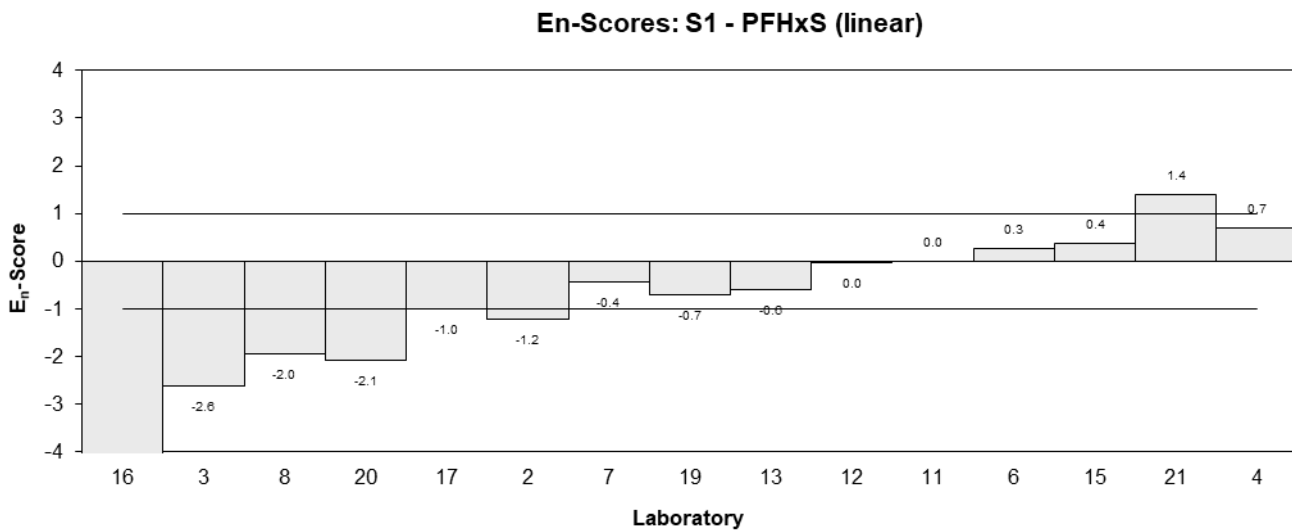
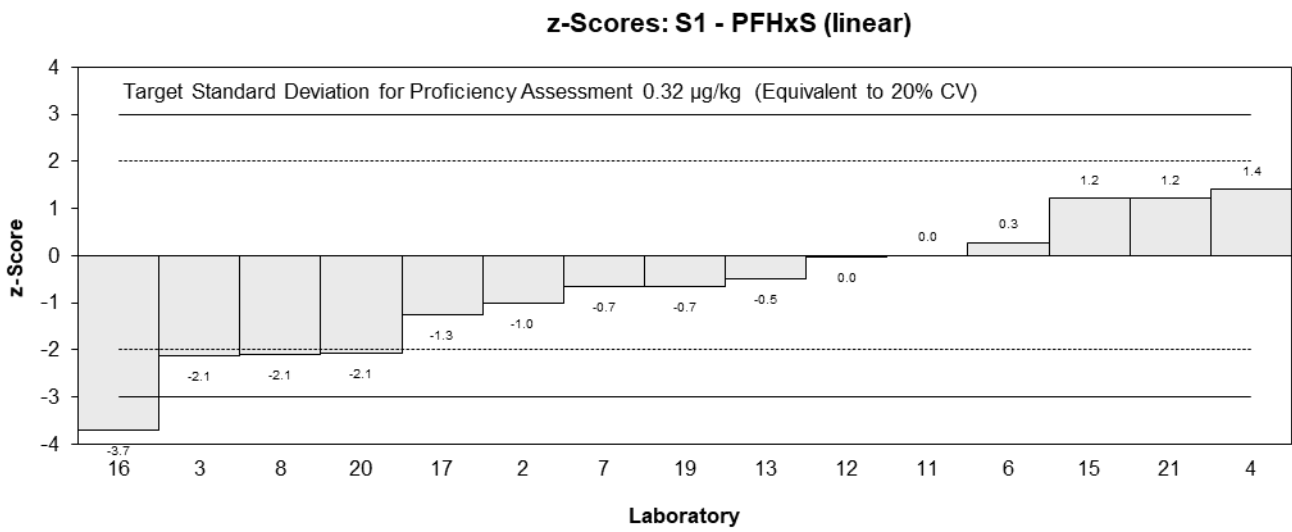
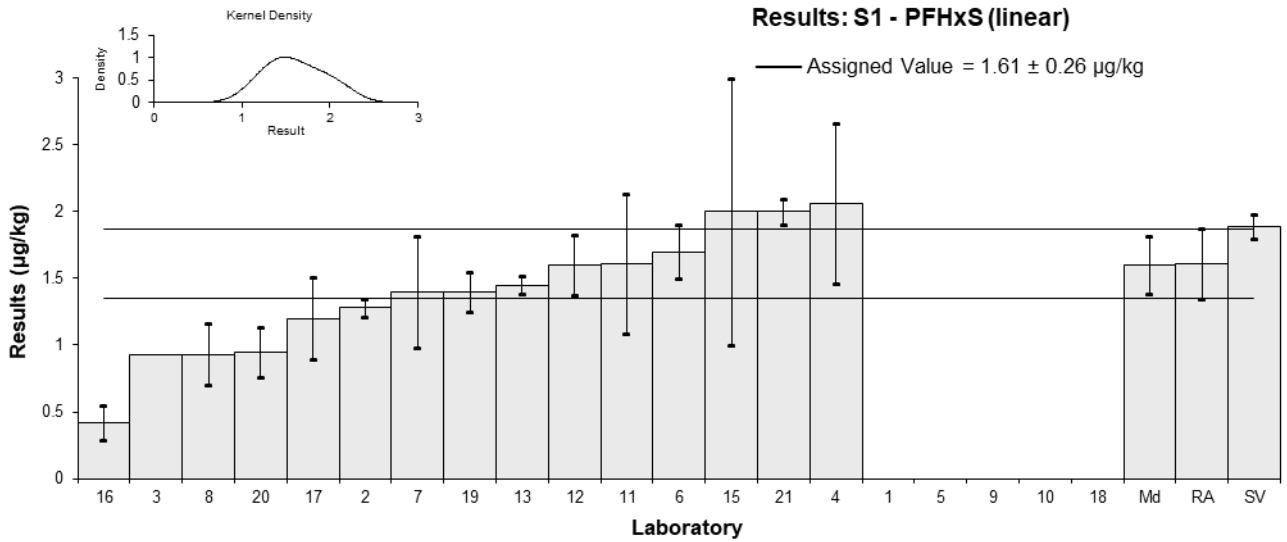


Figure 5

Table 10

## Sample Details

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <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   |       |                |
| 2         | 1.20   | 0.077       | 97   | -1.41 | -1.35          |
| 3**       | 0.967  | NR          | NR   | -2.10 | -2.07          |
| 4         | 1.73   | 0.5         | NR   | 0.18  | 0.10           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | 2.3    | 0.15        | NT   | 1.89  | 1.70           |
| 7         | 1.40   | 0.42        | 85   | -0.81 | -0.50          |
| 8**       | 0.99   | 0.30        | NR   | -2.04 | -1.50          |
| 9         | NT     | NT          | NT   |       |                |
| 10        | <1     | NR          | NR   |       |                |
| 11        | 1.65   | 0.416       | 94.0 | -0.06 | -0.04          |
| 12        | 1.99   | 0.298       | 83.7 | 0.96  | 0.71           |
| 13        | 1.34   | 0.067       | 106  | -0.99 | -0.95          |
| 15        | 2      | 1           | 89   | 0.99  | 0.31           |
| 16**      | 0.418  | 0.1254      | 147  | -3.75 | -3.45          |
| 17        | 1.2    | 0.29        | 89   | -1.41 | -1.05          |
| 18        | <1     | NR          | 151  |       |                |
| 19        | 1.4    | 0.12        | 107  | -0.81 | -0.75          |
| 20**      | 0.811  | 0.162       | 80   | -2.57 | -2.28          |
| 21        | 2.2    | 0.15        | NT   | 1.59  | 1.43           |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |      |      |
|-----------------------|------|------|
| <b>Assigned Value</b> | 1.67 | 0.34 |
| <b>Spike Value</b>    | 2.00 | 0.10 |
| <b>Robust Average</b> | 1.67 | 0.34 |
| <b>Median</b>         | 1.65 | 0.38 |
| <b>Mean</b>           | 1.67 |      |
| <b>N</b>              | 11   |      |
| <b>Max</b>            | 2.3  |      |
| <b>Min</b>            | 1.2  |      |
| <b>Robust SD</b>      | 0.45 |      |
| <b>Robust CV</b>      | 27%  |      |

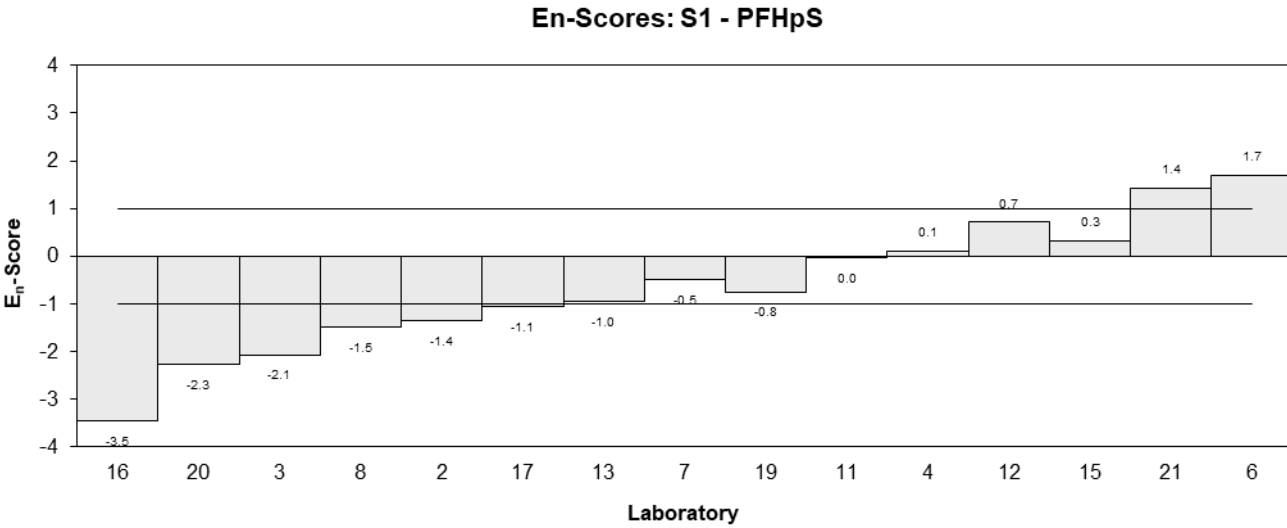
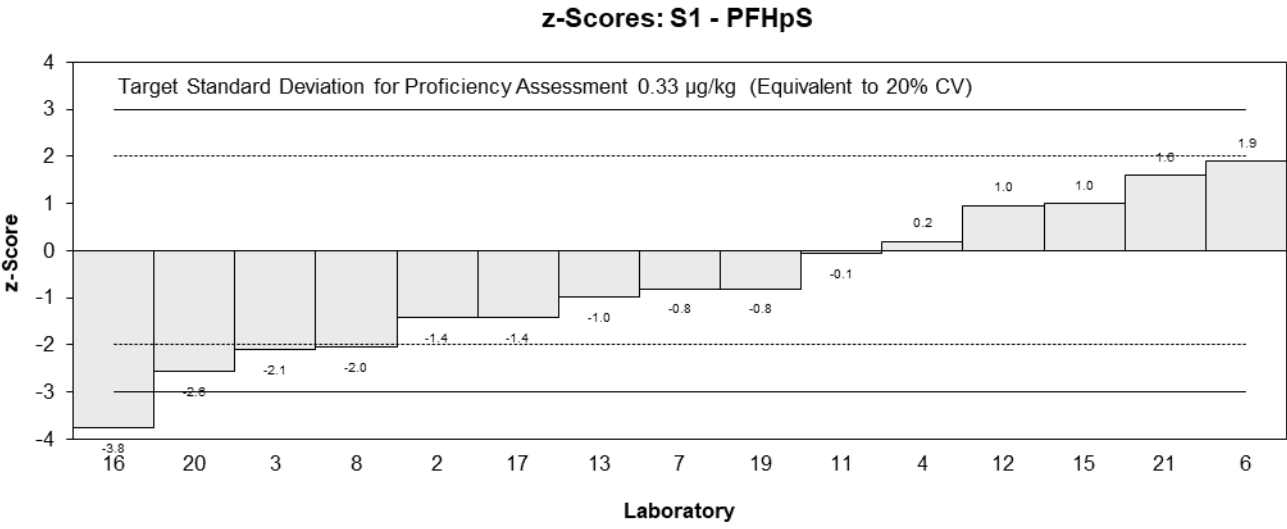
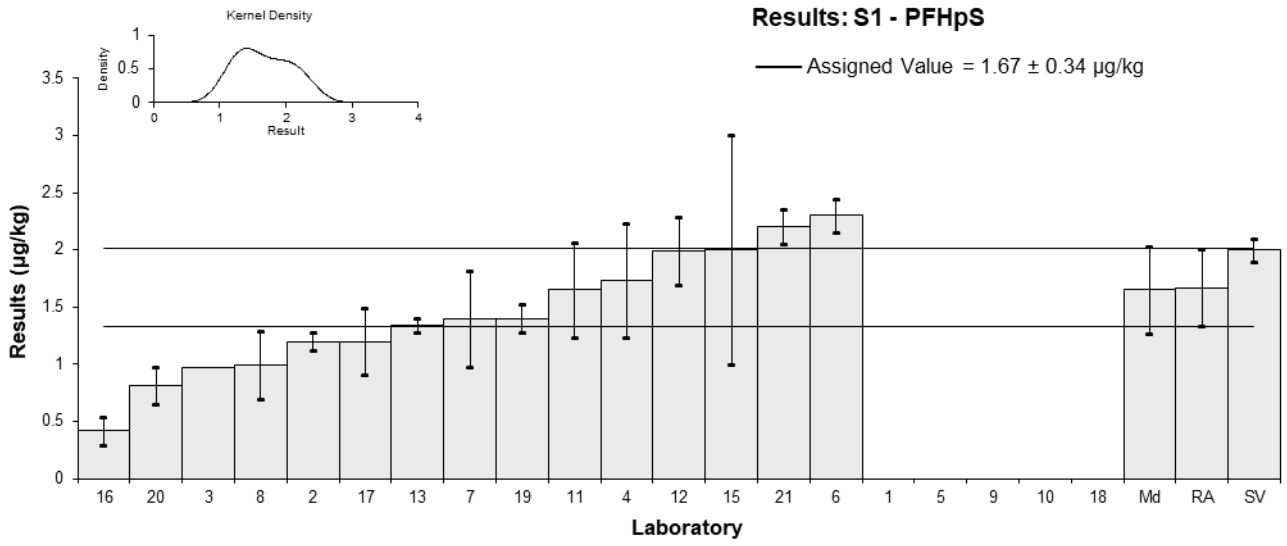


Figure 6

Table 11

## Sample Details

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | PFOS  |
| <b>Unit</b>       | µg/kg |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 2      | 1           | 111  | -2.24 | -1.34          |
| 2         | 2.93   | 0.183       | 88   | -0.95 | -0.98          |
| 3         | NT     | NT          | NT   |       |                |
| 4         | 4.43   | 1           | 74   | 1.12  | 0.67           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | 4.3    | 0.25        | 107  | 0.94  | 0.94           |
| 7         | 3.4    | 1.0         | 78   | -0.30 | -0.18          |
| 8**       | 2.40   | 0.60        | 106  | -1.69 | -1.35          |
| 9         | 3.2    | 1.6         | 77   | -0.58 | -0.24          |
| 10**      | 2.3    | 0.9         | NR   | -1.82 | -1.17          |
| 11        | 3.97   | 0.881       | 93.2 | 0.48  | 0.31           |
| 12        | 5.01   | 0.851       | 83.7 | 1.92  | 1.28           |
| 13        | 3.52   | 0.233       | 104  | -0.14 | -0.14          |
| 15        | 5      | 2           | 91   | 1.91  | 0.65           |
| 16**      | 1.13   | 0.339       | 136  | -3.44 | -3.28          |
| 17        | 2.9    | 0.74        | 89   | -0.99 | -0.72          |
| 18        | <1     | NR          | 151  |       |                |
| 19        | 3.1    | 0.87        | 102  | -0.72 | -0.47          |
| 20        | NR     | NR          | 69   |       |                |
| 21        | 3.2    | 0.2         | 107  | -0.58 | -0.59          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |      |      |
|-----------------------|------|------|
| <b>Assigned Value</b> | 3.62 | 0.68 |
| <b>Spike Value</b>    | 4.77 | 0.24 |
| <b>Robust Average</b> | 3.62 | 0.68 |
| <b>Median</b>         | 3.40 | 0.51 |
| <b>Mean</b>           | 3.61 |      |
| <b>N</b>              | 13   |      |
| <b>Max</b>            | 5.01 |      |
| <b>Min</b>            | 2    |      |
| <b>Robust SD</b>      | 0.98 |      |
| <b>Robust CV</b>      | 27%  |      |

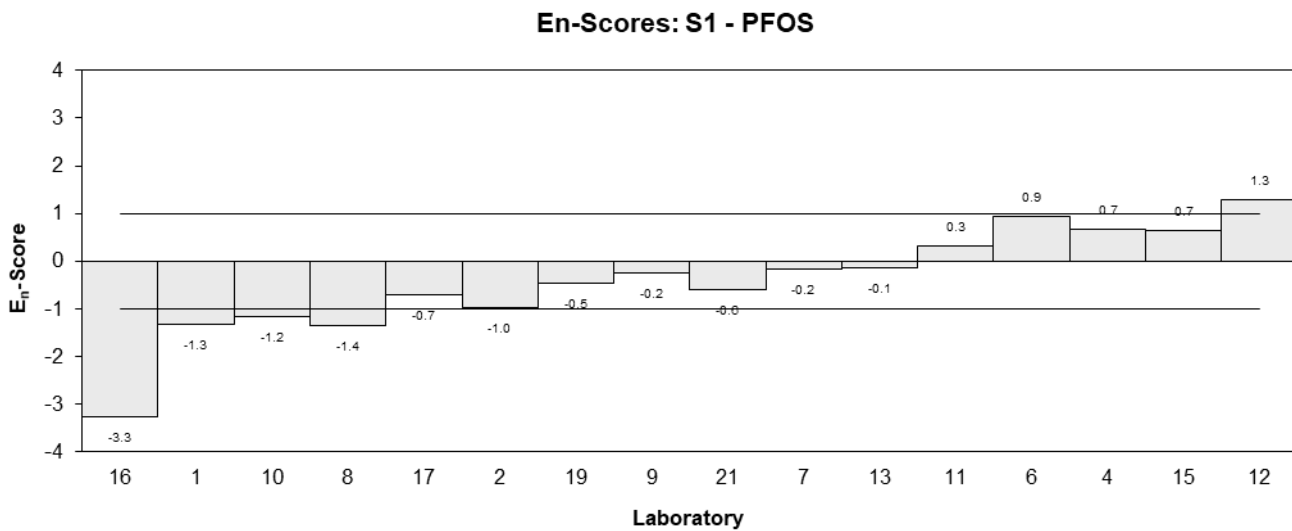
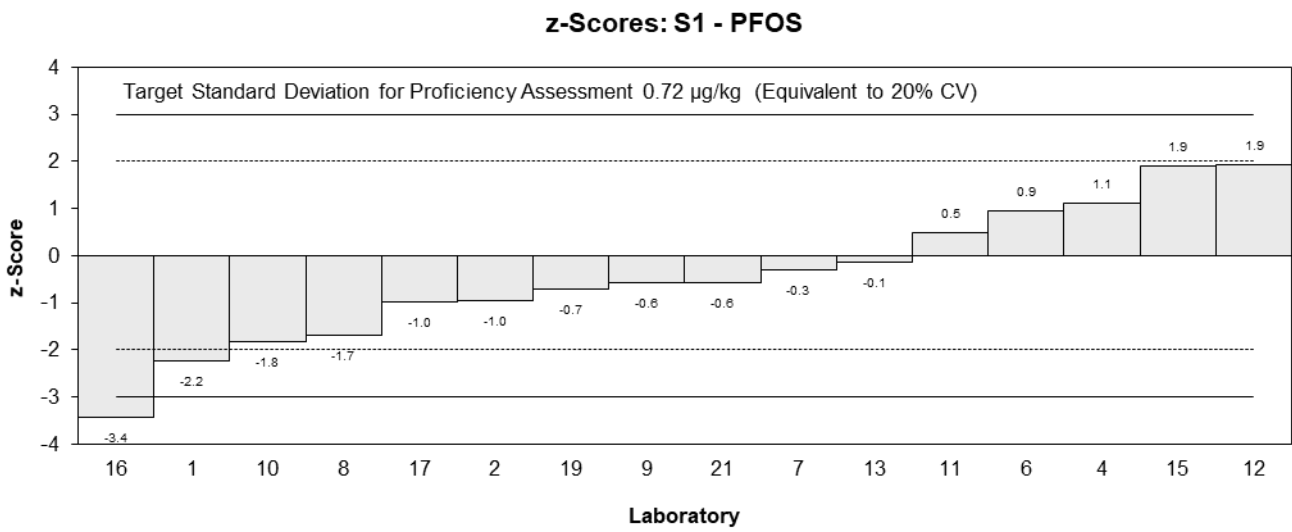
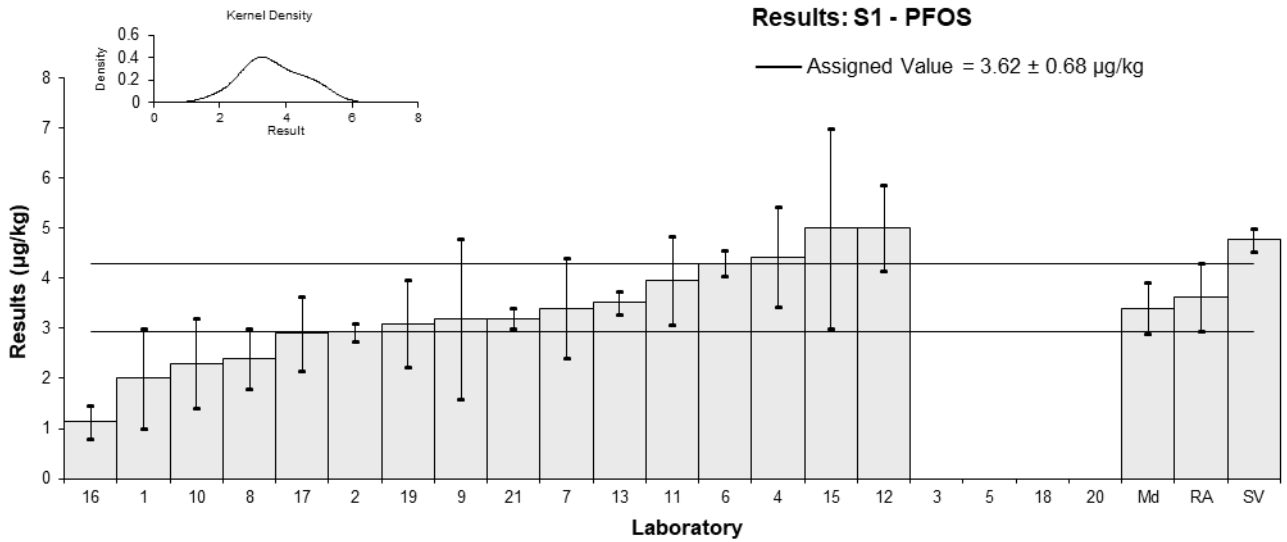


Figure 7

Table 12

## Sample Details

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

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 2.3    | 1.1         | NR   | -1.93 | -1.09          |
| 2         | NR     | NR          | NR   |       |                |
| 3**       | 2.015  | NR          | NR   | -2.31 | -2.33          |
| 4         | 4.43   | 1           | NR   | 0.92  | 0.55           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | 4.3    | 0.25        | 107  | 0.75  | 0.72           |
| 7         | 3.4    | 1.0         | 78   | -0.45 | -0.27          |
| 8**       | 2.40   | 0.60        | 106  | -1.79 | -1.41          |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NR     | NR          | NR   |       |                |
| 11        | 3.97   | 0.881       | 93.2 | 0.31  | 0.20           |
| 12        | 4.96   | 0.851       | 83.7 | 1.63  | 1.08           |
| 13        | 3.54   | 0.162       | 104  | -0.27 | -0.26          |
| 15        | 5      | 2           | 91   | 1.68  | 0.59           |
| 16**      | 1.13   | 0.339       | 136  | -3.49 | -3.21          |
| 17        | 2.9    | 0.74        | 89   | -1.12 | -0.80          |
| 18        | <1     | NR          | 151  |       |                |
| 19        | 3.1    | 0.87        | 102  | -0.86 | -0.56          |
| 20**      | 1.97   | 0.394       | 69   | -2.37 | -2.11          |
| 21        | 3.2    | 0.2         | 107  | -0.72 | -0.70          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |      |      |
|-----------------------|------|------|
| <b>Assigned Value</b> | 3.74 | 0.74 |
| <b>Spike Value</b>    | 4.77 | 0.24 |
| <b>Robust Average</b> | 3.74 | 0.74 |
| <b>Median</b>         | 3.54 | 0.72 |
| <b>Mean</b>           | 3.74 |      |
| <b>N</b>              | 11   |      |
| <b>Max</b>            | 5    |      |
| <b>Min</b>            | 2.3  |      |
| <b>Robust SD</b>      | 0.99 |      |
| <b>Robust CV</b>      | 26%  |      |

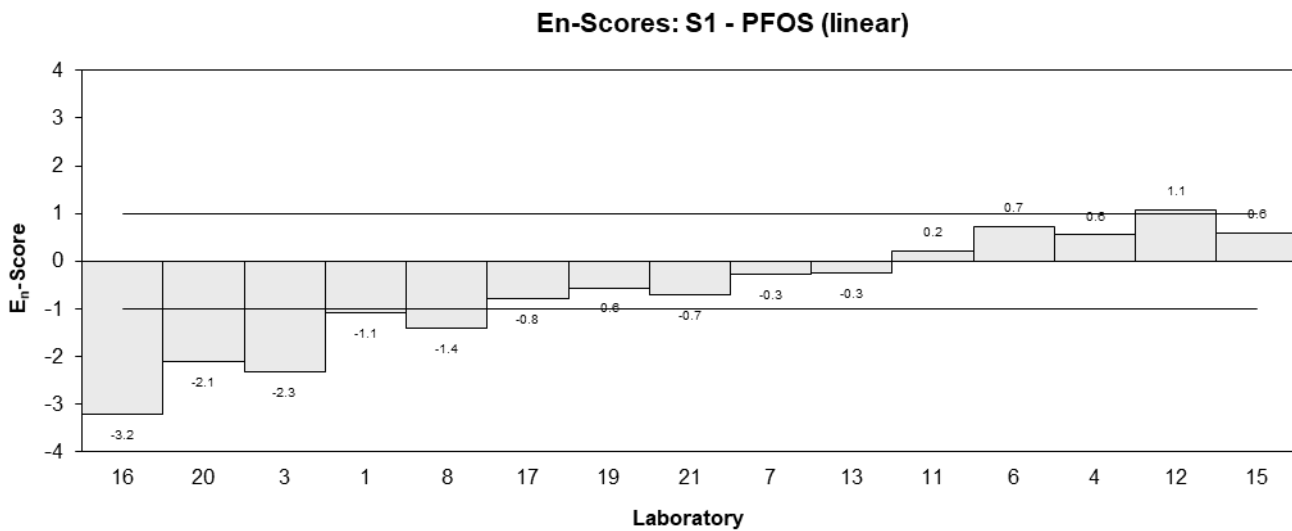
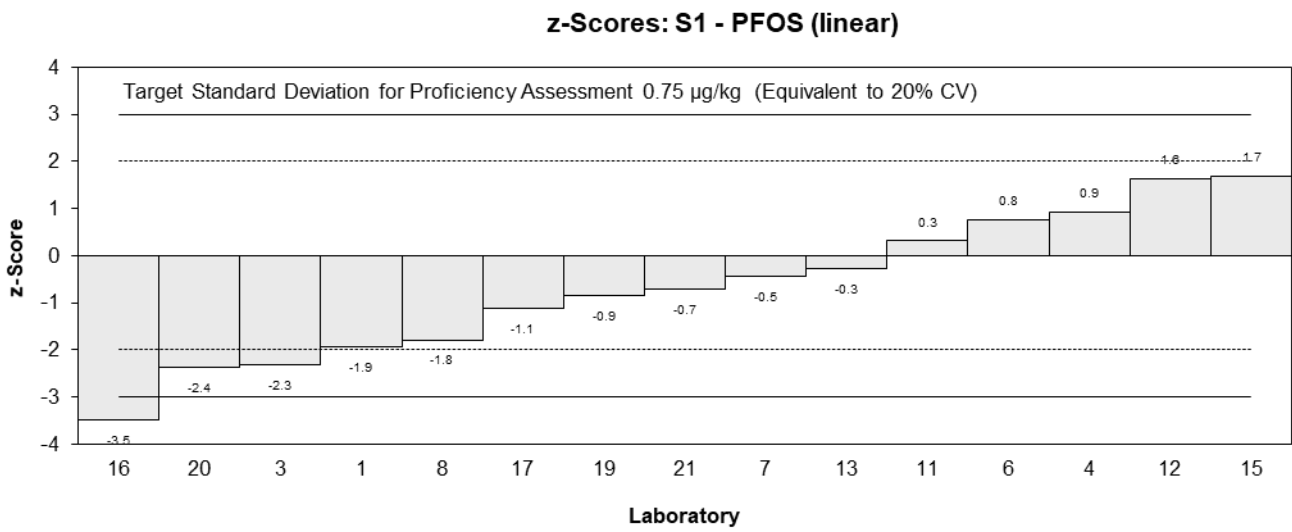
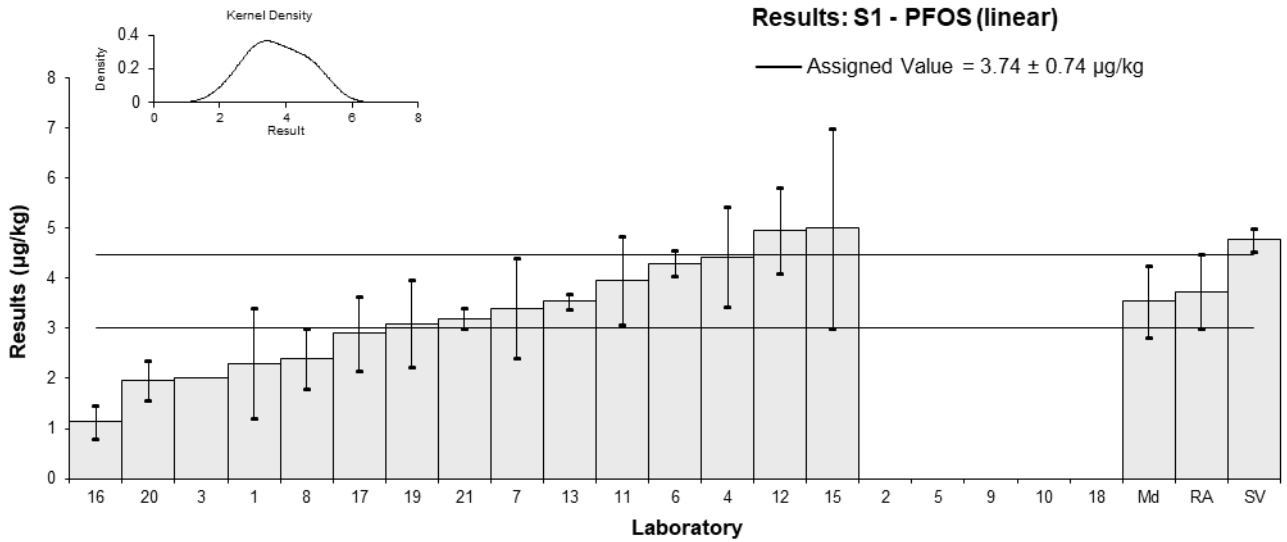


Figure 8

Table 13

## Sample Details

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | PFNS  |
| <b>Unit</b>       | µg/kg |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 5      | 2.5         | NR   | -1.88 | -0.96          |
| 2         | 7.25   | 0.314       | 88   | -0.47 | -0.39          |
| 3         | NT     | NT          | NT   |       |                |
| 4         | 11.3   | 3           | NR   | 2.00▼ | 0.93           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | 6.4    | 0.35        | NT   | -1.00 | -0.83          |
| 7         | 7.63   | 2.3         | 82   | -0.23 | -0.12          |
| 8**       | 4.49   | 1.79        | NR   | -2.19 | -1.34          |
| 9         | NT     | NT          | NT   |       |                |
| 10**      | 3.7    | 1.8         | NR   | -2.69 | -1.64          |
| 11        | 9.24   | 6.23        | 93.2 | 0.78  | 0.19           |
| 12        | 10.8   | 2.26        | 83.7 | 1.75  | 0.95           |
| 13        | 5.89   | 1.25        | 104  | -1.32 | -0.93          |
| 15        | 11     | 4           | 91   | 1.88  | 0.68           |
| 16        | NT     | NT          | NT   |       |                |
| 17        | 9.3    | 2.3         | 89   | 0.81  | 0.44           |
| 18        | NT     | NT          | NT   |       |                |
| 19        | 7.2    | 1.6         | 102  | -0.50 | -0.32          |
| 20**      | 3.58   | 0.715       | 69   | -2.76 | -2.18          |
| 21        | 4.9    | 0.3         | NT   | -1.94 | -1.61          |

\*\* Not included in robust average calculations, see Section 4.2; ▼ Adjusted Score, see Section 6.3

## Statistics

|                              |      |     |
|------------------------------|------|-----|
| <b>Assigned Value</b>        | 8.0  | 1.9 |
| <b>Spike Value</b>           | 11.5 | 0.6 |
| <b>Robust Average</b>        | 8.0  | 1.9 |
| <b>Max Acceptable Result</b> | 14.7 |     |
| <b>Median</b>                | 7.4  | 2.0 |
| <b>Mean</b>                  | 8.0  |     |
| <b>N</b>                     | 12   |     |
| <b>Max</b>                   | 11.3 |     |
| <b>Min</b>                   | 4.9  |     |
| <b>Robust SD</b>             | 2.6  |     |
| <b>Robust CV</b>             | 33%  |     |



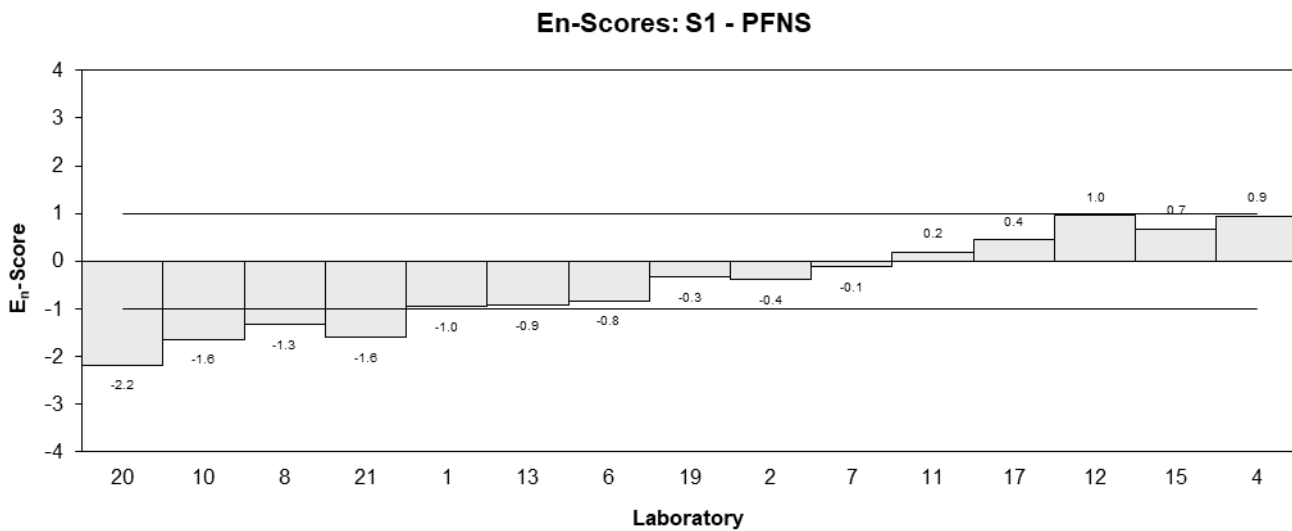
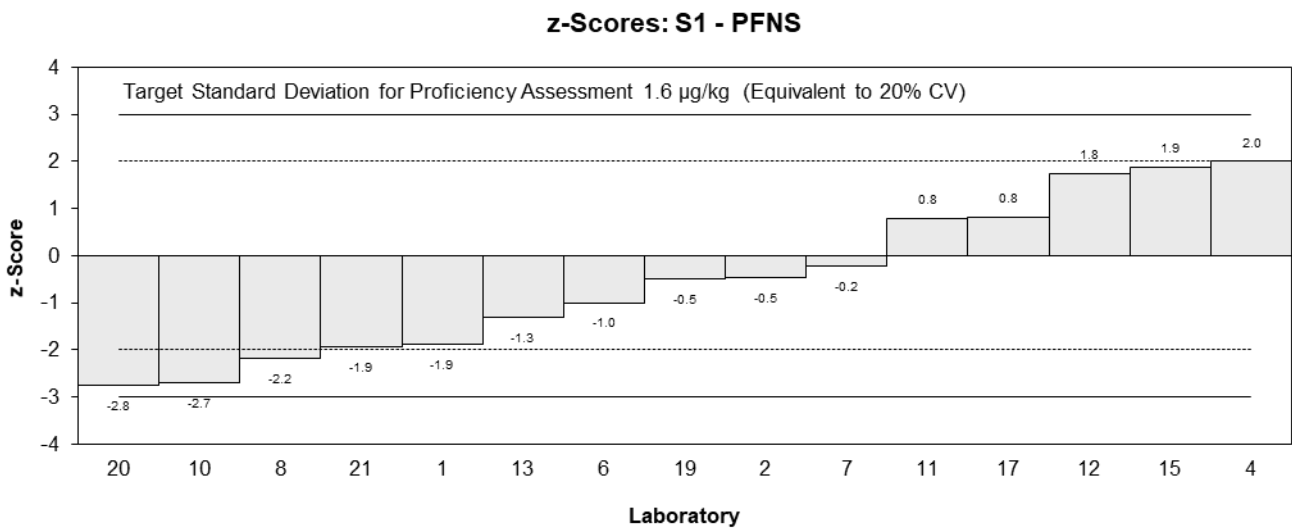
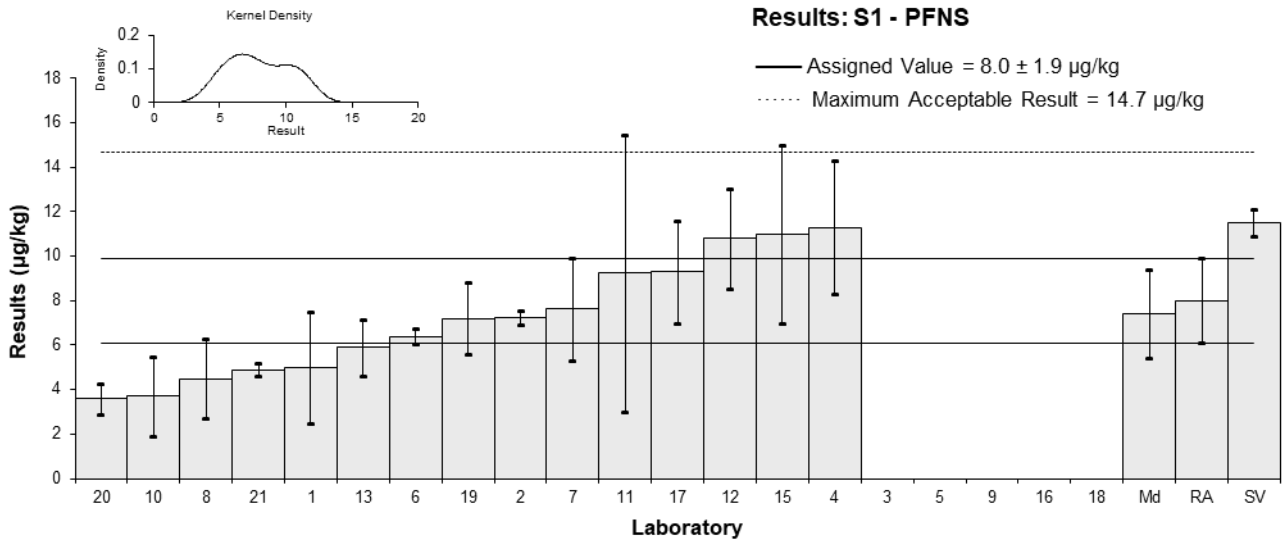


Figure 9

Table 14

## Sample Details

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | PFBA  |
| <b>Unit</b>       | µg/kg |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | < 2    | 1           | 103  |       |                |
| 2         | 1.48   | 0.051       | 67   | -1.35 | -1.12          |
| 3**       | 1.104  | NR          | NR   | -2.28 | -1.89          |
| 4         | 2.67   | 1           | 77   | 1.58  | 0.57           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | <5     | NR          | 105  |       |                |
| 7         | 1.86   | 0.56        | 79   | -0.42 | -0.23          |
| 8**       | 1.16   | 0.29        | 88   | -2.14 | -1.53          |
| 9         | NT     | NT          | NT   |       |                |
| 10**      | 1.5    | 0.7         | NR   | -1.31 | -0.62          |
| 11        | 2.89   | 0.680       | 95.7 | 2.00▼ | 1.00▼          |
| 12        | 2.21   | 0.199       | 85.4 | 0.44  | 0.34           |
| 13        | 1.79   | 0.035       | 104  | -0.59 | -0.49          |
| 15        | 2      | 1           | 64   | -0.07 | -0.03          |
| 16**      | 0.582  | 0.1746      | 102  | -3.57 | -2.78          |
| 17        | 1.3    | 0.94        | 89   | -1.80 | -0.69          |
| 18        | <5     | NR          | 128  |       |                |
| 19        | 2.1    | 0.14        | 123  | 0.17  | 0.14           |
| 20        | NT     | NT          | NT   |       |                |
| 21        | <5     | NR          | 105  |       |                |

\*\* Not included in robust average calculations, see Section 4.2; ▼ Adjusted Score, see Section 6.3

## Statistics

|                              |      |      |
|------------------------------|------|------|
| <b>Assigned Value</b>        | 2.03 | 0.49 |
| <b>Spike Value</b>           | 2.96 | 0.15 |
| <b>Robust Average</b>        | 2.03 | 0.49 |
| <b>Max Acceptable Result</b> | 3.8  |      |
| <b>Median</b>                | 2.00 | 0.26 |
| <b>Mean</b>                  | 2.03 |      |
| <b>N</b>                     | 9    |      |
| <b>Max</b>                   | 2.89 |      |
| <b>Min</b>                   | 1.3  |      |
| <b>Robust SD</b>             | 0.58 |      |
| <b>Robust CV</b>             | 29%  |      |

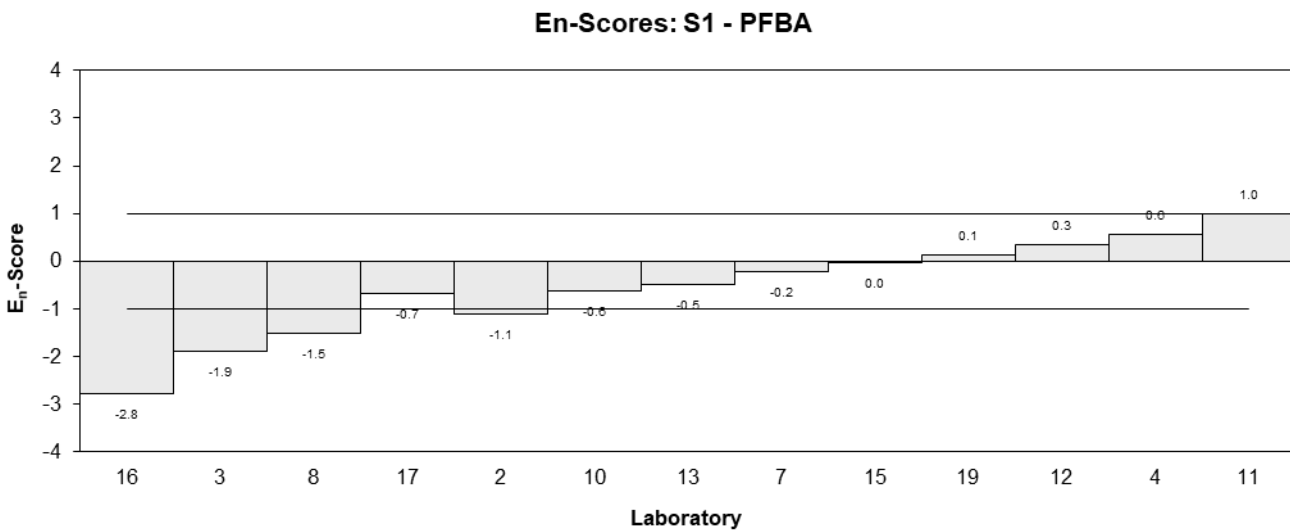
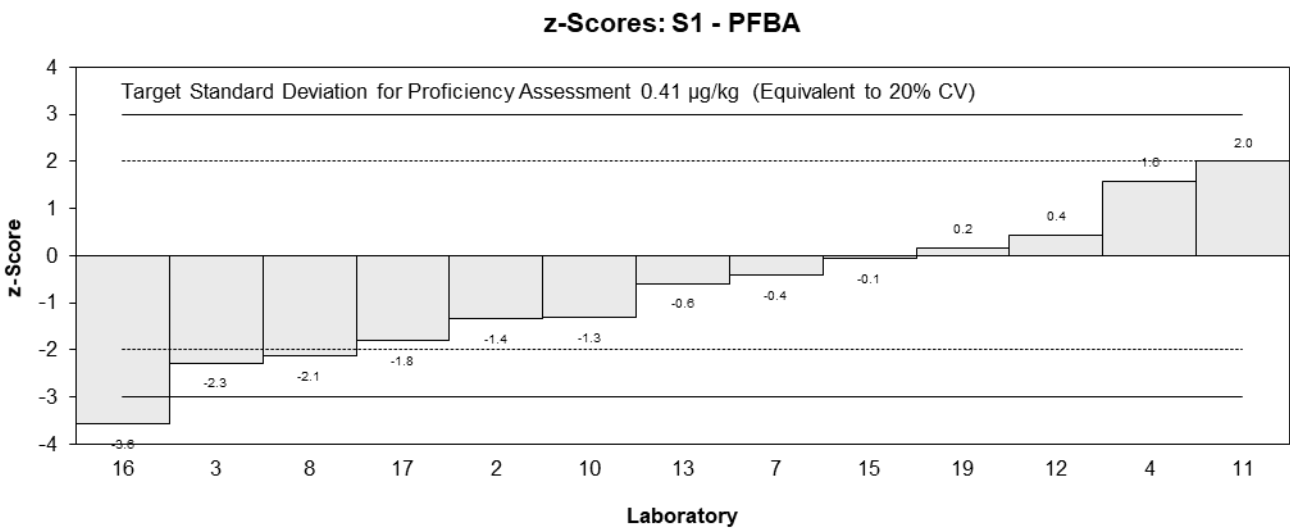
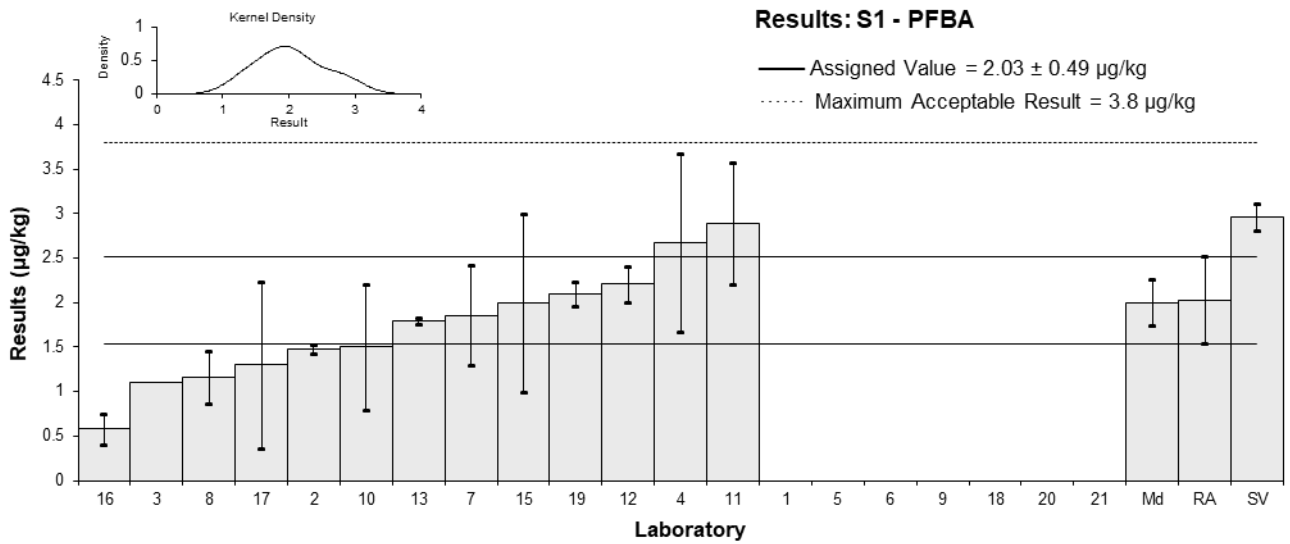


Figure 10

Table 15

## Sample Details

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | PFPeA |
| <b>Unit</b>       | µg/kg |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | < 2    | 1           | 106  |       |                |
| 2*        | 0.479  | 0.032       | 73   | -2.70 | -3.66          |
| 3**       | 0.562  | NR          | NR   | -2.30 | -3.19          |
| 4         | 1.16   | 0.3         | 71   | 0.58  | 0.36           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | <1     | NR          | NT   |       |                |
| 7         | 0.885  | 0.27        | 85   | -0.75 | -0.50          |
| 8**       | 0.55   | 0.14        | 98   | -2.36 | -2.39          |
| 9         | NT     | NT          | NT   |       |                |
| 10        | <1     | NR          | NR   |       |                |
| 11        | 1.05   | 0.196       | 93.2 | 0.05  | 0.04           |
| 12        | 1.15   | 0.115       | 101  | 0.53  | 0.58           |
| 13        | 0.878  | 0.021       | 106  | -0.78 | -1.07          |
| 15        | <2     | NR          | 76   |       |                |
| 16        | < 0.3  | NR          | 133  |       |                |
| 17        | 1.1    | 0.27        | 89   | 0.29  | 0.19           |
| 18        | <2     | NR          | 138  |       |                |
| 19        | < 1.0  | NR          | 118  |       |                |
| 20        | NT     | NT          | NT   |       |                |
| 21        | <1     | NR          | NT   |       |                |

\* Outlier, \*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |       |      |
|-----------------------|-------|------|
| <b>Assigned Value</b> | 1.04  | 0.15 |
| <b>Spike Value</b>    | 1.13  | 0.06 |
| <b>Robust Average</b> | 0.99  | 0.19 |
| <b>Median</b>         | 1.05  | 0.15 |
| <b>Mean</b>           | 0.96  |      |
| <b>N</b>              | 7     |      |
| <b>Max</b>            | 1.16  |      |
| <b>Min</b>            | 0.479 |      |
| <b>Robust SD</b>      | 0.20  |      |
| <b>Robust CV</b>      | 20%   |      |

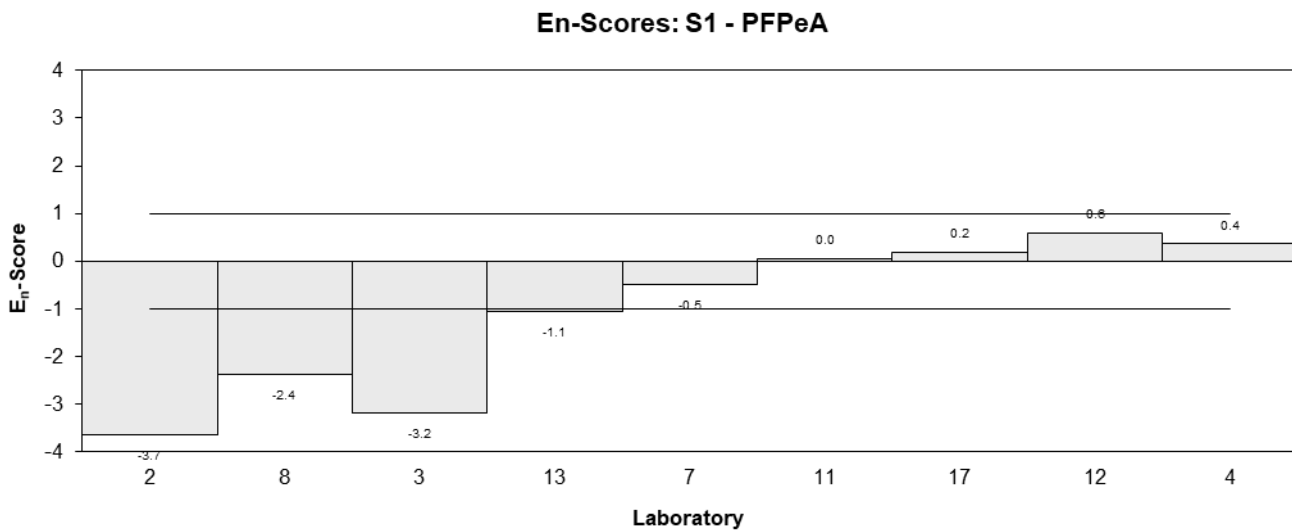
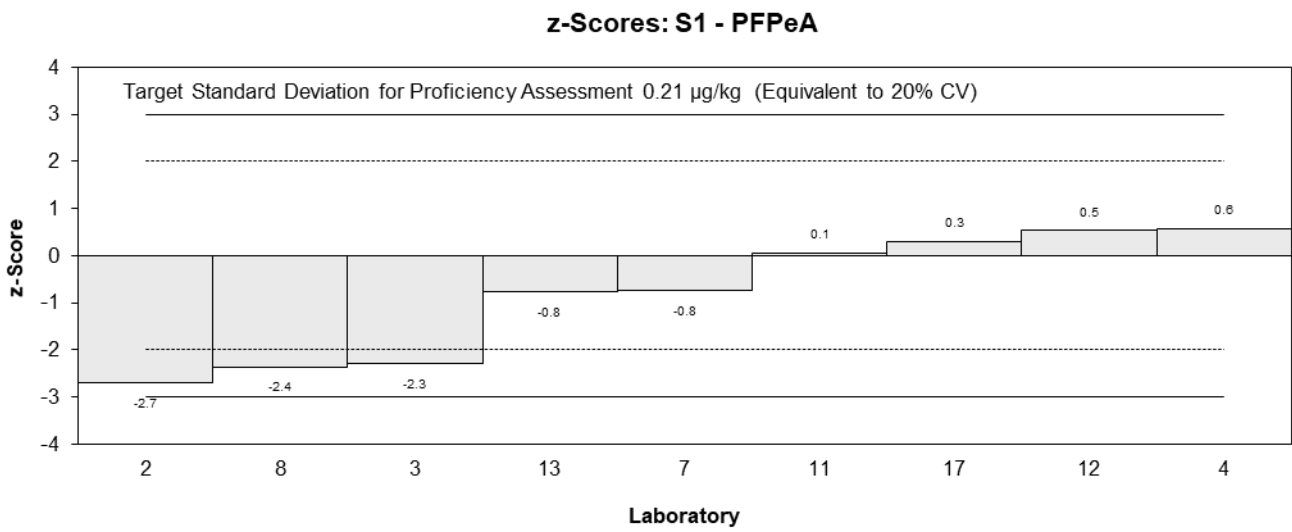
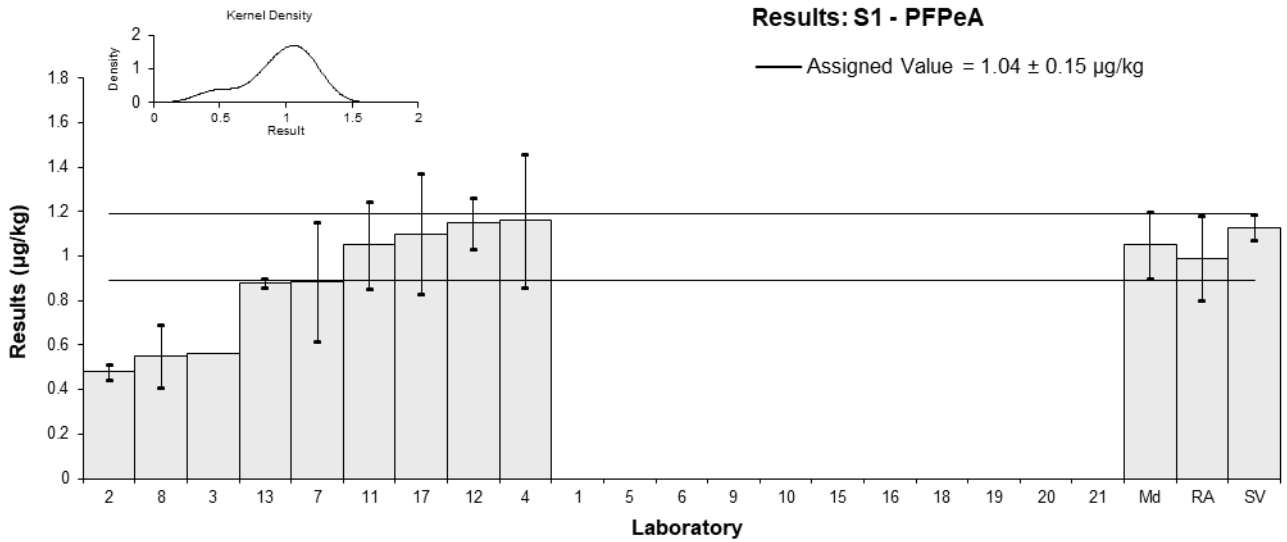


Figure 11

Table 16

## Sample Details

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | PFHxA |
| <b>Unit</b>       | µg/kg |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 4      | 2           | 100  | -0.66 | -0.29          |
| 2         | 3.42   | 0.265       | 73   | -1.29 | -1.84          |
| 3**       | 2.979  | NR          | NR   | -1.77 | -2.76          |
| 4         | 5.4    | 2           | 84   | 0.86  | 0.38           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | 4.9    | 0.25        | NT   | 0.31  | 0.45           |
| 7         | 4.32   | 1.3         | 91   | -0.31 | -0.20          |
| 8**       | 2.80   | 0.70        | 104  | -1.96 | -1.98          |
| 9         | 4.8    | 1.0         | 95   | 0.21  | 0.16           |
| 10**      | 2.4    | 0.9         | NR   | -2.40 | -2.05          |
| 11        | 4.70   | 1.15        | 96.4 | 0.10  | 0.07           |
| 12        | 5      | 0.7         | 83.3 | 0.42  | 0.43           |
| 13        | 4.49   | 0.092       | 116  | -0.13 | -0.20          |
| 15        | 6      | 2           | 85   | 1.51  | 0.67           |
| 16**      | 1.38   | 0.414       | 141  | -3.50 | -4.48          |
| 17        | 3.9    | 0.99        | 89   | -0.77 | -0.62          |
| 18        | 3.34   | 1.3         | 124  | -1.38 | -0.89          |
| 19        | 4.7    | 0.46        | 113  | 0.10  | 0.12           |
| 20**      | 3.41   | 0.682       | 68   | -1.30 | -1.33          |
| 21        | 5.7    | 0.3         | NT   | 1.18  | 1.65           |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |      |      |
|-----------------------|------|------|
| <b>Assigned Value</b> | 4.61 | 0.59 |
| <b>Spike Value</b>    | 5.31 | 0.27 |
| <b>Robust Average</b> | 4.61 | 0.59 |
| <b>Median</b>         | 4.70 | 0.54 |
| <b>Mean</b>           | 4.62 |      |
| <b>N</b>              | 14   |      |
| <b>Max</b>            | 6    |      |
| <b>Min</b>            | 3.34 |      |
| <b>Robust SD</b>      | 0.88 |      |
| <b>Robust CV</b>      | 19%  |      |

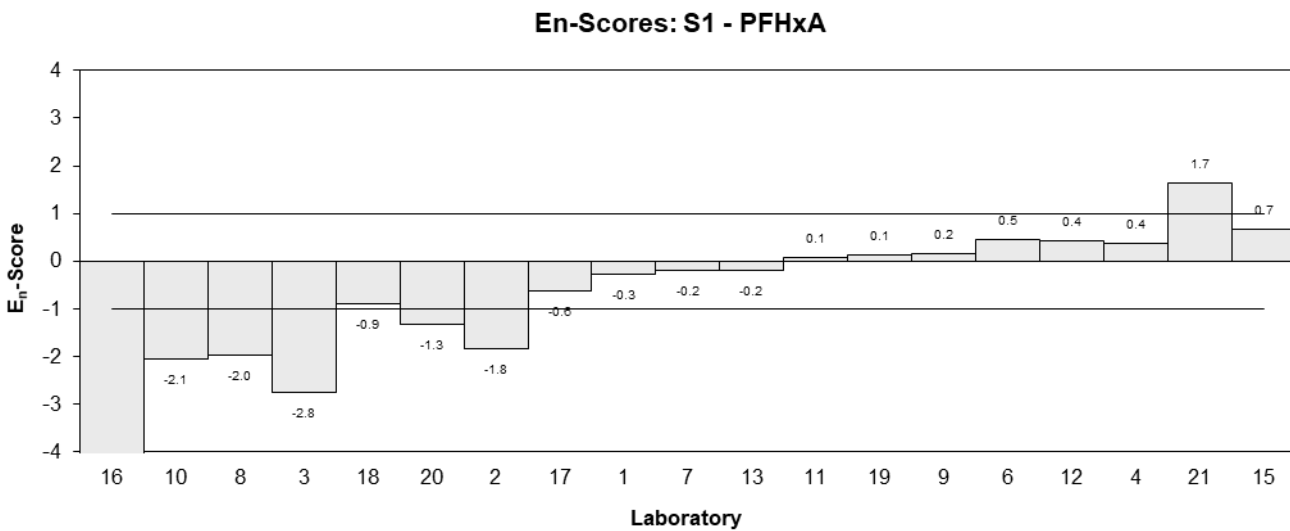
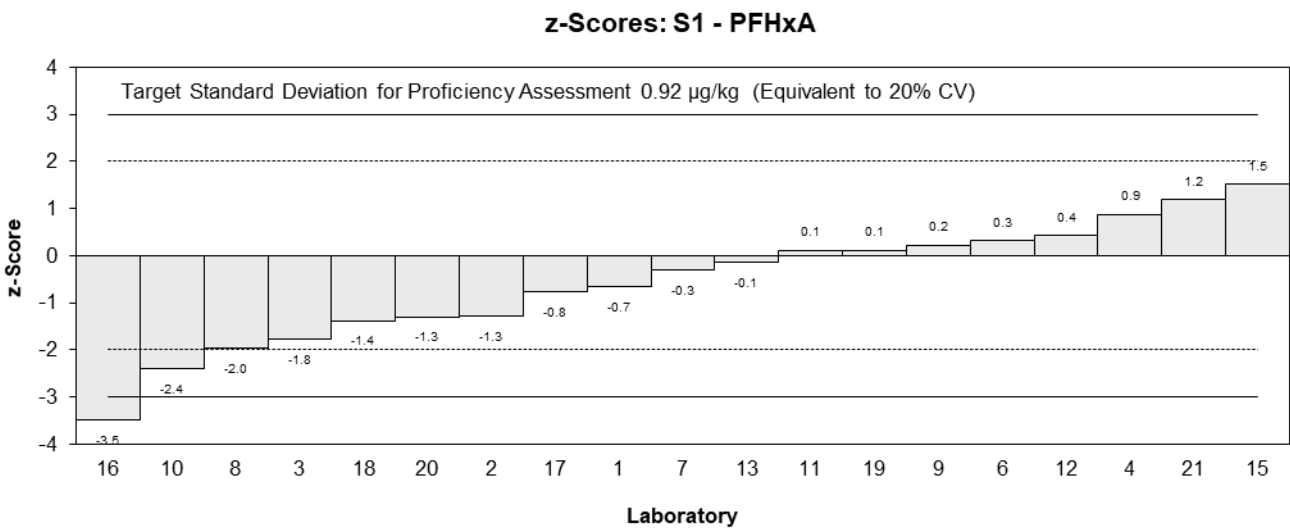
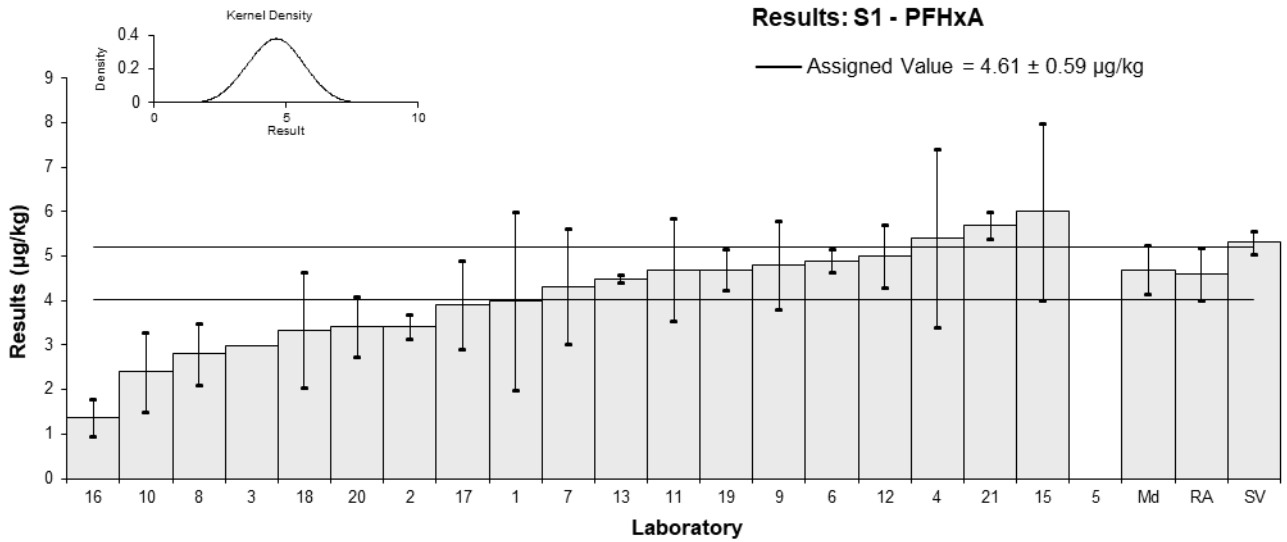


Figure 12

Table 17

## Sample Details

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | PFHpA |
| <b>Unit</b>       | µg/kg |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 4      | 2           | 111  | -1.68 | -0.95          |
| 2         | 4.85   | 0.330       | 73   | -0.97 | -1.46          |
| 3**       | 3.958  | NR          | NR   | -1.71 | -2.82          |
| 4         | 8.13   | 2           | 81   | 1.75  | 0.99           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | 6.0    | 0.3         | NT   | -0.02 | -0.03          |
| 7         | 5.58   | 1.7         | 82   | -0.37 | -0.24          |
| 8**       | 4.33   | 1.08        | 103  | -1.40 | -1.30          |
| 9         | 5.5    | 1.5         | 105  | -0.43 | -0.31          |
| 10**      | 3.7    | 1.2         | NR   | -1.93 | -1.65          |
| 11        | 6.82   | 1.92        | 94.9 | 0.66  | 0.39           |
| 12        | 6.43   | 0.965       | 89.3 | 0.34  | 0.34           |
| 13        | 6.15   | 0.161       | 104  | 0.11  | 0.17           |
| 15        | 8      | 3           | 87   | 1.64  | 0.64           |
| 16**      | 1.88   | 0.564       | 142  | -3.44 | -4.49          |
| 17        | 5.6    | 1.4         | 89   | -0.35 | -0.27          |
| 18*       | 2.72   | 1           | 122  | -2.74 | -2.67          |
| 19        | 5.8    | 1.3         | 119  | -0.18 | -0.15          |
| 20**      | 3.85   | 0.772       | 71   | -1.80 | -2.04          |
| 21        | 5.9    | 0.3         | NT   | -0.10 | -0.15          |

\* Outlier, \*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |      |      |
|-----------------------|------|------|
| <b>Assigned Value</b> | 6.02 | 0.73 |
| <b>Spike Value</b>    | 7.54 | 0.38 |
| <b>Robust Average</b> | 5.88 | 0.90 |
| <b>Median</b>         | 5.85 | 0.46 |
| <b>Mean</b>           | 5.82 |      |
| <b>N</b>              | 14   |      |
| <b>Max</b>            | 8.13 |      |
| <b>Min</b>            | 2.72 |      |
| <b>Robust SD</b>      | 1.3  |      |
| <b>Robust CV</b>      | 23%  |      |



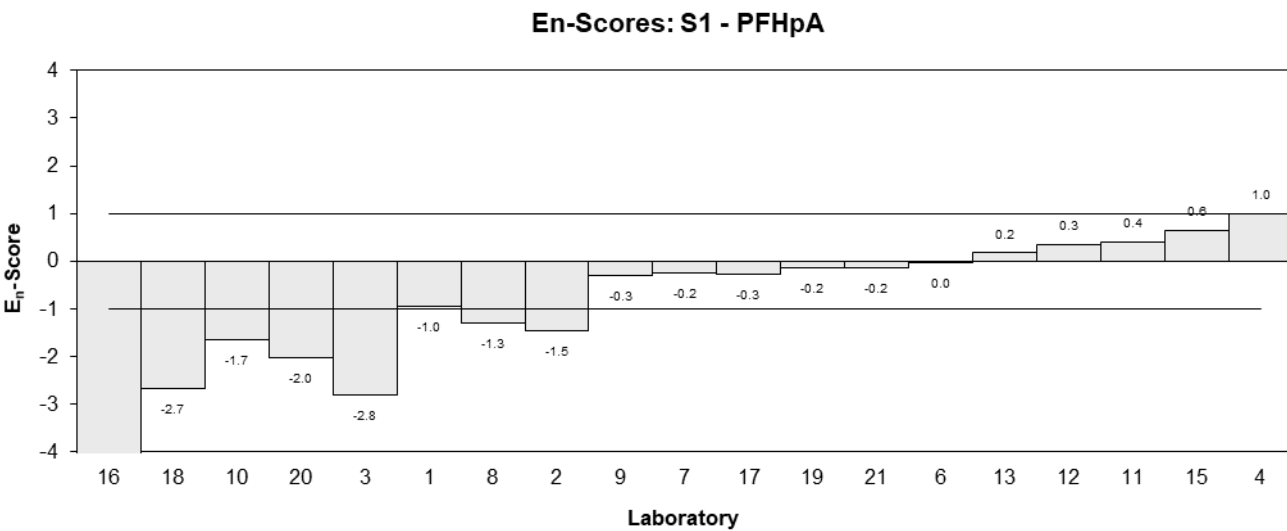
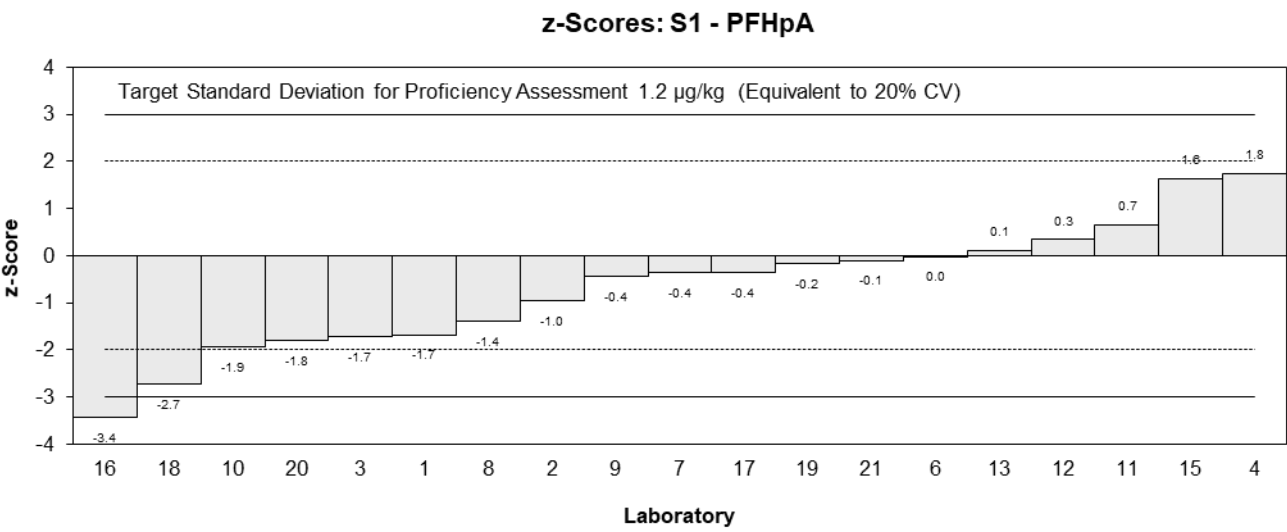
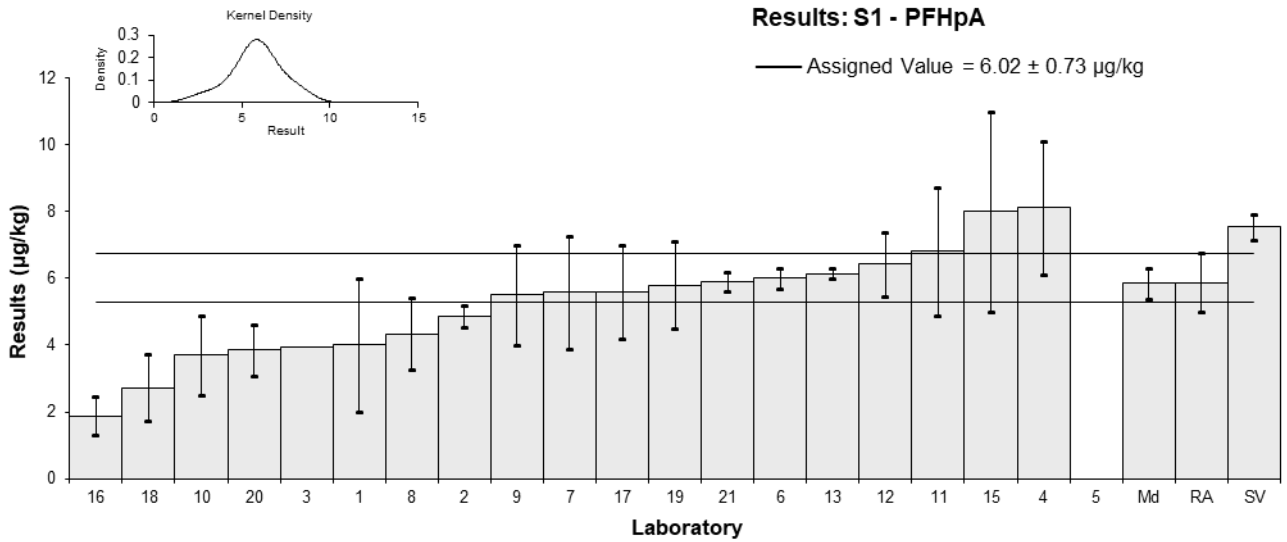


Figure 13

Table 18

## Sample Details

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <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         | 103  |       |                |
| 2         | 4.91   | 0.202       | 76   | -1.19 | -1.75          |
| 3**       | 3.426  | NR          | NR   | -2.34 | -3.55          |
| 4         | 8.38   | 2           | 73   | 1.51  | 0.89           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | 6.4    | 0.35        | 108  | -0.03 | -0.04          |
| 7         | 6.03   | 1.8         | 80   | -0.32 | -0.21          |
| 8**       | 4.26   | 1.06        | 111  | -1.69 | -1.60          |
| 9         | 5.7    | 1.5         | 95   | -0.57 | -0.43          |
| 10**      | 4.3    | 1.8         | NR   | -1.66 | -1.08          |
| 11        | 6.86   | 1.31        | 93.1 | 0.33  | 0.27           |
| 12        | 7.7    | 1.23        | 88.5 | 0.98  | 0.84           |
| 13        | 6.11   | 0.284       | 110  | -0.26 | -0.37          |
| 15        | 8      | 3           | 94   | 1.21  | 0.50           |
| 16**      | 1.98   | 0.594       | 152  | -3.46 | -4.30          |
| 17        | 5.5    | 1.4         | 89   | -0.73 | -0.57          |
| 18*       | 1.64   | 0.6         | 136  | -3.73 | -4.61          |
| 19        | 5.7    | 0.24        | 112  | -0.57 | -0.84          |
| 20**      | 3.92   | 0.784       | 64   | -1.96 | -2.18          |
| 21        | 6.2    | 0.4         | 108  | -0.19 | -0.26          |

\* Outlier, \*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |      |      |
|-----------------------|------|------|
| <b>Assigned Value</b> | 6.44 | 0.85 |
| <b>Spike Value</b>    | 7.92 | 0.40 |
| <b>Robust Average</b> | 6.28 | 0.92 |
| <b>Median</b>         | 6.11 | 0.63 |
| <b>Mean</b>           | 6.09 |      |
| <b>N</b>              | 13   |      |
| <b>Max</b>            | 8.38 |      |
| <b>Min</b>            | 1.64 |      |
| <b>Robust SD</b>      | 1.3  |      |
| <b>Robust CV</b>      | 21%  |      |

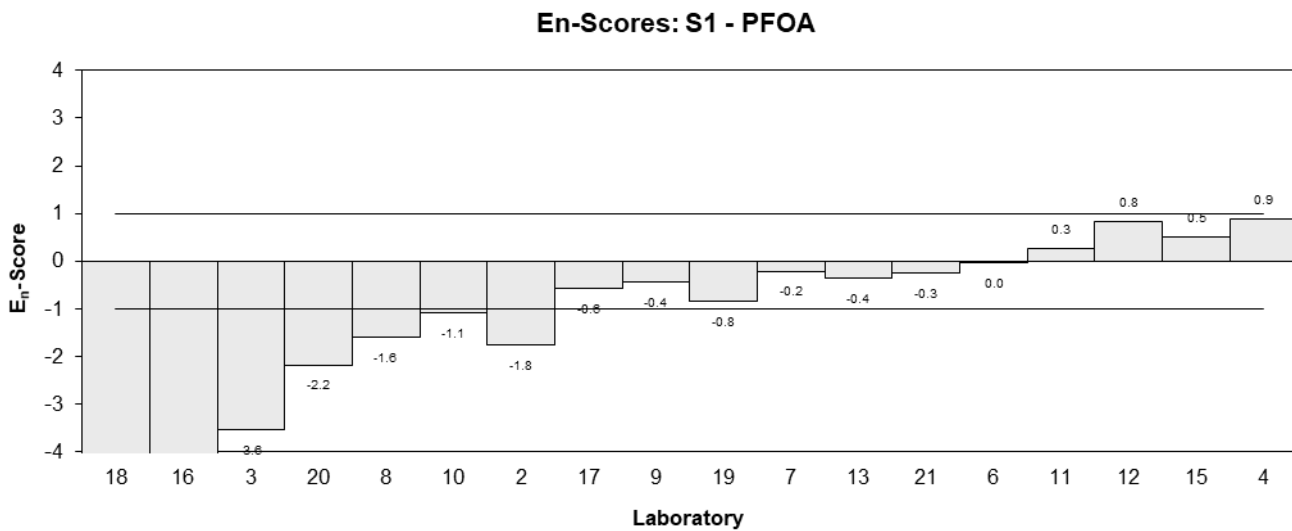
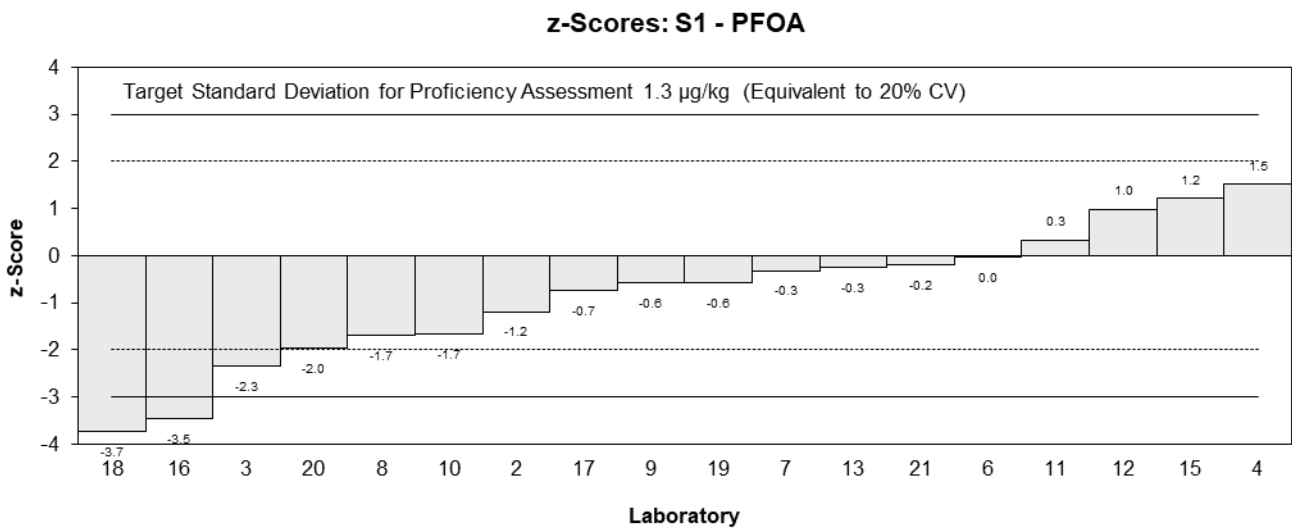
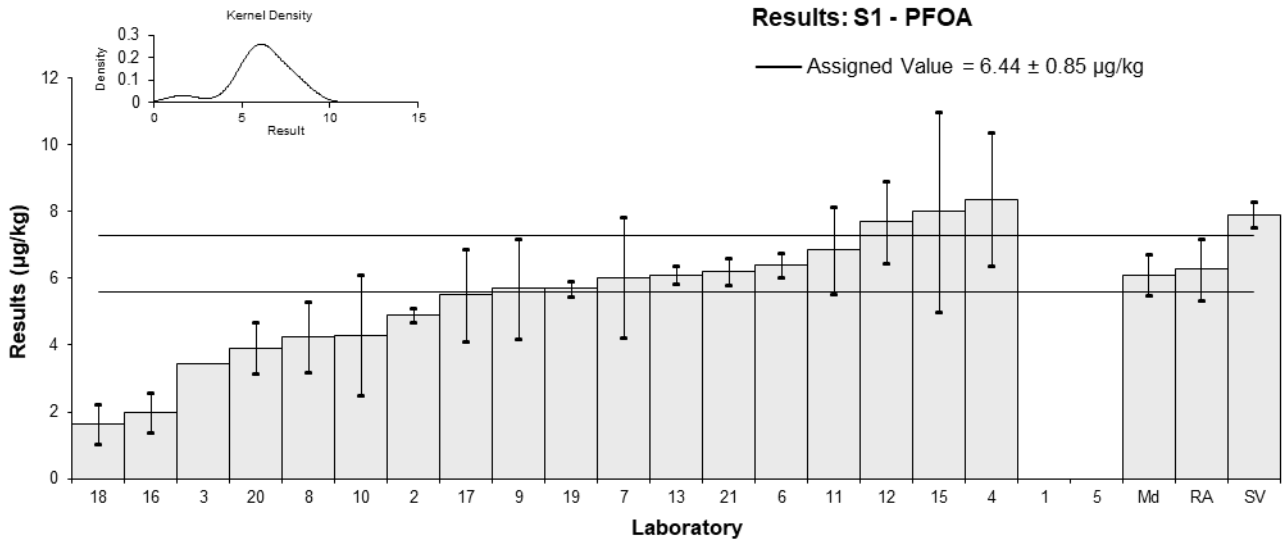


Figure 14

Table 19

## Sample Details

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | PFNA  |
| <b>Unit</b>       | µg/kg |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | < 2    | 1           | 111  |       |                |
| 2*        | 0.188  | 0.011       | 76   | -2.80 | -2.96          |
| 3**       | 0.206  | NR          | NR   | -2.59 | -2.76          |
| 4         | 0.523  | 0.2         | 70   | 1.12  | 0.45           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | <1     | NR          | NT   |       |                |
| 7         | 0.42   | 0.13        | 93   | -0.08 | -0.05          |
| 8         | <0.5   | 0.15        | 95   |       |                |
| 9         | 0.4    | 0.3         | 85   | -0.32 | -0.09          |
| 10        | <1     | NR          | NR   |       |                |
| 11        | 0.441  | 0.130       | 94.5 | 0.16  | 0.09           |
| 12        | 0.521  | 0.0834      | 86.7 | 1.10  | 0.81           |
| 13        | 0.343  | 0.029       | 102  | -0.98 | -0.99          |
| 15        | <1     | NR          | 93   |       |                |
| 16**      | 0.111  | 0.0333      | 171  | -3.70 | -3.65          |
| 17        | 0.34   | 0.24        | 89   | -1.02 | -0.34          |
| 18        | <1     | NR          | 149  |       |                |
| 19        | < 1.0  | NR          | 118  |       |                |
| 20**      | 0.224  | 0.045       | 74   | -2.38 | -2.21          |
| 21        | <1     | NR          | NT   |       |                |

\* Outlier, \*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |       |       |
|-----------------------|-------|-------|
| <b>Assigned Value</b> | 0.427 | 0.080 |
| <b>Spike Value</b>    | 0.503 | 0.025 |
| <b>Robust Average</b> | 0.404 | 0.095 |
| <b>Median</b>         | 0.410 | 0.090 |
| <b>Mean</b>           | 0.397 |       |
| <b>N</b>              | 8     |       |
| <b>Max</b>            | 0.523 |       |
| <b>Min</b>            | 0.188 |       |
| <b>Robust SD</b>      | 0.11  |       |
| <b>Robust CV</b>      | 27%   |       |

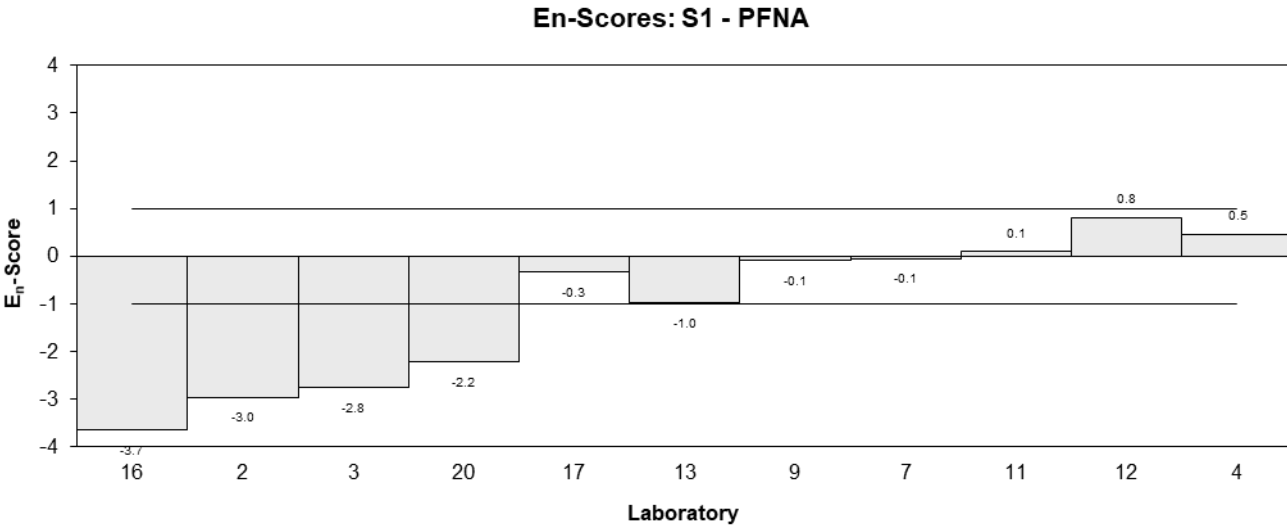
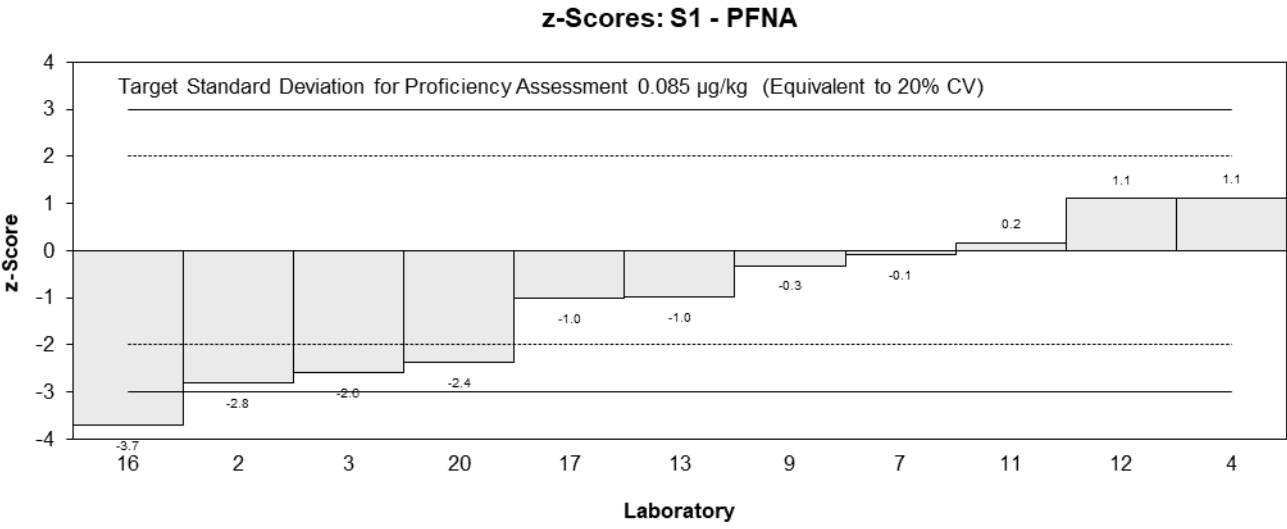
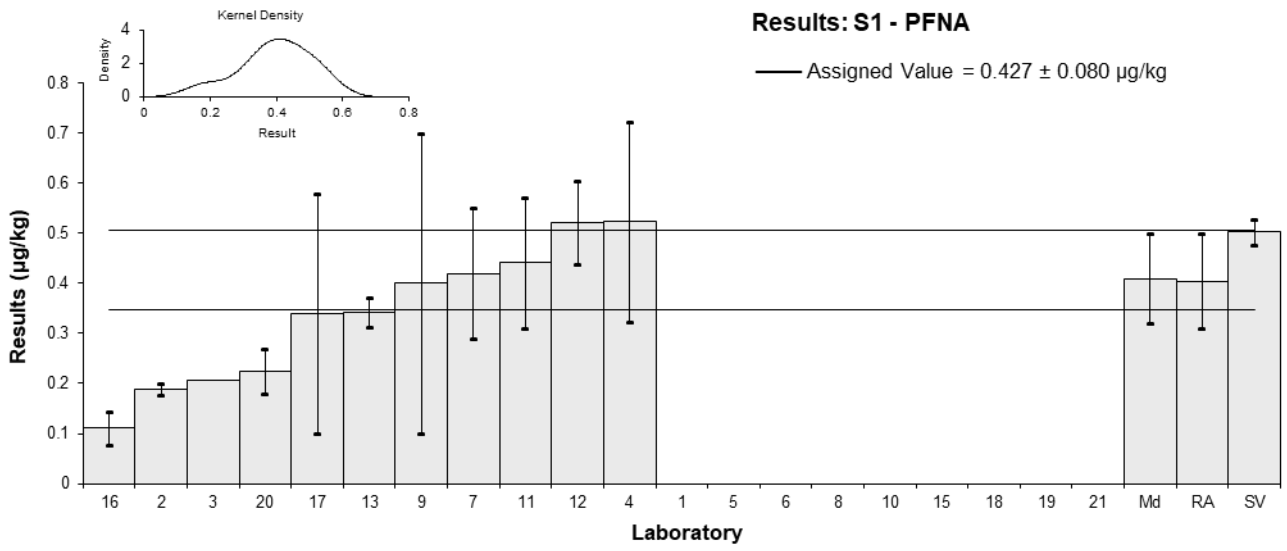


Figure 15

Table 20

**Sample Details**

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | PFDA  |
| <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           | 2.5                | 109        |          |                      |
| 2                | 0.438         | 0.028              | 76         | -2.04    | -1.86                |
| 3**              | 0.304         | NR                 | NR         | -2.95    | -2.72                |
| 4                | 0.986         | 0.3                | 70         | 1.66     | 0.72                 |
| 5                | NS            | NS                 | NS         |          |                      |
| 6                | <1            | NR                 | 101        |          |                      |
| 7                | 0.741         | 0.22               | 95         | 0.01     | 0.00                 |
| 8                | <0.5          | 0.15               | 120        |          |                      |
| 9                | 0.6           | 0.4                | 78         | -0.95    | -0.32                |
| 10               | NR            | NR                 | NR         |          |                      |
| 11               | 0.776         | 0.210              | 94.4       | 0.24     | 0.14                 |
| 12               | 0.913         | 0.137              | 85         | 1.17     | 0.82                 |
| 13               | 0.705         | 0.014              | 87         | -0.24    | -0.22                |
| 15               | <2            | NR                 | 90         |          |                      |
| 16**             | 0.19          | 0.057              | 154        | -3.72    | -3.24                |
| 17               | 0.75          | 0.19               | 89         | 0.07     | 0.04                 |
| 18               | <1            | NR                 | 168        |          |                      |
| 19               | < 1.0         | NR                 | 112        |          |                      |
| 20**             | 0.346         | 0.042              | 75         | -2.66    | -2.38                |
| 21               | <1            | NR                 | 101        |          |                      |

\*\* Not included in robust average calculations, see Section 4.2

**Statistics**

|                       |       |       |
|-----------------------|-------|-------|
| <b>Assigned Value</b> | 0.74  | 0.16  |
| <b>Spike Value</b>    | 0.902 | 0.045 |
| <b>Robust Average</b> | 0.74  | 0.16  |
| <b>Median</b>         | 0.75  | 0.12  |
| <b>Mean</b>           | 0.74  |       |
| <b>N</b>              | 8     |       |
| <b>Max</b>            | 0.986 |       |
| <b>Min</b>            | 0.438 |       |
| <b>Robust SD</b>      | 0.19  |       |
| <b>Robust CV</b>      | 25%   |       |

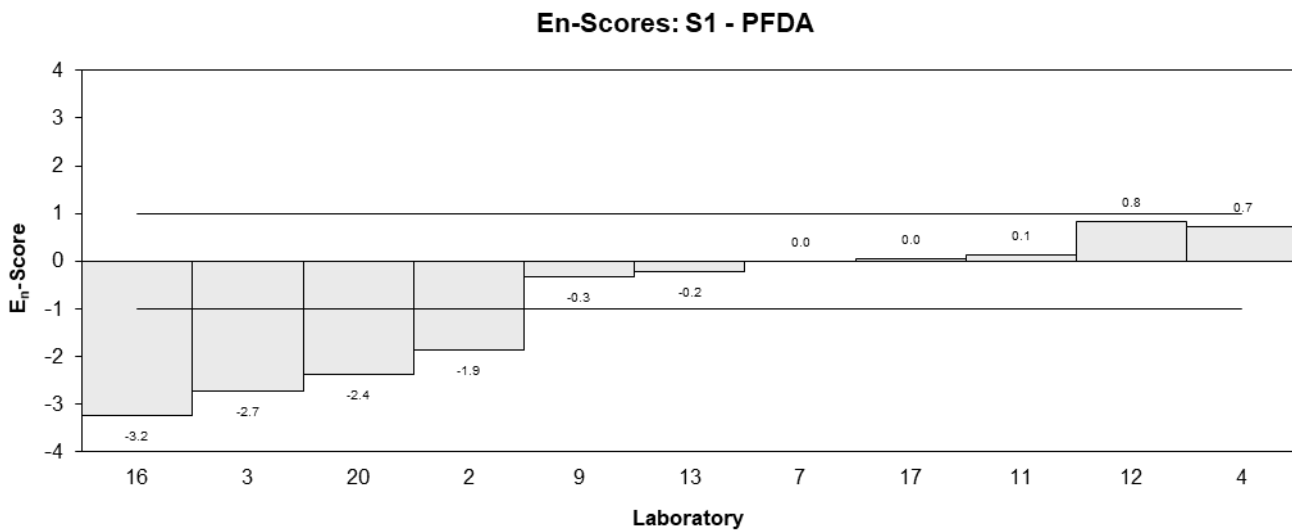
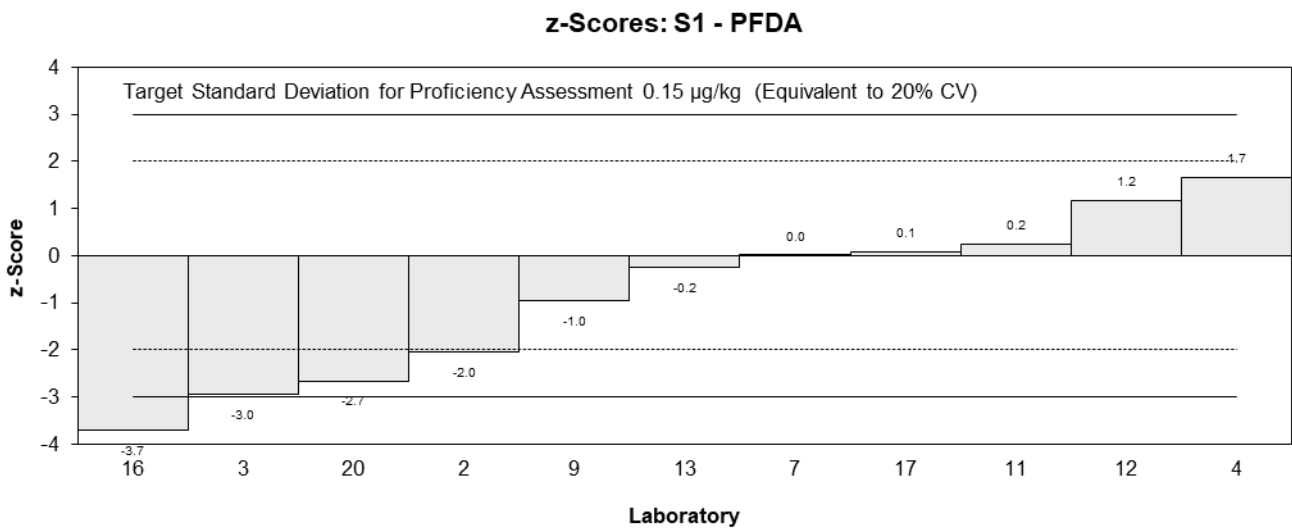
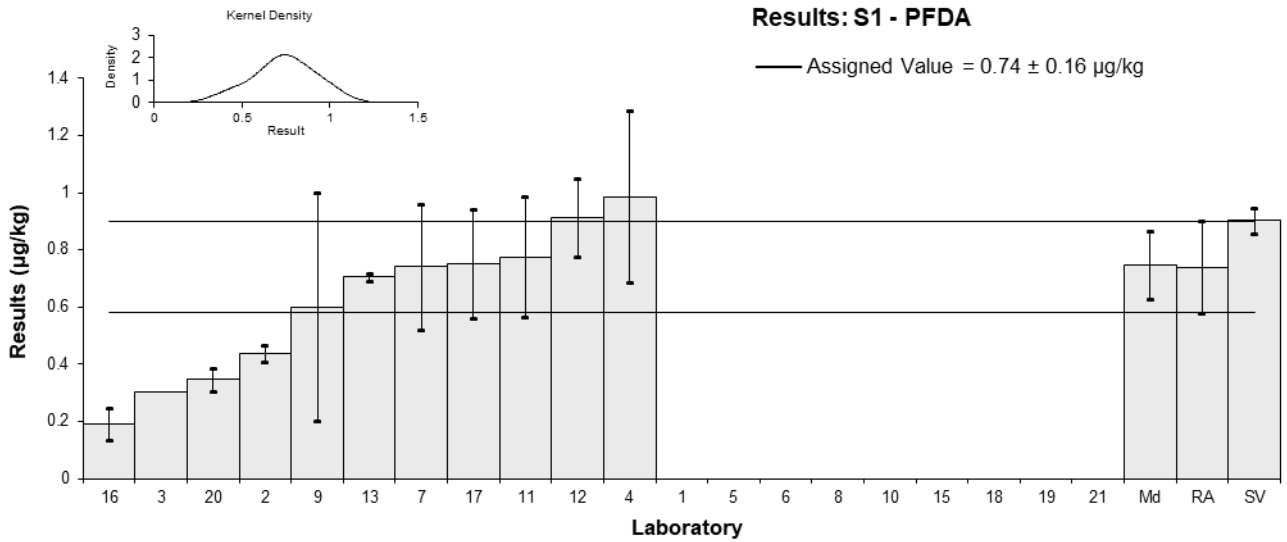


Figure 16

Table 21

## Sample Details

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | PFUdA |
| <b>Unit</b>       | µg/kg |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec   | z     | E <sub>n</sub> |
|-----------|--------|-------------|-------|-------|----------------|
| 1         | < 2    | 1           | 115   |       |                |
| 2         | 0.760  | 0.047       | 66    | -1.61 | -1.23          |
| 3         | NT     | NT          | NT    |       |                |
| 4         | 1.52   | 0.5         | 58    | 1.79  | 0.69           |
| 5         | NS     | NS          | NS    |       |                |
| 6         | <1     | NR          | NT    |       |                |
| 7         | 0.994  | 0.30        | 81    | -0.56 | -0.30          |
| 8**       | 0.63   | 0.19        | 109   | -2.19 | -1.41          |
| 9         | NT     | NT          | NT    |       |                |
| 10        | <1     | NR          | NR    |       |                |
| 11        | 1.05   | 0.427       | 101.1 | -0.31 | -0.14          |
| 12        | 1.43   | 0.243       | 76    | 1.38  | 0.82           |
| 13        | 0.953  | 0.051       | 71    | -0.75 | -0.57          |
| 15        | <2     | NR          | 91    |       |                |
| 16**      | 0.246  | 0.0738      | 184   | -3.90 | -2.92          |
| 17        | 1.1    | 0.26        | 89    | -0.09 | -0.05          |
| 18        | <1     | NR          | 159   |       |                |
| 19        | < 1.0  | NR          | 107   |       |                |
| 20**      | 0.450  | 0.090       | 76    | -2.99 | -2.21          |
| 21        | <1     | NR          | NT    |       |                |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |      |      |
|-----------------------|------|------|
| <b>Assigned Value</b> | 1.12 | 0.29 |
| <b>Spike Value</b>    | 1.21 | 0.06 |
| <b>Robust Average</b> | 1.12 | 0.29 |
| <b>Median</b>         | 1.05 | 0.14 |
| <b>Mean</b>           | 1.12 |      |
| <b>N</b>              | 7    |      |
| <b>Max</b>            | 1.52 |      |
| <b>Min</b>            | 0.76 |      |
| <b>Robust SD</b>      | 0.31 |      |
| <b>Robust CV</b>      | 27%  |      |



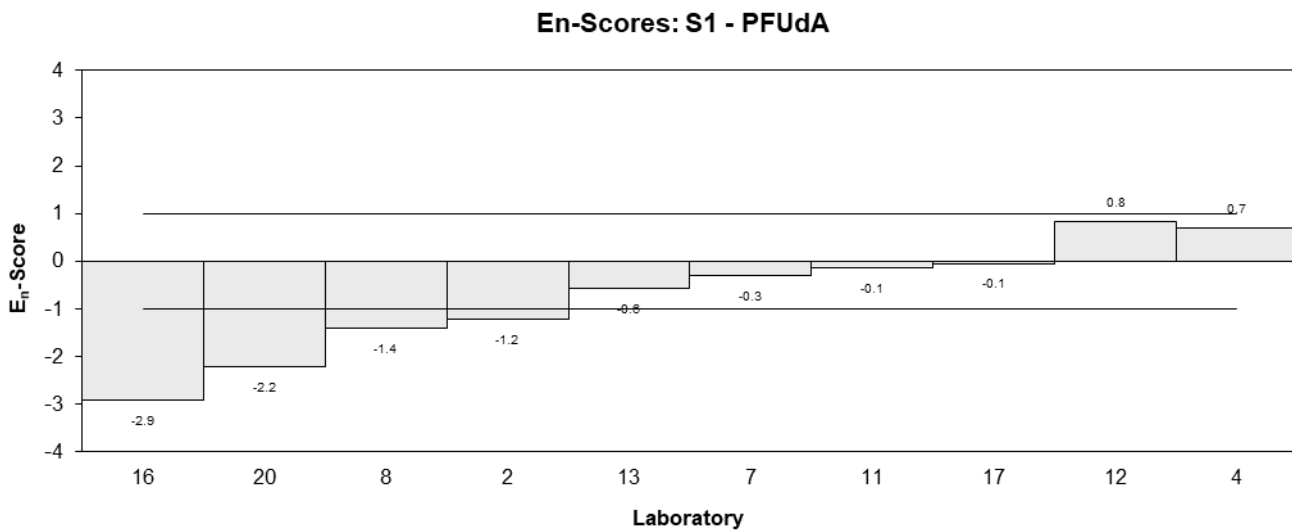
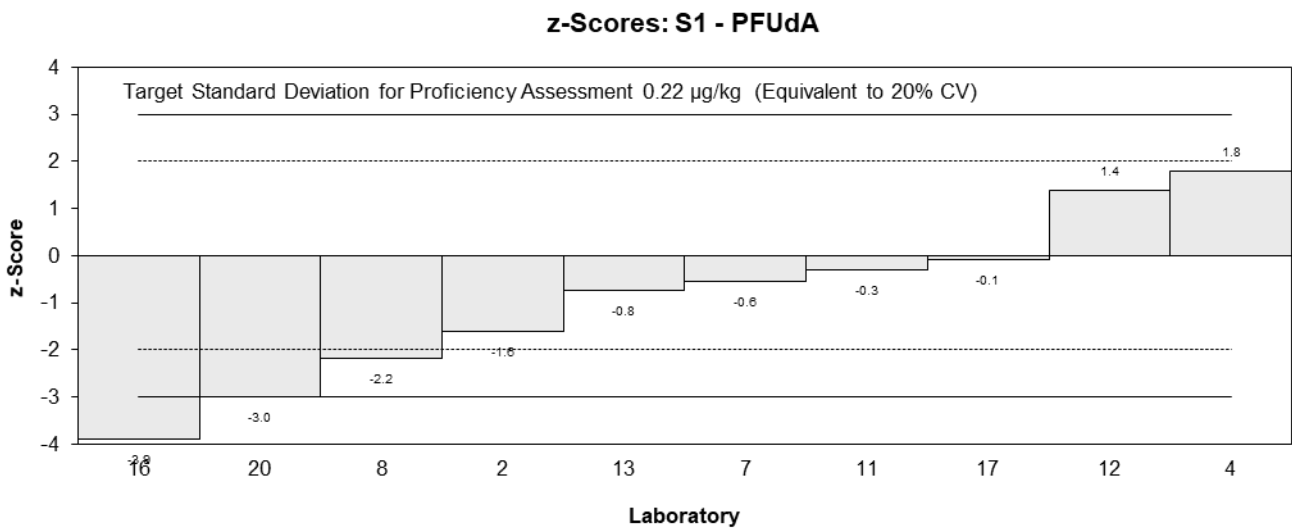
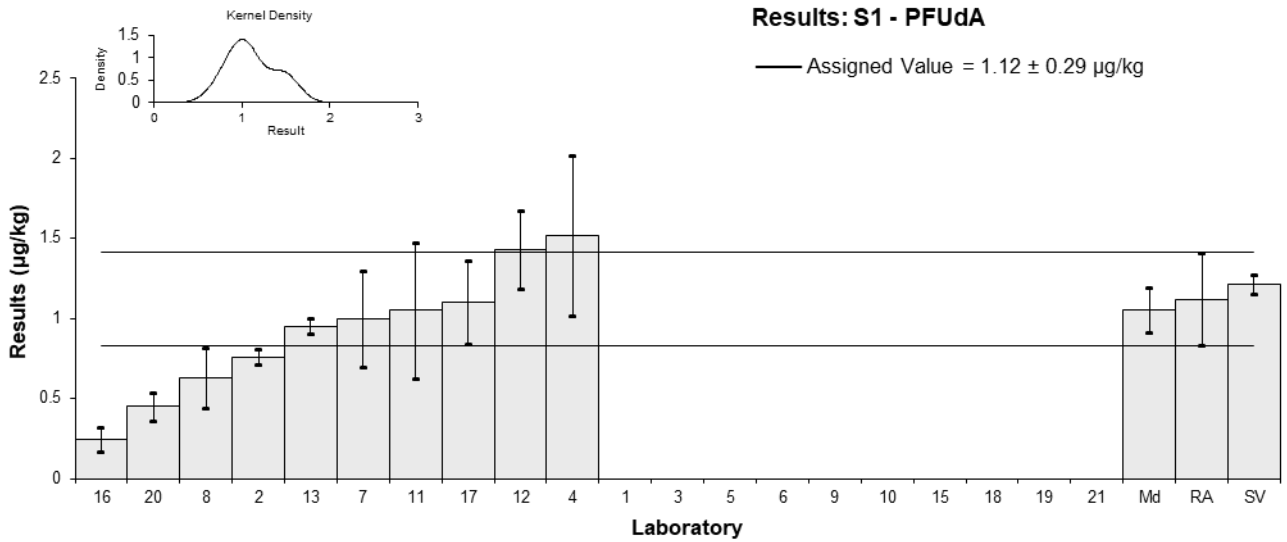


Figure 17

Table 22

**Sample Details**

|                   |         |
|-------------------|---------|
| <b>Sample No.</b> | S1      |
| <b>Matrix</b>     | Prawn   |
| <b>Analyte</b>    | PFTTrDA |
| <b>Unit</b>       | µg/kg   |

**Participant Results**

| <b>Lab. Code</b> | <b>Result</b> | <b>Uncertainty</b> | <b>Rec</b> |
|------------------|---------------|--------------------|------------|
| 1                | 3             | 1.5                | NR         |
| 2                | 2.64          | 0.291              | 56         |
| 3                | NT            | NT                 | NT         |
| 4                | 10.1          | 3                  | NR         |
| 5                | NS            | NS                 | NS         |
| 6                | 5.3           | 0.3                | NT         |
| 7                | 4.75          | 1.4                | 81         |
| 8**              | 4.01          | 2.01               | NR         |
| 9                | NT            | NT                 | NT         |
| 10               | NR            | NR                 | NR         |
| 11               | 4.20          | 1.49               | 69.1       |
| 12               | 9.59          | 2.21               | 62.8       |
| 13               | 5.03          | 1.05               | 32         |
| 15               | 10            | 3                  | 99         |
| 16**             | 1.63          | 0.489              | 129        |
| 17               | 3.7           | 0.92               | 89         |
| 18               | <2            | NR                 | 149        |
| 19               | 3.9           | 0.25               | 103        |
| 20**             | 2.141         | 0.428              | 76         |
| 21               | 2.1           | 0.25               | NT         |

\*\* Not included in robust average calculations, see Section 4.2

**Statistics**

|                       |         |      |
|-----------------------|---------|------|
| <b>Assigned Value</b> | Not Set |      |
| <b>Spike Value</b>    | 8.17    | 0.41 |
| <b>Robust Average</b> | 5.4     | 2.4  |
| <b>Median</b>         | 4.5     | 1.2  |
| <b>Mean</b>           | 5.4     |      |
| <b>N</b>              | 12      |      |
| <b>Max</b>            | 10.1    |      |
| <b>Min</b>            | 2.1     |      |
| <b>Robust SD</b>      | 3.3     |      |
| <b>Robust CV</b>      | 61%     |      |

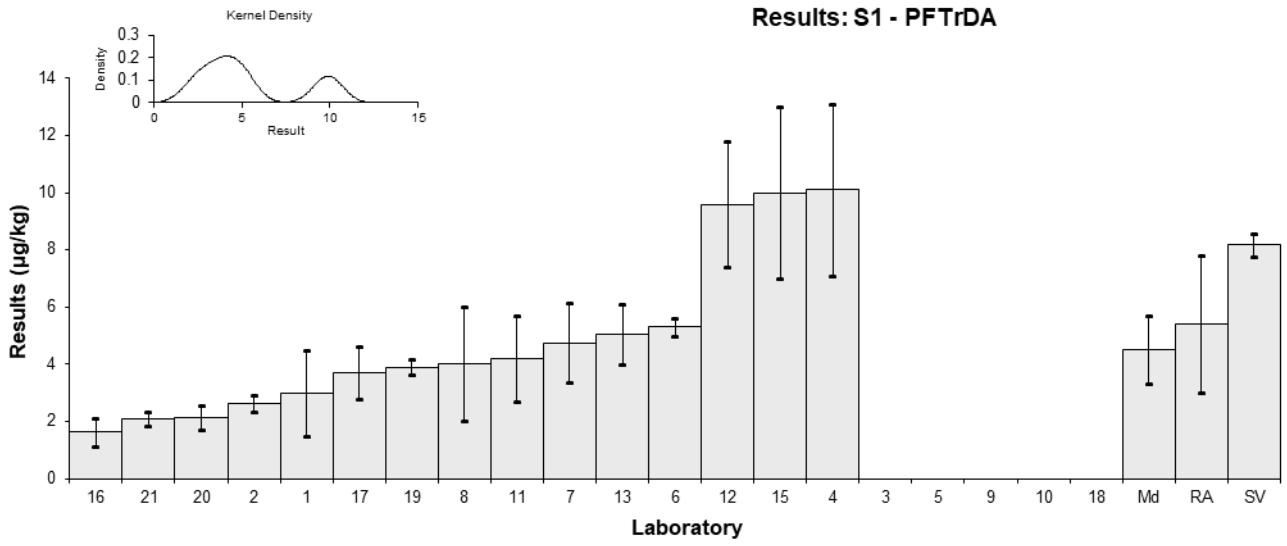


Figure 18

Table 23

**Sample Details**

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | PFOSA |
| <b>Unit</b>       | µg/kg |

**Participant Results**

| <b>Lab. Code</b> | <b>Result</b> | <b>Uncertainty</b> | <b>Rec</b> |
|------------------|---------------|--------------------|------------|
| 1                | 2             | 1                  | 111        |
| 2                | 2.51          | 0.135              | 92         |
| 3                | NT            | NT                 | NT         |
| 4                | 4.29          | 1                  | 76         |
| 5                | NS            | NS                 | NS         |
| 6                | 3.2           | 0.2                | NT         |
| 7                | 2.41          | 0.72               | 83         |
| 8**              | 1.72          | 0.43               | 89         |
| 9                | NT            | NT                 | NT         |
| 10               | NR            | NR                 | NR         |
| 11               | 3.03          | 0.563              | 19.5       |
| 12               | 4.37          | 0.612              | 112        |
| 13               | 3.16          | 0.227              | 76         |
| 15               | 5             | 2                  | 92         |
| 16**             | 0.844         | 0.2532             | 105        |
| 17               | 2.4           | 0.59               | 89         |
| 18               | <5            | NR                 | 114        |
| 19               | 2.4           | 0.32               | 105        |
| 20               | NT            | NT                 | NT         |
| 21               | 1.6           | 0.2                | NT         |

\*\* Not included in robust average calculations, see Section 4.2

**Statistics**

|                       |         |      |
|-----------------------|---------|------|
| <b>Assigned Value</b> | Not Set |      |
| <b>Spike Value</b>    | 4.46    | 0.22 |
| <b>Robust Average</b> | 3.00    | 0.81 |
| <b>Median</b>         | 2.77    | 0.44 |
| <b>Mean</b>           | 3.03    |      |
| <b>N</b>              | 12      |      |
| <b>Max</b>            | 5       |      |
| <b>Min</b>            | 1.6     |      |
| <b>Robust SD</b>      | 1.1     |      |
| <b>Robust CV</b>      | 37%     |      |

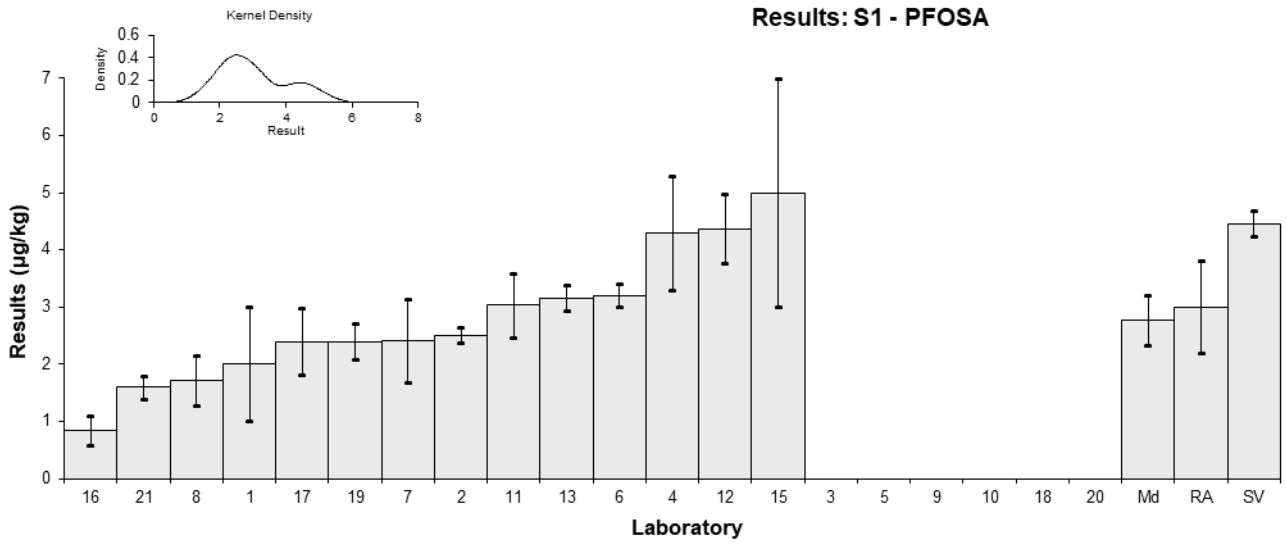


Figure 19

Table 24

**Sample Details**

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S1     |
| <b>Matrix</b>     | Prawn  |
| <b>Analyte</b>    | MeFOSA |
| <b>Unit</b>       | µg/kg  |

**Participant Results**

| <b>Lab. Code</b> | <b>Result</b> | <b>Uncertainty</b> | <b>Rec</b> |
|------------------|---------------|--------------------|------------|
| 1                | < 5           | 2.5                | 110        |
| 2                | NT            | NT                 | NT         |
| 3                | NT            | NT                 | NT         |
| 4                | 5.26          | 2                  | 6          |
| 5                | NS            | NS                 | NS         |
| 6                | 5.1           | 0.3                | NT         |
| 7                | 2.84          | 0.85               | 75         |
| 8**              | 2.40          | 0.60               | 115        |
| 9                | NT            | NT                 | NT         |
| 10               | NR            | NR                 | NR         |
| 11               | 2.21          | 0.478              | 19.5       |
| 12               | 4.72          | 1.18               | 56.9       |
| 13               | 3.59          | 1.28               | 19         |
| 15               | 5             | 2                  | 88         |
| 16**             | 1.32          | 0.396              | 144        |
| 17               | 3.3           | 0.81               | 89         |
| 18               | <5            | NR                 | 194        |
| 19               | 3.0           | 0.47               | 115        |
| 20**             | NT            | NT                 | NT         |
| 21               | 2.1           | 0.25               | NT         |

\*\* Not included in robust average calculations, see Section 4.2

**Statistics**

|                       |         |      |
|-----------------------|---------|------|
| <b>Assigned Value</b> | Not Set |      |
| <b>Spike Value</b>    | 4.99    | 0.25 |
| <b>Robust Average</b> | 3.7     | 1.1  |
| <b>Median</b>         | 3.4     | 1.5  |
| <b>Mean</b>           | 3.71    |      |
| <b>N</b>              | 10      |      |
| <b>Max</b>            | 5.26    |      |
| <b>Min</b>            | 2.1     |      |
| <b>Robust SD</b>      | 1.4     |      |
| <b>Robust CV</b>      | 37%     |      |

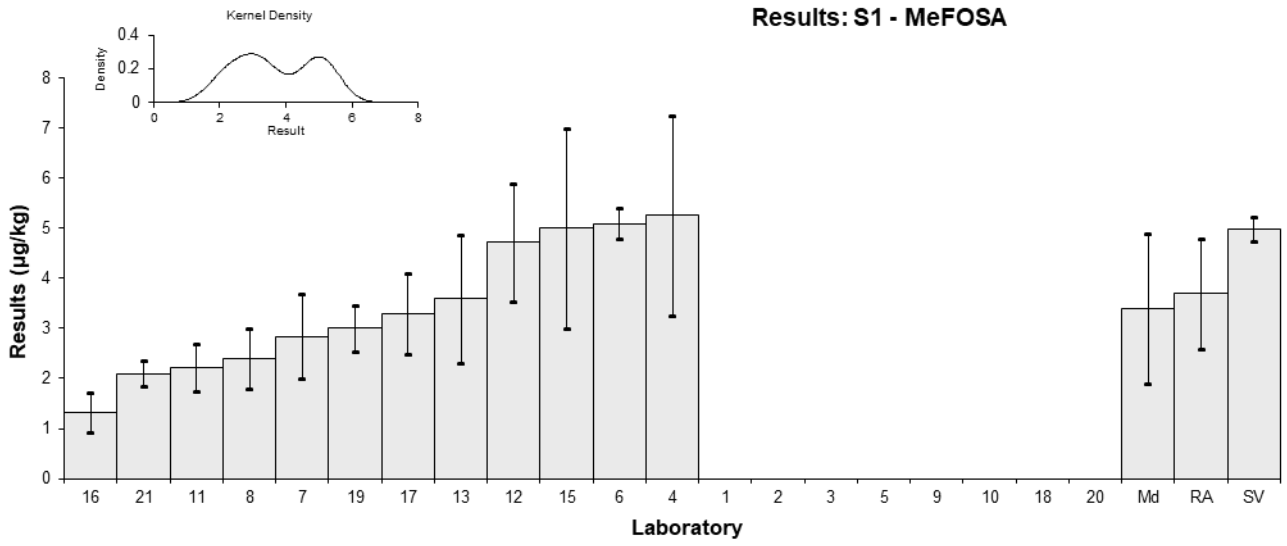


Figure 20

Table 25

**Sample Details**

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S1     |
| <b>Matrix</b>     | Prawn  |
| <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                | 115        |
| 2                | NT            | NT                 | NT         |
| 3                | NT            | NT                 | NT         |
| 4                | <5            | NR                 | 2          |
| 5                | NS            | NS                 | NS         |
| 6                | 5.1           | 0.3                | NT         |
| 7                | 1.64          | 0.49               | 73         |
| 8**              | 1.88          | 0.56               | 126        |
| 9                | NT            | NT                 | NT         |
| 10               | NR            | NR                 | NR         |
| 11               | 2.70          | 0.453              | 19.5       |
| 12               | 3.84          | 0.614              | 58.8       |
| 13               | 2.7           | 0.313              | 11         |
| 15               | <5            | NR                 | 90         |
| 16**             | 1.34          | 0.402              | 98         |
| 17               | 2.2           | 0.54               | 89         |
| 18               | <2            | NR                 | 100        |
| 19               | 2.3           | 0.50               | 112        |
| 20               | NT            | NT                 | NT         |
| 21               | 1.6           | 0.2                | NT         |

\*\* Not included in robust average calculations, see Section 4.2

**Statistics**

|                       |         |      |
|-----------------------|---------|------|
| <b>Assigned Value</b> | Not Set |      |
| <b>Spike Value</b>    | 3.99    | 0.20 |
| <b>Robust Average</b> | 2.66    | 0.98 |
| <b>Median</b>         | 2.50    | 0.76 |
| <b>Mean</b>           | 2.76    |      |
| <b>N</b>              | 8       |      |
| <b>Max</b>            | 5.1     |      |
| <b>Min</b>            | 1.6     |      |
| <b>Robust SD</b>      | 1.1     |      |
| <b>Robust CV</b>      | 41%     |      |



Results: S1 - EtFOSA

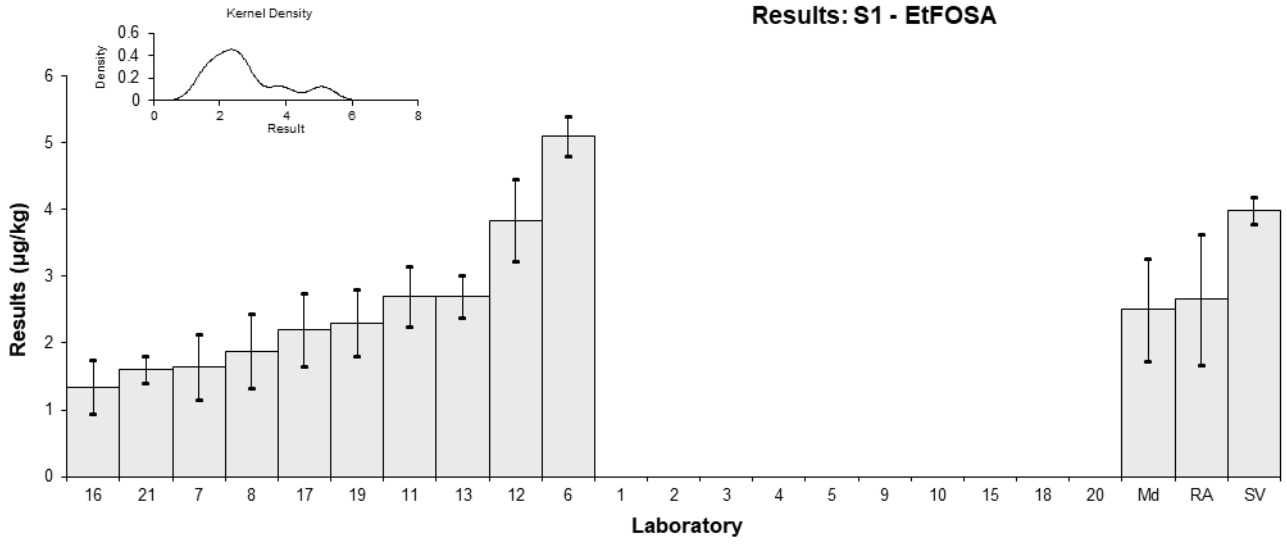


Figure 21

Table 26

**Sample Details**

|                   |       |
|-------------------|-------|
| <b>Sample No.</b> | S1    |
| <b>Matrix</b>     | Prawn |
| <b>Analyte</b>    | ADONA |
| <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                | 3.69          | 0.183              | 76         | -0.90    | -0.67                |
| 3**              | 3.013         | NR                 | NR         | -1.65    | -1.24                |
| 4*               | 7.31          | 2                  | NR         | 3.12     | 1.20                 |
| 5                | NS            | NS                 | NS         |          |                      |
| 6                | NT            | NT                 | NT         |          |                      |
| 7                | 3.48          | 1.1                | 87         | -1.13    | -0.63                |
| 8**              | 3.25          | 1.30               | NR         | -1.39    | -0.71                |
| 9                | NT            | NT                 | NT         |          |                      |
| 10               | NR            | NR                 | NR         |          |                      |
| 11               | NT            | NT                 | NT         |          |                      |
| 12               | 6.04          | 1.63               | 78.7       | 1.71     | 0.76                 |
| 13               | 4.11          | 0.099              | 110        | -0.43    | -0.32                |
| 15               | 6             | 2                  | 91         | 1.67     | 0.64                 |
| 16               | NR            | NR                 | NR         |          |                      |
| 17               | 3.6           | 0.92               | 89         | -1.00    | -0.60                |
| 18               | NT            | NT                 | NT         |          |                      |
| 19               | 4.4           | 0.46               | 119        | -0.11    | -0.08                |
| 20               | NT            | NT                 | NT         |          |                      |
| 21               | NT            | NT                 | NT         |          |                      |

\* Outlier, \*\* Not included in robust average calculations, see Section 4.2

**Statistics**

|                       |      |      |
|-----------------------|------|------|
| <b>Assigned Value</b> | 4.5  | 1.2  |
| <b>Spike Value</b>    | 5.64 | 0.28 |
| <b>Robust Average</b> | 4.8  | 1.4  |
| <b>Median</b>         | 4.26 | 0.94 |
| <b>Mean</b>           | 4.8  |      |
| <b>N</b>              | 8    |      |
| <b>Max</b>            | 7.31 |      |
| <b>Min</b>            | 3.48 |      |
| <b>Robust SD</b>      | 1.6  |      |
| <b>Robust CV</b>      | 33%  |      |

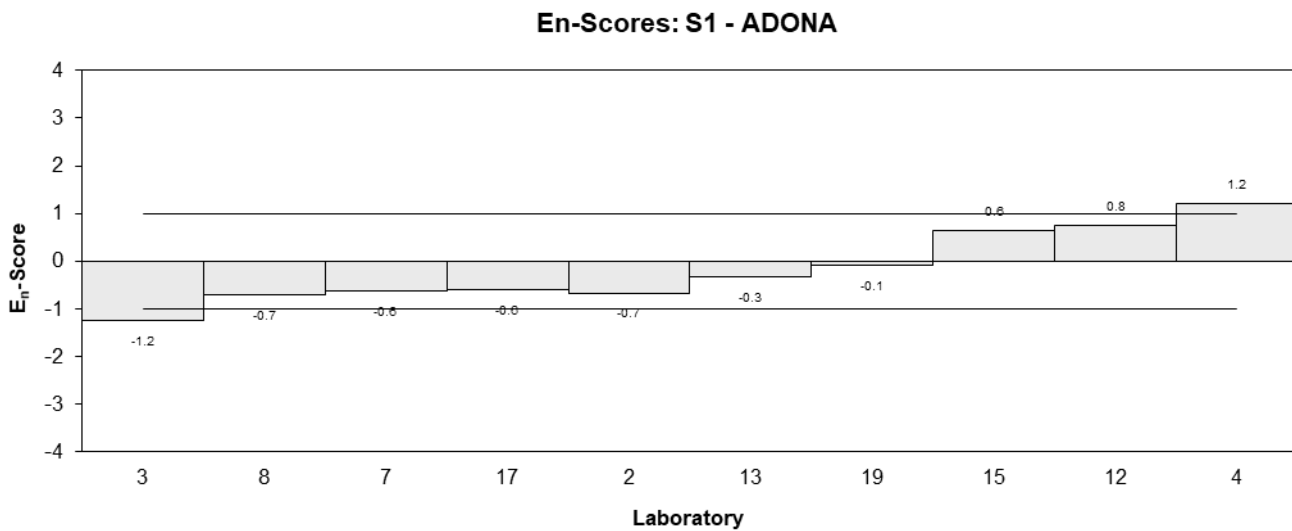
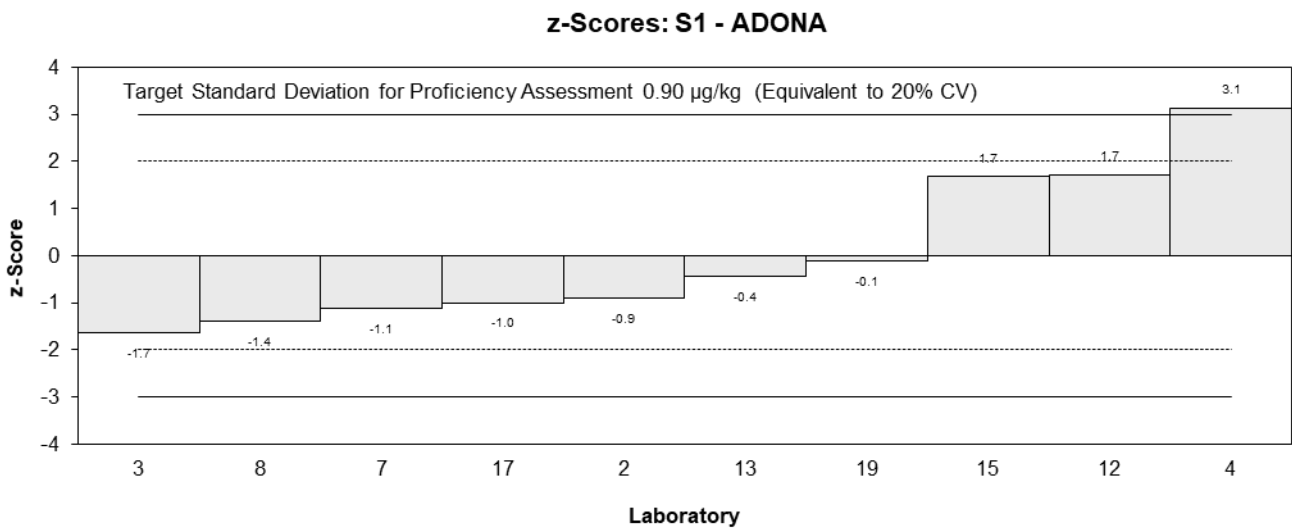
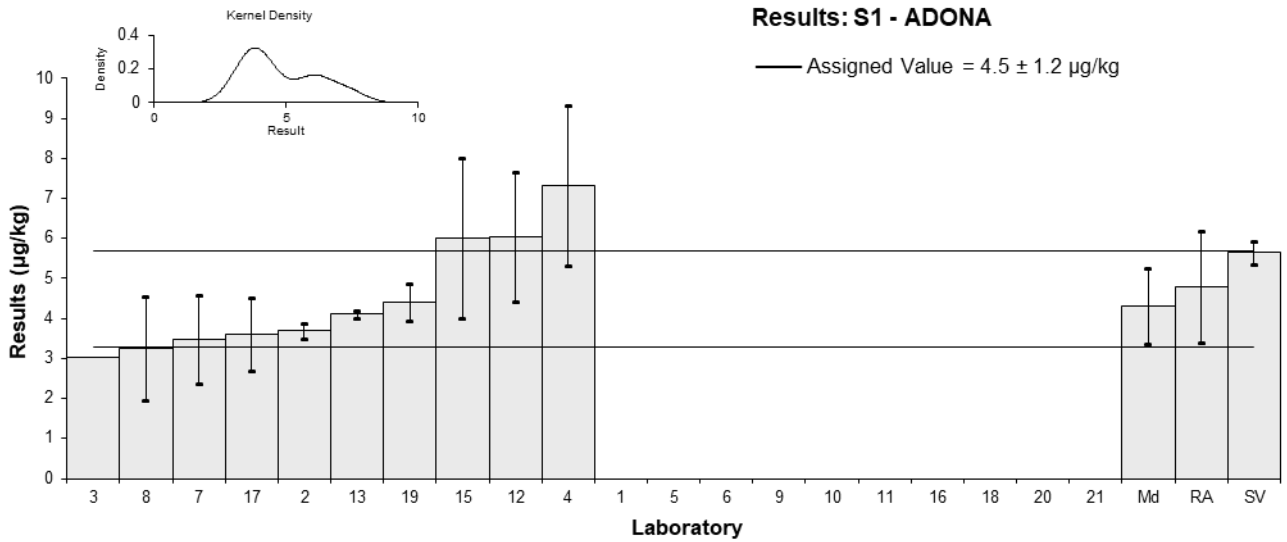


Figure 22

Table 27

## Sample Details

|                   |            |
|-------------------|------------|
| <b>Sample No.</b> | S1         |
| <b>Matrix</b>     | Prawn      |
| <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         | 7.47   | 0.673       | 97   | -1.60 | -1.53          |
| 3**       | 6.268  | NR          | NR   | -2.15 | -2.15          |
| 4         | 12.6   | 5           | NR   | 0.73  | 0.29           |
| 5         | NS     | NS          | NS   |       |                |
| 6         | NT     | NT          | NT   |       |                |
| 7         | 12.1   | 3.6         | 82   | 0.50  | 0.26           |
| 8**       | 5.88   | 1.47        | NR   | -2.33 | -1.94          |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NR     | NR          | NR   |       |                |
| 11        | NT     | NT          | NT   |       |                |
| 12        | 12.7   | 3.55        | 78.7 | 0.77  | 0.41           |
| 13        | 10.1   | 0.692       | 104  | -0.41 | -0.39          |
| 15        | 13     | 4           | 91   | 0.91  | 0.44           |
| 16        | NR     | NR          | NR   |       |                |
| 17        | 12     | 6.4         | 89   | 0.45  | 0.15           |
| 18        | NT     | NT          | NT   |       |                |
| 19        | 8.3    | 1.6         | 102  | -1.23 | -0.99          |
| 20        | NT     | NT          | NT   |       |                |
| 21        | NT     | NT          | NT   |       |                |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |      |     |
|-----------------------|------|-----|
| <b>Assigned Value</b> | 11.0 | 2.2 |
| <b>Spike Value</b>    | 14.4 | 0.7 |
| <b>Robust Average</b> | 11.0 | 2.2 |
| <b>Median</b>         | 12.1 | 1.0 |
| <b>Mean</b>           | 11.0 |     |
| <b>N</b>              | 8    |     |
| <b>Max</b>            | 13   |     |
| <b>Min</b>            | 7.47 |     |
| <b>Robust SD</b>      | 2.4  |     |
| <b>Robust CV</b>      | 22%  |     |

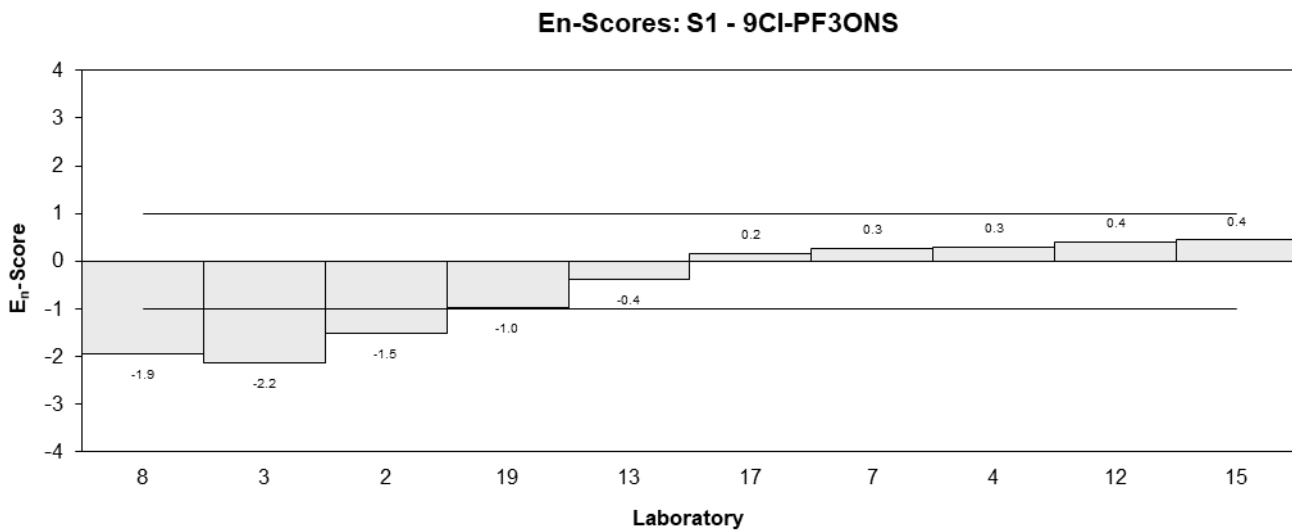
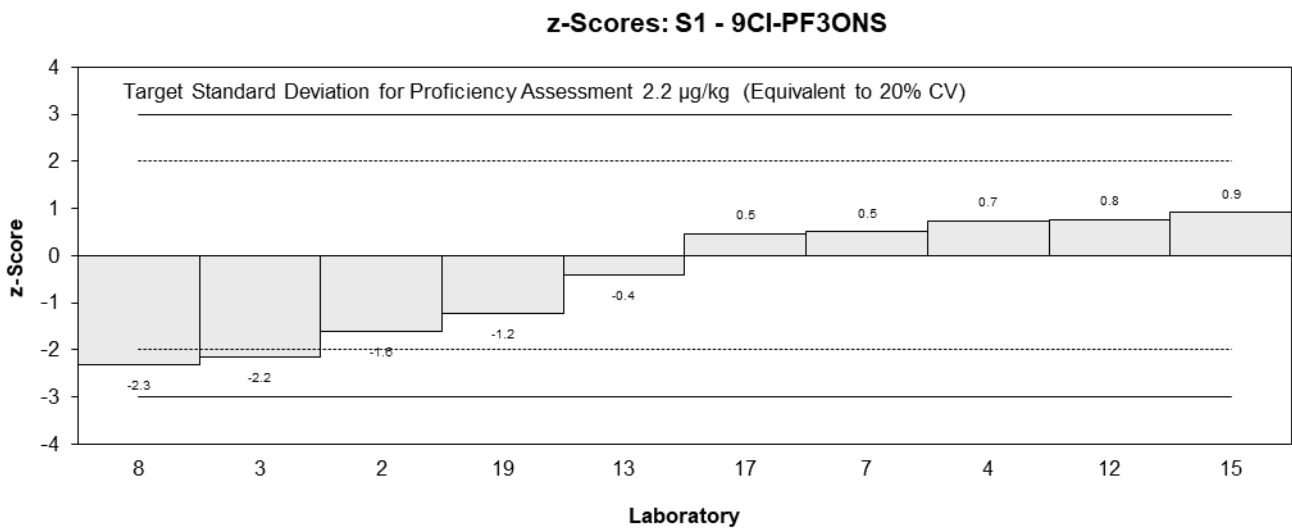
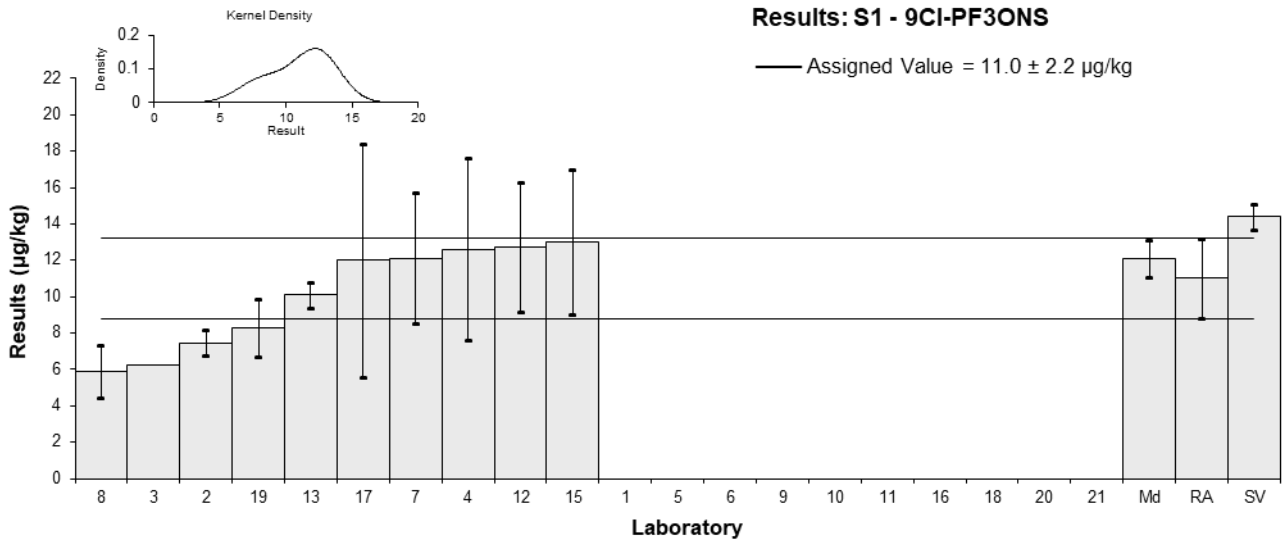


Figure 23

Table 28

## Sample Details

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

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  |
|-----------|--------|-------------|------|
| 1         | NT     | NT          | NT   |
| 2         | 1.56   | 0.150       | 97   |
| 3**       | 1.522  | NR          | NR   |
| 4         | 2.94   | 1           | NR   |
| 5         | NS     | NS          | NS   |
| 6         | NT     | NT          | NT   |
| 7         | 2.67   | 0.80        | 82   |
| 8**       | 1.09   | 0.33        | NR   |
| 9         | NT     | NT          | NT   |
| 10        | NR     | NR          | NR   |
| 11        | NT     | NT          | NT   |
| 12        | 2.83   | 0.932       | 78.7 |
| 13        | 0.82   | 0.356       | 104  |
| 15        | 3      | 2           | 91   |
| 16        | NR     | NR          | NR   |
| 17        | 2.2    | 0.56        | 89   |
| 18        | NT     | NT          | NT   |
| 19        | 1.9    | 0.32        | 103  |
| 20        | NT     | NT          | NT   |
| 21        | NT     | NT          | NT   |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |         |      |
|-----------------------|---------|------|
| <b>Assigned Value</b> | Not Set |      |
| <b>Spike Value</b>    | 4.70    | 0.24 |
| <b>Robust Average</b> | 2.27    | 0.72 |
| <b>Median</b>         | 2.44    | 0.68 |
| <b>Mean</b>           | 2.24    |      |
| <b>N</b>              | 8       |      |
| <b>Max</b>            | 3       |      |
| <b>Min</b>            | 0.82    |      |
| <b>Robust SD</b>      | 0.81    |      |
| <b>Robust CV</b>      | 36%     |      |

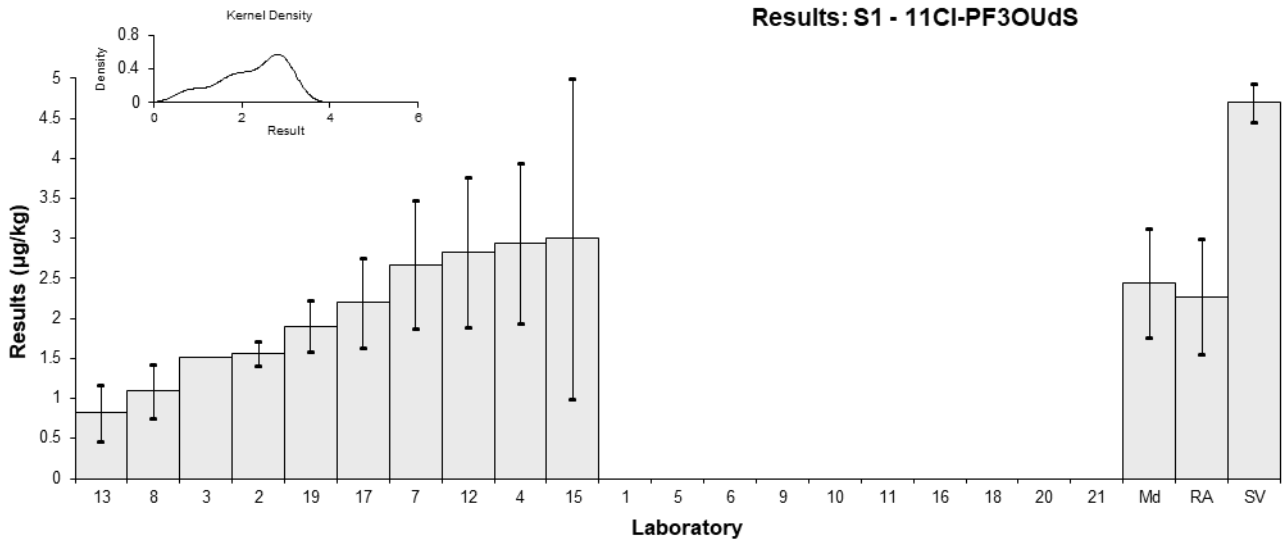


Figure 24

Table 29

**Sample Details**

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFBS   |
| <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                | 0.7           | 0.35               | 95         | -0.73    | -0.33                |
| 2                | 0.625         | 0.043              | 108        | -1.19    | -1.65                |
| 3                | NS            | NS                 | NS         |          |                      |
| 4                | 0.957         | 0.3                | 77         | 0.84     | 0.43                 |
| 5                | 0.99          | 0.54               | NR         | 1.04     | 0.31                 |
| 6                | <1            | NR                 | NT         |          |                      |
| 7                | 0.873         | 0.26               | 96         | 0.32     | 0.19                 |
| 8                | NS            | NS                 | NS         |          |                      |
| 9                | NT            | NT                 | NT         |          |                      |
| 10               | NS            | NS                 | NS         |          |                      |
| 11               | 0.709         | 0.170              | 86.4       | -0.68    | -0.55                |
| 12               | 0.787         | 0.11               | 91.2       | -0.20    | -0.21                |
| 13               | 0.782         | 0.025              | 145        | -0.23    | -0.34                |
| 15               | <1            | NR                 | 87         |          |                      |
| 16**             | 0.238         | 0.0714             | 151        | -3.55    | -4.44                |
| 17               | 0.89          | 0.22               | 94         | 0.43     | 0.28                 |
| 18               | <1            | NR                 | 128        |          |                      |
| 19               | NR            | NR                 | 103        |          |                      |
| 20               | 0.853         | 0.171              | 61         | 0.20     | 0.16                 |
| 21               | <1            | NR                 | NT         |          |                      |

\*\* Not included in robust average calculations, see Section 4.2

**Statistics**

|                          |       |       |
|--------------------------|-------|-------|
| <b>Assigned Value</b>    | 0.82  | 0.11  |
| <b>Spike Value</b>       | 0.891 | 0.045 |
| <b>Homogeneity Value</b> | 0.83  | 0.25  |
| <b>Robust Average</b>    | 0.82  | 0.11  |
| <b>Median</b>            | 0.82  | 0.11  |
| <b>Mean</b>              | 0.817 |       |
| <b>N</b>                 | 10    |       |
| <b>Max</b>               | 0.99  |       |
| <b>Min</b>               | 0.625 |       |
| <b>Robust SD</b>         | 0.13  |       |
| <b>Robust CV</b>         | 16%   |       |



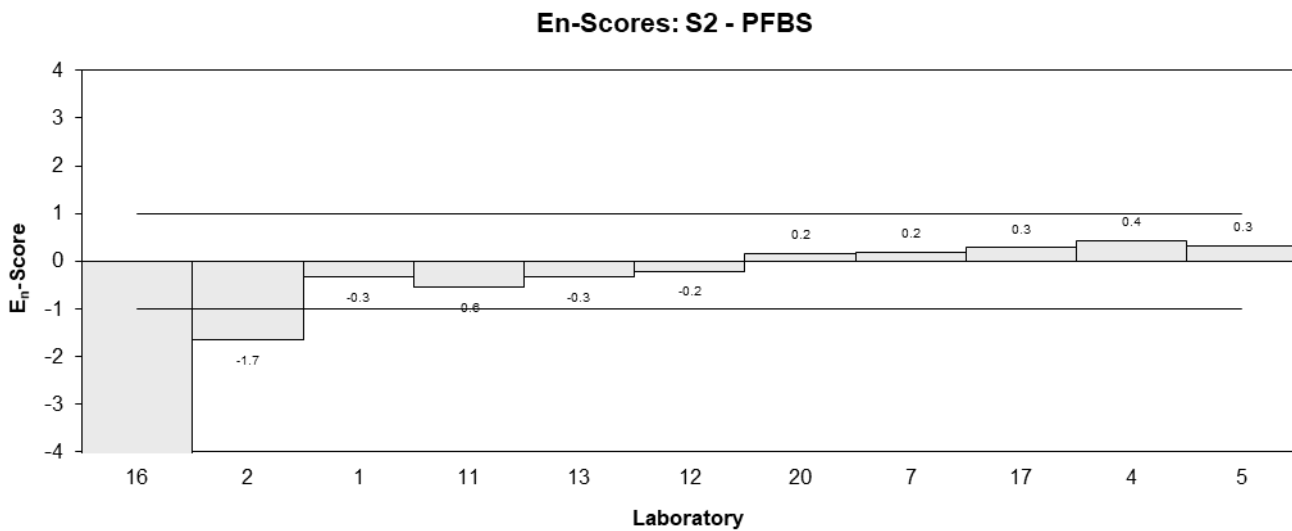
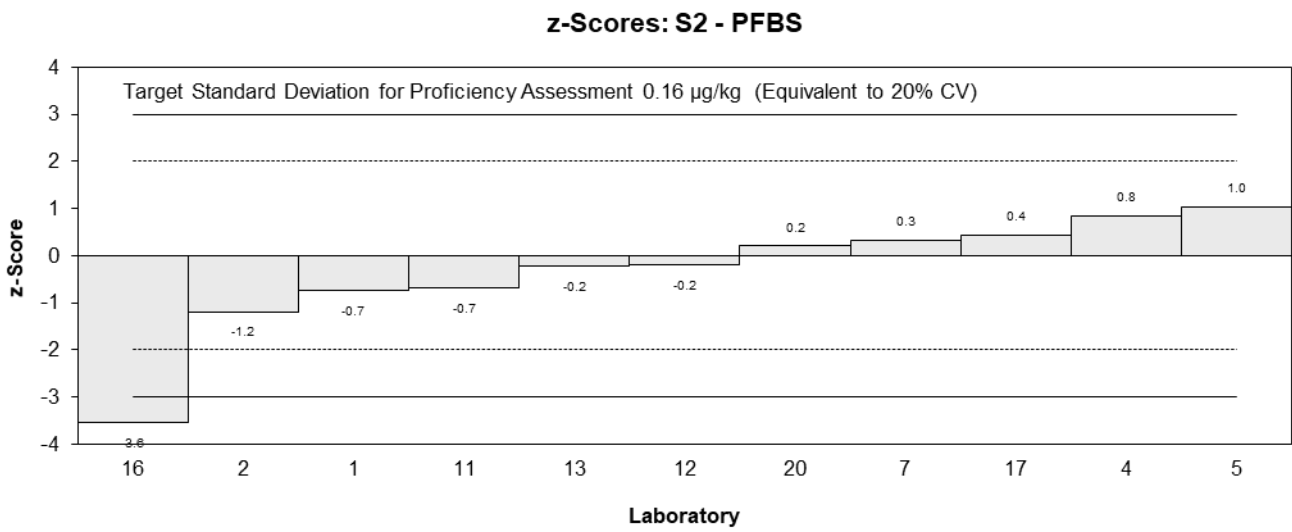
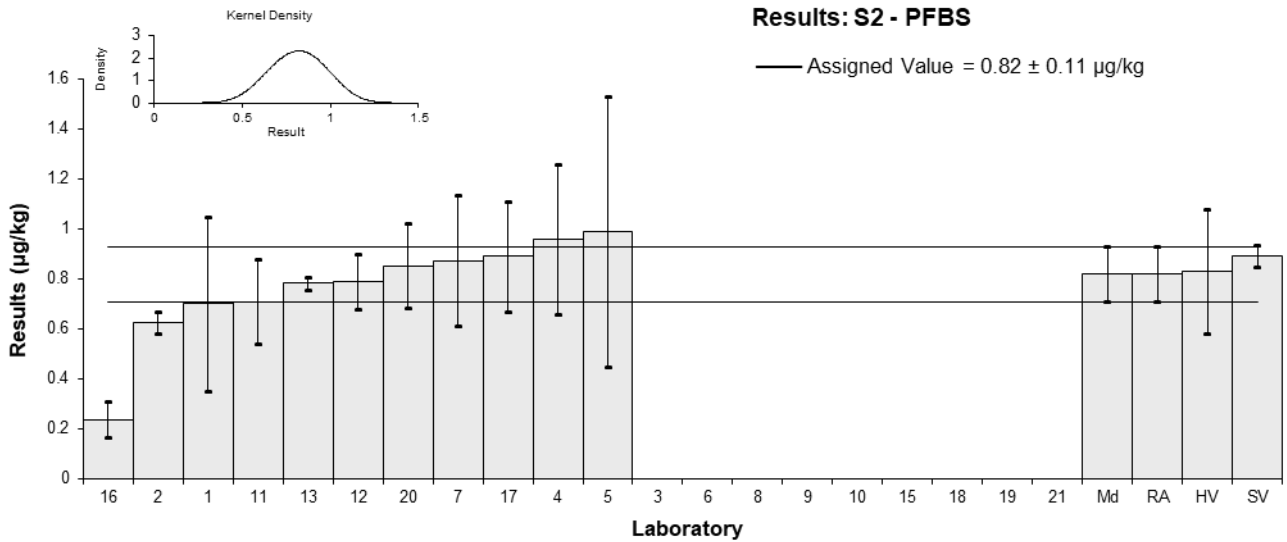


Figure 25

Table 30

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFPeS  |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 7.5    | 3.8         | NR   | -0.48 | -0.21          |
| 2         | 6.65   | 0.572       | 108  | -0.99 | -1.75          |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 8.47   | 3           | NR   | 0.10  | 0.05           |
| 5         | NT     | NT          | NT   |       |                |
| 6         | 9.7    | 0.5         | NT   | 0.84  | 1.55           |
| 7         | 8.68   | 2.6         | 85   | 0.23  | 0.14           |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | 8.04   | 2.28        | 86.4 | -0.16 | -0.11          |
| 12        | 8.78   | 0.966       | 87.6 | 0.29  | 0.39           |
| 13        | 8.08   | 0.347       | 117  | -0.13 | -0.27          |
| 15        | 9      | 3           | 88   | 0.42  | 0.23           |
| 16**      | 4.09   | 1.227       | 151  | -2.54 | -2.93          |
| 17        | 7.7    | 1.9         | 94   | -0.36 | -0.29          |
| 18        | 9.86   | 3.3         | 128  | 0.94  | 0.46           |
| 19        | 9.1    | 1.6         | 93   | 0.48  | 0.45           |
| 20        | 6.17   | 1.23        | 142  | -1.28 | -1.48          |
| 21        | 8.0    | 0.4         | NT   | -0.18 | -0.35          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 8.30 | 0.75 |
| <b>Spike Value</b>       | 7.47 | 0.37 |
| <b>Homogeneity Value</b> | 8.3  | 2.5  |
| <b>Robust Average</b>    | 8.30 | 0.75 |
| <b>Median</b>            | 8.28 | 0.64 |
| <b>Mean</b>              | 8.27 |      |
| <b>N</b>                 | 14   |      |
| <b>Max</b>               | 9.86 |      |
| <b>Min</b>               | 6.17 |      |
| <b>Robust SD</b>         | 1.1  |      |
| <b>Robust CV</b>         | 13%  |      |

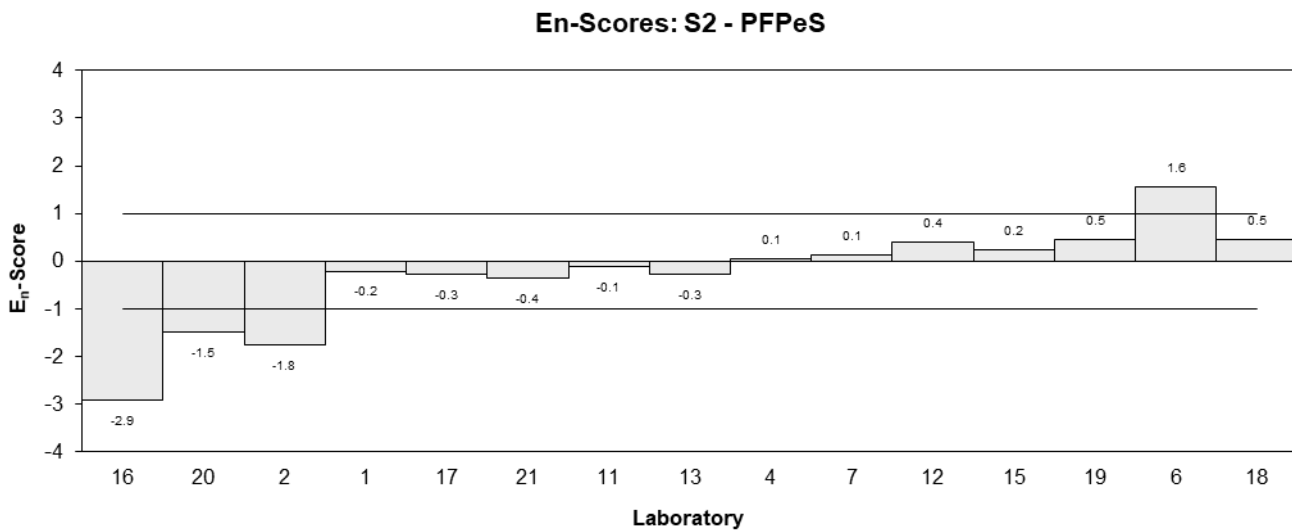
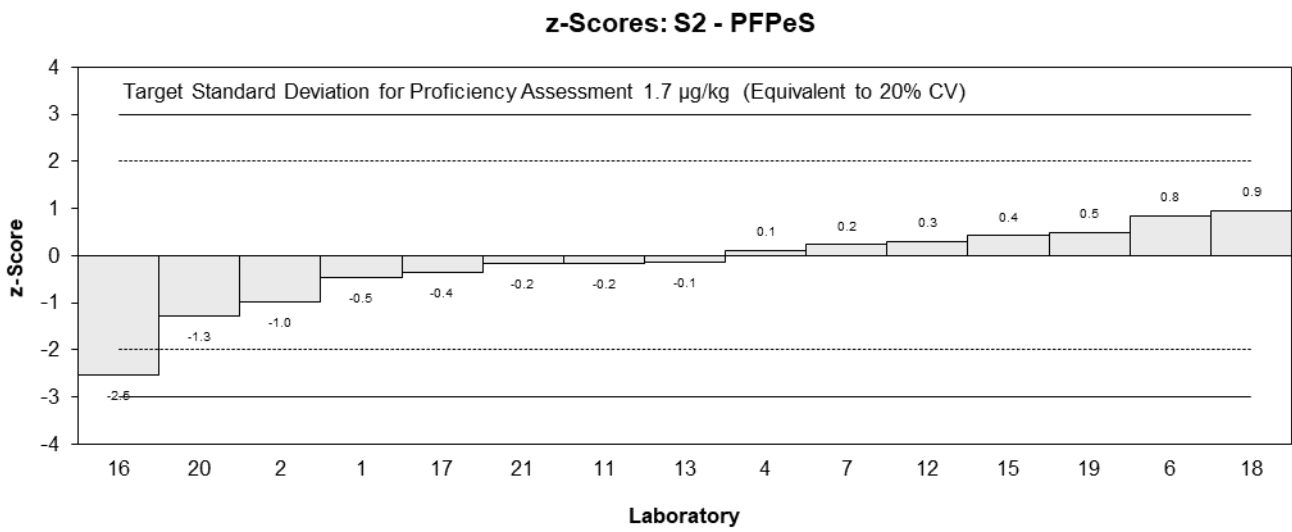
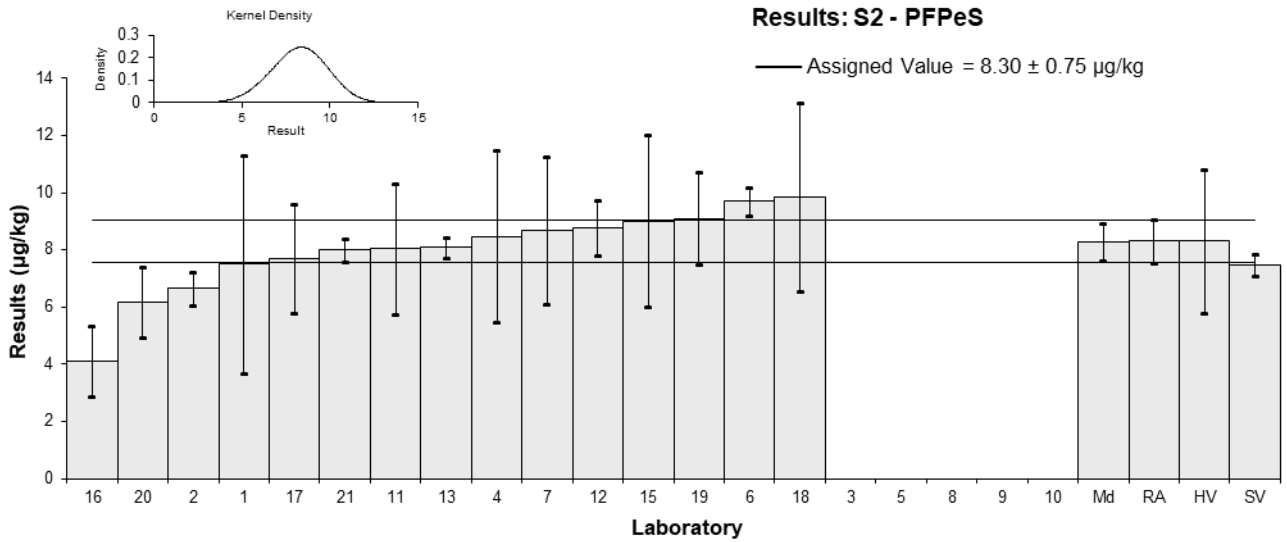


Figure 26

Table 31

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <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         | 98   | -0.72 | -0.33          |
| 2         | NR     | NR          | NR   |       |                |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 7.44   | 2           | 81   | 0.69  | 0.44           |
| 5         | NT     | NT          | NT   |       |                |
| 6         | 6.6    | 0.35        | NT   | 0.05  | 0.10           |
| 7         | 5.88   | 1.8         | 92   | -0.50 | -0.35          |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | NT     | NT          | NT   |       |                |
| 12        | 6.01   | 0.842       | 87.6 | -0.41 | -0.54          |
| 13        | 6.36   | 0.289       | 117  | -0.14 | -0.31          |
| 15        | 7      | 3           | 88   | 0.35  | 0.15           |
| 16**      | 2.07   | 0.621       | 134  | -3.42 | -5.56          |
| 17        | 7.1    | 1.8         | 94   | 0.43  | 0.30           |
| 18        | 7.3    | 2.5         | 136  | 0.58  | 0.30           |
| 19        | 6.8    | 1.6         | 106  | 0.20  | 0.15           |
| 20        | 6.70   | 1.34        | 142  | 0.12  | 0.11           |
| 21        | 5.7    | 0.3         | NT   | -0.64 | -1.42          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 6.54 | 0.51 |
| <b>Spike Value</b>       | 6.61 | 0.33 |
| <b>Homogeneity Value</b> | 5.9  | 1.8  |
| <b>Robust Average</b>    | 6.54 | 0.51 |
| <b>Median</b>            | 6.65 | 0.58 |
| <b>Mean</b>              | 6.54 |      |
| <b>N</b>                 | 12   |      |
| <b>Max</b>               | 7.44 |      |
| <b>Min</b>               | 5.6  |      |
| <b>Robust SD</b>         | 0.71 |      |
| <b>Robust CV</b>         | 11%  |      |

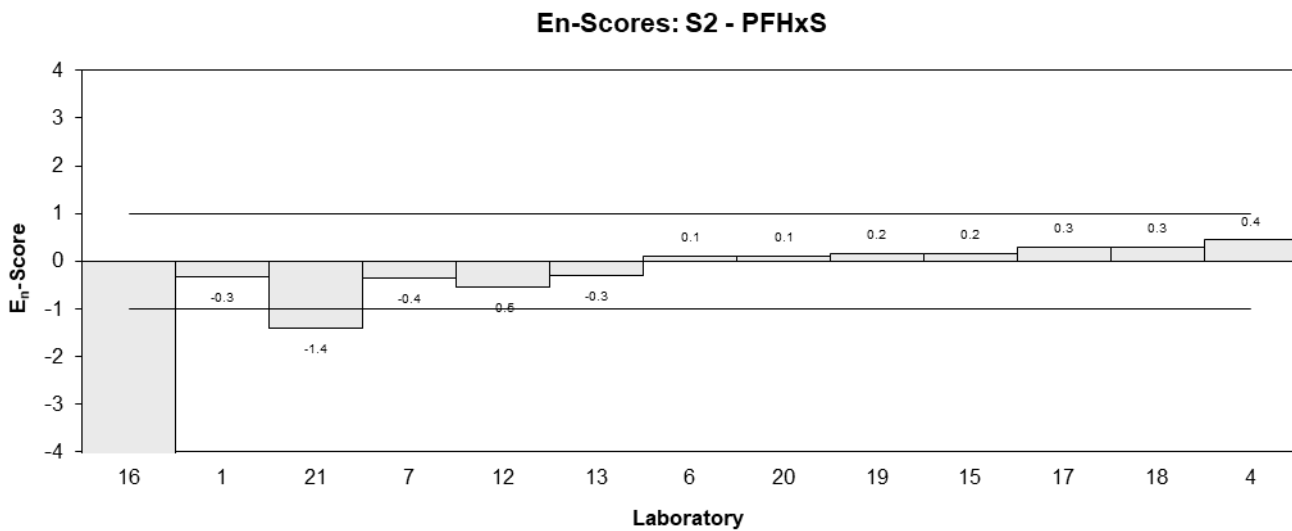
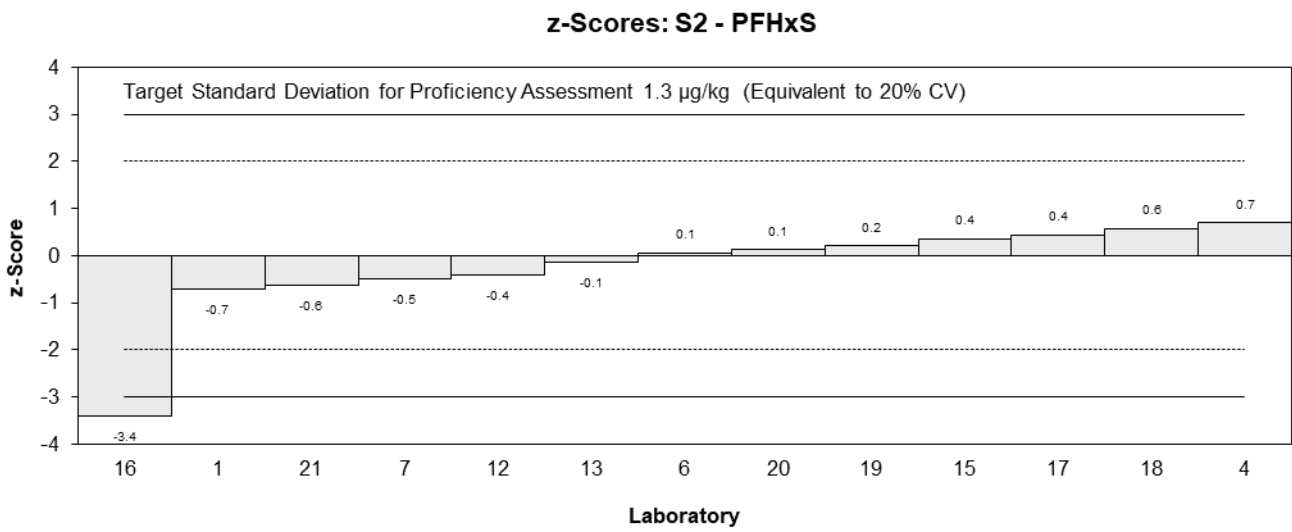
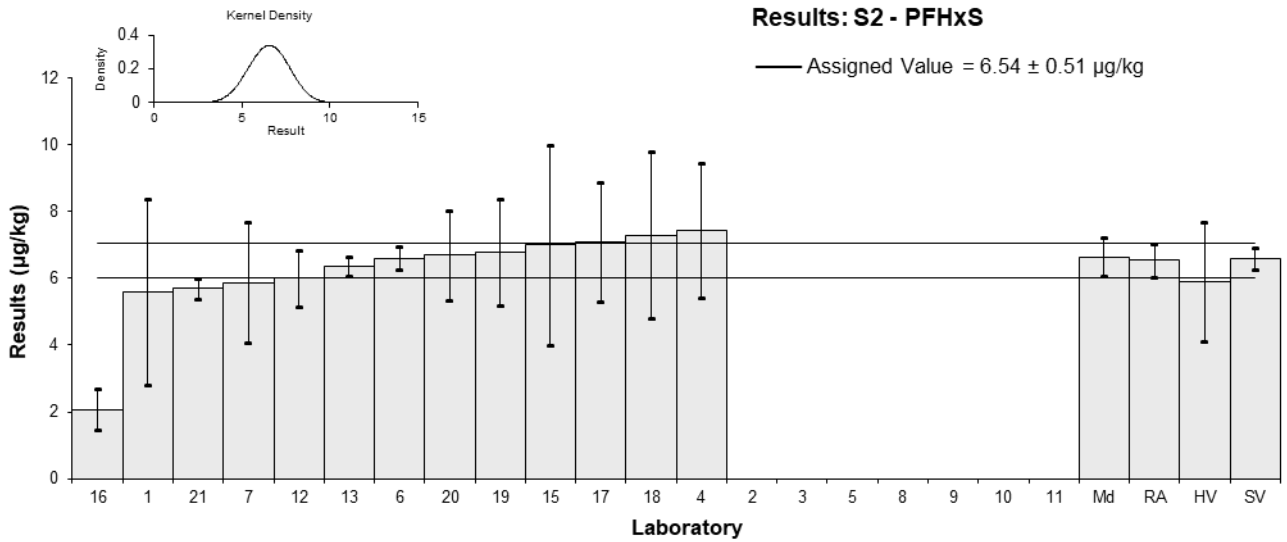


Figure 27

Table 32

## Sample Details

|                   |                |
|-------------------|----------------|
| <b>Sample No.</b> | S2             |
| <b>Matrix</b>     | Carrot         |
| <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    | NR          | NR   | -0.65 | -1.71          |
| 2         | 5.10   | 0.665       | 99   | -1.04 | -1.62          |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 7.44   | 2           | NR   | 0.78  | 0.49           |
| 5         | 7.18   | 2.3         | NR   | 0.57  | 0.31           |
| 6         | 6.6    | 0.35        | NT   | 0.12  | 0.27           |
| 7         | 5.88   | 1.8         | 92   | -0.43 | -0.30          |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | 6.51   | 2.11        | 86.4 | 0.05  | 0.03           |
| 12        | 6.01   | 0.842       | 87.6 | -0.33 | -0.44          |
| 13        | 6.34   | 0.248       | 117  | -0.08 | -0.18          |
| 15        | 7      | 3           | 88   | 0.43  | 0.18           |
| 16**      | 2.07   | 0.621       | 134  | -3.39 | -5.52          |
| 17        | 7.1    | 1.8         | 94   | 0.51  | 0.35           |
| 18        | NT     | NT          | NT   |       |                |
| 19        | 6.8    | 1.6         | 106  | 0.28  | 0.22           |
| 20        | 6.70   | 1.34        | 142  | 0.20  | 0.18           |
| 21        | 5.7    | 0.3         | NT   | -0.57 | -1.29          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 6.44 | 0.49 |
| <b>Spike Value</b>       | 6.61 | 0.33 |
| <b>Homogeneity Value</b> | 5.9  | 1.8  |
| <b>Robust Average</b>    | 6.44 | 0.49 |
| <b>Median</b>            | 6.56 | 0.54 |
| <b>Mean</b>              | 6.43 |      |
| <b>N</b>                 | 14   |      |
| <b>Max</b>               | 7.44 |      |
| <b>Min</b>               | 5.1  |      |
| <b>Robust SD</b>         | 0.74 |      |
| <b>Robust CV</b>         | 11%  |      |

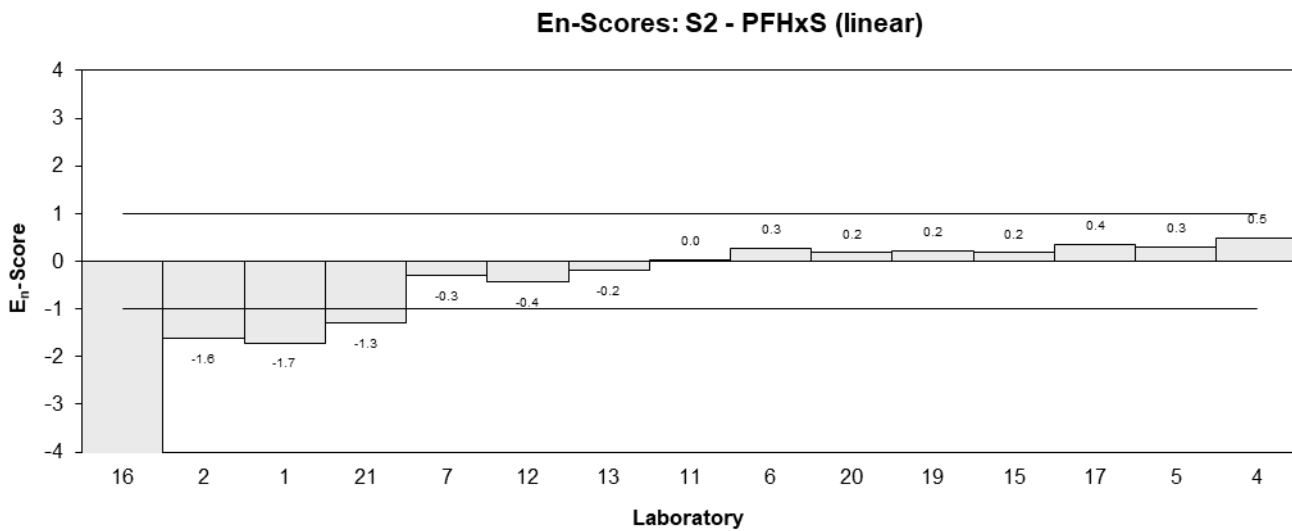
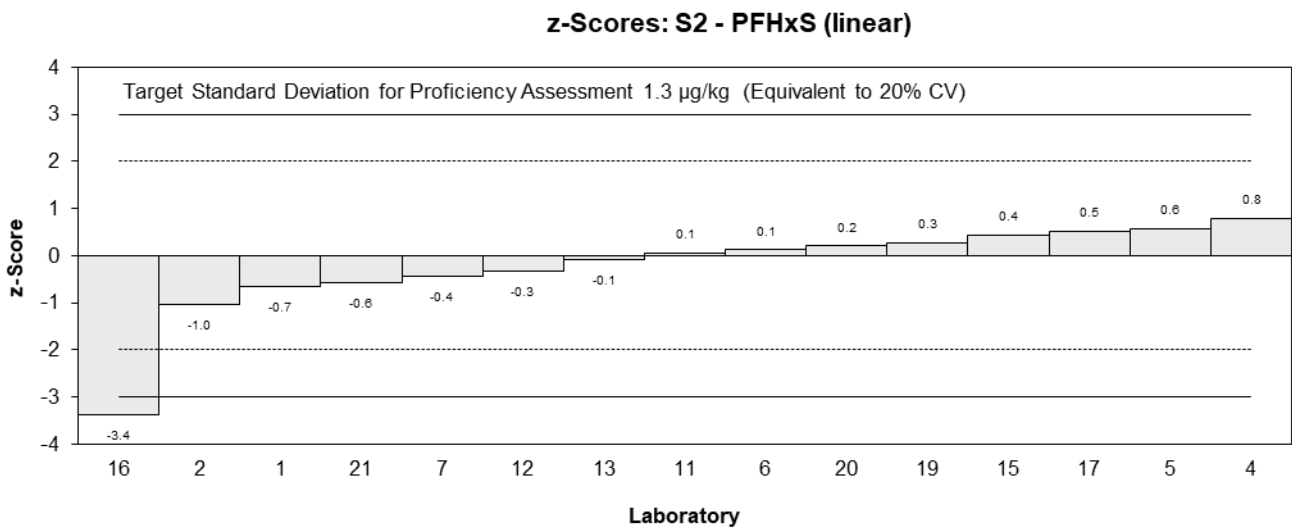
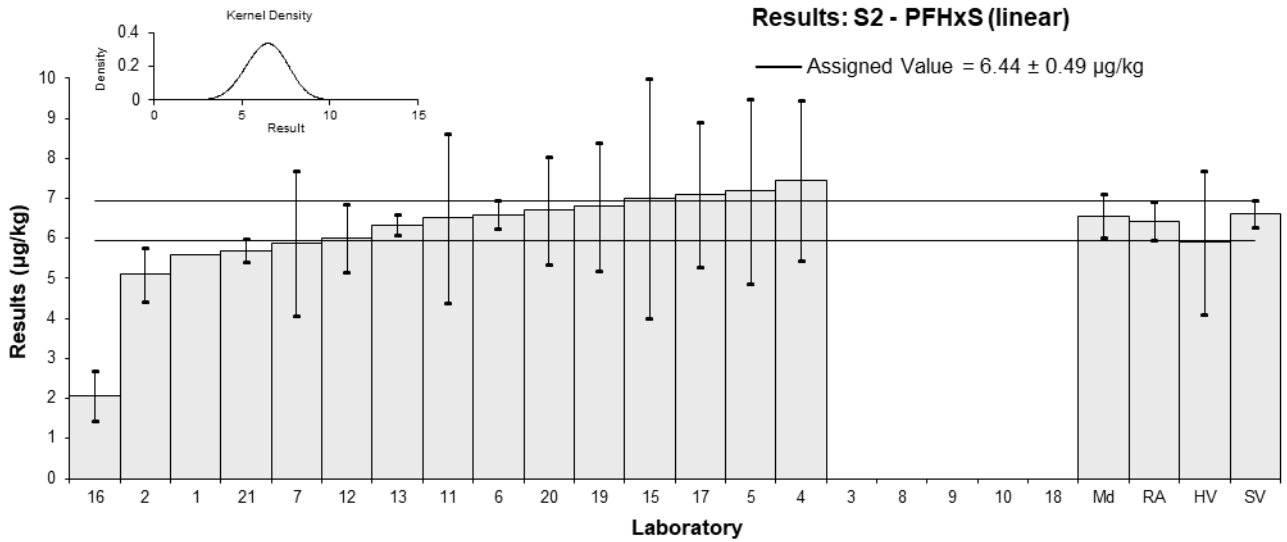


Figure 28

Table 33

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFHpS  |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 2.6    | 1.3         | NR   | -0.65 | -0.30          |
| 2         | 2.11   | 0.357       | 99   | -1.47 | -2.05          |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 2.87   | 0.9         | NR   | -0.20 | -0.13          |
| 5         | NT     | NT          | NT   |       |                |
| 6         | 3.1    | 0.2         | NT   | 0.18  | 0.35           |
| 7         | 2.64   | 0.79        | 82   | -0.59 | -0.42          |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | 3.32   | 0.840       | 86.4 | 0.55  | 0.38           |
| 12        | 3.22   | 0.483       | 92.1 | 0.38  | 0.43           |
| 13        | 3      | 0.186       | 117  | 0.02  | 0.03           |
| 15        | 3      | 2           | 88   | 0.02  | 0.00           |
| 16**      | 1.14   | 0.342       | 162  | -3.09 | -4.43          |
| 17        | 2.6    | 0.64        | 94   | -0.65 | -0.57          |
| 18        | 3.04   | 1           | 137  | 0.08  | 0.05           |
| 19        | 3.2    | 0.70        | 106  | 0.35  | 0.28           |
| 20        | 3.27   | 0.654       | 142  | 0.47  | 0.40           |
| 21        | 3.9    | 0.2         | NT   | 1.52  | 2.91           |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 2.99 | 0.24 |
| <b>Spike Value</b>       | 3.00 | 0.15 |
| <b>Homogeneity Value</b> | 2.62 | 0.79 |
| <b>Robust Average</b>    | 2.99 | 0.24 |
| <b>Median</b>            | 3.02 | 0.22 |
| <b>Mean</b>              | 2.99 |      |
| <b>N</b>                 | 14   |      |
| <b>Max</b>               | 3.9  |      |
| <b>Min</b>               | 2.11 |      |
| <b>Robust SD</b>         | 0.36 |      |
| <b>Robust CV</b>         | 12%  |      |



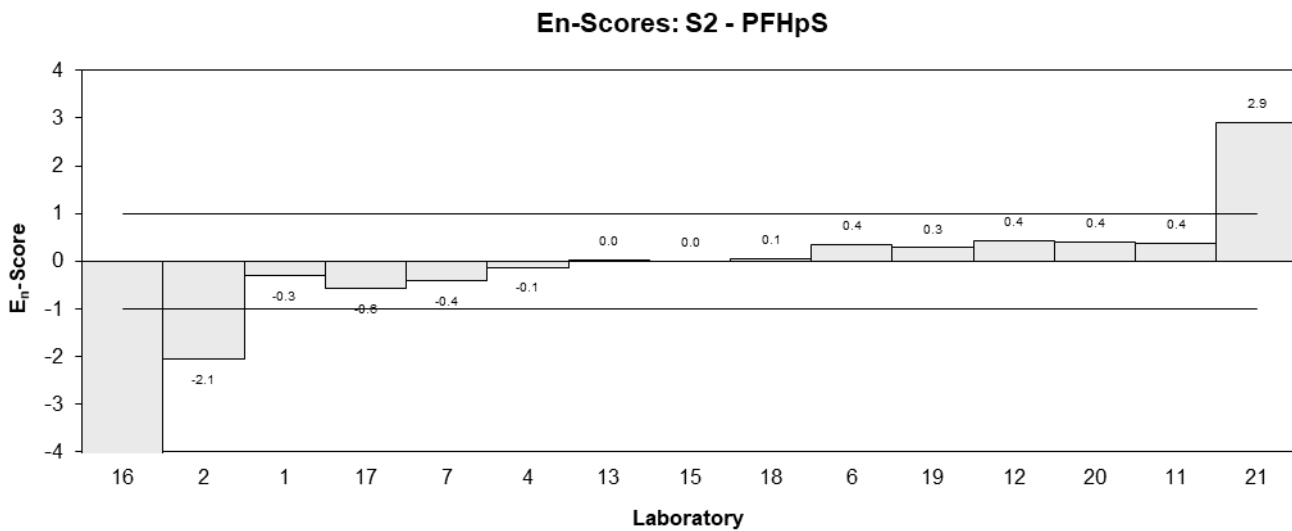
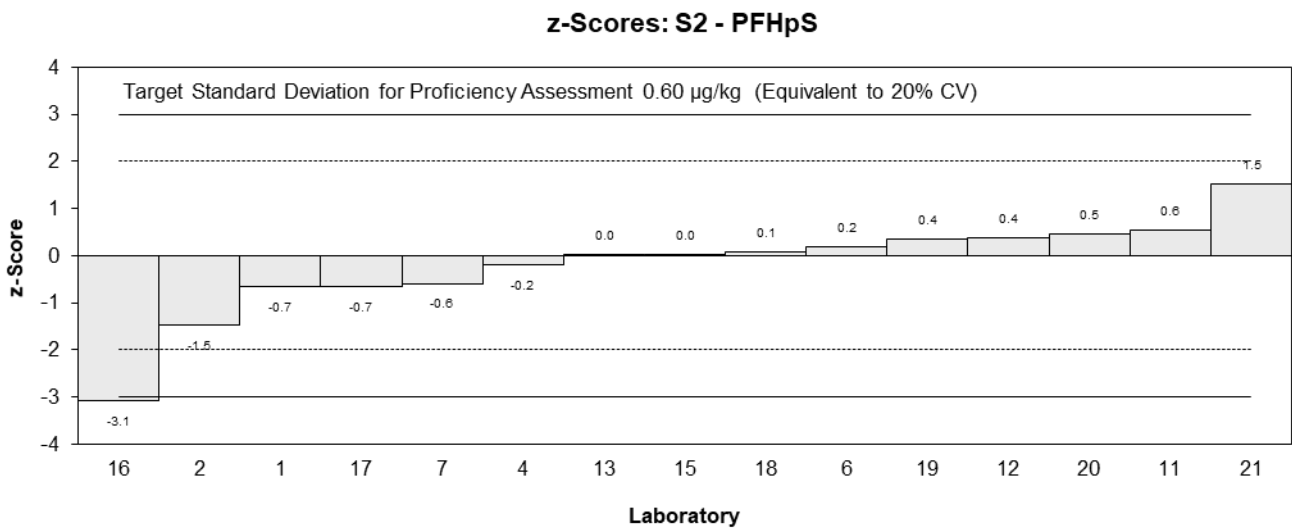
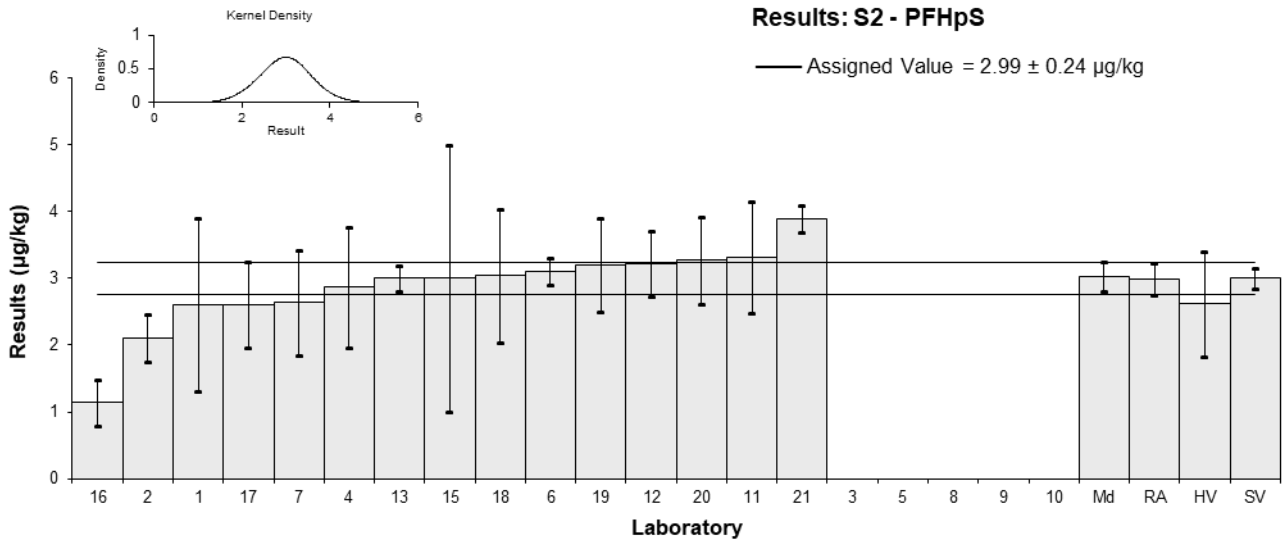


Figure 29

Table 34

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFOS   |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 1.7    | 0.85        | 100  | -0.64 | -0.29          |
| 2         | 1.53   | 0.211       | 88   | -1.08 | -1.55          |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 1.88   | 0.6         | 89   | -0.18 | -0.11          |
| 5         | NT     | NT          | NT   |       |                |
| 6         | 2.2    | 0.15        | 92   | 0.64  | 1.10           |
| 7         | 1.98   | 0.59        | 91   | 0.08  | 0.05           |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | 2.03   | 0.450       | 88.7 | 0.21  | 0.17           |
| 12        | 2.31   | 0.392       | 92.1 | 0.92  | 0.84           |
| 13        | 1.9    | 0.159       | 120  | -0.13 | -0.21          |
| 15        | 2      | 1           | 89   | 0.13  | 0.05           |
| 16**      | 0.651  | 0.1953      | 157  | -3.33 | -5.02          |
| 17        | 2.2    | 0.54        | 94   | 0.64  | 0.44           |
| 18        | 1.9    | 0.7         | 137  | -0.13 | -0.07          |
| 19        | 2.1    | 0.13        | 104  | 0.38  | 0.70           |
| 20        | 2.01   | 0.40        | 83   | 0.15  | 0.14           |
| 21        | 1.4    | 0.15        | 92   | -1.41 | -2.43          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 1.95 | 0.17 |
| <b>Spike Value</b>       | 2.12 | 0.11 |
| <b>Homogeneity Value</b> | 1.88 | 0.56 |
| <b>Robust Average</b>    | 1.95 | 0.17 |
| <b>Median</b>            | 1.99 | 0.11 |
| <b>Mean</b>              | 1.94 |      |
| <b>N</b>                 | 14   |      |
| <b>Max</b>               | 2.31 |      |
| <b>Min</b>               | 1.4  |      |
| <b>Robust SD</b>         | 0.25 |      |
| <b>Robust CV</b>         | 13%  |      |

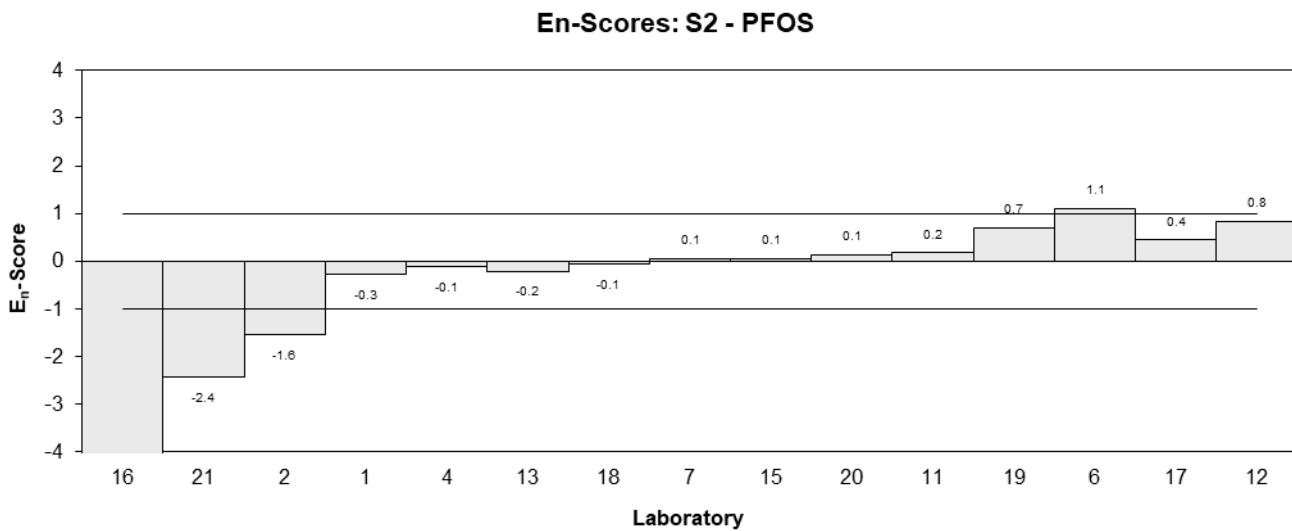
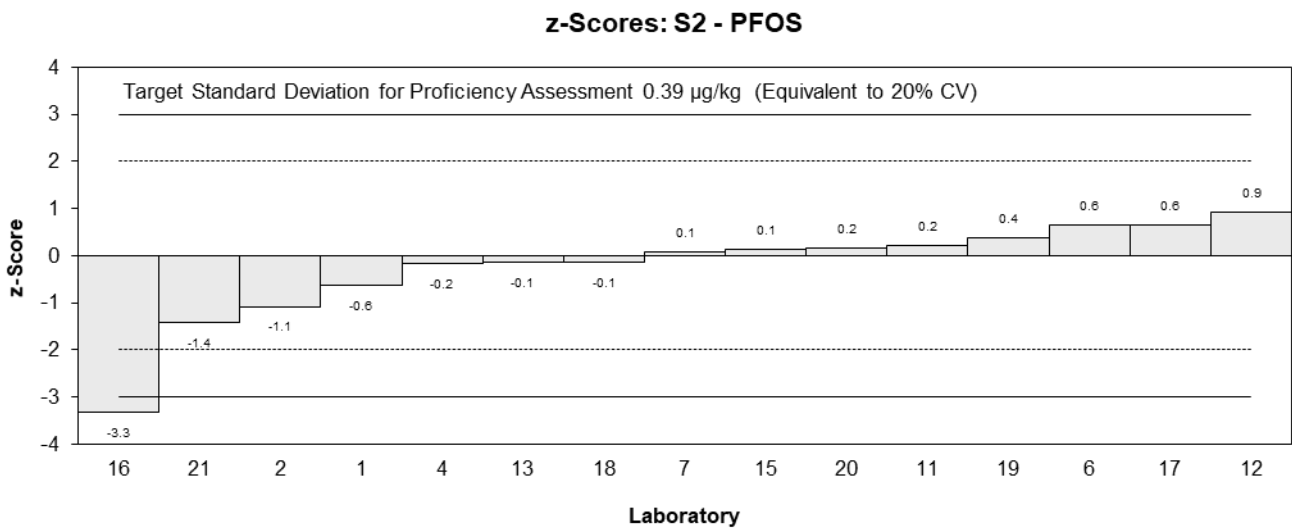
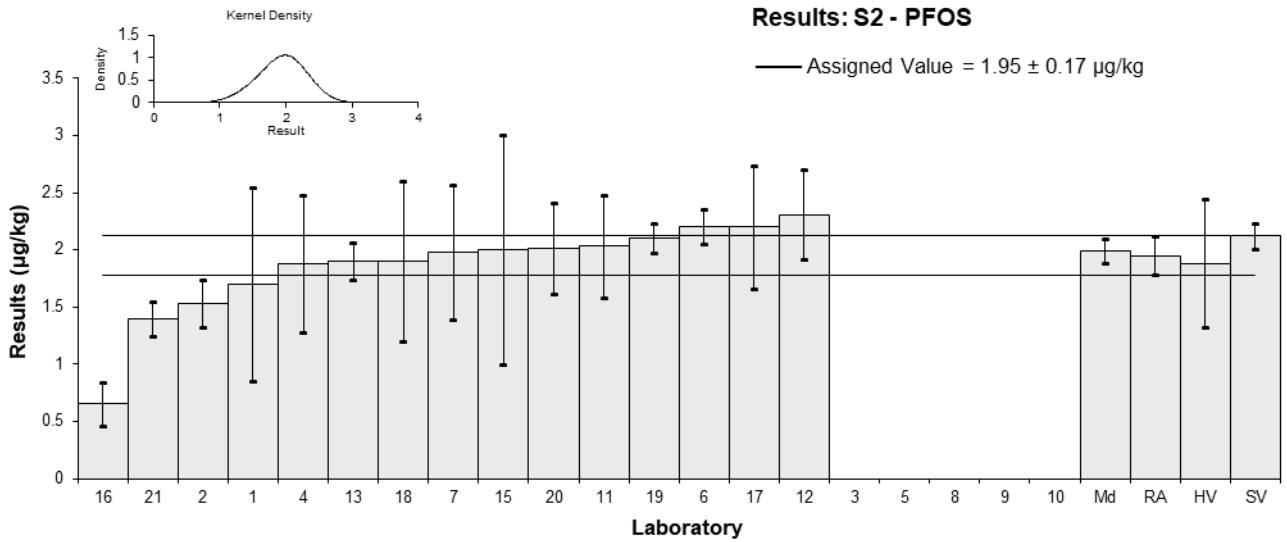


Figure 30

Table 35

## Sample Details

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

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 1.7    | NR          | NR   | -0.77 | -2.07          |
| 2         | NR     | NR          | NR   |       |                |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 1.88   | 0.6         | NR   | -0.32 | -0.21          |
| 5         | 2.33   | 0.50        | NR   | 0.80  | 0.61           |
| 6         | 2.2    | 0.15        | 92   | 0.47  | 0.90           |
| 7         | 1.98   | 0.59        | 91   | -0.07 | -0.05          |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | 2.03   | 0.450       | 88.7 | 0.05  | 0.04           |
| 12        | 2.3    | 0.392       | 92.1 | 0.72  | 0.69           |
| 13        | 1.85   | 0.146       | 120  | -0.40 | -0.76          |
| 15        | 2      | 1           | 89   | -0.02 | -0.01          |
| 16**      | 0.651  | 0.1953      | 157  | -3.38 | -5.52          |
| 17        | 2.2    | 0.54        | 94   | 0.47  | 0.34           |
| 18        | 1.9    | 0.7         | 137  | -0.27 | -0.15          |
| 19        | 2.1    | 0.13        | 104  | 0.22  | 0.45           |
| 20        | 2.01   | 0.40        | 83   | 0.00  | 0.00           |
| 21        | 1.4    | 0.15        | 92   | -1.52 | -2.88          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 2.01 | 0.15 |
| <b>Spike Value</b>       | 2.12 | 0.11 |
| <b>Homogeneity Value</b> | 1.88 | 0.56 |
| <b>Robust Average</b>    | 2.01 | 0.15 |
| <b>Median</b>            | 2.01 | 0.14 |
| <b>Mean</b>              | 1.99 |      |
| <b>N</b>                 | 14   |      |
| <b>Max</b>               | 2.33 |      |
| <b>Min</b>               | 1.4  |      |
| <b>Robust SD</b>         | 0.23 |      |
| <b>Robust CV</b>         | 11%  |      |

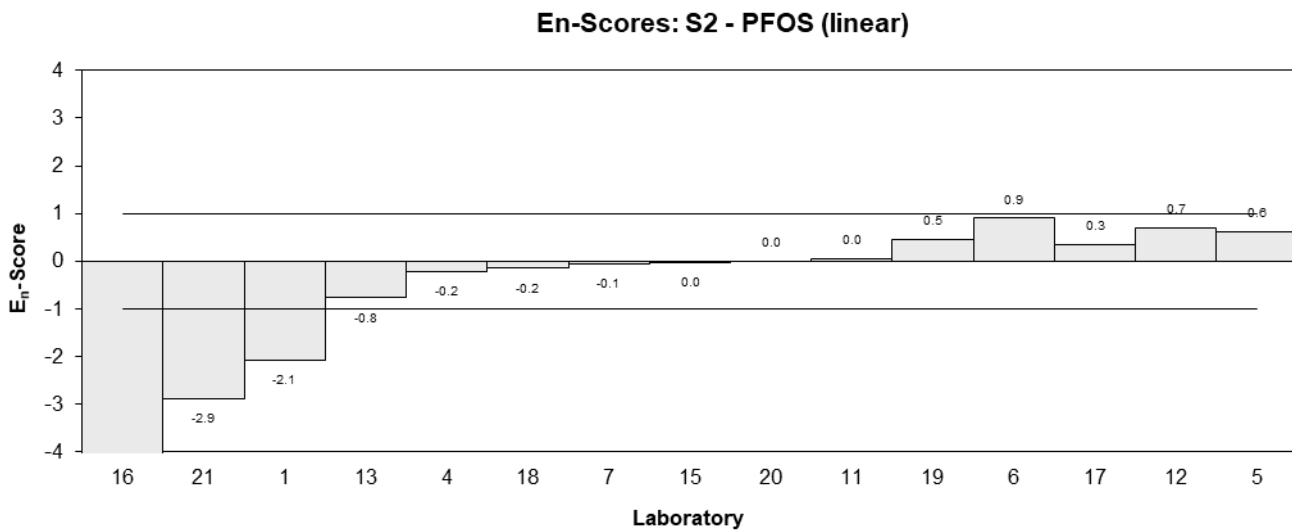
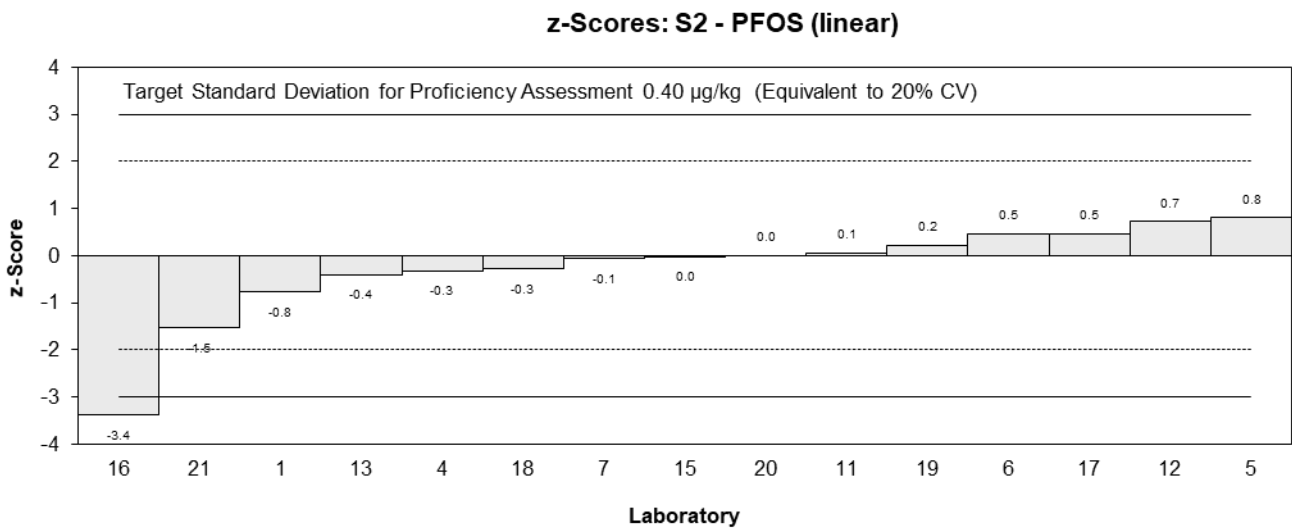
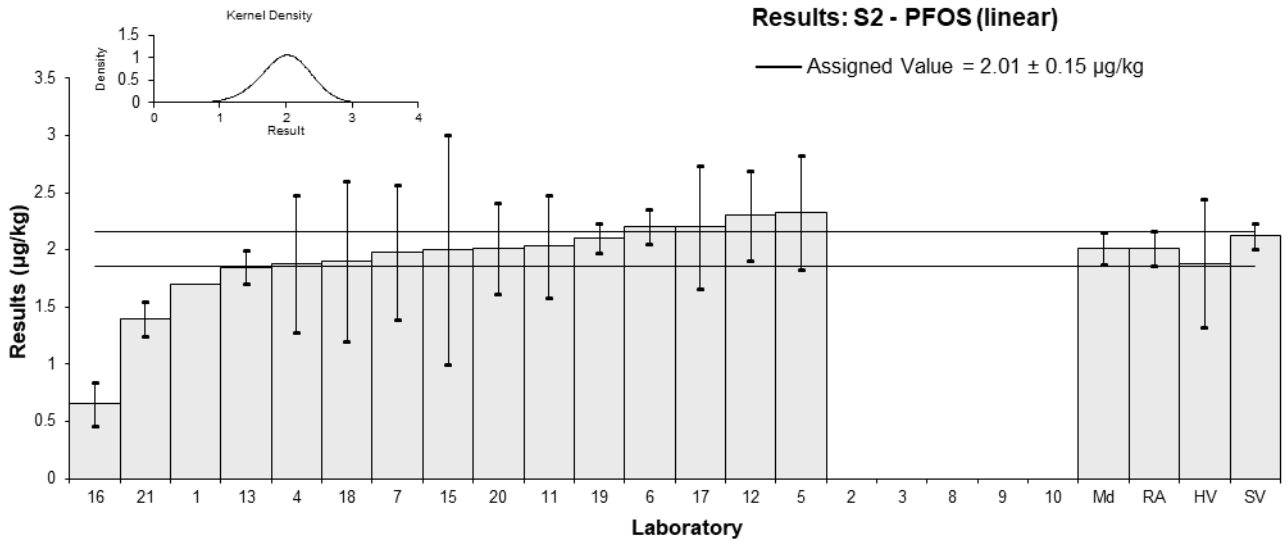


Figure 31

Table 36

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFNS   |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 1.2    | 0.6         | NR   | -1.05 | -0.50          |
| 2         | 1.44   | 0.140       | 88   | -0.26 | -0.31          |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 1.53   | 0.5         | NR   | 0.03  | 0.02           |
| 5         | NT     | NT          | NT   |       |                |
| 6         | <1     | NR          | NT   |       |                |
| 7         | 1.45   | 0.44        | 87   | -0.23 | -0.14          |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | 1.71   | 1.16        | 88.7 | 0.62  | 0.16           |
| 12        | 1.82   | 0.381       | 92.1 | 0.99  | 0.68           |
| 13        | 1.35   | 0.109       | 120  | -0.56 | -0.69          |
| 15        | 2      | 1           | 89   | 1.58  | 0.47           |
| 16        | NT     | NT          | NT   |       |                |
| 17        | 1.4    | 0.35        | 94   | -0.39 | -0.29          |
| 18        | NT     | NT          | NT   |       |                |
| 19        | 1.7    | 0.22        | 104  | 0.59  | 0.58           |
| 20        | 1.13   | 0.23        | 83   | -1.28 | -1.23          |
| 21        | <1     | NR          | NT   |       |                |

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 1.52 | 0.22 |
| <b>Spike Value</b>       | 1.72 | 0.09 |
| <b>Homogeneity Value</b> | 1.51 | 0.45 |
| <b>Robust Average</b>    | 1.52 | 0.22 |
| <b>Median</b>            | 1.45 | 0.28 |
| <b>Mean</b>              | 1.52 |      |
| <b>N</b>                 | 11   |      |
| <b>Max</b>               | 2    |      |
| <b>Min</b>               | 1.13 |      |
| <b>Robust SD</b>         | 0.29 |      |
| <b>Robust CV</b>         | 19%  |      |

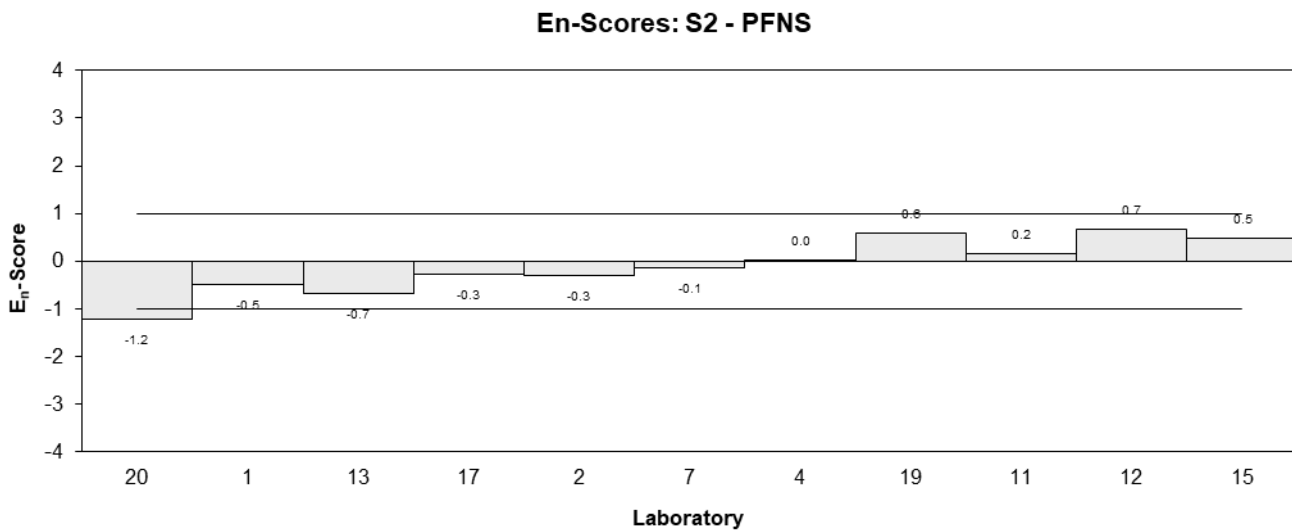
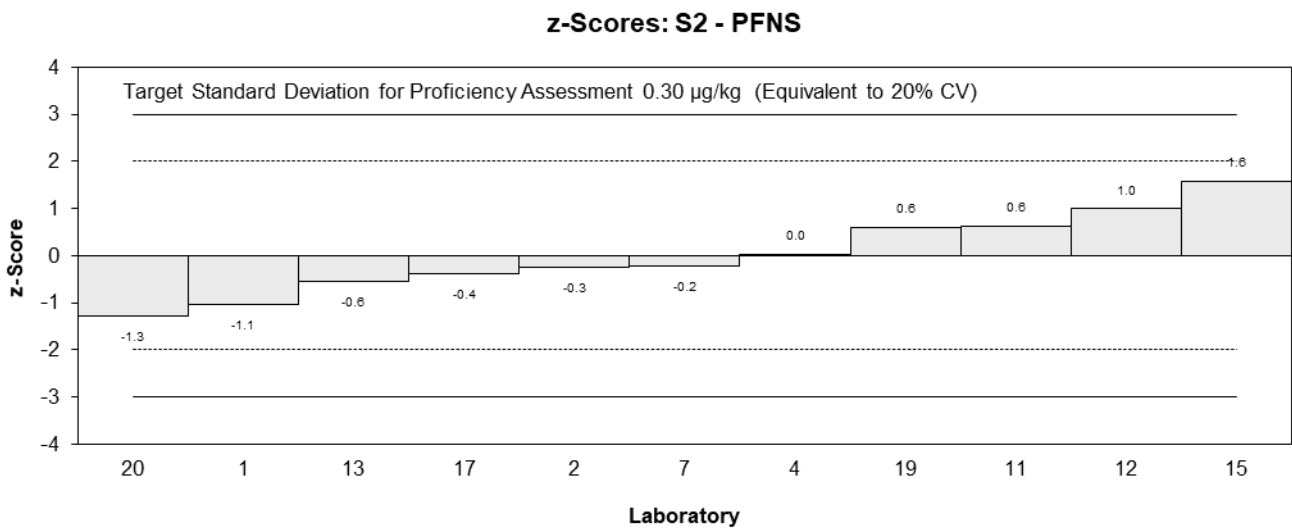
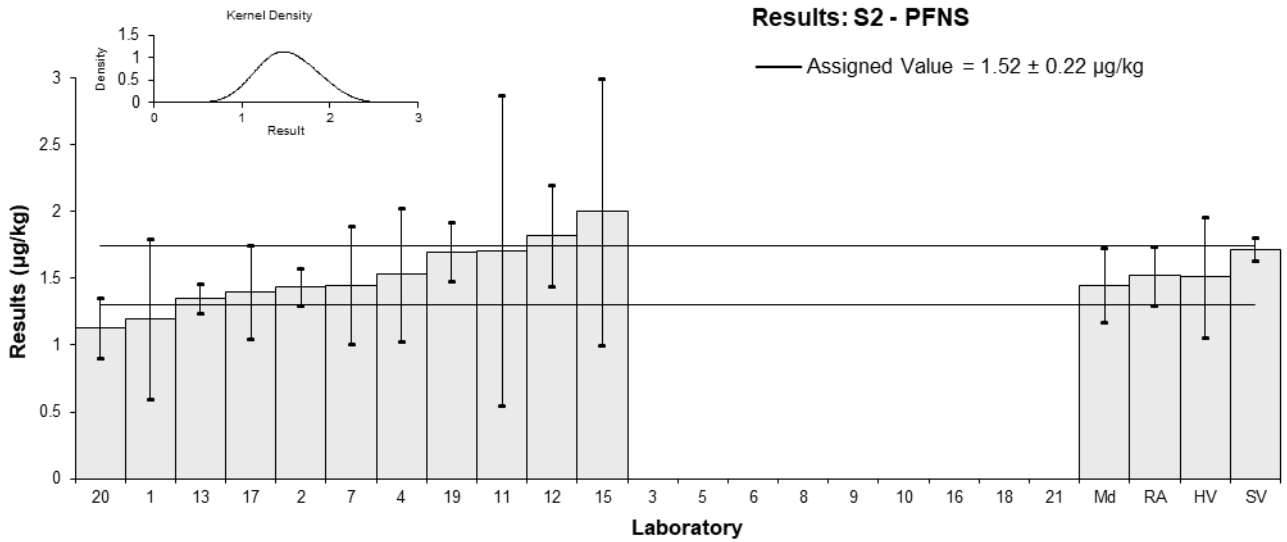


Figure 32

Table 37

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFDS   |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | NT     | NT          | NT   |       |                |
| 2         | 6.35   | 1.04        | 88   | -0.30 | -0.29          |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 7.22   | 3           | 89   | 0.34  | 0.15           |
| 5         | 7.86   | 2.1         | NR   | 0.81  | 0.48           |
| 6         | 6.6    | 0.35        | NT   | -0.12 | -0.16          |
| 7         | 6.80   | 2.0         | 86   | 0.03  | 0.02           |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | 8.08   | 5.24        | 88.7 | 0.98  | 0.25           |
| 12        | 8.67   | 1.91        | 92.1 | 1.41  | 0.89           |
| 13        | 5.57   | 0.631       | 120  | -0.88 | -1.04          |
| 15        | 8      | 3           | 89   | 0.92  | 0.39           |
| 16**      | 1.55   | 0.465       | 181  | -3.85 | -4.88          |
| 17        | 6.0    | 1.5         | 94   | -0.56 | -0.43          |
| 18        | 5.94   | 2.2         | 128  | -0.61 | -0.34          |
| 19        | 8.0    | 0.93        | 104  | 0.92  | 0.93           |
| 20        | 3.38   | 0.68        | 83   | -2.50 | -2.87          |
| 21        | 4.9    | 0.25        | NT   | -1.38 | -1.87          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 6.76 | 0.96 |
| <b>Spike Value</b>       | 6.80 | 0.34 |
| <b>Homogeneity Value</b> | 6.4  | 1.9  |
| <b>Robust Average</b>    | 6.76 | 0.96 |
| <b>Median</b>            | 6.7  | 1.1  |
| <b>Mean</b>              | 6.67 |      |
| <b>N</b>                 | 14   |      |
| <b>Max</b>               | 8.67 |      |
| <b>Min</b>               | 3.38 |      |
| <b>Robust SD</b>         | 1.4  |      |
| <b>Robust CV</b>         | 21%  |      |



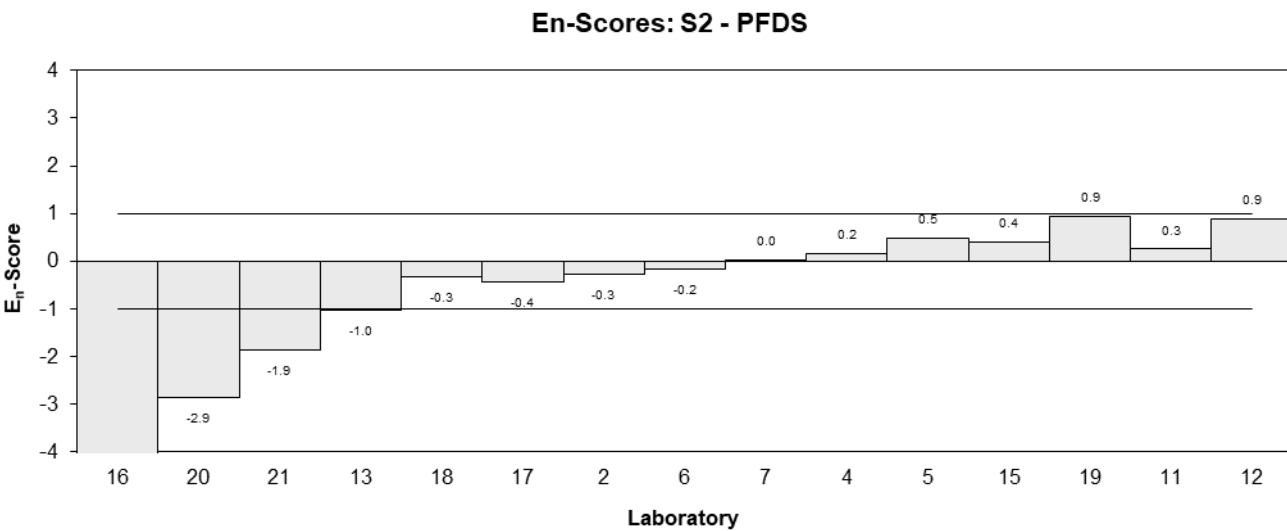
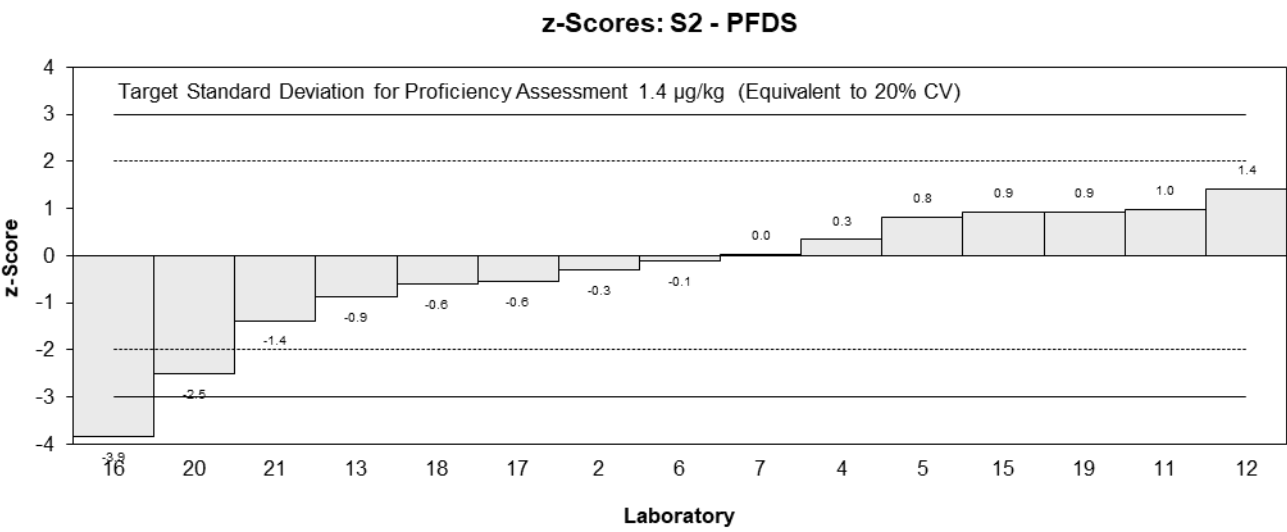
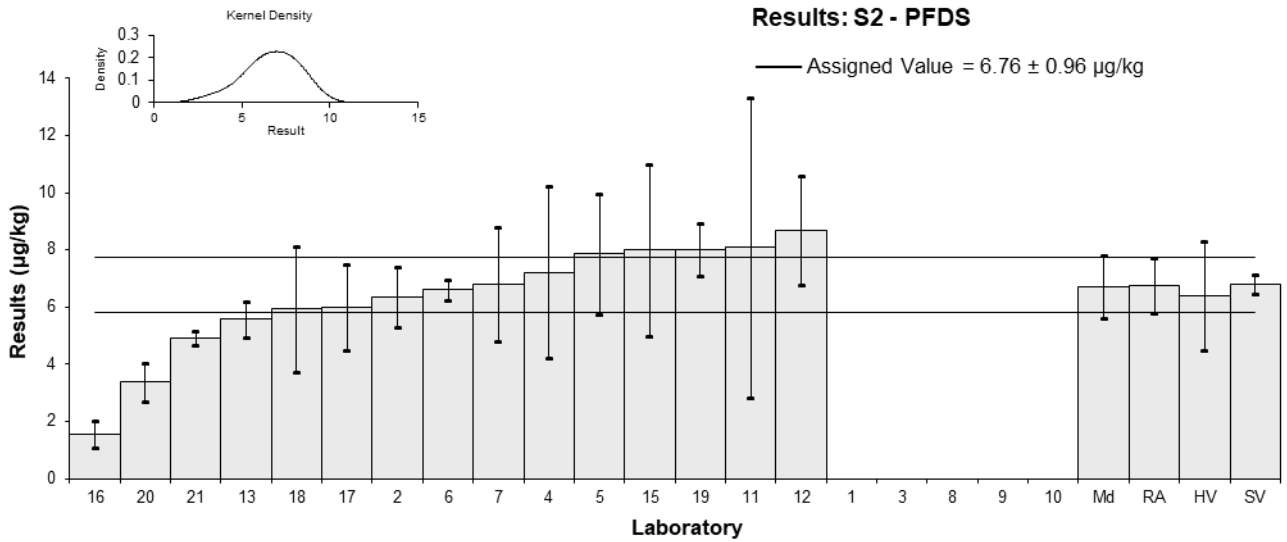


Figure 33

Table 38

**Sample Details**

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFBA   |
| <b>Unit</b>       | µg/kg  |

**Participant Results**

| <b>Lab. Code</b> | <b>Result</b> | <b>Uncertainty</b> | <b>Rec</b> |
|------------------|---------------|--------------------|------------|
| 1                | < 1           | 0.5                | 92         |
| 2                | 0.630         | 0.016              | 91         |
| 3                | NS            | NS                 | NS         |
| 4                | 0.861         | 0.3                | 85         |
| 5                | 1.26          | 0.97               | NR         |
| 6                | <5            | NR                 | 81         |
| 7                | 0.395         | 0.12               | 81         |
| 8                | NS            | NS                 | NS         |
| 9                | NT            | NT                 | NT         |
| 10               | NS            | NS                 | NS         |
| 11               | 0.908         | 0.213              | 74.3       |
| 12               | 1.09          | 0.0983             | 86.9       |
| 13               | 0.788         | 0.04               | 108        |
| 15               | <2            | NR                 | 86         |
| 16               | < 0.3         | NR                 | 97.3       |
| 17               | 0.31          | 0.22               | 94         |
| 18               | <5            | NR                 | 91         |
| 19               | NR            | NR                 | 112        |
| 20               | NT            | NT                 | NT         |
| 21               | <5            | NR                 | 81         |

**Statistics**

|                          |         |      |
|--------------------------|---------|------|
| <b>Assigned Value</b>    | Not Set |      |
| <b>Spike Value</b>       | 1.19    | 0.06 |
| <b>Homogeneity Value</b> | 0.81    | 0.24 |
| <b>Robust Average</b>    | 0.78    | 0.33 |
| <b>Median</b>            | 0.82    | 0.30 |
| <b>Mean</b>              | 0.78    |      |
| <b>N</b>                 | 8       |      |
| <b>Max</b>               | 1.26    |      |
| <b>Min</b>               | 0.31    |      |
| <b>Robust SD</b>         | 0.37    |      |
| <b>Robust CV</b>         | 47%     |      |

Results: S2 - PFBA

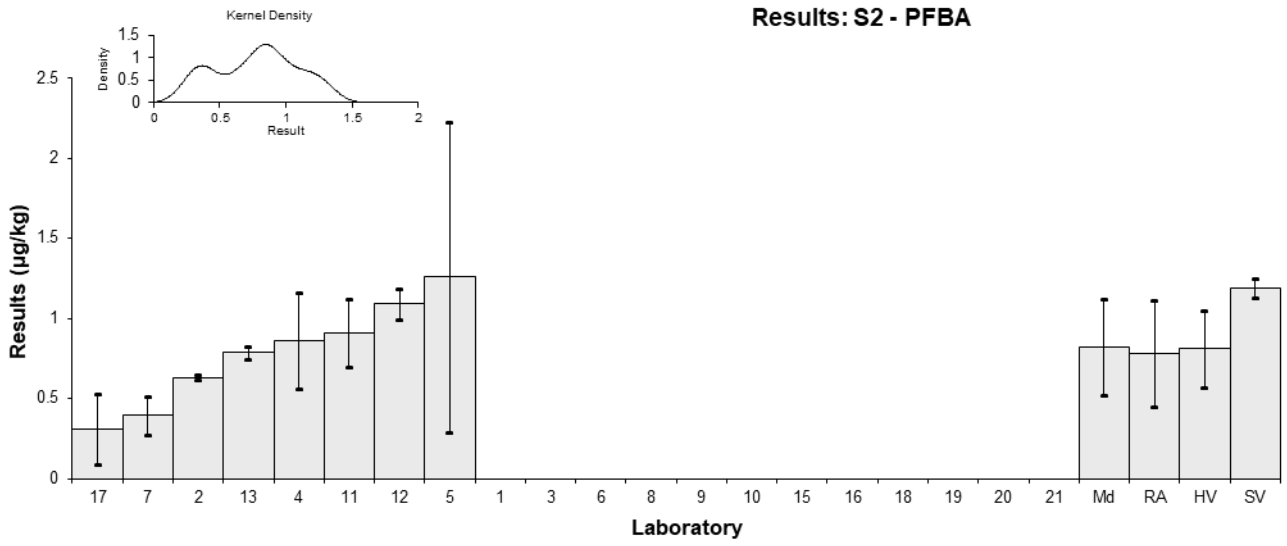


Figure 34

Table 39

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFPeA  |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec   | z     | E <sub>n</sub> |
|-----------|--------|-------------|-------|-------|----------------|
| 1         | 1.7    | 0.85        | 126   | -0.89 | -0.42          |
| 2         | 1.45   | 0.071       | 98    | -1.50 | -2.30          |
| 3         | NS     | NS          | NS    |       |                |
| 4         | 2.14   | 0.6         | 70    | 0.17  | 0.11           |
| 5         | 1.89   | 0.49        | NR    | -0.43 | -0.32          |
| 6         | <1     | NR          | NT    |       |                |
| 7         | 2.68   | 0.80        | 86    | 1.47  | 0.73           |
| 8         | NS     | NS          | NS    |       |                |
| 9         | NT     | NT          | NT    |       |                |
| 10        | NS     | NS          | NS    |       |                |
| 11        | 2.25   | 0.417       | 75.7  | 0.43  | 0.37           |
| 12        | 1.98   | 0.198       | 100.3 | -0.22 | -0.28          |
| 13        | 1.95   | 0.096       | 120   | -0.29 | -0.43          |
| 15        | 2      | 1           | 89    | -0.17 | -0.07          |
| 16**      | 0.612  | 0.1836      | 142   | -3.52 | -4.58          |
| 17*       | 4.4    | 1.1         | 94    | 5.63  | 2.06           |
| 18        | 2.4    | 0.8         | 133   | 0.80  | 0.39           |
| 19        | 2.3    | 0.24        | 98    | 0.56  | 0.65           |
| 20        | NT     | NT          | NT    |       |                |
| 21        | <1     | NR          | NT    |       |                |

\* Outlier, \*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 2.07 | 0.26 |
| <b>Spike Value</b>       | 2.20 | 0.11 |
| <b>Homogeneity Value</b> | 1.93 | 0.58 |
| <b>Robust Average</b>    | 2.13 | 0.31 |
| <b>Median</b>            | 2.07 | 0.22 |
| <b>Mean</b>              | 2.26 |      |
| <b>N</b>                 | 12   |      |
| <b>Max</b>               | 4.4  |      |
| <b>Min</b>               | 1.45 |      |
| <b>Robust SD</b>         | 0.42 |      |
| <b>Robust CV</b>         | 20%  |      |

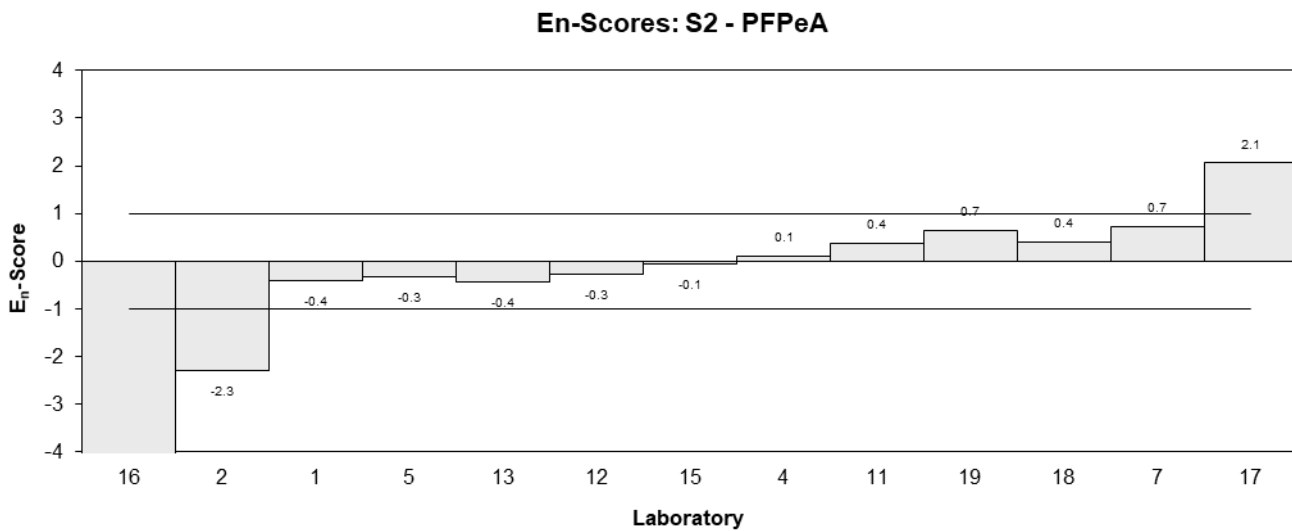
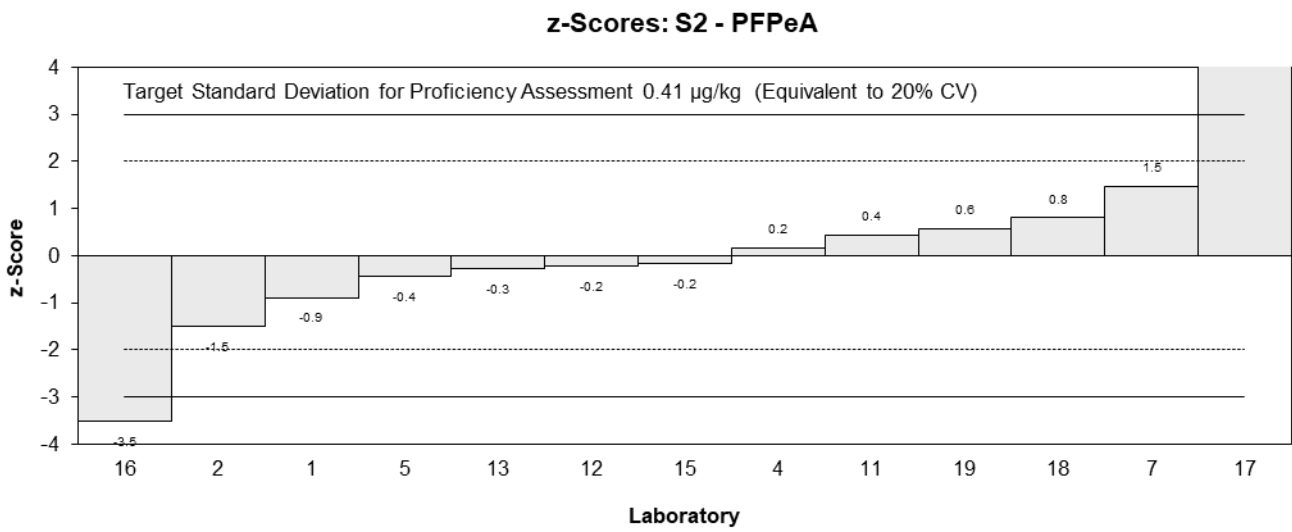
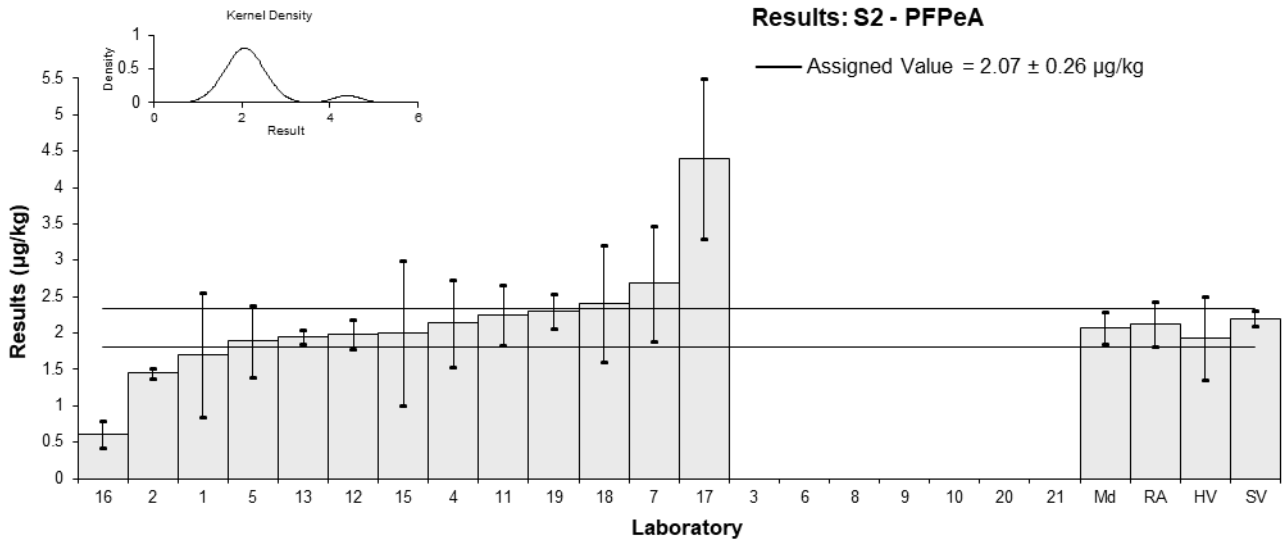


Figure 35

Table 40

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFHxA  |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec   | z     | E <sub>n</sub> |
|-----------|--------|-------------|-------|-------|----------------|
| 1         | 8      | 4           | 113   | 0.11  | 0.04           |
| 2         | 5.71   | 0.339       | 95    | -1.35 | -4.62          |
| 3         | NS     | NS          | NS    |       |                |
| 4         | 8.25   | 2           | 76    | 0.27  | 0.21           |
| 5         | 7.38   | 1.3         | NR    | -0.29 | -0.34          |
| 6         | 7.5    | 0.4         | NT    | -0.21 | -0.65          |
| 7         | 8.65   | 2.6         | 79    | 0.52  | 0.31           |
| 8         | NS     | NS          | NS    |       |                |
| 9         | NT     | NT          | NT    |       |                |
| 10        | NS     | NS          | NS    |       |                |
| 11        | 7.59   | 1.86        | 112.2 | -0.15 | -0.13          |
| 12        | 7.51   | 1.05        | 85.6  | -0.20 | -0.29          |
| 13        | 7.79   | 0.264       | 99    | -0.03 | -0.10          |
| 15        | 8      | 3           | 93    | 0.11  | 0.06           |
| 16**      | 2.44   | 0.732       | 153   | -3.44 | -6.78          |
| 17        | 10.1   | 2.5         | 94    | 1.45  | 0.90           |
| 18        | 7.92   | 3           | 122   | 0.06  | 0.03           |
| 19        | 8.0    | 1.1         | 93    | 0.11  | 0.15           |
| 20        | 7.95   | 1.59        | 78    | 0.08  | 0.07           |
| 21        | 7.4    | 0.4         | NT    | -0.27 | -0.85          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 7.83 | 0.31 |
| <b>Spike Value</b>       | 7.45 | 0.37 |
| <b>Homogeneity Value</b> | 7.3  | 2.2  |
| <b>Robust Average</b>    | 7.83 | 0.31 |
| <b>Median</b>            | 7.92 | 0.32 |
| <b>Mean</b>              | 7.85 |      |
| <b>N</b>                 | 15   |      |
| <b>Max</b>               | 10.1 |      |
| <b>Min</b>               | 5.71 |      |
| <b>Robust SD</b>         | 0.48 |      |
| <b>Robust CV</b>         | 6.1% |      |

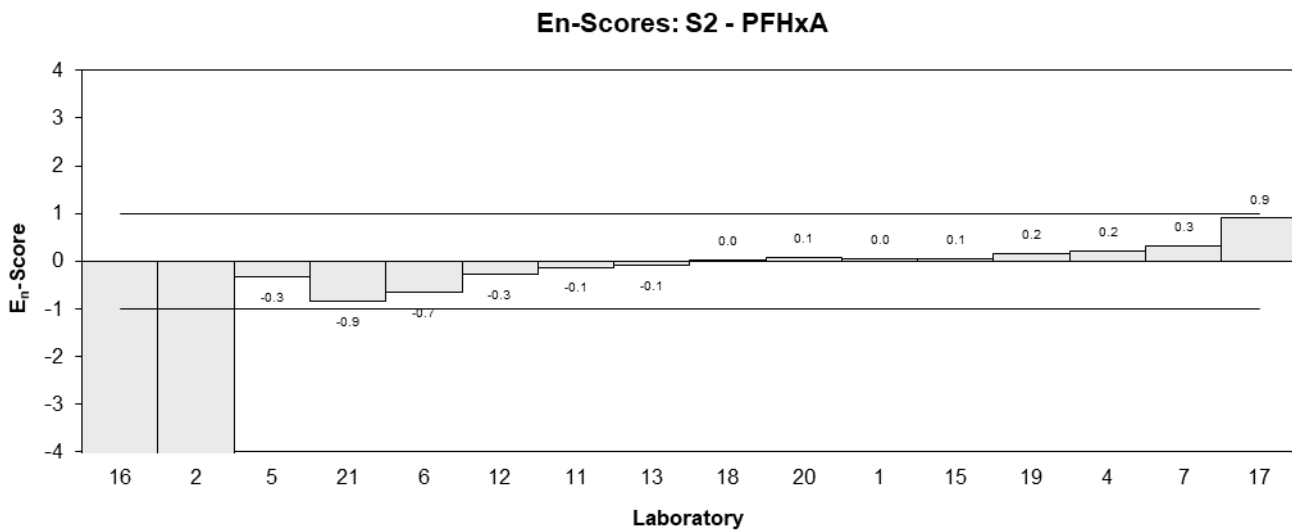
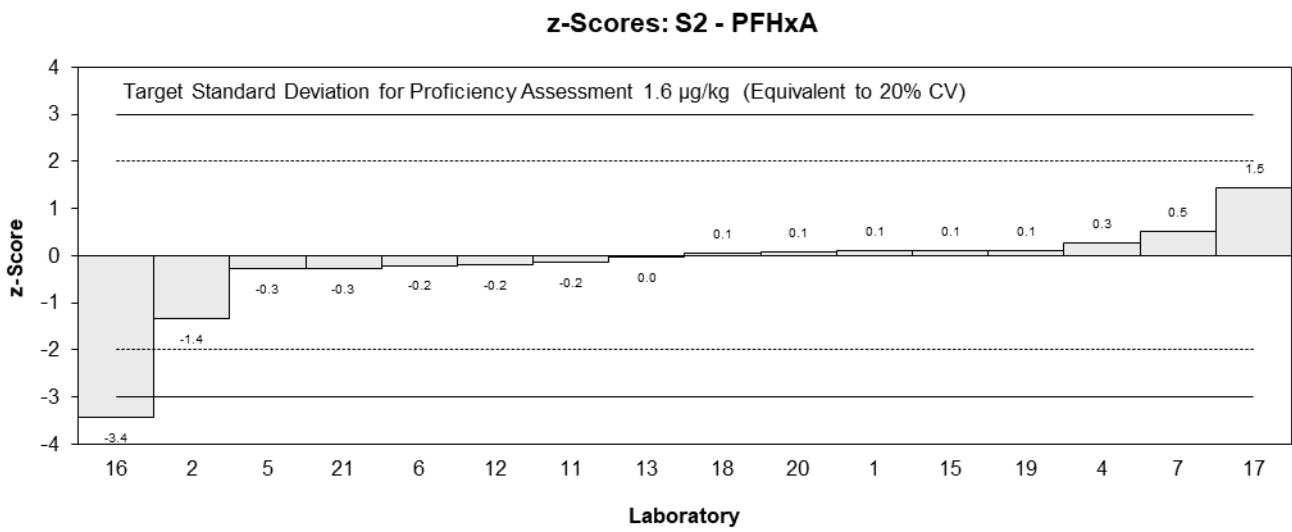
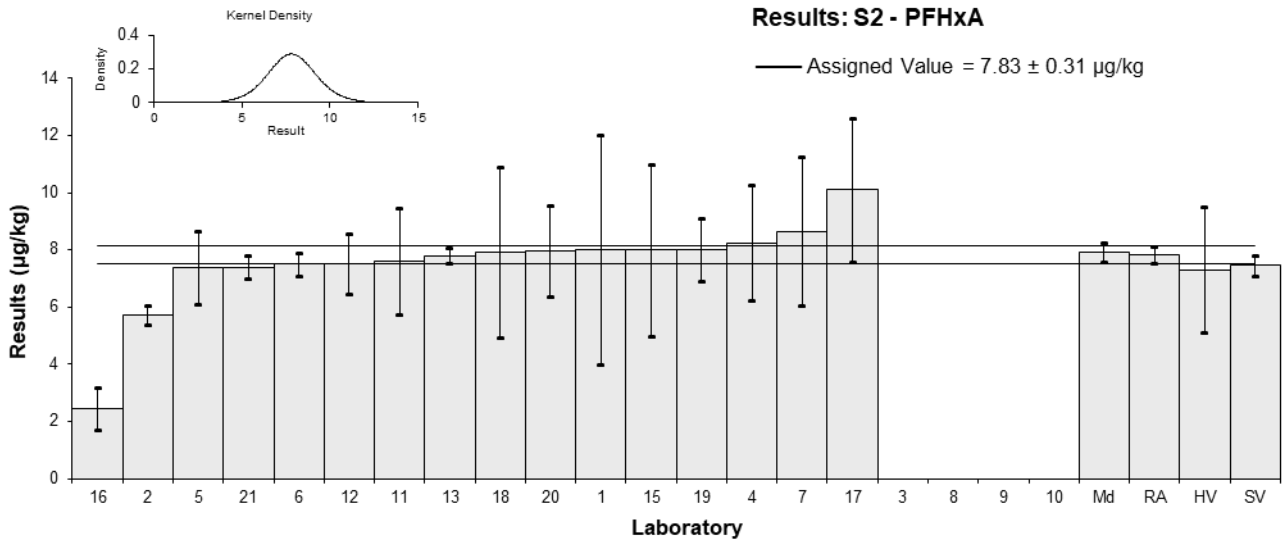


Figure 36

Table 41

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFHpA  |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 1.3    | 0.65        | 101  | -0.75 | -0.34          |
| 2         | 1.16   | 0.045       | 95   | -1.21 | -1.99          |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 1.8    | 0.5         | 76   | 0.88  | 0.51           |
| 5         | 1.19   | 0.39        | NR   | -1.11 | -0.79          |
| 6         | 1.4    | 0.1         | NT   | -0.42 | -0.63          |
| 7         | 1.62   | 0.49        | 80   | 0.29  | 0.17           |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | 1.66   | 0.470       | 86.3 | 0.42  | 0.26           |
| 12        | 1.38   | 0.207       | 87   | -0.49 | -0.55          |
| 13        | 1.59   | 0.029       | 72   | 0.20  | 0.33           |
| 15        | 2      | 1           | 91   | 1.54  | 0.46           |
| 16**      | 0.462  | 0.1386      | 162  | -3.49 | -4.70          |
| 17        | 1.8    | 0.44        | 94   | 0.88  | 0.57           |
| 18        | 1.68   | 0.6         | 125  | 0.49  | 0.24           |
| 19        | 1.7    | 0.45        | 99   | 0.56  | 0.35           |
| 20        | 1.56   | 0.311       | 98   | 0.10  | 0.08           |
| 21        | 1.2    | 0.2         | NT   | -1.08 | -1.23          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 1.53 | 0.18 |
| <b>Spike Value</b>       | 1.50 | 0.07 |
| <b>Homogeneity Value</b> | 1.52 | 0.46 |
| <b>Robust Average</b>    | 1.53 | 0.18 |
| <b>Median</b>            | 1.59 | 0.20 |
| <b>Mean</b>              | 1.54 |      |
| <b>N</b>                 | 15   |      |
| <b>Max</b>               | 2    |      |
| <b>Min</b>               | 1.16 |      |
| <b>Robust SD</b>         | 0.28 |      |
| <b>Robust CV</b>         | 18%  |      |



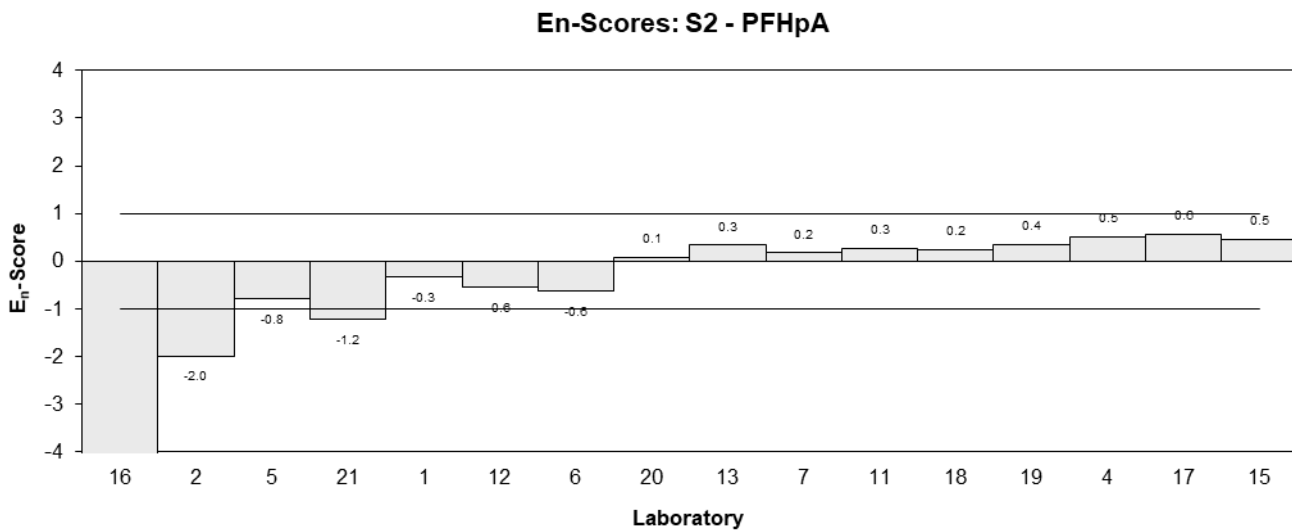
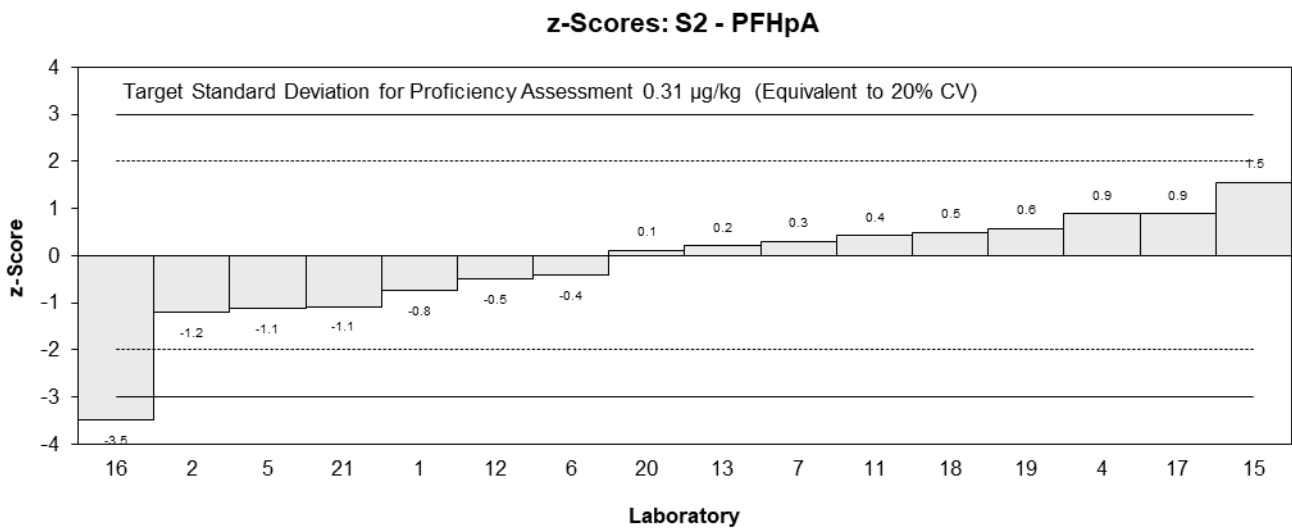
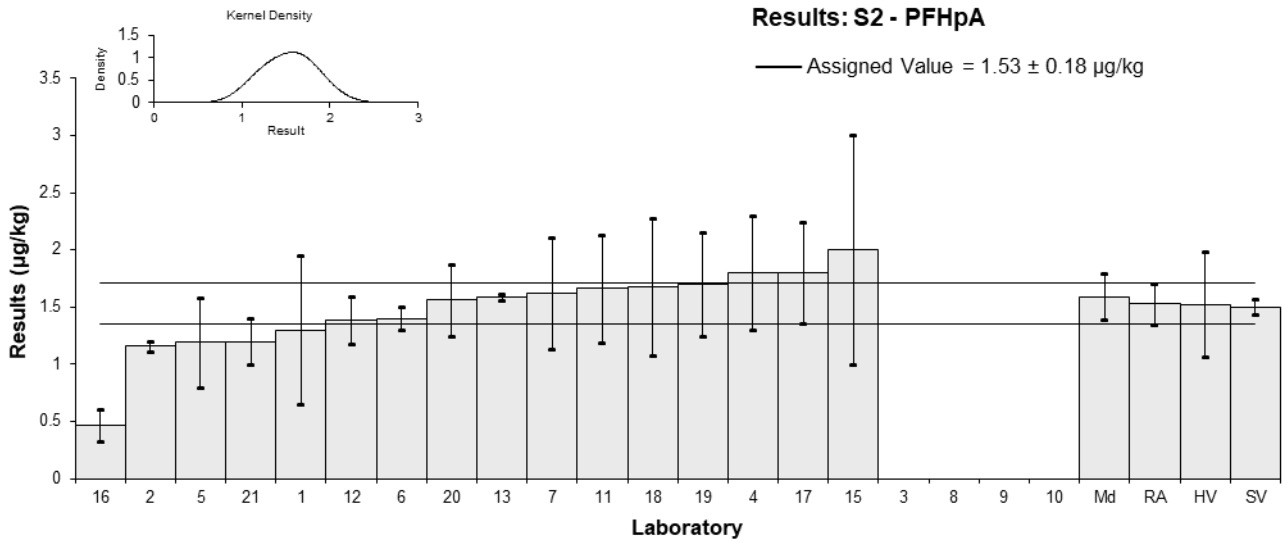


Figure 37

Table 42

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFOA   |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 1      | 0.5         | 111  | -0.97 | -0.46          |
| 2         | 0.935  | 0.068       | 85   | -1.23 | -1.96          |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 1.34   | 0.4         | 78   | 0.40  | 0.24           |
| 5         | 1.31   | 0.32        | NR   | 0.28  | 0.20           |
| 6         | 1.1    | 0.1         | 86   | -0.56 | -0.81          |
| 7         | 1.45   | 0.44        | 86   | 0.85  | 0.45           |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | 1.20   | 0.230       | 91.2 | -0.16 | -0.15          |
| 12        | 1.48   | 0.236       | 84.7 | 0.97  | 0.87           |
| 13        | 1.31   | 0.066       | 107  | 0.28  | 0.45           |
| 15        | 1      | 1           | 97   | -0.97 | -0.24          |
| 16**      | 0.381  | 0.1143      | 188  | -3.46 | -4.75          |
| 17        | 1.45   | 0.36        | 94   | 0.85  | 0.54           |
| 18        | 1.4    | 0.5         | 156  | 0.65  | 0.31           |
| 19        | 1.3    | 0.33        | 92   | 0.24  | 0.17           |
| 20        | 1.26   | 0.25        | 73   | 0.08  | 0.07           |
| 21        | 1.0    | 0.2         | 86   | -0.97 | -0.98          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |       |      |
|--------------------------|-------|------|
| <b>Assigned Value</b>    | 1.24  | 0.14 |
| <b>Spike Value</b>       | 1.20  | 0.06 |
| <b>Homogeneity Value</b> | 1.20  | 0.36 |
| <b>Robust Average</b>    | 1.24  | 0.14 |
| <b>Median</b>            | 1.30  | 0.14 |
| <b>Mean</b>              | 1.24  |      |
| <b>N</b>                 | 15    |      |
| <b>Max</b>               | 1.48  |      |
| <b>Min</b>               | 0.935 |      |
| <b>Robust SD</b>         | 0.21  |      |
| <b>Robust CV</b>         | 17%   |      |

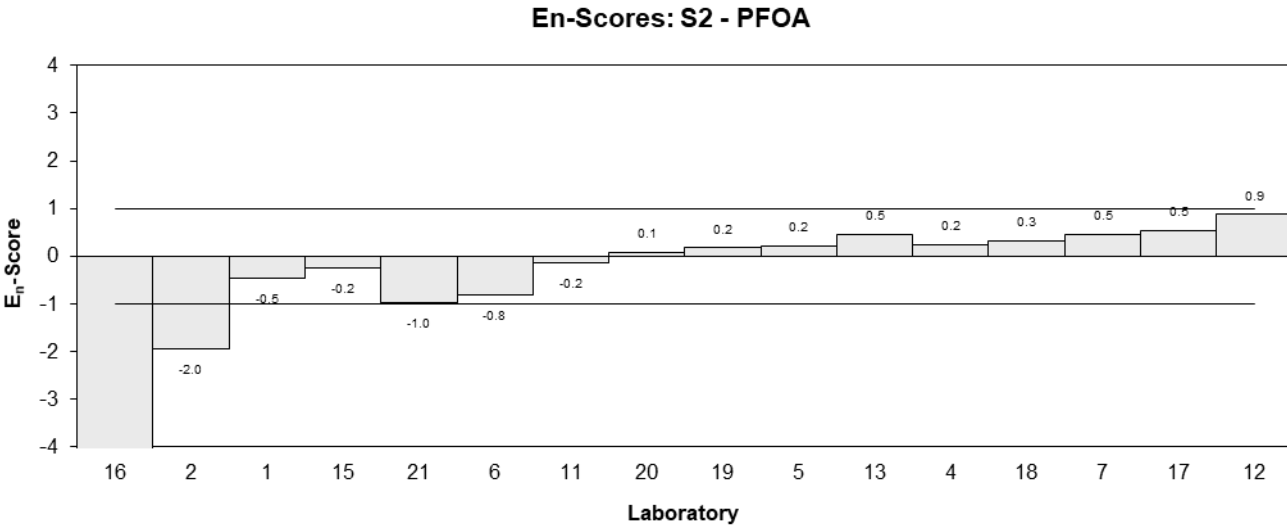
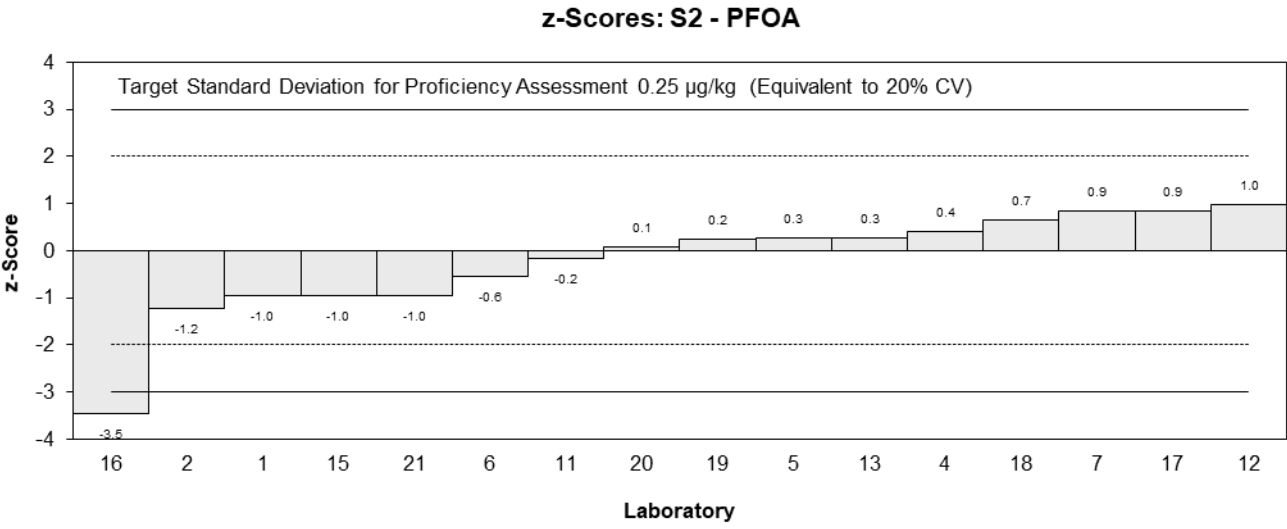
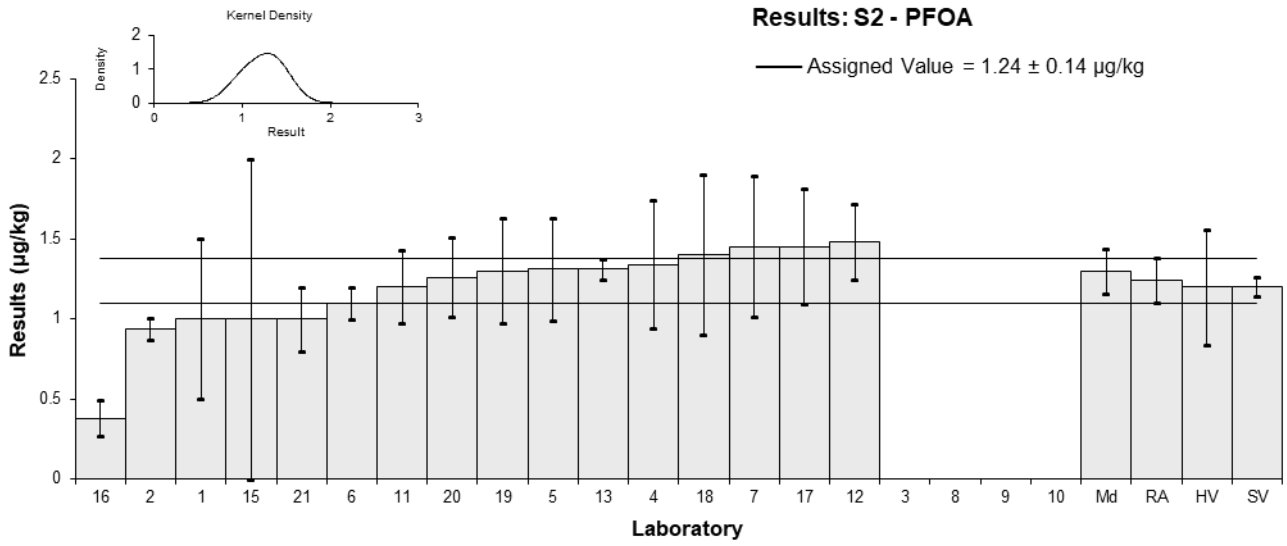


Figure 38

Table 43

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFNA   |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 2.0    | 1           | 124  | -0.76 | -0.34          |
| 2         | 1.69   | 0.220       | 85   | -1.42 | -1.80          |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 2.44   | 0.7         | 80   | 0.17  | 0.11           |
| 5         | 2.35   | 0.36        | NR   | -0.02 | -0.02          |
| 6         | 2.3    | 0.15        | NT   | -0.13 | -0.18          |
| 7         | 2.86   | 0.86        | 84   | 1.06  | 0.55           |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | 2.31   | 0.682       | 90.7 | -0.11 | -0.07          |
| 12        | 2.57   | 0.411       | 92.4 | 0.44  | 0.41           |
| 13        | 2.64   | 0.25        | 118  | 0.59  | 0.72           |
| 15        | 2      | 1           | 96   | -0.76 | -0.34          |
| 16**      | 0.652  | 0.1956      | 178  | -3.62 | -4.77          |
| 17        | 3.3    | 0.82        | 94   | 1.99  | 1.08           |
| 18        | 2.78   | 0.9         | 145  | 0.89  | 0.44           |
| 19        | 2.6    | 0.67        | 99   | 0.51  | 0.33           |
| 20        | 2.18   | 0.436       | 83   | -0.38 | -0.34          |
| 21        | 1.6    | 0.2         | NT   | -1.61 | -2.11          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 2.36 | 0.30 |
| <b>Spike Value</b>       | 2.31 | 0.12 |
| <b>Homogeneity Value</b> | 2.22 | 0.67 |
| <b>Robust Average</b>    | 2.36 | 0.30 |
| <b>Median</b>            | 2.35 | 0.28 |
| <b>Mean</b>              | 2.37 |      |
| <b>N</b>                 | 15   |      |
| <b>Max</b>               | 3.3  |      |
| <b>Min</b>               | 1.6  |      |
| <b>Robust SD</b>         | 0.46 |      |
| <b>Robust CV</b>         | 20%  |      |

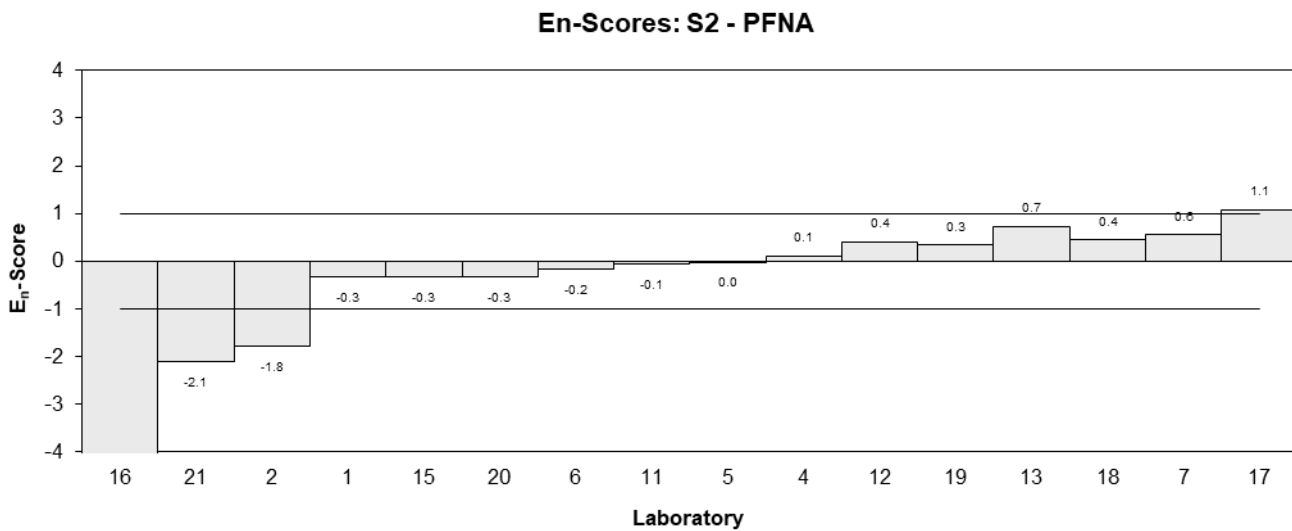
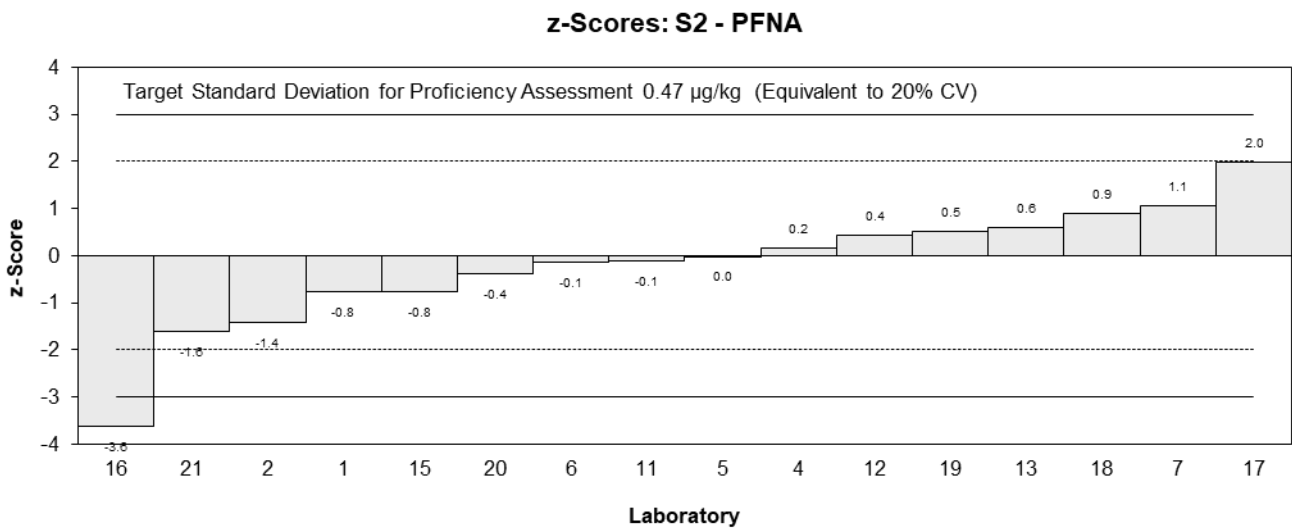
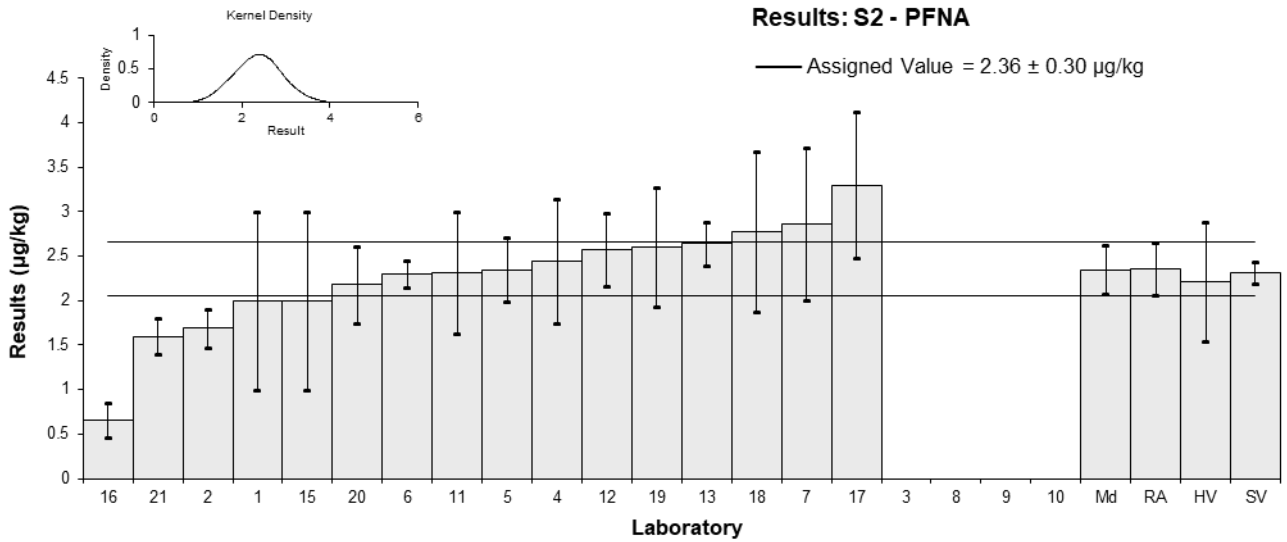


Figure 39

Table 44

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFDA   |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec   | z     | E <sub>n</sub> |
|-----------|--------|-------------|-------|-------|----------------|
| 1         | 8.9    | 4.5         | 102   | -0.35 | -0.15          |
| 2         | 7.72   | 1.04        | 85    | -0.97 | -1.41          |
| 3         | NS     | NS          | NS    |       |                |
| 4         | 11     | 3.3         | 80    | 0.74  | 0.42           |
| 5         | 8.89   | 2.3         | NR    | -0.36 | -0.28          |
| 6         | 9.2    | 0.5         | 84    | -0.20 | -0.40          |
| 7         | 9.88   | 3.0         | 81    | 0.16  | 0.10           |
| 8         | NS     | NS          | NS    |       |                |
| 9         | NT     | NT          | NT    |       |                |
| 10        | NS     | NS          | NS    |       |                |
| 11        | 9.97   | 2.70        | 101.6 | 0.20  | 0.14           |
| 12        | 11.1   | 1.66        | 91.7  | 0.79  | 0.82           |
| 13        | 9.42   | 0.241       | 145   | -0.08 | -0.19          |
| 15        | 11     | 4           | 93    | 0.74  | 0.35           |
| 16**      | 2.85   | 0.855       | 181   | -3.51 | -5.71          |
| 17        | 9.4    | 2.4         | 94    | -0.09 | -0.07          |
| 18        | 10.96  | 3.9         | 158   | 0.72  | 0.35           |
| 19        | 8.9    | 2.3         | 109   | -0.35 | -0.28          |
| 20        | 9.68   | 1.94        | 44    | 0.05  | 0.05           |
| 21        | 6.2    | 0.35        | 84    | -1.76 | -3.83          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 9.58 | 0.81 |
| <b>Spike Value</b>       | 9.47 | 0.47 |
| <b>Homogeneity Value</b> | 9.4  | 2.8  |
| <b>Robust Average</b>    | 9.58 | 0.81 |
| <b>Median</b>            | 9.42 | 0.51 |
| <b>Mean</b>              | 9.48 |      |
| <b>N</b>                 | 15   |      |
| <b>Max</b>               | 11.1 |      |
| <b>Min</b>               | 6.2  |      |
| <b>Robust SD</b>         | 1.3  |      |
| <b>Robust CV</b>         | 13%  |      |

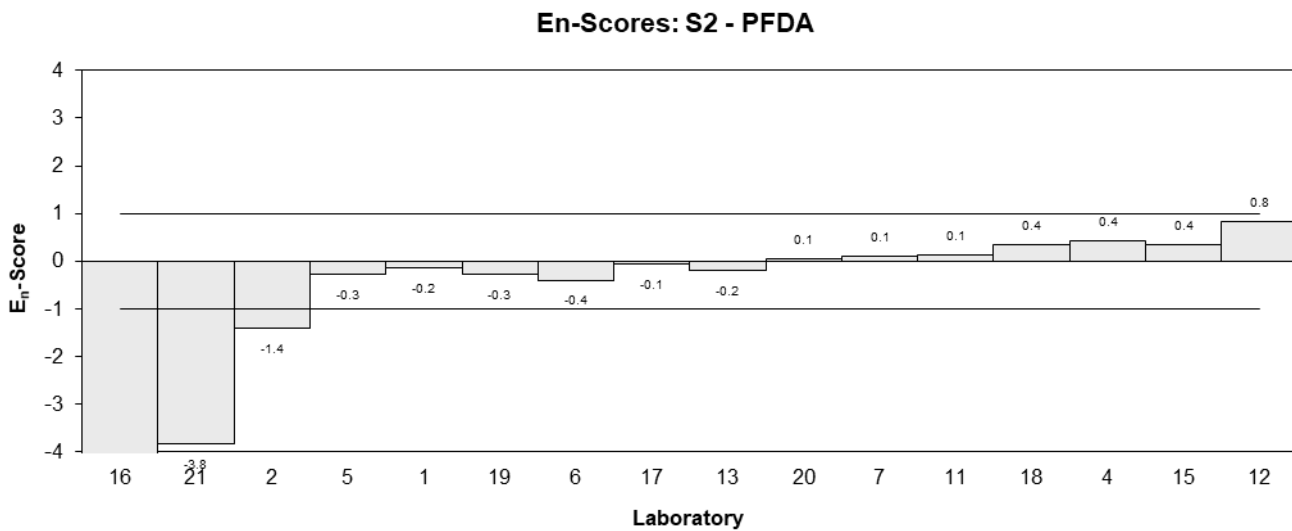
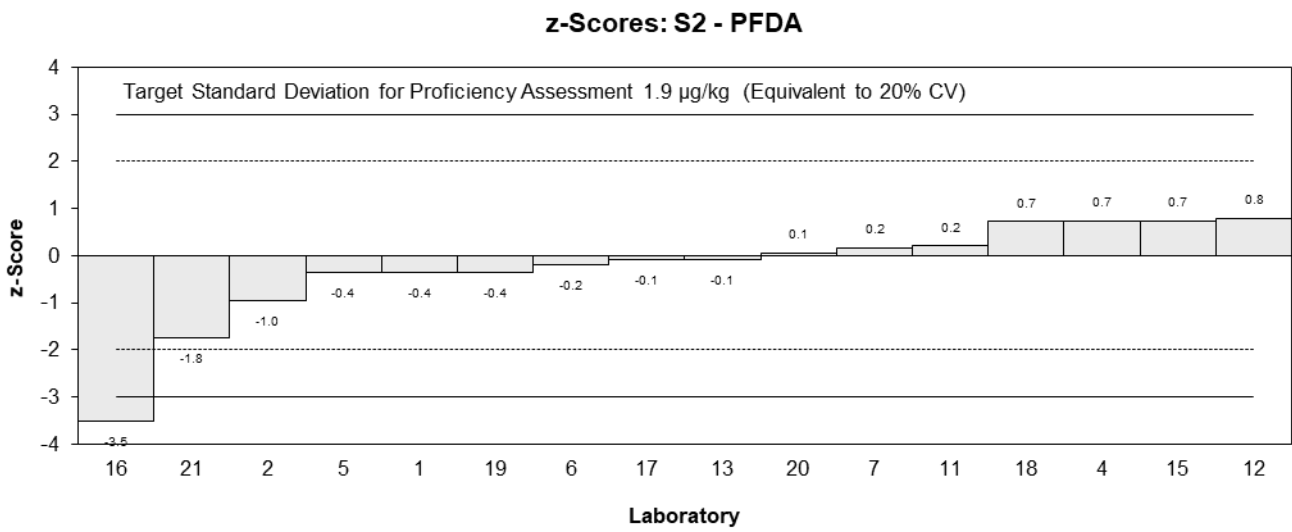
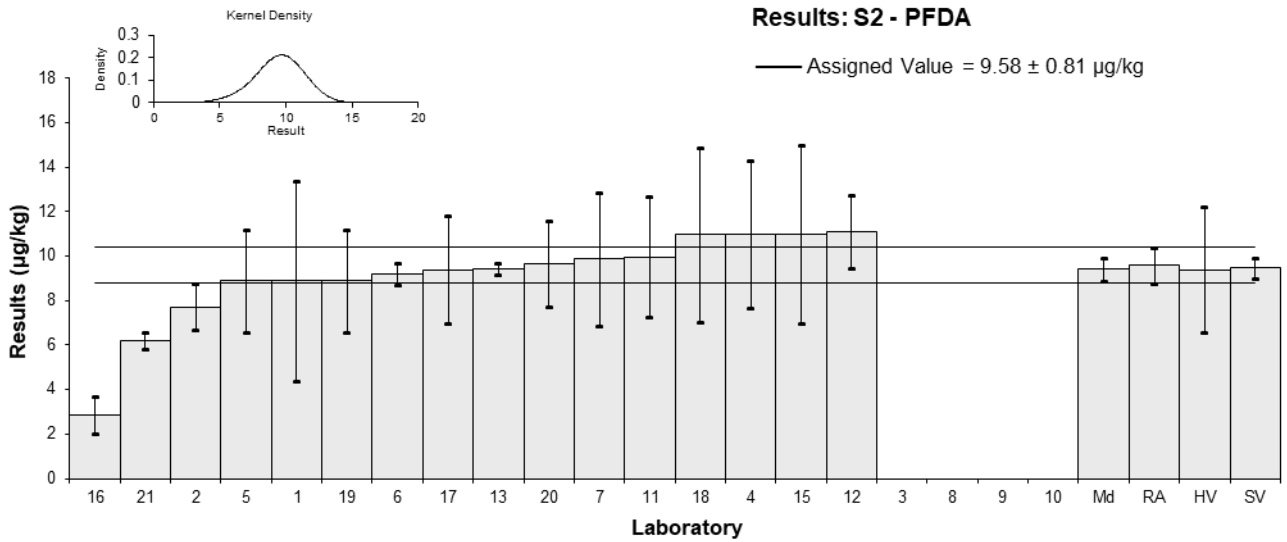


Figure 40

Table 45

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | PFOSA  |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 4      | 2           | 103  | -0.04 | -0.01          |
| 2         | 2.92   | 0.458       | 92   | -1.38 | -1.53          |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 4.41   | 1           | 85   | 0.47  | 0.33           |
| 5         | NT     | NT          | NT   |       |                |
| 6         | 3.8    | 0.2         | NT   | -0.29 | -0.39          |
| 7         | 4.12   | 1.2         | 91   | 0.11  | 0.07           |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | 3.54   | 0.658       | 41.6 | -0.61 | -0.57          |
| 12        | 4.64   | 0.651       | 108  | 0.76  | 0.71           |
| 13        | 4.04   | 0.198       | 87   | 0.01  | 0.02           |
| 15        | 5      | 2           | 93   | 1.20  | 0.47           |
| 16**      | 1.19   | 0.357       | 105  | -3.52 | -4.28          |
| 17        | 4.9    | 1.2         | 94   | 1.08  | 0.66           |
| 18        | <5     | NR          | 121  |       |                |
| 19        | 4.1    | 0.76        | 102  | 0.09  | 0.07           |
| 20        | NT     | NT          | NT   |       |                |
| 21        | 2.4    | 0.3         | NT   | -2.02 | -2.57          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 4.03 | 0.56 |
| <b>Spike Value</b>       | 4.95 | 0.25 |
| <b>Homogeneity Value</b> | 4.2  | 1.3  |
| <b>Robust Average</b>    | 4.03 | 0.56 |
| <b>Median</b>            | 4.07 | 0.47 |
| <b>Mean</b>              | 3.99 |      |
| <b>N</b>                 | 12   |      |
| <b>Max</b>               | 5    |      |
| <b>Min</b>               | 2.4  |      |
| <b>Robust SD</b>         | 0.77 |      |
| <b>Robust CV</b>         | 19%  |      |



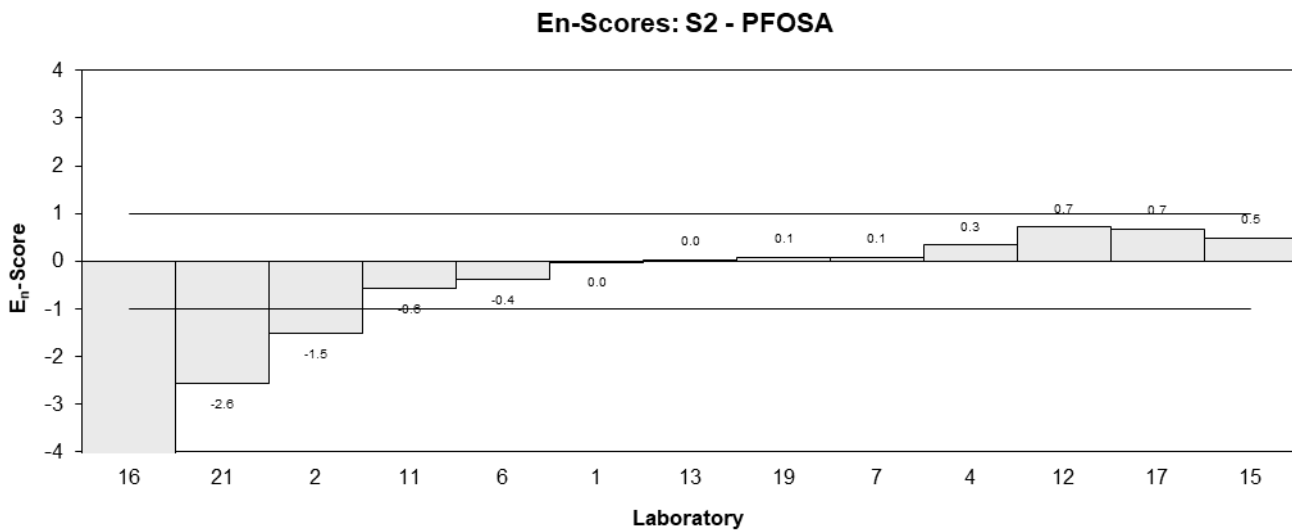
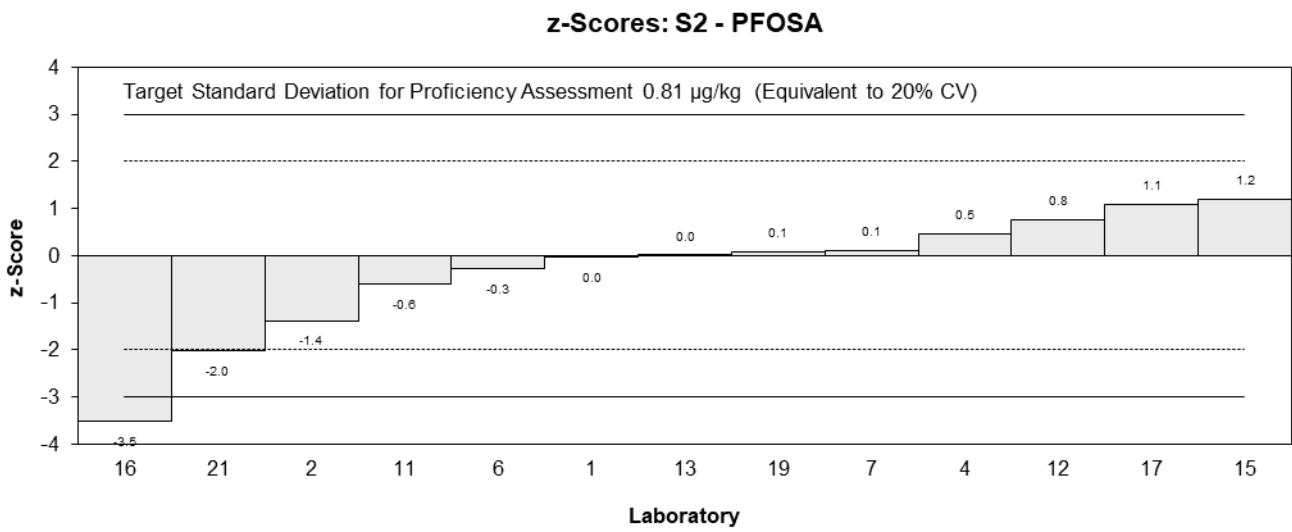
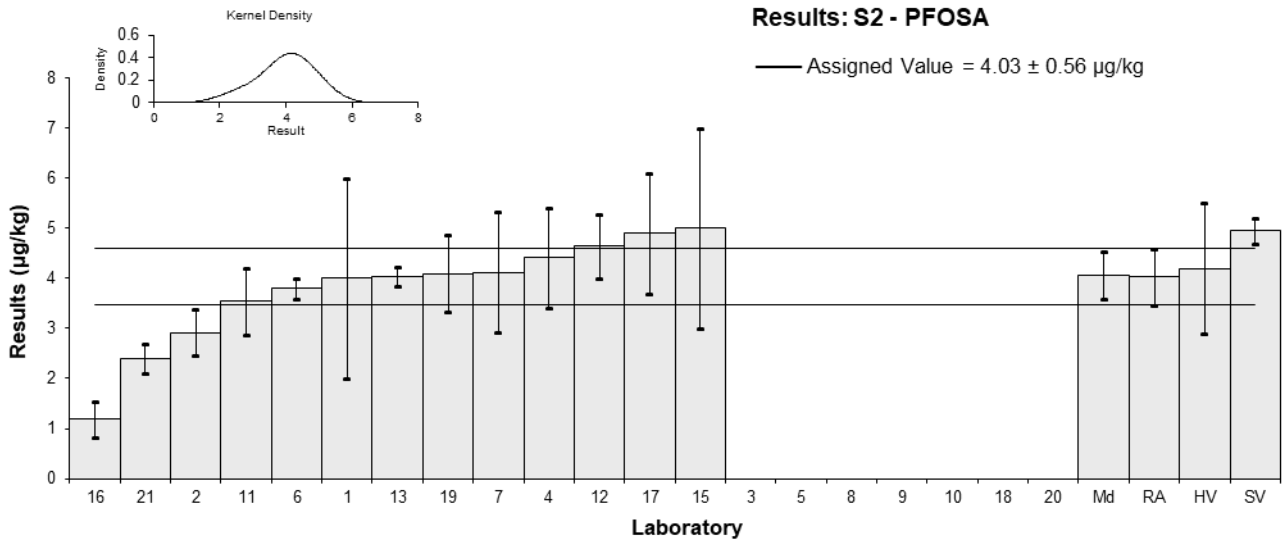


Figure 41

Table 46

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | MeFOSA |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 3      | 1.5         | 78   | -1.23 | -0.57          |
| 2         | NT     | NT          | NT   |       |                |
| 3         | NS     | NS          | NS   |       |                |
| 4         | <5     | NR          | 36   |       |                |
| 5         | NT     | NT          | NT   |       |                |
| 6         | 5.1    | 0.3         | NT   | 1.41  | 1.30           |
| 7         | 3.15   | 0.95        | 87   | -1.04 | -0.66          |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11*       | 0.620  | 0.215       | 41.6 | -4.22 | -4.01          |
| 12        | 4.8    | 0.651       | 93.5 | 1.03  | 0.79           |
| 13        | 3.91   | 0.34        | 45   | -0.09 | -0.08          |
| 15        | 5      | 2           | 90   | 1.28  | 0.47           |
| 16**      | 1.86   | 0.558       | 105  | -2.66 | -2.16          |
| 17        | 3.6    | 0.89        | 94   | -0.48 | -0.32          |
| 18        | <5     | NR          | 128  |       |                |
| 19        | 4.3    | 1.2         | 100  | 0.40  | 0.22           |
| 20        | NT     | NT          | NT   |       |                |
| 21        | 3.0    | 0.2         | NT   | -1.23 | -1.17          |

\* Outlier, \*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |      |      |
|-----------------------|------|------|
| <b>Assigned Value</b> | 3.98 | 0.81 |
| <b>Spike Value</b>    | 4.99 | 0.25 |
| <b>Robust Average</b> | 3.79 | 0.90 |
| <b>Median</b>         | 3.76 | 0.89 |
| <b>Mean</b>           | 3.65 |      |
| <b>N</b>              | 10   |      |
| <b>Max</b>            | 5.1  |      |
| <b>Min</b>            | 0.62 |      |
| <b>Robust SD</b>      | 1.1  |      |
| <b>Robust CV</b>      | 30%  |      |

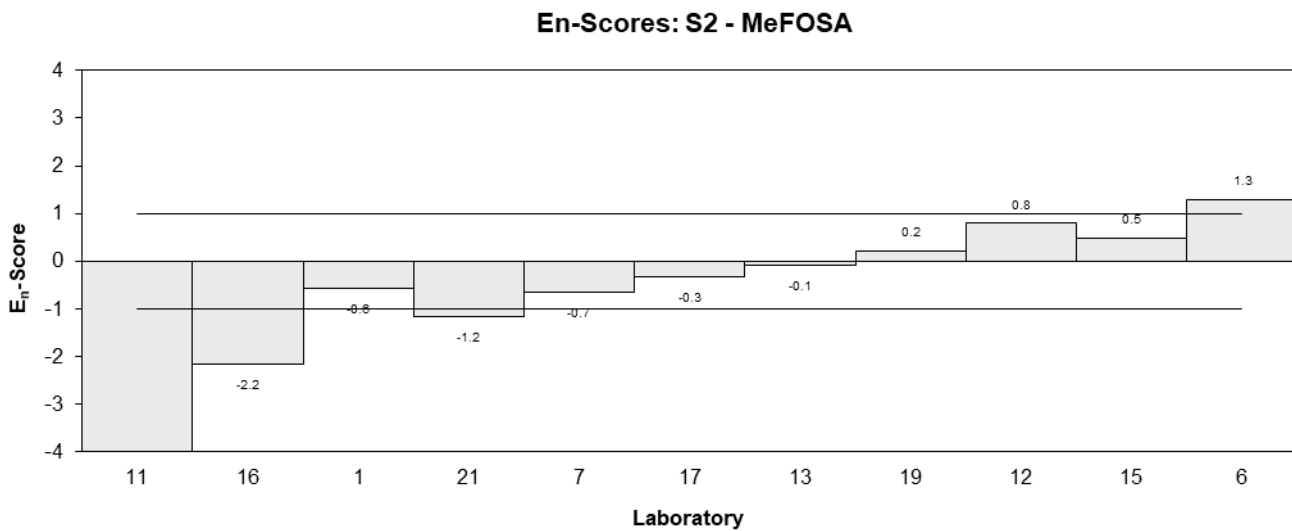
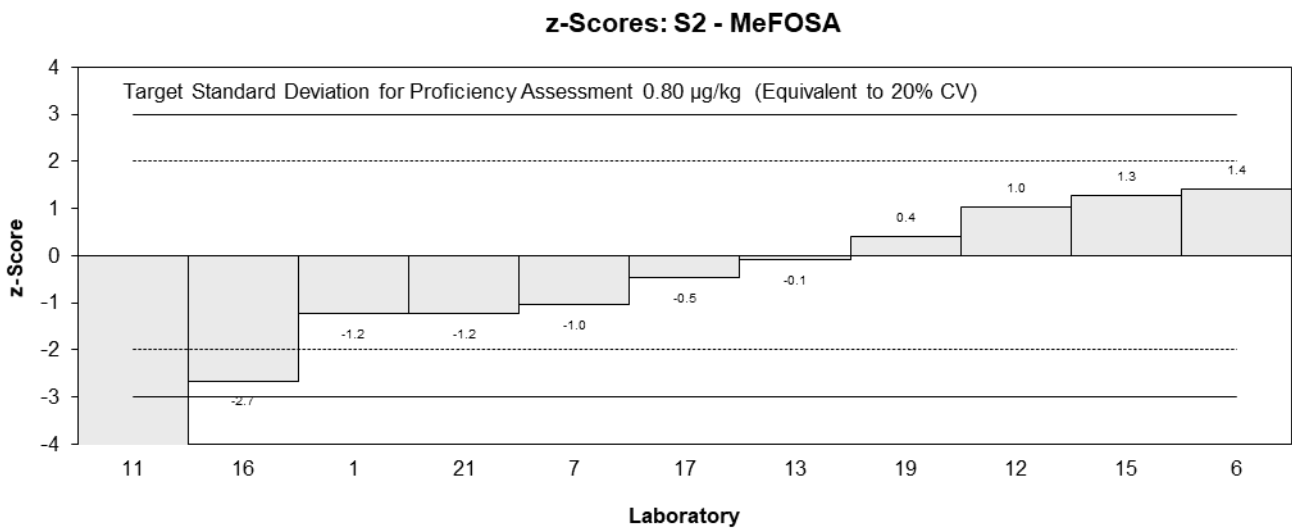
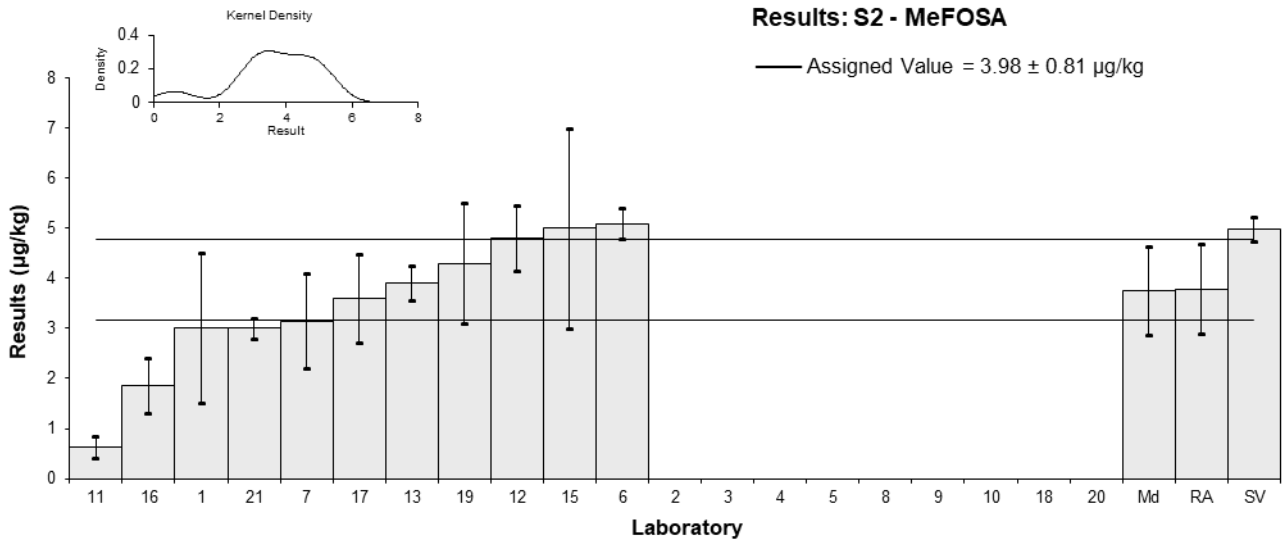


Figure 42

Table 47

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <b>Analyte</b>    | EtFOSA |
| <b>Unit</b>       | µg/kg  |

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 3      | 1.5         | 89   | -0.18 | -0.07          |
| 2         | NT     | NT          | NT   |       |                |
| 3         | NS     | NS          | NS   |       |                |
| 4         | <5     | NR          | 24   |       |                |
| 5         | NT     | NT          | NT   |       |                |
| 6*        | 5.1    | 0.3         | NT   | 3.20  | 3.13           |
| 7         | 2.06   | 0.62        | 89   | -1.69 | -1.26          |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11*       | 0.610  | 0.2         | 41.6 | -4.02 | -4.20          |
| 12        | 4.35   | 0.696       | 73.6 | 1.99  | 1.39           |
| 13        | 3.11   | 0.201       | 44   | 0.00  | 0.00           |
| 15        | <5     | NR          | 92   |       |                |
| 16**      | 1.63   | 0.489       | 92.4 | -2.38 | -1.99          |
| 17        | 2.8    | 0.71        | 94   | -0.50 | -0.34          |
| 18        | 2.94   | 1.1         | 105  | -0.27 | -0.14          |
| 19        | 3.6    | 0.61        | 100  | 0.79  | 0.59           |
| 20        | NT     | NT          | NT   |       |                |
| 21        | 3.2    | 0.2         | NT   | 0.14  | 0.15           |

\* Outlier, \*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                       |      |      |
|-----------------------|------|------|
| <b>Assigned Value</b> | 3.11 | 0.56 |
| <b>Spike Value</b>    | 3.99 | 0.20 |
| <b>Robust Average</b> | 3.13 | 0.86 |
| <b>Median</b>         | 3.06 | 0.47 |
| <b>Mean</b>           | 3.08 |      |
| <b>N</b>              | 10   |      |
| <b>Max</b>            | 5.1  |      |
| <b>Min</b>            | 0.61 |      |
| <b>Robust SD</b>      | 1.1  |      |
| <b>Robust CV</b>      | 35%  |      |

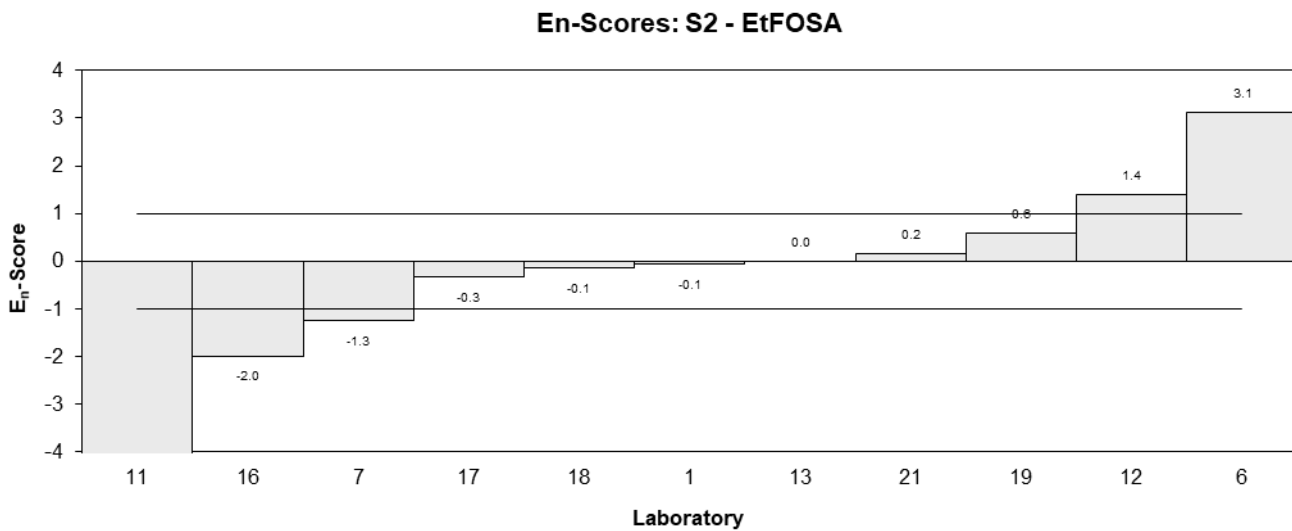
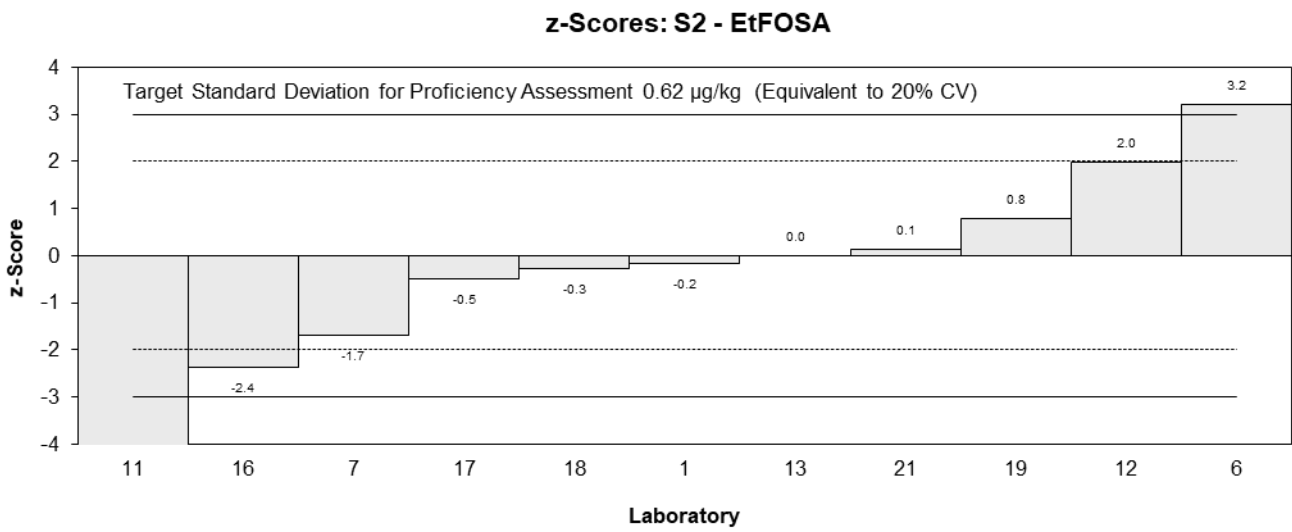
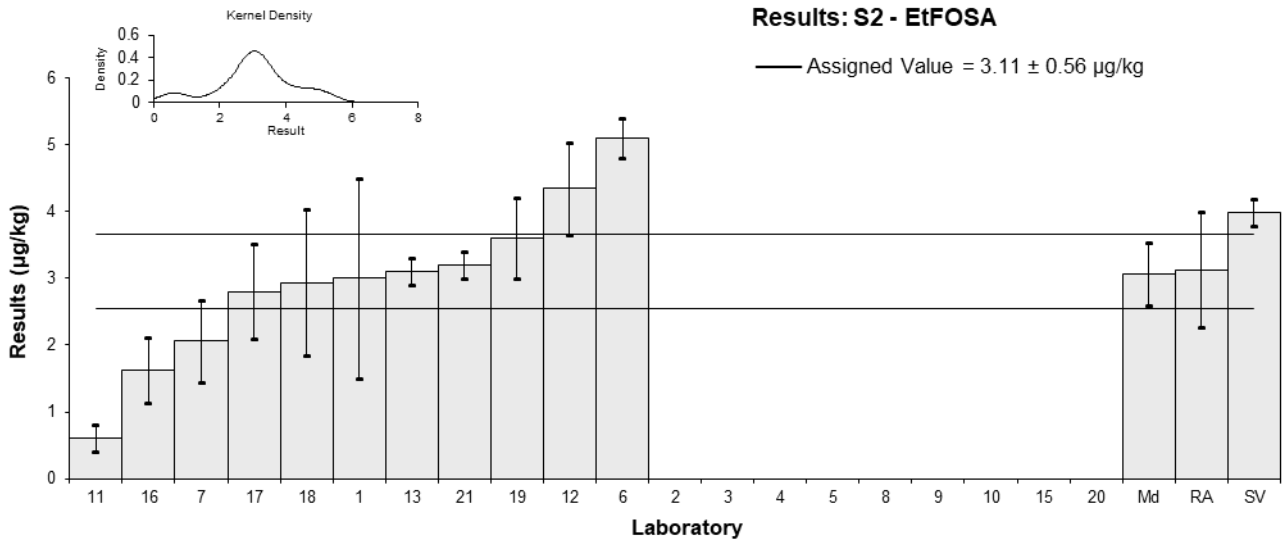


Figure 43

Table 48

## Sample Details

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

## Participant Results

| Lab. Code | Result | Uncertainty | Rec  | z     | E <sub>n</sub> |
|-----------|--------|-------------|------|-------|----------------|
| 1         | 1      | 0.5         | 313  | -2.04 | -1.23          |
| 2         | 1.62   | 0.142       | 63   | -0.21 | -0.24          |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 1.82   | 0.5         | 59   | 0.38  | 0.23           |
| 5         | NT     | NT          | NT   |       |                |
| 6         | 1.9    | 0.1         | NT   | 0.62  | 0.78           |
| 7         | 1.65   | 0.50        | 91   | -0.12 | -0.07          |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | NT     | NT          | NT   |       |                |
| 12        | 2.02   | 1.09        | 92.3 | 0.98  | 0.30           |
| 13        | 1.54   | 0.08        | 228  | -0.44 | -0.57          |
| 15        | 2      | 1           | 95   | 0.92  | 0.30           |
| 16**      | 0.618  | 0.1854      | 391  | -3.17 | -3.44          |
| 17        | 2.0    | 0.51        | 94   | 0.92  | 0.55           |
| 18        | 2.02   | 0.8         | 173  | 0.98  | 0.39           |
| 19        | 1.7    | 0.26        | 73   | 0.03  | 0.03           |
| 20        | 1.35   | 0.271       | 175  | -1.01 | -0.92          |
| 21        | 1.1    | 0.1         | NT   | -1.75 | -2.19          |

\*\* Not included in robust average calculations, see Section 4.2

## Statistics

|                          |      |      |
|--------------------------|------|------|
| <b>Assigned Value</b>    | 1.69 | 0.25 |
| <b>Spike Value</b>       | 1.89 | 0.09 |
| <b>Homogeneity Value</b> | 1.91 | 0.57 |
| <b>Robust Average</b>    | 1.69 | 0.25 |
| <b>Median</b>            | 1.70 | 0.31 |
| <b>Mean</b>              | 1.67 |      |
| <b>N</b>                 | 13   |      |
| <b>Max</b>               | 2.02 |      |
| <b>Min</b>               | 1    |      |
| <b>Robust SD</b>         | 0.36 |      |
| <b>Robust CV</b>         | 21%  |      |

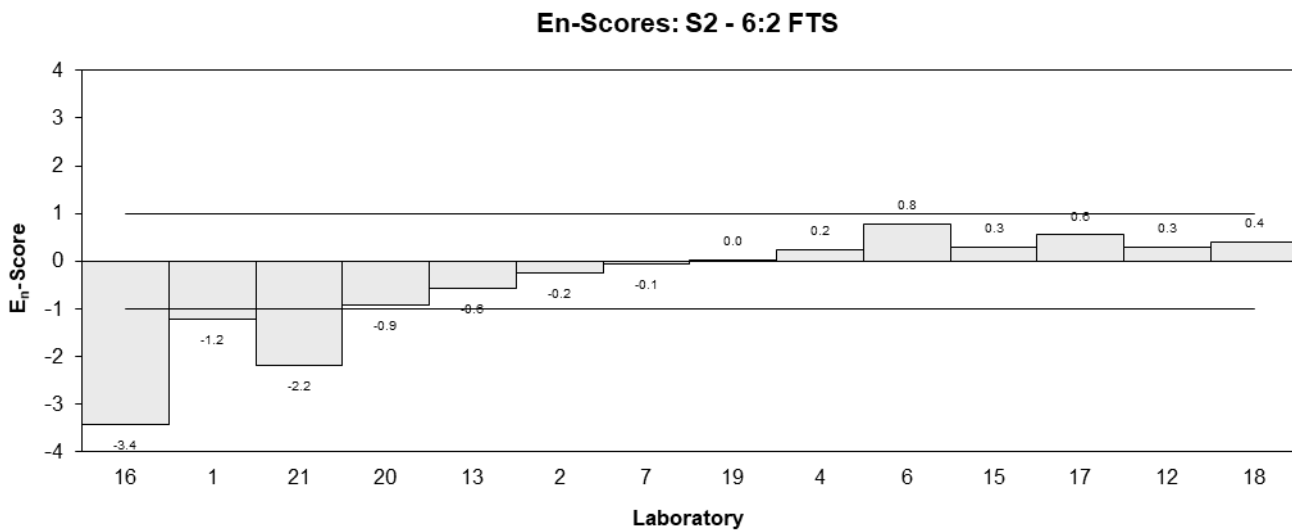
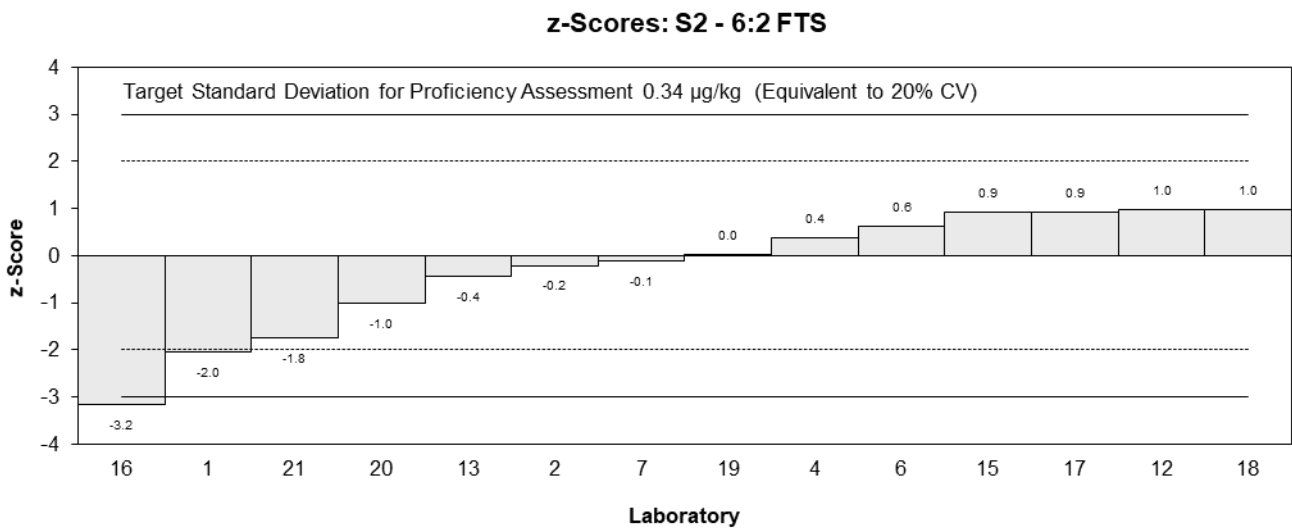
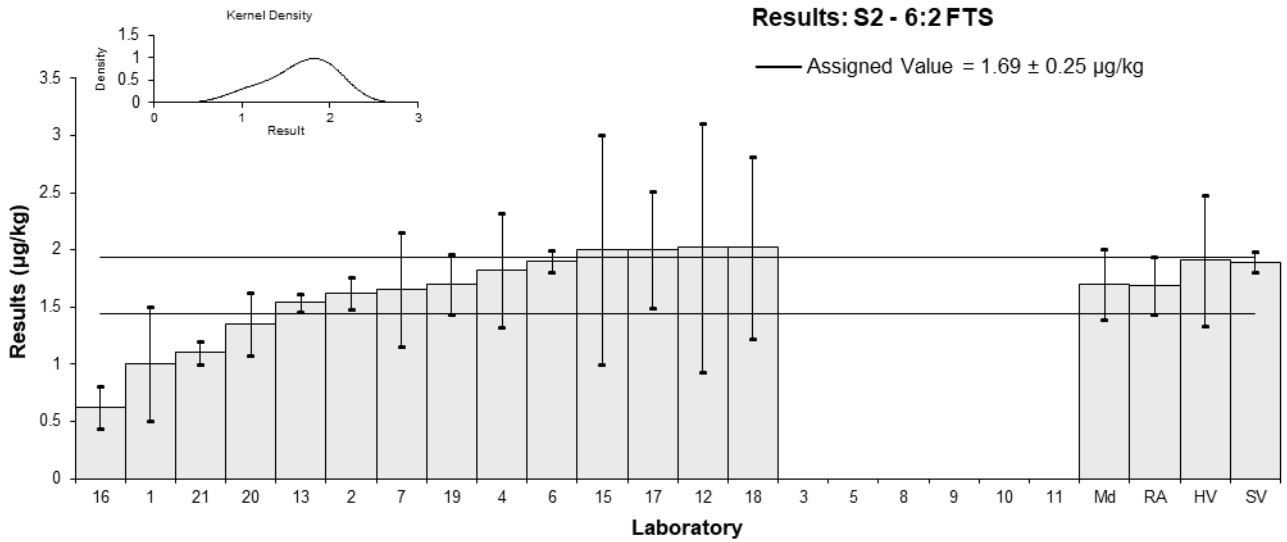


Figure 44

Table 49

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <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         | 7.34   | 0.427       | 99   | -1.26 | -1.58          |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 11.4   | 3           | 76   | 0.82  | 0.48           |
| 5         | NT     | NT          | NT   |       |                |
| 6         | <1     | NR          | NT   |       |                |
| 7         | 7.86   | 2.4         | 70   | -0.99 | -0.69          |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | NT     | NT          | NT   |       |                |
| 12        | 11     | 2.3         | 81.1 | 0.61  | 0.44           |
| 13*       | 4.19   | 0.382       | 107  | -2.86 | -3.62          |
| 15        | 10     | 5           | 89   | 0.10  | 0.04           |
| 16        | NR     | NR          | NR   |       |                |
| 17        | 10     | 5           | 94   | 0.10  | 0.04           |
| 18        | NT     | NT          | NT   |       |                |
| 19        | 11     | 0.72        | 76   | 0.61  | 0.72           |
| 20        | 9.90   | 1.98        | 36   | 0.05  | 0.04           |
| 21        | <1     | NR          | NT   |       |                |

\* Outlier, see Section 4.2

## Statistics

|                          |      |     |
|--------------------------|------|-----|
| <b>Assigned Value</b>    | 9.8  | 1.5 |
| <b>Spike Value</b>       | 11.1 | 0.6 |
| <b>Homogeneity Value</b> | 10.3 | 3.1 |
| <b>Robust Average</b>    | 9.4  | 1.7 |
| <b>Median</b>            | 10.0 | 1.2 |
| <b>Mean</b>              | 9.2  |     |
| <b>N</b>                 | 9    |     |
| <b>Max</b>               | 11.4 |     |
| <b>Min</b>               | 4.19 |     |
| <b>Robust SD</b>         | 2.0  |     |
| <b>Robust CV</b>         | 22%  |     |



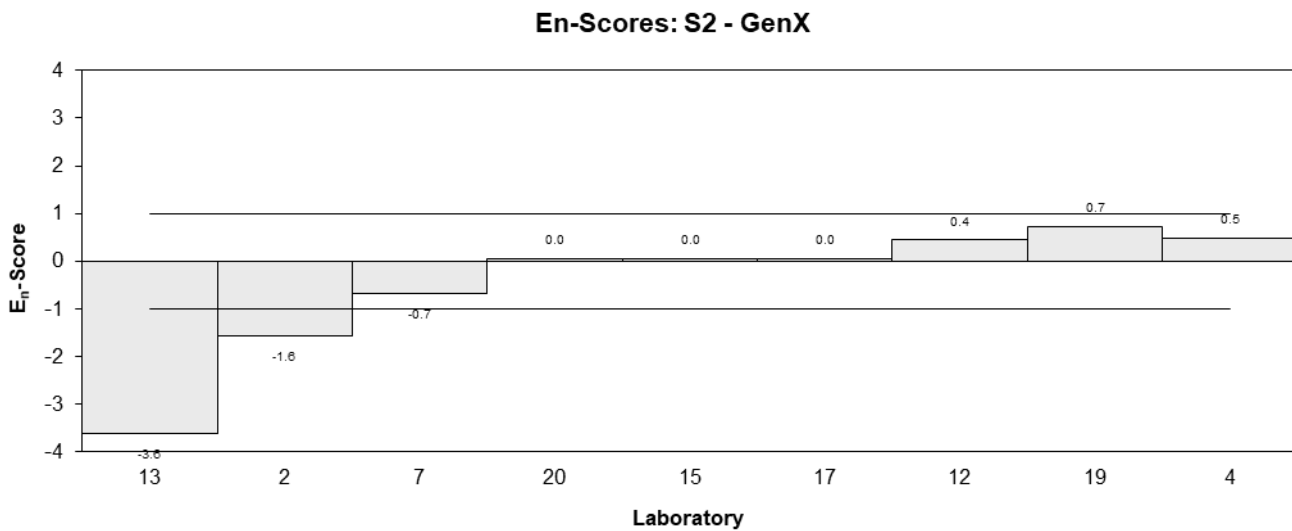
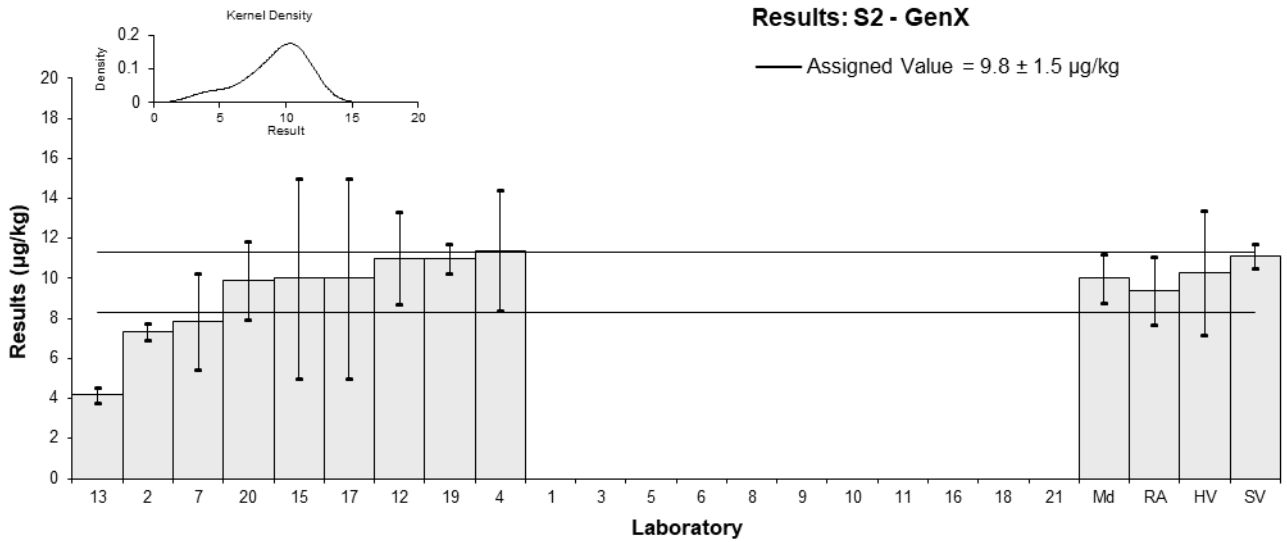


Figure 45

Table 50

## Sample Details

|                   |        |
|-------------------|--------|
| <b>Sample No.</b> | S2     |
| <b>Matrix</b>     | Carrot |
| <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         | 10.8   | 0.783       | 85   | -0.91 | -0.99          |
| 3         | NS     | NS          | NS   |       |                |
| 4         | 16.4   | 5           | NR   | 1.21  | 0.58           |
| 5         | NT     | NT          | NT   |       |                |
| 6         | <1     | NR          | NT   |       |                |
| 7         | 11.2   | 3.4         | 81   | -0.76 | -0.49          |
| 8         | NS     | NS          | NS   |       |                |
| 9         | NT     | NT          | NT   |       |                |
| 10        | NS     | NS          | NS   |       |                |
| 11        | NT     | NT          | NT   |       |                |
| 12        | 15.1   | 4.07        | 81.1 | 0.72  | 0.41           |
| 13        | 9.98   | 0.456       | 107  | -1.22 | -1.37          |
| 15        | 15     | 5           | 89   | 0.68  | 0.33           |
| 16        | NR     | NR          | NR   |       |                |
| 17        | 13     | 6           | 94   | -0.08 | -0.03          |
| 18        | NT     | NT          | NT   |       |                |
| 19        | 14     | 2.7         | 99   | 0.30  | 0.23           |
| 20        | NT     | NT          | NT   |       |                |
| 21        | NT     | NT          | NT   |       |                |

## Statistics

|                          |      |     |
|--------------------------|------|-----|
| <b>Assigned Value</b>    | 13.2 | 2.3 |
| <b>Spike Value</b>       | 14.0 | 0.7 |
| <b>Homogeneity Value</b> | 16.1 | 4.8 |
| <b>Robust Average</b>    | 13.2 | 2.3 |
| <b>Median</b>            | 13.5 | 2.6 |
| <b>Mean</b>              | 13.2 |     |
| <b>N</b>                 | 8    |     |
| <b>Max</b>               | 16.4 |     |
| <b>Min</b>               | 9.98 |     |
| <b>Robust SD</b>         | 2.6  |     |
| <b>Robust CV</b>         | 20%  |     |

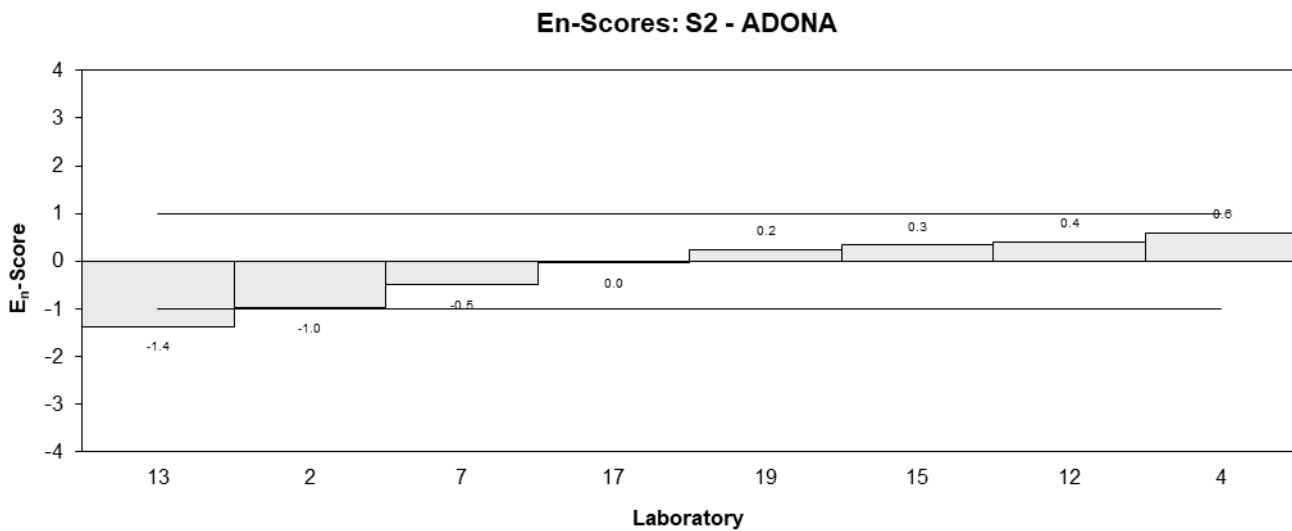
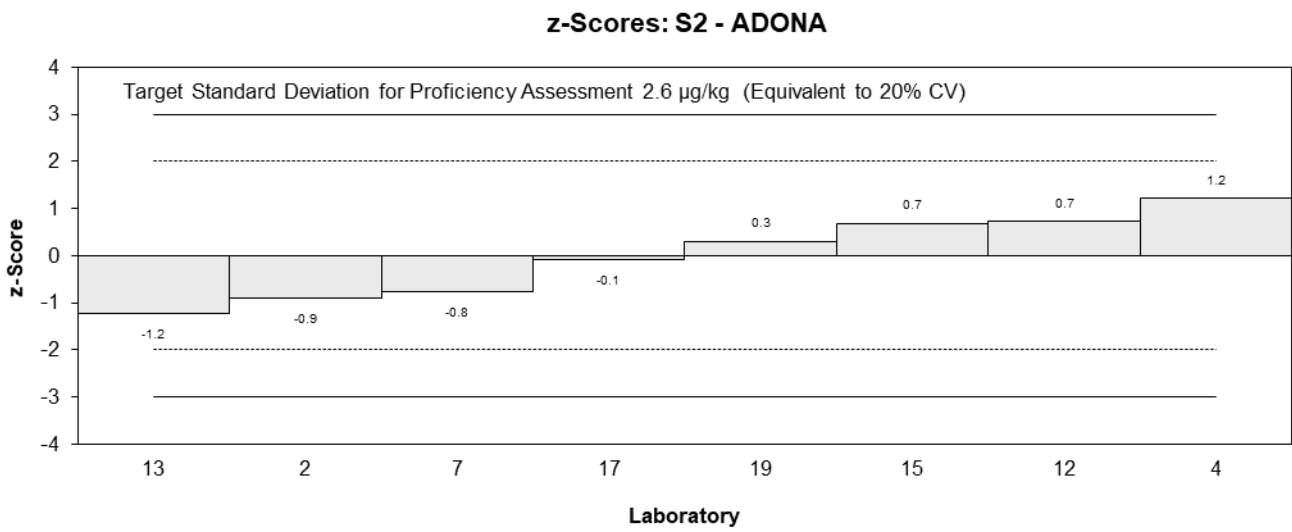
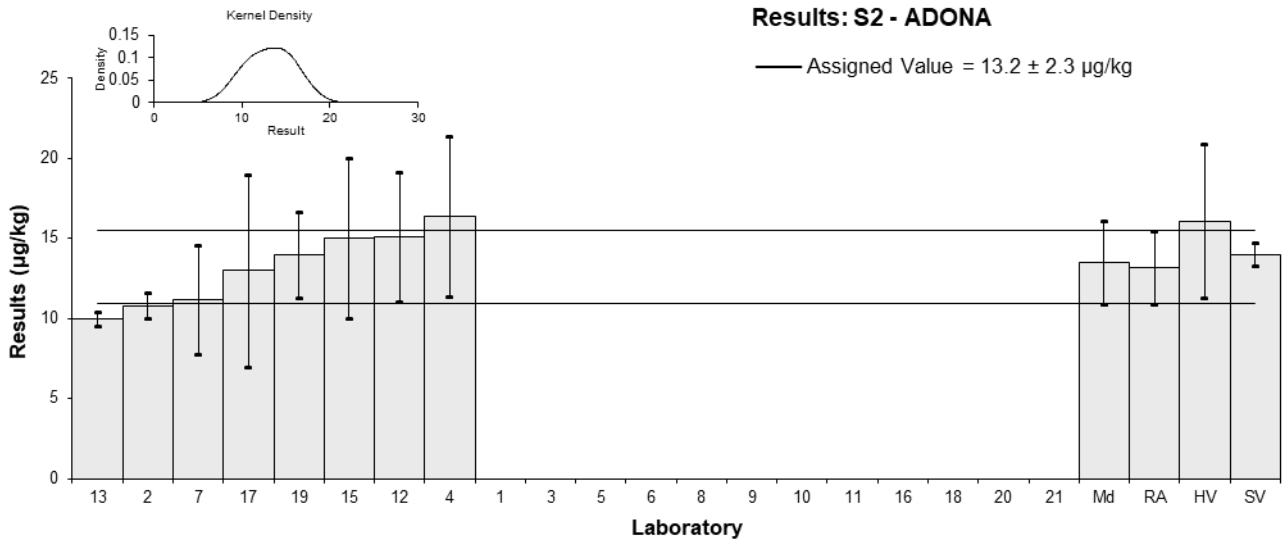


Figure 46

## 6 DISCUSSION OF RESULTS

### 6.1 Assigned Value

The robust averages of participants' results were used as the assigned values for scored analytes. The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528:2022.<sup>6</sup> Outliers, gross errors and results not included in robust average calculations were removed before the calculation of the assigned value (see Section 4.2 for additional information).<sup>3,4</sup> The calculation of the expanded uncertainty for the robust average is presented in Appendix 3, using Sample S2 PFHpS 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 S1 PFTTrDA, PFOSA, MeFOSA, EtFOSA, 11Cl-PF3OUdS, and Sample S2 PFBA, as reported results were too variable and recoveries were relatively low. 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 these analytes without assigned values, 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 51. For this study, the assigned values for scored analytes were within 69% to 93% and 78% to 111% of the spiked values for Samples S1 and S2 respectively. These recoveries are similar to previous NMI PFAS in biota and food PT studies, and provides good support for the assigned values and analyte stability.

Table 51 Comparison of Assigned Values (*Robust Averages*) and Spiked Values

| Sample        | Analyte        | Assigned Value ( <i>Robust Average</i> )<br>(µg/kg) | Spiked Value<br>(µg/kg) | Assigned Value ( <i>Robust Average</i> ) / Spiked Value<br>(%) |
|---------------|----------------|---|-------------------------|--|
| S1<br>(Prawn) | PFBS           | 0.302   | 0.399                   | 76   |
|               | PFPeS          | 4.19  | 4.65                    | 90   |
|               | PFHxS          | 1.64  | 1.89                    | 87   |
|               | PFHxS (linear) | 1.61  | 1.89                    | 85   |
|               | PFHpS          | 1.67  | 2.00                    | 84   |
|               | PFOS           | 3.62  | 4.77                    | 76   |
|               | PFOS (linear)  | 3.74  | 4.77                    | 78   |
|               | PFNS           | 8.0   | 11.5                    | 70   |
|               | PFBA           | 2.03  | 2.96                    | 69   |
|               | PFPeA          | 1.04  | 1.13                    | 92   |
|               | PFHxA          | 4.61  | 5.31                    | 87   |
|               | PFHpA          | 6.02  | 7.54                    | 80   |
|               | PFOA           | 6.44  | 7.92                    | 81   |
|               | PFNA           | 0.427   | 0.503                   | 85   |
|               | PFDA           | 0.74  | 0.902                   | 82   |
|               | PFUdA          | 1.12  | 1.21                    | 93   |
| PFTTrDA       | (5.4)          | 8.17  | (66)                    |  |

| Sample         | Analyte        | Assigned Value ( <i>Robust Average</i> ) (µg/kg) | Spiked Value (µg/kg) | Assigned Value ( <i>Robust Average</i> ) / Spiked Value (%) |
|----------------|----------------|--|----------------------|---|
|                | PFOSA          | (3.00)   | 4.46                 | (67)  |
|                | MeFOSA         | (3.7)  | 4.99                 | (74)  |
|                | EtFOSA         | (2.66)   | 3.99                 | (67)  |
|                | ADONA          | 4.5  | 5.64                 | 80  |
|                | 9Cl-PF3ONS     | 11.0   | 14.4                 | 76  |
|                | 11Cl-PF3OUdS   | (2.27)   | 4.70                 | (48)  |
| S2<br>(Carrot) | PFBS           | 0.82   | 0.891                | 92  |
|                | PFPeS          | 8.30   | 7.47                 | 111   |
|                | PFHxS          | 6.54   | 6.61                 | 99  |
|                | PFHxS (linear) | 6.44   | 6.61                 | 97  |
|                | PFHpS          | 2.99   | 3.00                 | 100   |
|                | PFOS           | 1.95   | 2.12                 | 92  |
|                | PFOS (linear)  | 2.01   | 2.12                 | 95  |
|                | PFNS           | 1.52   | 1.72                 | 88  |
|                | PFDS           | 6.76   | 6.80                 | 99  |
|                | PFBA           | (0.78)   | 1.19                 | (66)  |
|                | PFPeA          | 2.07   | 2.20                 | 94  |
|                | PFHxA          | 7.83   | 7.45                 | 105   |
|                | PFHpA          | 1.53   | 1.50                 | 102   |
|                | PFOA           | 1.24   | 1.20                 | 103   |
|                | PFNA           | 2.36   | 2.31                 | 102   |
|                | PFDA           | 9.58   | 9.47                 | 101   |
|                | PFOSA          | 4.03   | 4.95                 | 81  |
|                | MeFOSA         | 3.98   | 4.99                 | 80  |
|                | EtFOSA         | 3.11   | 3.99                 | 78  |
|                | 6:2 FTS        | 1.69   | 1.89                 | 89  |
| GenX           | 9.8            | 11.1   | 88                   |   |
| ADONA          | 13.2           | 14.0   | 94                   |   |

## 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 597 numeric results reported for spiked analytes in this study, 580 (97%) were reported with an uncertainty. Laboratory 1 did not report uncertainties for linear isomers PFHxS and PFOS in Sample S2 only (uncertainties were reported for these analytes in Sample S1); this participant reported they were accredited to ISO/IEC 17025. Laboratory 3 did not report any uncertainties; this participant reported that they were not accredited.

Laboratories **1** and **8** attached an estimate of MU to at least one non-numeric result reported. 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 3. Three participants reported using the NATA GAG Estimating and Reporting MU as their guide; NATA no longer publishes this document.<sup>10</sup>

The magnitude of the MUs for analytes in this study was within the range 1.8% 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 580 MUs, 436 (75%) were between 10% and 50% relative, 126 were less than 10% relative and 18 were greater than 50% relative.

Uncertainties associated with results returning a satisfactory *z*-score but an unsatisfactory *E<sub>n</sub>*-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  $4.51 \pm 1.2822 \mu\text{g/kg}$ , it is better to report this as  $4.5 \pm 1.3 \mu\text{g/kg}$ .<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 52.

Table 52 Comparison of Thompson-Horwitz CVs, Between-Laboratory CVs, and Target SDs

| Sample        | Analyte        | Assigned Value (µg/kg) | Thompson-Horwitz CV (%) | Between-Laboratory CV* (%) | Target SD (as PCV) (%) |
|---------------|----------------|------------------------|-------------------------|----------------------------|------------------------|
| S1<br>(Prawn) | PFBS           | 0.302                  | 22                      | 16                         | 20                     |
|               | PFPeS          | 4.19                   | 22                      | 21                         | 20                     |
|               | PFHxS          | 1.64                   | 22                      | 21                         | 20                     |
|               | PFHxS (linear) | 1.61                   | 22                      | 21                         | 20                     |
|               | PFHpS          | 1.67                   | 22                      | 27                         | 20                     |
|               | PFOS           | 3.62                   | 22                      | 27                         | 20                     |
|               | PFOS (linear)  | 3.74                   | 22                      | 26                         | 20                     |
|               | PFNS           | 8.0                    | 22                      | 33                         | 20                     |
|               | PFBA           | 2.03                   | 22                      | 29                         | 20                     |
|               | PFPeA          | 1.04                   | 22                      | 14                         | 20                     |
|               | PFHxA          | 4.61                   | 22                      | 19                         | 20                     |
|               | PFHpA          | 6.02                   | 22                      | 17                         | 20                     |
|               | PFOA           | 6.44                   | 22                      | 18                         | 20                     |
|               | PFNA           | 0.427                  | 22                      | 20                         | 20                     |
|               | PFDA           | 0.74                   | 22                      | 25                         | 20                     |
|               | PFUdA          | 1.12                   | 22                      | 27                         | 20                     |
|               | PFTTrDA        | Not Set                | NA                      | 61                         | Not Set                |
| PFOSA         | Not Set        | NA                     | 37                      | Not Set                    |                        |

| Sample         | Analyte        | Assigned Value (µg/kg) | Thompson-Horwitz CV (%) | Between-Laboratory CV* (%) | Target SD (as PCV) (%) |
|----------------|----------------|------------------------|-------------------------|----------------------------|------------------------|
|                | MeFOSA         | Not Set                | NA                      | 37                         | Not Set                |
|                | EtFOSA         | Not Set                | NA                      | 41                         | Not Set                |
|                | ADONA          | 4.5                    | 22                      | 28                         | 20                     |
|                | 9Cl-PF3ONS     | 11.0                   | 22                      | 22                         | 20                     |
|                | 11Cl-PF3OUdS   | Not Set                | NA                      | 36                         | Not Set                |
| S2<br>(Carrot) | PFBS           | 0.82                   | 22                      | 16                         | 20                     |
|                | PFPeS          | 8.30                   | 22                      | 13                         | 20                     |
|                | PFHxS          | 6.54                   | 22                      | 11                         | 20                     |
|                | PFHxS (linear) | 6.44                   | 22                      | 11                         | 20                     |
|                | PFHpS          | 2.99                   | 22                      | 12                         | 20                     |
|                | PFOS           | 1.95                   | 22                      | 13                         | 20                     |
|                | PFOS (linear)  | 2.01                   | 22                      | 11                         | 20                     |
|                | PFNS           | 1.52                   | 22                      | 19                         | 20                     |
|                | PFDS           | 6.76                   | 22                      | 21                         | 20                     |
|                | PFBA           | Not Set                | NA                      | 47                         | Not Set                |
|                | PFPeA          | 2.07                   | 22                      | 17                         | 20                     |
|                | PFHxA          | 7.83                   | 22                      | 6.1                        | 20                     |
|                | PFHpA          | 1.53                   | 22                      | 18                         | 20                     |
|                | PFOA           | 1.24                   | 22                      | 17                         | 20                     |
|                | PFNA           | 2.36                   | 22                      | 20                         | 20                     |
|                | PFDA           | 9.58                   | 22                      | 13                         | 20                     |
|                | PFOSA          | 4.03                   | 22                      | 19                         | 20                     |
|                | MeFOSA         | 3.98                   | 22                      | 24                         | 20                     |
|                | EtFOSA         | 3.11                   | 22                      | 20                         | 20                     |
|                | 6:2 FTS        | 1.69                   | 22                      | 21                         | 20                     |
| GenX           | 9.8            | 22                     | 17                      | 20                         |                        |
| ADONA          | 13.2           | 22                     | 20                      | 20                         |                        |

\* Robust between-laboratory CV (outliers removed where applicable). Shaded cells are between-laboratory CVs for scored 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 PFNS and PFBA. A maximum acceptable result was set to two target SDs more than the spiked value, and 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 laboratories reporting results close to the spiked value were not penalised.  $z$ -Scores for results higher than the maximum acceptable value were not adjusted, and  $z$ -scores less than 2.0 were left unaltered.

Of 528 results for which  $z$ -scores were calculated, 453 (86%) returned  $|z| \leq 2.0$ , indicating a satisfactory performance.

Sixteen participants analysed both samples, with Laboratories 7, 12, 13 and 17 reporting numeric results for all 39 scored analytes. Laboratories 7 and 12 returned satisfactory  $z$ -scores for all analytes. Laboratories 19 (33), 15 (32) and 9 (7) returned satisfactory  $z$ -scores for all reported numeric results.

Three participants were sent Sample S1 Prawn only. Laboratory 5 was sent Sample S2 carrot only, and returned satisfactory  $z$ -scores for all reported numeric results (10).

Laboratory 16 returned questionable or unsatisfactory  $z$ -scores for all results (31), with all being lower than the assigned value (negative bias;  $z$ -scores ranging from -3.90 to -2.38).

The dispersal of participants'  $z$ -scores is presented graphically by laboratory in Figure 47 and by analyte in Figure 48.

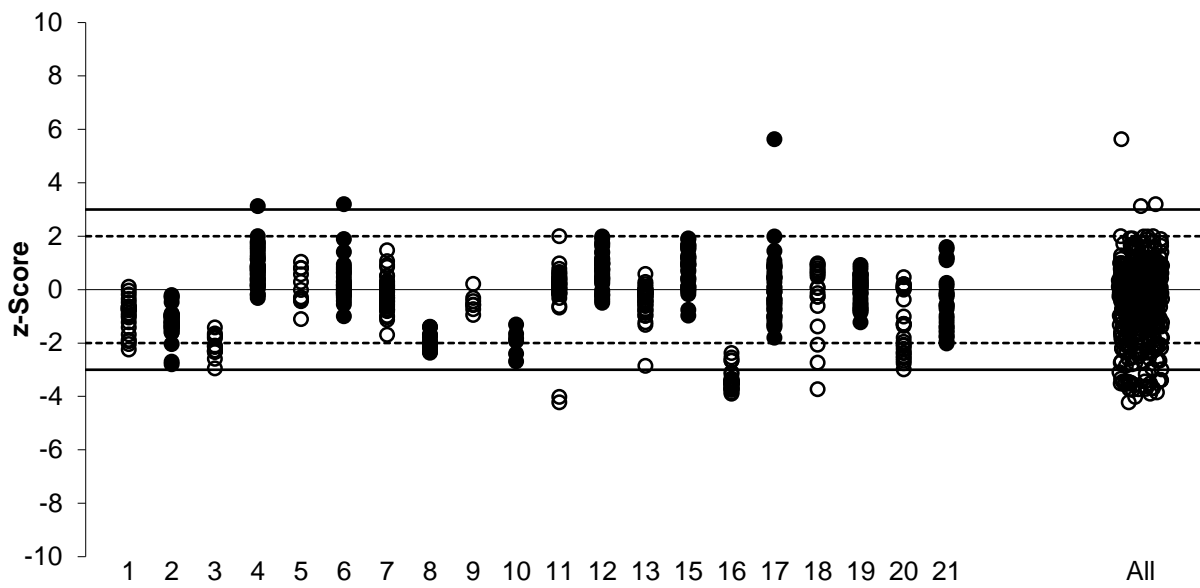


Figure 47  $z$ -Score Dispersal by Laboratory

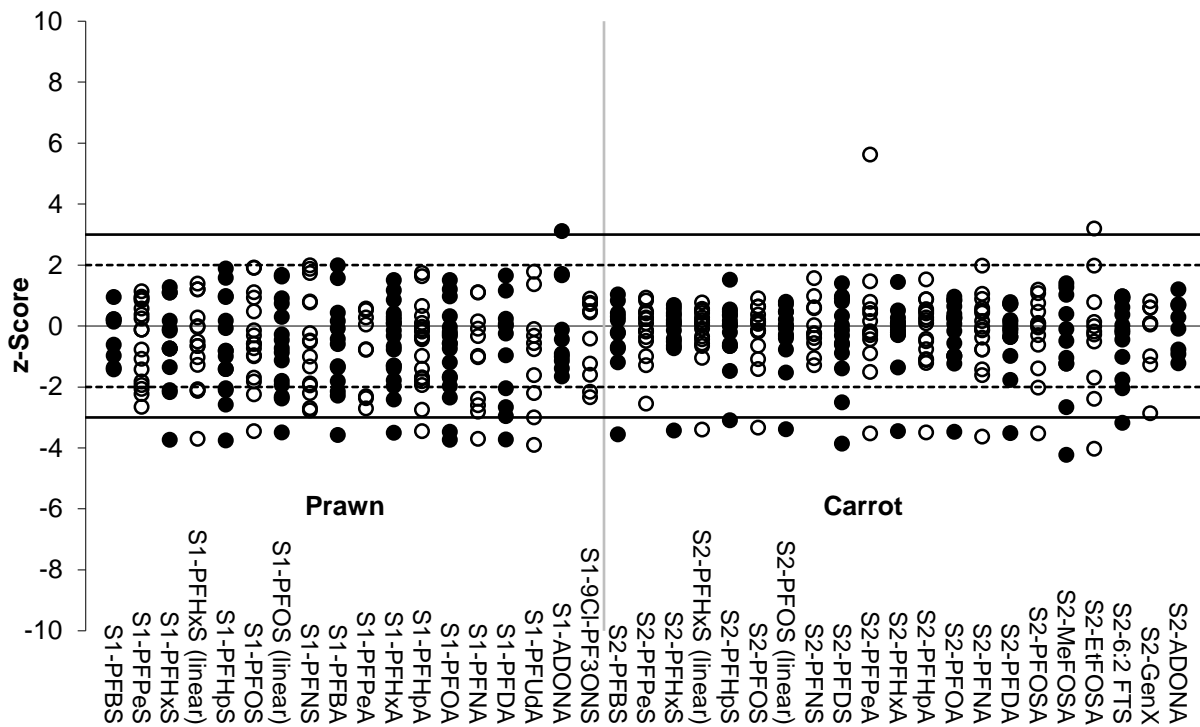


Figure 48  $z$ -Score Dispersal by Analyte



Scatter plots of  $z$ -scores for analytes present in both Samples S1 and S2 are presented in Figures 49 to 63. Scores are predominantly in the upper right and lower left quadrants, indicating that laboratory bias is the major contributor to the variability of results. Points close to the diagonal axis demonstrate excellent repeatability, while points close to the zero demonstrate excellent repeatability and accuracy.

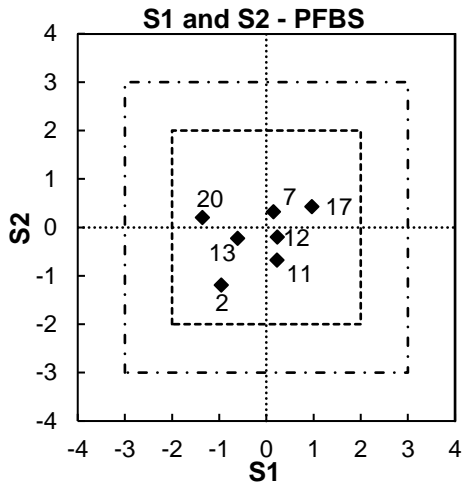


Figure 49  $z$ -Score Scatter Plot – PFBS

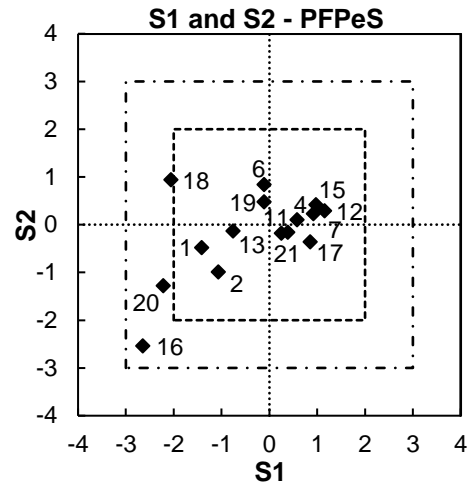


Figure 50  $z$ -Score Scatter Plot – PFPeS

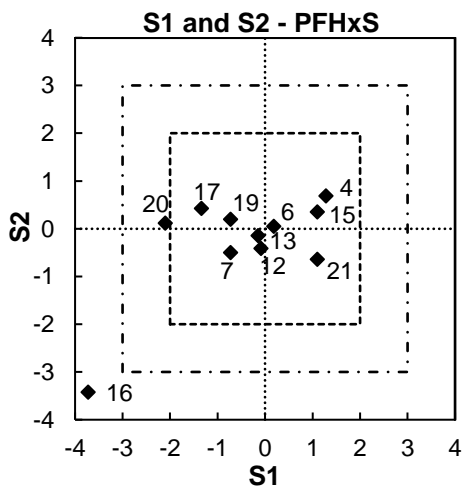


Figure 51  $z$ -Score Scatter Plot – PFHxS

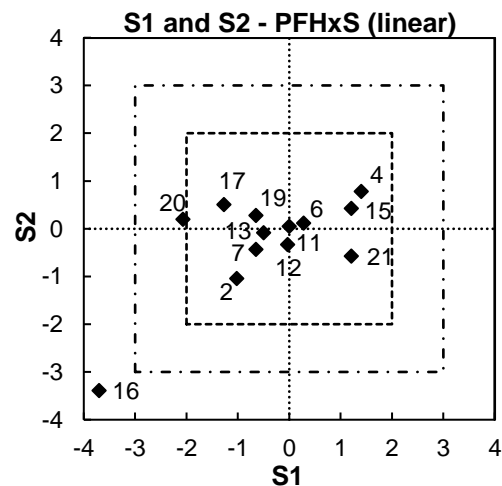


Figure 52  $z$ -Score Scatter Plot – PFHxS (linear)

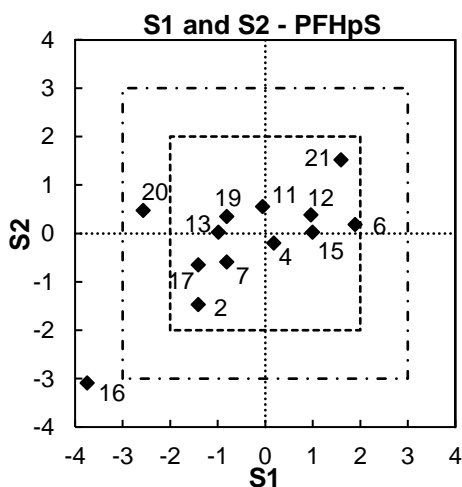


Figure 53  $z$ -Score Scatter Plot – PFHpS

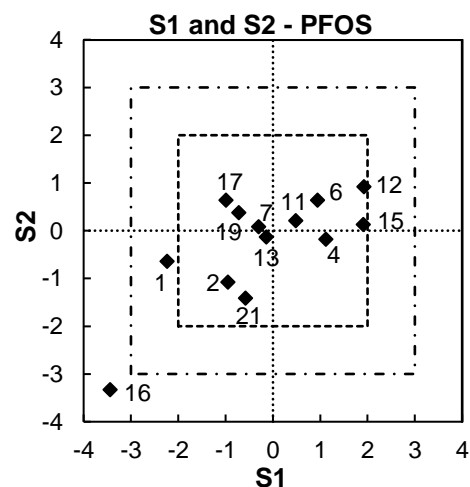


Figure 54  $z$ -Score Scatter Plot – PFOS

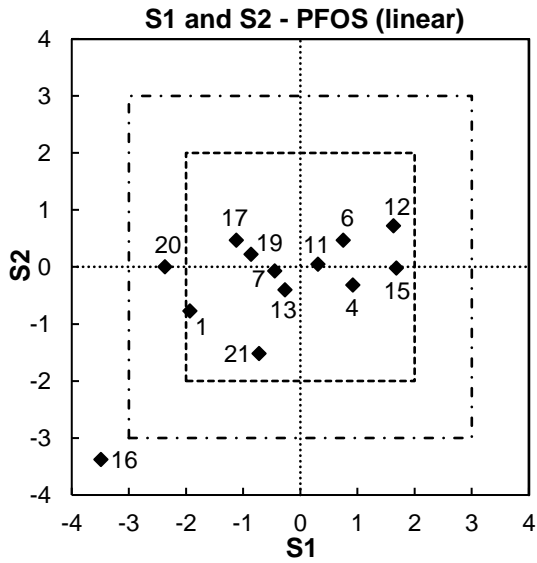


Figure 55 z-Score Scatter Plot – PFOS (linear)

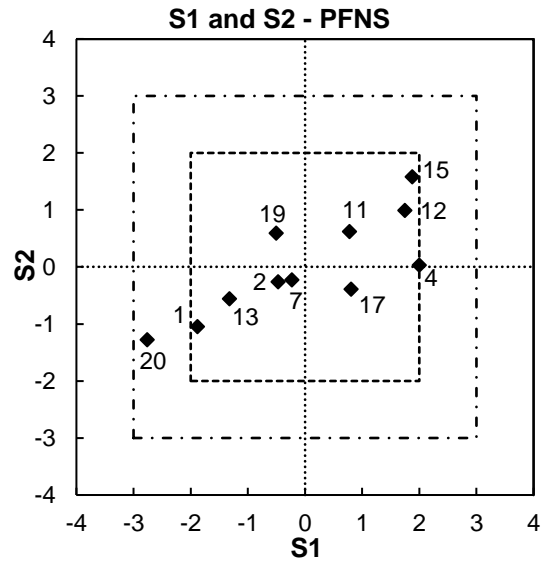
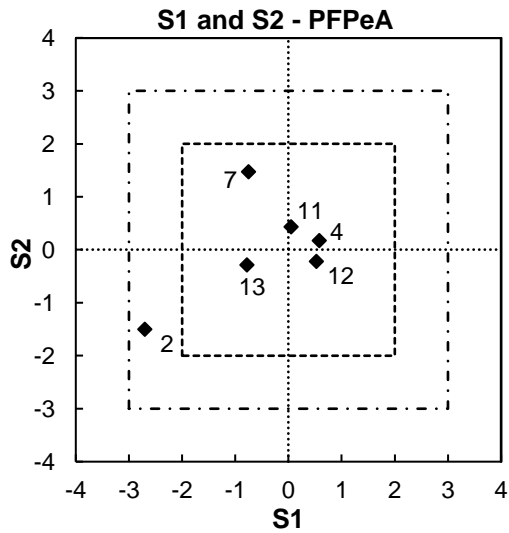


Figure 56 z-Score Scatter Plot – PFNS



Laboratory 17 is off-scale.

Figure 57 z-Score Scatter Plot – PFPeA

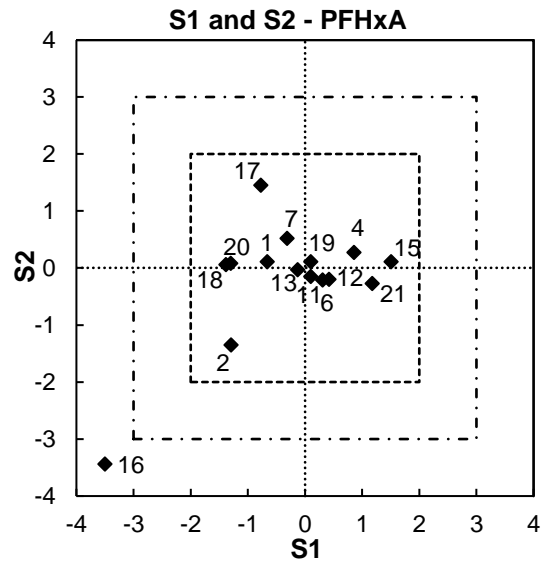


Figure 58 z-Score Scatter Plot – PFHxA

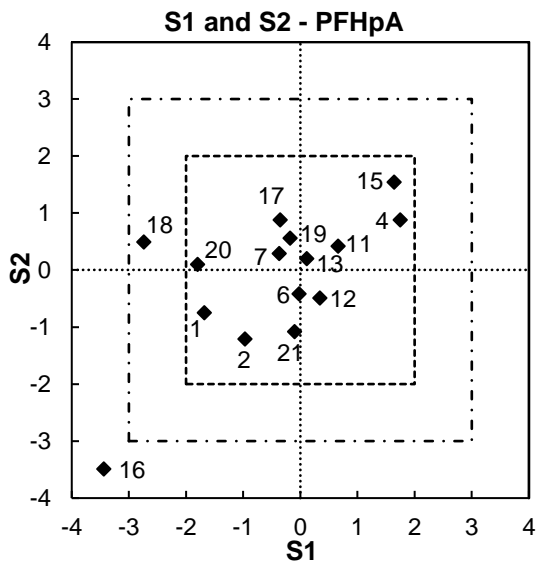


Figure 59 z-Score Scatter Plot – PFHpA

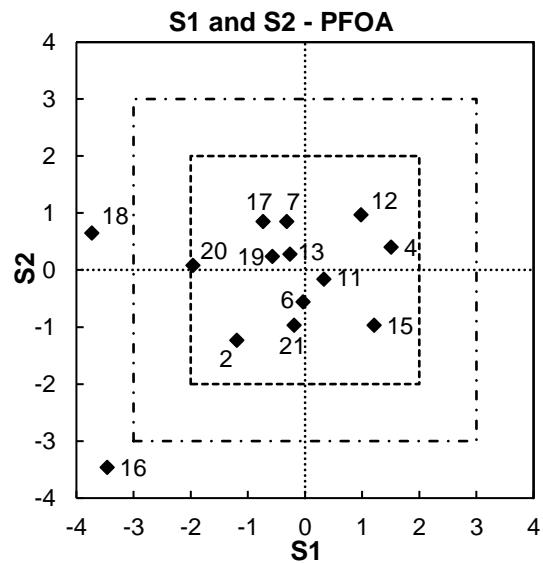


Figure 60 z-Score Scatter Plot – PFOA

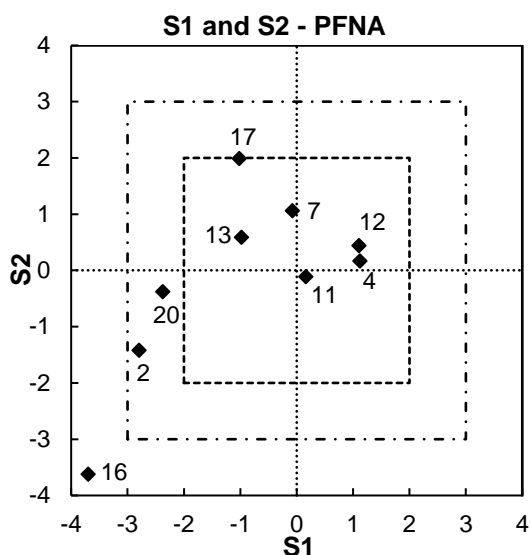


Figure 61  $z$ -Score Scatter Plot – PFNA

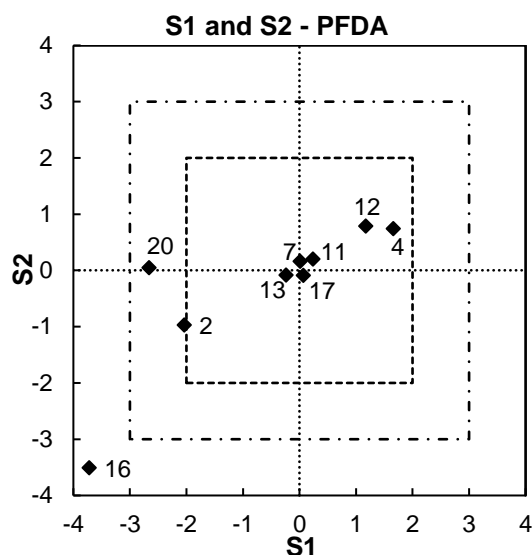


Figure 62  $z$ -Score Scatter Plot – PFDA

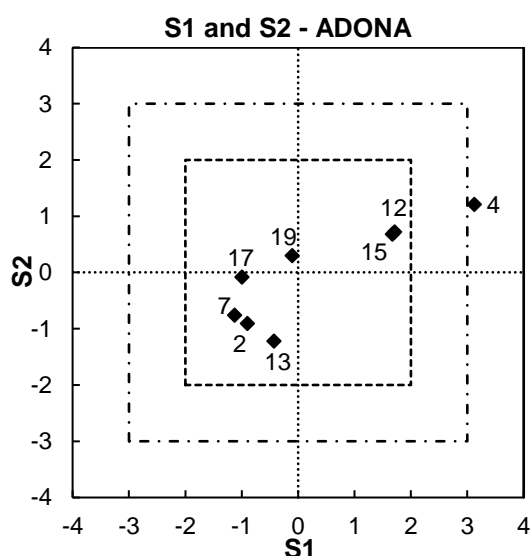


Figure 63  $z$ -Score Scatter Plot – ADONA

#### 6.4 $E_n$ -Score

$E_n$ -scores can be interpreted in conjunction with  $z$ -scores, as an unsatisfactory  $E_n$ -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.  $E_n$ -scores greater than 1.0 were set to 1.0 for results with  $z$ -scores that were adjusted as discussed in Section 6.3  $z$ -Score.

Of 528 results for which  $E_n$ -scores were calculated, 374 (71%) returned  $|E_n| \leq 1.0$ , indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

No participant returned satisfactory  $E_n$ -scores for all analytes of interest in this study. Of the participants analysing both matrices, Laboratories **19** (33), **15** (32) and **9** (7) returned satisfactory  $E_n$ -scores for all reported results. Of participants analysing Sample S2 carrot only, Laboratory **5** (10) returned satisfactory  $E_n$ -scores for all reported results.

Laboratories **16** (31) and **3** (14) returned unsatisfactory  $E_n$ -scores for all reported results.

The dispersal of participants'  $E_n$ -scores is presented graphically in Figure 64.

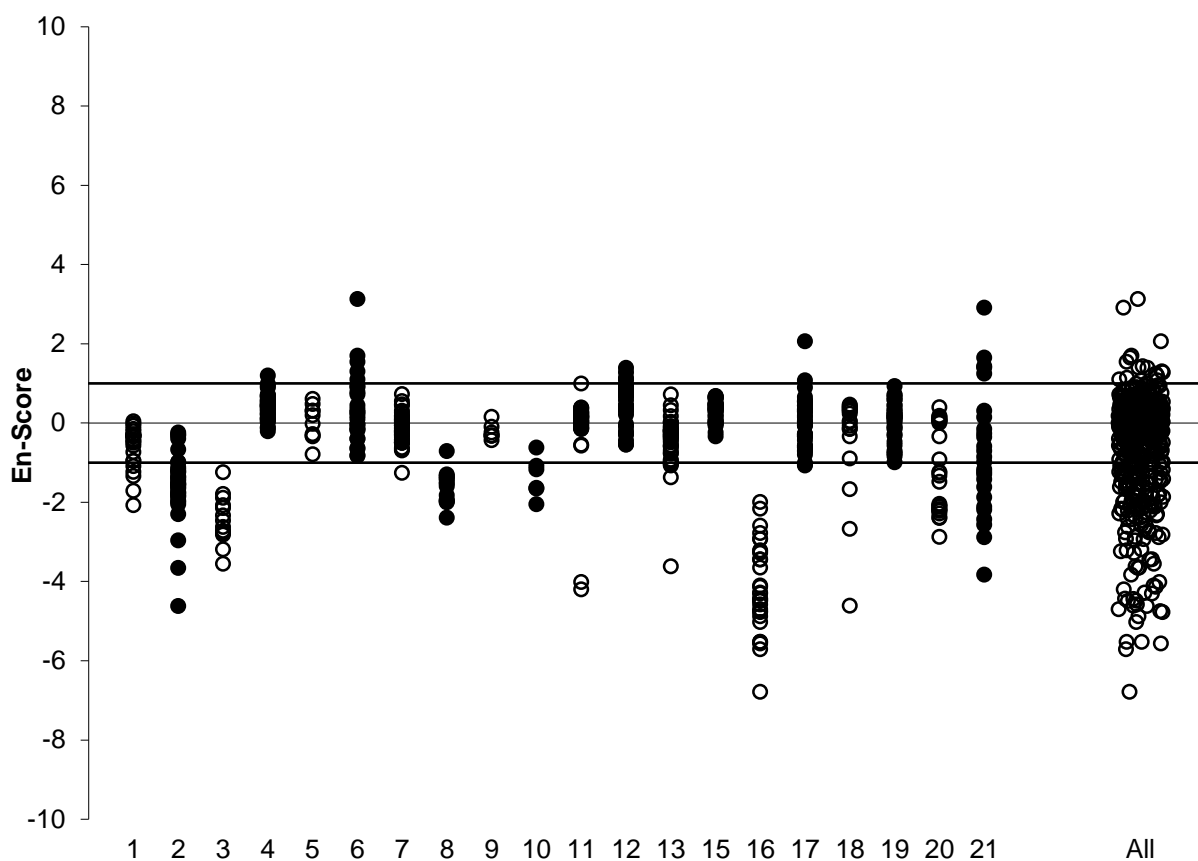


Figure 64  $E_n$ -Score Dispersal by Laboratory

### 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, 24 different analytes were spiked for this study, with 21 analytes being spiked into Sample S1, and 20 analytes being spiked into Sample S2. For PFHxS and PFOS, participants were requested to report for both linear isomers and total value, however both samples were spiked with linear only isomers. 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 53.

Of the participants who analysed both samples, Laboratories **4, 7, 12, 13, 15, 17** and **19** analysed for all spiked analytes. Of the participants who only received Sample S1 prawn, Laboratories **8** and **10** analysed for all spiked analytes in this sample. Laboratories **6** and **9** reported analysing some analytes in one sample but not the other. All participants tested for at least one spiked analyte, with the proportion of PFAS being analysed by each participant ranging from 31% to 100%.

Out of the spiked analytes in this study, PFBS, PFHxA, PFHpA, PFOA, PFNA and PFDA were tested for by the highest proportion of participants (100% for all). In general, perfluoroalkyl acids were very well represented by participants, with the overall proportion of analysis by participants being 89% and 95% for perfluoroalkane sulfonates and perfluoroalkyl carboxylic acids respectively. A lower proportion of participants analysed the perfluoroalkane sulfonamide (PFOSA), perfluoroalkane sulfonamido (MeFOSA and EtFOSA) and fluorotelomer (6:2 FTS) analytes, being 80%, 75% and 82% respectively. The PFAS replacement compounds (GenX, ADONA, 9Cl-PF3ONS and 11Cl-PF3OUdS) were analysed by the lowest proportion of participants (65%).

Table 53 Summary of PFAS Analysed by Participants<sup>a</sup>

| Lab. Code<br>Analyte | 1  | 2  | 3  | 4 | 5  | 6 | 7 | 8 | 9 <sup>d</sup> | 10 | 11 | 12 | 13 | 15 | 16 | 17 | 18 | 19 | 20 | 21 | Proportion of<br>Participants (%) |     |
|----------------------|----|----|----|---|----|---|---|---|----------------|----|----|----|----|----|----|----|----|----|----|----|-----------------------------------|-----|
| PFBS                 | ✓  | ✓  | ✓  | ✓ | ✓  | ✓ | ✓ | ✓ | ✓              | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 100 |
| PFPeS                | ✓  | ✓  | ✓  | ✓ | NT | ✓ | ✓ | ✓ | NT             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 90  |
| PFHxS                | ✓  | ✓  | NT | ✓ | NT | ✓ | ✓ | ✓ | ✓              | ✓  | NT | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 85  |
| PFHxS (linear)       | ✓  | ✓  | ✓  | ✓ | ✓  | ✓ | ✓ | ✓ | NT             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | NT | ✓  | ✓  | ✓  | ✓                                 | 90  |
| PFHpS                | ✓  | ✓  | ✓  | ✓ | NT | ✓ | ✓ | ✓ | NT             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 90  |
| PFOS                 | ✓  | ✓  | NT | ✓ | NT | ✓ | ✓ | ✓ | ✓              | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 90  |
| PFOS (linear)        | ✓  | ✓  | ✓  | ✓ | ✓  | ✓ | ✓ | ✓ | NT             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 95  |
| PFNS                 | ✓  | ✓  | NT | ✓ | NT | ✓ | ✓ | ✓ | NT             | ✓  | ✓  | ✓  | ✓  | ✓  | NT | ✓  | NT | ✓  | ✓  | ✓  | ✓                                 | 75  |
| PFDS <sup>c</sup>    | NT | ✓  | ■  | ✓ | ✓  | ✓ | ✓ | ■ | NT             | ■  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 88  |
| PFBA                 | ✓  | ✓  | ✓  | ✓ | ✓  | ✓ | ✓ | ✓ | NT             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | NT | ✓                                 | 90  |
| PFPeA                | ✓  | ✓  | ✓  | ✓ | ✓  | ✓ | ✓ | ✓ | NT             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | NT | ✓                                 | 90  |
| PFHxA                | ✓  | ✓  | ✓  | ✓ | ✓  | ✓ | ✓ | ✓ | ✓              | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 100 |
| PFHpA                | ✓  | ✓  | ✓  | ✓ | ✓  | ✓ | ✓ | ✓ | ✓              | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 100 |
| PFOA                 | ✓  | ✓  | ✓  | ✓ | ✓  | ✓ | ✓ | ✓ | ✓              | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 100 |
| PFNA                 | ✓  | ✓  | ✓  | ✓ | ✓  | ✓ | ✓ | ✓ | ✓              | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 100 |
| PFDA                 | ✓  | ✓  | ✓  | ✓ | ✓  | ✓ | ✓ | ✓ | ✓              | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 100 |
| PFUdA <sup>b</sup>   | ✓  | ✓  | NT | ✓ | ■  | ✓ | ✓ | ✓ | NT             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 89  |
| PFTrDA <sup>b</sup>  | ✓  | ✓  | NT | ✓ | ■  | ✓ | ✓ | ✓ | NT             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 89  |
| PFOSA                | ✓  | ✓  | NT | ✓ | NT | ✓ | ✓ | ✓ | NT             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | NT | ✓                                 | 80  |
| MeFOSA               | ✓  | NT | NT | ✓ | NT | ✓ | ✓ | ✓ | NT             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | NT | ✓                                 | 75  |
| EtFOSA               | ✓  | NT | NT | ✓ | NT | ✓ | ✓ | ✓ | NT             | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | NT | ✓                                 | 75  |
| 6:2 FTS <sup>c</sup> | ✓  | ✓  | ■  | ✓ | NT | ✓ | ✓ | ■ | NT             | ■  | NT | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓  | ✓                                 | 82  |

| Lab. Code<br>Analyte          | 1  | 2  | 3  | 4   | 5  | 6              | 7   | 8   | 9 <sup>d</sup> | 10  | 11 | 12  | 13  | 15  | 16 | 17  | 18 | 19  | 20 | 21 | Proportion of<br>Participants (%) |
|-------------------------------|----|----|----|-----|----|----------------|-----|-----|----------------|-----|----|-----|-----|-----|----|-----|----|-----|----|----|-----------------------------------|
| GenX <sup>c</sup>             | NT | ✓  |    | ✓   | NT | ✓              | ✓   |     | NT             |     | NT | ✓   | ✓   | ✓   | ✓  | ✓   | NT | ✓   | ✓  | ✓  | 71                                |
| ADONA                         | NT | ✓  | ✓  | ✓   | NT | ✓ <sup>e</sup> | ✓   | ✓   | NT             | ✓   | NT | ✓   | ✓   | ✓   | ✓  | ✓   | NT | ✓   | NT | NT | 65                                |
| 9CI-PF3ONS <sup>b</sup>       | NT | ✓  | ✓  | ✓   |    | NT             | ✓   | ✓   | NT             | ✓   | NT | ✓   | ✓   | ✓   | ✓  | ✓   | NT | ✓   | NT | NT | 63                                |
| 11CI-PF3OUdS <sup>b</sup>     | NT | ✓  | ✓  | ✓   |    | NT             | ✓   | ✓   | NT             | ✓   | NT | ✓   | ✓   | ✓   | ✓  | ✓   | NT | ✓   | NT | NT | 63                                |
| Proportion of<br>Analytes (%) | 81 | 92 | 65 | 100 | 50 | 92             | 100 | 100 | 31             | 100 | 77 | 100 | 100 | 100 | 96 | 100 | 77 | 100 | 69 | 88 |                                   |

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

<sup>b</sup> Spiked into Sample S1 only.

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

<sup>d</sup> Laboratory 9 enrolled for and was supplied both samples in this study, however reported 'NT' for all analytes in Sample S2. This participant's data in this table has been completed according to their Sample S1 results where available.

<sup>e</sup> Laboratory 6 reported analysing for ADONA in Sample S2 but not Sample S1.

## 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 54. With the relatively high trigger points for crustaceans, Sample S1 PFHxS, PFOS and PFOA were all well below the trigger points. For Sample S2, PFHxS and PFOS were above the trigger points, while PFOA was under the trigger point.

Table 54 Assigned Values and FSANZ Trigger Points for PFHxS, PFOS and PFOA<sup>11</sup>

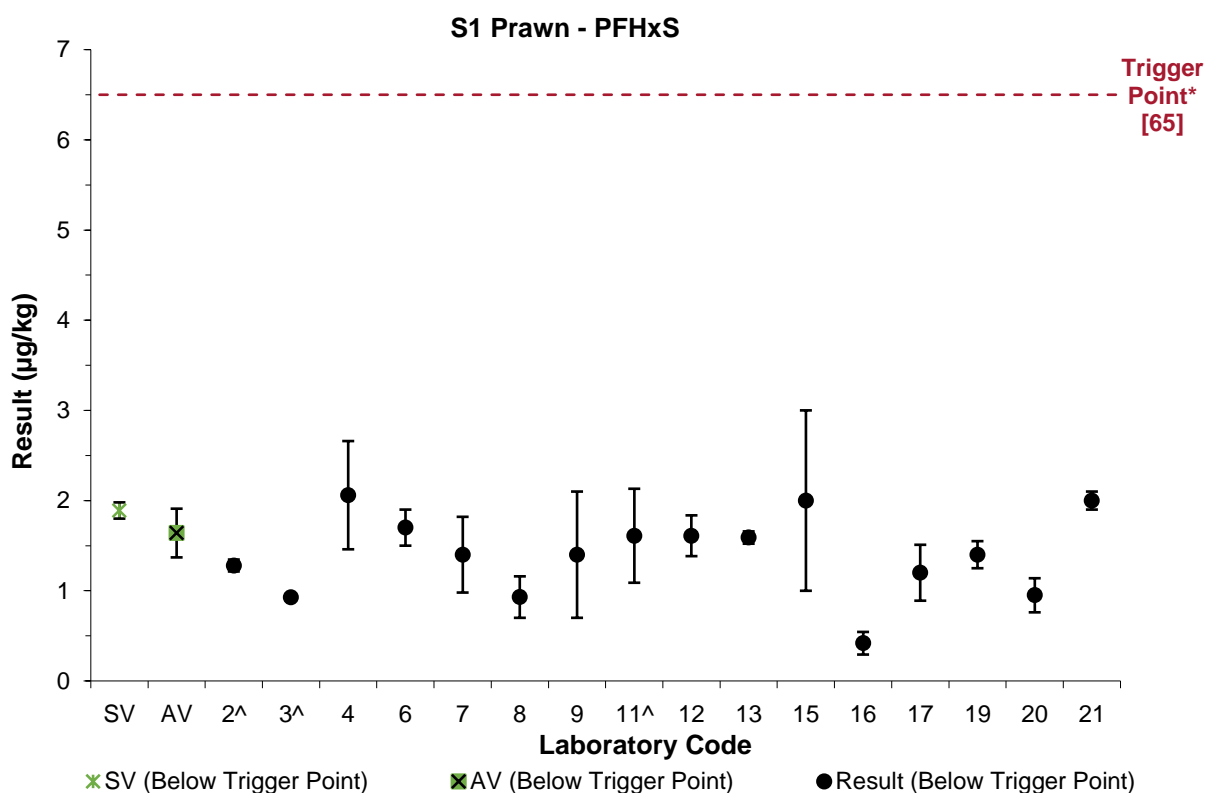
| Sample | Matrix | Classification | PFHxS (µg/kg)  |               | PFOS (µg/kg)   |               | PFOA (µg/kg)   |               |
|--------|--------|----------------|----------------|---------------|----------------|---------------|----------------|---------------|
|        |        |                | Assigned Value | Trigger Point | Assigned Value | Trigger Point | Assigned Value | Trigger Point |
| S1     | Prawn  | Crustaceans    | 1.64 ± 0.27    | 65            | 3.62 ± 0.68    | 65            | 6.44 ± 0.85    | 520           |
| S2     | Carrot | Vegetables     | 6.54 ± 0.51    | 1.1           | 1.95 ± 0.17    | 1.1           | 1.24 ± 0.14    | 8.8           |

Figures 65 to 70 show comparisons of the spiked values (SV), assigned values (AV), participants' results, and FSANZ trigger points for these analytes. Where no numeric result was reported, or if a LOR 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.

In this study, five of the six assessed analytes were either significantly higher or lower than the trigger points, and so it was expected that the vast majority of participants' results should match the assigned values with respect to being above or below the FSANZ trigger points. This was seen in this study, with 97 results of a total 100 (97%) being correctly above or below the trigger point inclusive of uncertainty, and a further two results being correctly above or below the trigger point with uncertainty spanning the trigger point.

Laboratories **2, 4, 6, 7, 11, 12, 13, 17, 19, 20** and **21** correctly identified whether the analyte mass fractions (inclusive of uncertainties) were above or below the trigger points for all six analytes, while Laboratories **18** (4), **3** (3), **5** (3), **8** (3), **9** (3) and **10** (2) did so for all analytes that they reported results for.

For Sample S2 PFOS, the assigned value above but closer to the trigger point as compared to the other analytes. In this sample, two participants reported results where the uncertainties spanned the trigger point. Laboratory **16** reported a result below the trigger point inclusive of uncertainty, which would have incorrectly indicated no need for further investigation.



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

^ Result for linear isomers only has been plotted.

Figure 65 Sample S1 Prawn PFHxS Spiked and Assigned Values, Participant Results and Trigger Point

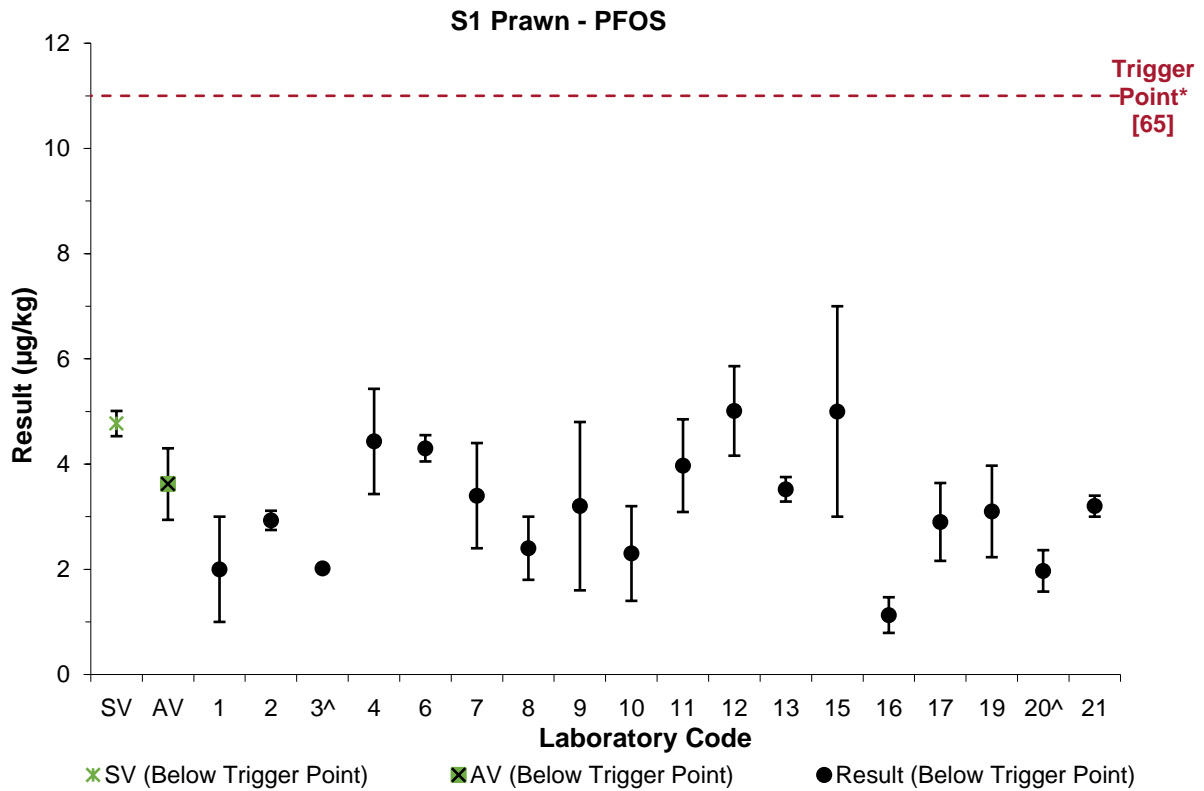


Figure 66 Sample S1 Prawn PFOS Spiked and Assigned Values, Participant Results and Trigger Point

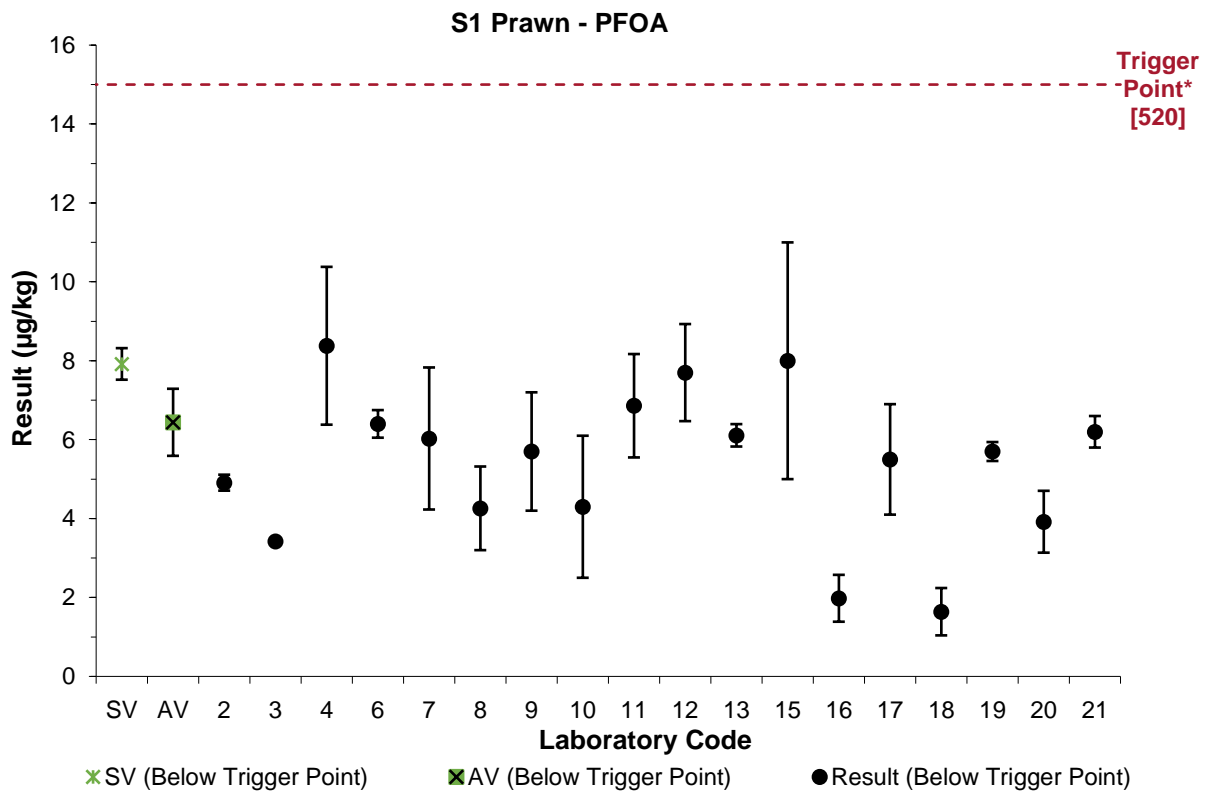
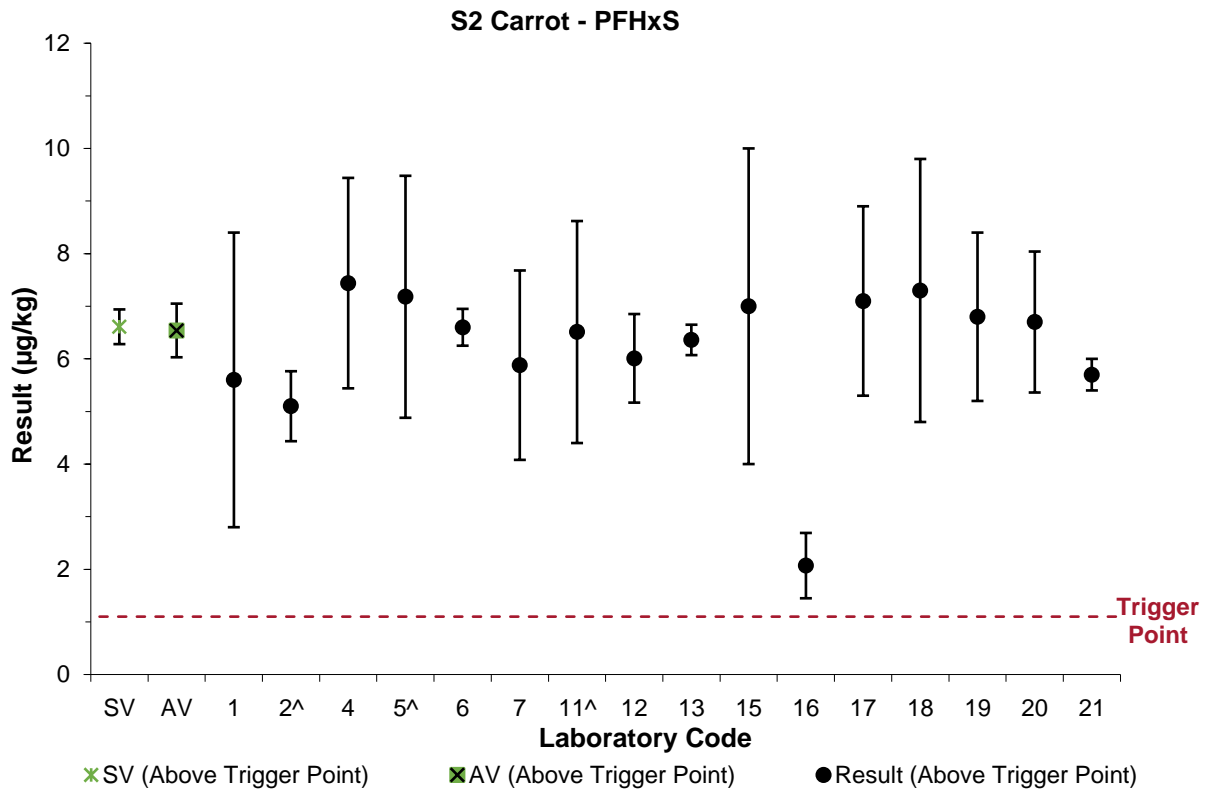


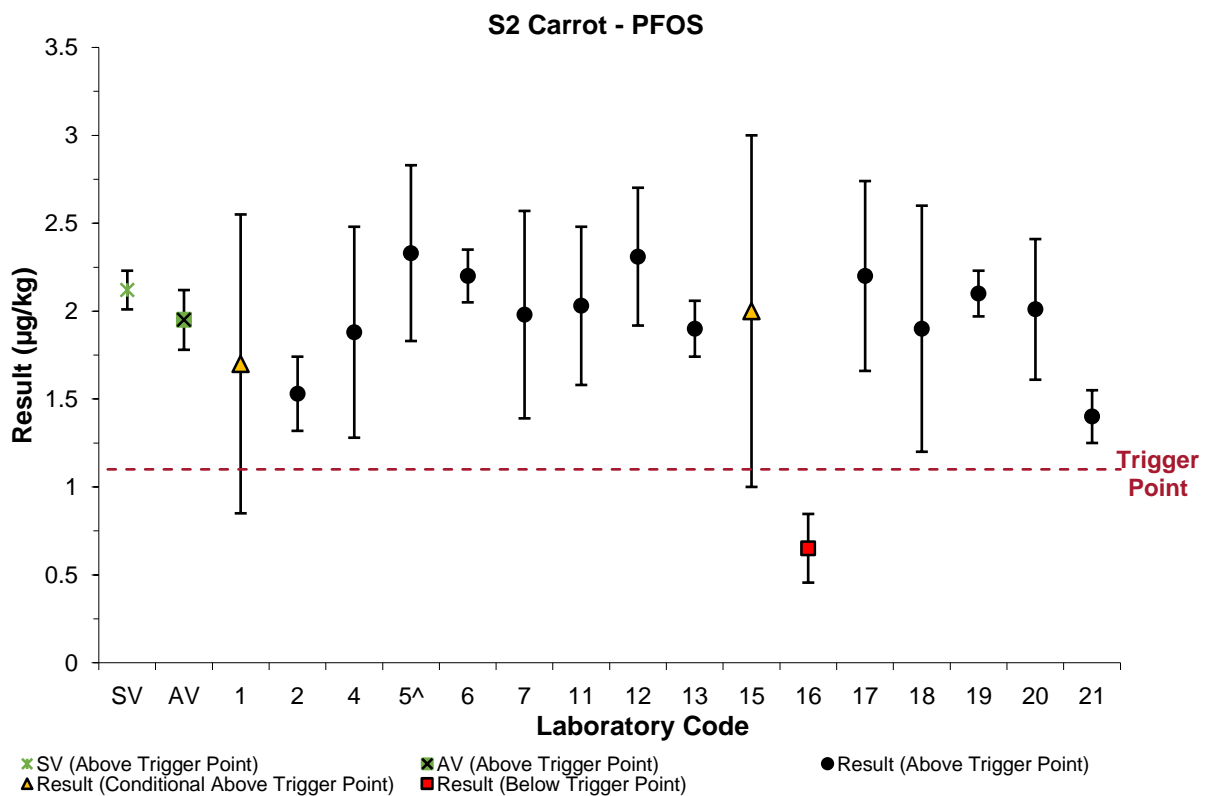
Figure 67 Sample S1 Prawn PFOA Spiked and Assigned Values, Participant Results and Trigger Point





^ Result for linear isomers only has been plotted.

Figure 68 Sample S2 Carrot PFHxS Spiked and Assigned Values, Participant Results and Trigger Point



^ Result for linear isomers only has been plotted.

Figure 69 Sample S2 Carrot PFOS Spiked and Assigned Values, Participant Results and Trigger Point

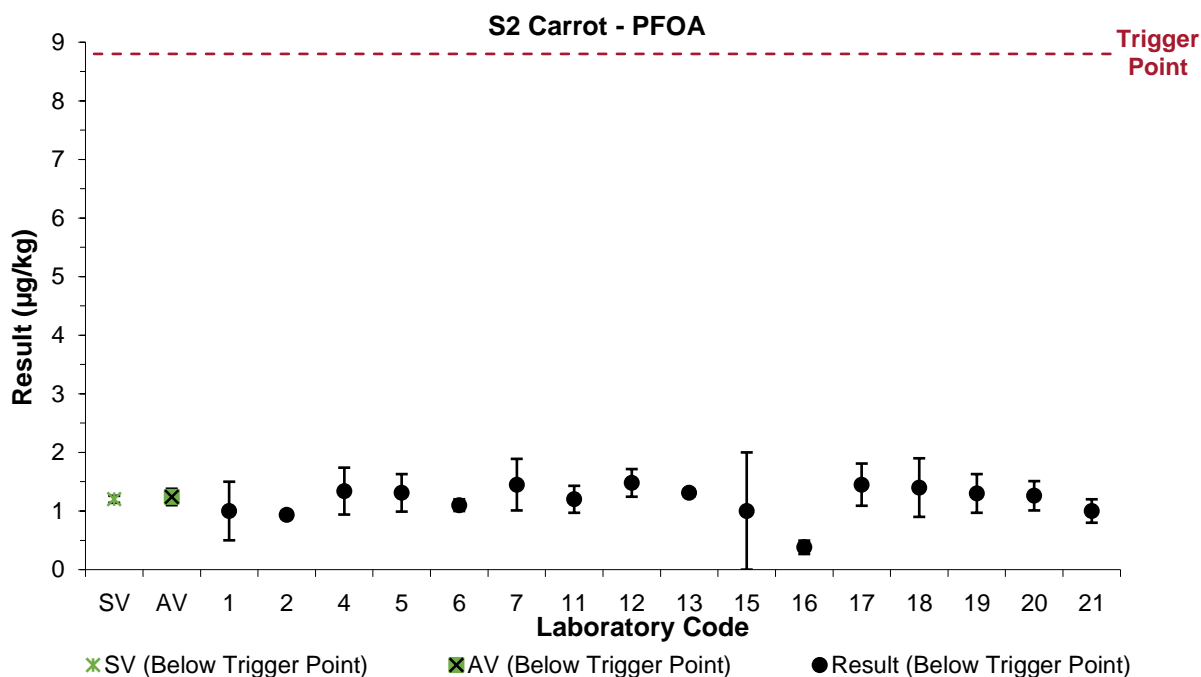


Figure 70 Sample S2 Carrot PFOA Spiked and Assigned Values, Participant Results and Trigger Point

### 6.7 False Negatives

Table 55 presents false negative results. These are analytes present in the samples which a participant tested for, but did not report a result; for example, when participants reported a 'less-than' result ( $< x$ ) when the assigned value was higher than their limit of reporting (LOR), or did not report anything. For analytes where no assigned value was set, results have only been considered to be false negatives where the robust average and spiked value were significantly higher than the participants' LOR, or if no value was reported.

Table 55 False Negatives

| Lab. Code | Sample | Analyte       | Assigned Value ( <i>Robust Average</i> ) (µg/kg) | Spiked Value (µg/kg) | Result* (µg/kg) |
|-----------|--------|---------------|--|----------------------|-----------------|
| 1         | S1     | PFHpS         | 1.67   | 2.00                 | < 1             |
|           |        | PFBA          | 2.03   | 2.96                 | < 2             |
|           |        | PFOA          | 6.44   | 7.92                 | < 5             |
| 2         | S1     | PFHxS         | 1.64   | 1.89                 | NR              |
|           |        | PFOS (linear) | 3.74   | 4.77                 | NR              |
|           | S2     | PFHxS         | 6.54   | 6.61                 | NR              |
|           |        | PFOS (linear) | 2.01   | 2.12                 | NR              |
| 6         | S1     | PFPeA         | 1.04   | 1.13                 | <1              |
|           |        | PFUdA         | 1.12   | 1.21                 | <1              |
|           | S2     | PFNS          | 1.52   | 1.72                 | <1              |
|           |        | PFPeA         | 2.07   | 2.20                 | <1              |
|           |        | GenX          | 9.8  | 11.1                 | <1              |
|           |        | ADONA         | 13.2   | 14.0                 | <1              |
| 8         | S1     | PFDA          | 0.74   | 0.902                | <0.5            |

| Lab. Code | Sample | Analyte        | Assigned Value ( <i>Robust Average</i> ) (µg/kg) | Spiked Value (µg/kg) | Result* (µg/kg) |
|-----------|--------|----------------|--|----------------------|-----------------|
| 10        | S1     | PFPeS          | 4.19   | 4.65                 | NR              |
|           |        | PFHxS          | 1.64   | 1.89                 | <1              |
|           |        | PFHxS (linear) | 1.61   | 1.89                 | NR              |
|           |        | PFHpS          | 1.67   | 2.00                 | <1              |
|           |        | PFOS (linear)  | 3.74   | 4.77                 | NR              |
|           |        | PFPeA          | 1.04   | 1.13                 | <1              |
|           |        | PFDA           | 0.74   | 0.902                | NR              |
|           |        | PFUdA          | 1.12   | 1.21                 | <1              |
|           |        | PFTTrDA        | (5.4)  | 8.17                 | NR              |
|           |        | PFOSA          | (3.00)   | 4.46                 | NR              |
|           |        | MeFOSA         | (3.7)  | 4.99                 | NR              |
|           |        | EtFOSA         | (2.66)   | 3.99                 | NR              |
|           |        | ADONA          | 4.5  | 5.64                 | NR              |
|           |        | 9Cl-PF3ONS     | 11.0   | 14.4                 | NR              |
|           |        | 11Cl-PF3OUdS   | (2.27)   | 4.70                 | NR              |
| 16        | S1     | PFBS           | 0.302  | 0.399                | < 0.1           |
|           |        | PFPeA          | 1.04   | 1.13                 | < 0.3           |
|           |        | ADONA          | 4.5  | 5.64                 | NR              |
|           |        | 9Cl-PF3ONS     | 11.0   | 14.4                 | NR              |
|           |        | 11Cl-PF3OUdS   | (2.27)   | 4.7                  | NR              |
|           | S2     | PFBA           | (0.78)   | 1.19                 | < 0.3           |
|           |        | GenX           | 9.8  | 11.1                 | NR              |
| 18        | S1     | PFHxS          | 1.64   | 1.89                 | <1              |
|           |        | PFHpS          | 1.67   | 2                    | <1              |
|           |        | PFOS           | 3.62   | 4.77                 | <1              |
|           |        | PFOS (linear)  | 3.74   | 4.77                 | <1              |
|           |        | PFUdA          | 1.12   | 1.21                 | <1              |
|           |        | PFTTrDA        | (5.4)  | 8.17                 | <2              |
| 19        | S1     | PFPeA          | 1.04   | 1.13                 | < 1.0           |
|           |        | PFUdA          | 1.12   | 1.21                 | < 1.0           |
|           | S2     | PFBS           | 0.82   | 0.891                | NR              |
|           |        | PFBA           | (0.78)   | 1.19                 | NR              |
| 20        | S1     | PFOS           | 3.62   | 4.77                 | NR              |
| 21        | S1     | PFPeA          | 1.04   | 1.13                 | <1              |
|           |        | PFUdA          | 1.12   | 1.21                 | <1              |
|           | S2     | PFNS           | 1.52   | 1.72                 | <1              |

| Lab. Code | Sample | Analyte | Assigned Value ( <i>Robust Average</i> ) (µg/kg) | Spiked Value (µg/kg) | Result* (µg/kg) |
|-----------|--------|---------|--|----------------------|-----------------|
|           |        | PFPeA   | 2.07   | 2.2                  | <1              |
|           |        | GenX    | 9.8  | 11.1                 | <1              |

\* NR results may or may not be false negatives, depending on the participant's actual LOR.

## 6.8 Reporting of Additional Analytes

Seven participants reported at least one analyte that was not spiked into the test samples by the study coordinator. These results are presented in Table 56.

Table 56 Non-Spiked Analytes Reported by Participants

| Lab. Code | Sample | Analyte            | Result (µg/kg) | Uncertainty (µg/kg) | Recovery (%) |
|-----------|--------|--------------------|----------------|---------------------|--------------|
| 2         | S1     | PFD <sub>o</sub> A | 0.025          | 0.006               | 56           |
| 7         | S1     | PFD <sub>o</sub> A | 0.120          | 0.12                | 81           |
| 11        | S1     | PFDS               | 0.0414         | 0.0268              | 93.2         |
|           |        | PFD <sub>o</sub> A | 0.155          | 0.035               | 127          |
|           | S2     | PFD <sub>o</sub> S | 0.0386         | 0.0204              | 148.5        |
| 12        | S1     | PFD <sub>o</sub> A | 0.19           | 0.0418              | 71.1         |
| 16        | S1     | PFD <sub>o</sub> A | 0.1            | 0.03                | 165          |
| 17        | S1     | PFD <sub>o</sub> A | 0.11           | 0.077               | 89           |
| 20        | S1     | PFD <sub>o</sub> A | 0.053          | 0.011               | 76           |
|           | S2     | PFD <sub>o</sub> S | 0.281          | 0.056               | 83           |
|           |        | PFD <sub>o</sub> A | 0.010          | 0.004               | 50           |

## 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 is presented below as technique (number of participants):

- Sample Weight
  - S1: < 1 g (2), 1 – 1.1 g (8), 2 – 2.5 g (3), 5 – 6 g (1)
  - S2: < 1 g (1), 1 – 1.1 g (3), 2 – 2.5 g (5), 5 – 6 g (2), 10 g (1)
- Pretreatment
  - S1: homogenisation (10), no pretreatment (2)
  - S2: homogenisation (9), freeze-drying (1), no pre-treatment (2)
- Extraction Technique
  - S1: alkaline digestion (5), QuEChERS (3), SLE (8)
  - S2: alkaline digestion (4), QuEChERS (3), SLE (7), Soxhlet (1)
- Extraction Solvent
  - S1: acetonitrile (4), acetonitrile/acid(/water) (3), acetonitrile/base (2), methanol/base (3), multiple/other (2)

- S2: acetonitrile (3), acetonitrile/acid(/water) (2), acetonitrile/base (1), methanol/base (3), multiple/other (3)
- Extraction Temperature
  - S1: room temperature (13), heated (2)
  - S2: room temperature (10), heated/boiling (3)
- Extraction Time (total)
  - S1: 8 min (1), 30 min (5), 40 min (1), 1 h (2), 2 h (1), 3 h (1), 8 h (1), 16 h (1)
  - S2: 8 min (1), 30 min (5), 1 h (2), 2 h (1), 4 h (1), 8 h (1), 16 h (1)
- Clean-Up
  - S1: SPE / dSPE (carbon: 8, WAX: 2, C18: 2, other / not specified: 6), centrifugation (1), filtration (1), LLE (1)
  - S2: SPE / dSPE (carbon: 7, WAX: 2, C18: 2, other / not specified: 4), centrifugation (1), filtration (1), ion pair separation (1)
- Instrument
  - S1: LC-MS/MS or LC-QQQ (14), LC-Orbitrap (2)
  - S2: LC-MS/MS or LC-QQQ (12), LC-Orbitrap (2)
- Dilution
  - S1: Yes (2), No (7)
  - S2: Yes (2), No (6)
- Guard Column
  - S1: Yes (10), No (5)
  - S2: Yes (11), No (3)
- Delay Column
  - S1: Yes (15), No (1)
  - S2: Yes (13), No (1)
- Blank Correction
  - S1: Yes (2), No (12)
  - S2: Yes (2), No (11)
- Labelled Standard Source
  - S1: Wellington Laboratories (15)
  - S2: Wellington Laboratories (13)
- Recovery Correction
 

Participants reported a very broad range of recoveries, ranging from 2% to 391%, though the vast majority were between 50% and 150%.

  - S1: Yes (11), No (5)
  - S2: Yes (9), No (5)

As discussed in Section 4.2, the results from Laboratories **3, 8, 10, 16** and **20** in Sample S1, and from Laboratory **16** in Sample S2, were consistently lower than the spiked value by around the same factor for all analytes. This is an indication of laboratory or method bias. These participants should check their sample or standard preparation/dilution procedures. Therefore, these results have been excluded from the following methodology analysis.

Comparisons of z-scores with various extraction and analysis parameters are given in Figures 71 to 78. The most popular methodology for this study was homogenisation as pretreatment, followed by alkaline digestion with basified methanol and SPE clean-up, and then analysis on LC-MS/MS.

In this study, it was observed that participants using basified methanol as their extraction solvent, or those who had long extraction times (> 8 h), generally achieved higher z-scores. As the ratios of assigned values to spiked values were generally below 100% in this study, higher satisfactory z-scores may correspond to more efficient extraction parameters.

A participant reported using 0.5 g for Sample S1 prawn analysis, and the results reported by this participant were biased low. Caution should be exercised when a small sample size (e.g. < 1 g) is taken for analysis, as this may not be a suitable representation of the whole sample.

One participant used QuEChERS for extraction; this participant's results were generally satisfactory though biased low. Another participant used MTBE as the extraction solvent; their results for Sample S1 prawn were biased low. These participants may need to review if their methodology introduced bias to their measurements.

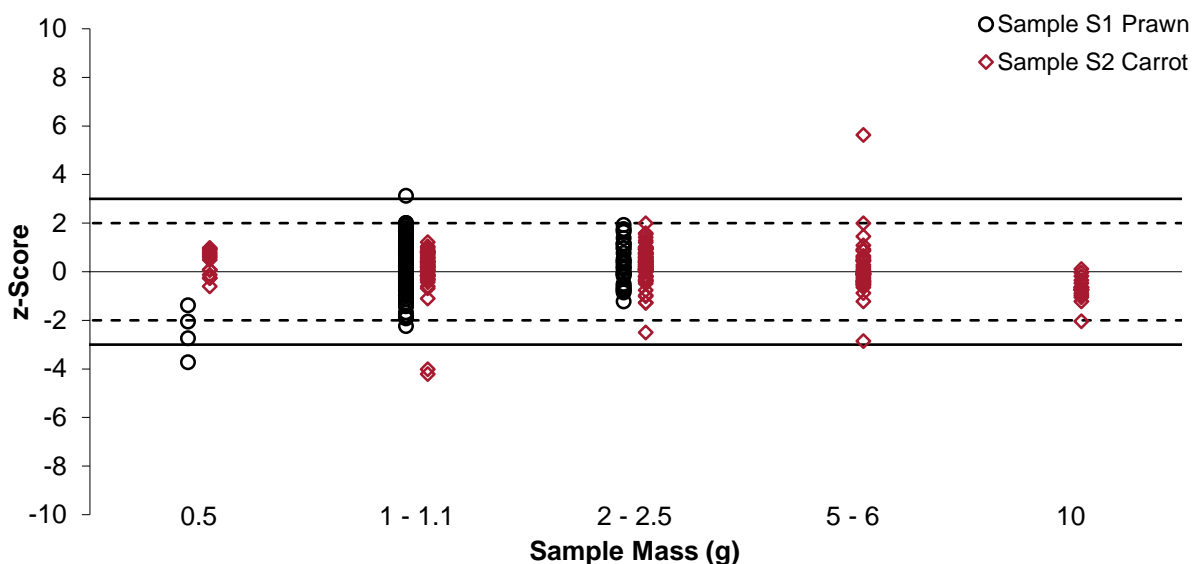


Figure 71 z-Score vs Sample Mass Used for Analysis

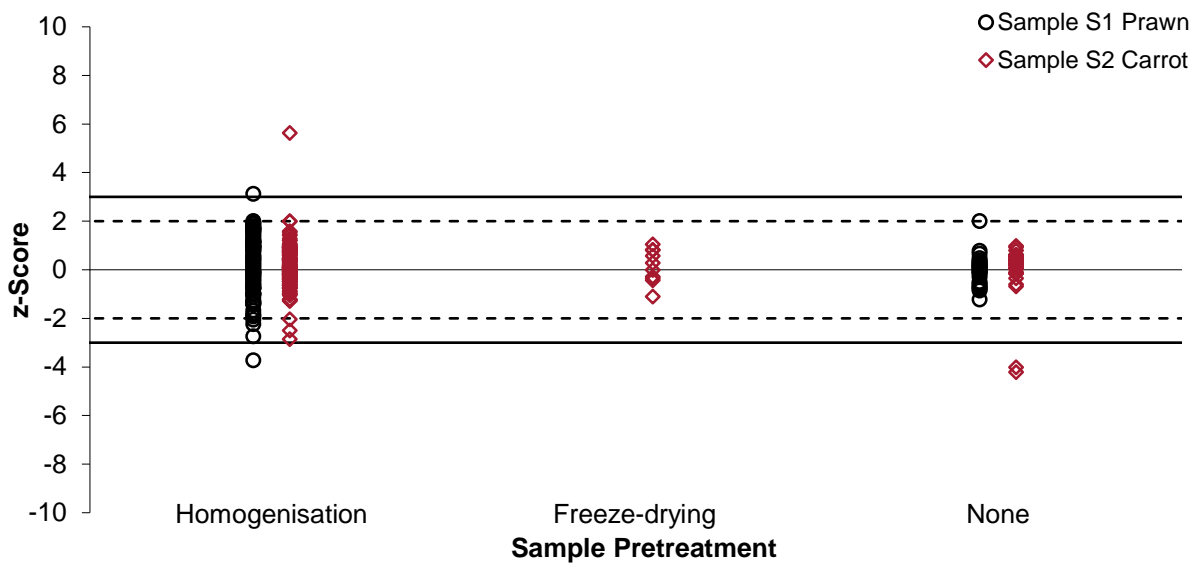


Figure 72 z-Score vs Pretreatment

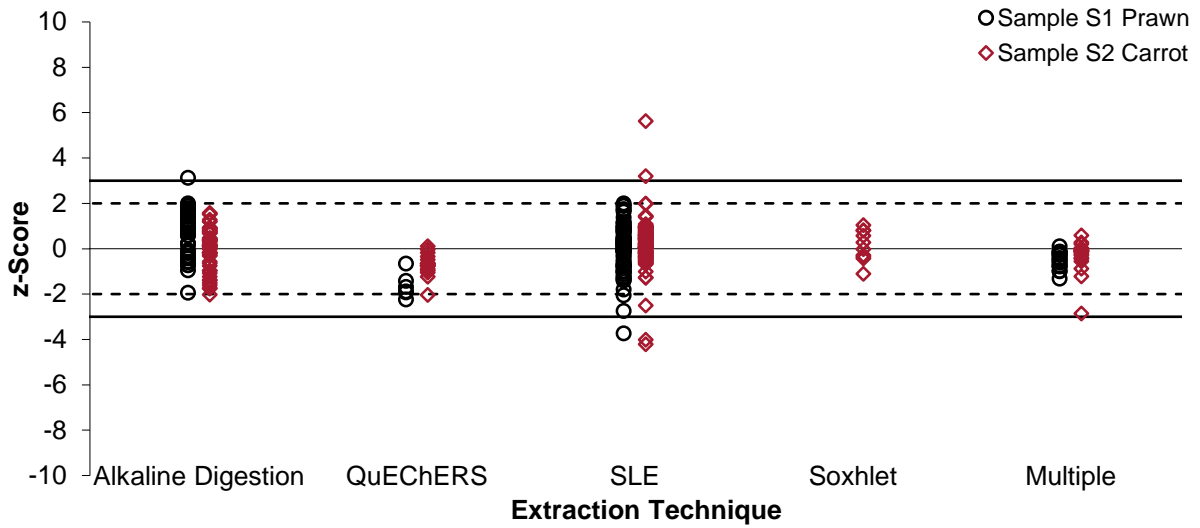


Figure 73 z-Score vs Extraction Technique

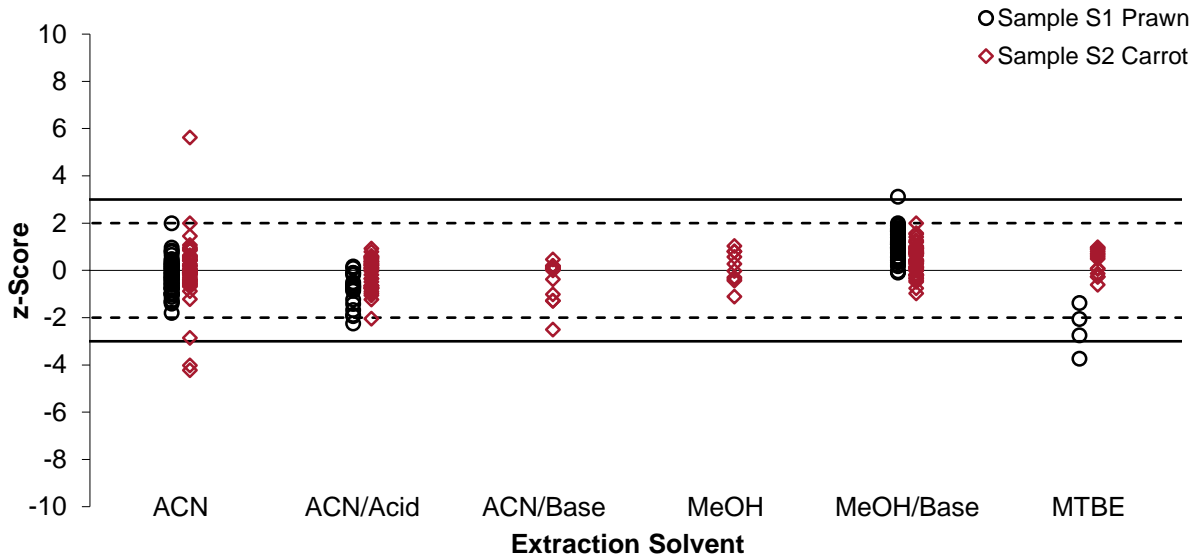


Figure 74 z-Score vs Extraction Solvent

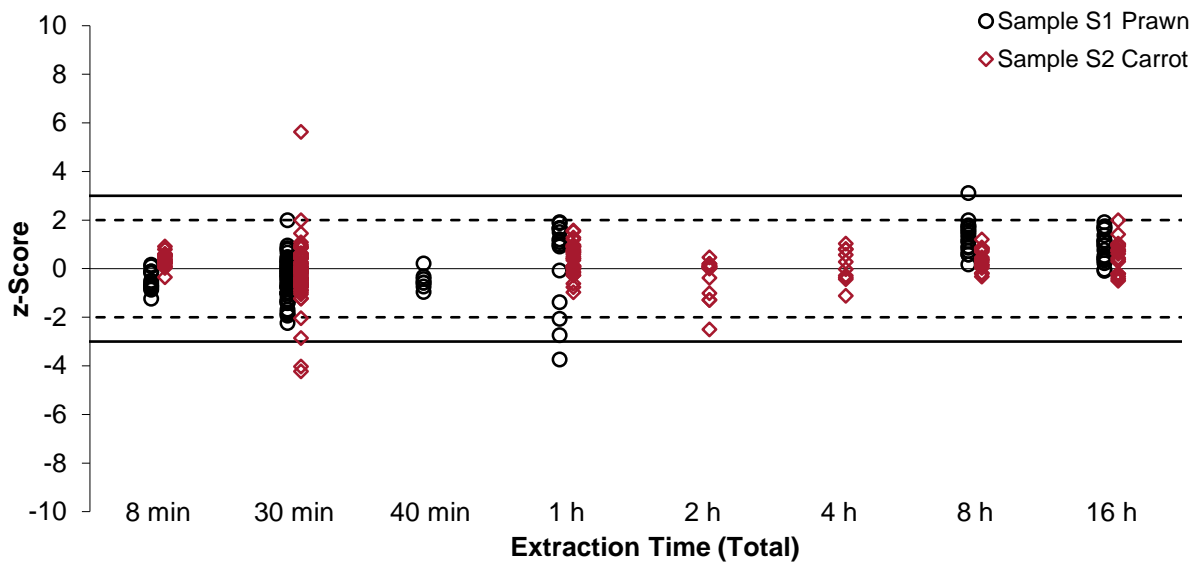


Figure 75 z-Score vs Extraction Time

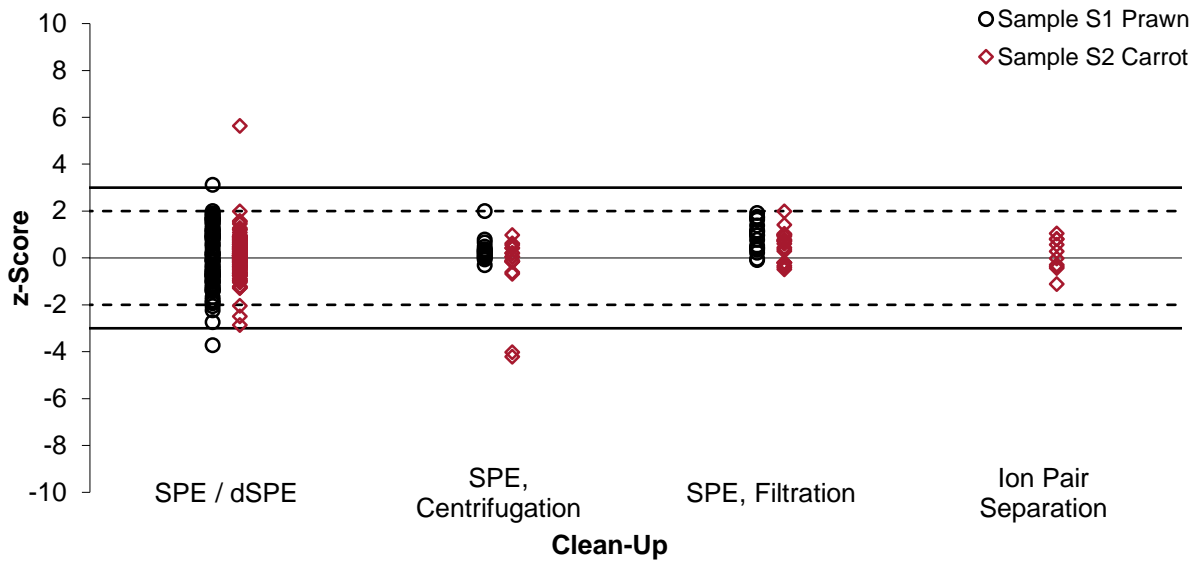


Figure 76 z-Score vs Clean-Up

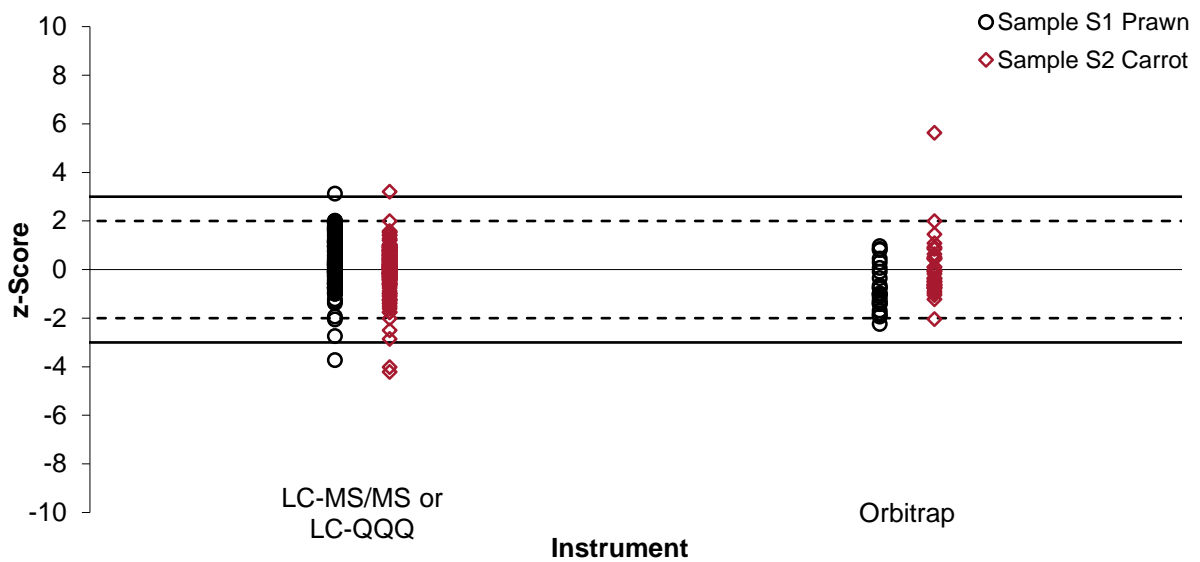


Figure 77 z-Score vs Measurement Instrument

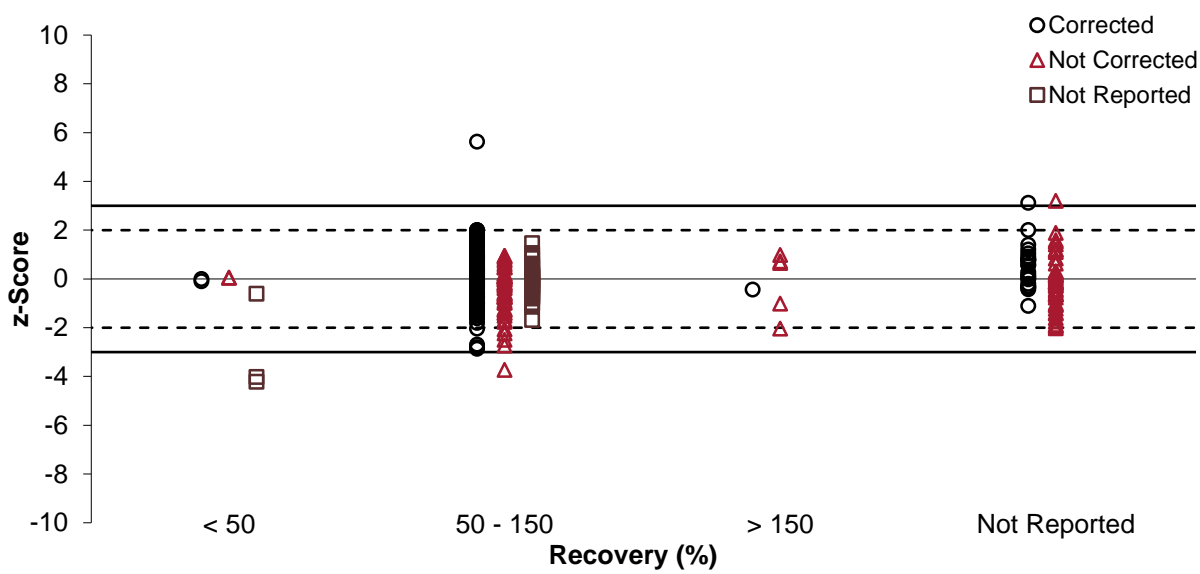


Figure 78 z-Score vs Recovery



Assigned values were not able to be set for a number of analytes in this study as the results reported by participants were not compatible (Section 6.1). For these analytes, it was seen that participants using basified methanol as their extraction solvent generally reported results closer to the spiked value (Figure 79), similar to what was observed for the scored analytes in this study.

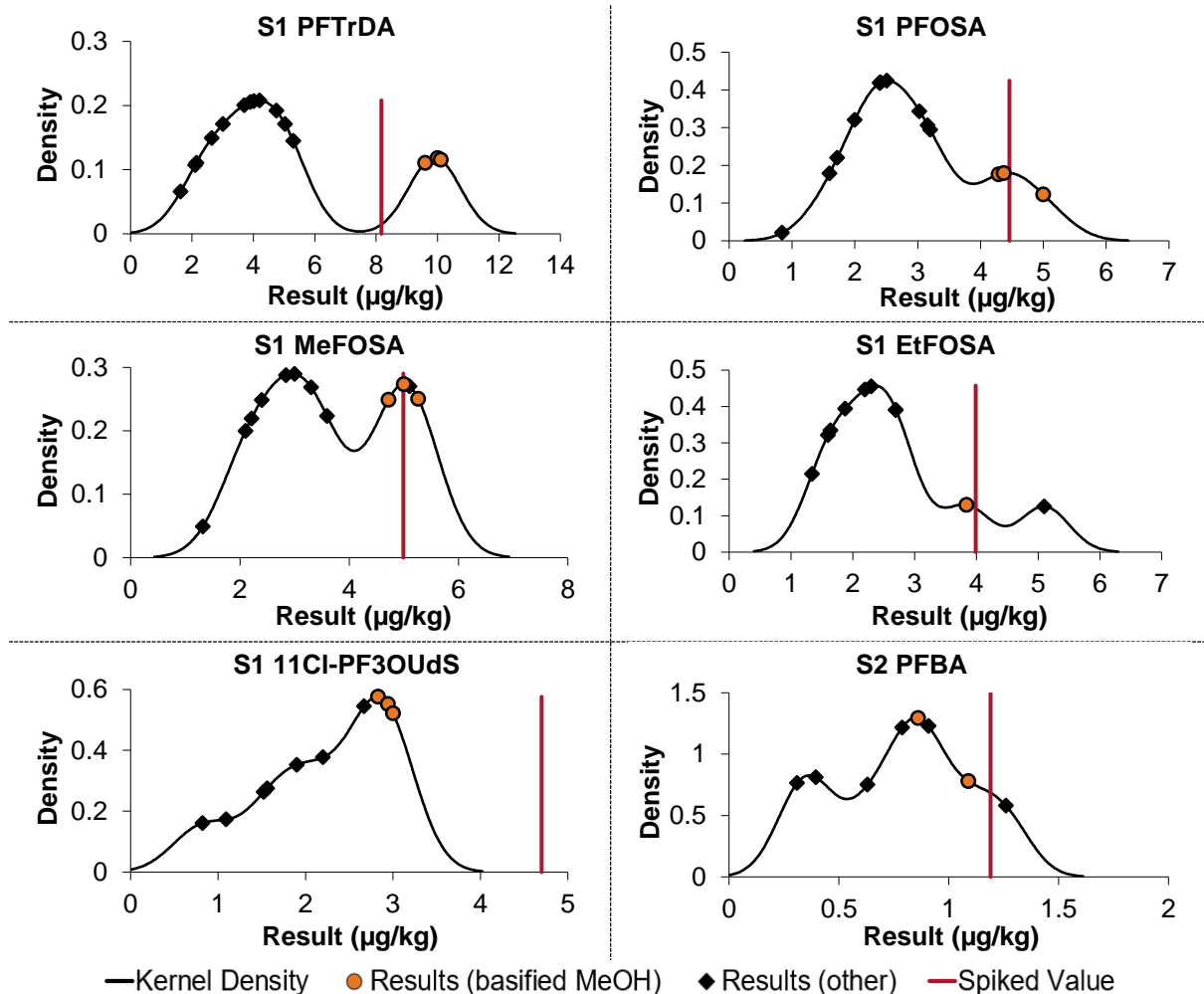


Figure 79 Results, Spiked Values and Kernel Densities for Non-Scored Analytes

### 6.10 Total vs Linear Isomers – PFHxS and PFOS

Participants were requested to report both the linear isomers and the total (sum of linear and branched isomers) for PFHxS and PFOS. A summary of results reported by participants is presented in Table 57. The majority of participants reported numeric results for both linear and total.

Table 57 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     | 12               | 3           | 1          | 13               | 2           | 3          |
| S2     | 12               | 3           | 1          | 14               | 1           | 1          |

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

## PFHxS

Summaries of participants' results for linear and total PFHxS in Samples S1 and S2 are presented in Figures 80 and 81.

Of the participants reporting numeric results for both linear and total PFHxS, the majority correctly reported the same result, or very similar results, for both. However, for Sample S1, Laboratory 13 reported a lower linear value compared to their total value (91% of total), and their results were not in agreement with each other within their respective uncertainties.

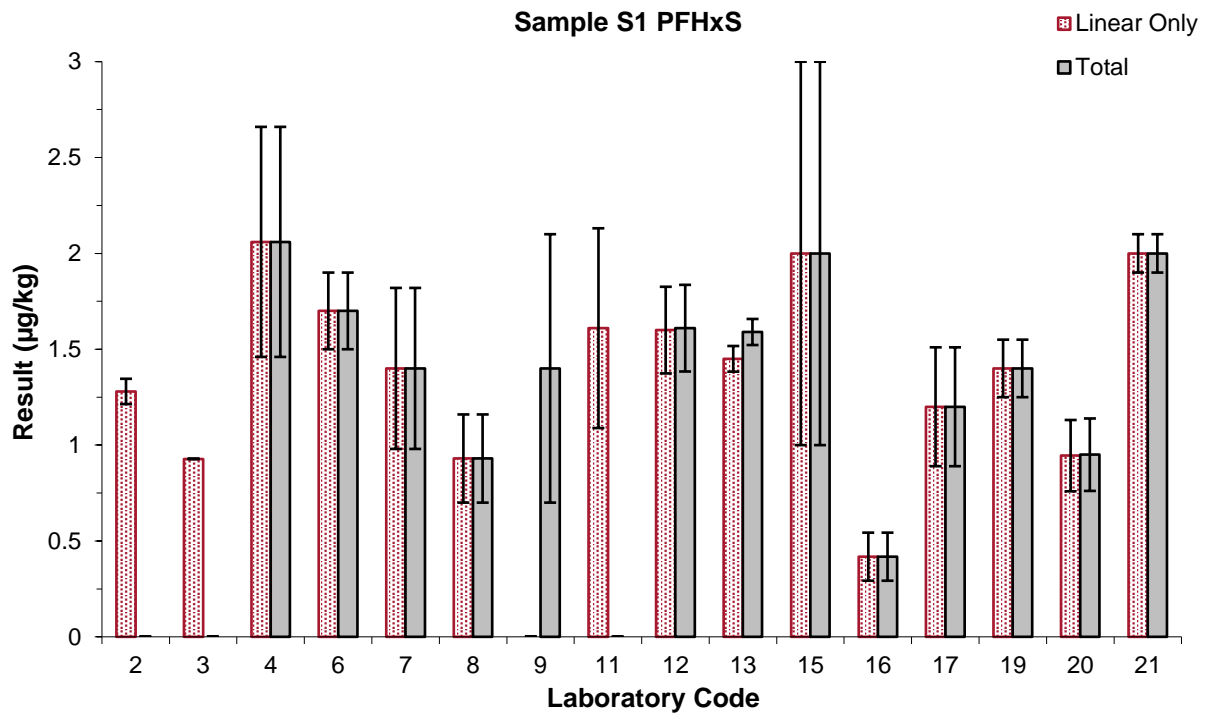


Figure 80 Participant Results for Sample S1 PFHxS (linear and total)

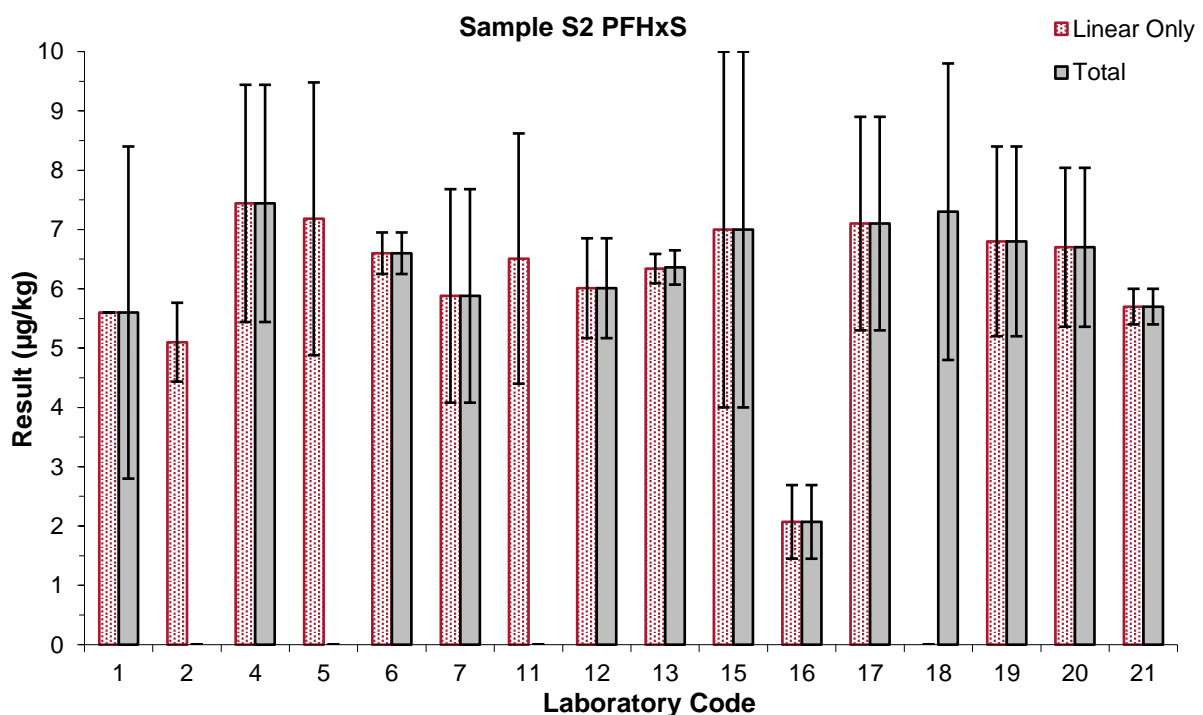


Figure 81 Participant Results for Sample S2 PFHxS (linear and total)

## PFOS

Summaries of participants' results for linear and total PFOS in Samples S1 and S2 are presented in Figures 82 and 83.

Laboratory **20** reported numeric results for both linear only and total PFOS in Sample S2, but only reported a linear result for Sample S1 PFOS (with their total result being 'NR').

Of the participants reporting numeric results for both linear and total PFOS, the majority of participants correctly reported the same result, or very similar results, for both, and all were in agreement with each other within their respective uncertainties. For Sample S1, Laboratory **1** reported a higher value for linear isomers (115% of total).

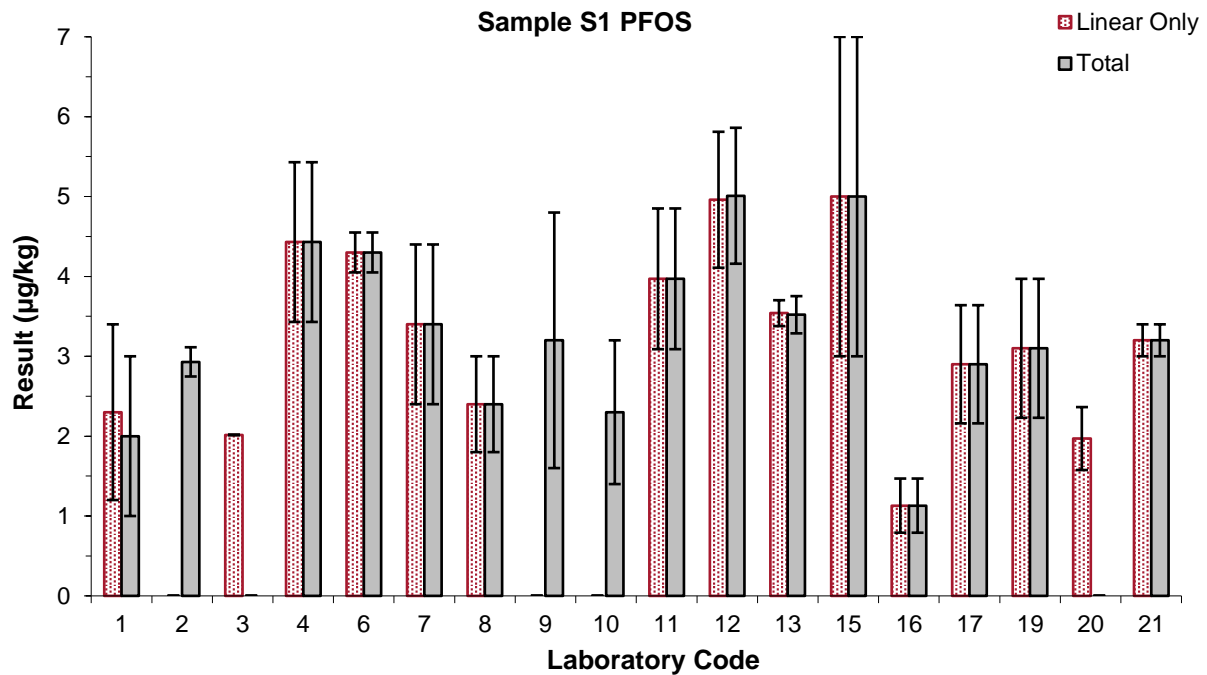


Figure 82 Participant Results for Sample S1 PFOS (linear and total)

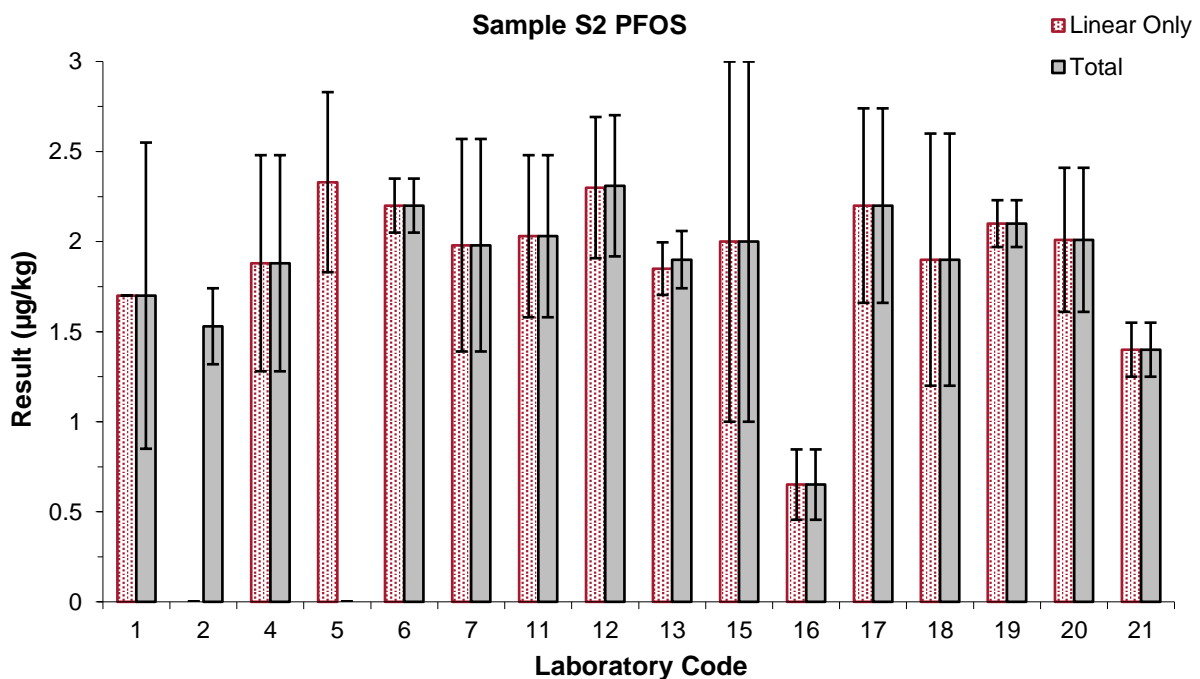


Figure 83 Participant Results for Sample S2 PFOS (linear and total)

### 6.11 Effects of Sample Matrix

The samples in this study were spiked prawn (Sample S1) and carrot (Sample S2). A summary of the results reported and  $z$ -scores obtained by matrix is presented in Table 58.

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

Table 58 Result Comparison by Matrix

| Sample | Matrix | Expected Number of Results | Numeric Results Reported | $z$ -Scores Calculated | Satisfactory $z$ -Scores |
|--------|--------|----------------------------|--------------------------|------------------------|--------------------------|
| S1     | Prawn  | 437                        | 305 (70%)                | 244                    | 195 (80%)                |
| S2     | Carrot | 374                        | 292 (78%)                | 284                    | 258 (91%)                |

## 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 59 and 60, and Figure 84.

Table 59 Summary of Participants' Sample S1 Results\*

| Lab. Code | PFBS  | PFPeS | PFHxS | PFHxS (linear) | PFHpS | PFOS | PFOS (linear) | PFNS | PFBA  | PFPeA | PFHxA | PFHpA | PFOA  | PFNA  | PFDA  | PFUdA | ADONA | 9CI-PF3ONS |
|-----------|-------|-------|-------|----------------|-------|------|---------------|------|-------|-------|-------|-------|-------|-------|-------|-------|-------|------------|
| AV        | 0.302 | 4.19  | 1.64  | 1.61           | 1.67  | 3.62 | 3.74          | 8.0  | 2.03  | 1.04  | 4.61  | 6.02  | 6.44  | 0.427 | 0.74  | 1.12  | 4.5   | 11.0       |
| SV        | 0.399 | 4.65  | 1.89  | 1.89           | 2.00  | 4.77 | 4.77          | 11.5 | 2.96  | 1.13  | 5.31  | 7.54  | 7.92  | 0.503 | 0.902 | 1.21  | 5.64  | 14.4       |
| 1         | < 2   | 3     | < 2   | < 2            | < 1   | 2    | 2.3           | 5    | < 2   | < 2   | 4     | 4     | < 5   | < 2   | < 5   | < 2   | NT    | NT         |
| 2         | 0.244 | 3.29  | NR    | 1.28           | 1.20  | 2.93 | NR            | 7.25 | 1.48  | 0.479 | 3.42  | 4.85  | 4.91  | 0.188 | 0.438 | 0.760 | 3.69  | 7.47       |
| 3         | 0.216 | 2.662 | NT    | 0.928          | 0.967 | NT   | 2.015         | NT   | 1.104 | 0.562 | 2.979 | 3.958 | 3.426 | 0.206 | 0.304 | NT    | 3.013 | 6.268      |
| 4         | <0.5  | 4.68  | 2.06  | 2.06           | 1.73  | 4.43 | 4.43          | 11.3 | 2.67  | 1.16  | 5.4   | 8.13  | 8.38  | 0.523 | 0.986 | 1.52  | 7.31  | 12.6       |
| 5         | NS    | NS    | NS    | NS             | NS    | NS   | NS            | NS   | NS    | NS    | NS    | NS    | NS    | NS    | NS    | NS    | NS    | NS         |
| 6         | <1    | 4.1   | 1.7   | 1.7            | 2.3   | 4.3  | 4.3           | 6.4  | <5    | <1    | 4.9   | 6.0   | 6.4   | <1    | <1    | <1    | NT    | NT         |
| 7         | 0.311 | 4.96  | 1.40  | 1.40           | 1.40  | 3.4  | 3.4           | 7.63 | 1.86  | 0.885 | 4.32  | 5.58  | 6.03  | 0.42  | 0.741 | 0.994 | 3.48  | 12.1       |
| 8         | <0.5  | 2.57  | 0.93  | 0.93           | 0.99  | 2.40 | 2.40          | 4.49 | 1.16  | 0.55  | 2.80  | 4.33  | 4.26  | <0.5  | <0.5  | 0.63  | 3.25  | 5.88       |
| 9         | <0.5  | NT    | 1.4   | NT             | NT    | 3.2  | NT            | NT   | NT    | NT    | 4.8   | 5.5   | 5.7   | 0.4   | 0.6   | NT    | NT    | NT         |
| 10        | <1    | NR    | <1    | NR             | <1    | 2.3  | NR            | 3.7  | 1.5   | <1    | 2.4   | 3.7   | 4.3   | <1    | NR    | <1    | NR    | NR         |
| 11        | 0.315 | 4.51  | NT    | 1.61           | 1.65  | 3.97 | 3.97          | 9.24 | 2.89  | 1.05  | 4.70  | 6.82  | 6.86  | 0.441 | 0.776 | 1.05  | NT    | NT         |
| 12        | 0.316 | 5.15  | 1.61  | 1.6            | 1.99  | 5.01 | 4.96          | 10.8 | 2.21  | 1.15  | 5     | 6.43  | 7.7   | 0.521 | 0.913 | 1.43  | 6.04  | 12.7       |
| 13        | 0.265 | 3.55  | 1.59  | 1.45           | 1.34  | 3.52 | 3.54          | 5.89 | 1.79  | 0.878 | 4.49  | 6.15  | 6.11  | 0.343 | 0.705 | 0.953 | 4.11  | 10.1       |
| 15        | <1    | 5     | 2     | 2              | 2     | 5    | 5             | 11   | 2     | <2    | 6     | 8     | 8     | <1    | <2    | <2    | 6     | 13         |
| 16        | < 0.1 | 1.97  | 0.418 | 0.418          | 0.418 | 1.13 | 1.13          | NT   | 0.582 | < 0.3 | 1.38  | 1.88  | 1.98  | 0.111 | 0.19  | 0.246 | NR    | NR         |
| 17        | 0.36  | 4.9   | 1.2   | 1.2            | 1.2   | 2.9  | 2.9           | 9.3  | 1.3   | 1.1   | 3.9   | 5.6   | 5.5   | 0.34  | 0.75  | 1.1   | 3.6   | 12         |
| 18        | <1    | 2.46  | <1    | NT             | <1    | <1   | <1            | NT   | <5    | <2    | 3.34  | 2.72  | 1.64  | <1    | <1    | <1    | NT    | NT         |
| 19        | < 1.0 | 4.1   | 1.4   | 1.4            | 1.4   | 3.1  | 3.1           | 7.2  | 2.1   | < 1.0 | 4.7   | 5.8   | 5.7   | < 1.0 | < 1.0 | < 1.0 | 4.4   | 8.3        |
| 20        | 0.220 | 2.33  | 0.950 | 0.945          | 0.811 | NR   | 1.97          | 3.58 | NT    | NT    | 3.41  | 3.85  | 3.92  | 0.224 | 0.346 | 0.450 | NT    | NT         |
| 21        | <1    | 4.4   | 2.0   | 2.0            | 2.2   | 3.2  | 3.2           | 4.9  | <5    | <1    | 5.7   | 5.9   | 6.2   | <1    | <1    | <1    | NT    | NT         |

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

Table 60 Summary of Participants' Sample S2 Results\*

| Lab. Code | PFBS  | PFPeS | PFHxS | PFHxS (linear) | PFHpS | PFOS  | PFOS (linear) | PFNS | PFDS | PFPeA | PFHxA | PFHpA | PFOA  | PFNA  | PFDA  | PFOSA | MeFOSA | EtFOSA | 6:2 FTS | GenX | ADONA |
|-----------|-------|-------|-------|----------------|-------|-------|---------------|------|------|-------|-------|-------|-------|-------|-------|-------|--------|--------|---------|------|-------|
| AV        | 0.82  | 8.30  | 6.54  | 6.44           | 2.99  | 1.95  | 2.01          | 1.52 | 6.76 | 2.07  | 7.83  | 1.53  | 1.24  | 2.36  | 9.58  | 4.03  | 3.98   | 3.11   | 1.69    | 9.8  | 13.2  |
| HV        | 0.83  | 8.3   | 5.9   | 5.9            | 2.62  | 1.88  | 1.88          | 1.51 | 6.4  | 1.93  | 7.3   | 1.52  | 1.20  | 2.22  | 9.4   | 4.2   | -      | -      | 1.91    | 10.3 | 16.1  |
| SV        | 0.891 | 7.47  | 6.61  | 6.61           | 3.00  | 2.12  | 2.12          | 1.72 | 6.80 | 2.20  | 7.45  | 1.50  | 1.20  | 2.31  | 9.47  | 4.95  | 4.99   | 3.99   | 1.89    | 11.1 | 14.0  |
| 1         | 0.7   | 7.5   | 5.6   | 5.6            | 2.6   | 1.7   | 1.7           | 1.2  | NT   | 1.7   | 8     | 1.3   | 1     | 2.0   | 8.9   | 4     | 3      | 3      | 1       | NT   | NT    |
| 2         | 0.625 | 6.65  | NR    | 5.10           | 2.11  | 1.53  | NR            | 1.44 | 6.35 | 1.45  | 5.71  | 1.16  | 0.935 | 1.69  | 7.72  | 2.92  | NT     | NT     | 1.62    | 7.34 | 10.8  |
| 3         | NS    | NS    | NS    | NS             | NS    | NS    | NS            | NS   | NS   | NS    | NS    | NS    | NS    | NS    | NS    | NS    | NS     | NS     | NS      | NS   | NS    |
| 4         | 0.957 | 8.47  | 7.44  | 7.44           | 2.87  | 1.88  | 1.88          | 1.53 | 7.22 | 2.14  | 8.25  | 1.8   | 1.34  | 2.44  | 11    | 4.41  | <5     | <5     | 1.82    | 11.4 | 16.4  |
| 5         | 0.99  | NT    | NT    | 7.18           | NT    | NT    | 2.33          | NT   | 7.86 | 1.89  | 7.38  | 1.19  | 1.31  | 2.35  | 8.89  | NT    | NT     | NT     | NT      | NT   | NT    |
| 6         | <1    | 9.7   | 6.6   | 6.6            | 3.1   | 2.2   | 2.2           | <1   | 6.6  | <1    | 7.5   | 1.4   | 1.1   | 2.3   | 9.2   | 3.8   | 5.1    | 5.1    | 1.9     | <1   | <1    |
| 7         | 0.873 | 8.68  | 5.88  | 5.88           | 2.64  | 1.98  | 1.98          | 1.45 | 6.80 | 2.68  | 8.65  | 1.62  | 1.45  | 2.86  | 9.88  | 4.12  | 3.15   | 2.06   | 1.65    | 7.86 | 11.2  |
| 8         | NS    | NS    | NS    | NS             | NS    | NS    | NS            | NS   | NS   | NS    | NS    | NS    | NS    | NS    | NS    | NS    | NS     | NS     | NS      | NS   | NS    |
| 9         | NT    | NT    | NT    | NT             | NT    | NT    | NT            | NT   | NT   | NT    | NT    | NT    | NT    | NT    | NT    | NT    | NT     | NT     | NT      | NT   | NT    |
| 10        | NS    | NS    | NS    | NS             | NS    | NS    | NS            | NS   | NS   | NS    | NS    | NS    | NS    | NS    | NS    | NS    | NS     | NS     | NS      | NS   | NS    |
| 11        | 0.709 | 8.04  | NT    | 6.51           | 3.32  | 2.03  | 2.03          | 1.71 | 8.08 | 2.25  | 7.59  | 1.66  | 1.20  | 2.31  | 9.97  | 3.54  | 0.620  | 0.610  | NT      | NT   | NT    |
| 12        | 0.787 | 8.78  | 6.01  | 6.01           | 3.22  | 2.31  | 2.3           | 1.82 | 8.67 | 1.98  | 7.51  | 1.38  | 1.48  | 2.57  | 11.1  | 4.64  | 4.8    | 4.35   | 2.02    | 11   | 15.1  |
| 13        | 0.782 | 8.08  | 6.36  | 6.34           | 3     | 1.9   | 1.85          | 1.35 | 5.57 | 1.95  | 7.79  | 1.59  | 1.31  | 2.64  | 9.42  | 4.04  | 3.91   | 3.11   | 1.54    | 4.19 | 9.98  |
| 15        | <1    | 9     | 7     | 7              | 3     | 2     | 2             | 2    | 8    | 2     | 8     | 2     | 1     | 2     | 11    | 5     | 5      | <5     | 2       | 10   | 15    |
| 16        | 0.238 | 4.09  | 2.07  | 2.07           | 1.14  | 0.651 | 0.651         | NT   | 1.55 | 0.612 | 2.44  | 0.462 | 0.381 | 0.652 | 2.85  | 1.19  | 1.86   | 1.63   | 0.618   | NR   | NR    |
| 17        | 0.89  | 7.7   | 7.1   | 7.1            | 2.6   | 2.2   | 2.2           | 1.4  | 6.0  | 4.4   | 10.1  | 1.8   | 1.45  | 3.3   | 9.4   | 4.9   | 3.6    | 2.8    | 2.0     | 10   | 13    |
| 18        | <1    | 9.86  | 7.3   | NT             | 3.04  | 1.9   | 1.9           | NT   | 5.94 | 2.4   | 7.92  | 1.68  | 1.4   | 2.78  | 10.96 | <5    | <5     | 2.94   | 2.02    | NT   | NT    |
| 19        | NR    | 9.1   | 6.8   | 6.8            | 3.2   | 2.1   | 2.1           | 1.7  | 8.0  | 2.3   | 8.0   | 1.7   | 1.3   | 2.6   | 8.9   | 4.1   | 4.3    | 3.6    | 1.7     | 11   | 14    |
| 20        | 0.853 | 6.17  | 6.70  | 6.70           | 3.27  | 2.01  | 2.01          | 1.13 | 3.38 | NT    | 7.95  | 1.56  | 1.26  | 2.18  | 9.68  | NT    | NT     | NT     | 1.35    | 9.90 | NT    |
| 21        | <1    | 8.0   | 5.7   | 5.7            | 3.9   | 1.4   | 1.4           | <1   | 4.9  | <1    | 7.4   | 1.2   | 1.0   | 1.6   | 6.2   | 2.4   | 3.0    | 3.2    | 1.1     | <1   | NT    |

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

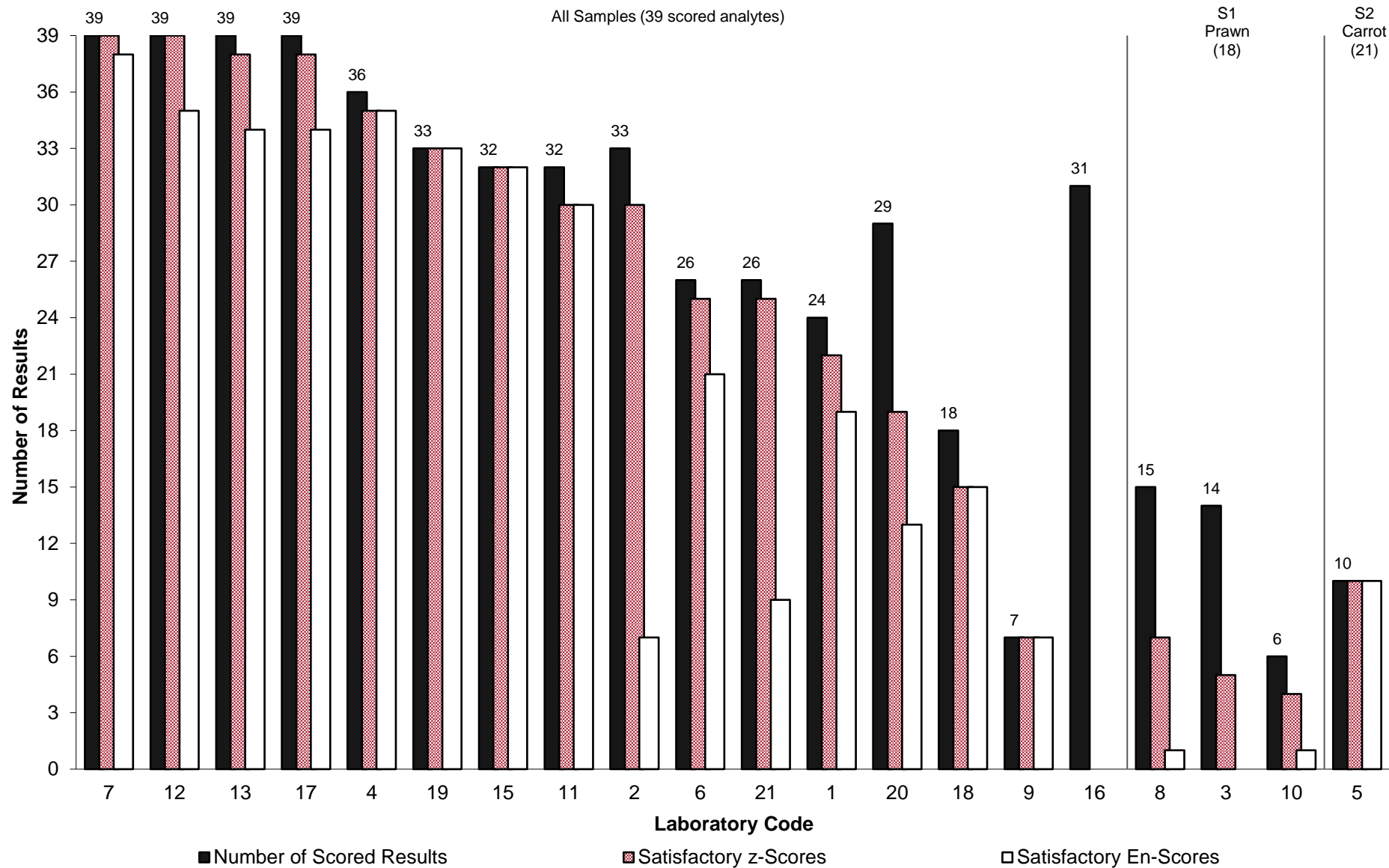


Figure 84 Summary of Participants' Performance

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

NMI has coordinated PFAS in Biota and Food PT studies since 2016. A summary of participation and reported results rates over the last seven studies (2016 to 2022) is presented in Figure 85. Proportions of PFAS analysed and numeric results reported have remained relatively high over this period, despite the increased number of spiked analytes as compared to the original studies.

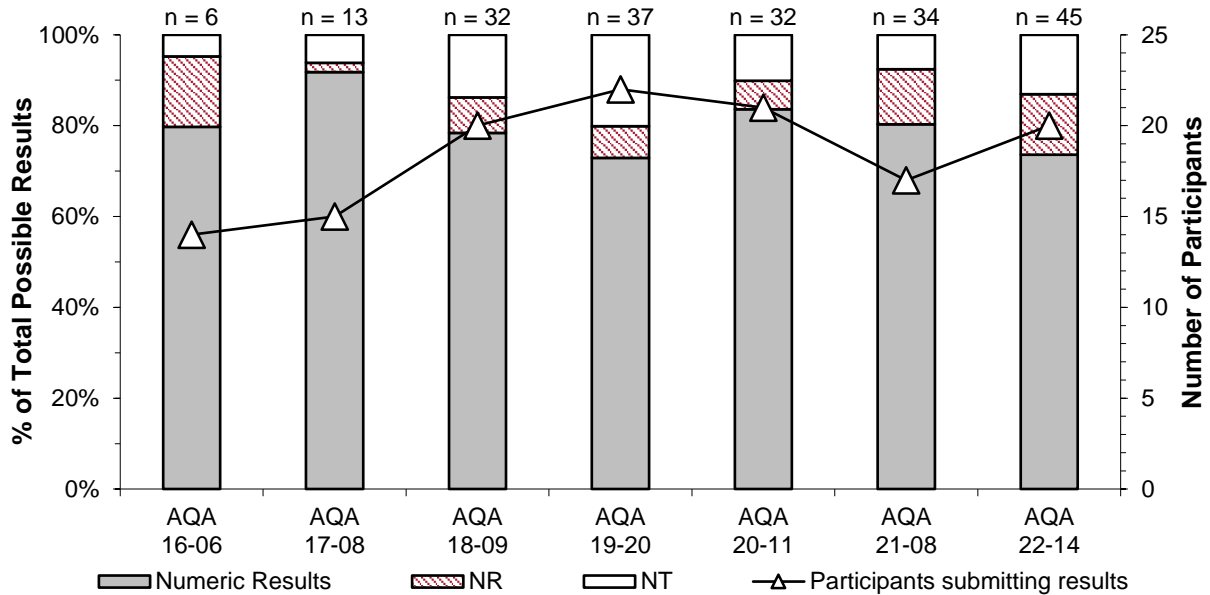


Figure 85 Summary of Participation and Reported Results in PFAS in Biota and Food PT Studies (n = number of spiked analytes).

A summary of the satisfactory performance (presented as a percentage of the total number of scores for each study) in PFAS in Biota and Food PT studies over the last seven studies (2016 to 2022) is presented in Figure 86. The target SD used to calculate z-scores has been kept constant at 20% PCV, which enables comparison between different studies. Proportions of satisfactory scores has remained relatively consistent, with the average proportion of satisfactory scores over this period being 89% for z-scores and 76% for  $E_n$ -scores.

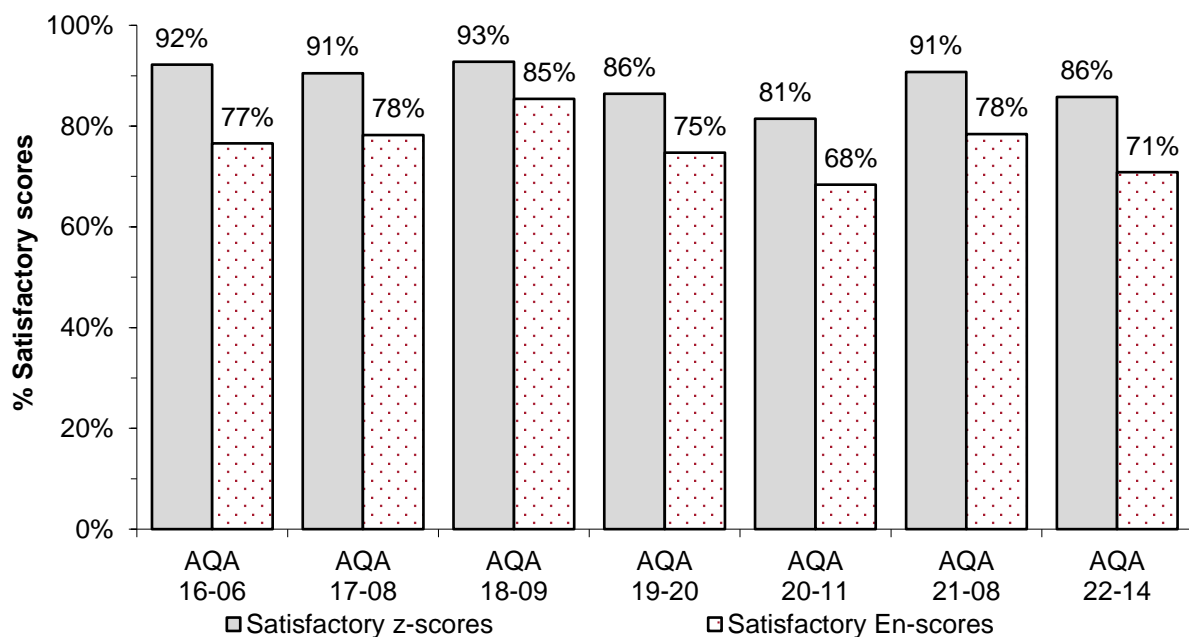


Figure 86 Summary of Participants' Performance for PFAS in Biota and Food PT Studies



The number of analytes assessed in each study has increased significantly as compared to the initial PFAS in Biota and Food study, and the studies have increased in size and complexity. As a point of comparison, PFOS and PFOA have been assessed in every study, and a summary of the proportion of satisfactory scores for these analytes over the last seven studies is presented in Figure 87.

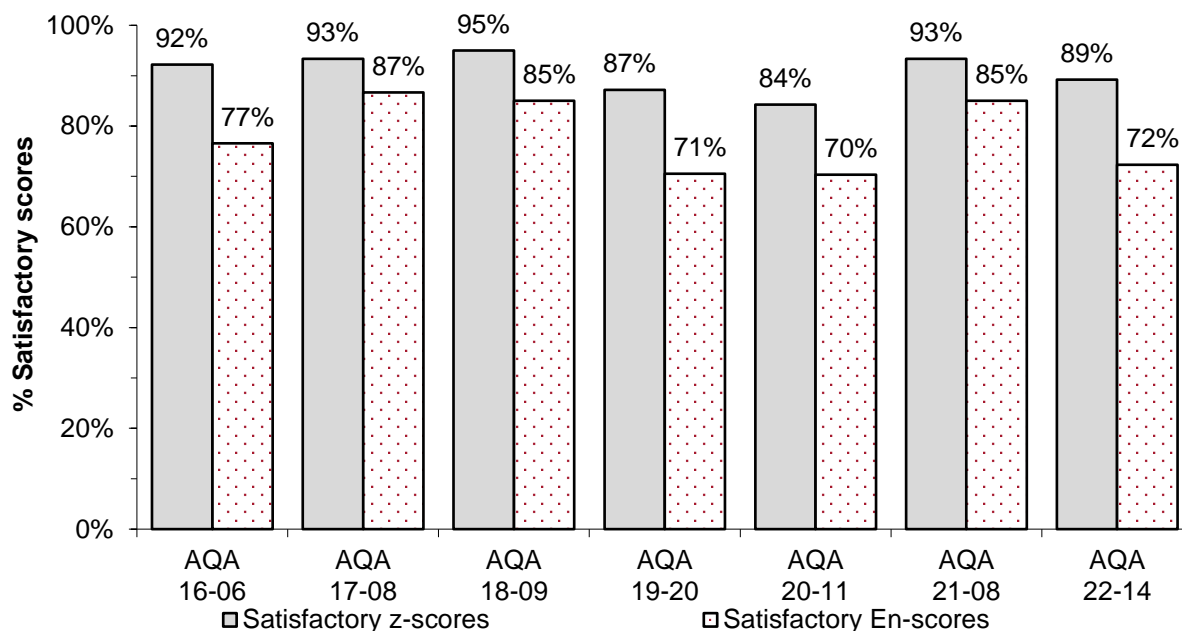


Figure 87 Summary of Participants' Performance for PFOS and PFOA in Biota and Food PT Studies

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 88 presents a summary of relative uncertainties as reported by participants over the last seven studies (2016 to 2022). Over this period, the vast majority of results were reported with uncertainties (95%), despite only around 60% participants reporting that they were accredited to ISO/IEC 17025. A small proportion of reported results consisted of numeric results with no uncertainty, or non-numeric results with uncertainties; the proportion of such results has reduced in recent studies as compared to the first PFAS in Biota and Food study run in 2016. Over the last three studies in particular, 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 satisfactory  $z$ -scores, but with smaller relative uncertainties and unsatisfactory  $E_n$ -scores, may need to assess whether their uncertainties have been underestimated.

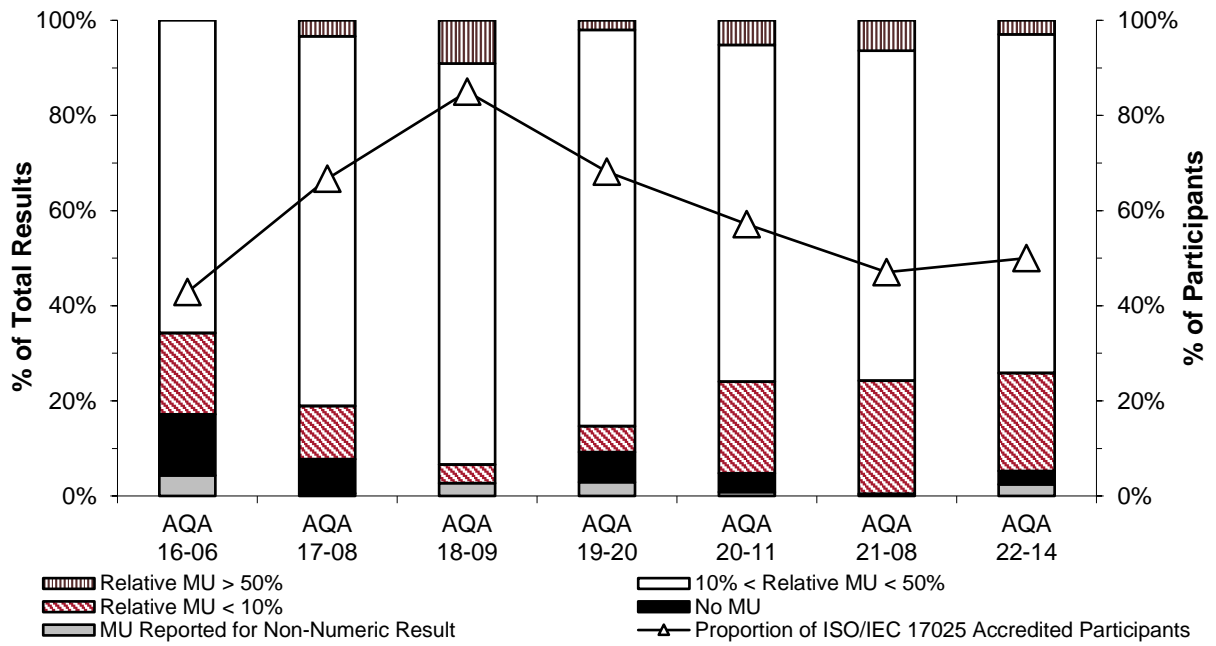


Figure 88 Summary of Participants' Relative Uncertainties for PFAS in Biota and Food PT Studies

## 7 REFERENCES

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## APPENDIX 1 SAMPLE PREPARATION

**Sample S1:** Prawns were blended to yield 550.6 g of puree. The pureed prawns were placed in a tray and sprayed with a spiking solution containing PFAS analytes in methanol. The prawns were thoroughly mixed, before being divided into patties of no more than 6 cm in diameter, placed on a tray, covered, and placed into the freezer overnight at -80 °C. The frozen patties were then ground using a Retsch SM2000 Knife Mill which was kept cold using liquid nitrogen and dry ice. The dry ice was then allowed to sublime off, before 5 g portions of the spiked prawns were packed into sample tubes. The tubes were labelled, shrink-wrapped, and then stored at -80 °C prior to dispatch.

**Sample S2:** Organic carrots were bought from a Sydney organic fruit and vegetable wholesaler. The carrots were rinsed, cut and blended. A stainless steel tray was lined with aluminium foil and the carrot was spread evenly over the tray. The tray was tilted at 45 degrees and a prepared composite in methanol was sprayed over the carrot with regular mixing steps to homogenise the carrot. The spiked carrot was then formed into patties of no more than 6 cm in diameter and placed on trays which were covered with baking paper. The trays were then placed into a freezer over the weekend 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 of at least 25 g of the spiked carrot was packed into sample tubes. The tubes were labelled, shrink-wrapped, and then stored at -80 °C prior to dispatch.

## APPENDIX 2 HOMOGENEITY AND STABILITY

No homogeneity or stability testing was conducted on Sample S1 prawn. This sample was prepared and packaged using a process previously demonstrated to produce homogeneous and stable PT samples; in particular, stability for PFAS analytes in prawn at room temperature has been demonstrated for at least two months previously.<sup>5</sup>

### A2.1 Homogeneity and Stability Testing – Sample S2 Carrot

#### Homogeneity Testing

Homogeneity and stability was conducted for Sample S2 carrot, which was prepared using a new method for PFAS in fresh produce, though the process used was similar to the preparation of PFAS in biota or meat samples.

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 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 61 to 76. Samples were found to be sufficiently homogeneous for use in a PT study with a target SD (as PCV) of 20%.

Table 61 Sample S2 PFBS Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 0.85           | 0.84        |
| 18               | 0.77           | 0.88        |
| 29               | 0.84           | 0.80        |
| 40               | 0.81           | 0.92        |
| 52               | 0.85           | 0.83        |
| 66               | 0.81           | 0.79        |
| 75               | 0.90           | 0.80        |
| Mean             | 0.83           |             |
| CV               | 5.2%           |             |

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

| Test                   | Value | Critical | Result      |
|------------------------|-------|----------|-------------|
| Cochran                | 0.331 | 0.727    | <b>Pass</b> |
| $s_{\text{an}}/\sigma$ | 0.309 | 0.500    | <b>Pass</b> |
| $s^2_{\text{sam}}$     | 0.000 | 0.009    | <b>Pass</b> |

Table 62 Sample S2 PFPeS Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 8.5            | 8.2         |
| 18               | 8.0            | 8.5         |
| 29               | 8.9            | 7.7         |
| 40               | 7.4            | 8.5         |
| 52               | 8.3            | 8.5         |
| 66               | 8.4            | 8.3         |
| 75               | 9.5            | 7.7         |
| Mean             | 8.3            |             |
| CV               | 6.4%           |             |

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

| Test                   | Value | Critical | Result      |
|------------------------|-------|----------|-------------|
| Cochran                | 0.519 | 0.727    | <b>Pass</b> |
| $s_{\text{an}}/\sigma$ | 0.404 | 0.500    | <b>Pass</b> |
| $s^2_{\text{sam}}$     | 0.000 | 1.171    | <b>Pass</b> |

Table 63 Sample S2 PFHxS Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 5.8            | 6.0         |
| 18               | 6.1            | 6.4         |
| 29               | 6.3            | 6.1         |
| 40*              | 5.1            | 6.3         |
| 52               | 5.6            | 6.2         |
| 66               | 5.8            | 5.8         |
| 75               | 5.7            | 5.4         |
| Mean             | 5.9            |             |
| CV               | 6.3%           |             |

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

| Test                   | Value | Critical | Result      |
|------------------------|-------|----------|-------------|
| Cochran                | 0.681 | 0.781    | <b>Pass</b> |
| $s_{\text{an}}/\sigma$ | 0.169 | 0.500    | <b>Pass</b> |
| $s^2_{\text{sam}}$     | 0.051 | 0.349    | <b>Pass</b> |

\* Results from container 40 were not included in the test for homogeneity, being identified as Cochran outliers due to the difference between replicates.<sup>12</sup>

Table 64 Sample S2 PFHpS Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 2.58           | 2.52        |
| 18               | 2.69           | 2.43        |
| 29               | 2.67           | 2.77        |
| 40               | 2.67           | 3.02        |
| 52               | 2.31           | 2.69        |
| 66               | 2.67           | 2.52        |
| 75               | 2.87           | 2.26        |
| Mean             | 2.62           |             |
| CV               | 7.8%           |             |

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

| Test            | Value | Critical | Result      |
|-----------------|-------|----------|-------------|
| Cochran         | 0.498 | 0.727    | <b>Pass</b> |
| $s_{an}/\sigma$ | 0.442 | 0.500    | <b>Pass</b> |
| $s^2_{sam}$     | 0.000 | 0.129    | <b>Pass</b> |

Table 65 Sample S2 PFOS Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 1.86           | 1.89        |
| 18               | 2.05           | 1.94        |
| 29               | 1.99           | 1.67        |
| 40               | 1.98           | 1.76        |
| 52               | 1.77           | 1.83        |
| 66               | 1.96           | 1.87        |
| 75               | 1.91           | 1.80        |
| Mean             | 1.88           |             |
| CV               | 5.6%           |             |

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

| Test            | Value | Critical | Result      |
|-----------------|-------|----------|-------------|
| Cochran         | 0.542 | 0.727    | <b>Pass</b> |
| $s_{an}/\sigma$ | 0.308 | 0.500    | <b>Pass</b> |
| $s^2_{sam}$     | 0.000 | 0.046    | <b>Pass</b> |

Table 66 Sample S2 PFNS Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 1.62           | 1.51        |
| 18               | 1.53           | 1.49        |
| 29               | 1.64           | 1.44        |
| 40               | 1.54           | 1.42        |
| 52               | 1.56           | 1.67        |
| 66               | 1.56           | 1.12        |
| 75               | 1.51           | 1.53        |
| Mean             | 1.51           |             |
| CV               | 8.8%           |             |

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

| Test            | Value | Critical | Result      |
|-----------------|-------|----------|-------------|
| Cochran         | 0.716 | 0.727    | <b>Pass</b> |
| $s_{an}/\sigma$ | 0.467 | 0.500    | <b>Pass</b> |
| $s^2_{sam}$     | 0.000 | 0.046    | <b>Pass</b> |

Table 67 Sample S2 PFDS Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 6.3            | 6.4         |
| 18               | 6.6            | 6.8         |
| 29               | 6.9            | 5.9         |
| 40               | 6.5            | 7.0         |
| 52               | 6.0            | 5.7         |
| 66               | 6.6            | 5.9         |
| 75               | 6.3            | 5.9         |
| Mean             | 6.4            |             |
| CV               | 6.4%           |             |

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

| Test            | Value | Critical | Result      |
|-----------------|-------|----------|-------------|
| Cochran         | 0.493 | 0.727    | <b>Pass</b> |
| $s_{an}/\sigma$ | 0.311 | 0.500    | <b>Pass</b> |
| $s^2_{sam}$     | 0.011 | 0.528    | <b>Pass</b> |

Table 68 Sample S2 PFPeA Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 1.84           | 1.96        |
| 18               | 1.98           | 1.80        |
| 29               | 1.97           | 1.93        |
| 40               | 2.05           | 1.88        |
| 52               | 1.99           | 1.87        |
| 66               | 1.80           | 1.96        |
| 75               | 2.04           | 2.00        |
| Mean             | 1.93           |             |
| CV               | 4.3%           |             |

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

| Test            | Value | Critical | Result      |
|-----------------|-------|----------|-------------|
| Cochran         | 0.285 | 0.727    | <b>Pass</b> |
| $s_{an}/\sigma$ | 0.243 | 0.500    | <b>Pass</b> |
| $s^2_{sam}$     | 0.000 | 0.041    | <b>Pass</b> |

Table 69 Sample S2 PFHxA Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 7.3            | 7.7         |
| 18               | 7.3            | 8.2         |
| 29               | 7.0            | 6.5         |
| 40               | 7.3            | 6.9         |
| 52               | 7.4            | 7.7         |
| 66               | 7.0            | 6.8         |
| 75               | 7.9            | 7.4         |
| Mean             | 7.3            |             |
| CV               | 6.3%           |             |

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

| Test            | Value | Critical | Result      |
|-----------------|-------|----------|-------------|
| Cochran         | 0.411 | 0.727    | <b>Pass</b> |
| $s_{an}/\sigma$ | 0.249 | 0.500    | <b>Pass</b> |
| $s^2_{sam}$     | 0.090 | 0.596    | <b>Pass</b> |



Table 70 Sample S2 PFHpA Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 1.58           | 1.47        |
| 18               | 1.64           | 1.40        |
| 29               | 1.52           | 1.46        |
| 40               | 1.52           | 1.69        |
| 52               | 1.58           | 1.46        |
| 66               | 1.46           | 1.53        |
| 75               | 1.40           | 1.55        |
| Mean             | 1.52           |             |
| CV               | 5.5%           |             |

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

| Test            | Value | Critical | Result      |
|-----------------|-------|----------|-------------|
| Cochran         | 0.397 | 0.727    | <b>Pass</b> |
| $s_{an}/\sigma$ | 0.331 | 0.500    | <b>Pass</b> |
| $s^2_{sam}$     | 0.000 | 0.032    | <b>Pass</b> |

Table 71 Sample S2 PFOA Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 1.17           | 1.19        |
| 18               | 1.22           | 1.19        |
| 29               | 1.22           | 1.27        |
| 40*              | 1.18           | 1.06        |
| 52               | 1.18           | 1.19        |
| 66               | 1.24           | 1.25        |
| 75               | 1.23           | 1.21        |
| Mean             | 1.20           |             |
| CV               | 4.1%           |             |

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

| Test            | Value | Critical | Result      |
|-----------------|-------|----------|-------------|
| Cochran         | 0.649 | 0.781    | <b>Pass</b> |
| $s_{an}/\sigma$ | 0.078 | 0.500    | <b>Pass</b> |
| $s^2_{sam}$     | 0.001 | 0.012    | <b>Pass</b> |

\* Results from container 40 were not included in the test for homogeneity, being identified as Cochran outliers due to the difference between replicates.<sup>12</sup>

Table 72 Sample S2 PFNA Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 2.32           | 2.25        |
| 18               | 2.17           | 2.23        |
| 29               | 2.22           | 2.02        |
| 40               | 2.25           | 2.11        |
| 52               | 2.35           | 2.25        |
| 66               | 2.24           | 2.17        |
| 75               | 2.35           | 2.20        |
| Mean             | 2.22           |             |
| CV               | 4.0%           |             |

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

| Test            | Value | Critical | Result      |
|-----------------|-------|----------|-------------|
| Cochran         | 0.380 | 0.727    | <b>Pass</b> |
| $s_{an}/\sigma$ | 0.193 | 0.500    | <b>Pass</b> |
| $s^2_{sam}$     | 0.001 | 0.048    | <b>Pass</b> |

Table 73 Sample S2 PFDA Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 9.5            | 10.1        |
| 18               | 9.4            | 9.5         |
| 29               | 9.1            | 9.6         |
| 40               | 10.2           | 9.5         |
| 52               | 9.0            | 9.3         |
| 66               | 9.8            | 8.4         |
| 75               | 9.1            | 8.8         |
| Mean             | 9.4            |             |
| CV               | 5.3%           |             |

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

| Test            | Value | Critical | Result      |
|-----------------|-------|----------|-------------|
| Cochran         | 0.615 | 0.727    | <b>Pass</b> |
| $s_{an}/\sigma$ | 0.263 | 0.500    | <b>Pass</b> |
| $s^2_{sam}$     | 0.002 | 1.015    | <b>Pass</b> |

Table 74 Sample S2 6:2 FTS Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 1.71           | 1.69        |
| 18               | 1.95           | 1.76        |
| 29               | 1.85           | 2.26        |
| 40               | 2.08           | 2.17        |
| 52               | 1.48           | 2.00        |
| 66               | 1.74           | 1.78        |
| 75               | 2.12           | 2.12        |
| Mean             | 1.91           |             |
| CV               | 12%            |             |

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

| Test            | Value | Critical | Result      |
|-----------------|-------|----------|-------------|
| Cochran         | 0.562 | 0.727    | <b>Pass</b> |
| $s_{an}/\sigma$ | 0.488 | 0.500    | <b>Pass</b> |
| $s^2_{sam}$     | 0.017 | 0.077    | <b>Pass</b> |

Table 75 Sample S2 GenX Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5                | 10.2           | 10.7        |
| 18               | 10.1           | 10.0        |
| 29               | 10.4           | 10.2        |
| 40               | 10.3           | 10.4        |
| 52               | 10.8           | 10.3        |
| 66               | 10.3           | 10.0        |
| 75               | 10.5           | 10.4        |
| Mean             | 10.3           |             |
| CV               | 2.3%           |             |

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

| Test            | Value | Critical | Result      |
|-----------------|-------|----------|-------------|
| Cochran         | 0.393 | 0.727    | <b>Pass</b> |
| $s_{an}/\sigma$ | 0.109 | 0.500    | <b>Pass</b> |
| $s^2_{sam}$     | 0.005 | 0.881    | <b>Pass</b> |

Table 76 Sample S2 ADONA Homogeneity Testing

| Container Number | Result (µg/kg) |             |
|------------------|----------------|-------------|
|                  | Replicate 1    | Replicate 2 |
| 5*               | 24.8           | 17.1        |
| 18               | 15.8           | 13.3        |
| 29               | 15.8           | 14.8        |
| 40               | 15.9           | 16.4        |
| 52               | 14.7           | 15.7        |
| 66               | 15.8           | 14.7        |
| 75               | 15.7           | 15.1        |
| Mean             | 16.1           |             |
| CV               | 17%            |             |

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

| Test            | Value | Critical | Result      |
|-----------------|-------|----------|-------------|
| Cochran         | 0.603 | 0.781    | <b>Pass</b> |
| $s_{an}/\sigma$ | 0.302 | 0.500    | <b>Pass</b> |
| $s^2_{sam}$     | 0.000 | 3.318    | <b>Pass</b> |

\* Results from container 5 were not included in the test for homogeneity, being identified as Cochran outliers due to the difference between replicates.<sup>12</sup>

### Stability Testing

Sample S2 carrot analysis was performed as described above.

The carrot samples were analysed at an initial time point in August 2022 (the start of the PT study). On the sample dispatch date, a sample was 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 the participants (October 2022), before being analysed, 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 October 2022 and November 2022 (the conclusion of the PT study).

Results were in good agreement with each other and the assigned value within their respective uncertainties (Figures 89 to 105, T = Transportation Stability and F = Freezer Stability). The samples were also shown to be adequately stable when assessed against the criteria specified in ISO 13528:2022.<sup>6</sup>

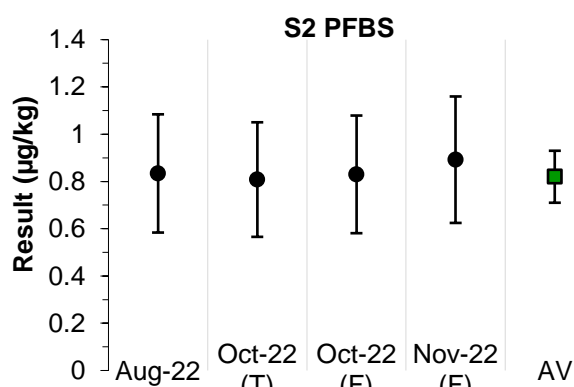


Figure 89 S2 PFBS Stability Testing

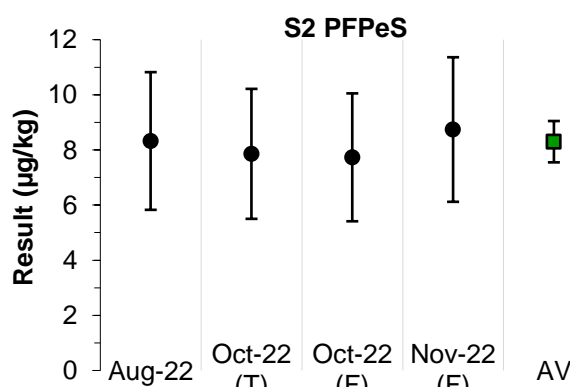


Figure 90 S2 PFPeS Stability Testing

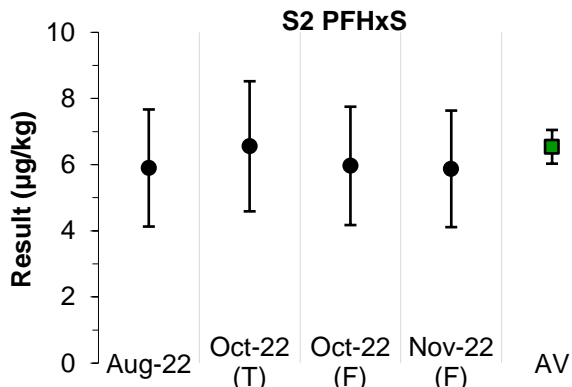


Figure 91 S2 PFHxS Stability Testing

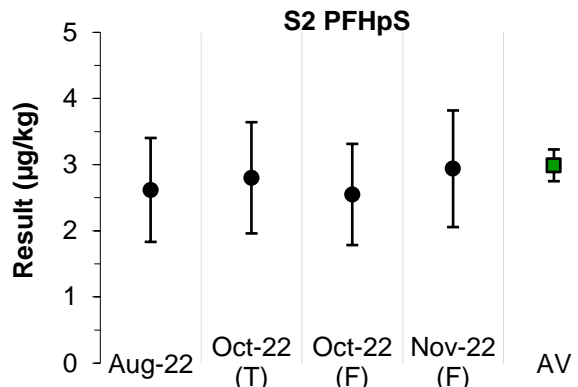


Figure 92 S2 PFHpS Stability Testing

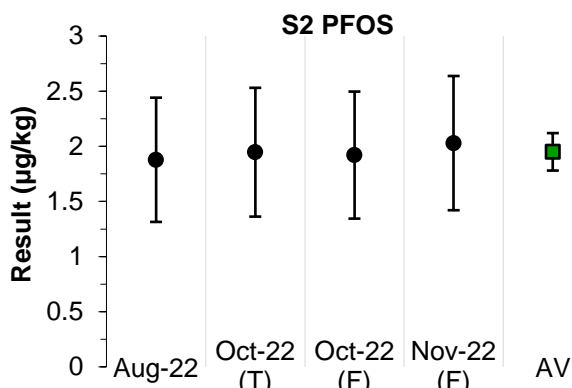


Figure 93 S2 PFOS Stability Testing

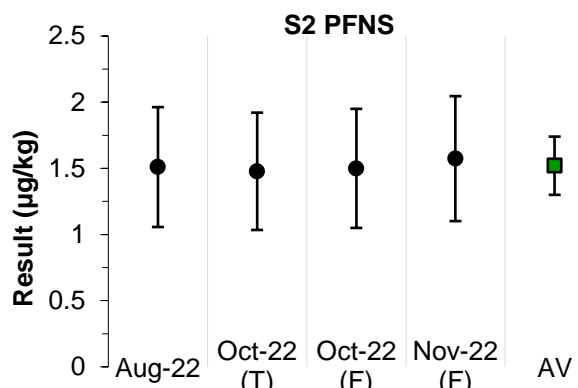


Figure 94 S2 PFNS Stability Testing

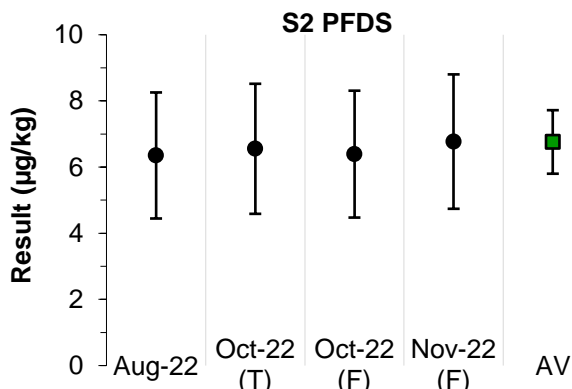


Figure 95 S2 PFDS Stability Testing

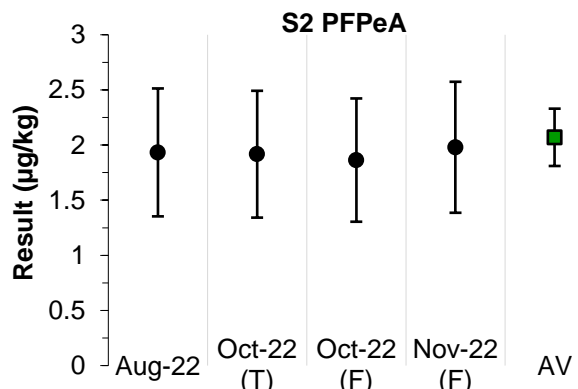


Figure 96 S2 PFPeA Stability Testing

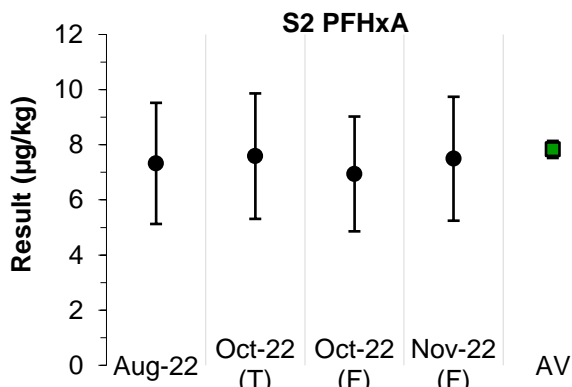


Figure 97 S2 PFHxA Stability Testing

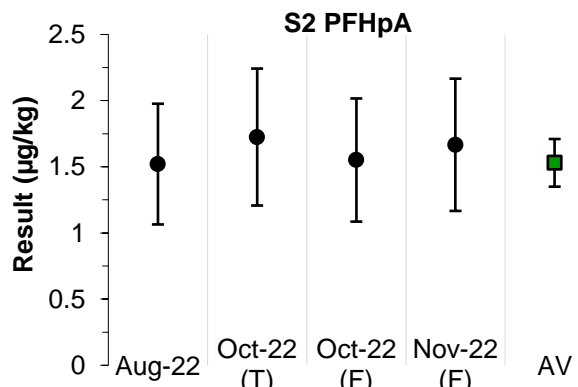


Figure 98 S2 PFHpA Stability Testing

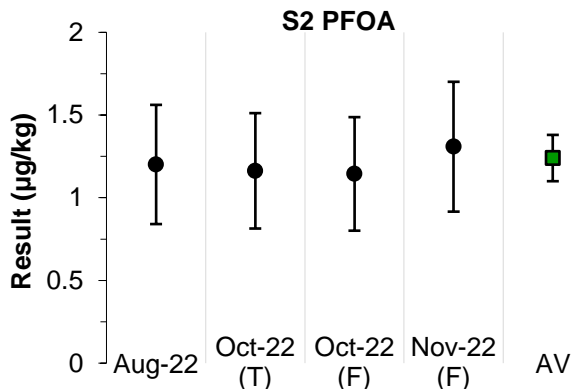


Figure 99 S2 PFOA Stability Testing

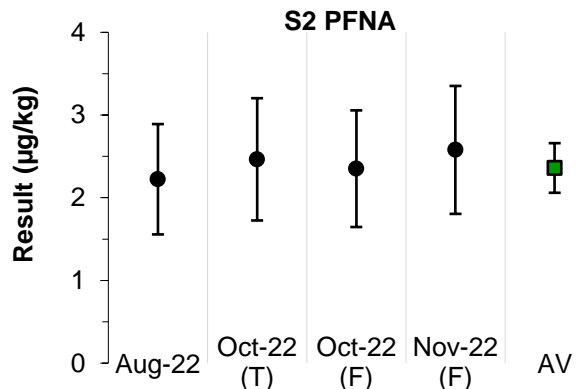


Figure 100 S2 PFNA Stability Testing

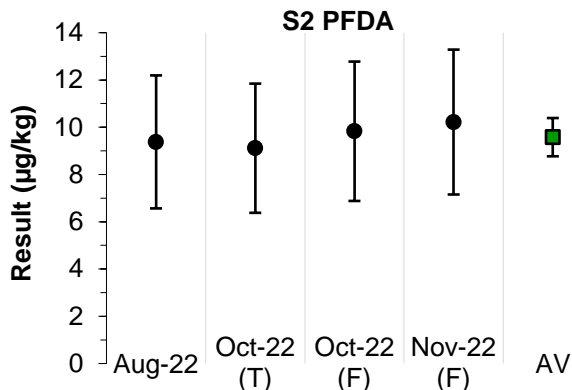


Figure 101 S2 PFDA Stability Testing

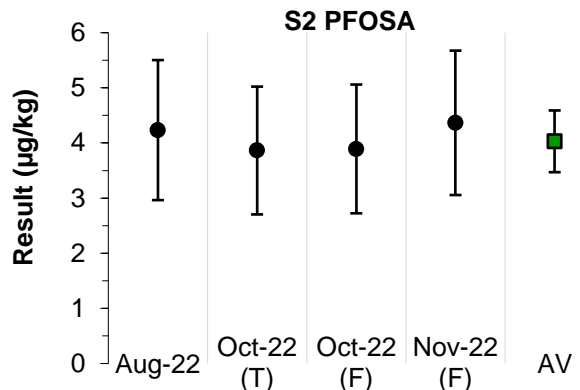


Figure 102 S2 PFOSA Stability Testing

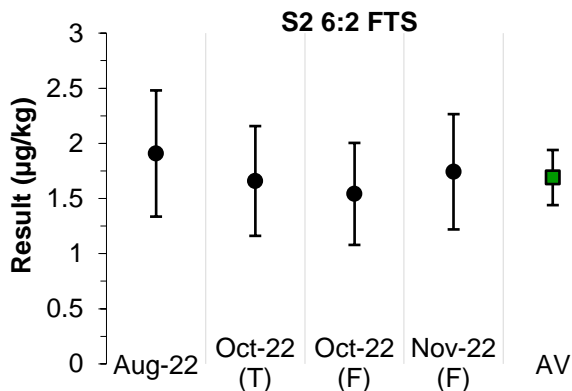


Figure 103 S2 6:2 FTS Stability Testing

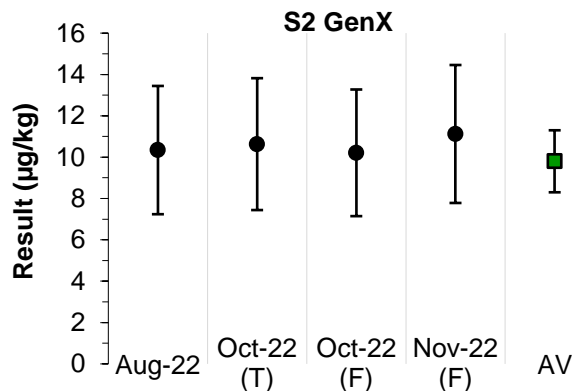


Figure 104 S2 GenX Stability Testing

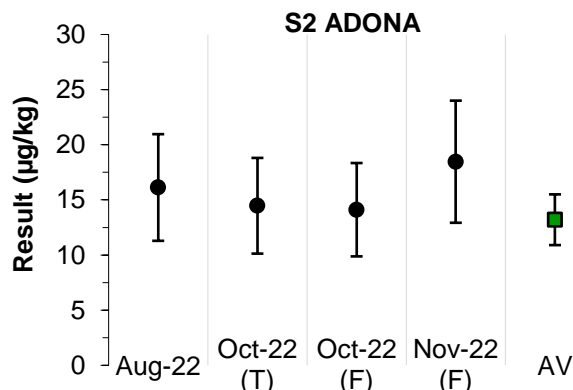


Figure 105 S2 ADONA Stability Testing

## A2.2 Comparison of Results and Container Numbers

Comparisons of z-scores obtained to the container number analysed by participants for all scored analytes are presented for information in Figures 106 to 144 (results have been included when the participant was sent one sample set only), with results excluded from statistical calculations as described in Section 4.2 being shaded.

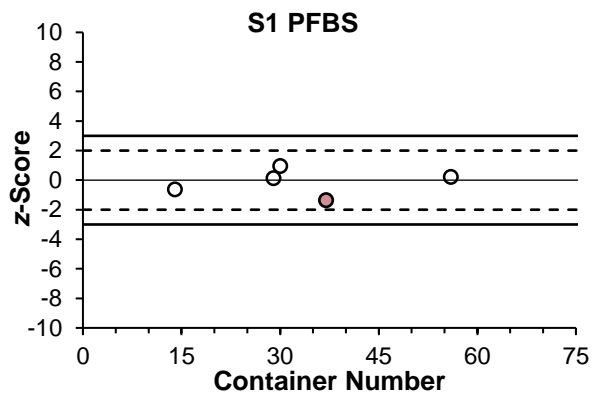


Figure 106 S1 PFBS z-Score vs Container

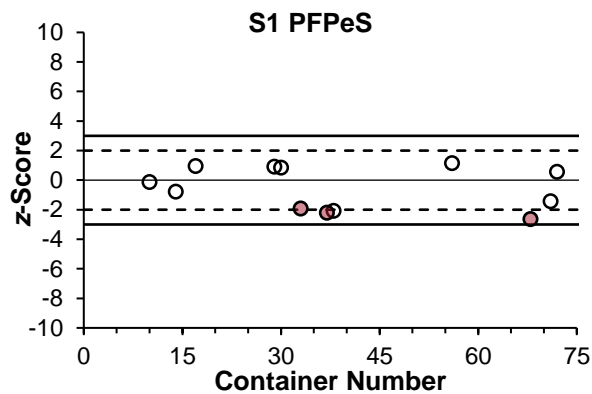


Figure 107 S1 PFPeS z-Score vs Container

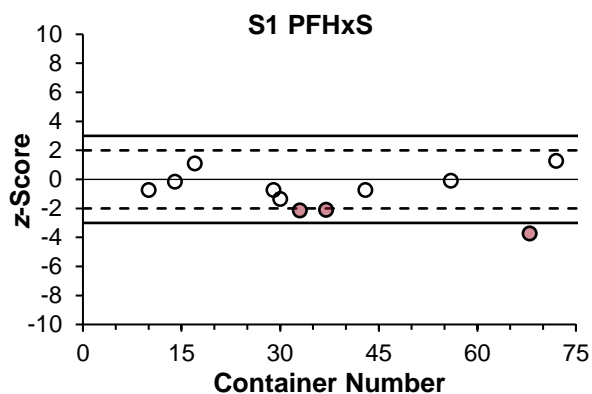


Figure 108 S1 PFHxS z-Score vs Container

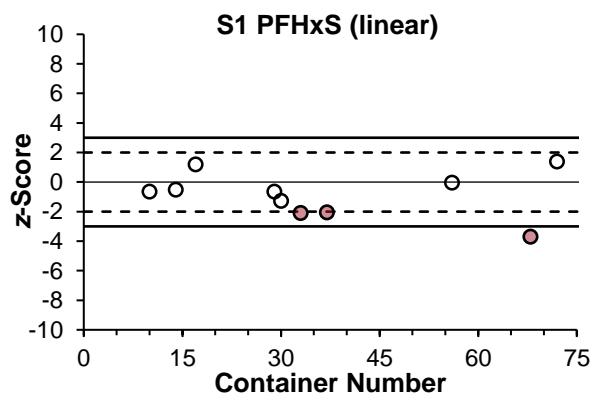


Figure 109 S1 PFHxS (linear) z-Score vs Container

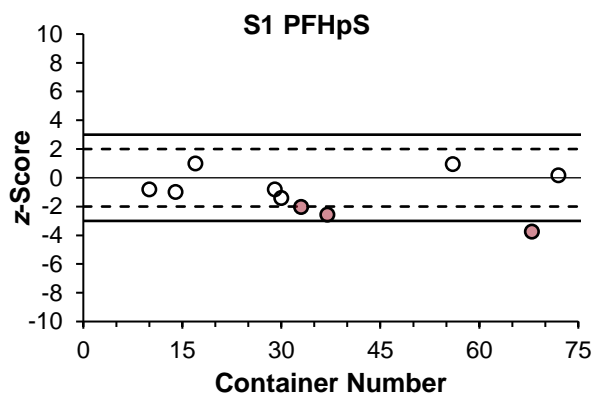


Figure 110 S1 PFHpS z-Score vs Container

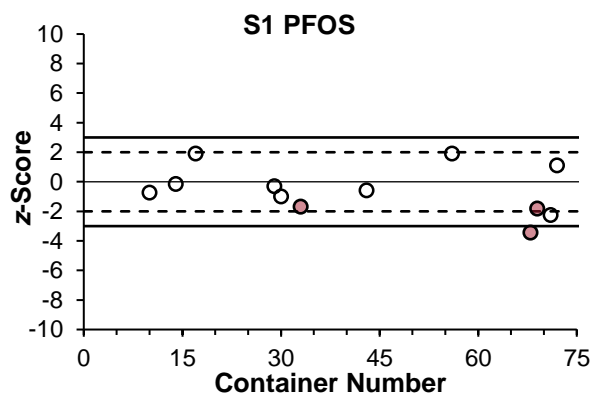


Figure 111 S1 PFOS z-Score vs Container

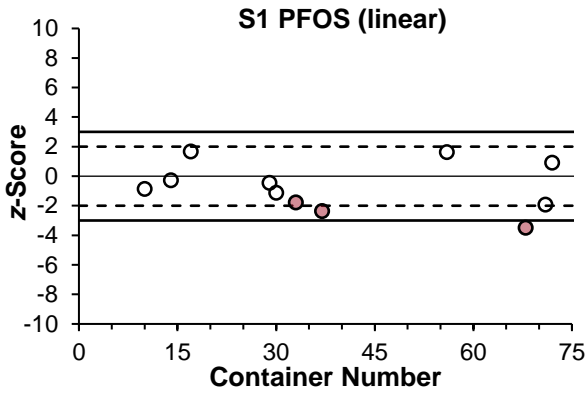


Figure 112 S1 PFOS (linear) z-Score vs Container

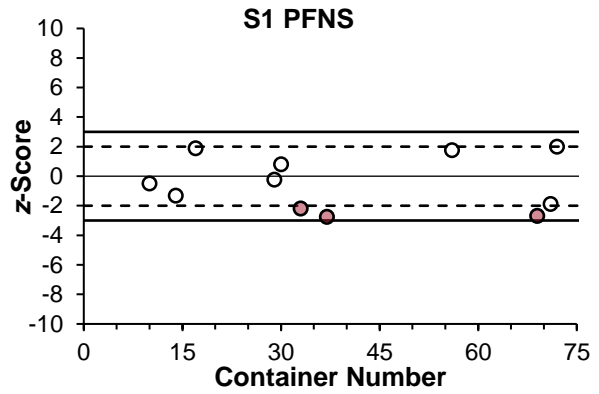


Figure 113 S1 PFNS z-Score vs Container

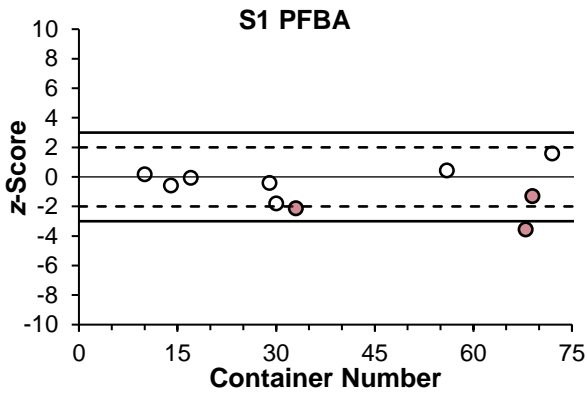


Figure 114 S1 PFBA z-Score vs Container

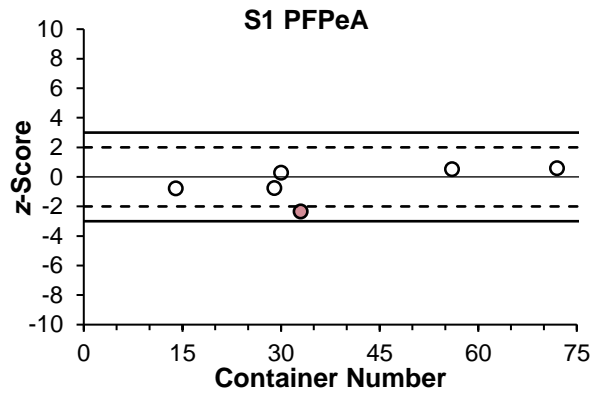


Figure 115 S1 PFPeA z-Score vs Container

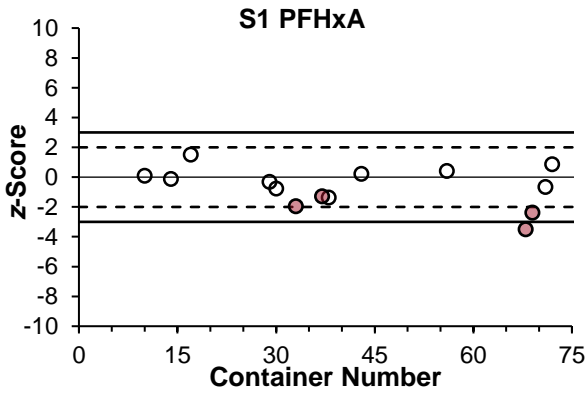


Figure 116 S1 PFHxA z-Score vs Container

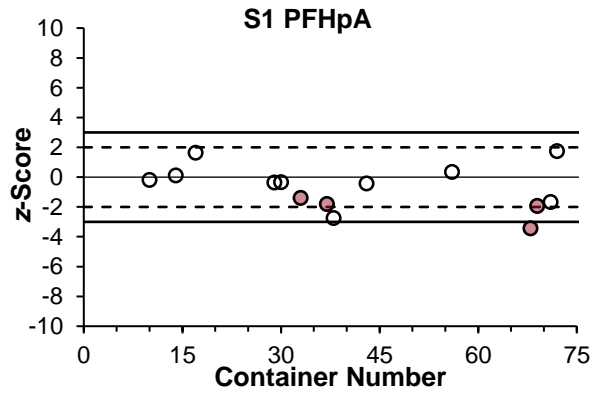


Figure 117 S1 PFHpA z-Score vs Container

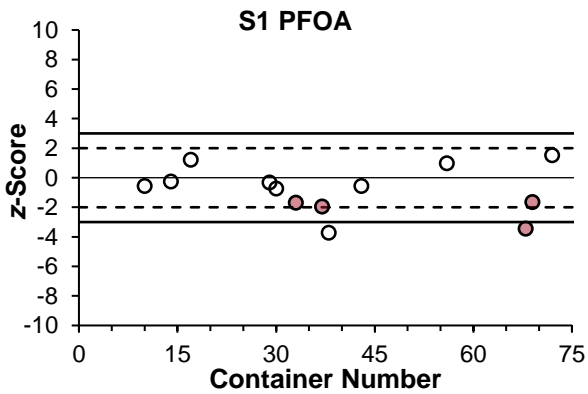


Figure 118 S1 PFOA z-Score vs Container

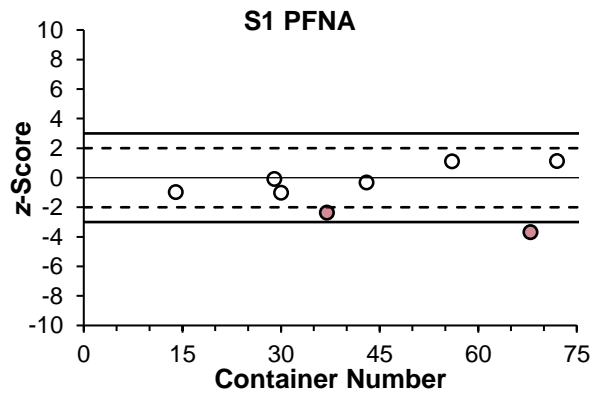


Figure 119 S1 PFNA z-Score vs Container

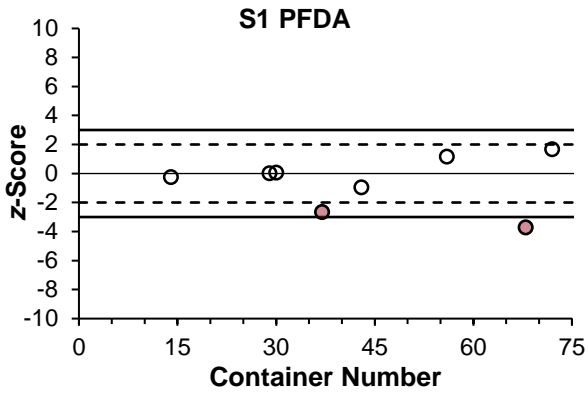


Figure 120 S1 PFDA z-Score vs Container

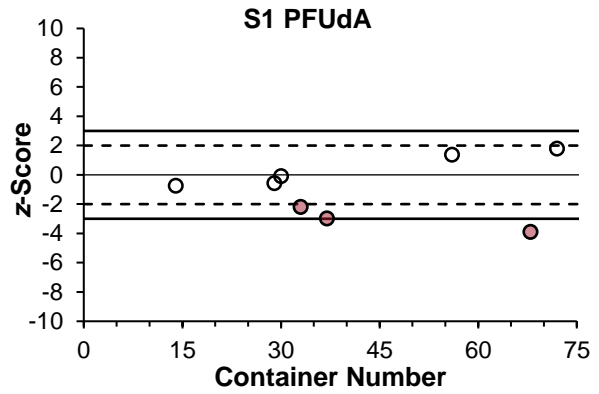


Figure 121 S1 PFUdA z-Score vs Container

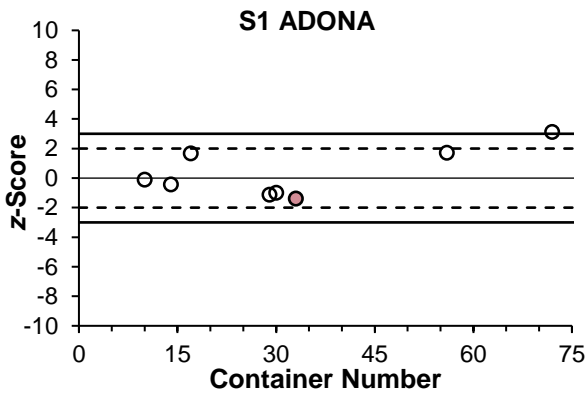


Figure 122 S1 ADONA z-Score vs Container

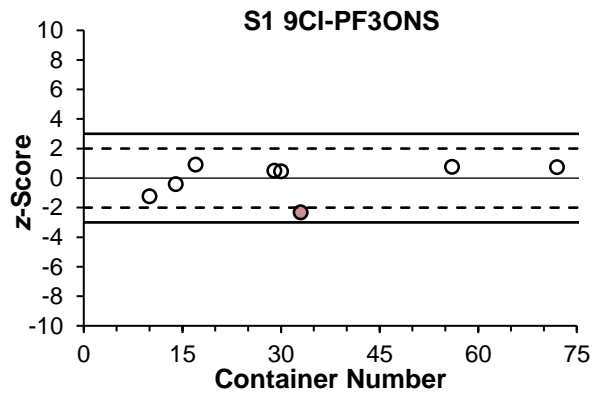


Figure 123 S1 9CI-PF3ONS z-Score vs Container

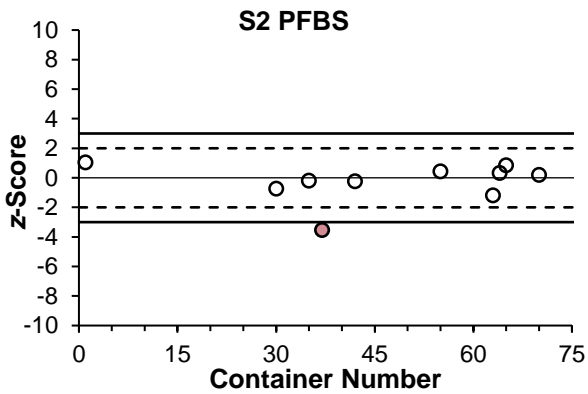


Figure 124 S2 PFBS z-Score vs Container

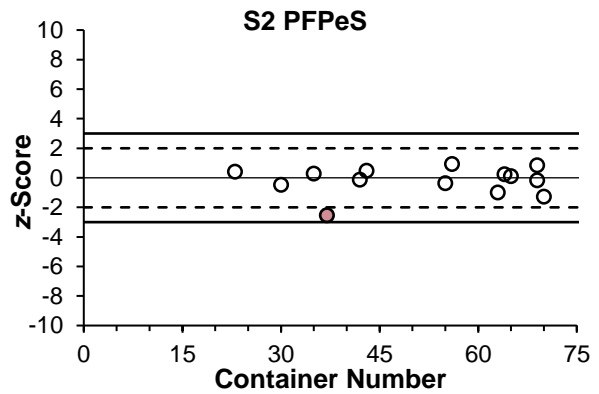


Figure 125 S2 PFPeS z-Score vs Container

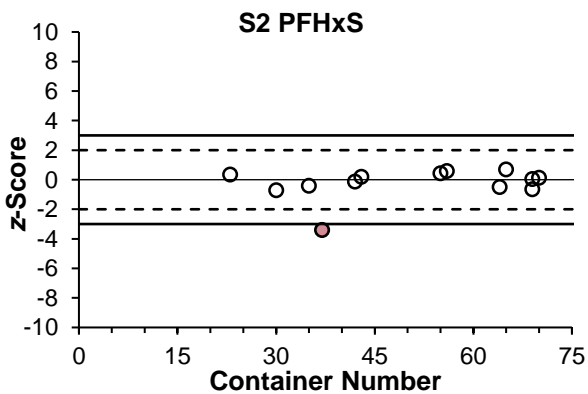


Figure 126 S2 PFHxS z-Score vs Container

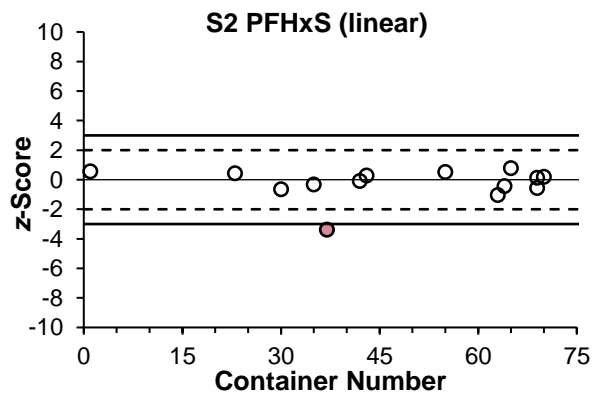


Figure 127 S2 PFHxS (linear) z-Score vs Container



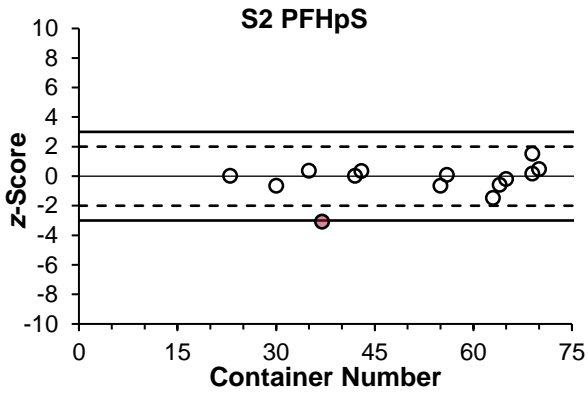


Figure 128 S2 PFHpS  $z$ -Score vs Container

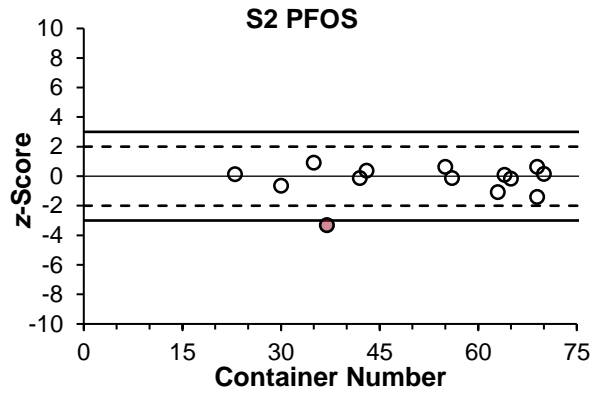


Figure 129 S2 PFOS  $z$ -Score vs Container

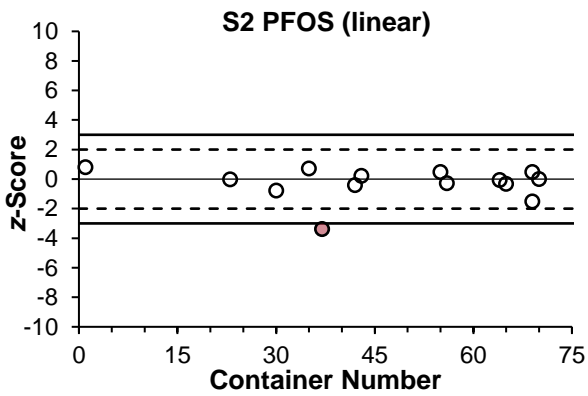


Figure 130 S2 PFOS (linear)  $z$ -Score vs Container

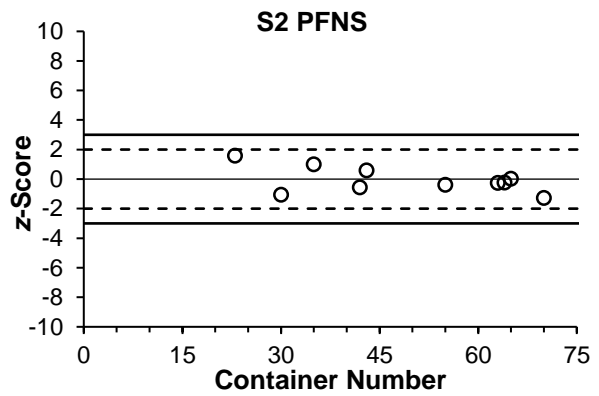


Figure 131 S2 PFNS  $z$ -Score vs Container

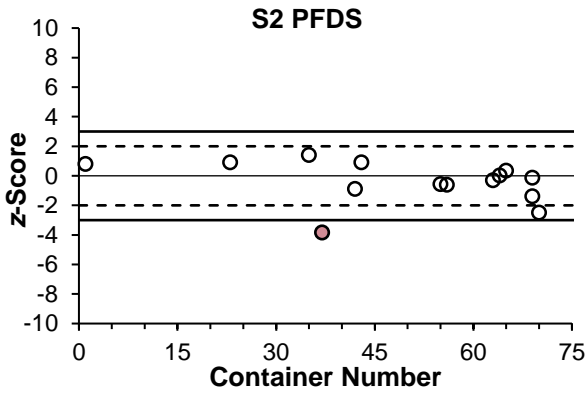


Figure 132 S2 PFDS  $z$ -Score vs Container

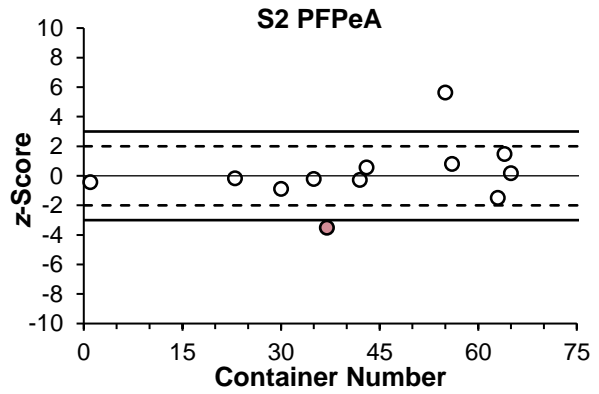


Figure 133 S2 PFPeA  $z$ -Score vs Container

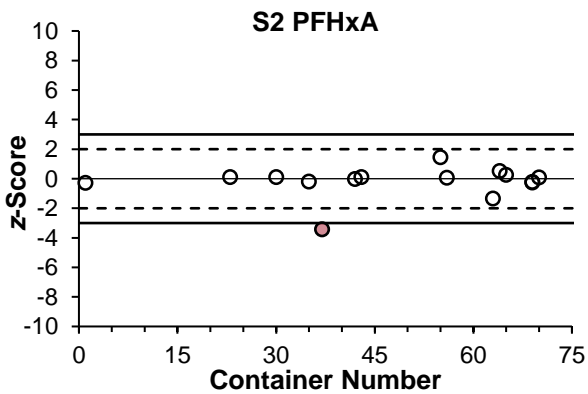


Figure 134 S2 PFHxA  $z$ -Score vs Container

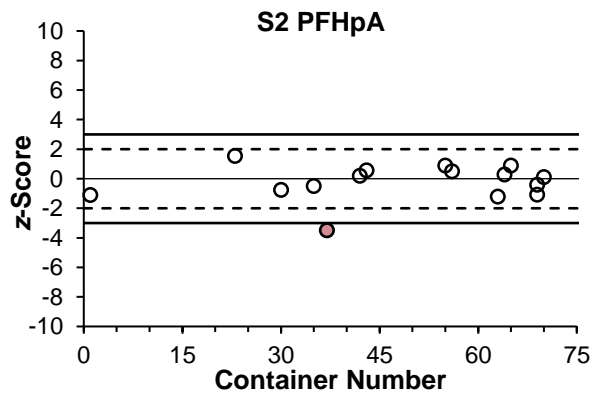


Figure 135 S2 PFHpA  $z$ -Score vs Container

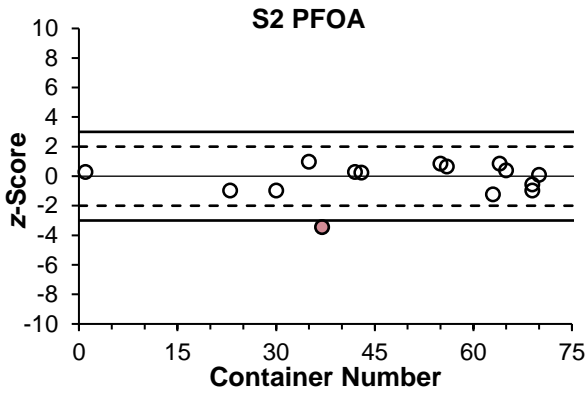


Figure 136 S2 PFOA z-Score vs Container

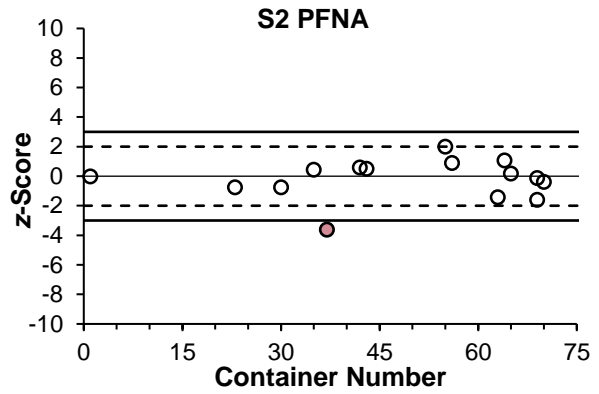


Figure 137 S2 PFNA z-Score vs Container

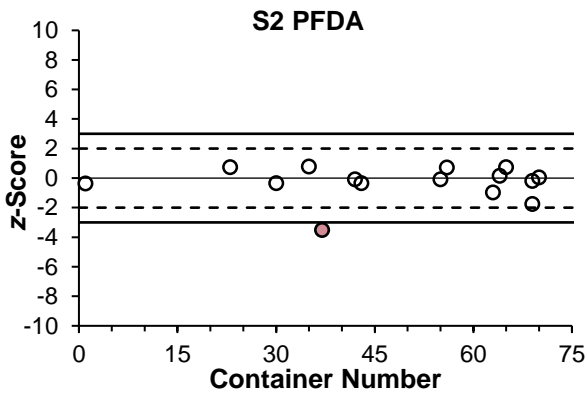


Figure 138 S2 PFDA z-Score vs Container

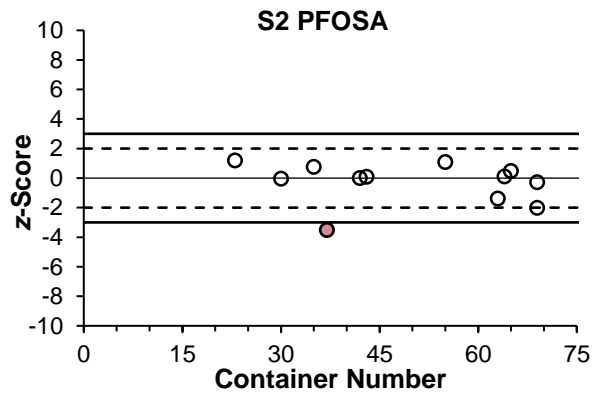


Figure 139 S2 PFOSA z-Score vs Container

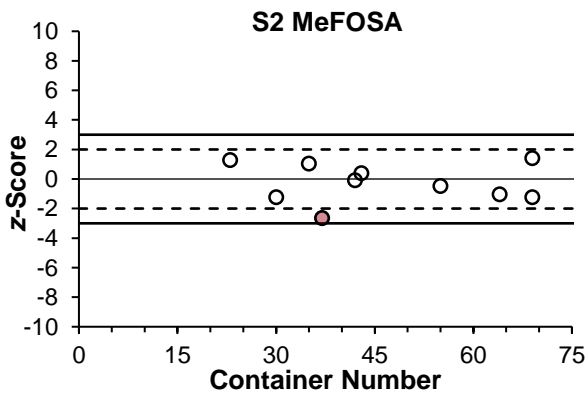


Figure 140 S2 MeFOSA z-Score vs Container

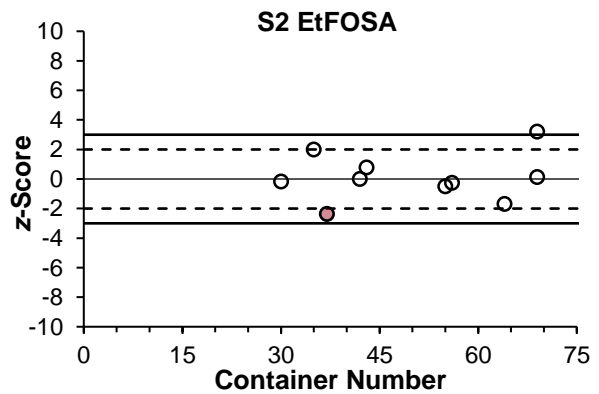


Figure 141 S2 EtFOSA z-Score vs Container

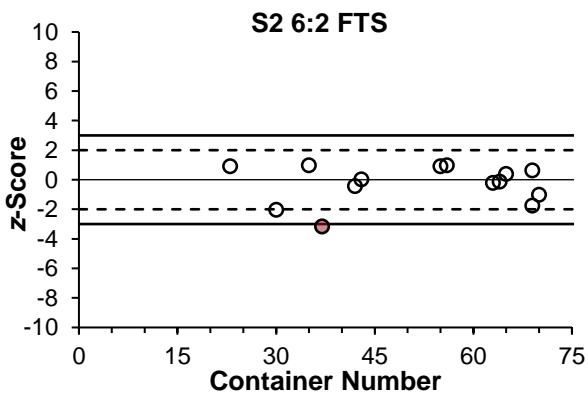


Figure 142 S2 6:2 FTS z-Score vs Container

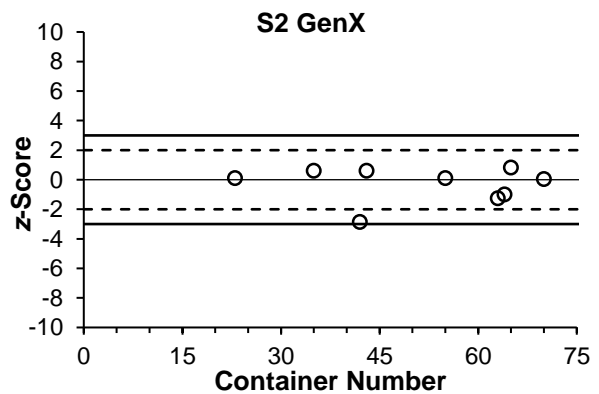


Figure 143 S2 GenX z-Score vs Container

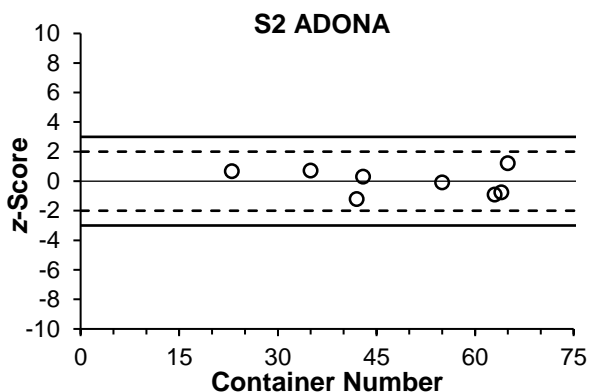


Figure 144 S2 ADONA z-Score vs Container

### A2.3 Comparison of Results and Days in Transit

Comparisons of participants' results to the number of days the samples spent in transit for all scored analytes are presented for information in Figures 145 to 183, with results excluded from statistical calculations as described in Section 4.2 being shaded.

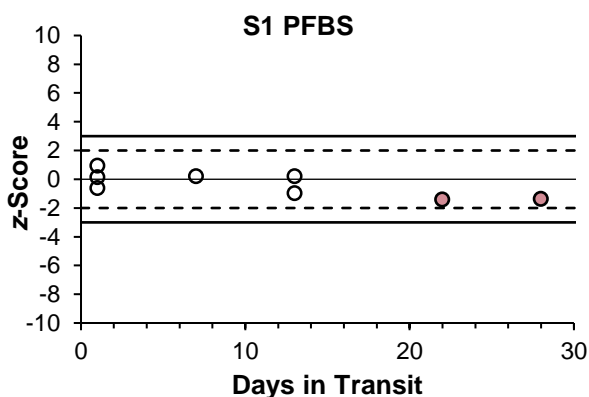


Figure 145 S1 PFBS z-Score vs Transit Days

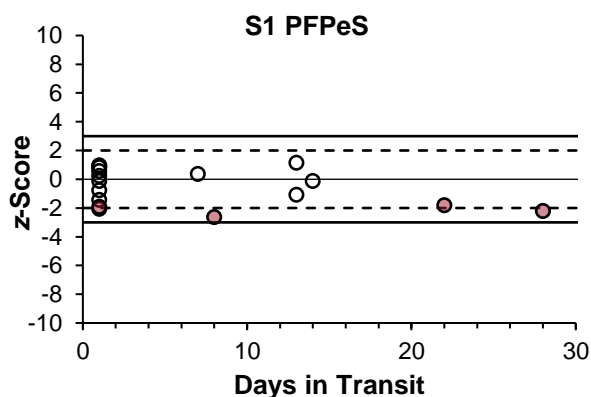


Figure 146 S1 PFPeS z-Score vs Transit Days

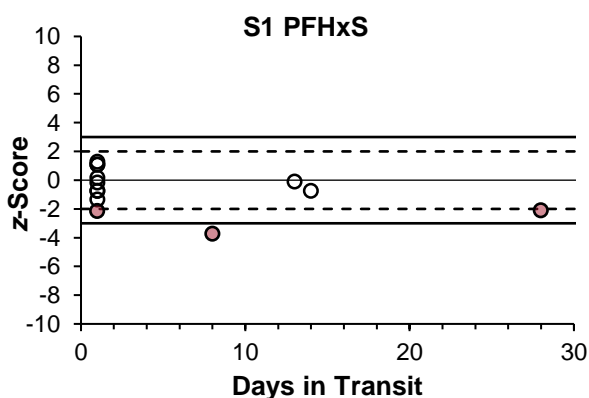


Figure 147 S1 PFHxS z-Score vs Transit Days

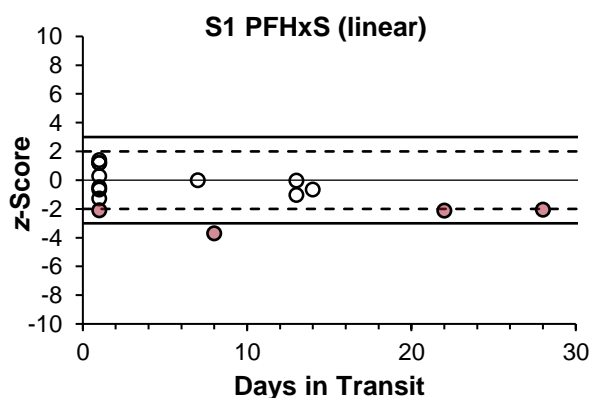


Figure 148 S1 PFHxS (linear) z-Score vs Transit Days

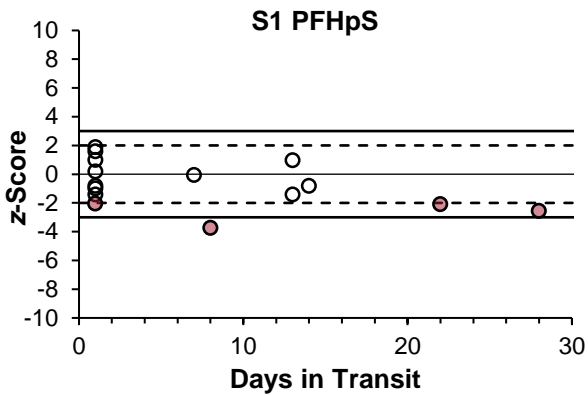


Figure 149 S1 PFHpS  $z$ -Score vs Transit Days

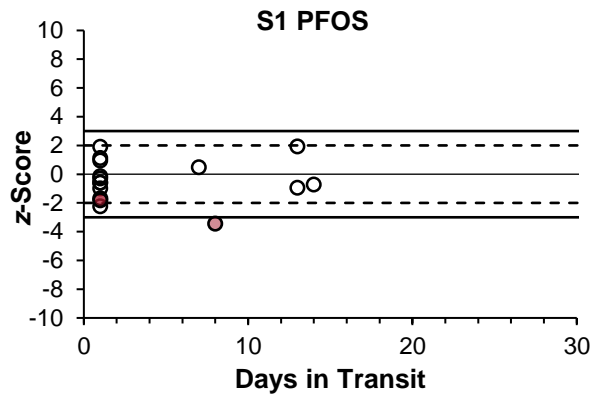


Figure 150 S1 PFOS  $z$ -Score vs Transit Days

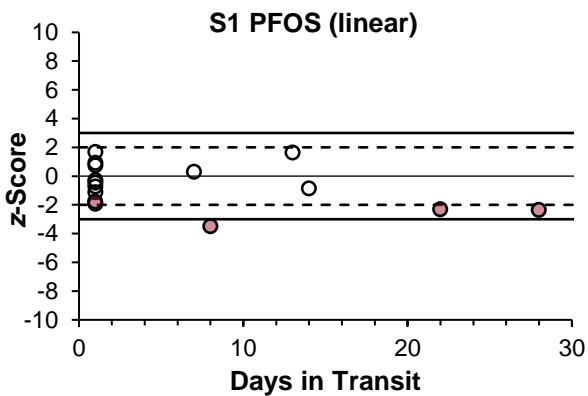


Figure 151 S1 PFOS (linear)  $z$ -Score vs Transit Days

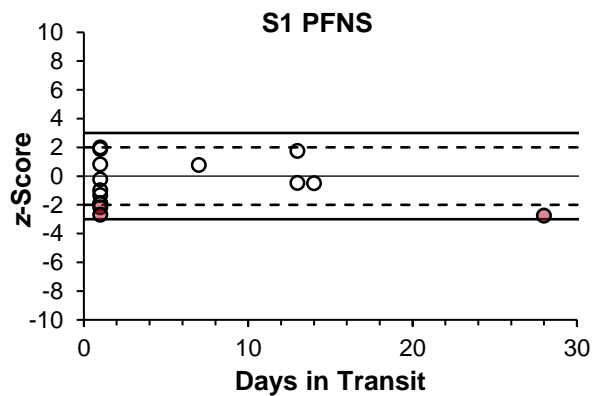


Figure 152 S1 PFNS  $z$ -Score vs Transit Days

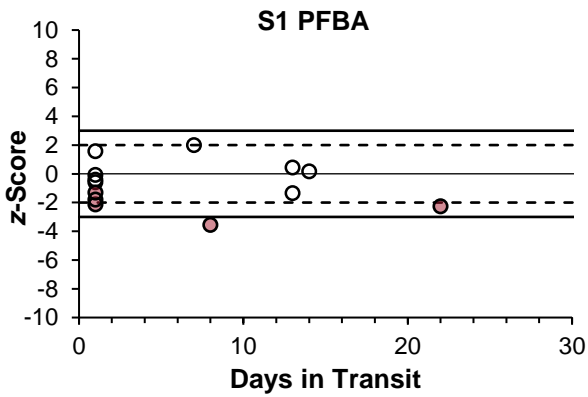


Figure 153 S1 PFBA  $z$ -Score vs Transit Days

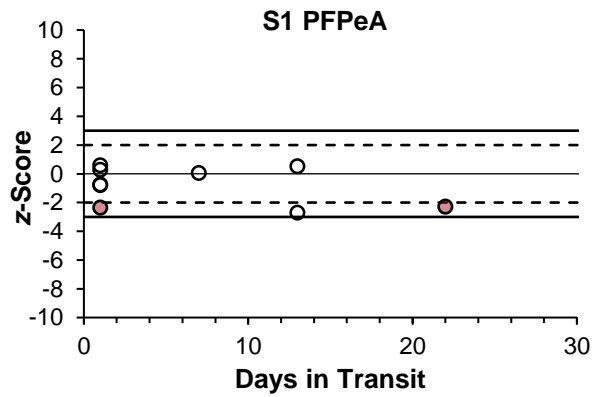


Figure 154 S1 PFPeA  $z$ -Score vs Transit Days

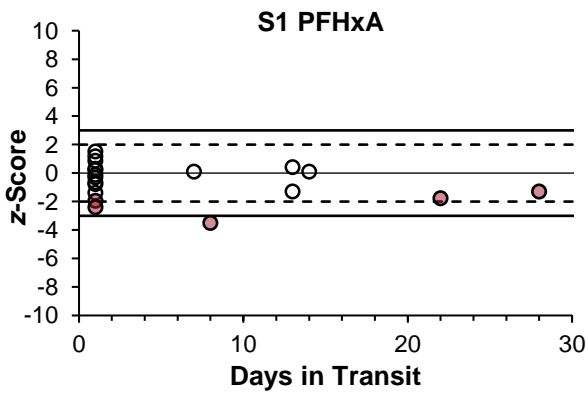


Figure 155 S1 PFHxA  $z$ -Score vs Transit Days

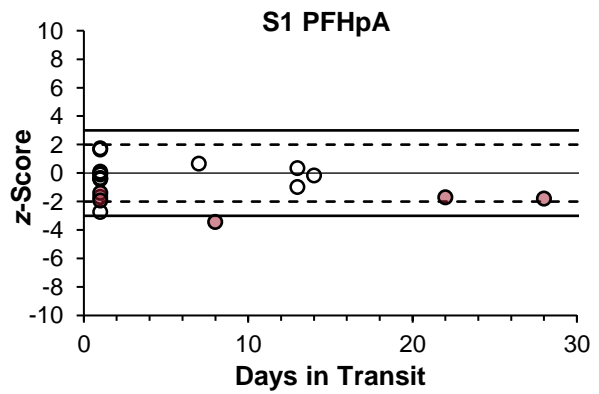


Figure 156 S1 PFHpA  $z$ -Score vs Transit Days

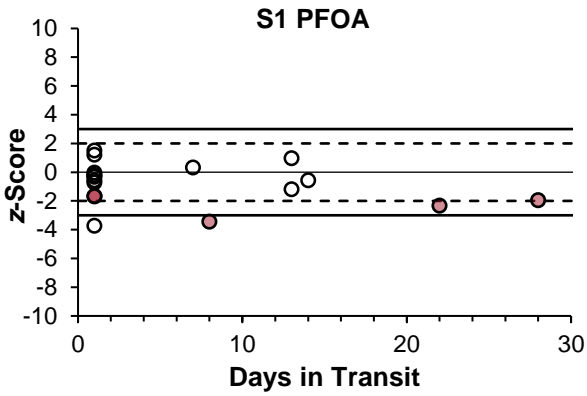


Figure 157 S1 PFOA z-Score vs Transit Days

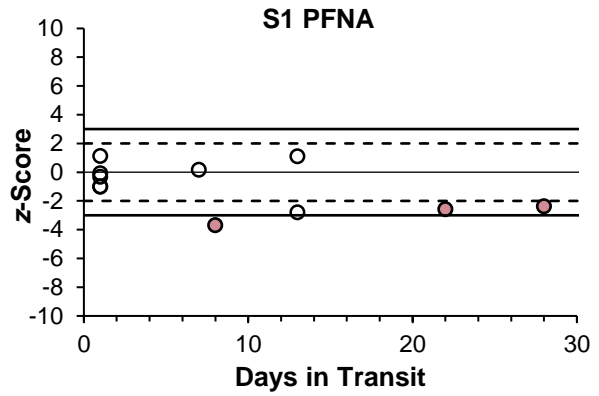


Figure 158 S1 PFNA z-Score vs Transit Days

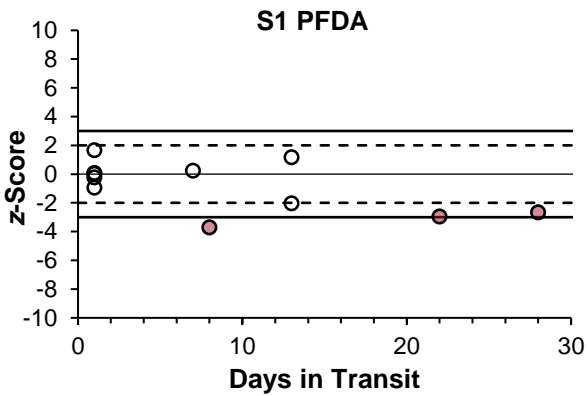


Figure 159 S1 PFDA z-Score vs Transit Days

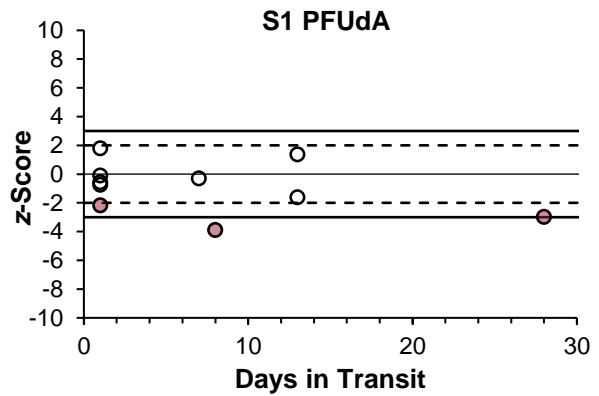


Figure 160 S1 PFUdA z-Score vs Transit Days

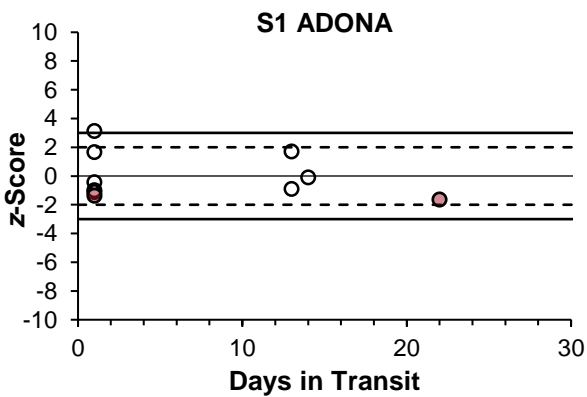


Figure 161 S1 ADONA z-Score vs Transit Days

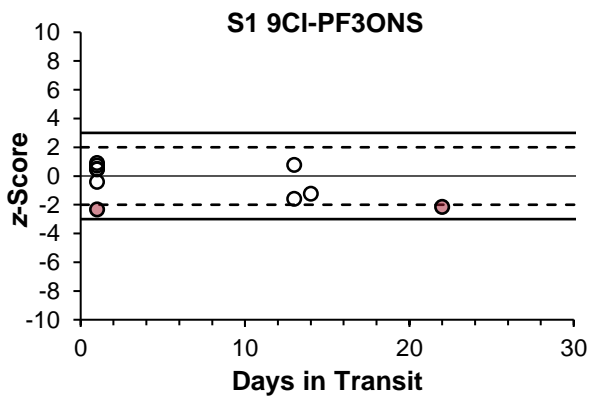


Figure 162 S1 9CI-PF3ONS z-Score vs Transit Days

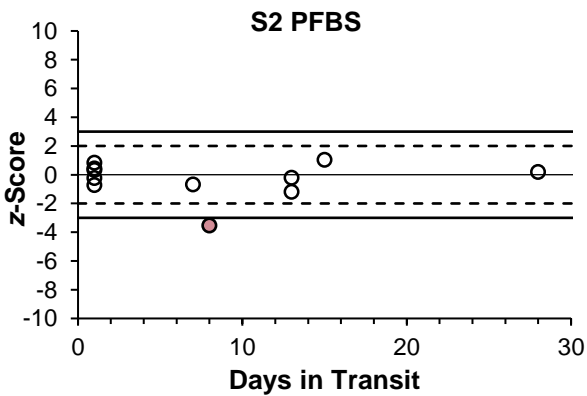


Figure 163 S2 PFBS z-Score vs Transit Days

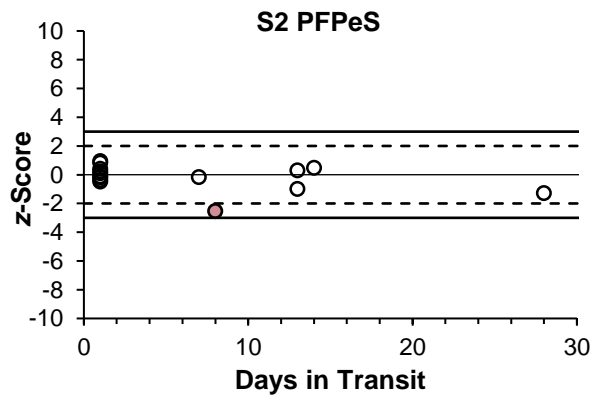


Figure 164 S2 PFPeS z-Score vs Transit Days

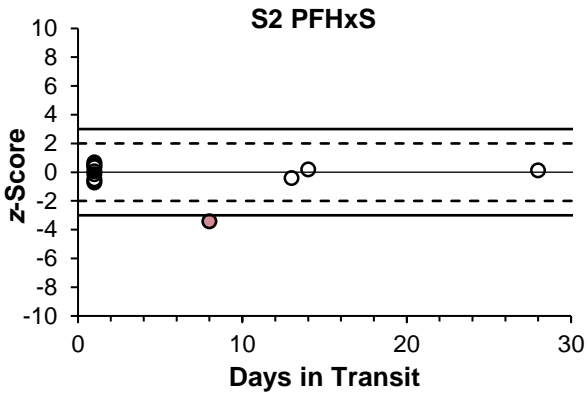


Figure 165 S2 PFHxS z-Score vs Transit Days

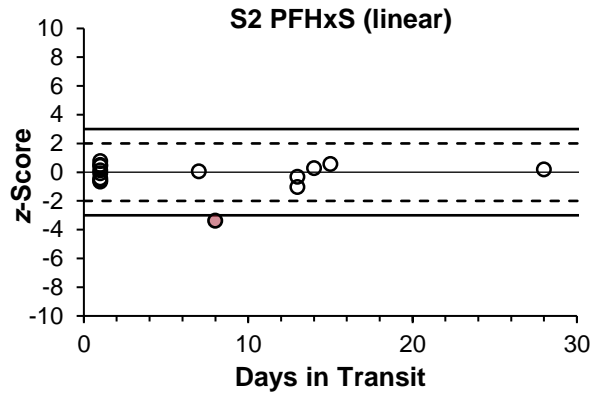


Figure 166 S2 PFHxS (linear) z-Score vs Transit Days

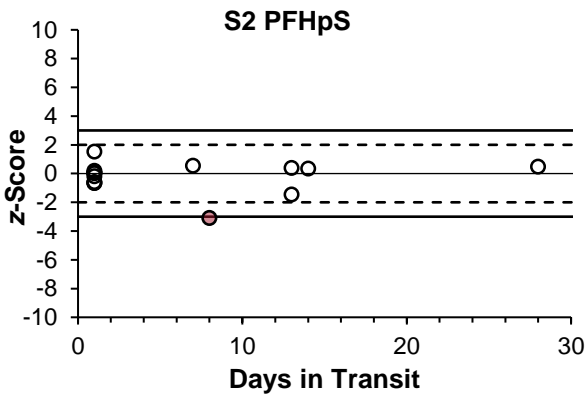


Figure 167 S2 PFHpS z-Score vs Transit Days

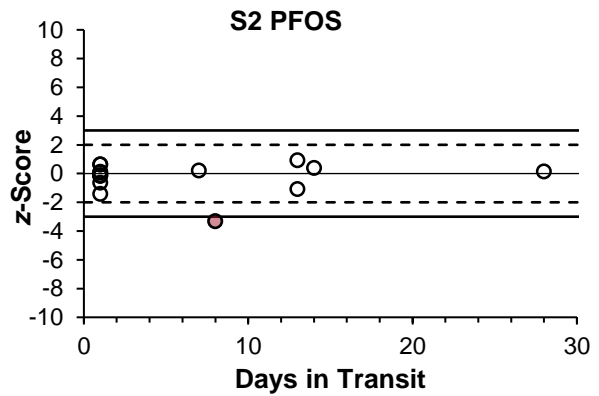


Figure 168 S2 PFOS z-Score vs Transit Days

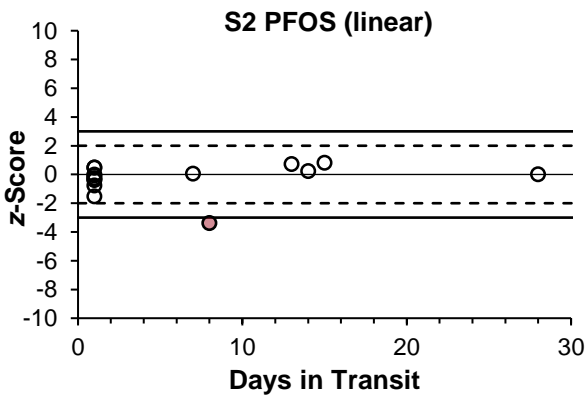


Figure 169 S2 PFOS (linear) z-Score vs Transit Days

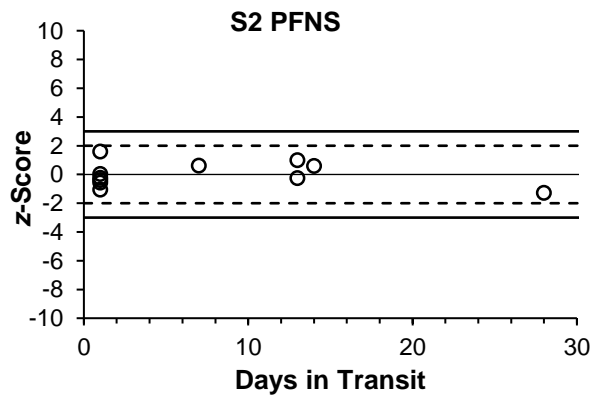


Figure 170 S2 PFNS z-Score vs Transit Days

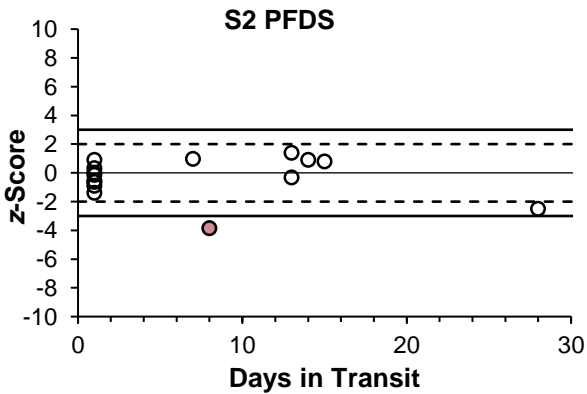


Figure 171 S2 PFDS z-Score vs Transit Days

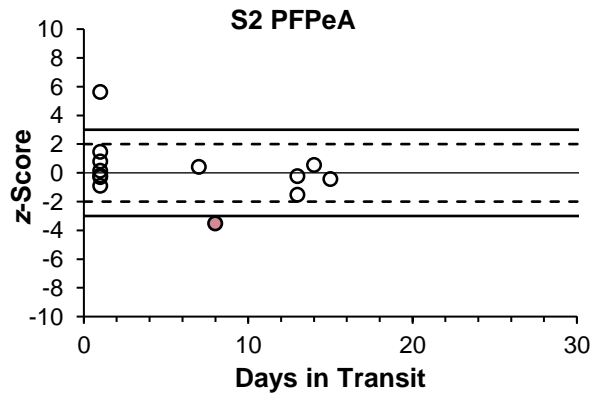


Figure 172 S2 PFPeA z-Score vs Transit Days

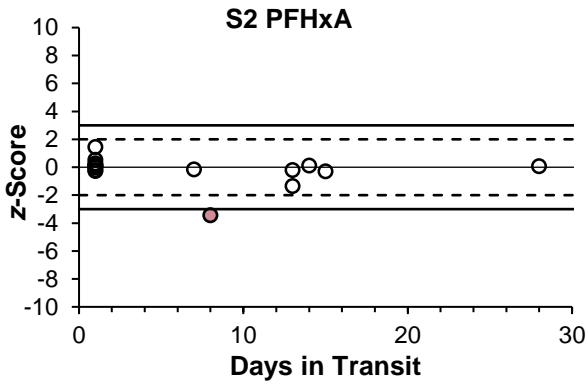


Figure 173 S2 PFHxA z-Score vs Transit Days

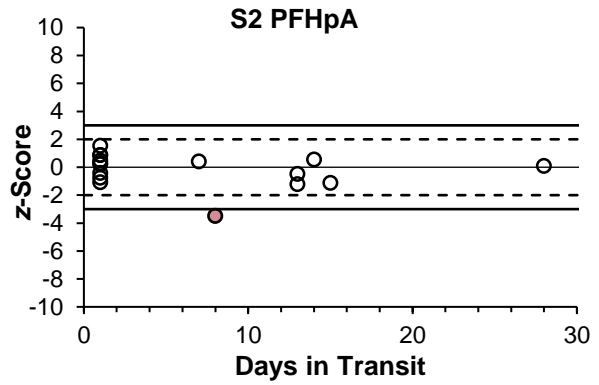


Figure 174 S2 PFHpA z-Score vs Transit Days

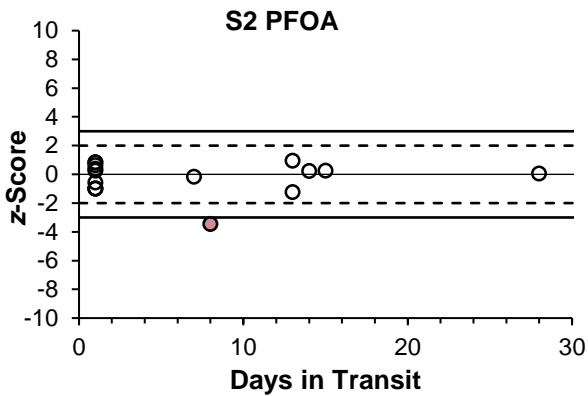


Figure 175 S2 PFOA z-Score vs Transit Days

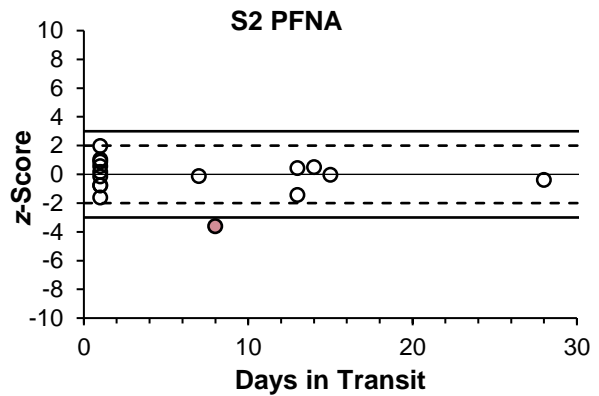


Figure 176 S2 PFNA z-Score vs Transit Days

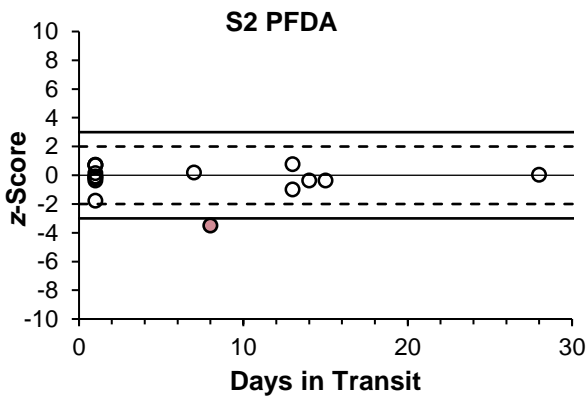


Figure 177 S2 PFDA z-Score vs Transit Days

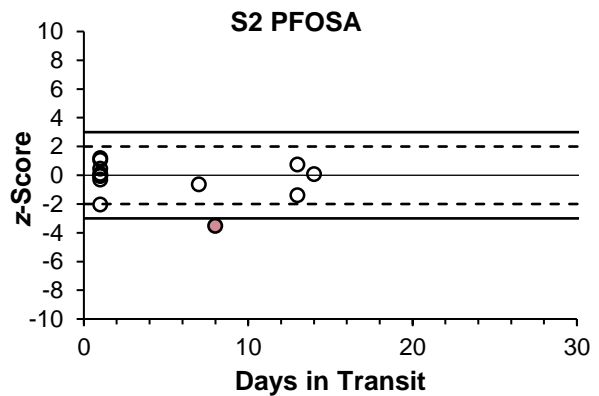


Figure 178 S2 PFOSA z-Score vs Transit Days

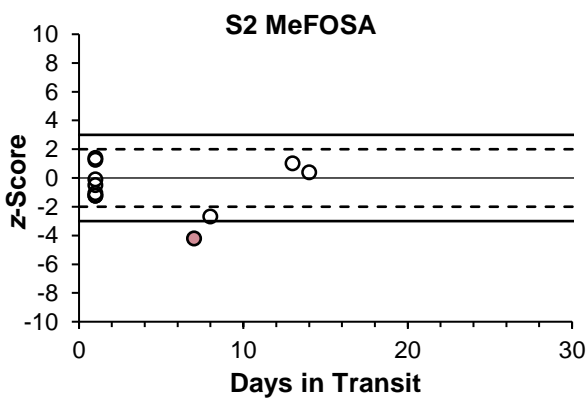


Figure 179 S2 MeFOSA z-Score vs Transit Days

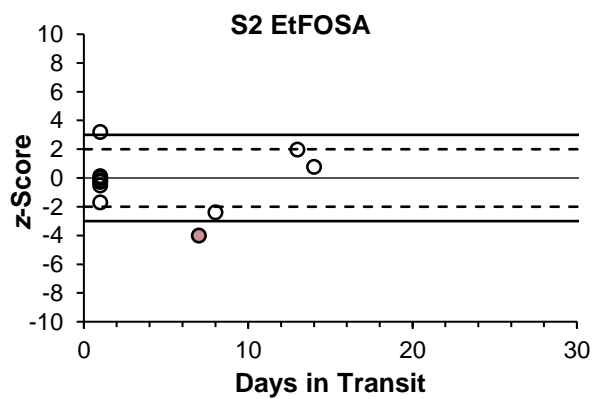


Figure 180 S2 EtFOSA z-Score vs Transit Days

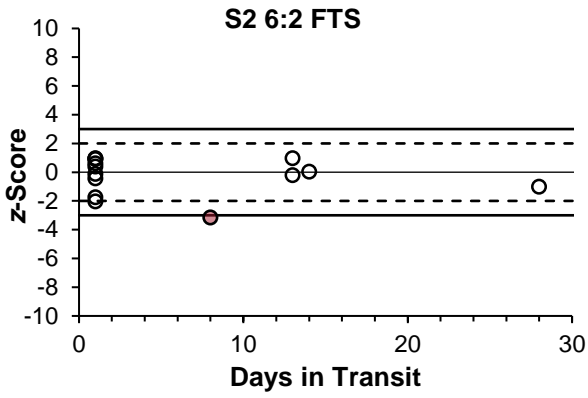


Figure 181 S2 6:2 FTS  $z$ -Score vs Transit Days

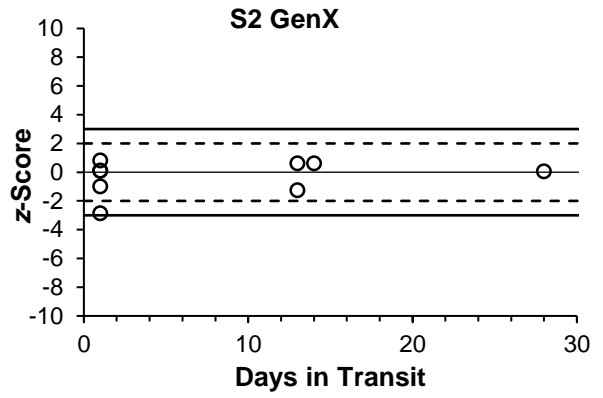


Figure 182 S2 GenX  $z$ -Score vs Transit Days

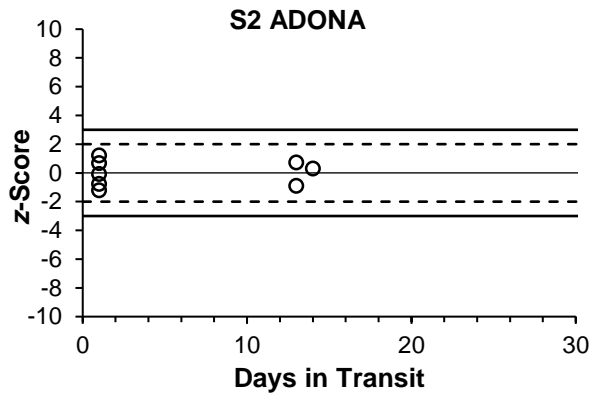


Figure 183 S2 ADONA  $z$ -Score vs Transit Days



## APPENDIX 3 ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, z-SCORE AND $E_n$ -SCORE CALCULATIONS

### A3.1 Robust Average and Associated Uncertainty

Robust averages were calculated using the procedure described in ISO 13528:2022.<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 77.

Table 77 Uncertainty Estimate for Robust Average of Sample S2 PFHpS

|                       |            |
|-----------------------|------------|
| Number of Results (p) | 14         |
| Robust Average        | 2.99 µg/kg |
| $S_{rob\ av}$         | 0.36 µg/kg |
| $u_{rob\ av}$         | 0.12 µg/kg |
| $k$                   | 2          |
| $U_{rob\ av}$         | 0.24 µg/kg |

Therefore, the robust average for Sample S2 PFHpS is  $2.99 \pm 0.24$  µg/kg.

### A3.2 z-Score and $E_n$ -Score Calculations

For each participant's result, a z-score and  $E_n$ -score are calculated according to Equations 2 and 3 respectively (Section 0).

A worked example is set out below in Table 78.

Table 78 z-Score and  $E_n$ -Score for Sample S1 PFBS Result Reported by Laboratory 2

| Participant Result (µg/kg) | Assigned Value (µg/kg) | Target Standard Deviation                                 | z-Score  | $E_n$ -Score   |
|----------------------------|------------------------|---|--|--|
| $0.244 \pm 0.011$          | $0.302 \pm 0.048$      | 20% as PCV, or:<br>$0.2 \times 0.302$<br>$= 0.0604$ µg/kg | $z\text{-Score} = \frac{0.244 - 0.302}{0.0604}$<br>$= -0.96$ | $E_n\text{-Score} = \frac{0.244 - 0.302}{\sqrt{0.011^2 + 0.048^2}}$<br>$= -1.18$ |

#### APPENDIX 4 PARTICIPANTS' TEST METHODS

Participants' responses to the methodology questionnaire are presented in Tables 79 to 129. Some responses may have been modified so that the participant cannot be identified.

Table 79 Participant Methodology – Sample S1 Prawn Extraction

| Lab. Code | S1 Sample Weight (g)  | Sample Pretreatment  | Extraction Technique        | Extraction Solvent                  | Extraction Temperature      | Extraction Time                        | Clean-Up                        |
|-----------|-----------------------|--|-----------------------------|-------------------------------------|-----------------------------|--|---------------------------------|
| 1         | 1                     | Homogenisation<br>Geno/Grinder<br>14min &<br>Centrifuge<br>10min | QuEChERS - modified<br>AOAC | Acetonitrile with 1%<br>Acetic Acid | Room                        | Sonicate 30<br>min at 30-35<br>degrees | envicarb                        |
| 2         |                       |  |                             |                                     |                             |  |                                 |
| 3         |                       |  |                             |                                     |                             |  |                                 |
| 4         | 1                     | Homogenisation   | Alkaline Digestion          | KOH-methanol                        | Room temp                   | 8 hrs                                  | Active carbon SPE               |
| 5         | NS                    |  |                             |                                     |                             |  |                                 |
| 6         |                       |  | Solid-Liquid Extraction     |                                     |                             |  |                                 |
| 7         |                       |  |                             |                                     |                             |  |                                 |
| 8         | 6.1                   | Homogenisation   | QuEChERS                    | Acetonitrile/Sodium<br>Hydroxide    | Room<br>temperature         | 180mins                                | Solid-Phase Extraction          |
| 9         | 1                     |  | Alkaline Digestion          | Acetonitrile                        | Room<br>temperature         | 2 x 20<br>minutes                      | GCB and WAX SPE                 |
| 10        | 2                     |  | Solid-Liquid Extraction     | Acidified<br>acetonitrile/water     | Room                        |  |                                 |
| 11        | 1                     | No   | Solid-Liquid Extraction     | Acetonitrile                        | Room<br>temperature         | 30 min                                 | SPE-WAX, ultracentrifugation    |
| 12        | 2.01 (as<br>received) | Homogenisation   | Solid-Liquid Extraction     | KOH-Methanol                        | Ambient Room<br>Temperature | 16 hours                               | Activated carbon/SPE/filtration |

| Lab. Code | S1 Sample Weight (g)        | Sample Pretreatment | Extraction Technique  | Extraction Solvent                  | Extraction Temperature | Extraction Time | Clean-Up  |
|-----------|-----------------------------|---------------------|---|-------------------------------------|------------------------|-----------------|---|
| 13        | 1                           | Homogenisation      | Digestion with 200mM NaOH in methanol, then extraction with acetonitrile. | Acetonitrile                        | Room Temperature       | 2 x 15min       | liquid-liquid extraction with n-hexane, then Bond Elut Carbon SPE |
| 15        | 1g                          | Homogenisation      | Alkaline Digestion  | Basified MeOH                       | Room                   | 60 mins         | Envicarb  |
| 16        | 1.1                         | Homogenisation      | QuEChERS  | Methanol + Ammonium-ACN and Acetone | 40 °C                  | 30 min          | Solid-Phase Extraction  |
| 17        | 1                           | Homogenisation      | Solid-Liquid Extraction   | Acetonitrile                        | Ambient                | 30 mins         | Solid-Phase Extraction  |
| 18        | 0.5 grams                   | Homogenisation      | Solid-Liquid Extraction   | MTBE                                | Room                   | 60 minutes      | C18 & Activated Carbon  |
| 19        | 2.076 and 2.106 (duplicate) | NA                  | Solid-Liquid Extraction Merris-Minimix shaker                             | 2% formic acid in acetonitrile      | Room temperature       | 8 min           | dSPE (C18, Envicarb, MgSO4)                                       |
| 20        | 0.7                         | Homogenisation      | Solid-Liquid Extraction   | 0.1 % NH3 in ACN                    | room temperature       | 2 h             | Solid-Phase Extraction two different SPE cartridges               |
| 21        |                             |                     | Alkaline Digestion  |                                     |                        |                 |   |

Table 80 Participant Methodology – Sample S1 Prawn Instrumental Technique and Analysis

| Lab. Code | Instrument        | Guard Column | Instrument Column          | Dilution Factor | Delay Column? | Blank Correction? | Standard Method?  |
|-----------|-------------------|--------------|----------------------------|-----------------|---------------|-------------------|---|
| 1         | Orbitrap          | C18 3mm      | Kinetex C18 100x3mm 2.6 um |                 | Yes           | Yes               | In house  |
| 2         |                   |              |                            |                 |               |                   | US FDA Foods Program Compendium of Analytical Laboratory Methods; method C-010.02 |
| 3         |                   |              |                            |                 |               |                   |   |
| 4         | LC-MSMS or LC-QQQ | No           | C18, 50 mm                 | No              | Yes           | No                | No  |
| 5         | NS                |              |                            |                 |               |                   |   |

| Lab. Code | Instrument        | Guard Column                            | Instrument Column   | Dilution Factor | Delay Column? | Blank Correction? | Standard Method?  |
|-----------|-------------------|---|---|-----------------|---------------|-------------------|-------------------|
| 6         | LC-MSMS or LC-QQQ | C18                                     | 2.1x100mn 1.9um   |                 | Yes           | No                | USEPA 537         |
| 7         |                   |   |   |                 |               |                   |                   |
| 8         | LC-MSMS or LC-QQQ | None                                    | InfinityLab Poroshell HPH-C18 column, 2.1x50mm, 2.7micron | 0.16393         | No            | No                |                   |
| 9         | LC-MSMS or LC-QQQ | Nil                                     | Shimadzu Shim-pack XR-ODS III (1.6 µm, 50 mm x 2.0 mm)    | No              | Yes           | No                | No                |
| 10        | LC-MSMS or LC-QQQ |   |   |                 | Yes           |                   |                   |
| 11        | LC-MSMS or LC-QQQ | Gemini NX-C18; 4mm x 2.0 mm ID          | NX-C18; 15cm x 2mm x 3um                                  | No              | Yes           | Yes               | No                |
| 12        | LC-MSMS or LC-QQQ | Phenomenex Evo C18 (2µm, 2 mm x 2.1 mm) | BEH C18 (1.7µm, 50 mm x 2.1 mm)                           | No              | Yes           | No                | No                |
| 13        | LC-MSMS or LC-QQQ | Phenomenex Evo C18 (2 mm x 2.1 mm)      | Phenomenex Evo C18 (100 mm x 2.1 mm x 2.6 um)             | No              | Yes           | No                | Isotopic Dilution |
| 15        | LC-MSMS or LC-QQQ | Pre-column Filter 0.2µm                 | C18 50mm x 2.1mm x 1.8µm                                  | 50              | Yes           | No                | No. In-house      |
| 16        | LC-MSMS or LC-QQQ | UltraShield UHPLC 0.2 µm Restek         | Raptor C18 1.8 µm 50 x 2.1 mm Restek                      |                 | yes           | no                |                   |
| 17        | Orbitrap          | C18                                     | C18   |                 | Yes           |                   |                   |
| 18        | LC-MSMS or LC-QQQ | nil                                     | C18 1.6µm, 2.0mm x 50mm                                   | No              | Yes           | No                |                   |
| 19        | LC-MSMS or LC-QQQ | NA                                      | Zorbax XDB-C18, 100 mm x 2.1 mm, 1.8µm                    | NA              | Yes           | No                | No                |
| 20        | LC-MSMS or LC-QQQ | C18 Column, 2.1 x 5 mm, 3.5 µm          | C18 Column, 130Å, 3 x 50 mm, 3.5 µm                       |                 | Yes           | No                |                   |
| 21        | LC-MSMS or LC-QQQ | C18                                     | 2.1x100mn 1.9um   |                 | Yes           | No                | USEPA 537         |

Table 81 Participant Methodology – Sample S1 Prawn Labelled Standards

| Lab. Code | Labelled Standard Source | Recovery Correction? | Labelled Standards Additional Information              |
|-----------|--------------------------|----------------------|--|
| 1         | Wellington               | No                   |  |
| 2         | Wellington               | Yes                  | d5NN EtFOSAA added before instrument analysis          |
| 3         |                          |                      |  |
| 4         | Wellington               | Yes                  |  |
| 5         | NS                       |                      |  |
| 6         | Wellington               | no                   |  |
| 7         |                          |                      |  |
| 8         | Wellington               | Yes                  |  |
| 9         | Wellington               | Yes                  |  |
| 10        |                          |                      |  |
| 11        | Wellington               | Yes                  |  |
| 12        | Wellington               | Yes                  |  |
| 13        | Wellington               | Yes                  |  |
| 15        | Wellington               | Yes                  |  |
| 16        |                          | Yes                  |  |
| 17        | Wellington               | Yes                  | Results corrected by ISTD added before instrumentation |
| 18        | Wellington Laboratories  | No                   |  |
| 19        | Wellington Laboratory    | Yes                  |  |
| 20        | Wellington               | No                   |  |
| 21        | Wellington               | no                   |  |

Table 82 Labelled Standards for S1 PFBS

| Lab. Code | Before Extraction   | Before Instrument Analysis |
|-----------|---|----------------------------|
| 1         | Sodium perfluoro-1-[2,3,4 13C3] butanesulfonate<br>M3PFBS |                            |
| 2         | M3 PFBS   |                            |
| 3         |   |                            |
| 4         | 13C3 PFBS   |                            |
| 5         | NS  |                            |
| 6         |   | M3PFBS                     |
| 7         |   |                            |
| 8         | 13C3-PFBS   |                            |
| 9         | 13C3-PFBS   | 13C8-PFOS                  |
| 10        |   |                            |
| 11        | 18O2-PFHxS  | 18O2-PFOS                  |
| 12        | 13C3-PFBS   | 18O2-PFHxS                 |
| 13        | 13C3-PFBS   | 13C3-PFHxS                 |
| 15        | 13C3-PFBS   | N/A                        |
| 16        | 13C3-PFBS   | 13C4-PFOA                  |
| 17        | PFOS-C8   | PFBS-13C3                  |
| 18        |   | 13C3-PFBS                  |
| 19        | M3PFBS  | NA                         |
| 20        | 13C3-PFBS   | 13C2-PFHxA                 |
| 21        |   | M3PFBS                     |

Table 83 Labelled Standards for S1 PFPeS

| Lab. Code | Before Extraction | Before Instrument Analysis |
|-----------|-------------------|----------------------------|
| 1         |                   |                            |
| 2         | M PFHxS           |                            |
| 3         |                   |                            |
| 4         | 13C3 PFBS         |                            |
| 5         | NS                |                            |
| 6         |                   |                            |
| 7         |                   |                            |
| 8         | 13C3-PFBS         |                            |
| 9         | NT                |                            |
| 10        |                   |                            |
| 11        | 18O2-PFHxS        | 18O2-PFOS                  |
| 12        | 13C3-PFHxS        | 18O2-PFHxS                 |
| 13        | 18O2-PFHxS        | 13C3-PFHxS                 |
| 15        | 18O2-PFHxS        | N/A                        |
| 16        | 13C3-PFBS         | 13C4-PFOA                  |
| 17        | PFOS-C8           | PFOS-C4                    |
| 18        |                   | 16O2-PFHxS                 |
| 19        | M5PFHxA           | NA                         |
| 20        | linear 13C3-PFHxS | 13C2-PFHxA                 |
| 21        |                   |                            |

Table 84 Labelled Standards for S1 PFHxS

| Lab. Code | Before Extraction  | Before Instrument Analysis |
|-----------|--|----------------------------|
| 1         | Sodium perfluoro-1-[1,2,3 13C3] hexanesulfonate<br>M3PFHxS |                            |
| 2         |  |                            |
| 3         | NT   |                            |
| 4         | 13C3 PFHxS   | PFHxS18O2                  |
| 5         | NS   |                            |
| 6         |  |                            |
| 7         |  |                            |
| 8         | 18O2-PFHxS   |                            |
| 9         | 18O2-PFHxS   | 13C8-PFOS                  |
| 10        |  |                            |
| 11        | NT   |                            |
| 12        | 13C3-PFHxS   | 18O2-PFHxS                 |
| 13        | 18O2-PFHxS   | 13C3-PFHxS                 |
| 15        | 18O2-PFHxS   | N/A                        |
| 16        | 18O2-PFHxS   | 13C4-PFOA                  |
| 17        | PFOS-C8  | PFHxS-18O2                 |
| 18        |  | 16O2-PFHxS                 |
| 19        | M3PFHxS  | NA                         |
| 20        | linear 13C3-PFHxS  |                            |
| 21        |  |                            |

Table 85 Labelled Standards for S1 PFHxS  
(linear)

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

Table 86 Labelled Standards for S1 PFHpS

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

Table 87 Labelled Standards for S1 PFOS

| Lab. Code | Before Extraction                                | Before Instrument Analysis |
|-----------|--|----------------------------|
| 1         | Sodium perfluoro-1-[13C8] octanesulfonate M8PFOS |                            |
| 2         | 13C PFOS   |                            |
| 3         | NT   |                            |
| 4         | 13C8 PFOS  | 13C4 PFOS                  |
| 5         | NS   |                            |
| 6         |  |                            |
| 7         |  |                            |
| 8         | 13C8-PFOS  |                            |
| 9         | 13C4-PFOS  | 13C8-PFOS                  |
| 10        |  |                            |
| 11        | 13C4-PFOS  | 18O2-PFOS                  |
| 12        | 13C8-PFOS  | 13C4-PFOS                  |
| 13        | 13C4-PFOS  | 13C8-PFOS                  |
| 15        | 13C4-PFOS  | N/A                        |
| 16        | 13C4-PFOS  | 13C4-PFOA                  |
| 17        | PFOS-C8  | PFOS-C4                    |
| 18        | 13C4-PFOS  | 13C8-PFOS                  |
| 19        | M8PFOS   | NA                         |
| 20        | linear 13C8-PFOS                                 | 13C5-PFNA                  |
| 21        |  |                            |

Table 88 Labelled Standards for S1 PFOS  
(linear)

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

Table 89 Labelled Standards for S1 PFNS

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

Table 90 Labelled Standards for S1 PFBA

| Lab. Code | Before Extraction                     | Before Instrument Analysis |
|-----------|---------------------------------------|----------------------------|
| 1         | Perfluoro-n-[13C4]butanoic acid MPFBA |                            |
| 2         | M3 PFBA                               |                            |
| 3         |                                       |                            |
| 4         | 13C4 PFBA                             | 13C3 PFBA                  |
| 5         | NS                                    |                            |
| 6         | M3PFBA                                | MPFBA                      |
| 7         |                                       |                            |
| 8         | 13C4-PFBA                             |                            |
| 9         | NT                                    |                            |
| 10        |                                       |                            |
| 11        | 13C4-PFBA                             | 13C8-PFOA                  |
| 12        | 13C4-PFBA                             | 13C3-PFBA                  |
| 13        | 13C4-PFBA                             | 13C3-PFBA                  |
| 15        | 13C4-PFBA                             | N/A                        |
| 16        | 13C4-PFBA                             | 13C4-PFOA                  |
| 17        | PFOS-C8                               | PFBA-13C4                  |
| 18        |                                       | 13C4-PFBA                  |
| 19        | M4PFBA                                | NA                         |
| 20        | NT                                    |                            |
| 21        | M3PFBA                                | MPFBA                      |



Table 91 Labelled Standards for S1 PFPeA

| Lab. Code | Before Extraction                        | Before Instrument Analysis |
|-----------|--|----------------------------|
| 1         | Perfluoro-n-[13C5]pentanoic acid M5PFPeA |                            |
| 2         | M3 PFPeA                                 |                            |
| 3         |  |                            |
| 4         | 13C5 PFPeA                               |                            |
| 5         | NS                                       |                            |
| 6         |  | M5PFPeA                    |
| 7         |  |                            |
| 8         | 13C5-PFPEA                               |                            |
| 9         | NT                                       |                            |
| 10        |  |                            |
| 11        | 13C5-PFPeA                               | 13C8-PFOA                  |
| 12        | 13C5-PFPeA                               | 13C2-PFHxA                 |
| 13        | 13C4-PFPeA                               | 13C5 -PFPeA                |
| 15        | 13C3-PFPeA                               | N/A                        |
| 16        | 13C5-PFPeA                               | 13C4-PFOA                  |
| 17        | PFOS-C8                                  | PFPeA-13C3                 |
| 18        |  | 13C5-PFPeA                 |
| 19        | M5PFPeA                                  | NA                         |
| 20        | NT                                       |                            |
| 21        |  | M5PFPeA                    |

Table 92 Labelled Standards for S1 PFHxA

| Lab. Code | Before Extraction                                 | Before Instrument Analysis |
|-----------|---|----------------------------|
| 1         | Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid M5PFHxA |                            |
| 2         | M3 PFHxA  |                            |
| 3         |   |                            |
| 4         | 13C5 PFHxA  | 13C2 PFHxA                 |
| 5         | NS  |                            |
| 6         |   | M5PFHxA                    |
| 7         |   |                            |
| 8         | 13C2-PFHxA  |                            |
| 9         | 13C2-PFHxA  | 13C8-PFOA                  |
| 10        |   |                            |
| 11        | 13C5-PFHxA  | 13C8-PFOA                  |
| 12        | 13C5-PFHxA  | 13C2-PFHxA                 |
| 13        | 13C2-PFHxA  | 13C5 -PFPeA                |
| 15        | 13C2-PFHxA  | N/A                        |
| 16        | 13C2-PFHxA  | 13C4-PFOA                  |
| 17        | PFOS-C8   | PFHxA-13C2                 |
| 18        |   | 13C5-PFHxA                 |
| 19        | M5PFHxA   | NA                         |
| 20        | 13C5-PFHxA  | 13C2-PFHxA                 |
| 21        |   | M5PFHxA                    |

Table 93 Labelled Standards for S1 PFHpA

| Lab. Code | Before Extraction                                | Before Instrument Analysis |
|-----------|--|----------------------------|
| 1         | Perfluoro-n-[1,2,3,4-13C4]heptanoic acid M4PFHpA |                            |
| 2         | M3 PFHxA   |                            |
| 3         |  |                            |
| 4         | 13C4 PFHpA                                       |                            |
| 5         | NS   |                            |
| 6         |  | M4PFHpA                    |
| 7         |  |                            |
| 8         | 13C4-PFHpA                                       |                            |
| 9         | 13C4-PFHpA                                       | 13C8-PFOA                  |
| 10        |  |                            |
| 11        | 13C4-PFHpA                                       | 13C8-PFOA                  |
| 12        | 13C4-PFHpA                                       | 13C4-PFOA                  |
| 13        | 13C3-PFHpA                                       | 13C8-PFOA                  |
| 15        | 13C4-PFHpA                                       | N/A                        |
| 16        | 13C4-PFHpA                                       | 13C4-PFOA                  |
| 17        | PFOS-C8  | PFHpA-13C4                 |
| 18        |  | 13C4-PFHpA                 |
| 19        | MPFHpA   | NA                         |
| 20        | 13C4-PFHpA                                       | 13C2-PFHxA                 |
| 21        |  | M4PFHpA                    |

Table 94 Labelled Standards for S1 PFOA

| Lab. Code | Before Extraction                      | Before Instrument Analysis |
|-----------|--|----------------------------|
| 1         | Perfluoro-n-[13C8]octanoic acid M8PFOA |                            |
| 2         | 13C PFOA                               |                            |
| 3         |  |                            |
| 4         | 13C8 PFOA                              | 13C4 PFOA                  |
| 5         | NS                                     |                            |
| 6         | M2PFOA                                 | M8PFOA                     |
| 7         |  |                            |
| 8         | 13C8-PFOA                              |                            |
| 9         | 13C4-PFOA                              | 13C8-PFOA                  |
| 10        |  |                            |
| 11        | 13C4-PFOA                              | 13C8-PFOA                  |
| 12        | 13C8-PFOA                              | 13C4-PFOA                  |
| 13        | 13C4-PFOA                              | 13C8-PFOA                  |
| 15        | 13C4-PFOA                              | N/A                        |
| 16        | 13C8-PFOA                              | 13C4-PFOA                  |
| 17        | PFOS-C8                                | PFOA-13C4                  |
| 18        | 13C8-PFOA                              | 13C4-PFOA                  |
| 19        | M8PFOA                                 | NA                         |
| 20        | 13C8-PFOA                              | 13C5-PFNA                  |
| 21        | M2PFOA                                 | M8PFOA                     |

Table 95 Labelled Standards for S1 PFNA

| Lab. Code | Before Extraction                      | Before Instrument Analysis |
|-----------|--|----------------------------|
| 1         | Perfluoro-n-[13C9]nonanoic acid M9PFNA |                            |
| 2         | 13C PFOA                               |                            |
| 3         |  |                            |
| 4         | 13C9 PFNA                              | 13C5 PFNA                  |
| 5         | NS                                     |                            |
| 6         |  | M9PFNA                     |
| 7         |  |                            |
| 8         | 13C5-PFNA                              |                            |
| 9         | 13C5-PFNA                              | 13C8-PFOA                  |
| 10        |  |                            |
| 11        | 13C9-PFNA                              | 13C5-PFNA                  |
| 12        | 13C9-PFNA                              | 13C5-PFNA                  |
| 13        | 13C5-PFNA                              | 13C8-PFOA                  |
| 15        | 13C5-PFNA                              | N/A                        |
| 16        | 13C5-PFNA                              | 13C4-PFOA                  |
| 17        | PFOS-C8                                | PFNA-13C5                  |
| 18        |  | 13C5-PFNA                  |
| 19        | M9PFNA                                 | NA                         |
| 20        | 13C9-PFNA                              | 13C5-PFNA                  |
| 21        |  | M9PFNA                     |

Table 96 Labelled Standards for S1 PFDA

| Lab. Code | Before Extraction                                | Before Instrument Analysis |
|-----------|--|----------------------------|
| 1         | Perfluoro-n-[1,2,3,4,6-13C6]decanoic acid M6PFDA |                            |
| 2         | 13C PFOA   |                            |
| 3         |  |                            |
| 4         | 13C6 PFDA  | 13C2 PFDA                  |
| 5         | NS   |                            |
| 6         | MPFDA  | M6PFDA                     |
| 7         |  |                            |
| 8         | 13C6-PFDA  |                            |
| 9         | 13C2-PFDA  | 13C8-PFOA                  |
| 10        |  |                            |
| 11        | 13C2-PFDA  | 13C5-PFNA                  |
| 12        | 13C6-PFDA  | 13C2-PFDA                  |
| 13        | 13C2-PFDA  | 13C8-PFOA                  |
| 15        | 13C2-PFDA  | N/A                        |
| 16        | 13C2-PFDA  | 13C4-PFOA                  |
| 17        | PFOS-C8  | PFDA-13C2                  |
| 18        |  | 13C6-PFDA                  |
| 19        | M6PFDA   | NA                         |
| 20        | 13C6-PFDA  | 13C5-PFNA                  |
| 21        | MPFDA  | M6PFDA                     |

Table 97 Labelled Standards for S1 PFUdA

| Lab. Code | Before Extraction   | Before Instrument Analysis           |
|-----------|---|--------------------------------------|
| 1         | Perfluoro-n-[1,2,3,4,6,7- <sup>13</sup> C <sub>7</sub> ]undecanoic acid M7PFUdA |                                      |
| 2         | MPFUdA  |                                      |
| 3         | NT  |                                      |
| 4         | <sup>13</sup> C <sub>7</sub> PFUnA  |                                      |
| 5         | NS  |                                      |
| 6         |   | M7PFUdA                              |
| 7         |   |                                      |
| 8         | <sup>13</sup> C <sub>2</sub> -PFUDA   |                                      |
| 9         | NT  |                                      |
| 10        |   |                                      |
| 11        | <sup>13</sup> C <sub>2</sub> -PFUdA   | <sup>13</sup> C <sub>5</sub> -PFNA   |
| 12        | <sup>13</sup> C <sub>7</sub> -PFUnA   | <sup>13</sup> C <sub>2</sub> -PFDA   |
| 13        | <sup>13</sup> C <sub>2</sub> -PFUdA   | <sup>13</sup> C <sub>8</sub> -PFOA   |
| 15        | <sup>13</sup> C <sub>2</sub> -PFUdA   | N/A                                  |
| 16        | <sup>13</sup> C <sub>2</sub> -PFUnA   | <sup>13</sup> C <sub>4</sub> -PFOA   |
| 17        | PFOS-C8   | PFUNDA- <sup>13</sup> C <sub>2</sub> |
| 18        |   | <sup>13</sup> C <sub>2</sub> -PFUnDA |
| 19        | M7PFUnDA  | NA                                   |
| 20        | <sup>13</sup> C <sub>7</sub> -PFUdA   | <sup>13</sup> C <sub>5</sub> -PFNA   |
| 21        |   | M7PFUdA                              |

Table 98 Labelled Standards for S1 PFTrDA

| Lab. Code | Before Extraction  | Before Instrument Analysis           |
|-----------|--|--------------------------------------|
| 1         |  |                                      |
| 2         | MPFDoA   |                                      |
| 3         | NT   |                                      |
| 4         | <sup>13</sup> C <sub>2</sub> PFDoA   |                                      |
| 5         | NS   |                                      |
| 6         |  |                                      |
| 7         |  |                                      |
| 8         | <sup>13</sup> C <sub>2</sub> -PFTEDA   |                                      |
| 9         | NT   |                                      |
| 10        |  |                                      |
| 11        | <sup>13</sup> C <sub>2</sub> -PFHxDA   | <sup>13</sup> C <sub>2</sub> -PFTeDA |
| 12        | <sup>13</sup> C <sub>2</sub> -PFDoA;<br><sup>13</sup> C <sub>2</sub> -PFTeDA | <sup>13</sup> C <sub>2</sub> -PFDA   |
| 13        | <sup>13</sup> C <sub>2</sub> -PFTeDA   | <sup>13</sup> C <sub>8</sub> -PFOA   |
| 15        | <sup>13</sup> C <sub>2</sub> -PFTeDA   | N/A                                  |
| 16        | <sup>13</sup> C <sub>2</sub> -PFTeDA   | <sup>13</sup> C <sub>4</sub> -PFOA   |
| 17        | PFOS-C8  | PFTeDA- <sup>13</sup> C <sub>2</sub> |
| 18        |  | <sup>13</sup> C <sub>2</sub> -PFTeDA |
| 19        | MPFDoDA  | NA                                   |
| 20        | <sup>13</sup> C <sub>2</sub> -PFDoA  |                                      |
| 21        |  |                                      |

Table 99 Labelled Standards for S1 PFOSA

| Lab. Code | Before Extraction  | Before Instrument Analysis           |
|-----------|--|--------------------------------------|
| 1         | Perfluoro-1-[ <sup>13</sup> C <sub>8</sub> ]otanesulfonamide |                                      |
| 2         | M8 FOSA  |                                      |
| 3         | NT   |                                      |
| 4         | <sup>13</sup> C <sub>8</sub> PFOSA                           |                                      |
| 5         | NS   |                                      |
| 6         |  | M8-FOSA                              |
| 7         |  |                                      |
| 8         | <sup>13</sup> C <sub>8</sub> -FOSA                           |                                      |
| 9         | NT   |                                      |
| 10        |  |                                      |
| 11        | <sup>13</sup> C <sub>8</sub> -PFOSA                          | <sup>13</sup> C <sub>2</sub> -PFTeDA |
| 12        | <sup>13</sup> C <sub>8</sub> -PFOSA                          | <sup>13</sup> C <sub>4</sub> -PFOS   |
| 13        | <sup>13</sup> C <sub>8</sub> -FOSA                           |                                      |
| 15        | <sup>13</sup> C <sub>8</sub> -FOSA                           | N/A                                  |
| 16        | <sup>13</sup> C <sub>8</sub> -PFOSA                          | <sup>13</sup> C <sub>4</sub> -PFOA   |
| 17        | PFOS-C8  | FOSA- <sup>13</sup> C <sub>8</sub>   |
| 18        |  | <sup>13</sup> C <sub>8</sub> -FOSA   |
| 19        | MPFOSA   | NA                                   |
| 20        | NT   |                                      |
| 21        |  | M8-FOSA                              |

Table 100 Labelled Standards for S1 MeFOSA

| Lab. Code | Before Extraction                         | Before Instrument Analysis |
|-----------|---|----------------------------|
| 1         | N-methyl-d3-perfluoro-1-octanesulfonamide |                            |
| 2         | NT  |                            |
| 3         | NT  |                            |
| 4         | d3-N-MeFOSA                               |                            |
| 5         | NS  |                            |
| 6         |   | d-N-MeFOSA                 |
| 7         |   |                            |
| 8         | d3-N-MEFOSA                               |                            |
| 9         | NT  |                            |
| 10        |   |                            |
| 11        | 13C8-PFOSA                                | 13C2-PFTeDA                |
| 12        | D3-N-MeFOSA                               | 13C4-PFOS                  |
| 13        | D3-N-Me FOSA                              |                            |
| 15        | D3-M PFOSA                                | N/A                        |
| 16        | d3-N-MeFOSA                               | 13C4-PFOA                  |
| 17        | PFOS-C8                                   | MeFOSA-D3                  |
| 18        |   | d3-MeFOSA                  |
| 19        | d-NMeFOSA-M                               | NA                         |
| 20        | NT  |                            |
| 21        |   | d-N-MeFOSA                 |

Table 101 Labelled Standards for S1 EtFOSA

| Lab. Code | Before Extraction                        | Before Instrument Analysis |
|-----------|--|----------------------------|
| 1         | N-ethyl-d5-perfluoro-1-octanesulfonamide |                            |
| 2         | NT                                       |                            |
| 3         | NT                                       |                            |
| 4         | d5-N-EtFOSA                              |                            |
| 5         | NS                                       |                            |
| 6         |  | d-N-EtFOSA                 |
| 7         |  |                            |
| 8         | d5-N-ETFOSA                              |                            |
| 9         | NT                                       |                            |
| 10        |  |                            |
| 11        | 13C8-PFOSA                               | 13C2-PFTeDA                |
| 12        | D5-N-EtFOSA                              | 13C4-PFOS                  |
| 13        | D5-N-Et FOSA                             |                            |
| 15        | D5-E PFOSA                               | N/A                        |
| 16        | d5-N-EtFOSA                              | 13C4-PFOA                  |
| 17        | PFOS-C8                                  | EtFOSA-D5                  |
| 18        |  | d5-EtFOSA                  |
| 19        | d-NEtFOSA-M                              | NA                         |
| 20        | NT                                       |                            |
| 21        |  | d-N-EtFOSA                 |

Table 102 Labelled Standards for S1 ADONA

| Lab. Code | Before Extraction | Before Instrument Analysis |
|-----------|-------------------|----------------------------|
| 1         | NT                |                            |
| 2         | 13C PFOA          |                            |
| 3         |                   |                            |
| 4         | 13C3 HFPO-DA      |                            |
| 5         | NS                |                            |
| 6         | NT                |                            |
| 7         |                   |                            |
| 8         | 13C8-PFOS         |                            |
| 9         | NT                |                            |
| 10        |                   |                            |
| 11        | NT                |                            |
| 12        | 13C3-HFPO-DA      | 13C2-PFHxA                 |
| 13        | 13C4-PFOA         |                            |
| 15        | 13C4-PFHpA        | N/A                        |
| 16        |                   |                            |
| 17        | PFOS-C8           | FOSA-13C8                  |
| 18        | NT                |                            |
| 19        | MPFHpA            | NA                         |
| 20        | NT                |                            |
| 21        | NT                |                            |

Table 103 Labelled Standards for S1  
9Cl-PF3ONS

| Lab. Code | Before Extraction | Before Instrument Analysis |
|-----------|-------------------|----------------------------|
| 1         | NT                |                            |
| 2         | MPFHxS            |                            |
| 3         |                   |                            |
| 4         | 13C3 HFPO-DA      |                            |
| 5         | NS                |                            |
| 6         | NT                |                            |
| 7         |                   |                            |
| 8         | 13C8-PFOS         |                            |
| 9         | NT                |                            |
| 10        |                   |                            |
| 11        | NT                |                            |
| 12        | 13C3-HFPO-DA      | 13C2-PFHxA                 |
| 13        | 13C4-PFOS         | 13C8-PFOS                  |
| 15        | 13C4-PFOS         | N/A                        |
| 16        |                   |                            |
| 17        | PFOS-C8           |                            |
| 18        | NT                |                            |
| 19        | M8PFOS            | NA                         |
| 20        | NT                |                            |
| 21        | NT                |                            |

Table 104 Labelled Standards for S1  
11Cl-PF3OUdS

| Lab. Code | Before Extraction | Before Instrument Analysis |
|-----------|-------------------|----------------------------|
| 1         | NT                |                            |
| 2         | MPFHxS            |                            |
| 3         |                   |                            |
| 4         | 13C3 HFPO-DA      |                            |
| 5         | NS                |                            |
| 6         | NT                |                            |
| 7         |                   |                            |
| 8         | 13C2-PFHXA        |                            |
| 9         | NT                |                            |
| 10        |                   |                            |
| 11        | NT                |                            |
| 12        | 13C3-HFPO-DA      | 13C2-PFHxA                 |
| 13        | 13C4-PFOS         | 13C8-PFOS                  |
| 15        | 13C4-PFOS         | N/A                        |
| 16        |                   |                            |
| 17        | PFOS-C8           |                            |
| 18        | NT                |                            |
| 19        | MPFDoDA           | NA                         |
| 20        | NT                |                            |
| 21        | NT                |                            |

Table 105 Participant Methodology – Sample S2 Carrot Extraction

| Lab. Code | S2 Sample Weight (g)  | Sample Pretreatment   | Extraction Technique                                   | Extraction Solvent                        | Extraction Temperature      | Extraction Time                        | Clean-Up  |
|-----------|-----------------------|---|--|---|-----------------------------|--|---|
| 1         | 10                    | Homogenisation<br>Geno/Grinder<br>14min &<br>Centrifuge 10min | QuEChERS - modified AOAC                               | Acetonitrile with<br>1% Acetic Acid       | Room                        | Sonicate 30<br>min at 30-35<br>degrees | envicarb  |
| 2         |                       |   |  |   |                             |  |   |
| 3         | NS                    |   |  |   |                             |  |   |
| 4         | 1                     | Homogenisation  | Alkaline Digestion                                     | KOH-methanol                              | Room temp                   | 8 hrs                                  | Active carbon SPE   |
| 5         | 1                     | Freeze-drying   | Soxhlet  | MeOH                                      | boiling point               | 4h                                     | ion pair separation                                       |
| 6         |                       |   | Solid-Liquid Extraction                                |   |                             |  |   |
| 7         |                       |   |  |   |                             |  |   |
| 8         | NS                    |   |  |   |                             |  |   |
| 9         |                       |   |  |   |                             |  |   |
| 10        | NS                    |   |  |   |                             |  |   |
| 11        | 1                     | No  | Solid-Liquid Extraction                                | Acetonitrile                              | Room temperature            | 30 min                                 | SPE-WAX,<br>ultracentrifugation                           |
| 12        | 2.14 (as<br>received) | Homogenisation  | Solid-Liquid Extraction                                | KOH-Methanol                              | Ambient Room<br>Temperature | 16 hours                               | Activated<br>Carbon/SPE/Filtration                        |
| 13        | 5                     | Homogenisation  | Digestion with 10mM NaOH<br>before QuEChERS extraction | Acetonitrile                              | Room temperature            | 30 min                                 | Bond Elut Carbon, then SPE<br>with Strata X-AW cartridges |
| 15        | 2g                    | Homogenisation  | Alkaline Digestion                                     | Basified MeOH                             | Room                        | 60 mins                                | Envicarb  |
| 16        | 2.5                   | Homogenisation  | QuEChERS   | Methanol +<br>Ammonium-ACN<br>and Acetone | 40 °C                       | 30 min                                 | Solid-Phase Extraction                                    |
| 17        | 5                     | Homogenisation  | Solid-Liquid Extraction                                | Acetonitrile                              | Ambient                     | 30                                     | Solid-Phase Extraction                                    |
| 18        | 0.5 grams             | Homogenisation  | Solid-Liquid Extraction                                | MTbE                                      | Room                        | 60 minutes                             | C18 & Activated Carbon                                    |

| Lab. Code | S2 Sample Weight (g)        | Sample Pretreatment | Extraction Technique                                   | Extraction Solvent             | Extraction Temperature | Extraction Time | Clean-Up   |
|-----------|-----------------------------|---------------------|--|--------------------------------|------------------------|-----------------|--|
| 19        | 2.024 and 2.082 (duplicate) | NA                  | Solid-Liquid Extraction (SLE)<br>Merris-Minimix shaker | 2% formic acid in acetonitrile | Room temperature       | 8 min           | dSPE (C18, Envicarb, MgSO4)                            |
| 20        | 2.5                         | Homogenisation      | Solid-Liquid Extraction                                | 0.1 % NH3 in ACN               | room temperature       | 2 hours         | Solid-Phase Extraction<br>two different SPE cartridges |
| 21        |                             |                     | Alkaline Digestion                                     |                                |                        |                 |  |

Table 106 Participant Methodology – Sample S2 Carrot Instrumental Technique and Analysis

| Lab. Code | Instrument        | Guard Column | Instrument Column          | Dilution Factor | Delay Column? | Blank Correction? | Standard Method?   |
|-----------|-------------------|--------------|----------------------------|-----------------|---------------|-------------------|--|
| 1         | Orbitrap          | C18 3mm      | Kinetex C18 100x3mm 2.6 um |                 | Yes           | Yes               | In house   |
| 2         |                   |              |                            |                 |               |                   | US FDA Foods Program<br>Compendium of Analytical<br>Laboratory Methods;<br>method C-010.02 |
| 3         | NS                |              |                            |                 |               |                   |  |
| 4         | LC-MSMS or LC-QQQ | No           | C18, 50 mm                 | No              | Yes           | No                | No   |
| 5         | LC-MSMS or LC-QQQ | C18          | Biphenyl 150x 2.5 mm       | yes 1:10; 1:100 | No            | No                | no   |
| 6         | LC-MSMS or LC-QQQ | C18          | 2.1x100mm 1.9um            |                 | Yes           | No                | USEPA 537  |
| 7         |                   |              |                            |                 |               |                   |  |
| 8         | NS                |              |                            |                 |               |                   |  |
| 9         |                   |              |                            |                 |               |                   |  |
| 10        | NS                |              |                            |                 |               |                   |  |

| Lab. Code | Instrument        | Guard Column                            | Instrument Column                             | Dilution Factor | Delay Column? | Blank Correction? | Standard Method?  |
|-----------|-------------------|---|---|-----------------|---------------|-------------------|-------------------|
| 11        | LC-MSMS or LC-QQQ | Gemini NX-C18; 4mm x 2.0mm ID           | NX-C18; 15cm x 2mm x 3um                      | No              | Yes           | Yes               |                   |
| 12        | LC-MSMS or LC-QQQ | Phenomenex Evo C18 (2µm, 2 mm x 2.1 mm) | BEH C18 (1.7µm, 50 mm x 2.1 mm)               | No              | Yes           | No                | No                |
| 13        | LC-MSMS or LC-QQQ | Phenomenex Evo C18 (2 mm x 2.1 mm)      | Phenomenex Evo C18 (100 mm x 2.1 mm x 2.6 um) | No              | Yes           | No                | Isotopic Dilution |
| 15        | LC-MSMS or LC-QQQ | Pre-column Filter 0.2µm                 | C18 50mm x 2.1mm x 1.8µm                      | 50              | Yes           | No                | No. In-house      |
| 16        | LC-MSMS or LC-QQQ | UltraShield UHPLC 0.2 µm Restek         | Raptor C18 1.8 µm 50 x 2.1 mm Restek          |                 | yes           | no                |                   |
| 17        | Orbitrap          | C18                                     | C18   |                 | Yes           |                   |                   |
| 18        | LC-MSMS or LC-QQQ | nil                                     | C18 1.6µm, 2.0mm x 50mm                       | No              | Yes           | No                |                   |
| 19        | LC-MSMS or LC-QQQ | NA                                      | Zorbax XDB-C18, 100 mm x 2.1 mm, 1.8µm        | NA              | Yes           | No                | No                |
| 20        | LC-MSMS or LC-QQQ | C18 Column, 2.1 x 5 mm, 3.5 µm          | C18 Column, 130Å, 3 x 50 mm, 3.5 µm           |                 | Yes           | No                |                   |
| 21        | LC-MSMS or LC-QQQ | C18                                     | 2.1x100mn 1.9um                               |                 | Yes           | No                | USEPA 537         |

Table 107 Participant Methodology – Sample S2 Carrot Labelled Standards

| Lab. Code | Labelled Standard Source | Recovery Correction? | Labelled Standards Additional Information     |
|-----------|--------------------------|----------------------|---|
| 1         | Wellington               | No                   |   |
| 2         | Wellington               | Yes                  | d5NN EtFOSAA added before instrument analysis |
| 3         | NS                       |                      |   |
| 4         | Wellington               | Yes                  |   |



| Lab. Code | Labelled Standard Source | Recovery Correction? | Labelled Standards Additional Information              |
|-----------|--------------------------|----------------------|--|
| 5         | Wellington               | Yes                  |  |
| 6         | Wellington               | no                   |  |
| 7         |                          |                      |  |
| 8         |                          |                      | NS   |
| 9         |                          |                      |  |
| 10        |                          |                      | NS   |
| 11        |                          |                      |  |
| 12        | Wellington               | Yes                  |  |
| 13        | Wellington               | Yes                  |  |
| 15        | Wellington               | Yes                  |  |
| 16        |                          | Yes                  |  |
| 17        | Wellington               | Yes                  | Results corrected by ISTD added before instrumentation |
| 18        | Wellington Laboratories  | No                   |  |
| 19        | Wellington Laboratory    | Yes                  |  |
| 20        | Wellington               | No                   |  |
| 21        | Wellington               | no                   |  |

Table 108 Labelled Standards for S2 PFBS

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

Table 109 Labelled Standards for S2 PFPeS

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

Table 110 Labelled Standards for S2 PFHxS

| Lab. Code | Before Extraction   | Before Instrument Analysis          |
|-----------|---|-------------------------------------|
| 1         | Sodium perfluoro-1-[1,2,3 <sup>13</sup> C <sub>3</sub> ] hexanesulfonate<br>M3PFHxS |                                     |
| 2         |   |                                     |
| 3         | NS  |                                     |
| 4         | <sup>13</sup> C <sub>3</sub> PFHxS  | PFHxS18O <sub>2</sub>               |
| 5         | NT  |                                     |
| 6         |   |                                     |
| 7         |   |                                     |
| 8         | NS  |                                     |
| 9         | NT  |                                     |
| 10        | NS  |                                     |
| 11        | NT  |                                     |
| 12        | <sup>13</sup> C <sub>3</sub> -PFHxS   | 18O <sub>2</sub> -PFHxS             |
| 13        | 18O <sub>2</sub> -PFHxS   | <sup>13</sup> C <sub>3</sub> -PFHxS |
| 15        | 18O <sub>2</sub> -PFHxS   | N/A                                 |
| 16        | 18O <sub>2</sub> -PFHxS   | <sup>13</sup> C <sub>4</sub> -PFOA  |
| 17        | PFOS-C8   | PFHxS-18O <sub>2</sub>              |
| 18        |   | 16O <sub>2</sub> -PFHxS             |
| 19        | M3PFHxS   | NA                                  |
| 20        | linear <sup>13</sup> C <sub>3</sub> -PFHxS  |                                     |
| 21        |   |                                     |

Table 111 Labelled Standards for S2 PFHxS (linear)

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

Table 112 Labelled Standards for S2 PFHpS

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

Table 113 Labelled Standards for S2 PFOS

| Lab. Code | Before Extraction                                | Before Instrument Analysis |
|-----------|--|----------------------------|
| 1         | Sodium perfluoro-1-[13C8] octanesulfonate M8PFOS |                            |
| 2         | 13C PFOS   |                            |
| 3         | NS   |                            |
| 4         | 13C8 PFOS  | 13C4 PFOS                  |
| 5         | NT   |                            |
| 6         |  |                            |
| 7         |  |                            |
| 8         | NS   |                            |
| 9         | NT   |                            |
| 10        | NS   |                            |
| 11        | 13C4-PFOS  | 18O2-PFOS                  |
| 12        | 13C8-PFOS  | 13C4-PFOS                  |
| 13        | 13C4-PFOS  | 13C8-PFOS                  |
| 15        | 13C4-PFOS  | N/A                        |
| 16        | 13C4-PFOS  | 13C4-PFOA                  |
| 17        | PFOS-C8  | PFOS-C4                    |
| 18        | 13C4-PFOS  | 13C8-PFOS                  |
| 19        | M8PFOS   | NA                         |
| 20        | linear 13C8-PFOS                                 | 13C5-PFNA                  |
| 21        |  |                            |

Table 114 Labelled Standards for S2 PFOS  
(linear)

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

Table 115 Labelled Standards for S2 PFNS

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

Table 116 Labelled Standards for S2 PFDS

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

Table 117 Labelled Standards for S2 PFBA

| Lab. Code | Before Extraction                     | Before Instrument Analysis |
|-----------|---------------------------------------|----------------------------|
| 1         | Perfluoro-n-[13C4]butanoic acid MPFBA |                            |
| 2         | M3 PFBA                               |                            |
| 3         | NS                                    |                            |
| 4         | 13C4 PFBA                             | 13C3 PFBA                  |
| 5         | yes                                   |                            |
| 6         | M3PFBA                                | MPFBA                      |
| 7         |                                       |                            |
| 8         | NS                                    |                            |
| 9         | NT                                    |                            |
| 10        | NS                                    |                            |
| 11        | 13C4-PFBA                             | 13C8-PFOA                  |
| 12        | 13C4-PFBA                             | 13C3-PFBA                  |
| 13        | 13C4-PFBA                             | 13C3-PFBA                  |
| 15        | 13C4-PFBA                             | N/A                        |
| 16        | 13C4-PFBA                             | 13C4-PFOA                  |
| 17        | PFOS-C8                               | PFBA-13C4                  |
| 18        |                                       | 13C4-PFBA                  |
| 19        | M4PFBA                                | NA                         |
| 20        | NT                                    |                            |
| 21        | M3PFBA                                | MPFBA                      |

Table 118 Labelled Standards for S2 PFPeA

| Lab. Code | Before Extraction                        | Before Instrument Analysis |
|-----------|--|----------------------------|
| 1         | Perfluoro-n-[13C5]pentanoic acid M5PFPeA |                            |
| 2         | M3 PFPeA                                 |                            |
| 3         | NS                                       |                            |
| 4         | 13C5 PFPeA                               |                            |
| 5         |  |                            |
| 6         |  | M5PFPeA                    |
| 7         |  |                            |
| 8         | NS                                       |                            |
| 9         | NT                                       |                            |
| 10        | NS                                       |                            |
| 11        | 13C5-PFPeA                               | 13C8-PFOA                  |
| 12        | 13C5-PFPeA                               | 13C2-PFHxA                 |
| 13        | 13C4-PFPeA                               | 13C5 -PFPeA                |
| 15        | 13C3-PFPeA                               | N/A                        |
| 16        | 13C5-PFPeA                               | 13C4-PFOA                  |
| 17        | PFOS-C8                                  | PFPeA-13C3                 |
| 18        |  | 13C5-PFPeA                 |
| 19        | M5PFPeA                                  | NA                         |
| 20        | NT                                       |                            |
| 21        |  | M5PFPeA                    |

Table 119 Labelled Standards for S2 PFHxA

| Lab. Code | Before Extraction                                 | Before Instrument Analysis |
|-----------|---|----------------------------|
| 1         | Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid M5PFHxA |                            |
| 2         | M3 PFHxA  |                            |
| 3         | NS  |                            |
| 4         | 13C5 PFHxA  | 13C2 PFHxA                 |
| 5         | yes   |                            |
| 6         |   | M5PFHxA                    |
| 7         |   |                            |
| 8         | NS  |                            |
| 9         | NT  |                            |
| 10        | NS  |                            |
| 11        | 13C5-PFHxA  | 13C8-PFOA                  |
| 12        | 13C5-PFHxA  | 13C2-PFHxA                 |
| 13        | 13C2-PFHxA  | 13C5 -PFPeA                |
| 15        | 13C2-PFHxA  | N/A                        |
| 16        | 13C2-PFHxA  | 13C4-PFOA                  |
| 17        | PFOS-C8   | PFHxA-13C2                 |
| 18        |   | 13C5-PFHxA                 |
| 19        | M5PFHxA   | NA                         |
| 20        | 13C5-PFHxA  | 13C2-PFHxA                 |
| 21        |   | M5PFHxA                    |

Table 120 Labelled Standards for S2 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         | M3 PFHxA   |                                     |
| 3         | NS   |                                     |
| 4         | <sup>13</sup> C <sub>4</sub> PFHpA   |                                     |
| 5         |  |                                     |
| 6         |  | M4PFHpA                             |
| 7         |  |                                     |
| 8         | NS   |                                     |
| 9         | NT   |                                     |
| 10        | NS   |                                     |
| 11        | <sup>13</sup> C <sub>4</sub> -PFHpA  | <sup>13</sup> C <sub>8</sub> -PFOA  |
| 12        | <sup>13</sup> C <sub>4</sub> -PFHpA  | <sup>13</sup> C <sub>4</sub> -PFOA  |
| 13        | <sup>13</sup> C <sub>3</sub> -PFHpA  | <sup>13</sup> C <sub>8</sub> -PFOA  |
| 15        | <sup>13</sup> C <sub>4</sub> -PFHpA  | N/A                                 |
| 16        | <sup>13</sup> C <sub>4</sub> -PFHpA  | <sup>13</sup> C <sub>4</sub> -PFOA  |
| 17        | PFOS-C8  | PFHpA- <sup>13</sup> C <sub>4</sub> |
| 18        |  | <sup>13</sup> C <sub>4</sub> -PFHpA |
| 19        | MPFHpA   | NA                                  |
| 20        | <sup>13</sup> C <sub>4</sub> -PFHpA  | <sup>13</sup> C <sub>2</sub> -PFHxA |
| 21        |  | M4PFHpA                             |

Table 121 Labelled Standards for S2 PFOA

| Lab. Code | Before Extraction  | Before Instrument Analysis         |
|-----------|--|------------------------------------|
| 1         | Perfluoro-n-[ <sup>13</sup> C <sub>8</sub> ]octanoic acid M8PFOA |                                    |
| 2         | <sup>13</sup> C PFOA   |                                    |
| 3         | NS   |                                    |
| 4         | <sup>13</sup> C <sub>8</sub> PFOA                                | <sup>13</sup> C <sub>4</sub> PFOA  |
| 5         | yes  |                                    |
| 6         | M2PFOA   | M8PFOA                             |
| 7         |  |                                    |
| 8         | NS   |                                    |
| 9         | NT   |                                    |
| 10        | NS   |                                    |
| 11        | <sup>13</sup> C <sub>4</sub> -PFOA                               | <sup>13</sup> C <sub>8</sub> -PFOA |
| 12        | <sup>13</sup> C <sub>8</sub> -PFOA                               | <sup>13</sup> C <sub>4</sub> -PFOA |
| 13        | <sup>13</sup> C <sub>4</sub> -PFOA                               | <sup>13</sup> C <sub>8</sub> -PFOA |
| 15        | <sup>13</sup> C <sub>4</sub> -PFOA                               | N/A                                |
| 16        | <sup>13</sup> C <sub>8</sub> -PFOA                               | <sup>13</sup> C <sub>4</sub> -PFOA |
| 17        | PFOS-C8  | PFOA- <sup>13</sup> C <sub>4</sub> |
| 18        | <sup>13</sup> C <sub>8</sub> -PFOA                               | <sup>13</sup> C <sub>4</sub> -PFOA |
| 19        | M8PFOA   | NA                                 |
| 20        | <sup>13</sup> C <sub>8</sub> -PFOA                               | <sup>13</sup> C <sub>5</sub> -PFNA |
| 21        | M2PFOA   | M8PFOA                             |

Table 122 Labelled Standards for S2 PFNA

| Lab. Code | Before Extraction  | Before Instrument Analysis         |
|-----------|--|------------------------------------|
| 1         | Perfluoro-n-[ <sup>13</sup> C <sub>9</sub> ]nonanoic acid M9PFNA |                                    |
| 2         | <sup>13</sup> C PFOA   |                                    |
| 3         | NS   |                                    |
| 4         | <sup>13</sup> C <sub>9</sub> PFNA                                | <sup>13</sup> C <sub>5</sub> PFNA  |
| 5         | yes  |                                    |
| 6         |  | M9PFNA                             |
| 7         |  |                                    |
| 8         | NS   |                                    |
| 9         | NT   |                                    |
| 10        | NS   |                                    |
| 11        | <sup>13</sup> C <sub>9</sub> -PFNA                               | <sup>13</sup> C <sub>5</sub> -PFNA |
| 12        | <sup>13</sup> C <sub>9</sub> -PFNA                               | <sup>13</sup> C <sub>5</sub> -PFNA |
| 13        | <sup>13</sup> C <sub>5</sub> -PFNA                               | <sup>13</sup> C <sub>8</sub> -PFOA |
| 15        | <sup>13</sup> C <sub>5</sub> -PFNA                               | N/A                                |
| 16        | <sup>13</sup> C <sub>5</sub> -PFNA                               | <sup>13</sup> C <sub>4</sub> -PFOA |
| 17        | PFOS-C8  | PFNA- <sup>13</sup> C <sub>5</sub> |
| 18        |  | <sup>13</sup> C <sub>5</sub> -PFNA |
| 19        | M9PFNA   | NA                                 |
| 20        | <sup>13</sup> C <sub>9</sub> -PFNA                               | <sup>13</sup> C <sub>5</sub> -PFNA |
| 21        |  | M9PFNA                             |

Table 123 Labelled Standards for S2 PFDA

| Lab. Code | Before Extraction                                   | Before Instrument Analysis |
|-----------|---|----------------------------|
| 1         | Perfluoro-n-[1,2,3,4,6-13C6]decanoic acid<br>M6PFDA |                            |
| 2         | 13C PFOA  |                            |
| 3         | NS  |                            |
| 4         | 13C6 PFDA   | 13C2 PFDA                  |
| 5         | yes   |                            |
| 6         | MPFDA   | M6PFDA                     |
| 7         |   |                            |
| 8         | NS  |                            |
| 9         | NT  |                            |
| 10        | NS  |                            |
| 11        | 13C2-PFDA   | 13C5-PFNA                  |
| 12        | 13C6-PFDA   | 13C2-PFDA                  |
| 13        | 13C2-PFDA   | 13C8-PFOA                  |
| 15        | 13C2-PFDA   | N/A                        |
| 16        | 13C2-PFDA   | 13C4-PFOA                  |
| 17        | PFOS-C8   | PFDA-13C2                  |
| 18        |   | 13C6-PFDA                  |
| 19        | M6PFDA  | NA                         |
| 20        | 13C6-PFDA   | 13C5-PFNA                  |
| 21        | MPFDA   | M6PFDA                     |

Table 124 Labelled Standards for S2 PFOSA

| Lab. Code | Before Extraction                  | Before Instrument Analysis |
|-----------|------------------------------------|----------------------------|
| 1         | Perfluoro-1-[13C8]otanesulfonamide |                            |
| 2         | M8 FOSA                            |                            |
| 3         | NS                                 |                            |
| 4         | 13C8 PFOSA                         |                            |
| 5         | NT                                 |                            |
| 6         |                                    | M8-FOSA                    |
| 7         |                                    |                            |
| 8         | NS                                 |                            |
| 9         | NT                                 |                            |
| 10        | NS                                 |                            |
| 11        | 13C8-PFOSA                         | 13C2-PFTeDA                |
| 12        | 13C8-PFOSA                         | 13C4-PFOS                  |
| 13        | 13C8-FOSA                          |                            |
| 15        | 13C8-FOSA                          | N/A                        |
| 16        | 13C8-PFOSA                         | 13C4-PFOA                  |
| 17        | PFOS-C8                            | FOSA-13C8                  |
| 18        |                                    | 13C8-FOSA                  |
| 19        | MPFOSA                             | NA                         |
| 20        | NT                                 |                            |
| 21        |                                    | M8-FOSA                    |

Table 125 Labelled Standards for S2 MeFOSA

| Lab. Code | Before Extraction                         | Before Instrument Analysis |
|-----------|---|----------------------------|
| 1         | N-methyl-d3-perfluoro-1-octanesulfonamide |                            |
| 2         | NT  |                            |
| 3         | NS  |                            |
| 4         | d3-N-MeFOSA                               |                            |
| 5         | NT  |                            |
| 6         |   | d-N-MeFOSA                 |
| 7         |   |                            |
| 8         | NS  |                            |
| 9         | NT  |                            |
| 10        | NS  |                            |
| 11        | 13C8-PFOSA                                | 13C2-PFTeDA                |
| 12        | D3-N-MeFOSA                               | 13C4-PFOS                  |
| 13        | D3-N-Me FOSA                              |                            |
| 15        | D3-M PFOSA                                | N/A                        |
| 16        | d3-N-MeFOSA                               | 13C4-PFOA                  |
| 17        | PFOS-C8                                   | MeFOSA-D3                  |
| 18        |   | d3-MeFOSA                  |
| 19        | d-NMeFOSA-M                               | NA                         |
| 20        | NT  |                            |
| 21        |   | d-N-MeFOSA                 |

Table 126 Labelled Standards for S2 EtFOSA

| Lab. Code | Before Extraction                        | Before Instrument Analysis |
|-----------|--|----------------------------|
| 1         | N-ethyl-d5-perfluoro-1-octanesulfonamide |                            |
| 2         | NT                                       |                            |
| 3         | NS                                       |                            |
| 4         | d5-N-EtFOSA                              |                            |
| 5         | NT                                       |                            |
| 6         |  | d-N-EtFOSA                 |
| 7         |  |                            |
| 8         | NS                                       |                            |
| 9         | NT                                       |                            |
| 10        | NS                                       |                            |
| 11        | 13C8-PFOSA                               | 13C2-PFTeDA                |
| 12        | D5-N-EtFOSA                              | 13C4-PFOS                  |
| 13        | D5-N-Et FOSA                             |                            |
| 15        | D5-E PFOSA                               | N/A                        |
| 16        | d5-N-EtFOSA                              | 13C4-PFOA                  |
| 17        | PFOS-C8                                  | EtFOSA-D5                  |
| 18        |  | d5-EtFOSA                  |
| 19        | d-NEtFOSA-M                              | NA                         |
| 20        | NT                                       |                            |
| 21        |  | d-N-EtFOSA                 |

Table 127 Labelled Standards for S2 6:2 FTS

| Lab. Code | Before Extraction  | Before Instrument Analysis |
|-----------|--|----------------------------|
| 1         | M2-6:2FTS - 1H,1H,2H,2H-perfluoro1-[1,2-13C2]-octane sulfonate (6:2) |                            |
| 2         | 13C2D4 6:2 FTS   |                            |
| 3         | NS   |                            |
| 4         | 13C2 6:2 FTS   |                            |
| 5         | NT   |                            |
| 6         |  | M2-6:2FTS                  |
| 7         |  |                            |
| 8         | NS   |                            |
| 9         | NT   |                            |
| 10        | NS   |                            |
| 11        | NT   |                            |
| 12        | 13C2-6:2 FTS   | 13C2-D4-6:2 FTS            |
| 13        | 13C2-6:2 FTS   |                            |
| 15        | 13C2,12C6 6:2-FTS  | N/A                        |
| 16        | 13C2-6:2FTS  | 13C4-PFOA                  |
| 17        | PFOS-C8  | 6:2 FTS-13C2               |
| 18        |  | 13C2-6:2 FTS               |
| 19        | M6:2 FTS   | NA                         |
| 20        | 13C2-6:2 FTS   |                            |
| 21        |  | M2-6:2FTS                  |

Table 128 Labelled Standards for S2 GenX

| Lab. Code | Before Extraction | Before Instrument Analysis |
|-----------|-------------------|----------------------------|
| 1         | NT                |                            |
| 2         | M3 HFPO           |                            |
| 3         | NS                |                            |
| 4         | 13C3 HFPO-DA      |                            |
| 5         | NT                |                            |
| 6         |                   |                            |
| 7         |                   |                            |
| 8         | NS                |                            |
| 9         | NT                |                            |
| 10        | NS                |                            |
| 11        | NT                |                            |
| 12        | 13C3-HFPO-DA      | 13C2-PFHxA                 |
| 13        | 13C4-PFOA         |                            |
| 15        | 13C312C3HF11O3    | N/A                        |
| 16        |                   |                            |
| 17        | PFOS-C8           | PFPeA-13C3                 |
| 18        | NT                |                            |
| 19        | M3HFPO-DA         | NA                         |
| 20        | 13C3-GenX         |                            |
| 21        |                   |                            |



Table 129 Labelled Standards for S2 ADONA

| Lab. Code | Before Extraction | Before Instrument Analysis |
|-----------|-------------------|----------------------------|
| 1         | NT                |                            |
| 2         | 13C PFOA          |                            |
| 3         | NS                |                            |
| 4         | 13C3 HFPO-DA      |                            |
| 5         | NT                |                            |
| 6         |                   |                            |
| 7         |                   |                            |
| 8         | NS                |                            |
| 9         | NT                |                            |
| 10        | NS                |                            |
| 11        | NT                |                            |
| 12        | 13C3-HFPO-DA      | 13C2-PFHxA                 |
| 13        | 13C4-PFOA         |                            |
| 15        | 13C4-PFHpA        | N/A                        |
| 16        |                   |                            |
| 17        | PFOS-C8           | FOSA-13C8                  |
| 18        | NT                |                            |
| 19        | MPFHpA            | NA                         |
| 20        | NT                |                            |
| 21        | NT                |                            |

## APPENDIX 5 ACRONYMS AND ABBREVIATIONS

|              |   |
|--------------|---|
| 4:2 FTS      | 4:2 Fluorotelomer sulfonate                                       |
| 6:2 FTS      | 6:2 Fluorotelomer sulfonate                                       |
| 8:2 FTS      | 8:2 Fluorotelomer sulfonate                                       |
| 10:2 FTS     | 10:2 Fluorotelomer sulfonate                                      |
| 9Cl-PF3ONS   | 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate                    |
| 11Cl-PF3OUdS | 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate                   |
| ACN          | Acetonitrile  |
| ADONA        | Ammonium 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         | Ammonium 2,3,3,3-tetrafluoro-2-(heptafluoropropoxy) propanoate    |
| 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                               |
| MTBE   | Methyl <i>tert</i> -butyl ether                        |
| 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                                 |
| PFOS   | Perfluorooctane sulfonate                              |
| PFOSA  | Perfluorooctane sulfonamide                            |
| PFPeA  | Perfluoropentanoic acid                                |
| PFPeS  | Perfluoropentane sulfonate                             |
| PFTeDA | Perfluorotetradecanoic acid                            |
| PFTrDA | Perfluorotridecanoic acid                              |
| PFTrDS | Perfluorotridecane sulfonate                           |
| PFUdA  | Perfluoroundecanoic acid                               |
| PFUdS  | Perfluoroundecane sulfonate                            |
| PT     | Proficiency 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  |
| $\sigma$  | Target standard deviation for proficiency assessment             |

**END OF REPORT**