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Proficiency Test Final Report AQA 26-21 PFAS, EOF, and TF in Consumer Goods

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SUMMARY

This report presents the results of the proficiency testing (PT) study, AQA 26-21 PFAS, EOF, and TF in Consumer Goods. This study was designed based on current and emerging legislation, and participants' requests. It is focused on the measurement of total fluorine (TF) and 19 per- and polyfluoroalkyl substances (PFAS): PFBA, PFPeA, PFHxA, PFOA, PFNA, PFDA, PFUdA, PFDoA, PFTrDA, PFTeDA, PFBS, PFHxS, PFOS, N-EtFOSAA, 6:2FTS, 8:2FTS, 8:2diPAP, GenX, and ADONA in cardboard packaging material. The study also included two pilot samples, a carboard packaging material (pilot Sample S2) to be analysed for Extractable Organic Fluorine (EOF) and a cosmetic (foundation) material (pilot Sample S4) to be analysed for individual PFAS analytes, EOF, and Total Fluorine (TF).

The sample set consisted of 4 samples:

- Sample S1: 3 g of cardboard packaging material spiked with 19 PFAS contaminants.
- Pilot Sample S2: 3 g of cardboard packaging material spiked with 19 PFAS contaminants to be analysed for EOF only.
- Sample S3: 3 g of cardboard packaging material to be analysed for TF.
- Pilot Sample S4: 20 g of cosmetic (foundation) material spiked with 10 PFAS contaminants, to be analysed for PFAS, EOF and TF.

No assigned values were set for the tests conducted on the pilot samples S2 and S4. These samples were not intended for performance assessment, but to assist laboratories with method development for EOF in packaging materials and for individual PFAS, EOF, and TF in cosmetic products (foundation).

The assigned values for Samples S1 and S3 were the robust averages of participants' results. The associated uncertainties were evaluated from the robust standard deviations of the participants' results.

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

i. compare laboratories' performance and assess their capabilities to measure PFAS, and TF in packaging material;

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

Of 151 z-scores, 130 (86%) returned $|z| \leq 2.0$, indicating an acceptable performance.

Of 151 E_n-scores, 116 (77%) returned $|E_n| < 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Laboratory 8 returned acceptable z-scores and E_n-scores for all 15 analytes for which z-scores were calculated.

One laboratory did not report numeric results for analytes that they tested for and were present in the study samples while 8 participants reported results for analytes that were not spiked into the test samples.

ii. evaluate laboratories' methods used in determination of PFAS, EOF, and TF in packaging material and cosmetics;

The most popular method used for the PFAS analysis in cardboard material sample S1 consisted of solid liquid extraction (SLE), a sample size of 3 g (entire sample as instructed), methanol base as extraction solvent, 40°C to 45°C for extract concentration, and no cleanup. LC-MS/MS was the instrumental technique of choice for all participants.

The majority of the results reported by laboratories which did not add isotopically labelled internal standards directly to the original container of Sample S1 were biased low or high, likely due to uncorrected errors introduced during sample preparation.

There may be a relationship between low-biased PFDA, PFUdA, and PFDoA results and the use of ACN, particularly in the absence of methanol, as the extraction reagent. PFAS extraction from matrices requires strong solvent-matrix disruption and ACN has limited ability to disrupt strongly bound PFAS.

EOF is not yet routinely analysed by laboratories. Only 4 participants reported numeric results for EOF in the cardboard pilot Sample S2, and all used different method parameters. However 3 of the 4 reported results were in good agreement with each other and with the calculated spiked value.

Participants used a wide range of sample sizes for TF analysis in the cardboard packaging material sample S3, from 18 mg to 550 mg. Despite this variation, all reported results returned acceptable z-scores.

The most popular method used for PFAS analysis in the cosmetic pilot Sample S4 consisted of solid liquid extraction, a sample size of 1 g, methanol base as extraction solvent, 40°C for extract concentration, and SPE cleanup. The majority of reported results were in good agreement with each other and the spiked value.

Participants used a variety of sample sizes for analysis of TF in the pilot Sample S4, from 14 mg to 100 mg. Most of the reported results were in good agreement with each other.

iii. develop the practical application of measurement uncertainty, and provide participants with information that will be useful in assessing their measurement uncertainty evaluations;

Laboratories should review their uncertainty evaluation procedure as some laboratories have reported unrealistically small or large relative uncertainties for routine PFAS analyses. The magnitude of these expanded measurement uncertainties was within the range 0.2% to 63% of the reported value.

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

The study samples S1 and S3 are homogeneous and stable and are well characterised. Surplus test samples are available for purchase from NMIA and can be used for quality control and method development purposes.

TABLE OF CONTENTS

1 INTRODUCTION 1
1.1 NMIA Proficiency Testing Program 1
1.2 Study Background 1
1.3 Study Aims 2
1.4 Study Conduct 2
2 STUDY INFORMATION 2
2.1 Study Timetable 2
2.2 Participation and Laboratory Code 3
2.3 Selection of Analytes and Test Material Preparation 3
2.4 Test Material Homogeneity and Stability Testing 4
2.5 Sample Storage, Dispatch, and Receipt 4
2.6 Instructions to Participants 4
2.7 Interim Report and Preliminary Report 5
3 PARTICIPANT LABORATORY INFORMATION 6
3.1 Test Methods Reported by Participants 6
3.2 Basis of Participants' Measurement Uncertainty Evaluations 6
3.3 Participants' Comments 7
4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS 8
4.1 Results Summary 8
4.2 Outliers and Extreme Outliers 8
4.3 Assigned Value 8
4.4 Robust Average and Robust Between-Laboratory Coefficient of Variation 8
4.5 Standard Deviation for Proficiency Assessment 9
4.6 z-Score 9
5 TABLES AND FIGURES 10
6 DISCUSSION OF RESULTS 76
6.1 Assigned Value 76
6.2 Measurement Uncertainty Reported by Participants 77
6.3 z-Score 77
6.4 E_n-Score 80
6.5 Summary of Participants' Results and Performances 81
6.6 Participants' Results and Analytical Methods for PFAS in Cardboard Packaging Material Sample S1 86
6.7 Participants' Results and Analytical Methods for Extractable Organic Fluorine in Cardboard 94
6.8 Participants' Results and Analytical Methods for Total Fluorine in Cardboard 96
6.9 Participants' Results and Analytical Methods for PFAS in Cosmetics (Foundation) 96
6.10 Participants' Results and Analytical Methods for Extractable Organic Fluorine in Cosmetics (Foundation) 99
6.11 Participants' Results and Analytical Methods for TF in Cosmetics (Foundation) 102
6.12 False Negatives 102
7 REFERENCES 103
APPENDIX 1 – SAMPLE PREPARATION, SAMPLE ANALYSIS and HOMOGENEITY ASSESSMENT, and STABILITY ASESSMENT 106

A1.1 Sample Preparation 106
A1.2 Sample Analysis and Homogeneity Assessment 106
A1.3 Stability Assessment 111
APPENDIX 2– ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, Z-SCORE, AND E_N-SCORE CALCULATIONS 117
A2.1 Robust Average and Associated Uncertainty 117
A2.2 z-Score and E_n-Score Calculations 117
APPENDIX 3 – ADDITIONAL ANALYTES 118
APPENDIX 4 – FALSE NEGATIVES 119
APPENDIX 5 – ASSESSMENT OF RESULTS COMPATIBILITY FOR PFAS IN PILOT SAMPLE S4 120
APPENDIX 6 – PARTICIPANTS' TEST METHODS 124
A6.1 Participants' Test Methods for Total Fluorine 124
A6.2 Participants' Test Methods for Extractable Organic Fluorine 126
A6.3 Participants' Test Methods for PFAS in Sample S1 128
A6.4 Participants' Test Methods for PFAS in Pilot Sample S4 136
APPENDIX 7 – ACRONYMS AND ABBREVIATIONS 141

1 INTRODUCTION

1.1 NMIA Proficiency Testing Program

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

Proficiency testing (PT) is the: 'evaluation of participant performance against pre-established criteria by means of interlaboratory comparison.'[1] NMIA PT studies target chemical testing in areas of high public significance such as trade, environment, law enforcement, and food safety. NMIA offers studies in:

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

1.2 Study Background

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

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

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

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

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

PFAS in cosmetics is also an emerging global concern, with an increasing number of countries starting to investigate potential health risks. Although the United States does not have a federal ban on PFAS in cosmetics, California was the first state to prohibit from 1 January 2025 the manufacture, sale, delivery, holding, or offering for sale of cosmetic products containing intentionally added PFAS.[6] Other states are also introducing similar restrictions.[7]

The EU does not have a specific regulation for cosmetics, but rather a proposal under its Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) regulation. The proposal was raised by authorities in Denmark, Germany, the Netherlands, Norway, and Sweden. It focuses on restricting the entire group of PFAS substances to avoid one PFAS being replaced by another.[8] The Risk Assessment Committee of the European Chemicals Agency (ECHA) adopted their opinion in March 2026, and the proposal is now being reviewed by the Socio-Economic Analysis Committee of the ECHA. Their opinion is to be published in 2026 and if adopted, EU member states will vote as to whether the legislation will enter into force.[8,9]

Regardless of the outcome, France has already enacted a national law banning the manufacture, import, export, and sale of cosmetic products containing intentionally added PFAS which came into force on 1 January 2026.[10] Japan and New Zealand have also implemented their own bans for PFAS in cosmetics.[11, 12] Currently, Australia has no legislation specifically aimed at PFAS in cosmetics, but the import, export, and manufacture is limited under IChEMS.[5]

1.3 Study Aims

The aims of the study were to:

i. compare laboratories' performance and assess their capabilities to measure PFAS, and TF in packaging material;
ii. evaluate laboratories' methods used in determination of PFAS, EOF, and TF in packaging material and cosmetics;
iii. develop the practical application of measurement uncertainty, and provide participants with information that will be useful in assessing their measurement uncertainty evaluations; and
iv. produce materials that can be used in method validation and as control samples.

1.4 Study Conduct

The conduct of NMIA PT studies is described in the NMIA Study Protocol for Proficiency Testing.[13] The statistical methods used are described in the NMIA Chemical Proficiency Testing Statistical Manual.[14] These documents have been prepared with reference to ISO/IEC 17043[1] and The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.[15]

NMIA is accredited by the National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043 as a provider of proficiency testing schemes.[1] This study falls within the scope of NMIA's accreditation (Accreditation No. 198; Site No. 14485).

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of the study was:

Invitations sent	13 April 2026
Samples sent	1 June 2026
Results due	9 July 2026
Interim Report	16 July 2026
Preliminary Report	20 July 2026

2.2 Participation and Laboratory Code

Fourteen laboratories participated in this study, and all submitted results. A confidential laboratory code number was assigned to each of these 14 participants.

2.3 Selection of Analytes and Test Material Preparation

When selecting analytes and spiking values for this study, consideration was given to:

- current and emerging legislation;[2-12]
- feedback from participants and other stakeholders.

Four test samples were provided for analysis.

- Sample S1 consisted of two identical containers of 3 g cardboard packaging material each;
- Pilot Sample S2 was the same material used for preparation of Sample S1 and consisted of 3 g cardboard packaging material;
- Sample S3 consisted of 3 g cardboard packaging material; and
- Pilot Sample S4 consisted of 20 g cosmetic (foundation) material.

Details of the spiked analytes and levels for Sample S1 and pilot Samples S2 and S4 are presented in Table 1 and sample preparation details for all samples are in Appendix 1.

Table 1 Spiked Values of Test Samples

Analyte	Sample S1 and Pilot Sample S2 Spiked Values (µg/kg)	Pilot Sample S4 Spiked Values (µg/kg)
TFA	10000	9390
PFBA	20.2	96.2
PFPeA	5.19	Not spiked
PFHxA	8.44	224
PFOA	1.01	1.93
PFDA	15.6	Not spiked
PFUdA	15.2	Not spiked
PFDoA	15.2	38.5
PFTrDA	15.2	Not spiked
PFTeDA	15.2	Not spiked
PFBS*	3.04	4.85
PFHxS*	10.2	14.6
PFOS*	15.2	14.4
N-EtFOSAA	20.2	Not spiked
6:2FTS*	46.6	38.6
8:2FTS*	47.0	Not spiked
8:2diPAP*	20.3	72.2
GenX	20.2	Not spiked
ADONA*	15.2	Not spiked
Extractable Organic Fluorine**	5200	5020

*Values for these analytes are the anion concentration. **EOF spiked values were calculated as the sum of the fluorine component of all analytes spiked into each sample.

2.4 Test Material Homogeneity and Stability Testing

The same validated preparation procedure was followed for Samples S1, S3, and pilot Sample S2 as in previous studies.[13] Test samples from previous studies were demonstrated to be sufficiently homogenous for the evaluation of participants' performance. Therefore, no homogeneity or stability testing was conducted for these samples.

As this is the first time that cosmetics (foundation) have been introduced in a PT study, a full homogeneity and stability study was conducted for this sample except for EOF and TF. The stability and homogeneity of these tests was assumed based on stability of individual PFAS compounds.

Further details on the homogeneity and stability assessment are given in Appendix 1.

2.5 Sample Storage, Dispatch, and Receipt

Before dispatch, Sample S1 and pilot Samples S2 and S4 were stored refrigerated at 4ºC, whilst Sample S3 was stored at room temperature in a dry location. The samples were sent by courier on 1 June 2026. 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

The instructions provided to participants were as follows:

- Quantitatively analyse the samples using your routine test method and report results:
  - on as received basis in units of µg/kg for PFAS in Sample S1,
  - on as received basis in units of µg/kg for EOF in pilot Sample S2,
  - on as received basis units of mg/kg for TF in Sample S3, and
  - on as received basis in units of µg/kg for PFAS and EOF and in units of mg/kg for TF in pilot Sample S4.
- The PFAS analyte concentration range in Sample S1 is 0.5-75 µg/kg and in pilot Sample S4 is 0.5-400 µg/kg. The concentration range for EOF in pilot Sample S2 is >1000 µg/kg and in pilot Sample S4 is >500 µg/kg. The concentration range of TF in Sample S3 is >25 mg/kg and in pilot Sample S4 is >15 mg/kg.
- If samples cannot be analysed on the day of receipt, please store Sample S1 and pilot Samples S2 and S4 refrigerated and Sample S3 covered at room temperature in a dry location.
- For Sample S1 and pilot Sample S2, participants will be asked to use the entire sample and to rinse the sample container with the extraction reagent. For the calculation of results, the sample mass of Sample S1 and pilot sample S2 should be assumed to be 3.00 g. Two identical containers have been provided for Sample S1 so that laboratories can repeat the measurement if required. For pilot Sample S4, participants are asked to mix the sample well before subsampling.
- For Sample S1 and pilot Sample S4, PFAS analytes that may be present in the samples are given in the table below.

Possible PFAS (total) analytes in AQA 26-21 Sample S1 and Pilot Sample S4					
PFBA	PFTrDA	PFOS*	N-MeFOSAA	8:2diPAP*	PFMPA
PFPeA	PFTeDA	PFNS*	N-EtFOSAA	6:2FTOH	PFMBA
PFHxA	PFHxDA	PFDS*	N-MeFOSE	8:2FTOH	NFDHA
PFHpA	PFODA	PFUdS*	N-EtFOSE	10:2FTOH	9Cl-PF3ONS*
PFOA	FOUEA	PFDoS*	4:2FTS*	3:3FTCA	11Cl-PF3OUdS*
PFNA	PFBS*	PFTrDS*	6:2FTS*	5:3FTCA	PFEESA*
PFDA	PFPeS*	PFOSA	8:2FTS*	7:3FTCA	PFECHS*
PFUdA	PFHxS*	N-MeFOSA	10:2FTS*	GenX	
PFDoA	PFHpS*	N-EtFOSA	6:2diPAP*	ADONA*	

* Participants will be asked to submit data calculated on the anion-basis, not calculated on the salt basis (e.g. 8:2FTS anion + counter ion).

- Please report total PFAS: e.g. for analytes that contain linear and branched isomers you will be requested to report the sum of linear and branched isomers. Participants may elect not to test for all listed analytes, and not all analytes may be present in the sample.
- Report results as you would report to a client (i.e. with the number of significant figures you would normally report, corrected for recovery or not according to your standard procedure) and apply the limit of reporting of the method used for analysis.
- For each analyte report the associated expanded measurement uncertainty as µg/kg for PFAS and EOF in Sample S1 and pilot Samples S2 and S4 or as mg/kg for TF in Sample S3 and pilot Sample S4 e.g., 0.532 ± 0.021 µg/kg or 0.532 ± 0.021 mg/kg.
- Report any listed analyte not tested as NT.
- Please complete the method details and report the basis of your uncertainty evaluations as required by the results sheet.
- If determined, report your internal standard percentage recovery. This will be presented in the report for information only.
- Please return the completed results sheet by email to proficiency@measurement.gov.au by 26 June 2026. Late results may not be accepted.

The results due date was later extended to 9 July 2026 for all participants to allow laboratories sufficient time to develop their methods for PFAS measurements in the cosmetics (foundation) matrix

2.7 Interim Report and Preliminary Report

An Interim Report was emailed to participants on 16 July 2026.

A Preliminary Report was emailed to participants on 20 July 2026. This report included: a summary of results reported by all laboratories, assigned values, performance coefficients of variation, z-scores and E_n-scores for each analyte tested by participants.

No data from the Preliminary Report, including all scoring, has changed in the present Final Report except for pilot Sample S4.

Laboratory 2 submitted results for pilot Sample S4 after the release of the Preliminary Report. These results were still included in this Final Report, as the purpose of pilot Sample S4 was to support method development and not to assess performance. The statistics for the analytes in this sample have been updated accordingly (Tables 26 to 37).

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Methods Reported by Participants

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

3.2 Basis of Participants' Measurement Uncertainty Evaluations

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

Table 2 Basis of Participants' Uncertainty Evaluations

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
1	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - RM Duplicate analysis	CRM Laboratory bias from PT studies Recoveries of SS	Nordtest Report TR537
2	Standard deviation of replicate analyses multiplied by 2 or 3 k = 2	Duplicate analysis Instrument calibration	Recoveries of SS	
3	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - RM Duplicate analysis Instrument calibration	Instrument calibration	Eurachem/CITAC Guide
4	Coverage factor not reported			
5	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis Instrument calibration	CRM	Eurachem/CITAC Guide
6	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control samples - SS Duplicate analysis Instrument calibration	Recoveries of SS	Eurachem/CITAC Guide
7	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis	Recoveries of SS	Nordtest Report TR537
8	Top Down - precision and evaluations of the method and laboratory bias k = 2	Control samples - SS	Recoveries of SS	NATA-Estimating and reporting MU of chemical test results
9**	Top Down - precision and evaluations of the method and laboratory bias Coverage factor not reported	Control samples - SS Duplicate analysis Instrument calibration	Instrument calibration Laboratory bias from PT studies Recoveries of SS Standard purity	EOF: In-house validation document; TF: Quantitative Chemical Analysis, 5th ed. D.C. Harris. 1997 Freeman & Co. Ch 4.
10	Bottom Up (ISO/GUM, fish bone/cause and effect diagram) k = 2	Control samples - SS	Recoveries of SS	ISO/GUM
11	Top Down - precision and evaluations of the method and laboratory bias k = 2	Duplicate analysis Instrument calibration	Recoveries of SS	ISO/GUM

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
12	Professional judgment k = 2	Control samples - SS Duplicate analysis	Laboratory bias from PT studies Recoveries of SS	Overview of the Guidance Documents developed and published by the network of the European Union Reference Laboratory (EURL) and the National Reference Laboratories (NRLs) for halogenated persistent organic pollutants (POPs) in Feed and Food and the cluster of the four EURLs working in the field of contaminants.
13**	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control samples - SS		In-house Establishing Control Limits SOP that is based on ISO17025
14	Coverage factor not reported			

*SS = Spiked Samples, RM = Reference Material, CRM = Certified Reference Material. **Additional Information in Table 3.

Table 3 Uncertainty Evaluation Additional Information

Lab Code	Approach to Evaluating MU
9	S2: Uncertainty calculated by In-House standard validation spreadsheet, n=1 S3: Uncertainty calculated from text reference. n=6. DF=5, s=standard deviation, double tailed student's t value = 2.571 S4: EOF: n=3; TF: n=3
13	All Samples: Uncertainty is in %

3.3 Participants' Comments

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

Table 4 Participants' Comments

Lab Code	Participants' Comments	Study Coordinator's Response
6	S1: Using 3g of sample is a lot! We had to modify our normal method extraction solvent volume to accommodate this. We usually use 1g for extractions so would be good to have this option rather than using the whole container.	Thank you for your feedback. We will consider adjusting the sample size in future studies. If other participants have a preferred sample size, we would welcome their feedback as well.

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 5 to 37 with resultant summary statistics. Bar charts of results and performance scores are presented in Figures 2 to 34. An example chart with interpretation guide is shown in Figure 1.

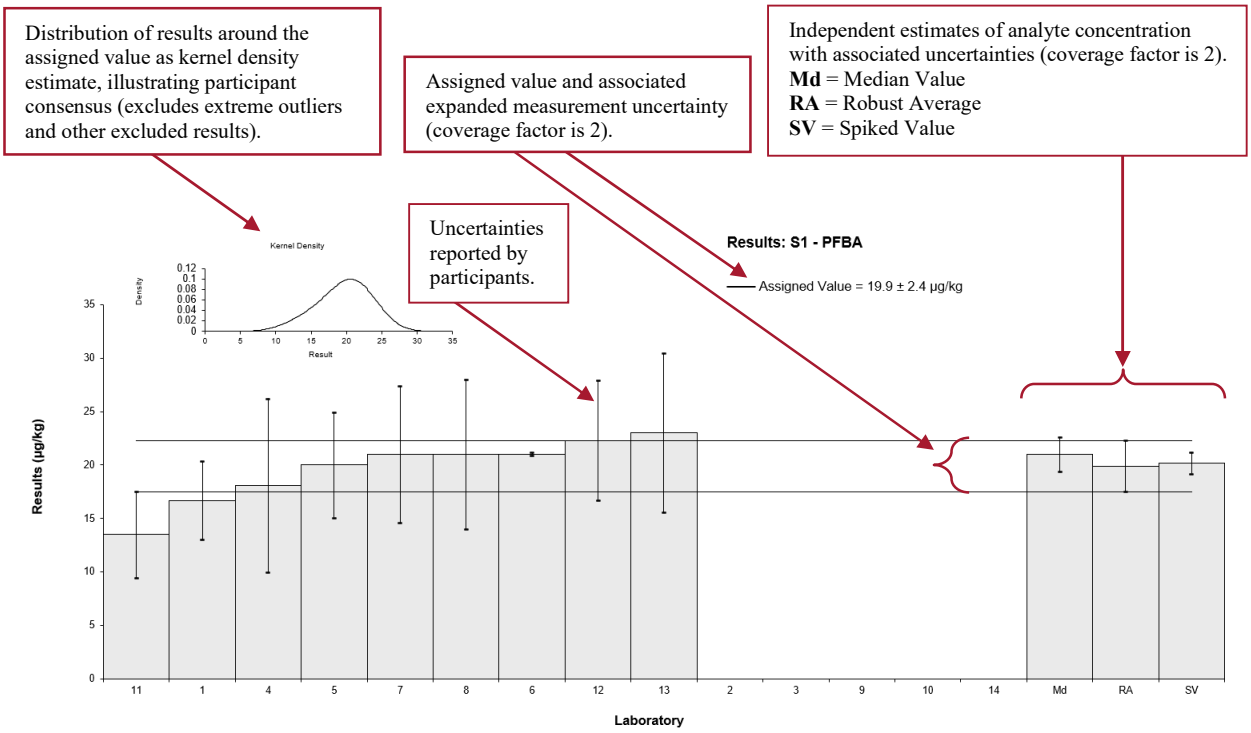


Figure 1 Guide to Presentation of Results

4.2 Outliers and Extreme Outliers

Outliers were results less than 50% and greater than 150% of the robust average and were removed before assigned value calculation. Extreme outliers (gross errors) were obvious blunders, such as those with incorrect units, decimal placement errors, or results from a different PT item, and were removed before the calculation of summary statistics.[15, 16]

4.3 Assigned Value

The assigned value is defined as the 'value attributed to a particular property or characteristic of a proficiency testing item'.[1] In this study the property is the mass fraction of analyte. Assigned values were the robust average of participants' results (after the removal of outliers) and the expanded uncertainties were evaluated from the associated robust standard deviations.[15, 16] An example of the assigned value calculation using data from the present study is given in Appendix 2.

4.4 Robust Average and Robust Between-Laboratory Coefficient of Variation

The robust averages and associated expanded measurement uncertainties, and robust CVs (a measure of the variability of participants' results) were calculated using the procedure described in ISO 13528.[16] The robust CVs as presented in the Section 5 Statistics Tables are the robust between-laboratory CVs including outliers but excluding extreme outliers. Everywhere else in the report, the robust between-laboratory CV refers to the robust CV calculated with both outliers and extreme outliers removed.

4.5 Standard Deviation for Proficiency Assessment

The standard deviation for proficiency assessment (SDPA, σ) is the product of the assigned value *(X)* and the performance coefficient of variation (PCV). This value is used for calculation of participant z-scores and provides scaling for laboratory deviation from the assigned value.

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

It is important to note that the PCV is a set value and is not the standard deviation of participants' results. The fixed value set for PCV is based on the existing regulation, the acceptance criteria indicated by the methods, the matrix, the concentration level of analyte, and on experience from previous studies. It is backed up by mathematical models such as the Thompson/Horwitz equation.[17]

4.6 z-Score

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

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

where:

z is z-score;
χ is a participants' result;
X is the assigned value;
σ is the SDPA from Equation 1

For a z-score with the absolute value (|z|): $|z| \leq 2.0$ is acceptable; $2.0 < |z| < 3.0$ is questionable; and $|z| \geq 3.0$ is unacceptable.

4.7 E_n-Score

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

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

where:

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

For an E_n-score with the absolute value ($|E_n|$): $|E_n| < 1.0$ is acceptable; and $|E_n| \geq 1.0$ is unacceptable.

4.8 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC 17025 must establish and demonstrate the traceability and measurement uncertainty associated with their test results.[18] Guidelines for quantifying uncertainty in analytical measurements are described in the Eurachem/CITAC Guide.[19]

5 TABLES AND FIGURES

Table 5

Sample Details

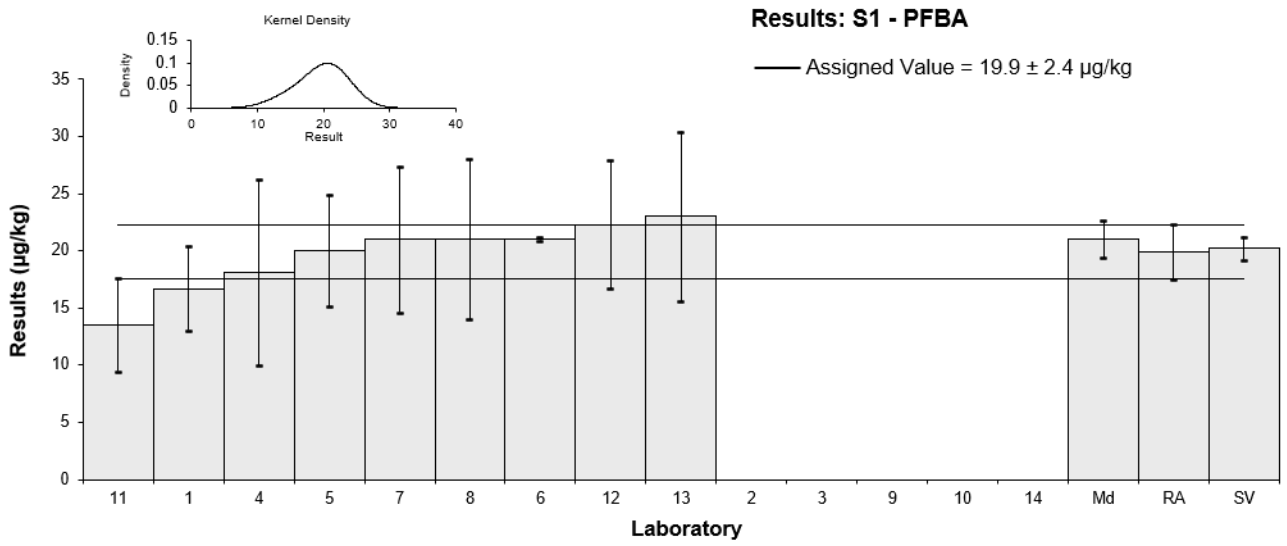
Sample No.	S1
Matrix	Cardboard
Analyte	PFBA
Unit	µg/kg

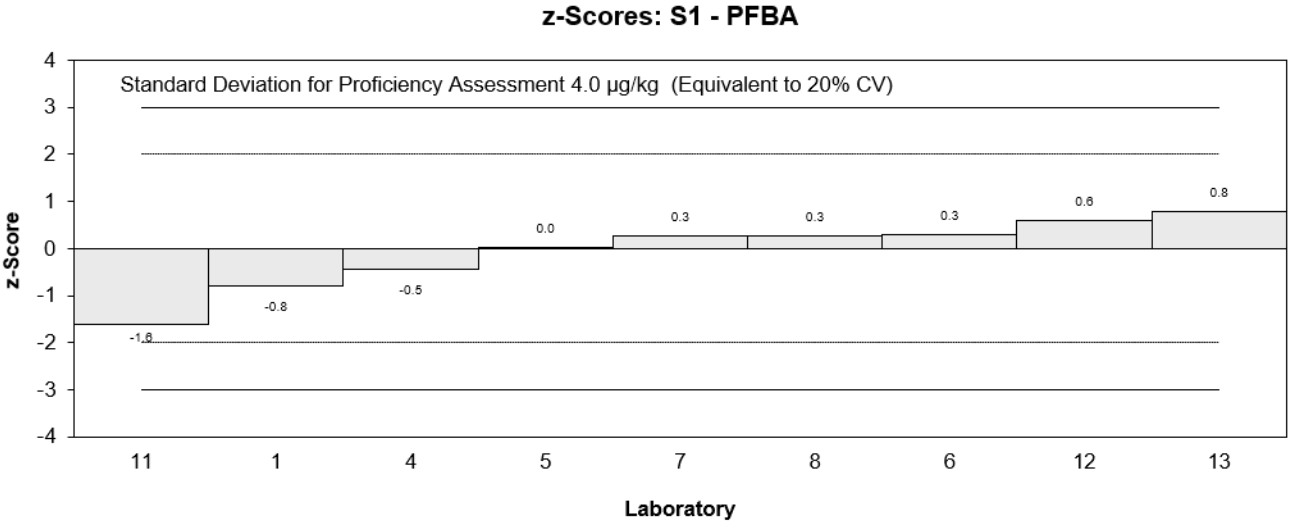
Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	16.7	3.66	47.1	-0.80	-0.73
2	NT	NT	NT		
3	NT	NT	NT		
4	18.1	8.14	NR	-0.45	-0.21
5	20	4.9	118	0.03	0.02
6	21.04	0.135	69	0.29	0.47
7	21	6.4	NR	0.28	0.16
8	21	7	92	0.28	0.15
9	NS	NS	NS		
10	NT	NT	NT		
11	13.5	4.06	59	-1.61	-1.36
12	22.3	5.6	47	0.60	0.39
13	23	7.429	59.7	0.78	0.40
14	NS	NS	NS		

Statistics

Assigned Value	19.9	2.4
Spiked Value	20.2	1.0
Robust Average	19.9	2.4
Median	21.0	1.6
Mean	19.6	
N	9	
Max	23	
Min	13.5	
Robust SD	2.9	
Robust CV	15%	


Results: S1 - PFBA
Assigned Value = 19.9 ± 2.4 µg/kg
Kernel Density
Density
Result
Results (µg/kg)
Laboratory
11 1 4 5 7 8 6 12 13 2 3 9 10 14 Md RA SV


z-Scores: S1 - PFBA
Standard Deviation for Proficiency Assessment 4.0 µg/kg (Equivalent to 20% CV)
z-Score
-1.6
-0.8
-0.5
0.0
0.3
0.3
0.3
0.6
0.8
Laboratory
11 1 4 5 7 8 6 12 13

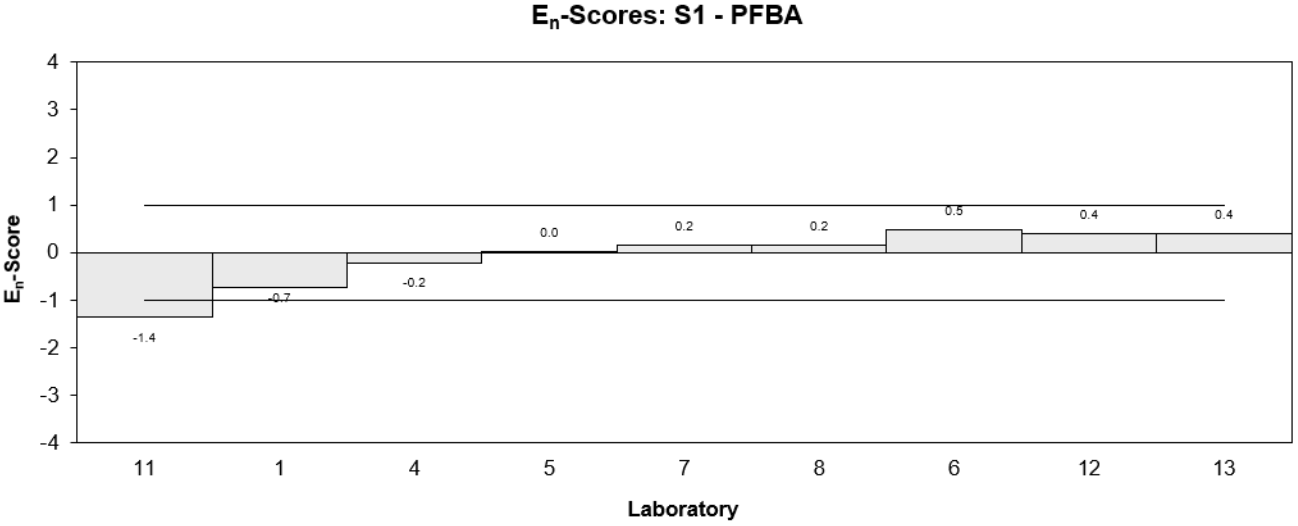

En-Scores: S1 - PFBA
En-Score
-1.4
-0.7
-0.2
0.0
0.2
0.2
0.5
0.4
0.4
Laboratory
11 1 4 5 7 8 6 12 13

Figure 2

Table 6

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	PFPeA
Unit	µg/kg

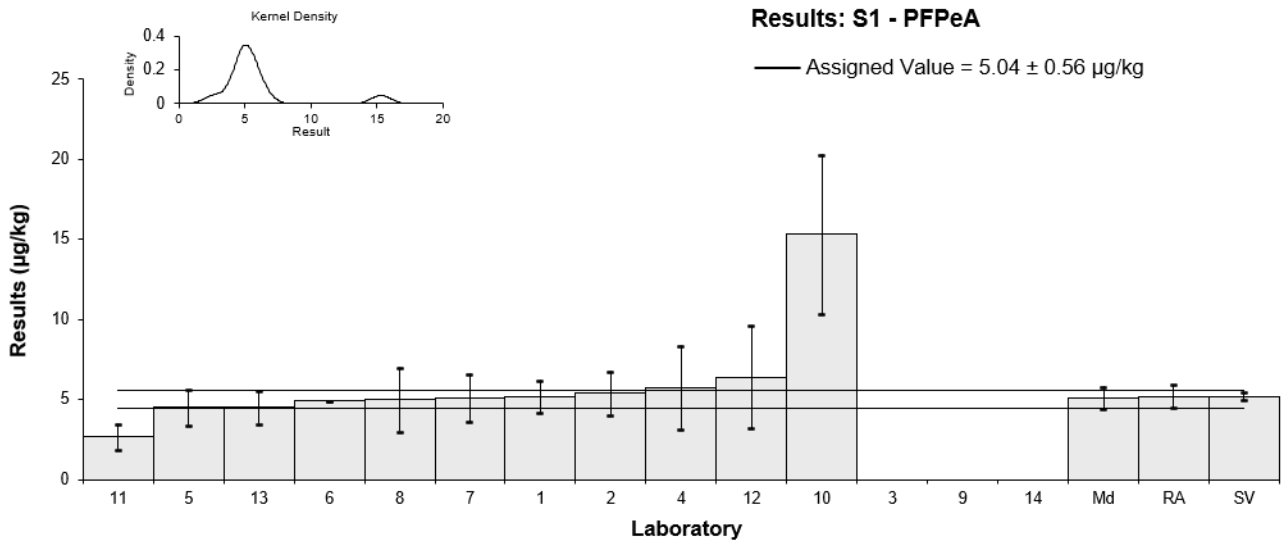
Participant Results

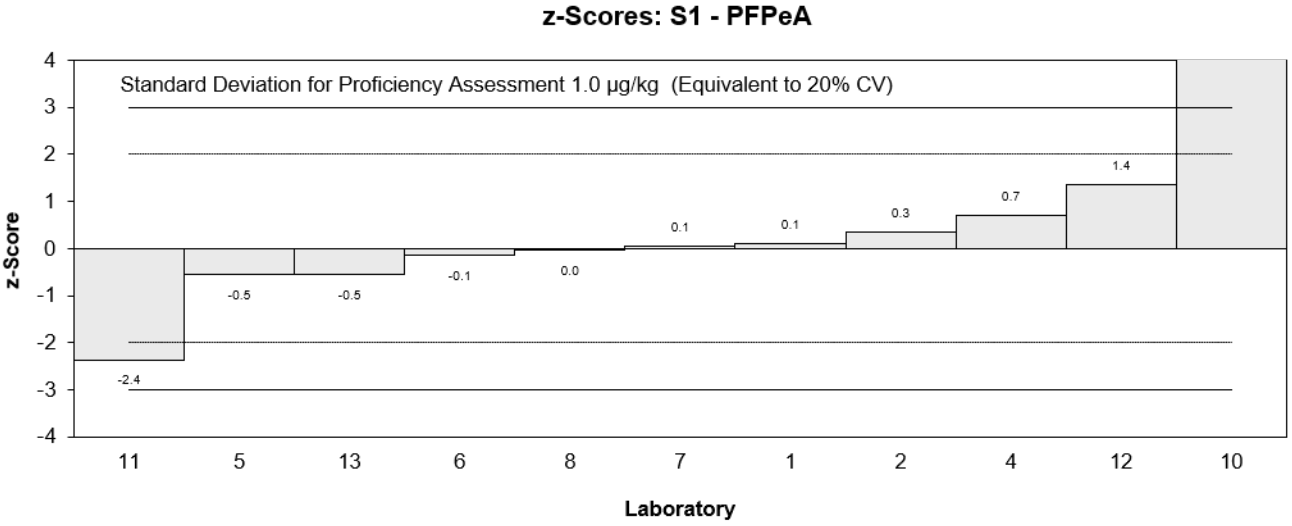
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	5.15	1.02	66	0.11	0.09
2	5.38	1.35	NR	0.34	0.23
3	NT	NT	NT		
4	5.75	2.587	NR	0.70	0.27
5	4.5	1.1	114	-0.54	-0.44
6	4.9	0.01	64	-0.14	-0.25
7	5.1	1.5	NR	0.06	0.04
8	5	2	98	-0.04	-0.02
9	NS	NS	NS		
10*	15.3	5.0	70	10.18	2.04
11	2.66	0.798	69	-2.36	-2.44
12	6.4	3.2	102	1.35	0.42
13	4.5	1.0215	76.9	-0.54	-0.46
14	NS	NS	NS		

* Outlier, see Section 4.2

Statistics

Assigned Value	5.04	0.56
Spiked Value	5.19	0.26
Robust Average	5.19	0.71
Median	5.10	0.67
Mean	5.9	
N	11	
Max	15.3	
Min	2.66	
Robust SD	0.94	
Robust CV	18%	


Kernel Density
Density
Result
Results: S1 - PFPeA
Assigned Value = 5.04 ± 0.56 µg/kg
Results (µg/kg)
Laboratory
11 5 13 6 8 7 1 2 4 12 10 3 9 14 Md RA SV


z-Scores: S1 - PFPeA
Standard Deviation for Proficiency Assessment 1.0 µg/kg (Equivalent to 20% CV)
z-Score
-2.4 -0.5 -0.5 -0.1 0.0 0.1 0.1 0.3 0.7 1.4
Laboratory
11 5 13 6 8 7 1 2 4 12 10

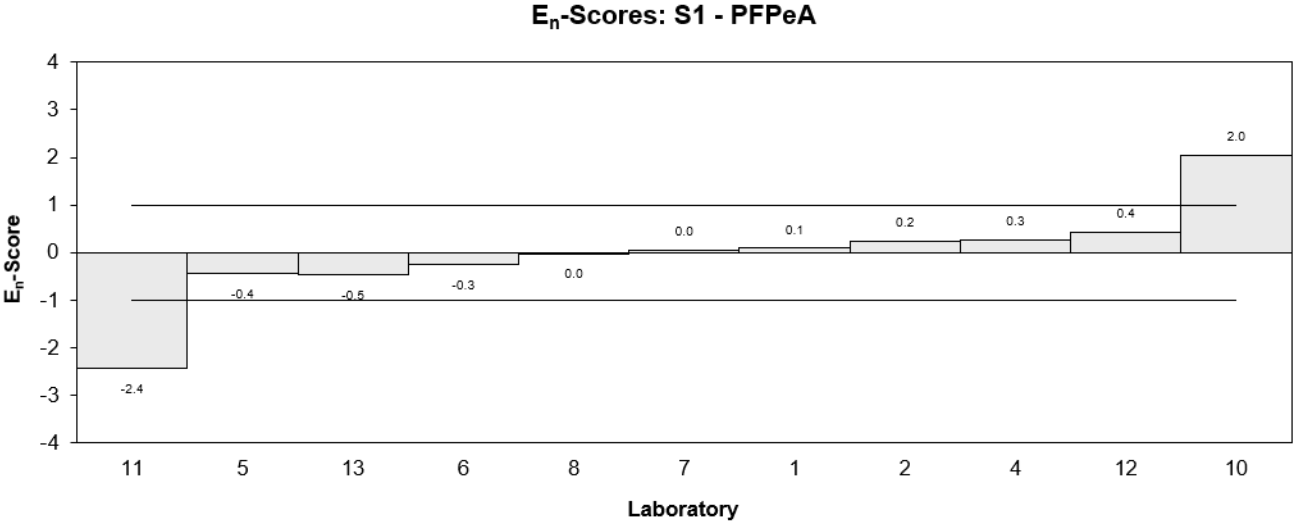

En-Scores: S1 - PFPeA
En-Score
-2.4 -0.4 -0.5 -0.3 0.0 0.0 0.1 0.2 0.3 0.4 2.0
Laboratory
11 5 13 6 8 7 1 2 4 12 10

Figure 3

Table 7

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	PFHxA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	6.67	1.55	86.2	0.13	0.08
2	4.62	1.16	NR	-1.45	-1.08
3	NT	NT	NT		
4*	11.9	5.36	NR	4.15	0.98
5	7.3	1.8	108	0.62	0.36
6	8.59	0.125	67	1.61	1.60
7	7.1	2.1	NR	0.46	0.24
8	6.5	2	93	0.00	0.00
9	NS	NS	NS		
10*	21.5	7.5	75	11.54	1.97
11	3.98	1.19	88	-1.94	-1.43
12	7.1	1.8	89	0.46	0.27
13	6.7	1.6415	74.9	0.15	0.10
14	NS	NS	NS		

* Outlier, see Section 4.2

Statistics

Assigned Value	6.5	1.3
Spiked Value	8.44	0.42
Robust Average	7.4	2.0
Median	7.10	0.67
Mean	8.4	
N	11	
Max	21.5	
Min	3.98	
Robust SD	2.6	
Robust CV	35%	

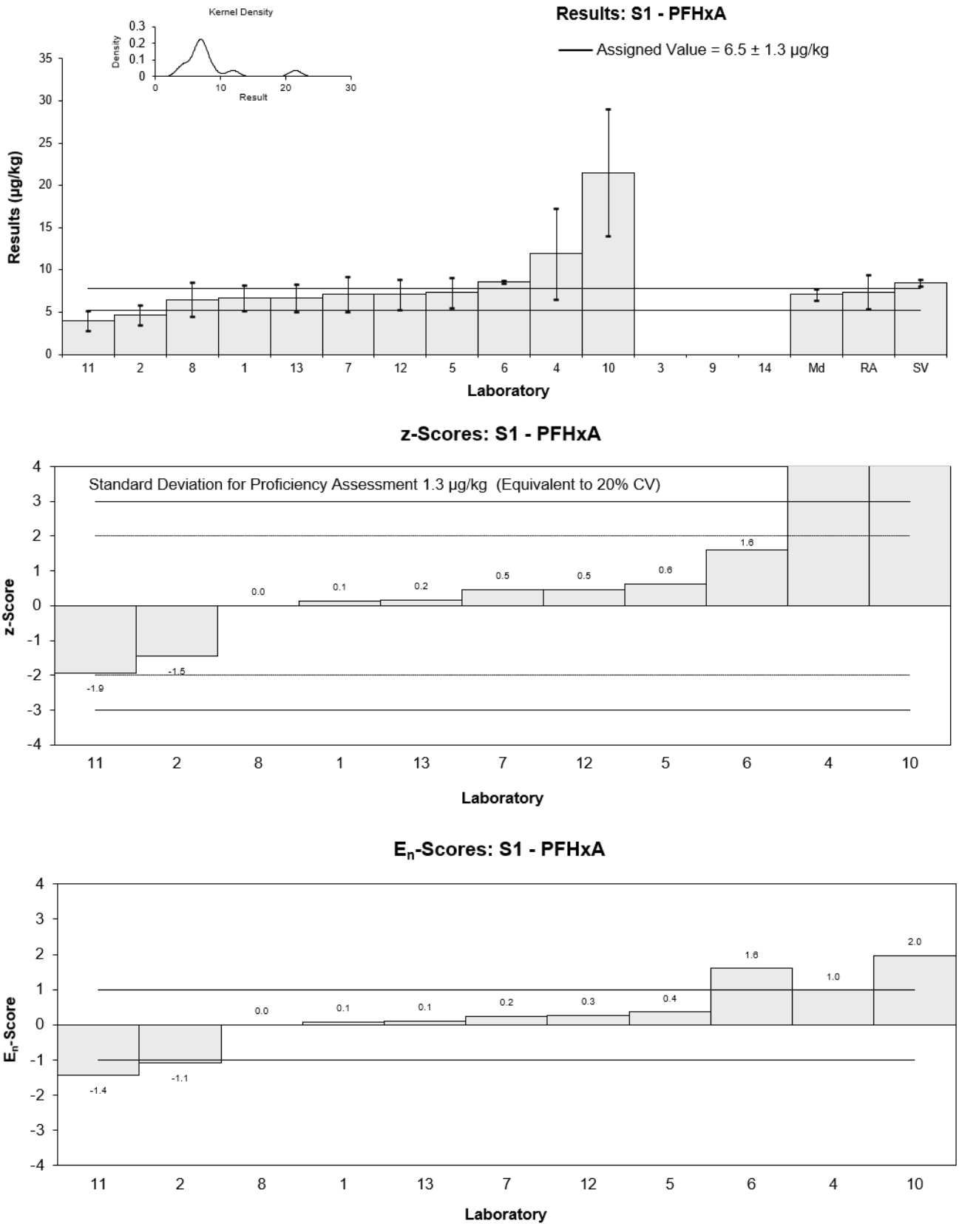

Kernel Density
Density
Result
Results: S1 - PFHxA
Assigned Value = 6.5 ± 1.3 µg/kg
Results (µg/kg)
Laboratory
11 2 8 1 13 7 12 5 6 4 10 3 9 14 Md RA SV
z-Scores: S1 - PFHxA
Standard Deviation for Proficiency Assessment 1.3 µg/kg (Equivalent to 20% CV)
z-Score
-1.9 -1.5 0.0 0.1 0.2 0.5 0.5 0.6 1.6
Laboratory
11 2 8 1 13 7 12 5 6 4 10
En-Scores: S1 - PFHxA
En-Score
-1.4 -1.1 0.0 0.1 0.1 0.2 0.3 0.4 1.6 1.0 2.0
Laboratory
11 2 8 1 13 7 12 5 6 4 10

Figure 4

Table 8

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	PFOA
Unit	µg/kg

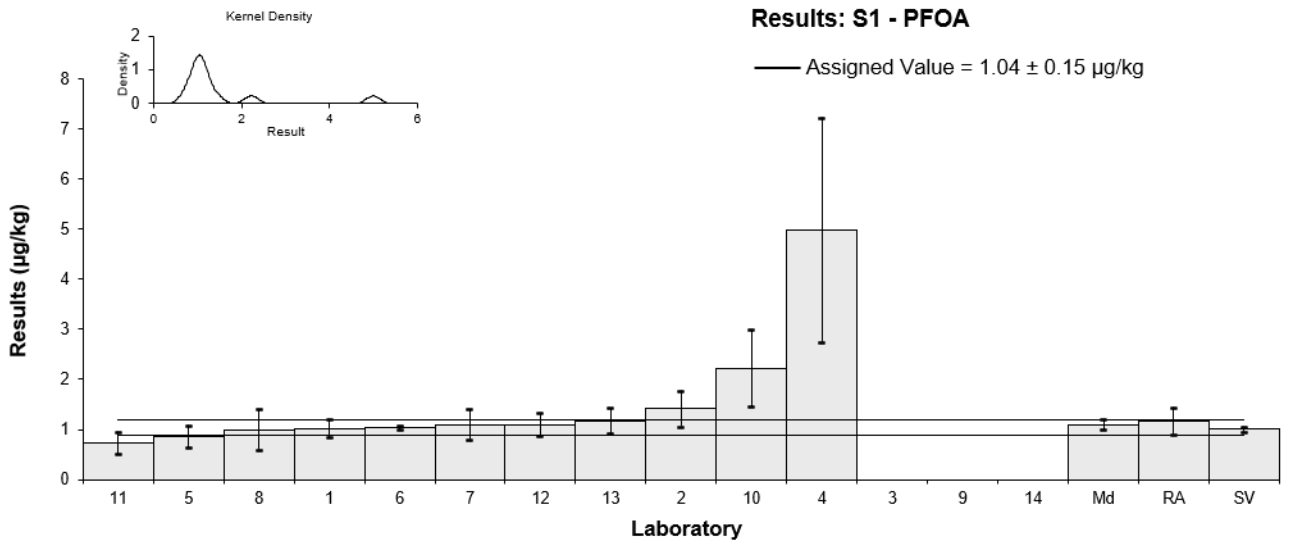
Participant Results

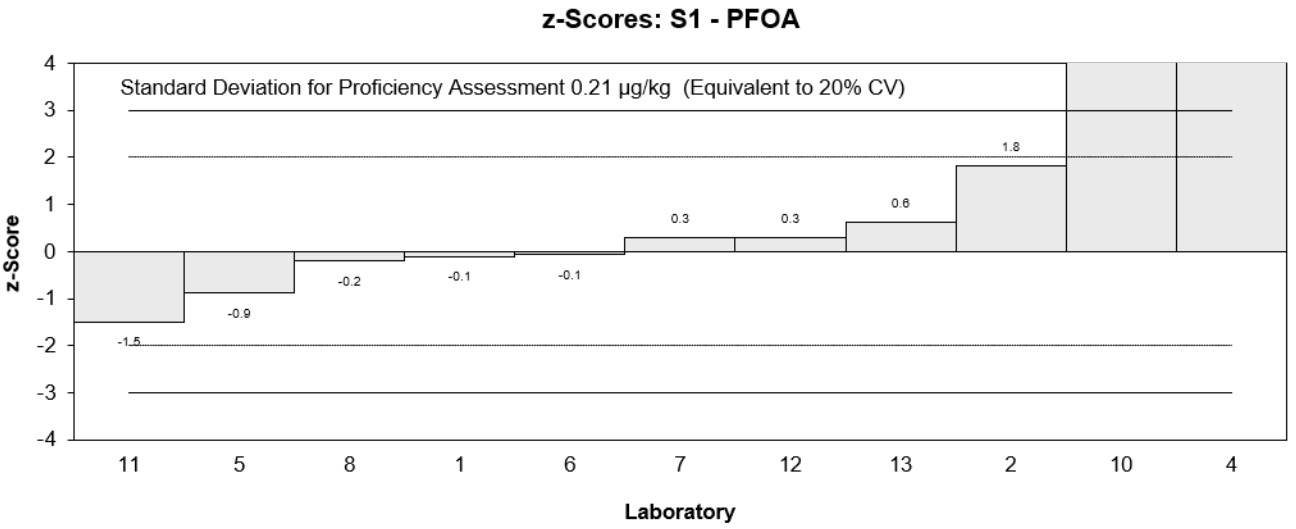
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	1.02	0.186	92	-0.10	-0.08
2	1.42	0.36	NR	1.83	0.97
3	NT	NT	NT		
4*	4.99	2.245	NR	18.99	1.76
5	0.86	0.22	132	-0.87	-0.68
6	1.03	0.04	56	-0.05	-0.06
7	1.1	0.3	NR	0.29	0.18
8	1	0.4	114	-0.19	-0.09
9	NS	NS	NS		
10*	2.22	0.77	75	5.67	1.50
11	0.731	0.22	100	-1.49	-1.16
12	1.1	0.23	90	0.29	0.22
13	1.17	0.25623	84.7	0.62	0.44
14	NS	NS	NS		

* Outlier, see Section 4.2

Statistics

Assigned Value	1.04	0.15
Spiked Value	1.01	0.05
Robust Average	1.17	0.27
Median	1.10	0.11
Mean	1.51	
N	11	
Max	4.99	
Min	0.731	
Robust SD	0.36	
Robust CV	31%	


Kernel Density
Density
Result
Results: S1 - PFOA
Assigned Value = 1.04 ± 0.15 µg/kg
Results (µg/kg)
Laboratory
11 5 8 1 6 7 12 13 2 10 4 3 9 14 Md RA SV


z-Scores: S1 - PFOA
Standard Deviation for Proficiency Assessment 0.21 µg/kg (Equivalent to 20% CV)
z-Score
-1.5
-0.9
-0.2
-0.1
-0.1
0.3
0.3
0.6
1.8
Laboratory
11 5 8 1 6 7 12 13 2 10 4

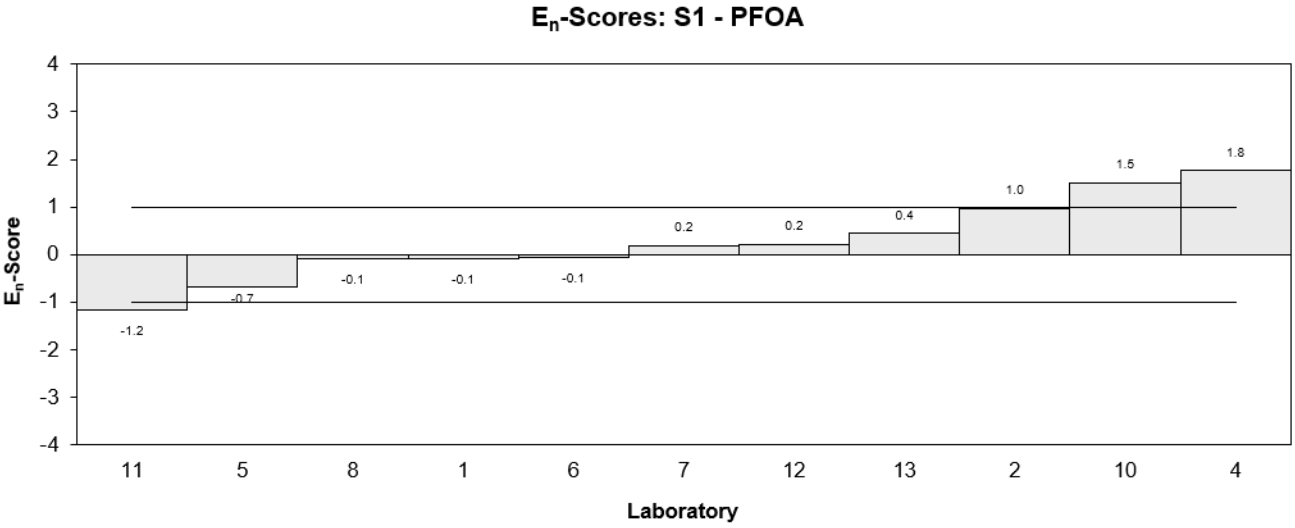

En-Scores: S1 - PFOA
En-Score
-1.2
-0.7
-0.1
-0.1
-0.1
0.2
0.2
0.4
1.0
1.5
1.8
Laboratory
11 5 8 1 6 7 12 13 2 10 4

Figure 5

Table 9

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	PFNA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	0.138	0.037	85.6
2	NT	NT	NT
3	NT	NT	NT
4	<0.50	NR	NR
5	<0.1	NR	NR
6	0.148	0.005	36
7	<1	0.3	NR
8	<0.5	NR	123
9	NS	NS	NS
10	NR	NR	72
11	< 0.1	NR	104
12	0.13	0.03	93
13	0.162	0.029646	77.4
14	NS	NS	NS

Statistics

Assigned Value	Not Set	
Spiked Value	Not Spiked	
Robust Average	NA (N<6)	
Median	0.143	0.017
Mean	0.145	
N	4	
Max	0.162	
Min	0.13	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

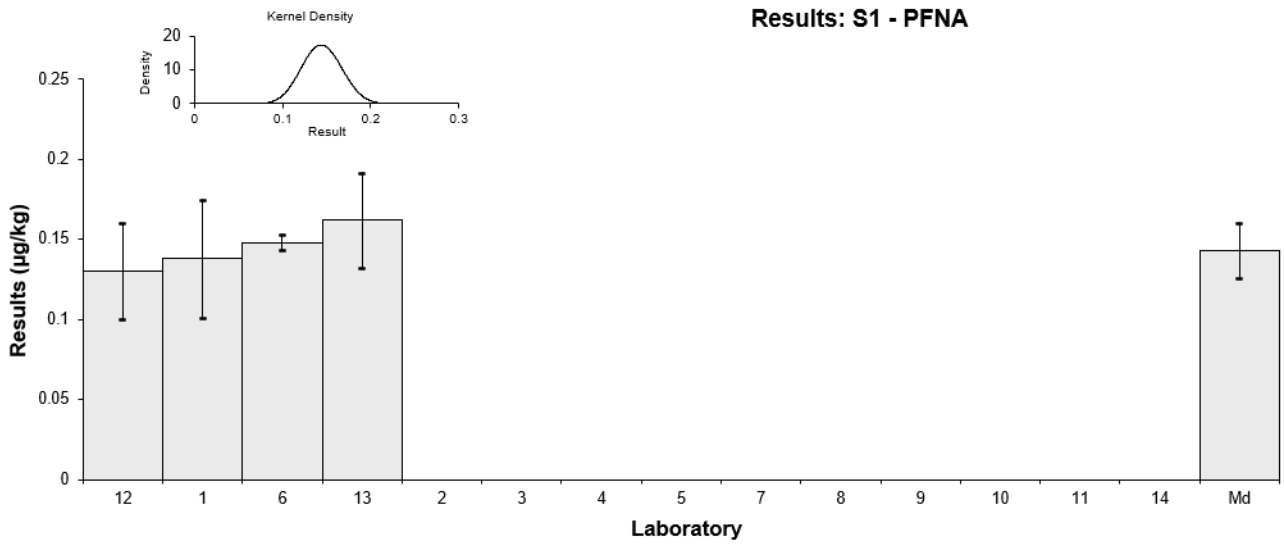

Results: S1 - PFNA
Kernel Density
Density
20
10
0
0
0.1
0.2
0.3
Result
Results (µg/kg)
0.25
0.2
0.15
0.1
0.05
0
12
1
6
13
2
3
4
5
7
8
9
10
11
14
Md
Laboratory

Figure 6

Table 10

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	PFDA
Unit	µg/kg

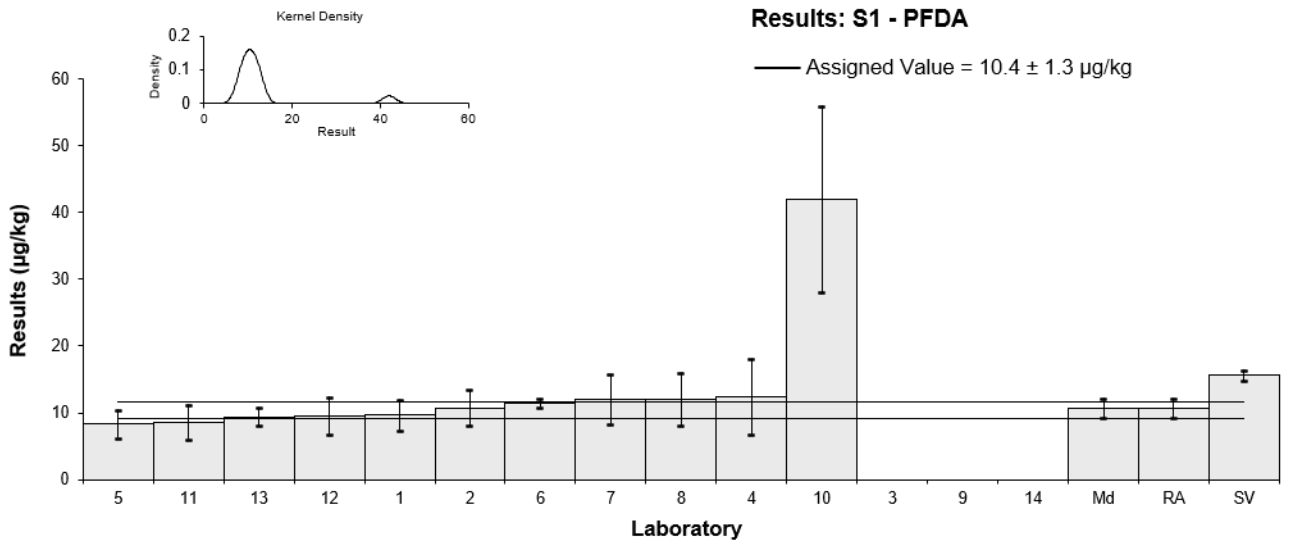
Participant Results

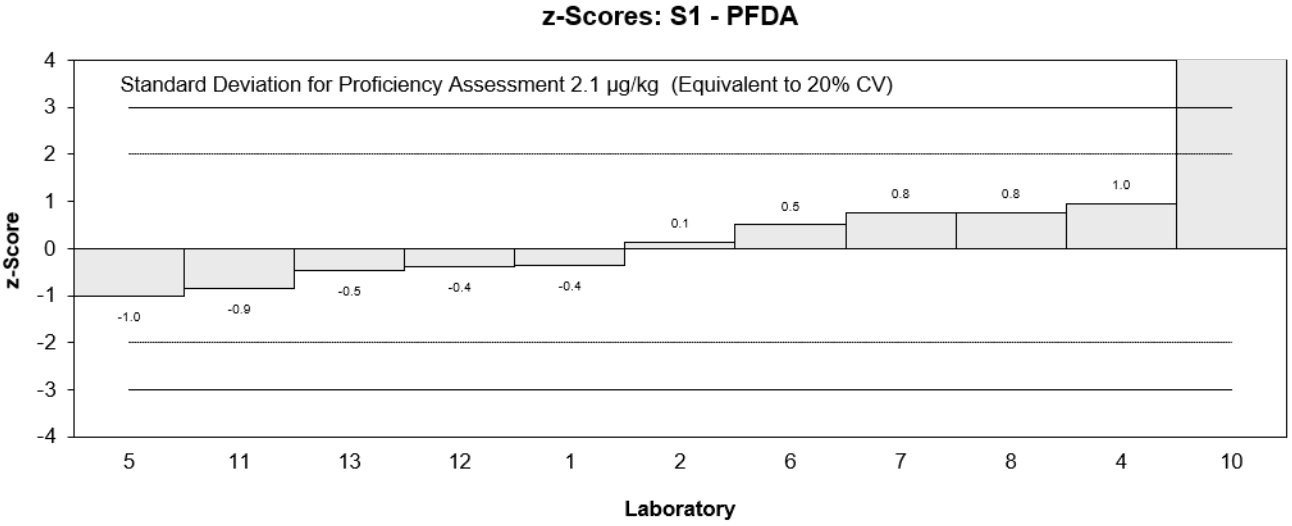
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	9.65	2.31	133	-0.36	-0.28
2	10.7	2.68	NR	0.14	0.10
3	NT	NT	NT		
4	12.4	5.58	NR	0.96	0.35
5	8.3	2.1	143	-1.01	-0.85
6	11.45	0.59	11	0.50	0.74
7	12	3.7	NR	0.77	0.41
8	12	4	116	0.77	0.38
9	NS	NS	NS		
10*	42	14	70	15.19	2.25
11	8.62	2.59	116	-0.86	-0.61
12	9.6	2.8	74	-0.38	-0.26
13	9.42	1.38474	72.6	-0.47	-0.52
14	NS	NS	NS		

* Outlier, see Section 4.2

Statistics

Assigned Value	10.4	1.3
Spiked Value	15.6	0.8
Robust Average	10.7	1.5
Median	10.7	1.5
Mean	13.3	
N	11	
Max	42	
Min	8.3	
Robust SD	1.9	
Robust CV	18%	


Kernel Density
Density
Result
Results: S1 - PFDA
Assigned Value = 10.4 ± 1.3 µg/kg
Results (µg/kg)
Laboratory
5 11 13 12 1 2 6 7 8 4 10 3 9 14 Md RA SV


z-Scores: S1 - PFDA
Standard Deviation for Proficiency Assessment 2.1 µg/kg (Equivalent to 20% CV)
z-Score
-1.0 -0.9 -0.5 -0.4 -0.4 0.1 0.5 0.8 0.8 1.0
Laboratory
5 11 13 12 1 2 6 7 8 4 10

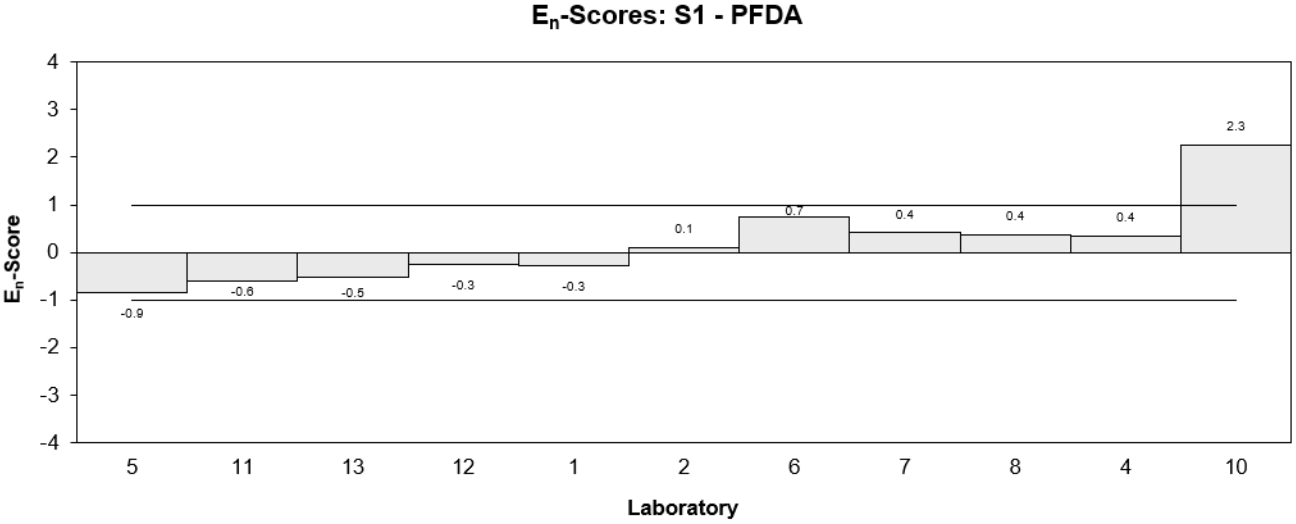

En-Scores: S1 - PFDA
En-Score
-0.9 -0.6 -0.5 -0.3 -0.3 0.1 0.7 0.4 0.4 0.4 2.3
Laboratory
5 11 13 12 1 2 6 7 8 4 10

Figure 7

Table 11

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	PFUdA
Unit	µg/kg

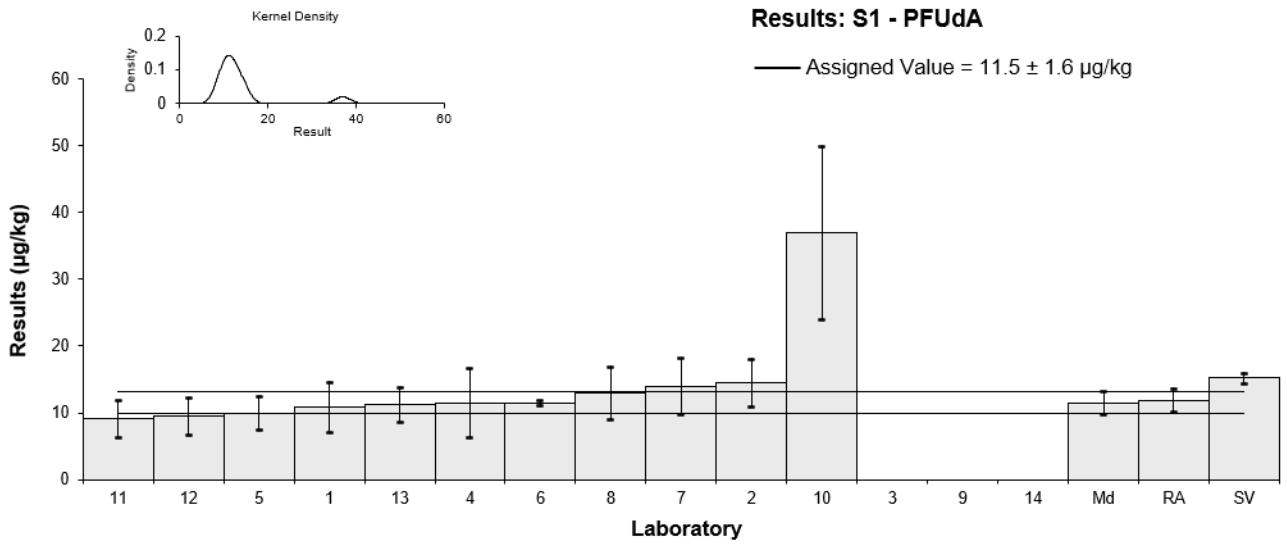
Participant Results

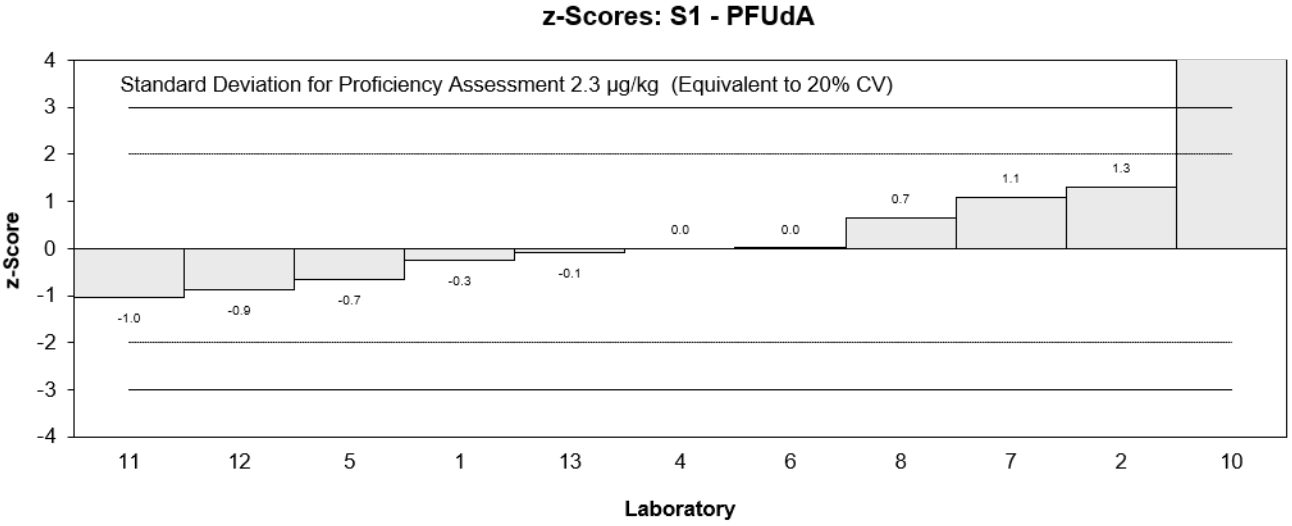
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	10.9	3.79	171	-0.26	-0.15
2	14.5	3.63	NR	1.30	0.76
3	NT	NT	NT		
4	11.5	5.17	NR	0.00	0.00
5	10	2.5	126	-0.65	-0.51
6	11.52	0.37	6	0.01	0.01
7	14	4.2	NR	1.09	0.56
8	13	4	138	0.65	0.35
9	NS	NS	NS		
10*	37	13	71	11.09	1.95
11	9.11	2.74	103	-1.04	-0.75
12	9.5	2.8	34	-0.87	-0.62
13	11.3	2.5764	53.8	-0.09	-0.07
14	NS	NS	NS		

* Outlier, see Section 4.2

Statistics

Assigned Value	11.5	1.6
Spiked Value	15.2	0.8
Robust Average	11.9	1.8
Median	11.5	1.7
Mean	13.8	
N	11	
Max	37	
Min	9.11	
Robust SD	2.4	
Robust CV	20%	


Kernel Density
Density
Result
Results: S1 - PFUdA
Assigned Value = 11.5 ± 1.6 µg/kg
Results (µg/kg)
Laboratory
11 12 5 1 13 4 6 8 7 2 10 3 9 14 Md RA SV


z-Scores: S1 - PFUdA
Standard Deviation for Proficiency Assessment 2.3 µg/kg (Equivalent to 20% CV)
z-Score
-1.0 -0.9 -0.7 -0.3 -0.1 0.0 0.0 0.7 1.1 1.3
Laboratory
11 12 5 1 13 4 6 8 7 2 10

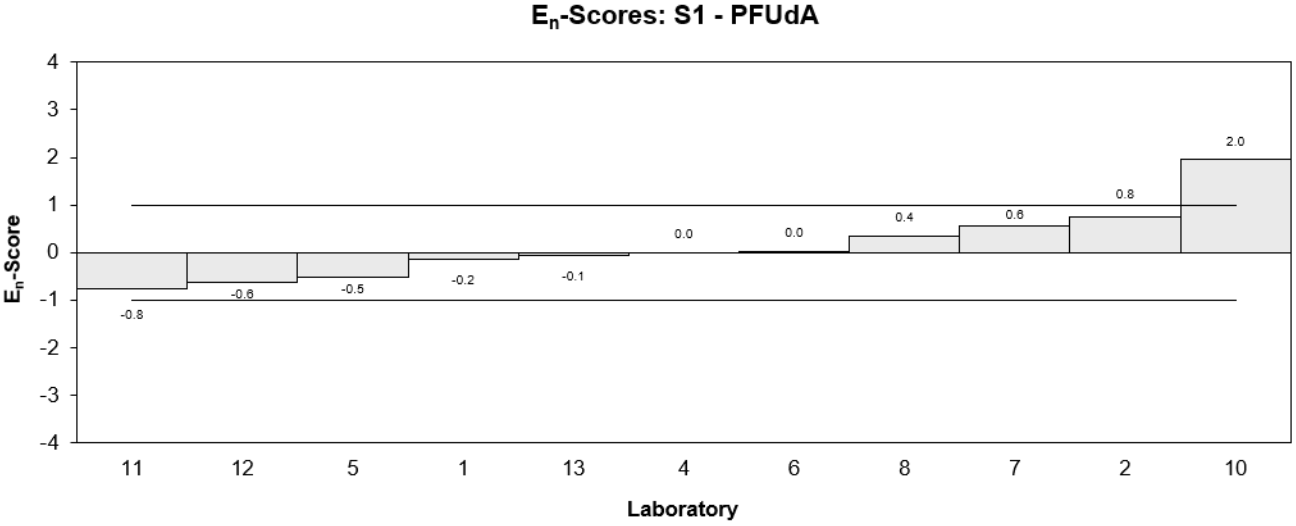

En-Scores: S1 - PFUdA
En-Score
-0.8 -0.6 -0.5 -0.2 -0.1 0.0 0.0 0.4 0.6 0.8 2.0
Laboratory
11 12 5 1 13 4 6 8 7 2 10

Figure 8

Table 12

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	PFDoA
Unit	µg/kg

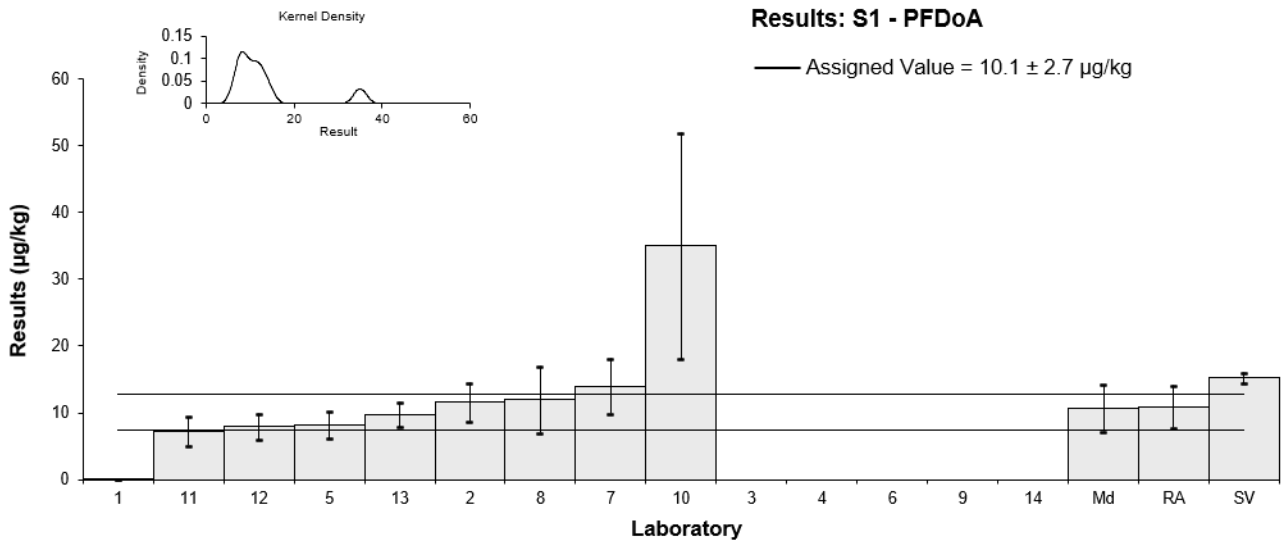
Participant Results

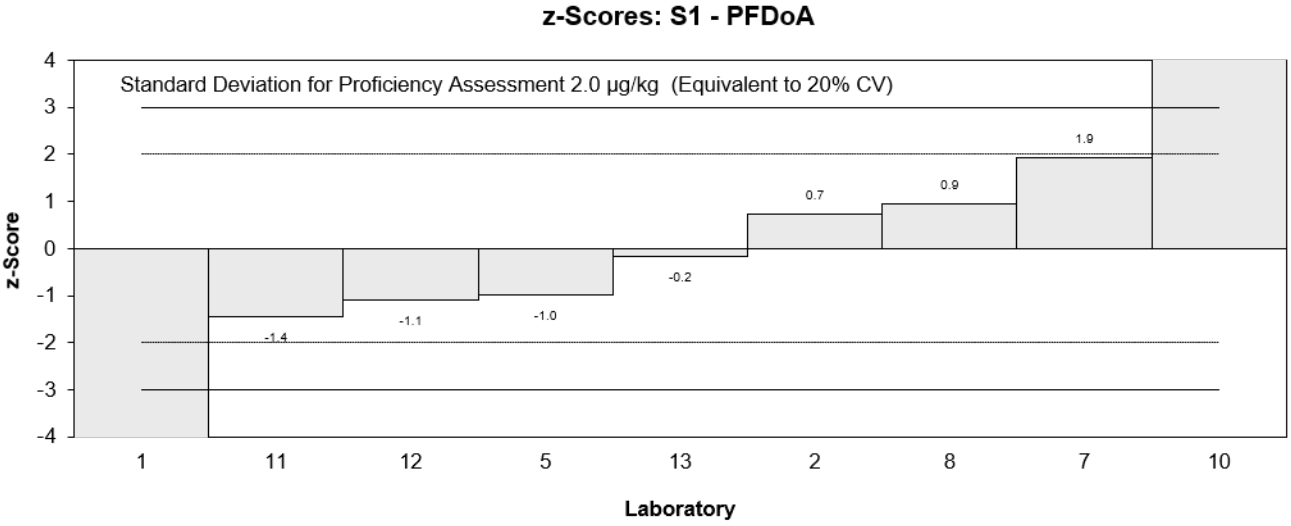
Lab. Code	Result	Uncertainty	Rec	z	E_n
1**	0.011	0.004	152	-4.99	-3.74
2	11.6	2.90	NR	0.74	0.38
3	NT	NT	NT		
4	<0.50	NR	NR		
5	8.1	2.0	131	-0.99	-0.60
6	NT	NT	NT		
7	14	4.1	NR	1.93	0.79
8	12	5	113	0.94	0.33
9	NS	NS	NS		
10*	35	17	72	12.33	1.45
11	7.19	2.16	67	-1.44	-0.84
12	7.9	2	31	-1.09	-0.65
13	9.74	1.87008	59.2	-0.18	-0.11
14	NS	NS	NS		

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	10.1	2.7
Spiked Value	15.2	0.8
Robust Average	10.9	3.2
Median	10.7	3.5
Mean	13.2	
N	8	
Max	35	
Min	7.19	
Robust SD	3.7	
Robust CV	34%	


Kernel Density
Density
Result
Results: S1 - PFDoA
Assigned Value = 10.1 ± 2.7 µg/kg
Results (µg/kg)
Laboratory
1
11
12
5
13
2
8
7
10
3
4
6
9
14
Md
RA
SV


z-Scores: S1 - PFDoA
Standard Deviation for Proficiency Assessment 2.0 µg/kg (Equivalent to 20% CV)
z-Score
Laboratory
-1.4
-1.1
-1.0
-0.2
0.7
0.9
1.9

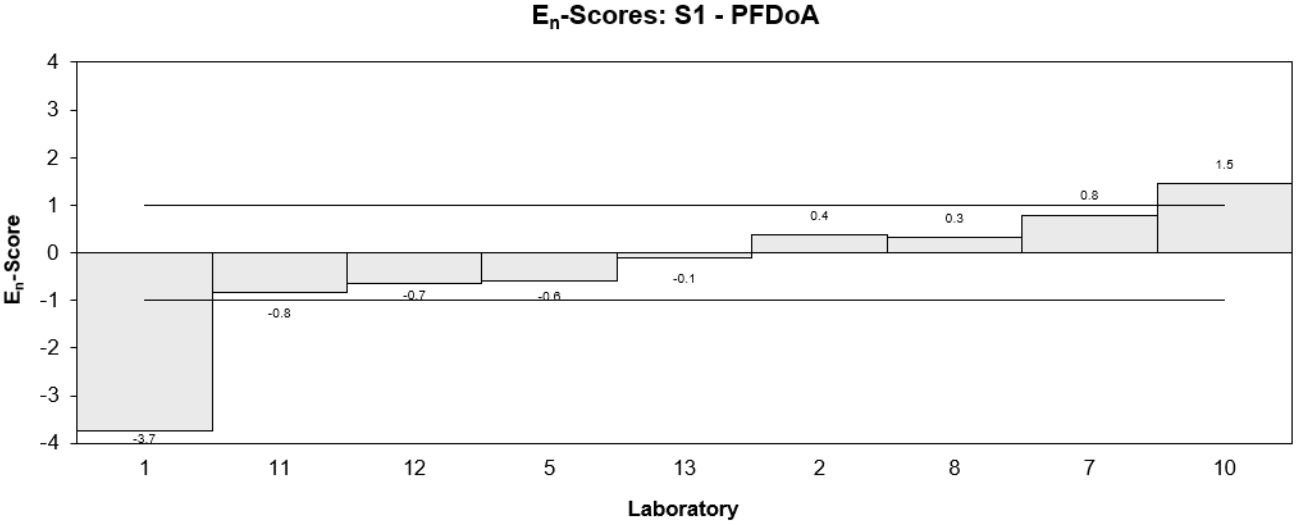

En-Scores: S1 - PFDoA
En-Score
Laboratory
-3.7
-0.8
-0.7
-0.6
-0.1
0.4
0.3
0.8
1.5

Figure 9

Table 13

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	PFTrDA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	7.46	2.68	64.6
2	10.5	2.63	NR
3	NT	NT	NT
4	6.26	2.817	NR
5	9.3	2.3	NR
6	NT	NT	NT
7	15	4.4	NR
8	9	5	95
9	NS	NS	NS
10	25.9	8.5	72
11	8.97	2.69	67
12	5.9	1.8	43
13	7.97	1.48242	70.9
14	NS	NS	NS

Statistics

Assigned Value	Not Set	
Spiked Value	15.2	0.8
Robust Average	9.5	2.8
Median	9.0	1.8
Mean	10.6	
N	10	
Max	25.9	
Min	5.9	
Robust SD	3.5	
Robust CV	37%	

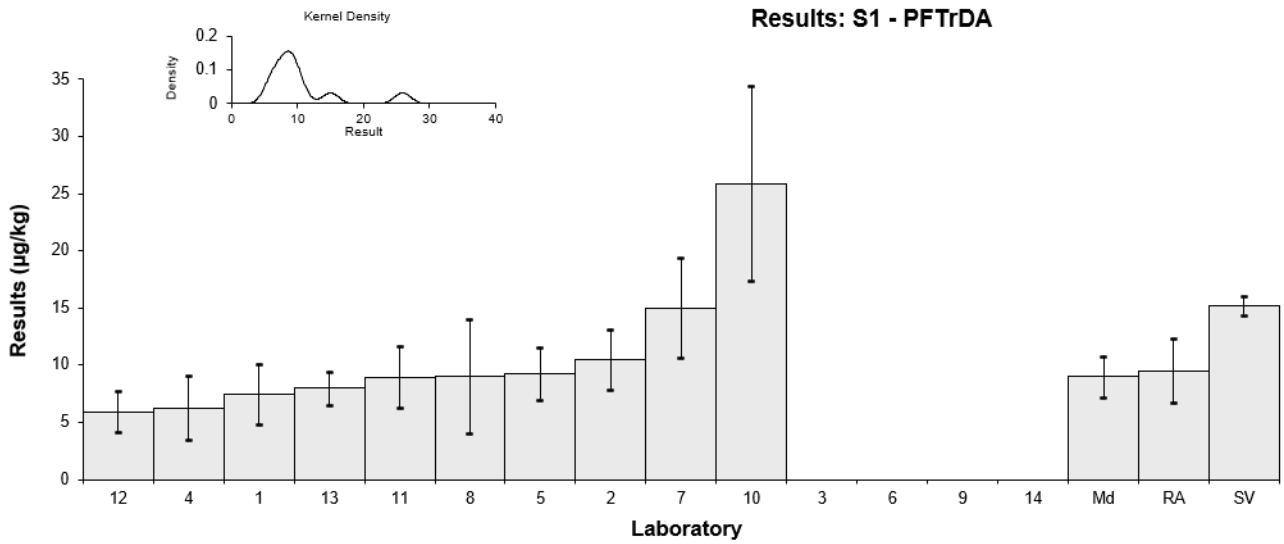

Results: S1 - PFTrDA
Kernel Density
Density
Result
Results (µg/kg)
Laboratory
12
4
1
13
11
8
5
2
7
10
3
6
9
14
Md
RA
SV

Figure 10

Table 14

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	PFTeDA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	17	6.41	22.1
2	NT	NT	NT
3	NT	NT	NT
4	6.85	3.083	NR
5	7.6	1.9	148
6	NT	NT	NT
7	13	4.0	NR
8	10	5	95
9	NS	NS	NS
10	27.5	9.1	72
11	4.61	1.39	122
12	4.9	1.3	47
13	8.96	2.84032	74.1
14	NS	NS	NS

Statistics

Assigned Value	Not Set	
Spiked Value	15.2	0.8
Robust Average	10.2	4.8
Median	9.0	5.0
Mean	11.2	
N	9	
Max	27.5	
Min	4.61	
Robust SD	5.8	
Robust CV	57%	

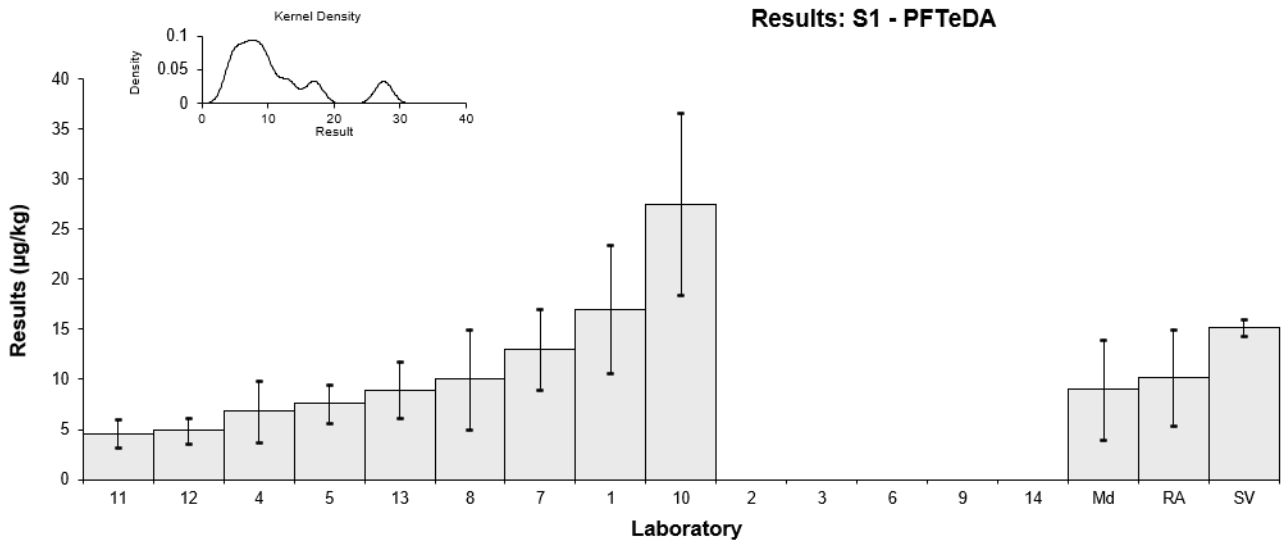

Results: S1 - PFTeDA
Kernel Density
Density
0.1
0.05
0
0
10
20
30
40
Result
Results (µg/kg)
40
35
30
25
20
15
10
5
0
11
12
4
5
13
8
7
1
10
2
3
6
9
14
Md
RA
SV
Laboratory

Figure 11

Table 15

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	PFBS
Unit	µg/kg

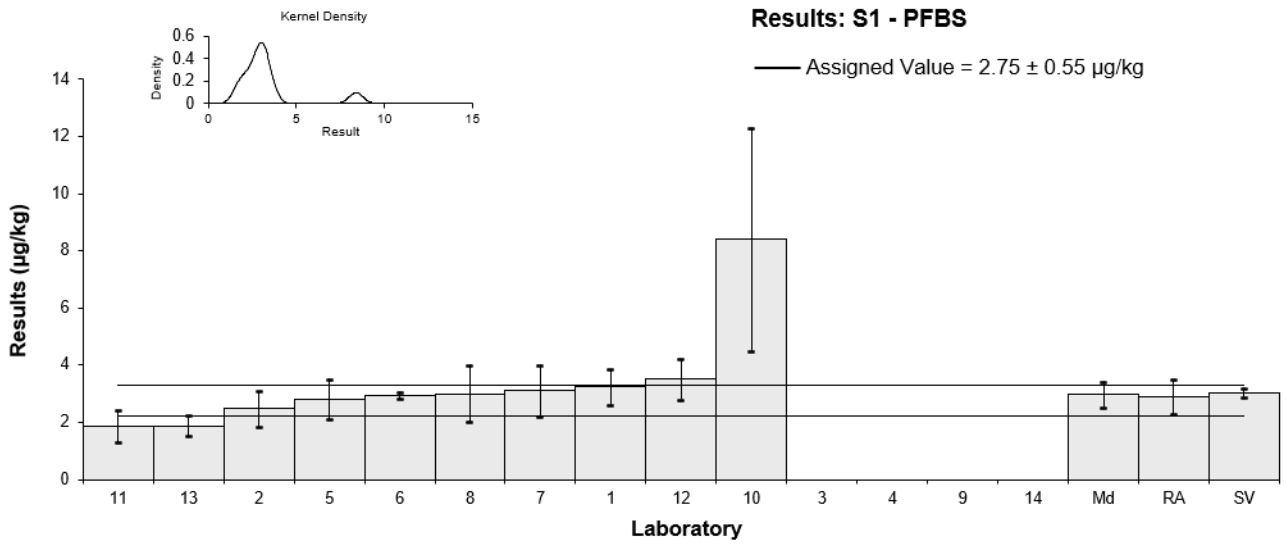
Participant Results

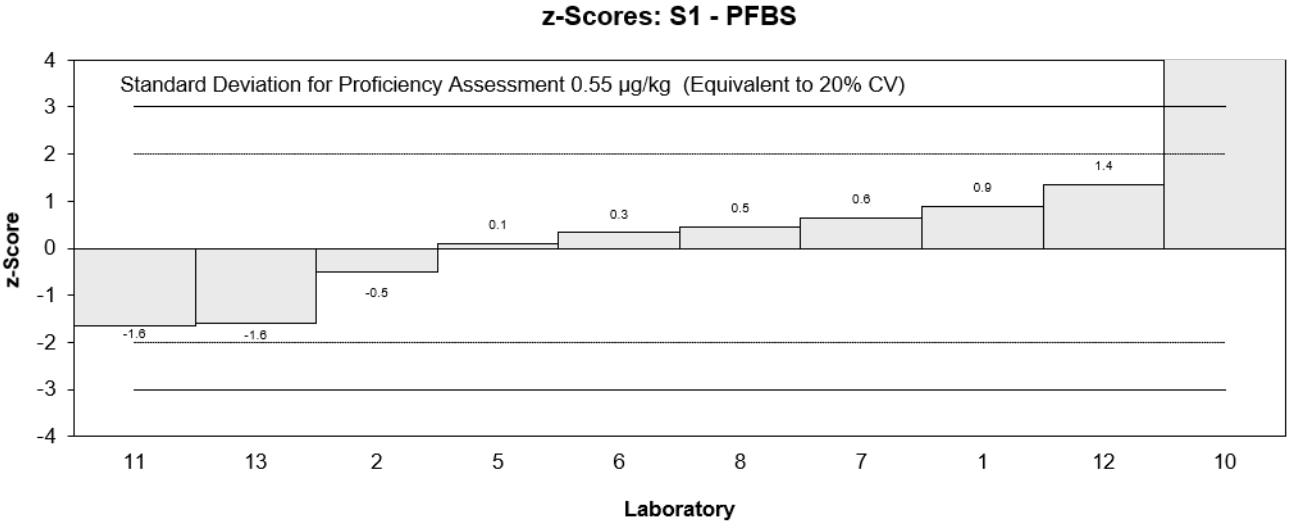
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	3.24	0.616	67.2	0.89	0.59
2	2.47	0.62	NR	-0.51	-0.34
3	NT	NT	NT		
4	<0.50	NR	NR		
5	2.8	0.70	147	0.09	0.06
6	2.93	0.125	86	0.33	0.32
7	3.1	0.9	NR	0.64	0.33
8	3	1	113	0.45	0.22
9	NS	NS	NS		
10*	8.4	3.9	73	10.27	1.43
11	1.85	0.556	83	-1.64	-1.15
12	3.5	0.71	102	1.36	0.84
13	1.87	0.35717	81.3	-1.60	-1.34
14	NS	NS	NS		

* Outlier, see Section 4.2

Statistics

Assigned Value	2.75	0.55
Spiked Value	3.04	0.15
Robust Average	2.88	0.61
Median	2.97	0.45
Mean	3.3	
N	10	
Max	8.4	
Min	1.85	
Robust SD	0.77	
Robust CV	27%	


Kernel Density
Density
Result
Results: S1 - PFBS
Assigned Value = 2.75 ± 0.55 µg/kg
Results (µg/kg)
Laboratory
11 13 2 5 6 8 7 1 12 10 3 4 9 14 Md RA SV


z-Scores: S1 - PFBS
Standard Deviation for Proficiency Assessment 0.55 µg/kg (Equivalent to 20% CV)
z-Score
-1.6 -1.6 -0.5 0.1 0.3 0.5 0.6 0.9 1.4
Laboratory
11 13 2 5 6 8 7 1 12 10

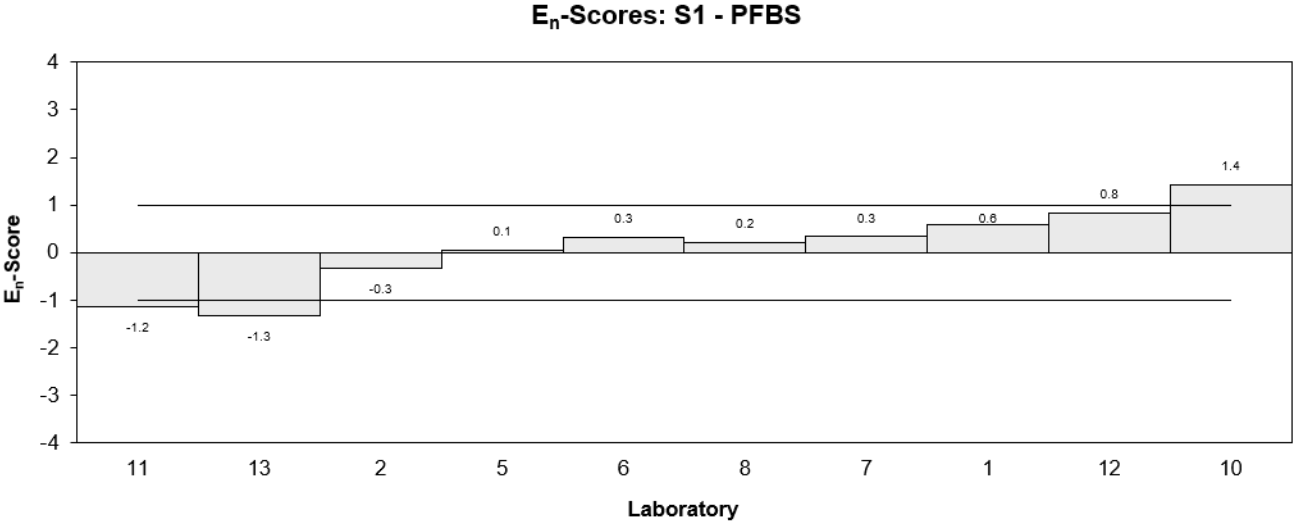

En-Scores: S1 - PFBS
En-Score
-1.2 -1.3 -0.3 0.1 0.3 0.2 0.3 0.6 0.8 1.4
Laboratory
11 13 2 5 6 8 7 1 12 10

Figure 12

Table 16

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	PFHxS
Unit	µg/kg

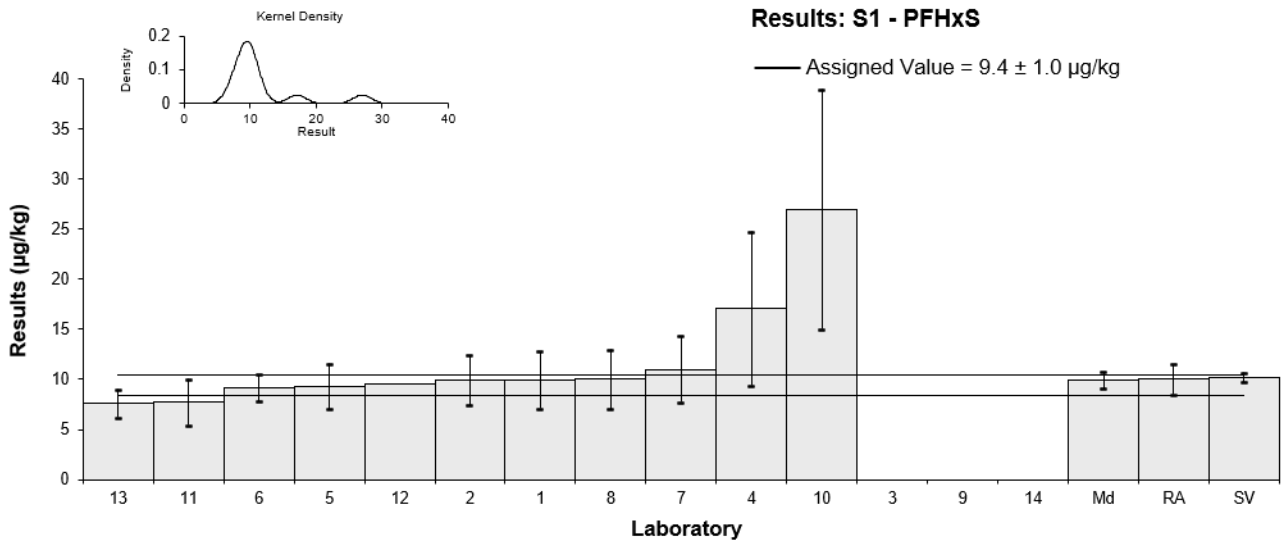
Participant Results

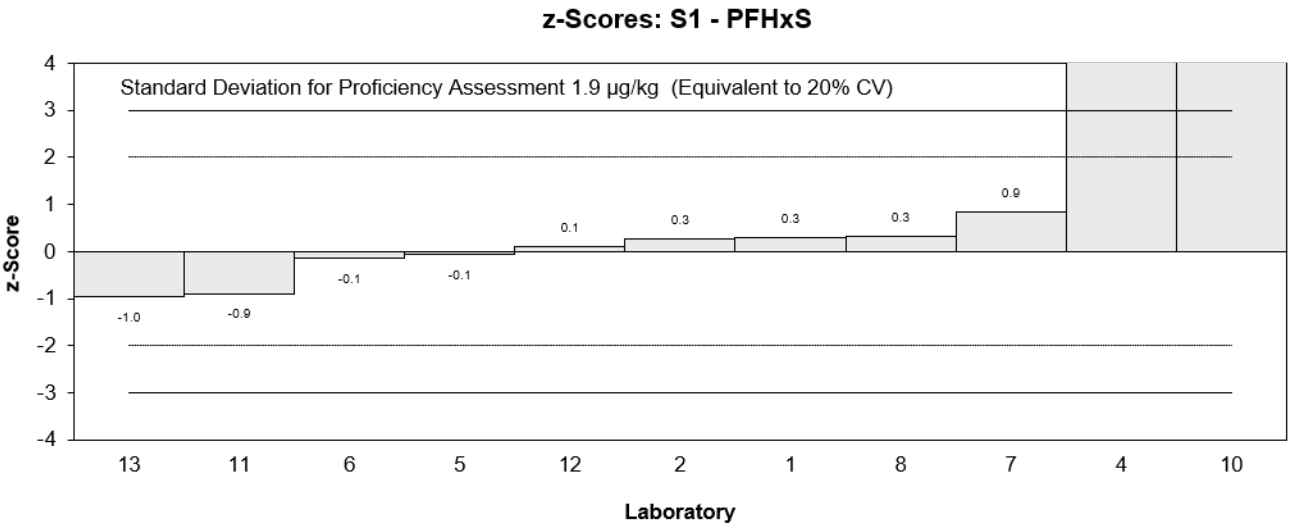
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	9.94	2.83	67.2	0.29	0.18
2	9.9	2.48	NR	0.27	0.19
3	NT	NT	NT		
4*	17.1	7.7	NR	4.10	0.99
5	9.3	2.3	141	-0.05	-0.04
6	9.14	1.37	65	-0.14	-0.15
7	11	3.3	NR	0.85	0.46
8	10	3	126	0.32	0.19
9	NS	NS	NS		
10*	27	12	72	9.36	1.46
11	7.72	2.32	80	-0.89	-0.66
12	9.6	NR	101	0.11	0.20
13	7.62	1.41732	80.3	-0.95	-1.03
14	NS	NS	NS		

* Outlier, see Section 4.2

Statistics

Assigned Value	9.4	1.0
Spiked Value	10.2	0.5
Robust Average	10.0	1.5
Median	9.90	0.85
Mean	11.7	
N	11	
Max	27	
Min	7.62	
Robust SD	2	
Robust CV	20%	


Kernel Density
Density
Result
Results: S1 - PFHxS
Assigned Value = 9.4 ± 1.0 µg/kg
Results (µg/kg)
Laboratory
13 11 6 5 12 2 1 8 7 4 10 3 9 14 Md RA SV


z-Scores: S1 - PFHxS
Standard Deviation for Proficiency Assessment 1.9 µg/kg (Equivalent to 20% CV)
z-Score
-1.0 -0.9 -0.1 -0.1 0.1 0.3 0.3 0.3 0.9
Laboratory
13 11 6 5 12 2 1 8 7 4 10

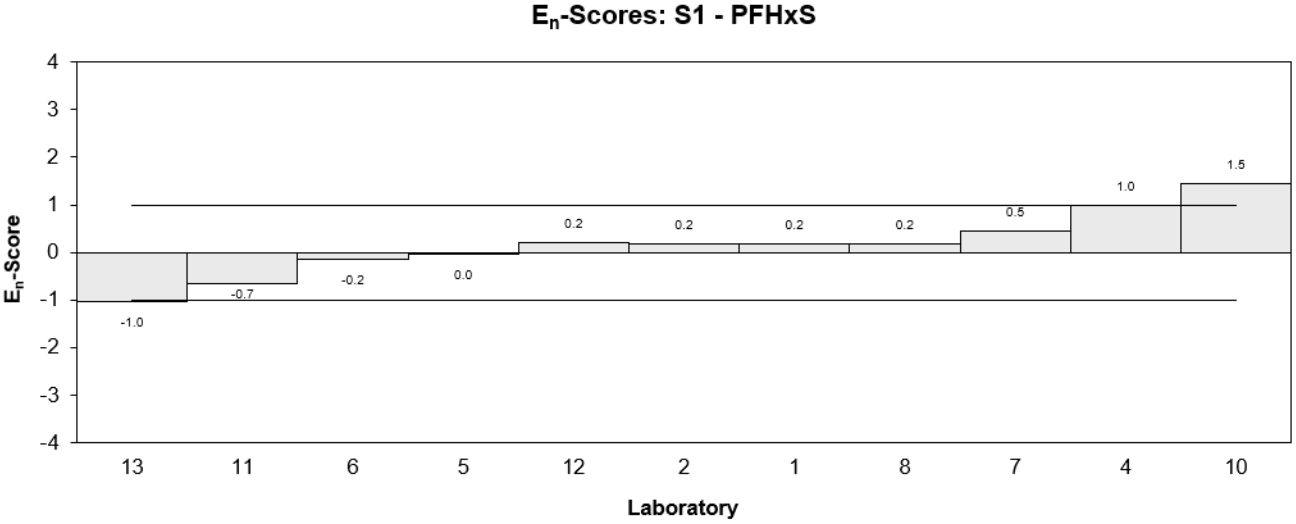

En-Scores: S1 - PFHxS
En-Score
-1.0 -0.7 -0.2 0.0 0.2 0.2 0.2 0.2 0.5 1.0 1.5
Laboratory
13 11 6 5 12 2 1 8 7 4 10

Figure 13

Table 17

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	PFOS
Unit	µg/kg

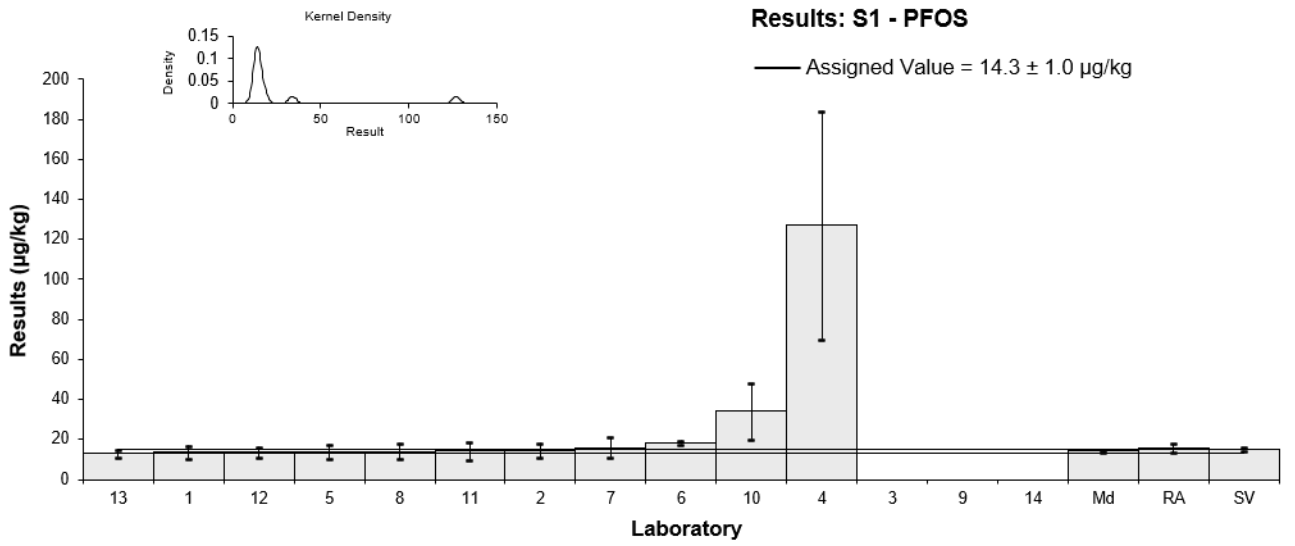
Participant Results

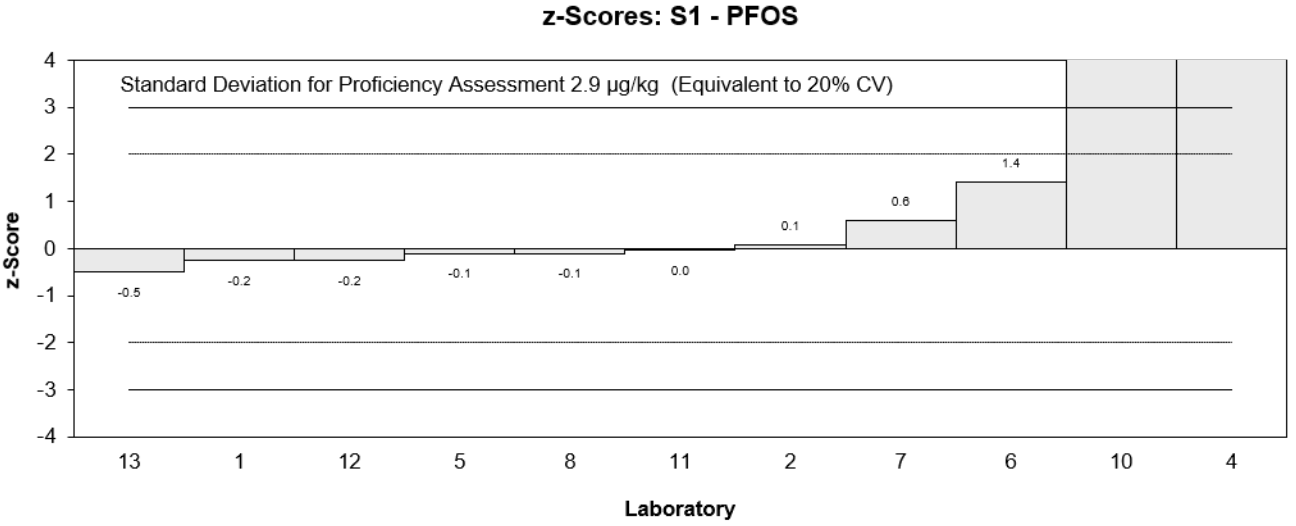
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	13.6	3.17	79.6	-0.24	-0.21
2	14.5	3.63	NR	0.07	0.05
3	NT	NT	NT		
4*	127	57	NR	39.41	1.98
5	14	3.5	109	-0.10	-0.08
6	18.37	1	63	1.42	2.88
7	16	4.9	NR	0.59	0.34
8	14	4	127	-0.10	-0.07
9	NS	NS	NS		
10*	34	14	70	6.89	1.40
11	14.2	4.27	96	-0.03	-0.02
12	13.6	2.7	85	-0.24	-0.24
13	12.9	1.9221	76.9	-0.49	-0.65
14	NS	NS	NS		

* Outlier, see Section 4.2

Statistics

Assigned Value	14.3	1.0
Spiked Value	15.2	0.8
Robust Average	15.6	2.3
Median	14.2	0.7
Mean	27	
N	11	
Max	127	
Min	12.9	
Robust SD	3.1	
Robust CV	20%	


Kernel Density
Density
Result
Results: S1 - PFOS
Assigned Value = 14.3 ± 1.0 µg/kg
Results (µg/kg)
Laboratory
13 1 12 5 8 11 2 7 6 10 4 3 9 14 Md RA SV


z-Scores: S1 - PFOS
Standard Deviation for Proficiency Assessment 2.9 µg/kg (Equivalent to 20% CV)
z-Score
-0.5 -0.2 -0.2 -0.1 -0.1 0.0 0.1 0.6 1.4
Laboratory
13 1 12 5 8 11 2 7 6 10 4

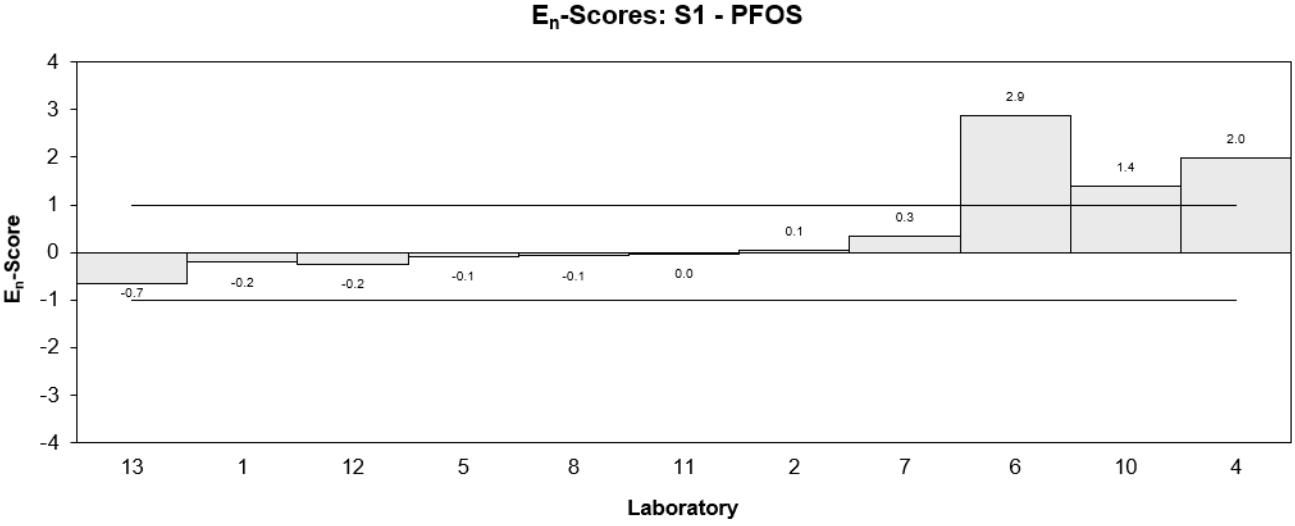

En-Scores: S1 - PFOS
En-Score
-0.7 -0.2 -0.2 -0.1 -0.1 0.0 0.1 0.3 2.9 1.4 2.0
Laboratory
13 1 12 5 8 11 2 7 6 10 4

Figure 14

Table 18

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	N-EtFOSAA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	34.8	6.06	32.1
2	NT	NT	NT
3	NT	NT	NT
4	<0.50	NR	NR
5	15	3.6	152
6	NT	NT	NT
7	19	5.8	NR
8	18	6	109
9	NS	NS	NS
10	NT	NT	NT
11	16.3	4.88	73
12	NT	NT	NT
13	18.5	3.922	77.5
14	NS	NS	NS

Statistics

Assigned Value	Not Set	
Spiked Value	20.2	1.0
Robust Average	18.3	3.1
Median	18.3	2.0
Mean	20.3	
N	6	
Max	34.8	
Min	15	
Robust SD	3	
Robust CV	17%	

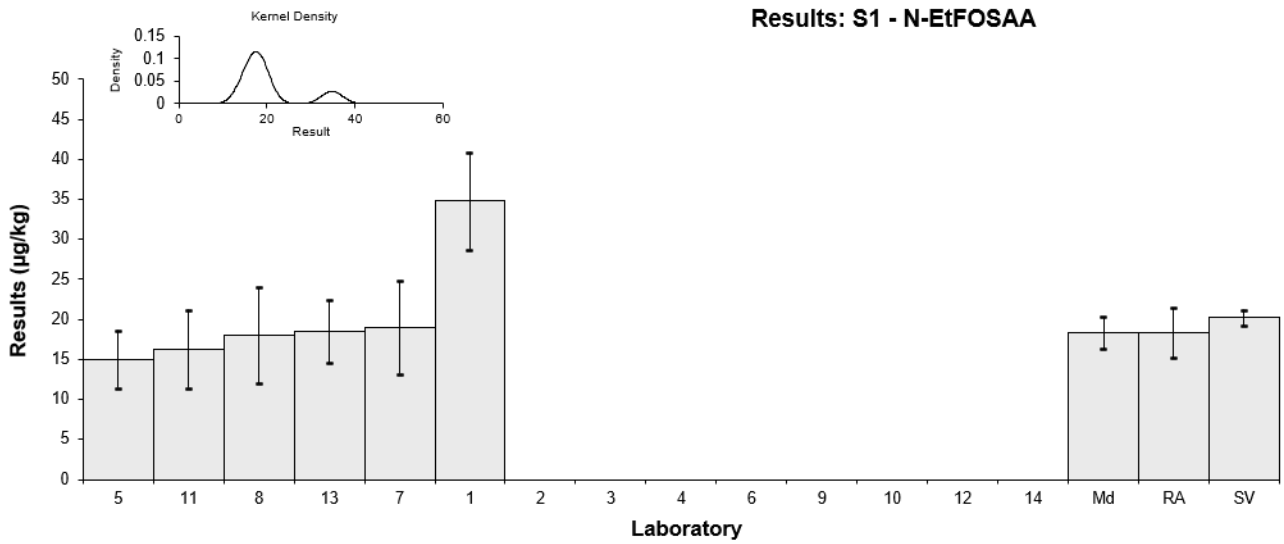

Results: S1 - N-EtFOSAA
Kernel Density
Density
0.15
0.1
0.05
0
0
20
40
60
Result
Results (µg/kg)
50
45
40
35
30
25
20
15
10
5
0
5
11
8
13
7
1
2
3
4
6
9
10
12
14
Md
RA
SV
Laboratory

Figure 15

Table 19

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	6:2FTS
Unit	µg/kg

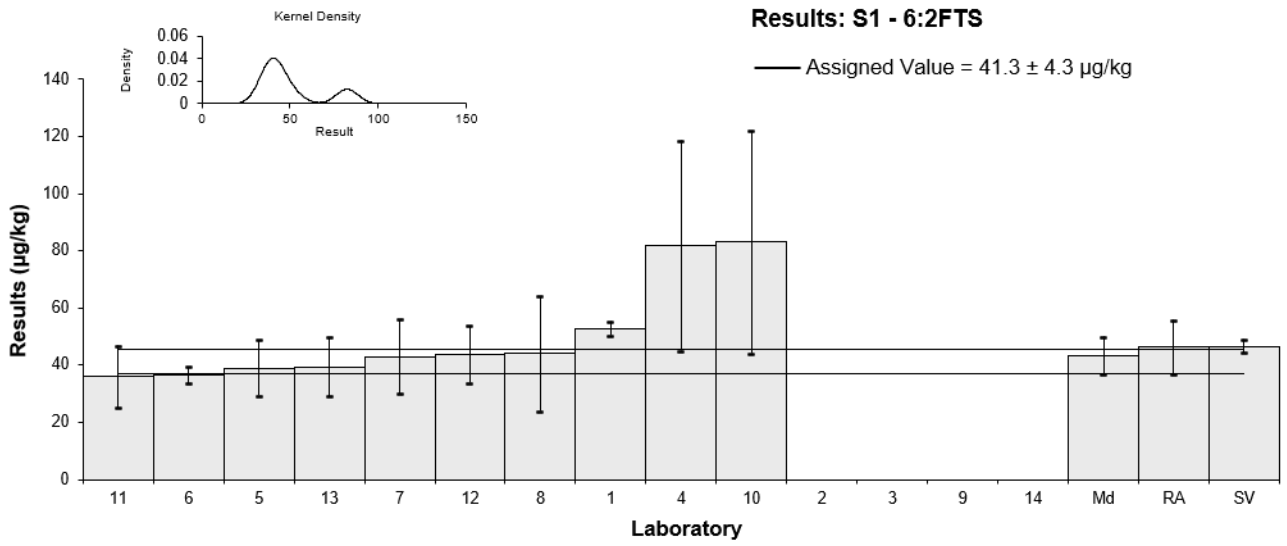
Participant Results

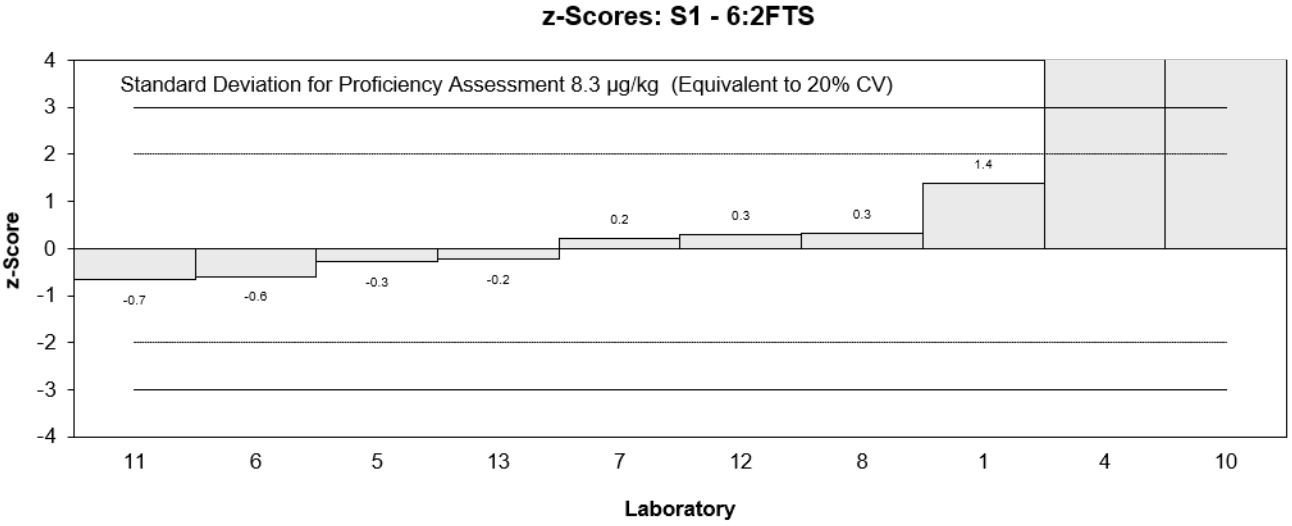
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	52.8	2.49	69.4	1.39	2.31
2	NT	NT	NT		
3	NT	NT	NT		
4*	81.8	36.81	NR	4.90	1.09
5	39	9.8	280	-0.28	-0.21
6	36.43	2.88	100	-0.59	-0.94
7	43	13	NR	0.21	0.12
8	44	20	124	0.33	0.13
9	NS	NS	NS		
10*	83	39	81	5.05	1.06
11	35.9	10.8	137	-0.65	-0.46
12	43.7	10	NR	0.29	0.22
13	39.4	10.244	169	-0.23	-0.17
14	NS	NS	NS		

* Outlier, see Section 4.2

Statistics

Assigned Value	41.3	4.3
Spiked Value	46.6	2.3
Robust Average	46.3	9.5
Median	43.4	6.6
Mean	50	
N	10	
Max	83	
Min	35.9	
Robust SD	12	
Robust CV	26%	


Kernel Density
Density
0.06
0.04
0.02
0
0
50
100
150
Result
Results: S1 - 6:2FTS
Assigned Value = 41.3 ± 4.3 µg/kg
Results (µg/kg)
140
120
100
80
60
40
20
0
11
6
5
13
7
12
8
1
4
10
2
3
9
14
Md
RA
SV
Laboratory


z-Scores: S1 - 6:2FTS
Standard Deviation for Proficiency Assessment 8.3 µg/kg (Equivalent to 20% CV)
z-Score
4
3
2
1
0
-1
-2
-3
-4
-0.7
-0.6
-0.3
-0.2
0.2
0.3
0.3
1.4
11
6
5
13
7
12
8
1
4
10
Laboratory

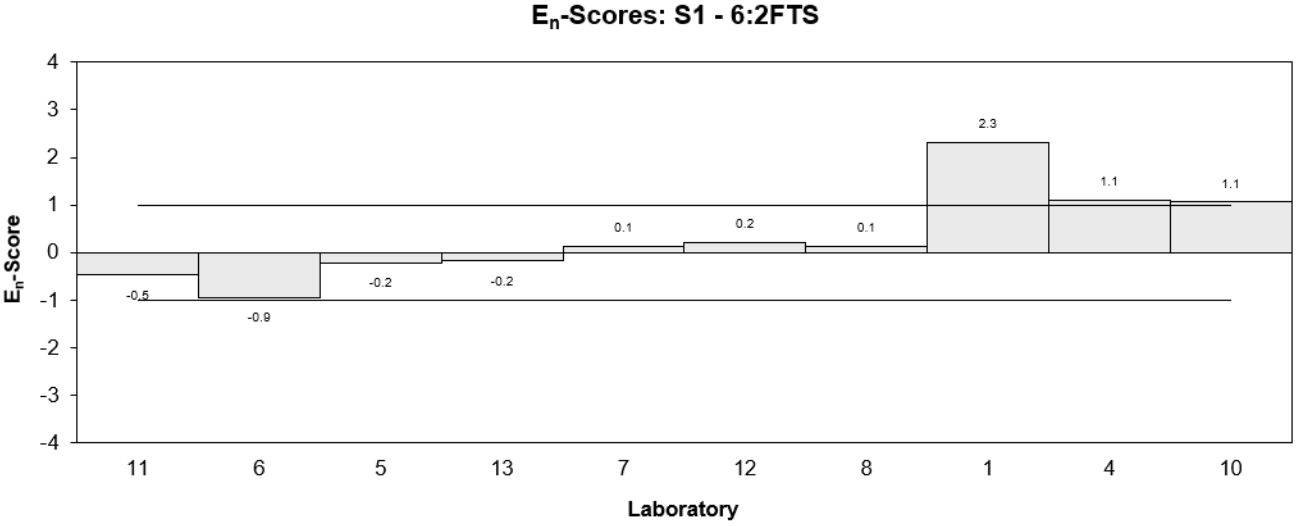

En-Scores: S1 - 6:2FTS
En-Score
4
3
2
1
0
-1
-2
-3
-4
-0.5
-0.9
-0.2
-0.2
0.1
0.2
0.1
2.3
1.1
1.1
11
6
5
13
7
12
8
1
4
10
Laboratory

Figure 16

Table 20

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	8:2FTS
Unit	µg/kg

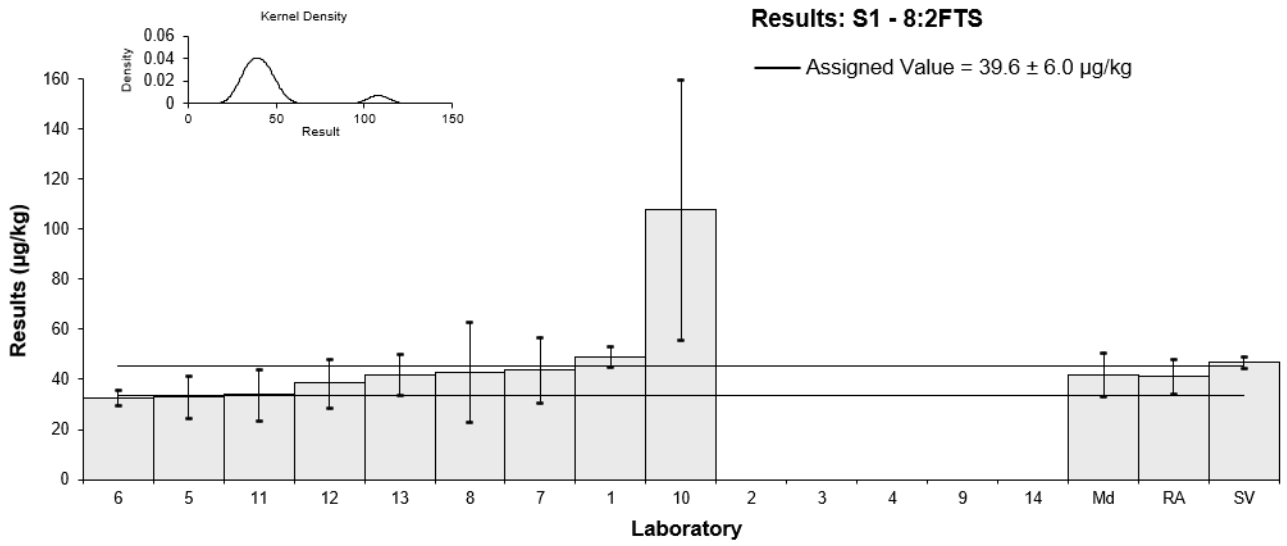
Participant Results

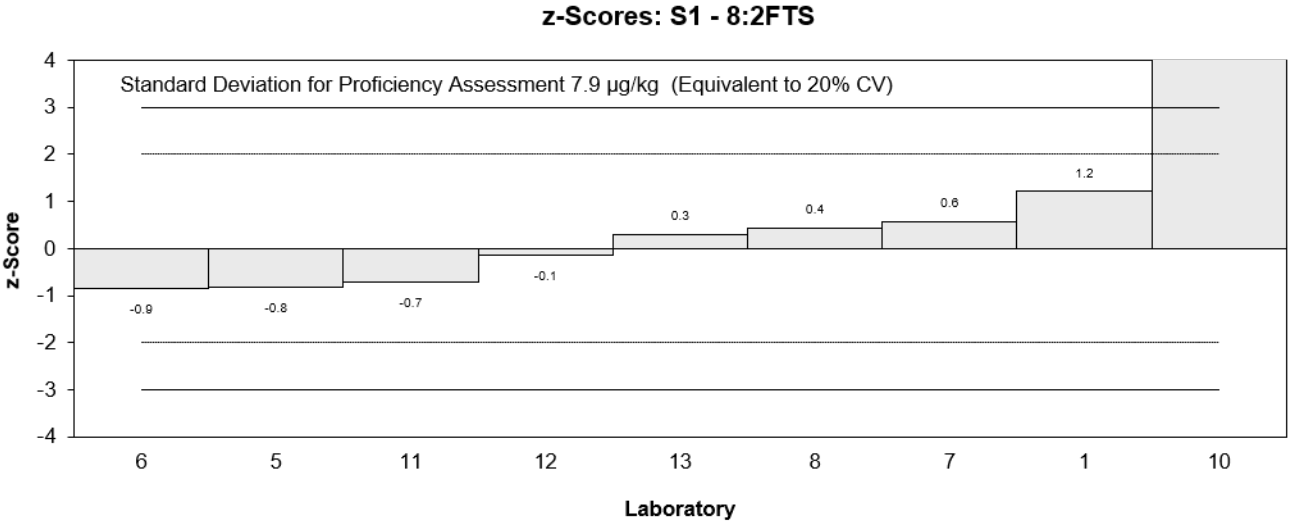
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	49.2	4.11	69.4	1.21	1.32
2	NT	NT	NT		
3	NT	NT	NT		
4	<0.50	NR	NR		
5	33	8.3	458	-0.83	-0.64
6	32.8	3.19	14	-0.86	-1.00
7	44	13	NR	0.56	0.31
8	43	20	97	0.43	0.16
9	NS	NS	NS		
10*	108	52	72	8.64	1.31
11	34	10.2	191	-0.71	-0.47
12	38.5	9.6	NR	-0.14	-0.10
13	42	8.232	138	0.30	0.24
14	NS	NS	NS		

* Outlier, see Section 4.2

Statistics

Assigned Value	39.6	6.0
Spiked Value	47.0	2.3
Robust Average	41.1	6.9
Median	42.0	8.9
Mean	47	
N	9	
Max	108	
Min	32.8	
Robust SD	8.3	
Robust CV	20%	


Kernel Density
Density
Result
Results: S1 - 8:2FTS
Assigned Value = 39.6 ± 6.0 µg/kg
Results (µg/kg)
Laboratory
6 5 11 12 13 8 7 1 10 2 3 4 9 14 Md RA SV


z-Scores: S1 - 8:2FTS
Standard Deviation for Proficiency Assessment 7.9 µg/kg (Equivalent to 20% CV)
z-Score
-0.9 -0.8 -0.7 -0.1 0.3 0.4 0.6 1.2
Laboratory
6 5 11 12 13 8 7 1 10

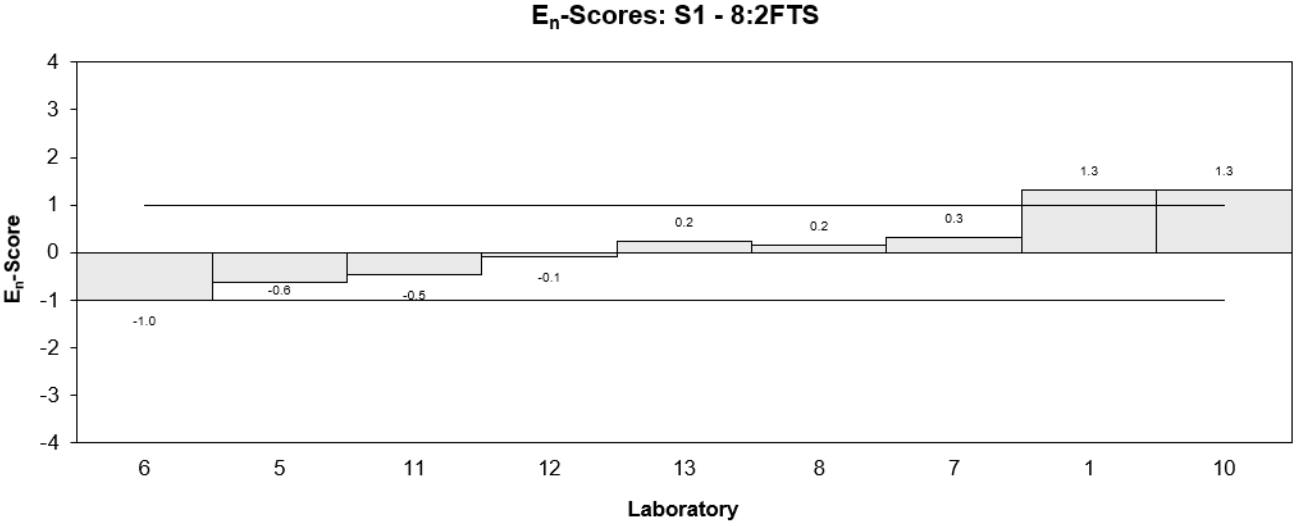

En-Scores: S1 - 8:2FTS
En-Score
-1.0 -0.6 -0.5 -0.1 0.2 0.2 0.3 1.3 1.3
Laboratory
6 5 11 12 13 8 7 1 10

Figure 17

Table 21

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	8:2diPAP
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	NT	NT	NT
2	NT	NT	NT
3	NT	NT	NT
4	16.4	7.38	NR
5	17	4.1	326
6	NT	NT	NT
7	22	6.5	NR
8	NT	NT	NT
9	NS	NS	NS
10	NT	NT	NT
11	NT	NT	NT
12	NT	NT	NT
13	18.9	4.3848	60.1
14	NS	NS	NS

Statistics

Assigned Value	Not Set	
Spiked Value	20.3	1.0
Robust Average	NA (N<6)	
Median	18.0	2.3
Mean	18.6	
N	4	
Max	22	
Min	16.4	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

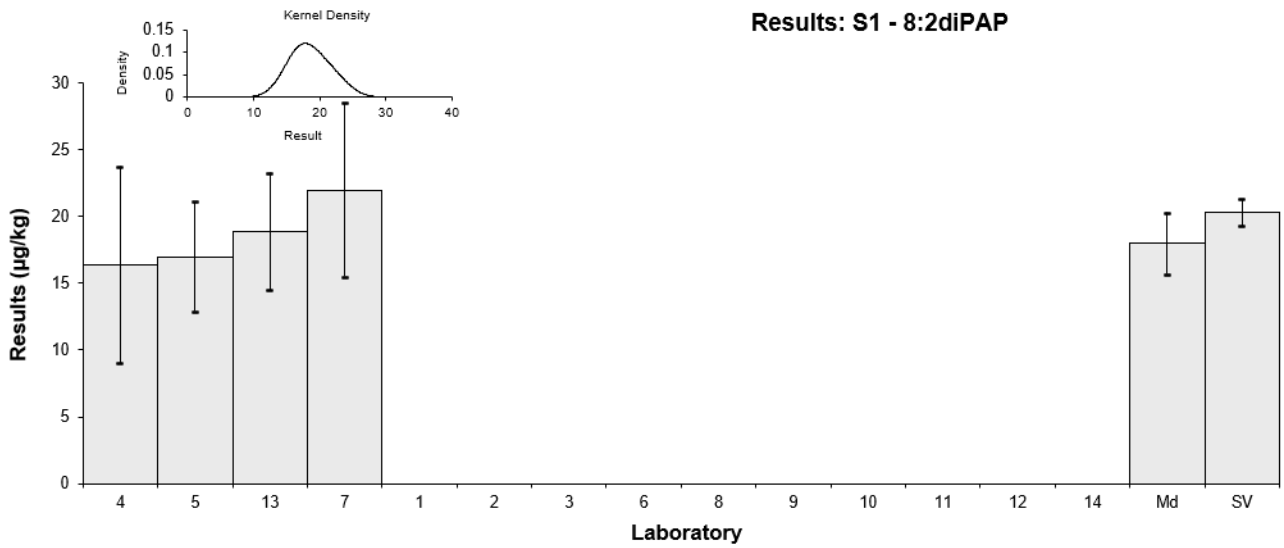

Results: S1 - 8:2diPAP
Kernel Density
Density
0.15
0.1
0.05
0
0
10
20
30
40
Result
Results (µg/kg)
30
25
20
15
10
5
0
4
5
13
7
1
2
3
6
8
9
10
11
12
14
Md
SV
Laboratory

Figure 18

Table 22

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	GenX
Unit	µg/kg

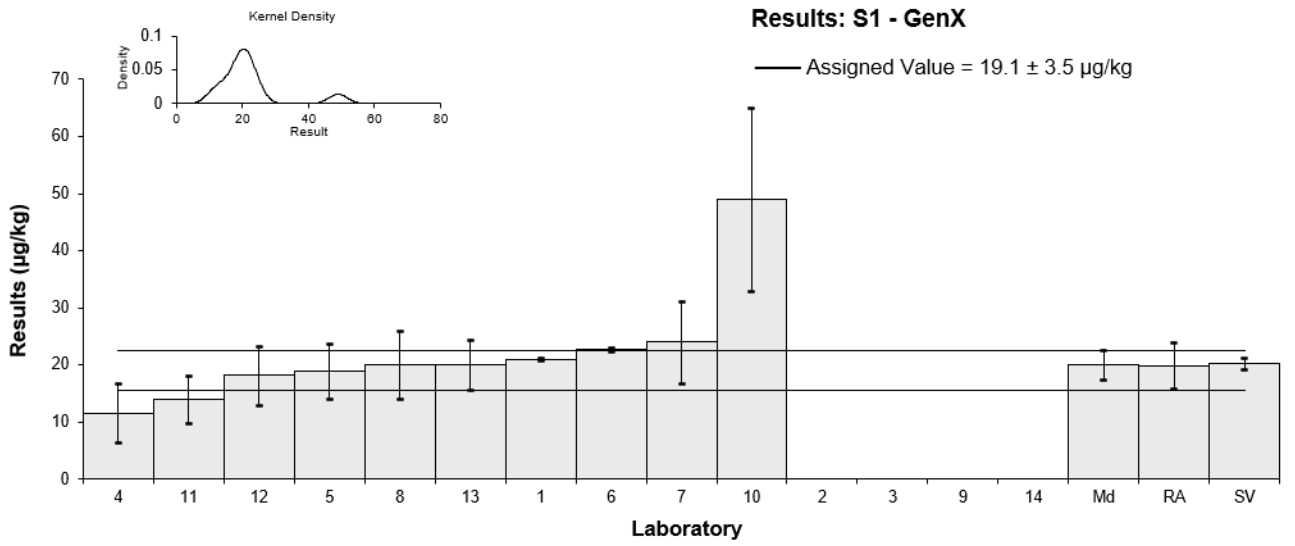
Participant Results

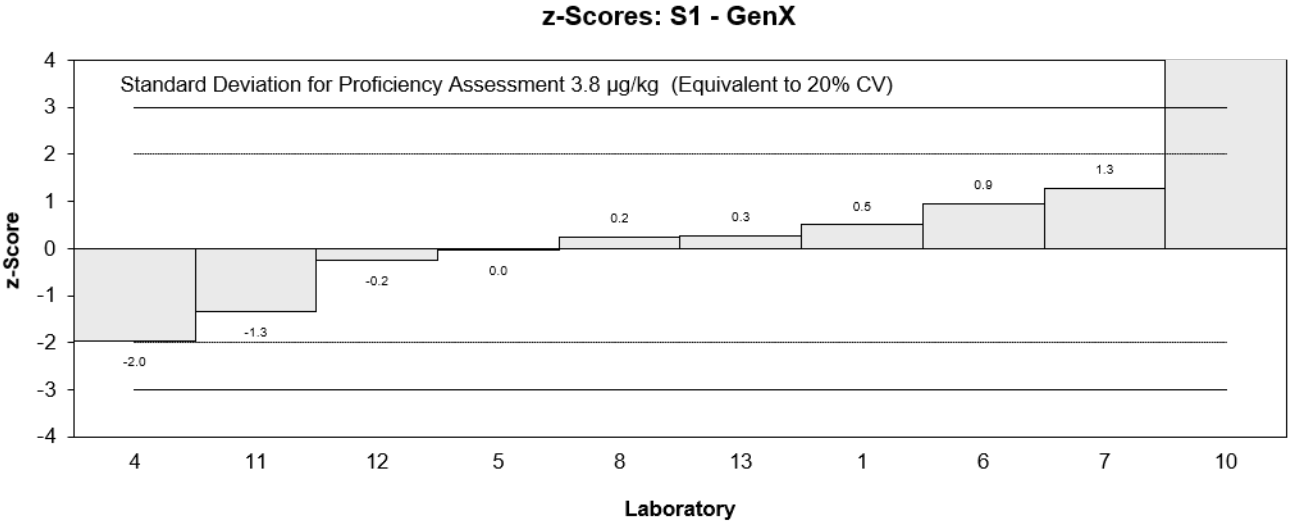
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	21	0.255	36.7	0.50	0.54
2	NT	NT	NT		
3	NT	NT	NT		
4	11.6	5.22	NR	-1.96	-1.19
5	19	4.8	77	-0.03	-0.02
6	22.7	0.355	26	0.94	1.02
7	24	7.1	NR	1.28	0.62
8	20	6	108	0.24	0.13
9	NS	NS	NS		
10*	49	16	72	7.83	1.83
11	14	4.19	23	-1.34	-0.93
12	18.2	5.1	66	-0.24	-0.15
13	20.1	4.4019	55	0.26	0.18
14	NS	NS	NS		

* Outlier, see Section 4.2

Statistics

Assigned Value	19.1	3.5
Spiked Value	20.2	1.0
Robust Average	19.9	4.0
Median	20.1	2.6
Mean	22.0	
N	10	
Max	49	
Min	11.6	
Robust SD	5.1	
Robust CV	26%	


Kernel Density
Density
Result
Results: S1 - GenX
Assigned Value = 19.1 ± 3.5 µg/kg
Results (µg/kg)
Laboratory
4 11 12 5 8 13 1 6 7 10 2 3 9 14 Md RA SV


z-Scores: S1 - GenX
Standard Deviation for Proficiency Assessment 3.8 µg/kg (Equivalent to 20% CV)
z-Score
-2.0
-1.3
-0.2
0.0
0.2
0.3
0.5
0.9
1.3
Laboratory
4 11 12 5 8 13 1 6 7 10

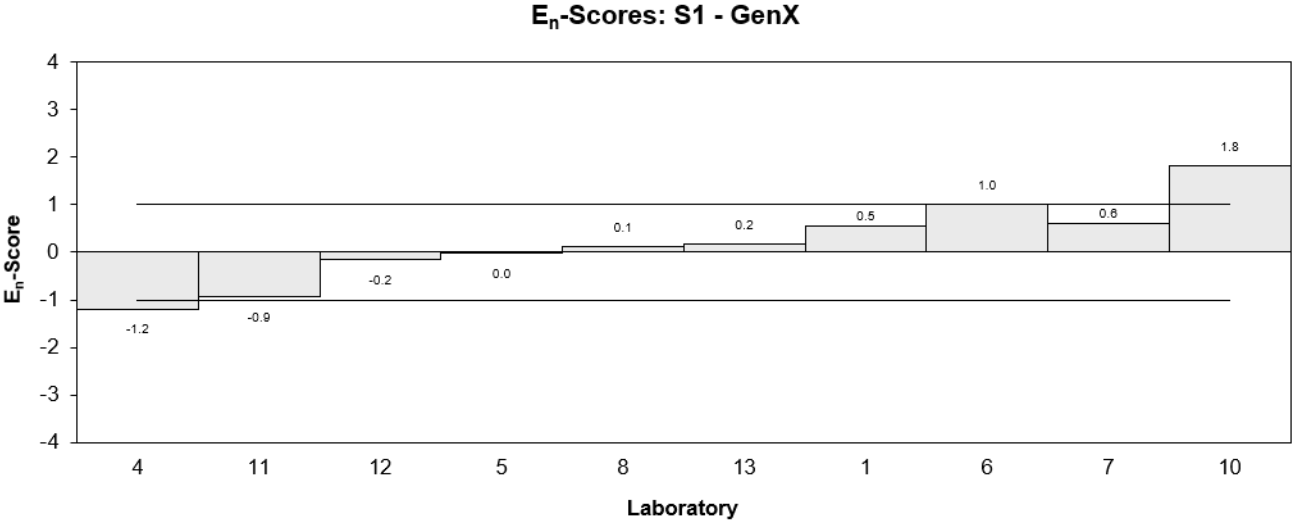

En-Scores: S1 - GenX
En-Score
-1.2
-0.9
-0.2
0.0
0.1
0.2
0.5
1.0
0.6
1.8
Laboratory
4 11 12 5 8 13 1 6 7 10

Figure 19

Table 23

Sample Details

Sample No.	S1
Matrix	Cardboard
Analyte	ADONA
Unit	µg/kg

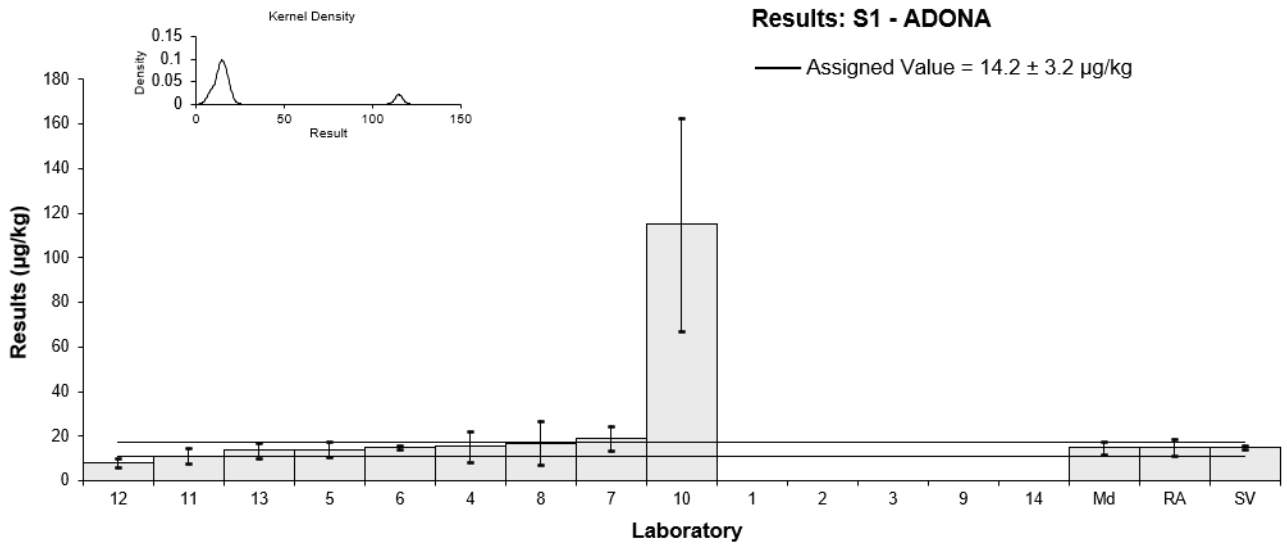
Participant Results

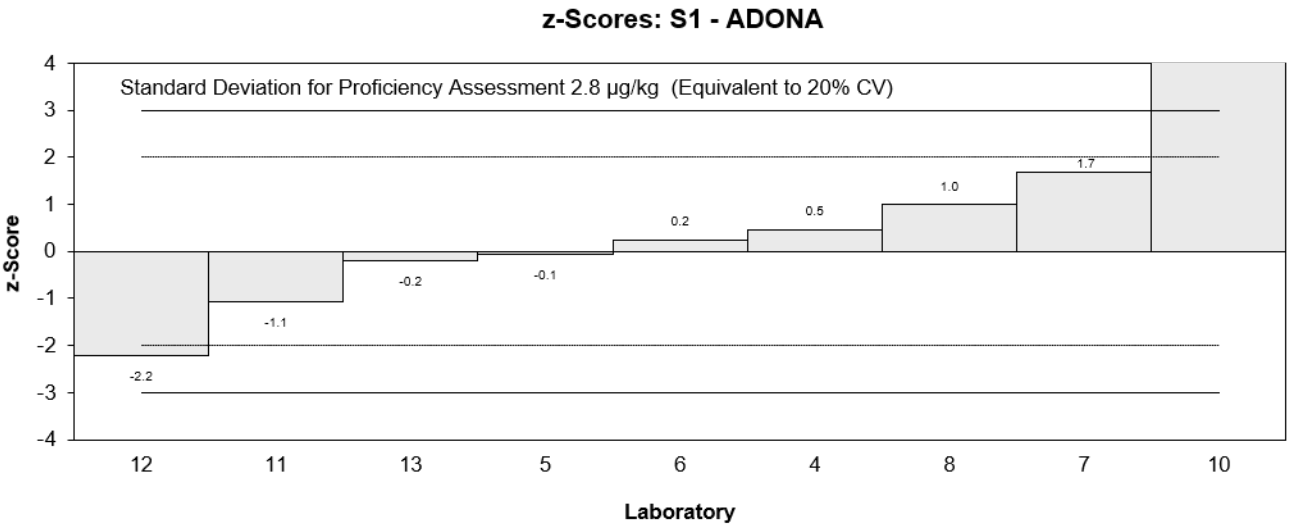
Lab. Code	Result	Uncertainty	Rec	z	E_n
1	NT	NT	NT		
2	NT	NT	NT		
3	NT	NT	NT		
4	15.5	6.97	NR	0.46	0.17
5	14	3.5	NR	-0.07	-0.04
6	14.84	0.87	67	0.23	0.19
7	19	5.6	NR	1.69	0.74
8	17	10	90	0.99	0.27
9	NS	NS	NS		
10*	115	48	72	35.49	2.10
11	11.2	3.35	96	-1.06	-0.65
12	7.9	2	66	-2.22	-1.67
13	13.6	3.2096	55	-0.21	-0.13
14	NS	NS	NS		

* Outlier, see Section 4.2

Statistics

Assigned Value	14.2	3.2
Spiked Value	15.2	0.8
Robust Average	15.0	3.9
Median	14.8	2.7
Mean	25	
N	9	
Max	115	
Min	7.9	
Robust SD	4.7	
Robust CV	31%	


Kernel Density
Density
Result
Results: S1 - ADONA
Assigned Value = 14.2 ± 3.2 µg/kg
Results (µg/kg)
Laboratory
12 11 13 5 6 4 8 7 10 1 2 3 9 14 Md RA SV


z-Scores: S1 - ADONA
Standard Deviation for Proficiency Assessment 2.8 µg/kg (Equivalent to 20% CV)
z-Score
-2.2
-1.1
-0.2
-0.1
0.2
0.5
1.0
1.7
Laboratory
12 11 13 5 6 4 8 7 10

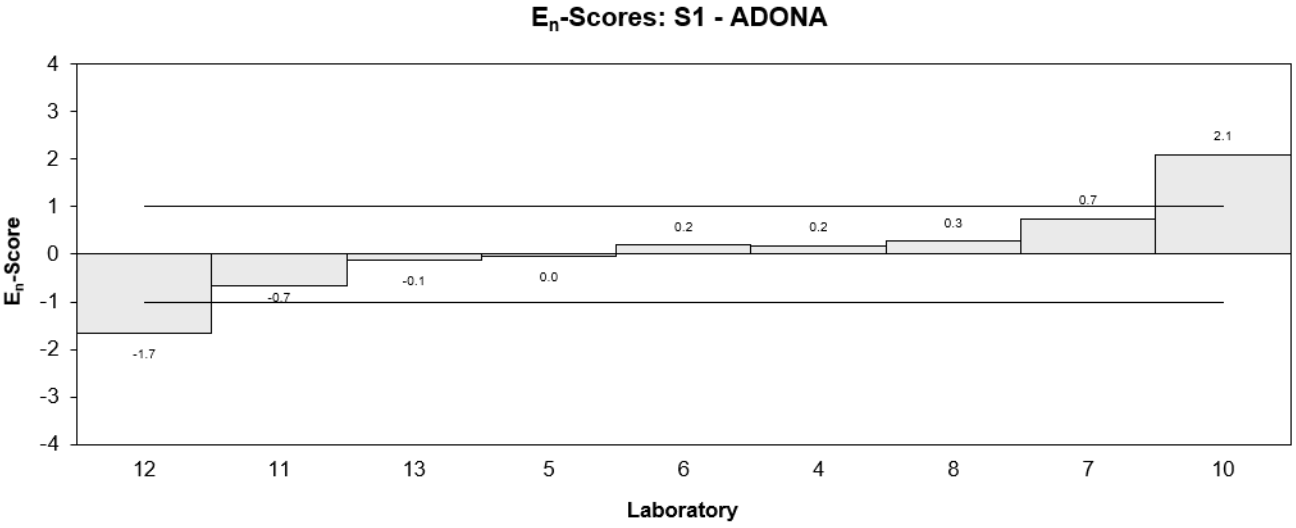

En-Scores: S1 - ADONA
En-Score
-1.7
-0.7
-0.1
0.0
0.2
0.2
0.3
0.7
2.1
Laboratory
12 11 13 5 6 4 8 7 10

Figure 20

Table 24

Sample Details

Sample No.	S2
Matrix	Cardboard (pilot sample)
Analyte	Extractable Organic Fluorine
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	NS	NS	NS
2	NS	NS	NS
3	NT	NT	NT
4	<256	NR	NR
5	4400	1300	NR
6	NS	NS	NS
7	NS	NS	NS
8	NT	NT	NT
9	4200	130	NR
10	NS	NS	NS
11	9690	2910	NR
12	NS	NS	NS
13	5340	865.08	NR
14	NS	NS	NS

Statistics

Assigned Value	Not Set	
Spiked Value	5200	260
Robust Average	NA (N<6)	
Median	4900	1100
Mean	5900	
N	4	
Max	9690	
Min	4200	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

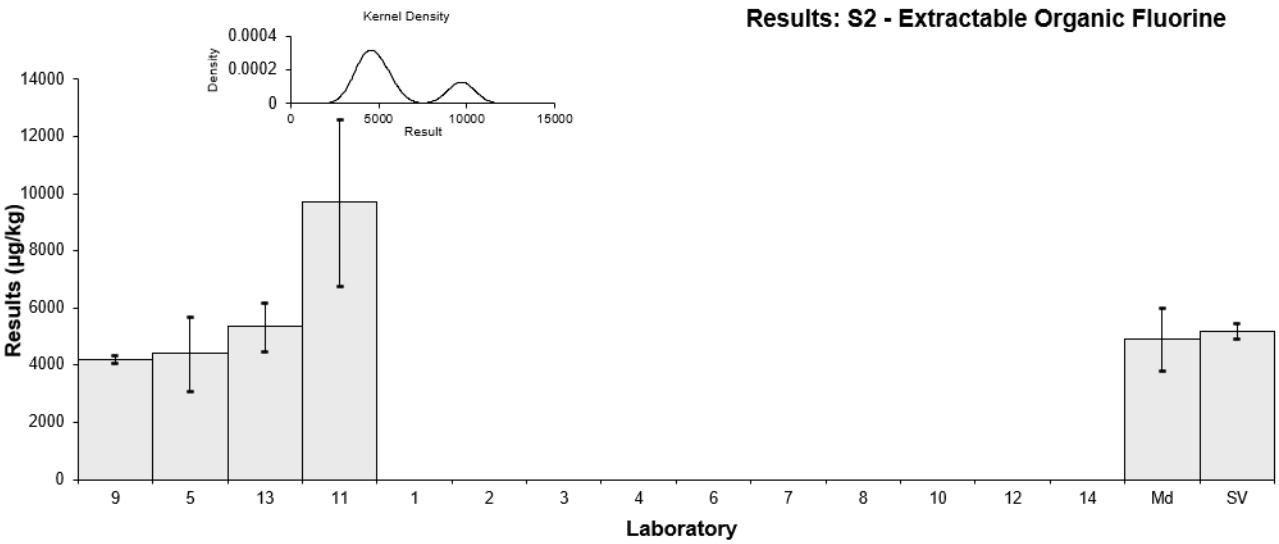

Results: S2 - Extractable Organic Fluorine
Kernel Density
Density
0.0004
0.0002
0
0
5000
10000
15000
Result
Results (µg/kg)
14000
12000
10000
8000
6000
4000
2000
0
9
5
13
11
1
2
3
4
6
7
8
10
12
14
Md
SV
Laboratory

Figure 21

Table 25

Sample Details

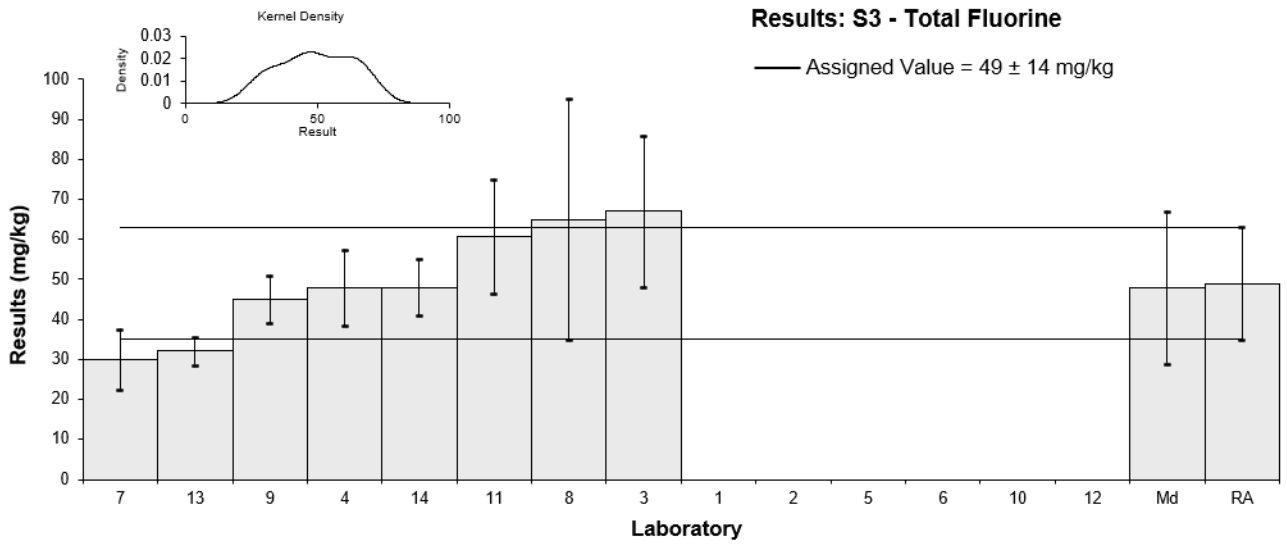
Sample No.	S3
Matrix	Cardboard
Analyte	Total Fluorine
Unit	mg/kg

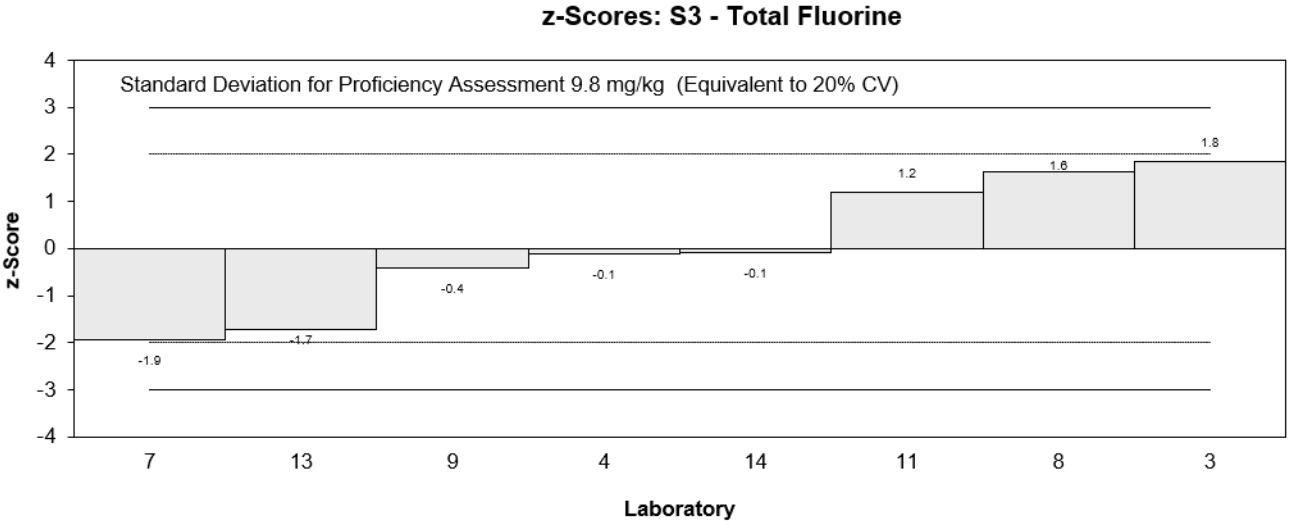
Participant Results

Lab. Code	Result	Uncertainty	Rec	z	E_n
1	NS	NS	NS		
2	NS	NS	NS		
3	67	19	NR	1.84	0.76
4	47.9	9.58	NR	-0.11	-0.06
5	NS	NS	NS		
6	NS	NS	NS		
7	30	7.5	NR	-1.94	-1.20
8	65	30	NR	1.63	0.48
9	45	6	NR	-0.41	-0.26
10	NS	NS	NS		
11	60.8	14.3	NR	1.20	0.59
12	NS	NS	NS		
13	32.1	3.4668	NR	-1.72	-1.17
14	48	7	NR	-0.10	-0.06

Statistics

Assigned Value	49	14
Robust Average	49	14
Median	48	19
Mean	49.5	
N	8	
Max	67	
Min	30	
Robust SD	16	
Robust CV	32%	


Results: S3 - Total Fluorine
Assigned Value = 49 ± 14 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
7
13
9
4
14
11
8
3
1
2
5
6
10
12
Md
RA


z-Scores: S3 - Total Fluorine
Standard Deviation for Proficiency Assessment 9.8 mg/kg (Equivalent to 20% CV)
z-Score
-1.9
-1.7
-0.4
-0.1
-0.1
1.2
1.6
1.8
Laboratory
7
13
9
4
14
11
8
3

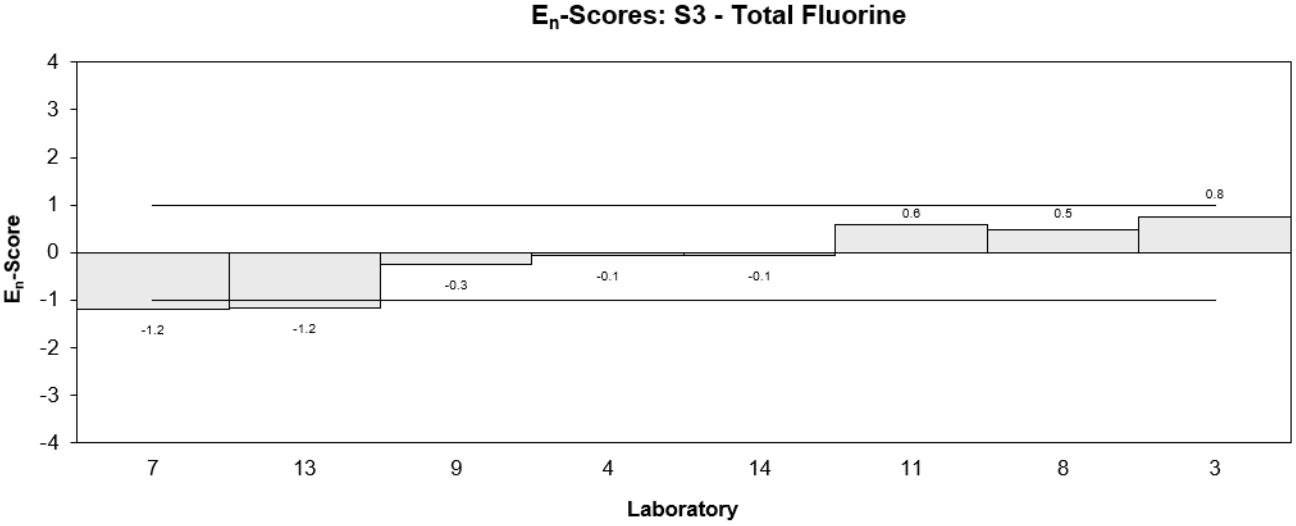

En-Scores: S3 - Total Fluorine
En-Score
-1.2
-1.2
-0.3
-0.1
-0.1
0.6
0.5
0.8
Laboratory
7
13
9
4
14
11
8
3

Figure 22

Table 26

Sample Details

Sample No.	S4
Matrix	Cosmetics (pilot sample)
Analyte	PFBA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	90.5	19.8	51.1
2	NT	NT	NT
3	NT	NT	NT
4	136	61	NR
5	80	20	96
6	103.4	2.145	83
7	98	29	NR
8	97	30	91
9	NT	NT	NT
10	NT	NT	NT
11	123	37	25
12	NS	NS	NS
13	73.4	23.7082	48.2
14	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spiked Value	96.2	4.8
Robust Average	100	21
Median	98	16
Mean	100	
N	8	
Max	136	
Min	73.4	
Robust SD	23	
Robust CV	23%	

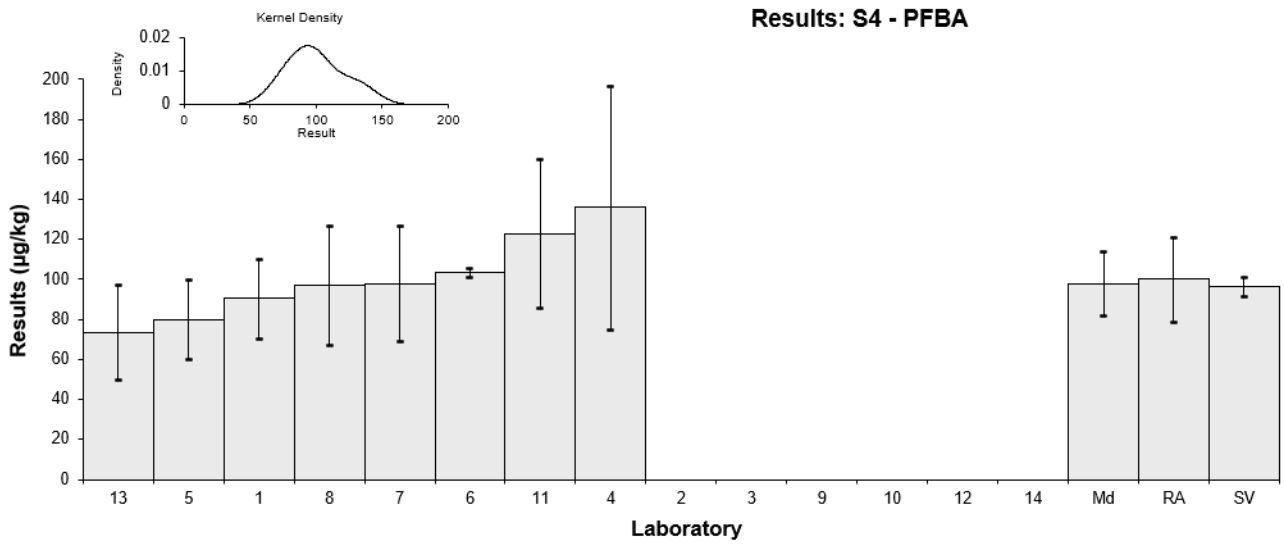

Results: S4 - PFBA
Kernel Density
Density
0.02
0.01
0
0
50
100
150
200
Result
Results (µg/kg)
200
180
160
140
120
100
80
60
40
20
0
13
5
1
8
7
6
11
4
2
3
9
10
12
14
Md
RA
SV
Laboratory

Figure 23

Table 27

Sample Details

Sample No.	S4
Matrix	Cosmetics (pilot sample)
Analyte	PFHxA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	212	49.4	66.9
2	1125	281	NR
3	NT	NT	NT
4	261	117	NR
5	200	49	67
6	223.2	26.6	70
7	239	72	NR
8	220	70	86
9	NT	NT	NT
10	NT	NT	NT
11	260	77.9	64
12	NS	NS	NS
13	186	45.57	46
14	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spiked Value	224	11
Robust Average	232	31
Median	223	29
Mean	330	
N	9	
Max	1125	
Min	186	
Robust SD	37	
Robust CV	16%	

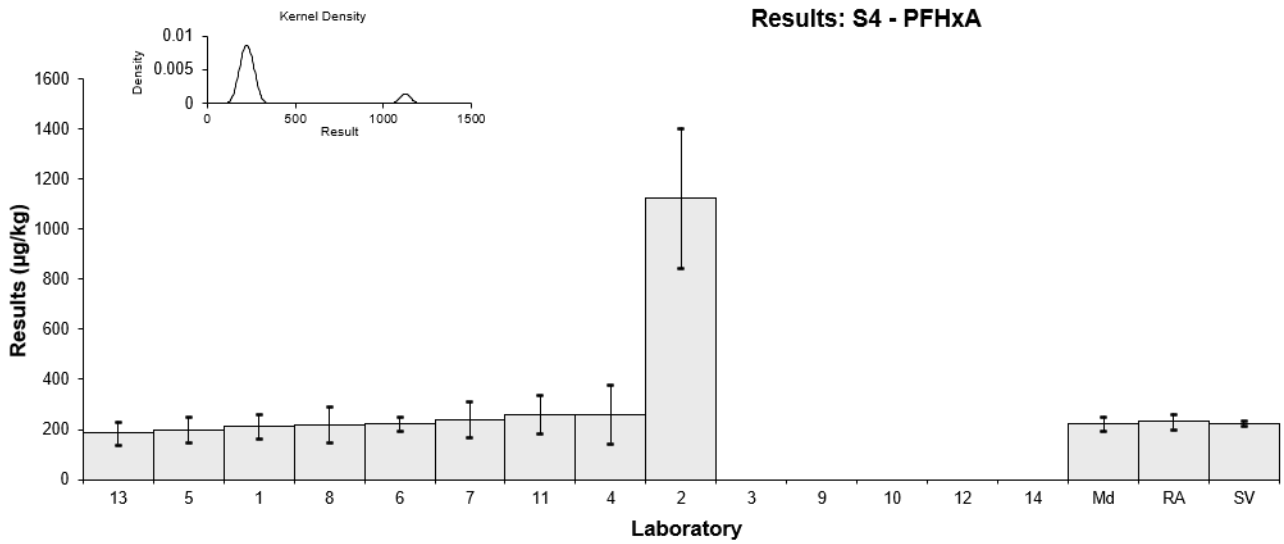

Results: S4 - PFHxA
Kernel Density
Density
0.01
0.005
0
0
500
1000
1500
Result
Results (µg/kg)
1600
1400
1200
1000
800
600
400
200
0
13
5
1
8
6
7
11
4
2
3
9
10
12
14
Md
RA
SV
Laboratory

Figure 24

Table 28

Sample Details

Sample No.	S4
Matrix	Cosmetics (pilot sample)
Analyte	PFOA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	2.33	0.424	61.8
2	22.2	5.6	NR
3	NT	NT	NT
4	3.08	1.386	NR
5	2.0	0.49	86
6	2.78	0.244	66
7	2.2	0.67	NR
8	2	0.6	105
9	NT	NT	NT
10	NT	NT	NT
11	2.2	0.66	90
12	NS	NS	NS
13	2.49	0.54531	64.9
14	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spiked Value	1.93	0.10
Robust Average	2.48	0.44
Median	2.33	0.41
Mean	4.6	
N	9	
Max	22.2	
Min	2	
Robust SD	0.53	
Robust CV	21%	

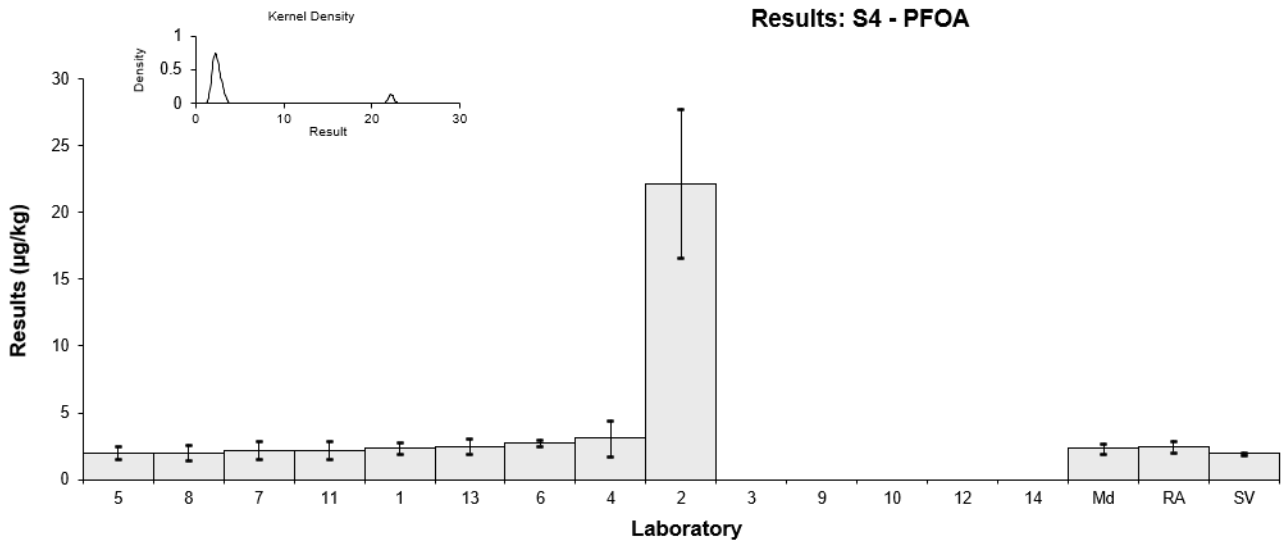

Results: S4 - PFOA
Kernel Density
Density
Result
Results (µg/kg)
Laboratory
5
8
7
11
1
13
6
4
2
3
9
10
12
14
Md
RA
SV

Figure 25

Table 29

Sample Details

Sample No.	S4
Matrix	Cosmetics (pilot sample)
Analyte	PFDoA
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	38	8.02	65.4
2	268	67	NR
3	NT	NT	NT
4	50.9	22.91	NR
5	31	7.8	123
6	40.7	3.6	60
7	38	12	NR
8	36	20	49
9	NT	NT	NT
10	NT	NT	NT
11	51.3	15.3	39
12	NS	NS	NS
13	36.3	6.9696	45.3
14	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spiked Value	38.5	1.9
Robust Average	42.1	8.3
Median	38.0	3.3
Mean	66	
N	9	
Max	268	
Min	31	
Robust SD	9.9	
Robust CV	24%	

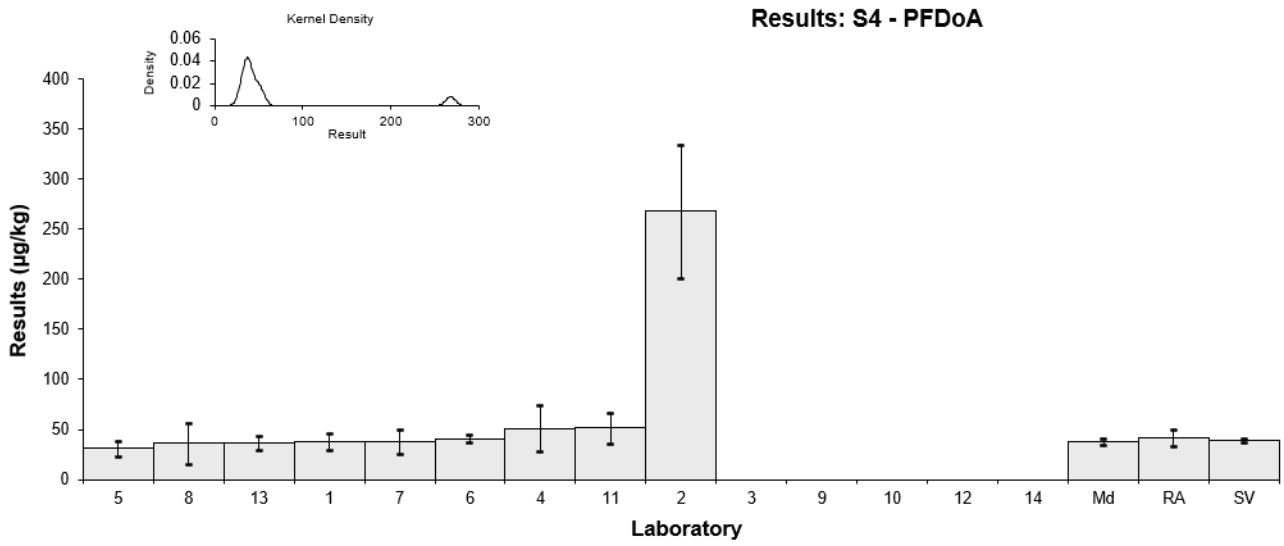

Results: S4 - PFDoA
Kernel Density
Density
0.06
0.04
0.02
0
0
100
200
300
Result
Results (µg/kg)
400
350
300
250
200
150
100
50
0
5
8
13
1
7
6
4
11
2
3
9
10
12
14
Md
RA
SV
Laboratory

Figure 26

Table 30

Sample Details

Sample No.	S4
Matrix	Cosmetics (pilot sample)
Analyte	PFBS
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	5.21	0.99	39.1
2	45.9	11.5	NR
3	NT	NT	NT
4	9.76	4.392	NR
5	4.6	1.1	86
6	5.053	0.427	84
7	4.8	1.4	NR
8	<5	NR	111
9	NT	NT	NT
10	NT	NT	NT
11	5.69	1.7	NR
12	NS	NS	NS
13	3.54	0.67614	70.9
14	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spiked Value	4.85	0.24
Robust Average	6.1	2.5
Median	5.13	0.71
Mean	11	
N	8	
Max	45.9	
Min	3.54	
Robust SD	2.9	
Robust CV	47%	

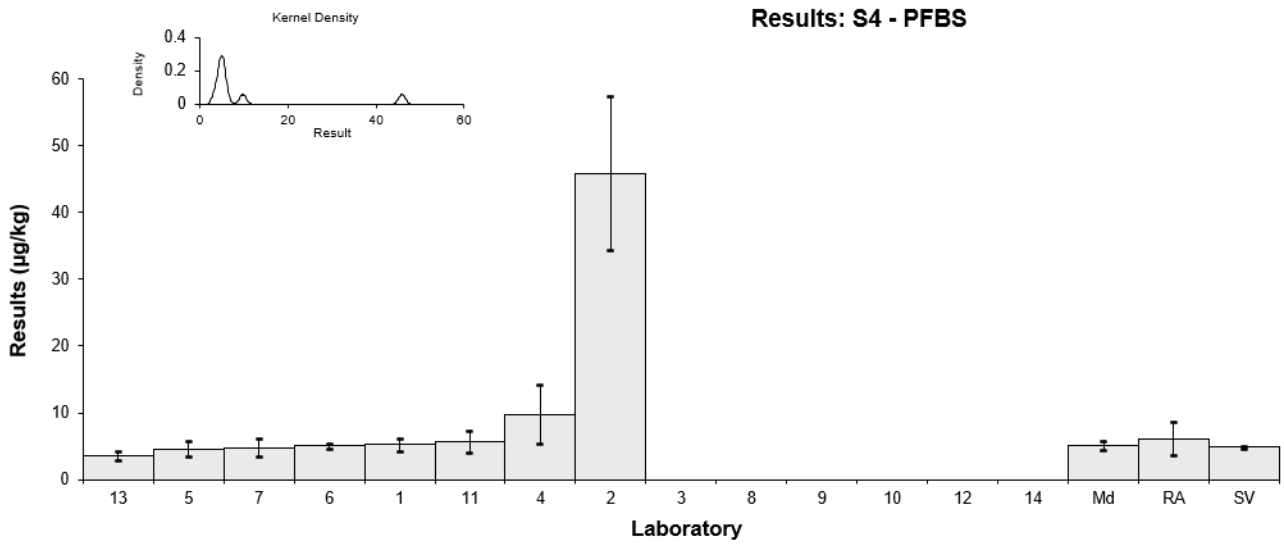

Results: S4 - PFBS
Kernel Density
Density
0.4
0.2
0
0
20
40
60
Result
Results (µg/kg)
60
50
40
30
20
10
0
13
5
7
6
1
11
4
2
3
8
9
10
12
14
Md
RA
SV
Laboratory

Figure 27

Table 31

Sample Details

Sample No.	S4
Matrix	Cosmetics (pilot sample)
Analyte	PFHxS
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	14.9	4.24	39.1
2**	152	38	NR
3	NT	NT	NT
4	24.3	10.94	NR
5	13	3.1	87
6	14.45	1.08	96
7	15	4.6	NR
8	14	5	116
9	NT	NT	NT
10	NT	NT	NT
11	18.6	5.5	77
12	NS	NS	NS
13	9.72	1.80792	65.2
14	NT	NT	NT

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	Not Set	
Spiked Value	14.6	0.7
Robust Average	15.1	3.4
Median	14.7	1.5
Mean	15.5	
N	8	
Max	24.3	
Min	9.72	
Robust SD	3.8	
Robust CV	25%	

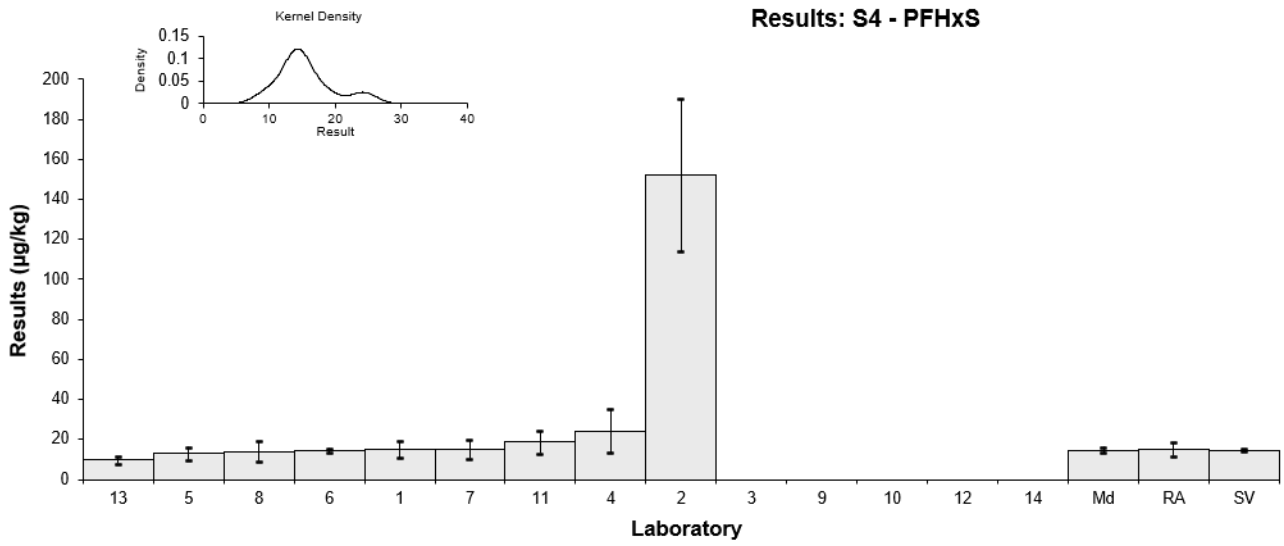

Results: S4 - PFHxS
Kernel Density
Density
0.15
0.1
0.05
0
0
10
20
30
40
Result
Results (µg/kg)
200
180
160
140
120
100
80
60
40
20
0
13
5
8
6
1
7
11
4
2
3
9
10
12
14
Md
RA
SV
Laboratory

Figure 28

Table 32

Sample Details

Sample No.	S4
Matrix	Cosmetics (pilot sample)
Analyte	PFHpS
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	2.83	0.672	39.1
2**	28.7	7.2	NR
3	NT	NT	NT
4	9.37	4.216	NR
5	2.4	0.60	NR
6	3.19	0.29	96
7	2.3	0.68	NR
8	1	0.5	116
9	NT	NT	NT
10	NT	NT	NT
11	3.51	1.05	NR
12	NS	NS	NS
13	2.21	0.32045	64.4
14	NT	NT	NT

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	Not Set	
Spiked Value	Not Spiked	
Robust Average	2.7	1.0
Median	2.62	0.64
Mean	3.4	
N	8	
Max	9.37	
Min	1	
Robust SD	1.2	
Robust CV	43%	

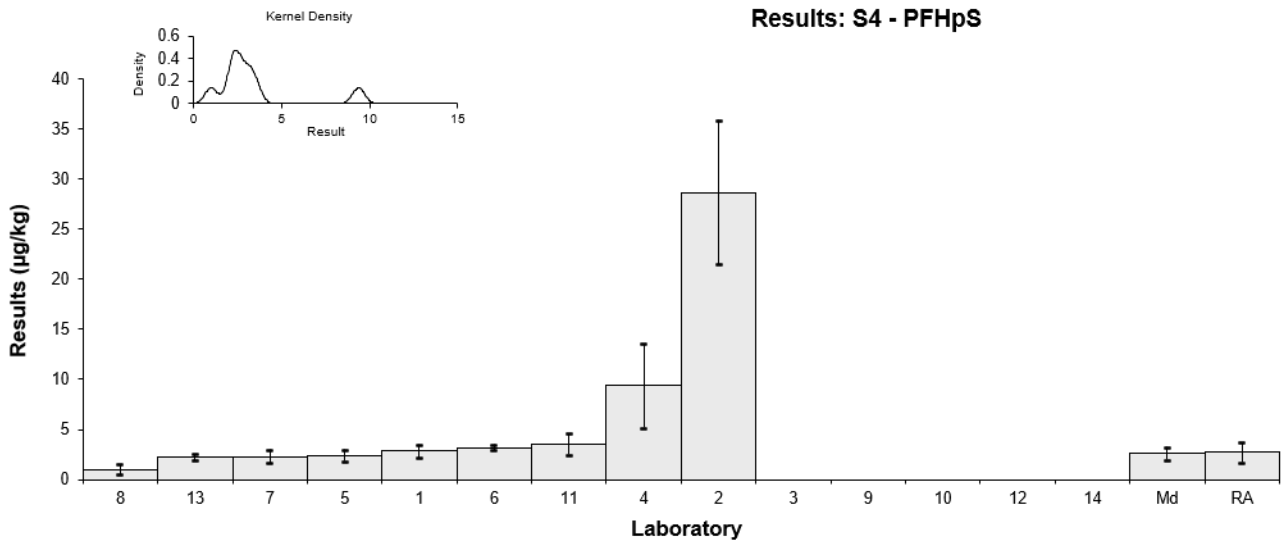

Results: S4 - PFHpS
Kernel Density
Density
Result
Results (µg/kg)
Laboratory
8
13
7
5
1
6
11
4
2
3
9
10
12
14
Md
RA

Figure 29

Table 33

Sample Details

Sample No.	S4
Matrix	Cosmetics (pilot sample)
Analyte	PFOS
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	19.2	4.65	57.4
2	116	29	NR
3	NT	NT	NT
4	41.3	18.59	NR
5	18	4.5	99
6	21.8	1.7	75
7	19	5.7	NR
8	18	6	113
9	NT	NT	NT
10	NT	NT	NT
11	28.3	8.48	55
12	NS	NS	NS
13	17.9	2.6671	64.4
14	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spiked Value	14.4	0.7
Robust Average	25.0	9.4
Median	19.2	1.6
Mean	33	
N	9	
Max	116	
Min	17.9	
Robust SD	11	
Robust CV	45%	

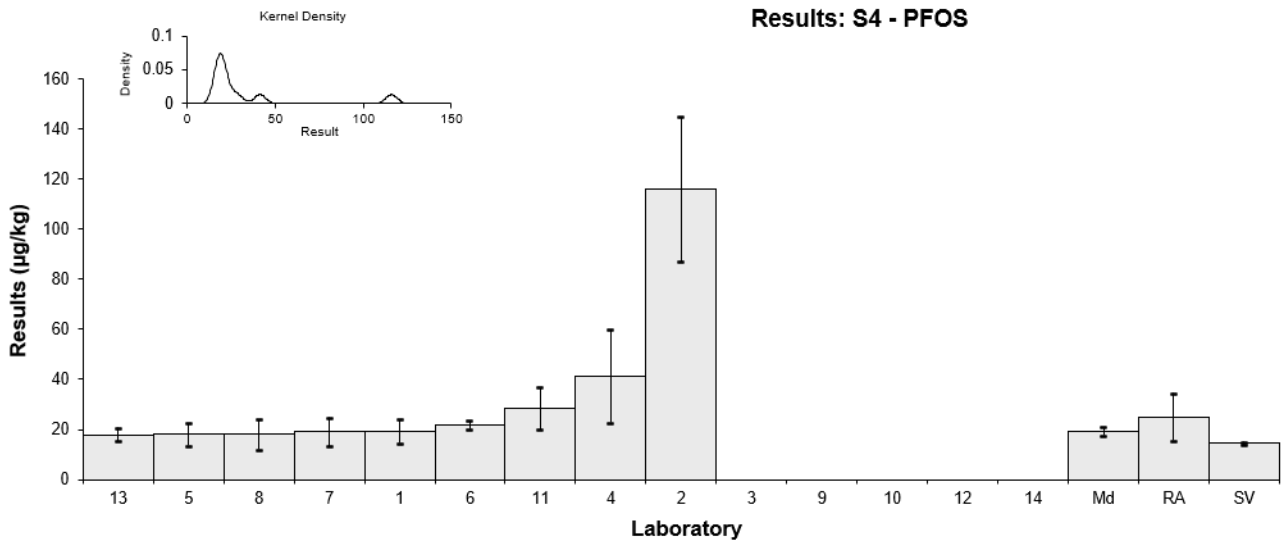

Results: S4 - PFOS
Kernel Density
Density
0.1
0.05
0
0
50
100
150
Result
Results (µg/kg)
160
140
120
100
80
60
40
20
0
13
5
8
7
1
6
11
4
2
3
9
10
12
14
Md
RA
SV
Laboratory

Figure 30

Table 34

Sample Details

Sample No.	S4
Matrix	Cosmetics (pilot sample)
Analyte	6:2FTS
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	42.6	5.75	31
2	NT	NT	NT
3	NT	NT	NT
4**	504	227	NR
5	30	7.5	767
6	40.47	3.35	44
7	35	11	NR
8	48	20	127
9	NT	NT	NT
10	NT	NT	NT
11	42.7	12.8	82
12	NS	NS	NS
13	31	8.06	164
14	NT	NT	NT

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	Not Set	
Spiked Value	38.6	1.9
Robust Average	38.5	7.2
Median	40.5	7.7
Mean	38.5	
N	7	
Max	48	
Min	30	
Robust SD	7.6	
Robust CV	20%	

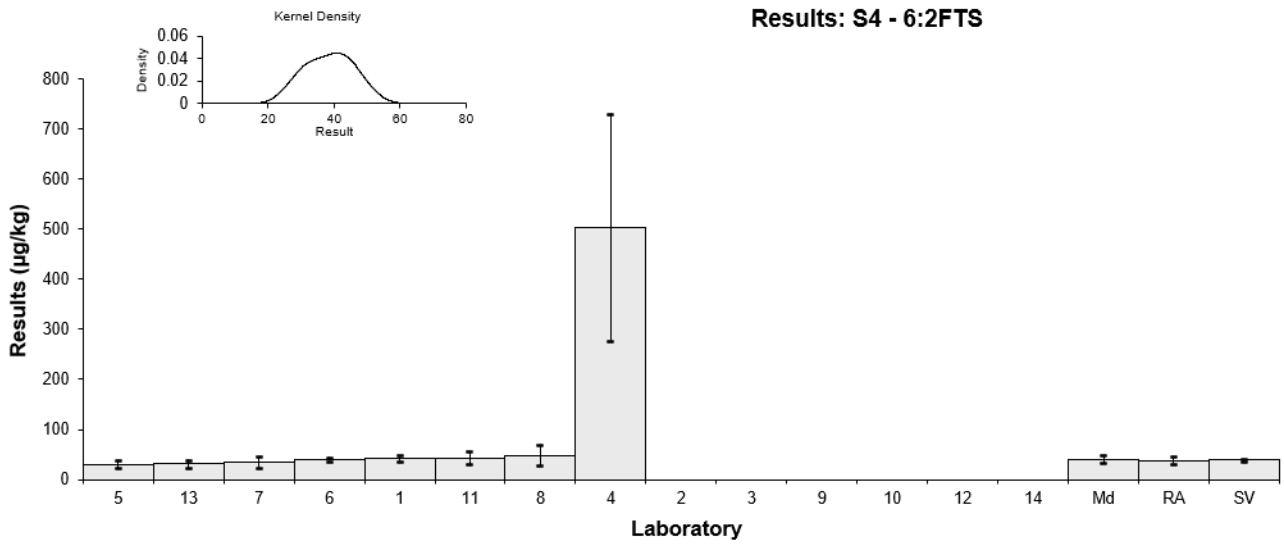

Results: S4 - 6:2FTS
Kernel Density
Density
0.06
0.04
0.02
0
0
20
40
60
80
Result
Results (µg/kg)
800
700
600
500
400
300
200
100
0
5
13
7
6
1
11
8
4
2
3
9
10
12
14
Md
RA
SV
Laboratory

Figure 31

Table 35

Sample Details

Sample No.	S4
Matrix	Cosmetics (pilot sample)
Analyte	8:2diPAP
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	NT	NT	NT
2	NT	NT	NT
3	NT	NT	NT
4	442	199	NR
5	59	15	270
6	67.7	4.9	311
7	73	22	NR
8	NT	NT	NT
9	NT	NT	NT
10	NT	NT	NT
11	NT	NT	NT
12	NS	NS	NS
13	64.8	15.0336	53.4
14	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spiked Value	72.2	3.6
Robust Average	NA (N<6)	
Median	67.7	8.8
Mean	140	
N	5	
Max	442	
Min	59	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

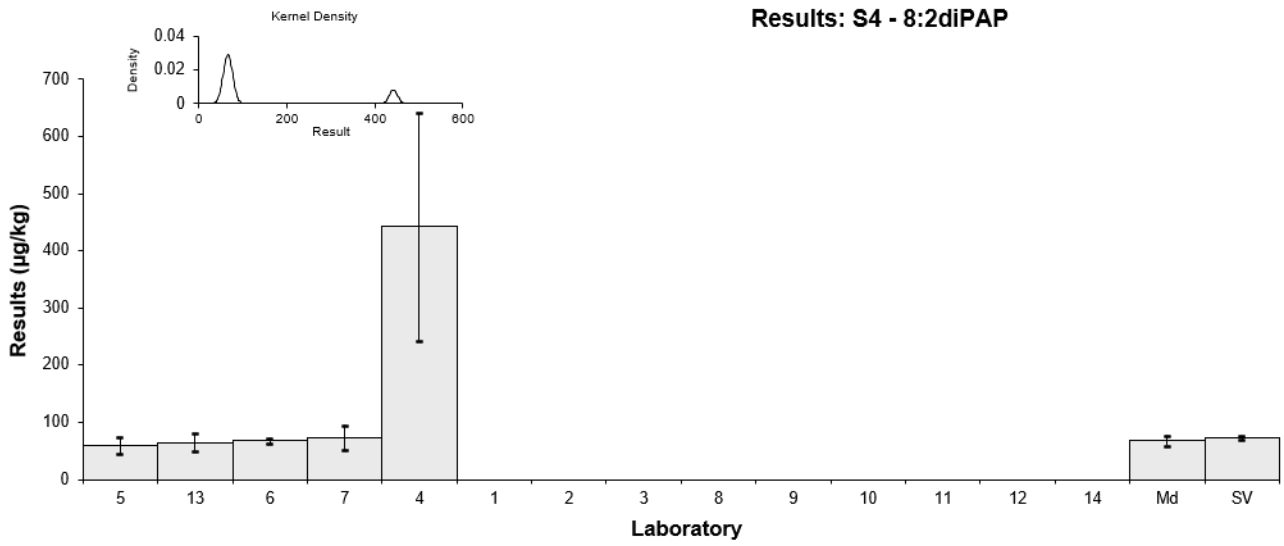

Results: S4 - 8:2diPAP
Kernel Density
Density
0.04
0.02
0
0
200
400
600
Result
Results (µg/kg)
700
600
500
400
300
200
100
0
5
13
6
7
4
1
2
3
8
9
10
11
12
14
Md
SV
Laboratory

Figure 32

Table 36

Sample Details

Sample No.	S4
Matrix	Cosmetics (pilot sample)
Analyte	Extractable Organic Fluorine
Unit	µg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	NT	NT	NT
2	1175	470	NR
3	NT	NT	NT
4	NR	NR	NR
5	4100	1200	NR
6	NT	NT	NT
7	NR	NR	NR
8	NT	NT	NT
9	1500	130	NR
10	NT	NT	NT
11	2800	840	NR
12	NS	NS	NS
13	500	81	NR
14	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spiked Value	5020	250
Robust Average	NA (N<6)	
Median	1500	1700
Mean	2000	
N	5	
Max	4100	
Min	500	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

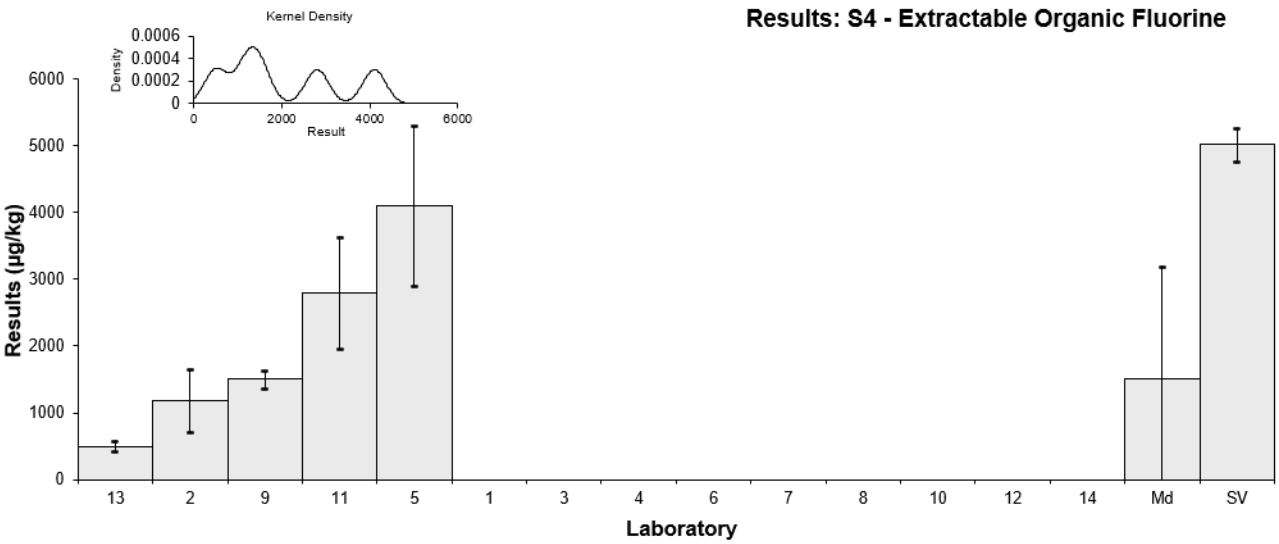

Results: S4 - Extractable Organic Fluorine
Kernel Density
Density
0.0006
0.0004
0.0002
0
0
2000
4000
6000
Result
Results (µg/kg)
6000
5000
4000
3000
2000
1000
0
13
2
9
11
5
1
3
4
6
7
8
10
12
14
Md
SV
Laboratory

Figure 33

Table 37

Sample Details

Sample No.	S4
Matrix	Cosmetics (pilot sample)
Analyte	Total Fluorine
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	Rec
1	NT	NT	NT
2	6.28	2.51	NR
3	48	13	NR
4	NR	NR	NR
5	NT	NT	NT
6	NT	NT	NT
7	NR	NR	NR
8	48	30	NR
9	36	0.5	NR
10	NT	NT	NT
11	48.5	11.4	NR
12	NS	NS	NS
13	32.2	3.4776	NR
14	32	5	NR

Statistics

Assigned Value	Not Set	
Robust Average	38	12
Median	36	17
Mean	36	
N	7	
Max	48.5	
Min	6.28	
Robust SD	13	
Robust CV	34%	

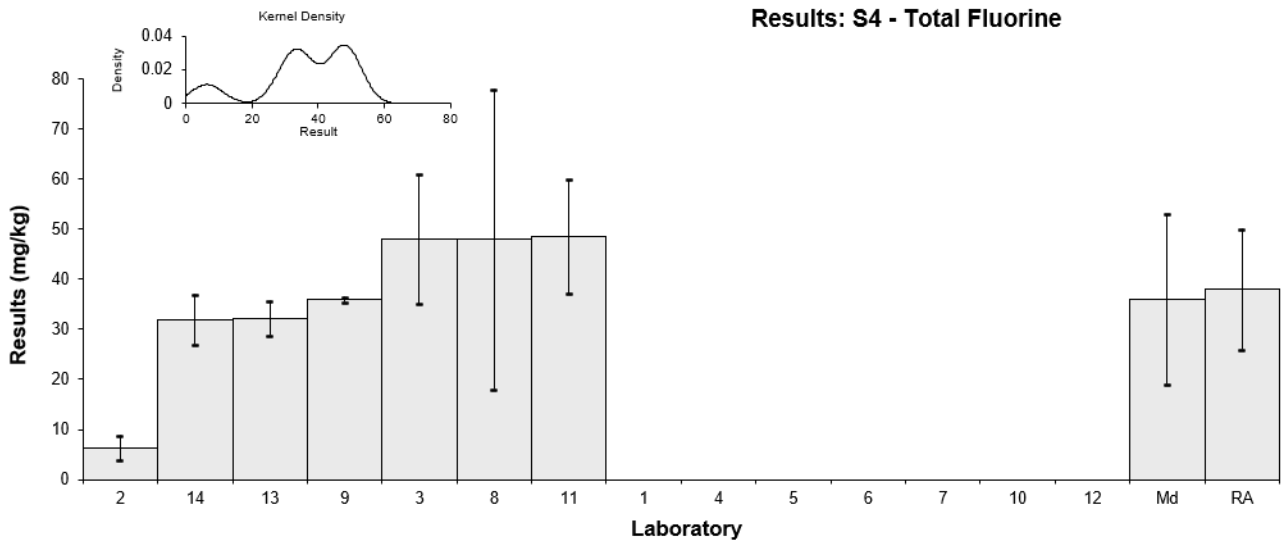

Results: S4 - Total Fluorine
Kernel Density
Density
0.04
0.02
0
0
20
40
60
80
Result
Results (mg/kg)
80
70
60
50
40
30
20
10
0
2
14
13
9
3
8
11
1
4
5
6
7
10
12
Md
RA
Laboratory

Figure 34

6 DISCUSSION OF RESULTS

6.1 Assigned Value

Assigned values for the tests in the study samples were the robust averages of participants' results. The robust averages and their associated expanded uncertainties were calculated using the procedures described in ISO 13528. Results less than 50% or more than 150% of the robust average were removed before calculation of the assigned value.[14, 16] Appendix 2 sets out the calculation for the expanded uncertainty of the robust average of PFBA in Sample S1.

No assigned values were set for PFNA, PFTrDA, PFTeDA, N-EtFOSAA, and 8:2diPAP in Sample S1, either because too few numeric results were reported or because the reported results were too variable. No assigned values were set for the tests in the pilot Samples S2 and S4 as the purpose of these samples was not to assess performance, but to assist laboratories in method development. Participants may compare their results with those reported by other participants, as well as with the spiked values, as presented in Section 5.

A comparison of the assigned or consensus value versus spiked value for all fortified analytes in Sample S1, and pilot Samples S2 and S4 is presented in Table 38. Incurred values are not included in the spiked value, and this may explain some of the high recoveries.

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

Table 38 Comparison of Assigned or Consensus Value and Spiked Value

Sample	Analyte	Assigned or Consensus Value / Spiked Value (%)
S1	PFBA	99
S1	PFPeA	97
S1	PFHxA	77
S1	PFOA	103
S1	PFDA	67
S1	PFUdA	76
S1	PFDoA	66
S1	PFTrDA	63*
S1	PFTeDA	67*
S1	PFBS	90
S1	PFHxS	92
S1	PFOS	94
S1	N-EtFOSAA	91*
S1	6:2FTS	89
S1	8:2FTS	84
S1	8:2diPAP	89*
S1	GenX	95
S1	ADONA	93
S2 (pilot sample)	Extractable Organic Fluorine	94*
S4 (pilot sample)	PFBA	104*
S4 (pilot sample)	PFHxA	100*
S4 (pilot sample)	PFOA	123*
S4 (pilot sample)	PFDoA	105*
S4 (pilot sample)	PFBS	100*
S4 (pilot sample)	PFHxS	97*
S4 (pilot sample)	PFOS	136*

Sample	Analyte	Assigned or Consensus Value / Spiked Value (%)
S4 (pilot sample)	6:2FTS	100*
S4 (pilot sample)	8:2diPAP	94*
S4 (pilot sample)	Extractable Organic Fluorine	30*

*Calculated using the robust average (outliers removed) or median as applicable (assigned value not set).

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an evaluation of the expanded measurement uncertainty (MU) associated with their results and the basis of this evaluation. It is a requirement of ISO/IEC 17025 that laboratories have procedures to evaluate and report the uncertainty of chemical measurements.[18]

Laboratory 13 reported uncertainty as relative uncertainty (x%). Where numerical results were provided, the study coordinator converted the reported uncertainties to absolute expanded uncertainties.

Participation in PT programs allows participants to check that their measurement uncertainty evaluation are reasonable. Results and the expanded MU are presented in the bar charts for each analyte in this study (Figures 2 to 34).

Of 283 numerical results, 282 were reported with an expanded measurement uncertainty. The magnitude of the reported expanded uncertainties was within the range 0.2% to 63% of the reported value. The participants used a wide variety of procedures to evaluate their expanded measurement uncertainties as presented in Tables 2 and 3. One participant reported using the NATA GAG Estimating and Reporting MU as their guide; this document has been officially removed from the NATA website and is considered obsolete.

Laboratories 1, 6, and 9 reported uncertainties lower than 10%. These laboratories should review their procedure for evaluating MU as these uncertainties are considered by the study coordinator to be unrealistically small for a routine PFAS measurement.

Laboratory 8 reported some relative uncertainties greater than 50%. They should also review their MU evaluation as uncertainties greater than 50% might not be fit-for-purpose.

Laboratory 7 attached an evaluation of the expanded MU to a result reported as being less than their limit of reporting. An evaluation of uncertainty expressed as a numerical value cannot be attached to a result expressed as a range.[19]

In some cases, results were reported with an inappropriate number of significant figures. The recommended format is to write uncertainty to no more than 2 significant figures and then to write the result with the corresponding number of decimal places. For example, a result of 4.62 ± 1.16 µg/kg should instead be expressed as 4.6 ± 1.2 µg/kg.[19]

6.3 z-Score

The z-score compares the participant's deviation from the assigned value with the standard deviation for proficiency assessment (SDPA). A SDPA equivalent to 20% PCV was used to calculate z-scores for all scored analytes. Unlike the standard deviation based on between-laboratory CV, setting the SDPA as a realistic set value enables z-scores to be used as fixed reference value points for assessment of individual laboratory performance, independent of the group's performance.

The between-laboratory CVs, CVs predicted by the Thompson-Horwitz equation,[17] and SDPAs as PCVs (when applicable) for scored and non-scored analytes are presented for comparison in Tables 39 and 40 respectively.

Table 39 Comparison of Between-Laboratory CVs, Thompson-Horwitz CVs, and SDPAs for Scored Analytes

Sample	Analyte	Numeric Results Reported*	Assigned Value (µg/kg)	Between-Laboratory CV* (%)	Thompson-Horwitz CV (%)	SPDA (as PCV, %)
S1	PFBA	9	19.9	15	22	20
S1	PFPeA	10	5.04	14	22	20
S1	PFHxA	9	6.5	23	22	20
S1	PFOA	9	1.04	18	22	20
S1	PFDA	10	10.4	16	22	20
S1	PFUdA	10	11.5	18	22	20
S1	PFDoA	7	10.1	29	22	20
S1	PFBS	9	2.75	24	22	20
S1	PFHxS	9	9.4	13	22	20
S1	PFOS	9	14.3	8.7	22	20
S1	6:2FTS	8	41.3	12	22	20
S1	8:2FTS	8	39.6	17	22	20
S1	GenX	9	19.1	22	22	20
S1	ADONA	8	14.2	25	22	20
S3	TF	8	49 (mg/kg)	32	8.9	20

*Values exclude outliers if applicable. Shaded cells represent between-laboratory CVs higher than both the SDPA and the Thompson-Horwitz CV.

Table 40 Comparison of Between-Laboratory CVs and Thompson-Horwitz CVs for Non-Scored Analytes

Sample	Analyte	Numeric Results Reported	Consensus Value* (µg/kg)	Between-Laboratory CV** (%)	Thompson-Horwitz CV (%)
S1	PFNA	4	0.143	9.3	22
S1	PFTrDA	10	9.5	37	22
S1	PFTeDA	9	10.2	57	22
S1	N-EtFOSAA	6	18.3	17	22
S1	8:2diPAP	4	18.0	10	22
S2	EOF	4	4870	17	13
S4	PFBA	8	100	23	22
S4	PFHxA	9	232	16	20
S4	PFOA	9	2.48	21	22
S4	PFDoA	9	42.1	24	22
S4	PFBS	8	6.1	47	22
S4	PFHxS	8	15.1	25	22
S4	PFHpS	8	2.7	43	22
S4	PFOS	9	25.0	45	22
S4	6:2FTS	7	38.5	20	22
S4	8:2diPAP	5	67.7	12	22
S4	EOF	5	1500	NA	15
S4	TF	7	38 (mg/kg)	34	9.3

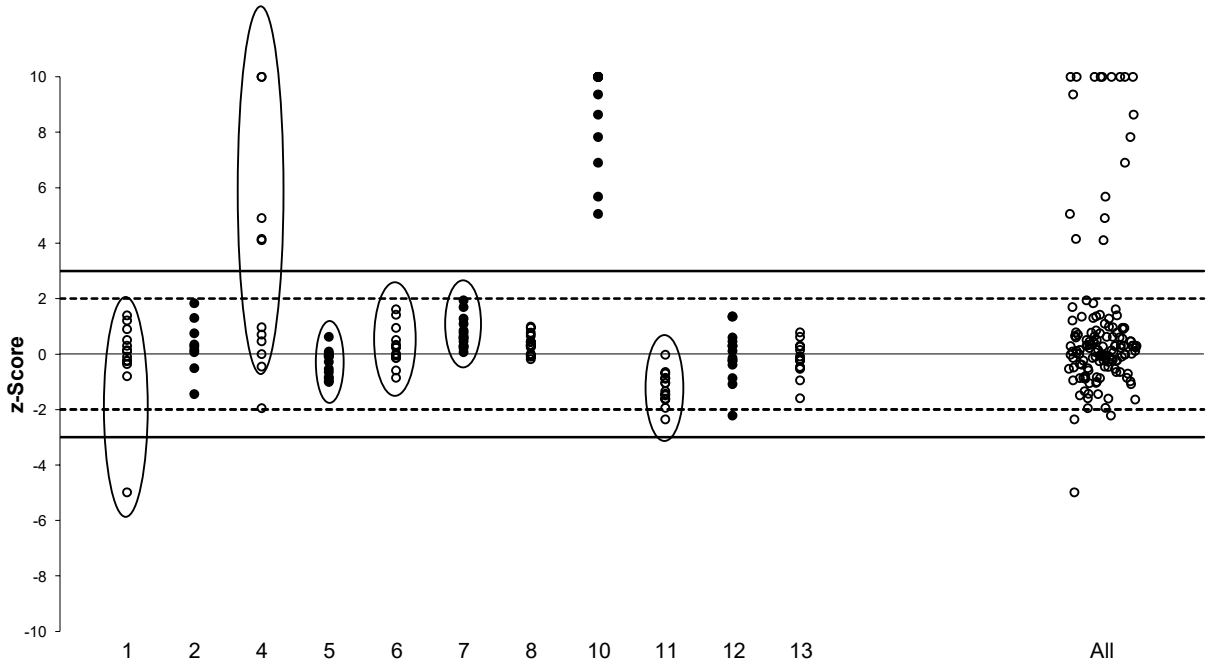
*Robust average or median as applicable (assigned value not set). ** Between Laboratory CV calculated as Robust CV or MADe. Shaded cells represent between-laboratory CVs higher than the Thompson-Horwitz CV. NA-not available

The dispersal of participants' z-scores is graphically presented by laboratory in Figures 35 and 36 and by analyte in Figure 37. Of the 151 results for which z-scores were calculated, 130 (86%) returned an acceptable z-score of $|z| \leq 2.0$ and 2 (1%) were questionable with a z-score of $2.0 < |z| < 3.0$. Participants with multiple z-scores larger than 2.0 or smaller than -2.0 should check for method or laboratory bias.

Laboratories 7, 8, and 13 returned acceptable z-scores for all scored analytes (15).

Laboratories 5 (14), 6 (13), and 2 (9) also received acceptable z-scores for all scored analytes they reported results for.

Laboratories 3, 9, and 14 were only scored for Total Fluorine in Sample S3 and returned acceptable z-scores.



Scores greater than 10 have been plotted as 10. Circled participants did not add labelled standards directly into the sample container (see Section 6.6).

Figure 35 S1 PFAS z-Scores Dispersal by Laboratory

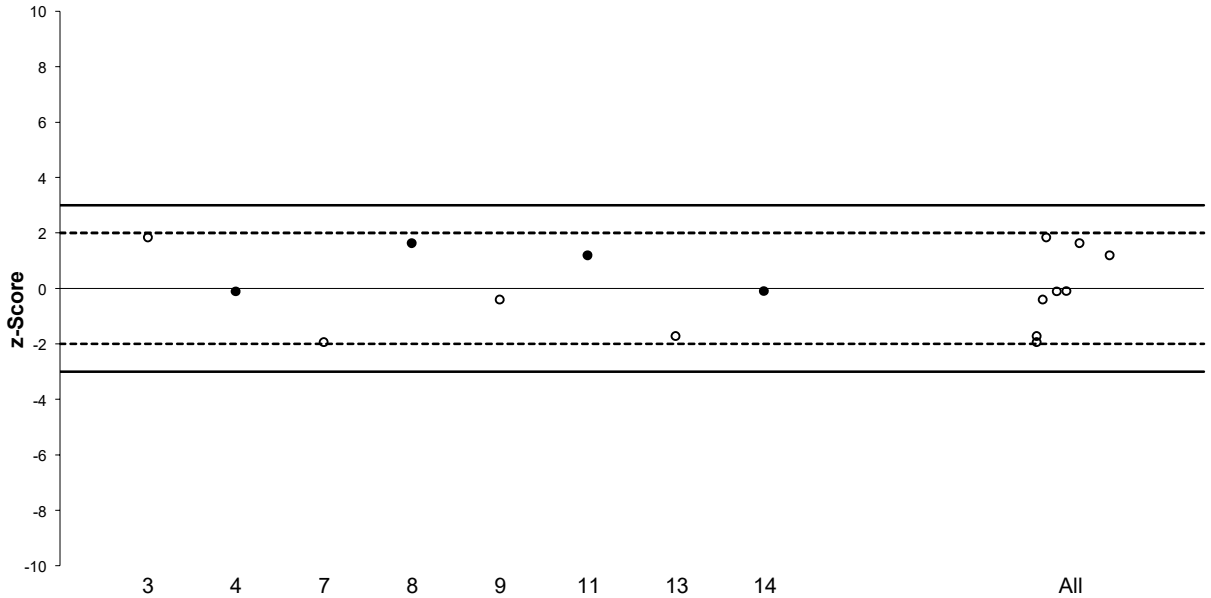
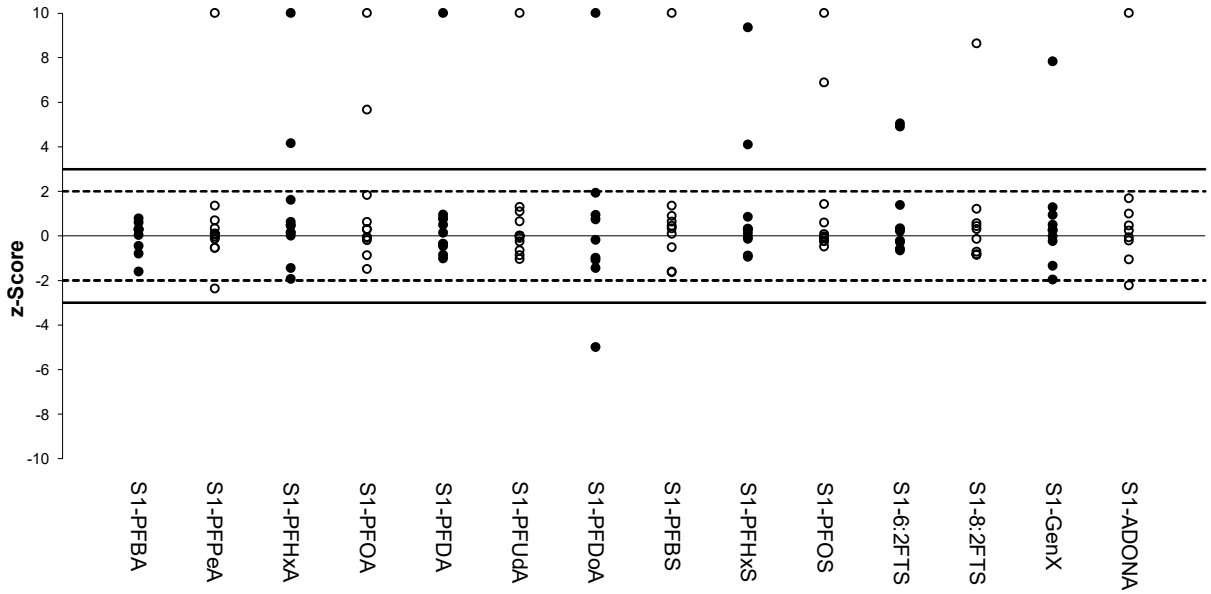


Figure 36 S3 Total Fluorine z-Scores Dispersal by Laboratory



Scores greater than 10 have been plotted as 10.

Figure 37 S1 PFAS z-Scores Dispersal by Analyte

6.4 E_n-Score

The E_n-score indicates how closely a result agrees with the assigned value, taking into consideration the respective uncertainties.

E_n-Scores can be interpreted in conjunction with z-scores, as a not acceptable E_n-score can either be caused by issue with measurement, an inappropriate evaluation of measurement uncertainty, or both.

Where a participant did not report an uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the E_n-score. E_n-Scores were calculated for Laboratory 13 using absolute expanded uncertainties which were converted by the study coordinator from the relative reported measurement uncertainties.

The dispersal of participants' E_n-scores is graphically presented in Figures 38 and 39. Of 151 results for which E_n-scores were calculated, 116 (77%) returned an acceptable score of $|E_n| < 1.0$ indicating agreement of the participants' results with the assigned values within their respective expanded measurement uncertainties.

Laboratory 8 returned acceptable E_n-scores for all 15 scored analytes.

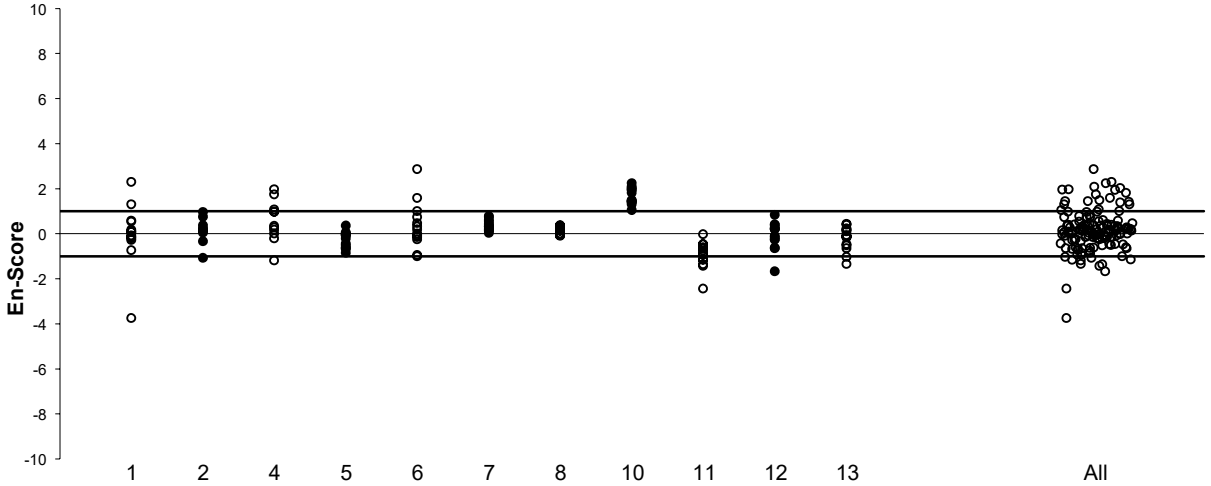


Figure 38 S1 PFAS E_n-Scores Dispersal by Laboratory

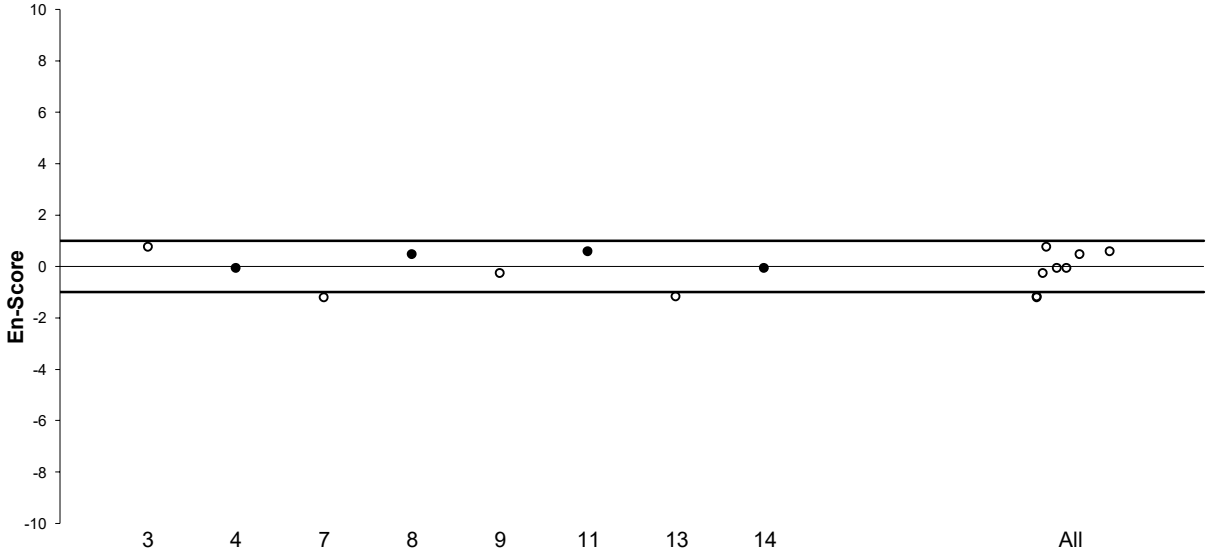


Figure 39 S3 Total Fluorine E_n-Scores Dispersal by Laboratory

6.5 Summary of Participants' Results and Performances

Summaries of participants' results and performance for scored analytes in this PT study are presented in Tables 41 and 42, and Figure 40.

Three laboratories (7, 8, and 13) returned acceptable z-scores for all scored analytes (15).

Laboratory 10 should check for method bias as they returned unacceptably high z-scores for all 13 reported results. Their results were not included in the analyses of extraction methods and of instrumental techniques employed by participants.

Eight laboratories reported at least one PFAS analyte that was not spiked into the test Sample S1. These results are presented in Appendix 3.

Laboratory 4 reported false negative results for 5 analytes (Appendix 4).

Table 41 Summary of Participants' Results and Performance for Sample S1, Pilot Sample S2, and Sample S3

Lab. Code	S1-PFBA (µg/kg)	S1-PFPeA (µg/kg)	S1-PFHxA (µg/kg)	S1-PFOA (µg/kg)	S1-PFNA (µg/kg)	S1-PFDA (µg/kg)	S1-PFUdA (µg/kg)	S1-PFDoA (µg/kg)	S1-PFTrDA (µg/kg)	S1-PFTeDA (µg/kg)	S1-PFBS (µg/kg)
AV	19.9	5.04	6.5	1.04	Not Set	10.4	11.5	10.1	Not Set	Not Set	2.75
1	16.7	5.15	6.67	1.02	0.138	9.65	10.9	0.011	7.46	17	3.24
2	NT	5.38	4.62	1.42	NT	10.7	14.5	11.6	10.5	NT	2.47
3	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
4	18.1	5.75	11.9	4.99	<0.50	12.4	11.5	<0.50	6.26	6.85	<0.50
5	20	4.5	7.3	0.86	<0.1	8.3	10	8.1	9.3	7.6	2.8
6	21.04	4.9	8.59	1.03	0.148	11.45	11.52	NT	NT	NT	2.93
7	21	5.1	7.1	1.1	<1	12	14	14	15	13	3.1
8	21	5	6.5	1	<0.5	12	13	12	9	10	3
9	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
10	NT	15.3	21.5	2.22	NR	42	37	35	25.9	27.5	8.4
11	13.5	2.66	3.98	0.731	< 0.1	8.62	9.11	7.19	8.97	4.61	1.85
12	22.3	6.4	7.1	1.1	0.13	9.6	9.5	7.9	5.9	4.9	3.5
13	23	4.5	6.7	1.17	0.162	9.42	11.3	9.74	7.97	8.96	1.87
14	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS

AV = Assigned Value, NS = Not Sent, NT = Not Tested, NR = Not Reported. Light grey shaded cells are results that were not scored. Dark grey shaded cells are results which returned a questionable or not acceptable z-score.

Table 41 Summary of Participants' Results and Performance for Sample S1, Pilot Sample S2, and Sample S3 (Continued)

Lab. Code	S1-PFHxS (µg/kg)	S1-PFOS (µg/kg)	S1-N-EtFOSAA (µg/kg)	S1-6:2FTS (µg/kg)	S1-8:2FTS (µg/kg)	S1-8:2diPAP (µg/kg)	S1-GenX (µg/kg)	S1-ADONA (µg/kg)	S2-Extractable Organic Fluorine (µg/kg)	S3-Total Fluorine (mg/kg)
AV	9.4	14.3	Not Set	41.3	39.6	Not Set	19.1	14.2	Not Set	49
1	9.94	13.6	34.8	52.8	49.2	NT	21	NT	NS	NS
2	9.9	14.5	NT	NT	NT	NT	NT	NT	NS	NS
3	NT	NT	NT	NT	NT	NT	NT	NT	NT	67
4	17.1	127	<0.50	81.8	<0.50	16.4	11.6	15.5	<256	47.9
5	9.3	14	15	39	33	17	19	14	4400	NS
6	9.14	18.37	NT	36.43	32.8	NT	22.7	14.84	NS	NS
7	11	16	19	43	44	22	24	19	NS	30
8	10	14	18	44	43	NT	20	17	NT	65
9	NS	NS	NS	NS	NS	NS	NS	NS	4200	45
10	27	34	NT	83	108	NT	49	115	NS	NS
11	7.72	14.2	16.3	35.9	34	NT	14	11.2	9690	60.8
12	9.6	13.6	NT	43.7	38.5	NT	18.2	7.9	NS	NS
13	7.62	12.9	18.5	39.4	42	18.9	20.1	13.6	5340	32.1
14	NS	NS	NS	NS	NS	NS	NS	NS	NS	48

AV = Assigned Value, NS = Not Sent, NT = Not Tested, NR = Not Reported. Light grey shaded cells are results that were not scored. Dark grey shaded cells are results which returned a questionable or not acceptable z-score.

Table 42 Summary of Participants' Results for Pilot Sample S4

Lab. Code	PFBA (µg/kg)	PFHxA (µg/kg)	PFOA (µg/kg)	PFDoA (µg/kg)	PFBS (µg/kg)	PFHxS (µg/kg)	PFHpS (µg/kg)	PFOS (µg/kg)	6:2FTS (µg/kg)	8:2diPAP (µg/kg)	Extractable Organic Fluorine (µg/kg)	Total Fluorine (mg/kg)
1	90.5	212	2.33	38	5.21	14.9	2.83	19.2	42.6	NT	NT	NT
2	NT	1125	22.2	268	45.9	152	28.7	116	NT	NT	1175	6.28
3	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	48
4	136	261	3.08	50.9	9.76	24.3	9.37	41.3	504	442	NR	NR
5	80	200	2.0	31	4.6	13	2.4	18	30	59	4100	NT
6	103.4	223.2	2.78	40.7	5.053	14.45	3.19	21.8	40.47	67.7	NT	NT
7	98	239	2.2	38	4.8	15	2.3	19	35	73	NR	NR
8	97	220	2	36	<5	14	1	18	48	NT	NT	48
9	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	1500	36
10	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
11	123	260	2.2	51.3	5.69	18.6	3.51	28.3	42.7	NT	2800	48.5
12	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS	NS
13	73.4	186	2.49	36.3	3.54	9.72	2.21	17.9	31	64.8	500	32.2
14	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	32

NS = Not Sent, NT = Not Tested, NR = Not Reported. Light grey shaded cells are results that were not scored.

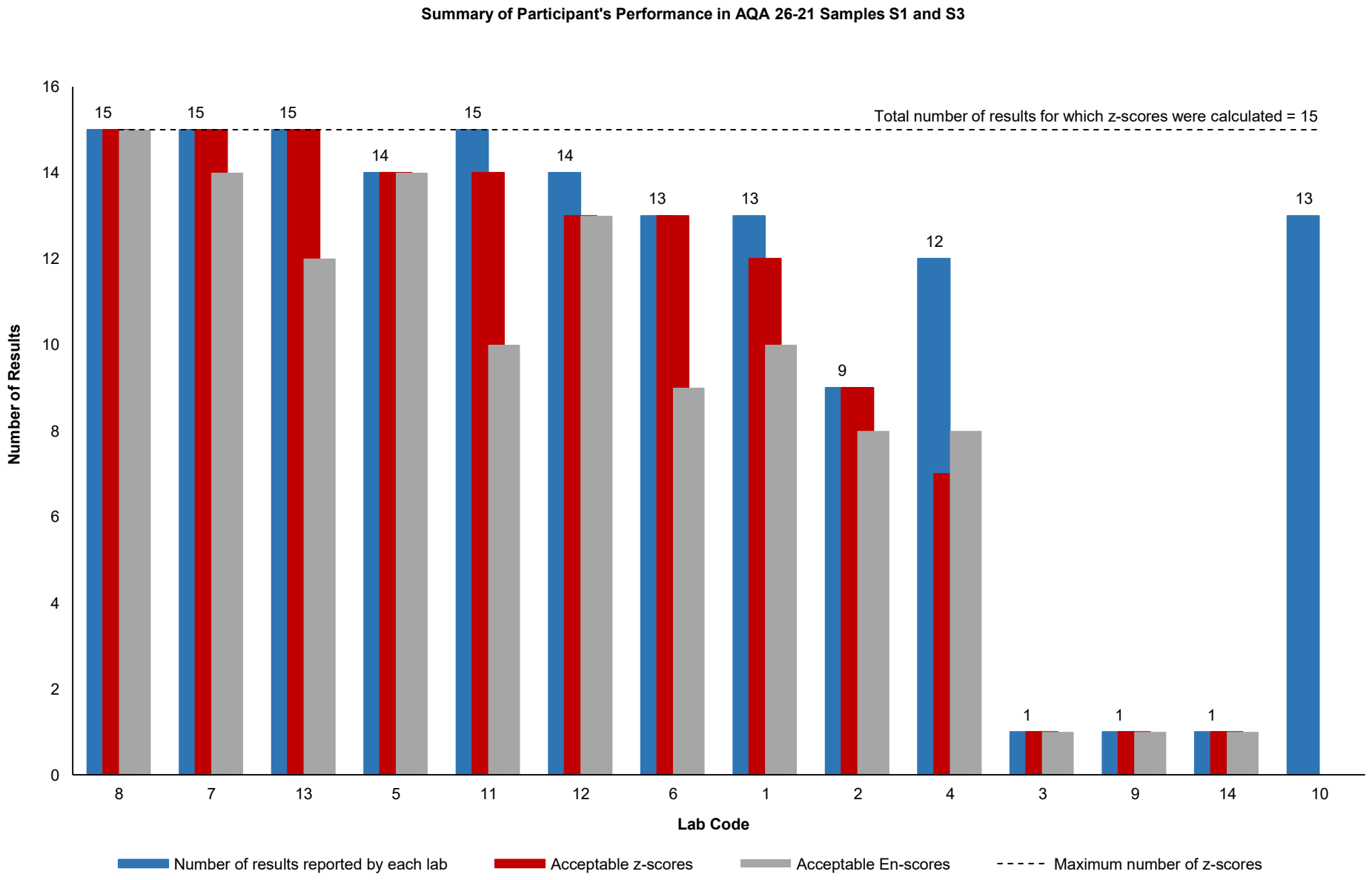


Figure 40 Summary of Participants' Performance in Samples S1 and S3

6.6 Participants' Results and Analytical Methods for PFAS in Cardboard Packaging Material Sample S1

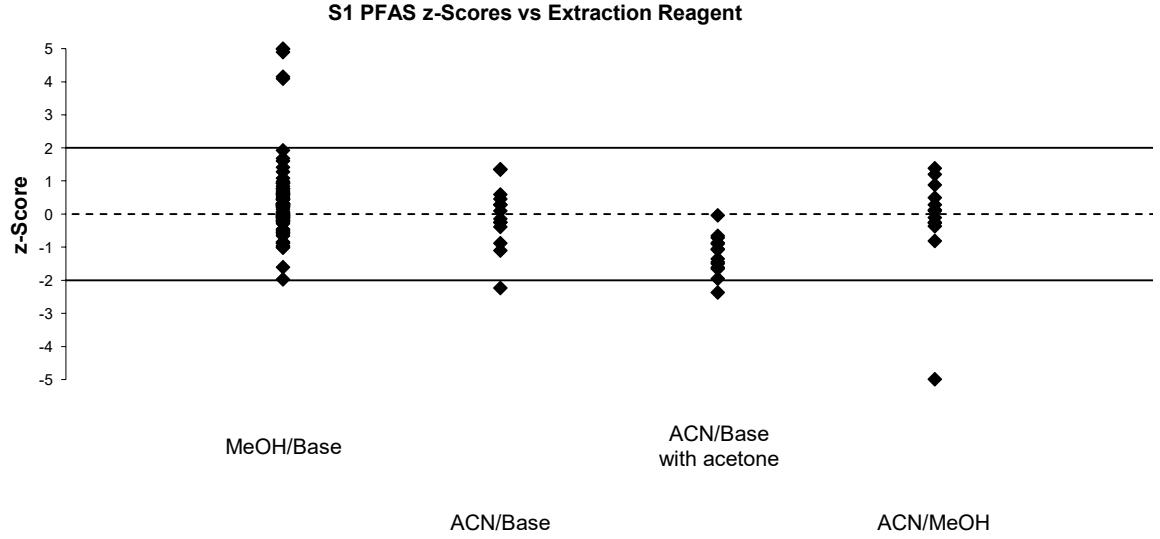
Sample S1 was a cardboard packaging material fortified with 19 individual PFAS compounds. The method descriptions provided by participants for PFAS measurements are presented in Appendix 6.

Participants were requested to analyse the samples using their normal test method but "to use the entire sample and to rinse the sample container with the reagent used for extraction". Two identical containers with the test sample were sent to participating laboratories for repeat analysis if required. All participants who reported results for PFAS in S1 reported using the entire container and rinsing.

Of 10 laboratories who reported results for PFAS in Sample S1, 6 did not add isotopically labelled internal standards directly into the original container to account for potential errors introduced during sample preparation.[20] Some results were biased high or low (Figure 35).

Equilibration times for internal standards varied between half a minute to 30 minutes. Two laboratories reported no equilibration time for internal standards. No trend was evident between the participant's results and the equilibration time reported.

Figure 41 presents plots of participants' performance versus the extraction reagent used. Base modified methanol was the preferred extraction reagent.



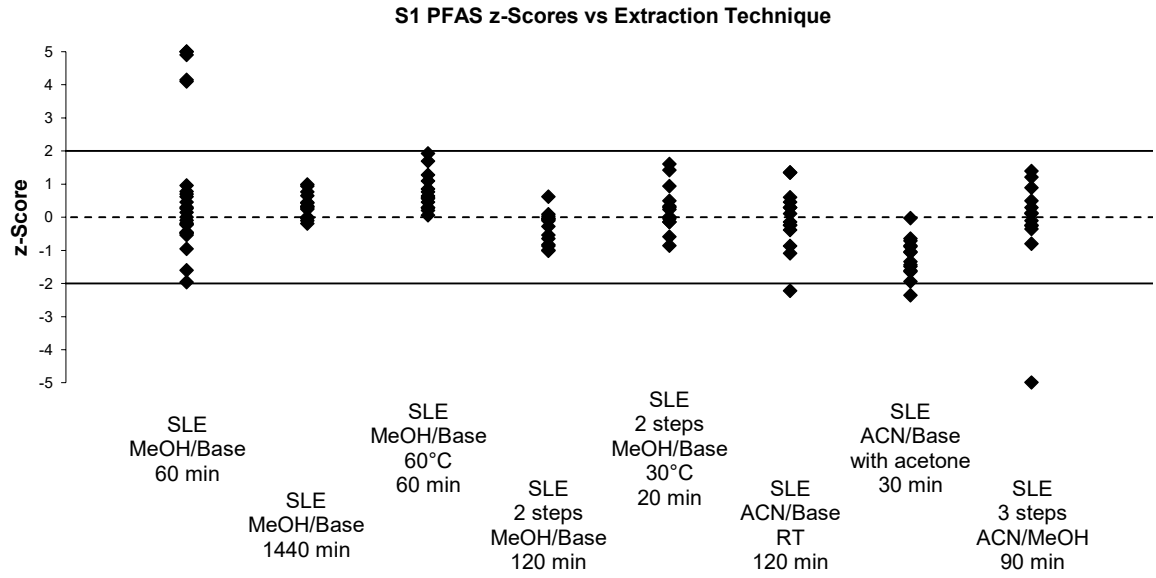
Scores greater than 5 have been plotted as 5.

Figure 41 S1 PFAS z-Scores vs Extraction Reagent

Plots of participants performance versus the extraction technique are presented in Figure 42. All participants used the Solid Liquid Extraction (SLE) technique. Three participants reported using a staggered extraction: 2 conducted SLE over 2 steps and one laboratory used a staggered SLE over 3 steps.

Only 2 laboratories reported applying heat for extraction. One laboratory extracted the sample at 30°C and one at 60°C. Extraction times ranged between 20 minutes and 1440 minutes.

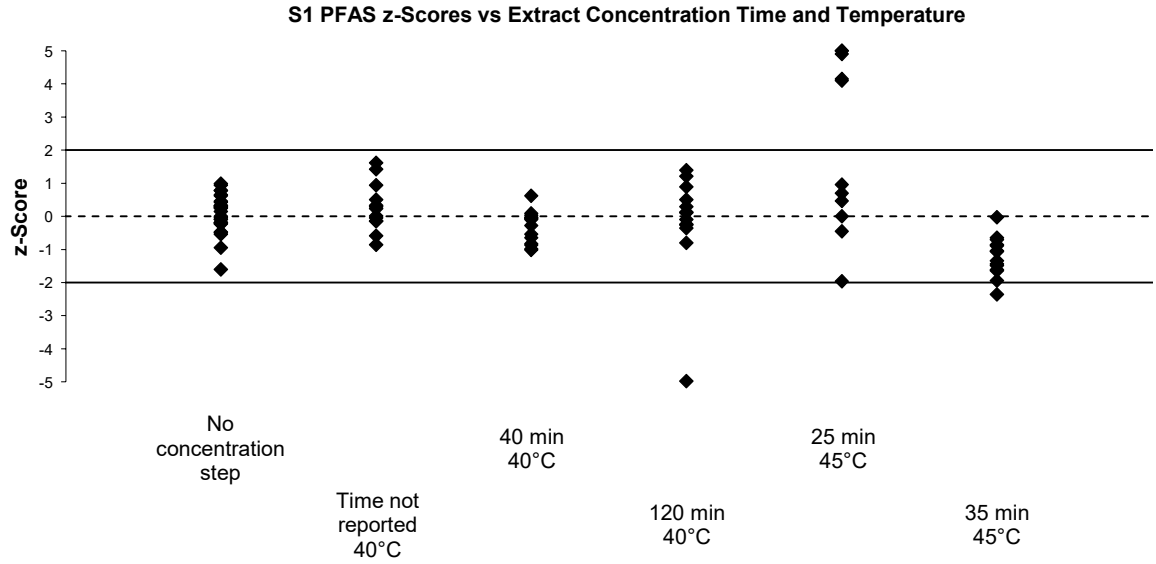
Due to the limited amount of data and wide variety of extraction techniques used, no significant trends were observed between the participant results and the technique used.



Scores greater than 5 have been plotted as 5.

Figure 42 S1 PFAS z-Scores vs Extraction Technique

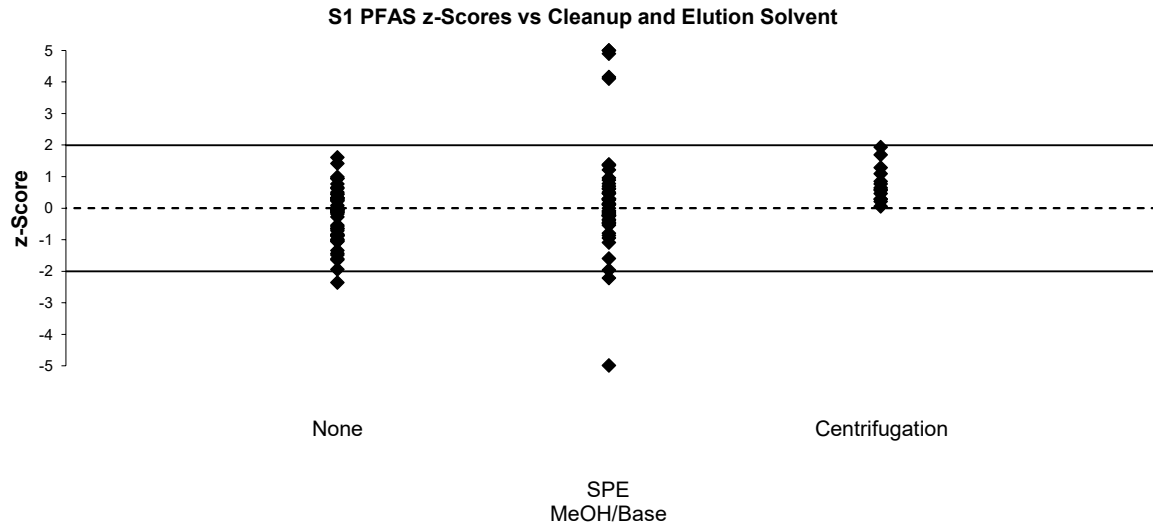
Figure 43 presents plots of participants' performance versus extract concentration time and temperature. All but 3 laboratories reported concentrating their sample extracts using temperatures between 40°C and 45°C.



Scores greater than 5 have been plotted as 5.

Figure 43 S1 PFAS z-Scores vs Extract Concentration Time and Temperature

Cleanup of the crude extracts is an important step in the removal of matrix constituents that may interfere in instrumental determination. Almost half of the laboratories who reported results for PFAS in Sample S1 have not conducted a cleanup step after extraction. There was no significant difference between participant's results who conducted a cleanup step and those who did not (Figure 44).



Scores greater than 5 have been plotted as 5.

Figure 44 S1 PFAS z-Scores vs Extract Cleanup Procedure

The most popular method used for the PFAS analysis in cardboard materials consisted of solid liquid extraction, a sample size of 3 g (entire sample as per instructions), methanol base as extraction solvent, 40°C to 45°C for extract concentration, and no cleanup.

LC-MS/MS was the instrumental technique of choice for all participants.

6.6.1 Individual PFCA Analytes in Packaging Material

The between-laboratory CVs for PFCA analytes in Sample S1 varied from 9.3% to 57%.

PFNA was an incurred analyte in the cardboard packaging material. Of 10 laboratories who measured PFAS in Sample S1, only 4 quantified this analyte

PFDA, PFUdA, and PFDoA Participant's performance for these analytes is presented in Figure 45. There may be a relationship between low-biased results and the use of ACN, particularly in the absence of methanol, as the extraction reagent. PFAS extraction from matrices requires strong solvent-matrix disruption and ACN has limited ability to disrupt strongly bound PFAS .[22]

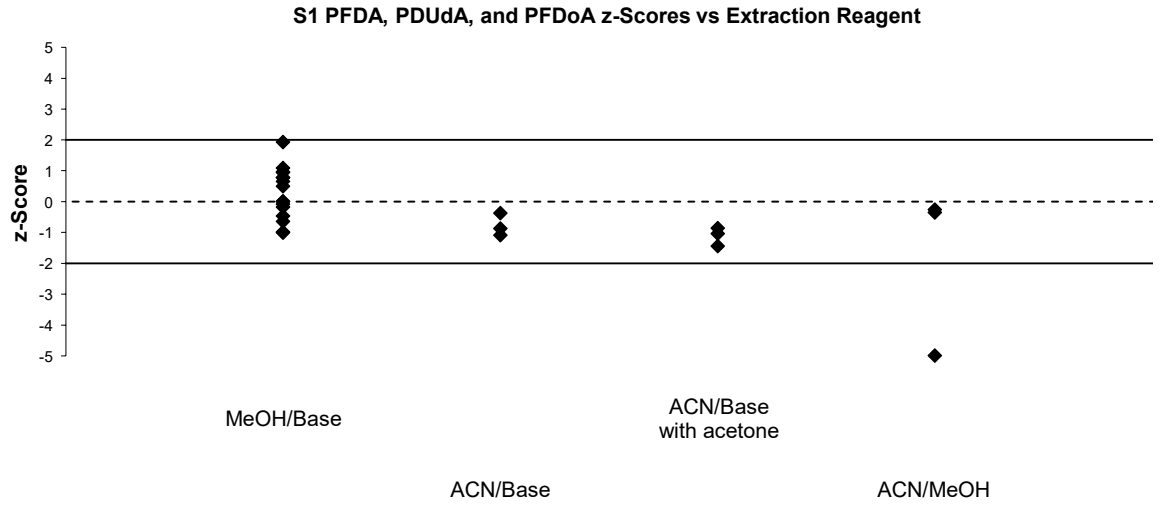


Figure 45 S1 PFDA, PFUdA, and PFDoA z-Scores vs Extraction Reagent

PFTrDA and **PFTeDA** were identified from literature as being analytes which are at risk of being adsorbed into the wall of the container during sample preparation and/or during analysis.[23-28].

No assigned value was set for these analytes as the results were too variable. Plots of participant's results versus extraction reagent are presented in Figures 46 and 47 respectively. No relationship between the results and extraction reagent used was evident. Most results were lower than the spiked value, an indication that losses may have occurred during the sample preparation and/or analysis.

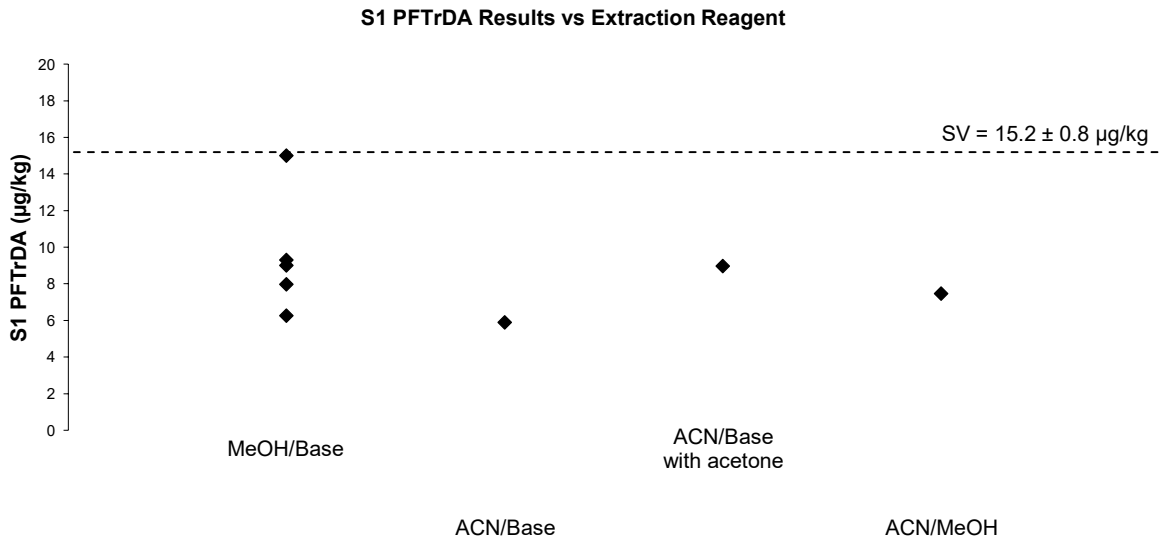


Figure 46 S1 PFTrDA Results vs Extraction Reagent

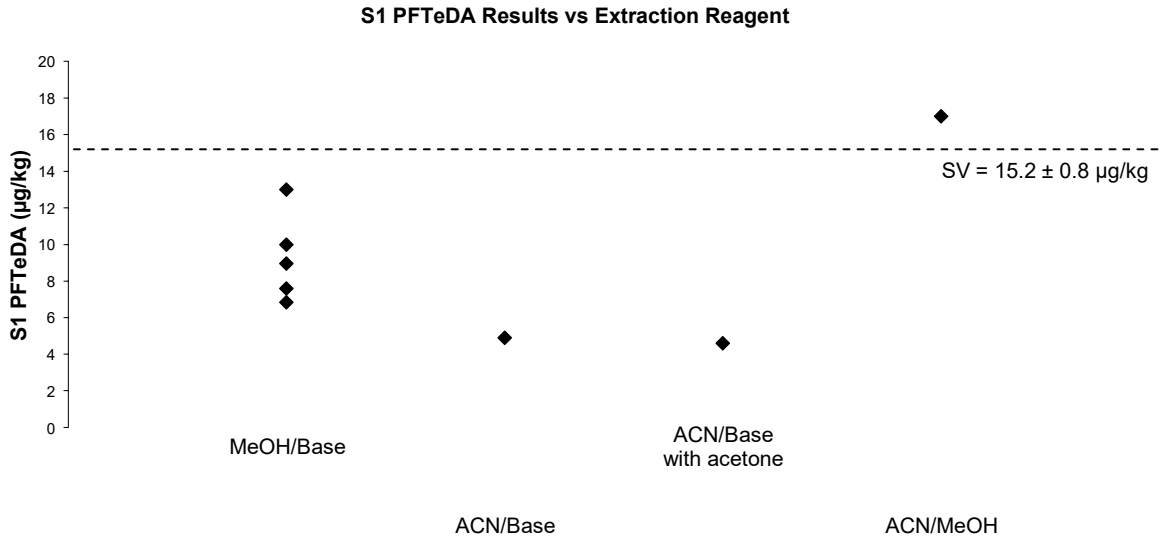


Figure 47 S1 PFTeDA Results vs Extraction Reagent

6.6.2 Individual PFSA Analytes in Packaging Material

PFSAs in Sample S1 did not present significant analytical difficulty to participants. The between-laboratory CVs for scored analytes were between 8.7% and 24%.

6.6.3 Individual PFAA Precursors or Related Compounds in Packaging Material

6:2FTS and 8:2FTS are now in the analytical suite of the majority of participating laboratories. Of 10 participants that reported results for PFAS in Sample S1, all reported results for 6:2FTS and 9 reported results for 8:2FTS. Except for one laboratory, the reported results were in good agreement with each other for both analytes.

Laboratory 4 should review its method for 8:2FTS, as they incorrectly reported the concentration of this analyte in S1 as being below their reporting limit of 0.50 mg/kg.

Plots of participants results versus the method used for 8:2FTS in S1 are presented in Figure 48. The result reported by Laboratory 4 as less than 0.50 mg/kg has been plotted as 0.5 mg/kg.

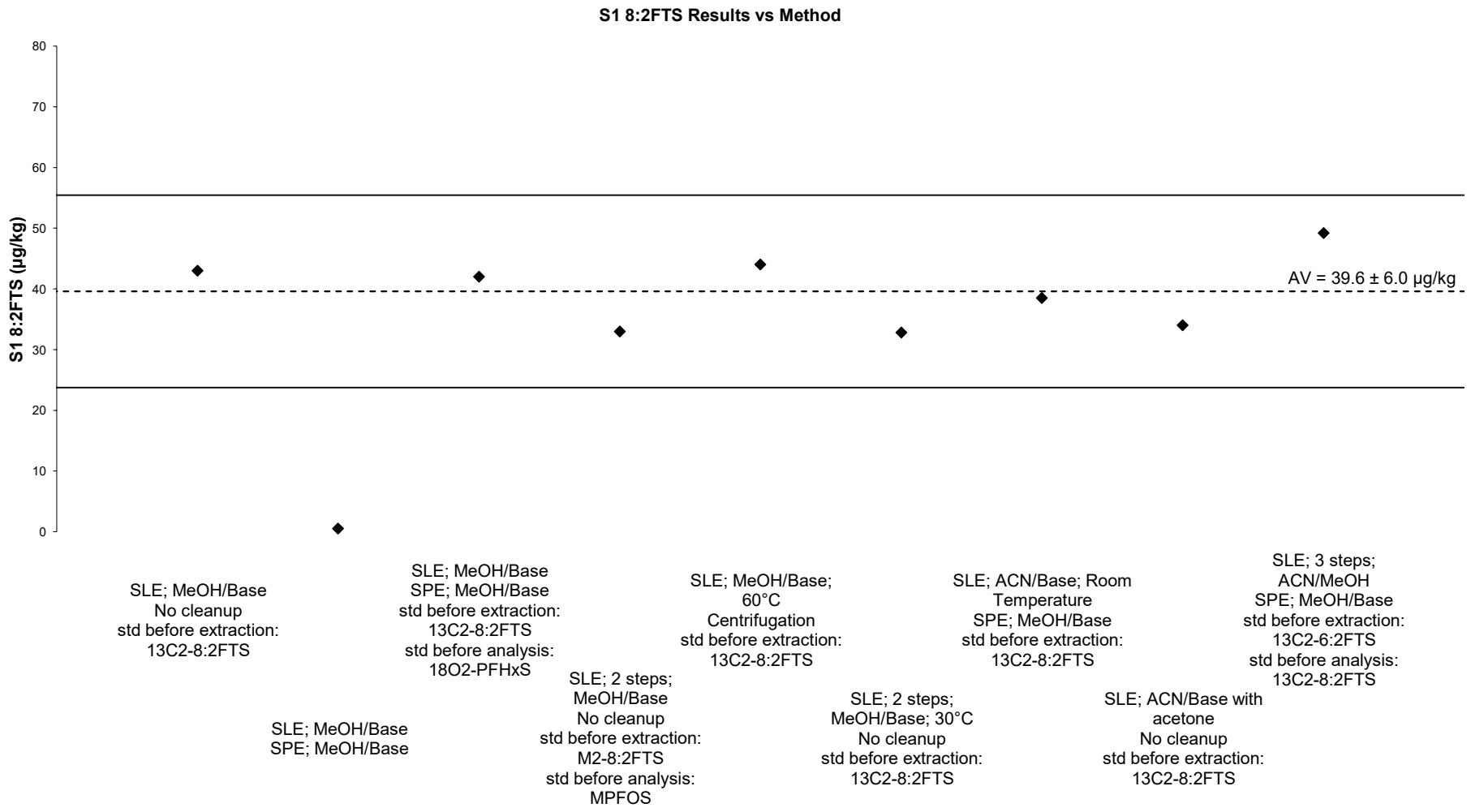
8:2diPAP is primarily used for its surfactant properties in the paper industry and was spiked in the study sample**.** Only 4 laboratories reported results for this analyte, and all reported results were in good agreement with each other. Recoveries of the spiked value were between 81% and 108%.

All laboratories reported adding labelled standards before extraction except one (Figure 49).

N-EtFOSAA is not commonly included in the analytical suite of participating laboratories. Of the 10 participants that reported PFAS results for Sample S1, only six reported results for this analyte. A summary of participants results versus the methods used for analysis are presented in Figure 50.

6.6.4 Individual PFECA Analytes in Packaging Material

PFECAs in Sample S1 did not present significant analytical challenges for participants. All reported results for GenX and ADONA returned satisfactory z-scores except for one (Laboratory 10 excluded). The result reported by Laboratory 12 for ADONA in S1 was approximately half the assigned value, suggesting a possible error in standard preparation or result calculation procedure.



Horizontal lines are equivalent to z-scores of 2 and -2 respectively. The result of less than 0.5 plotted as 0.5.

Figure 48 S1 8:2FTS Results vs Method

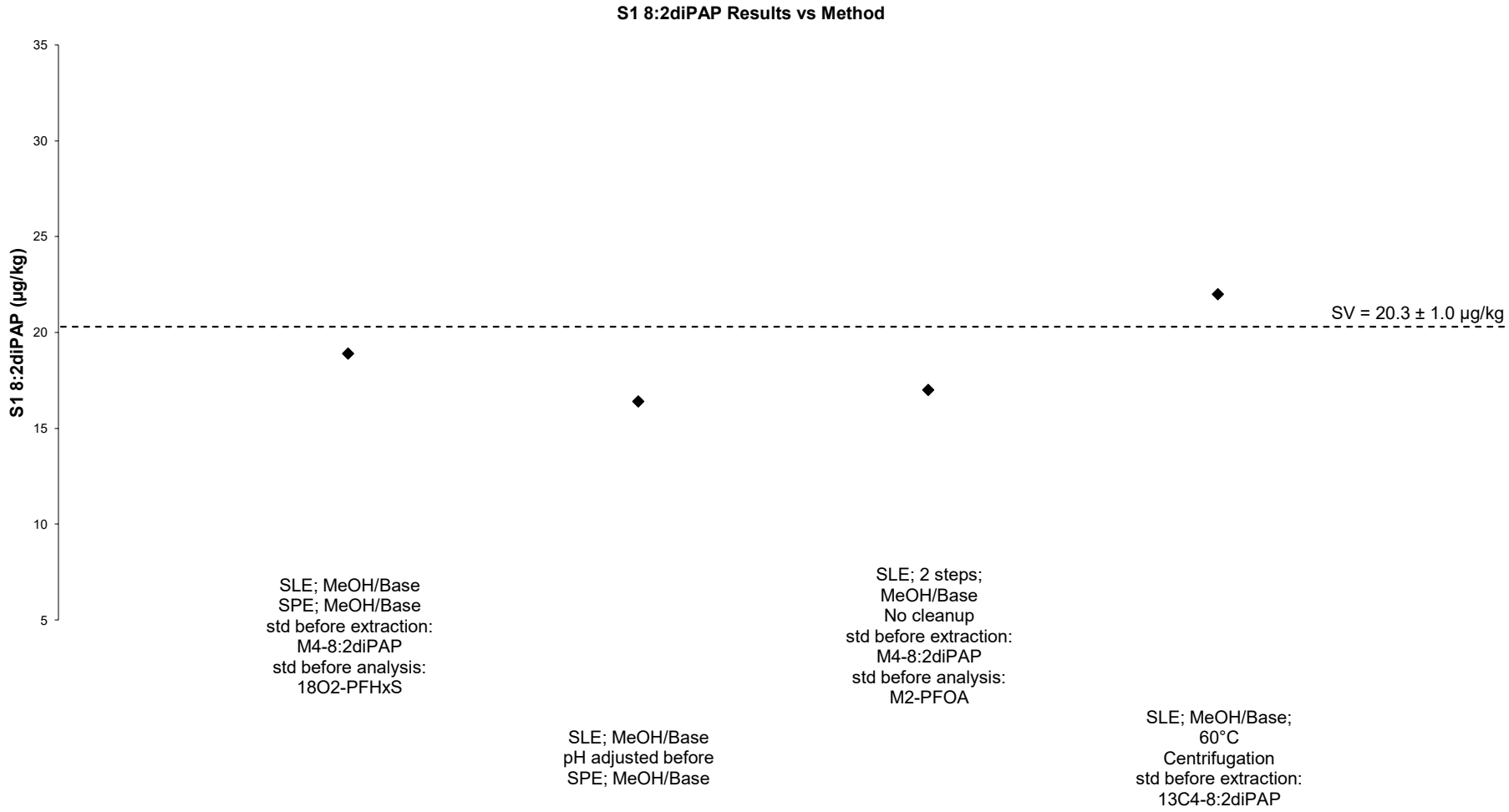


Figure 49 S1 8:2diPAP Results vs Method

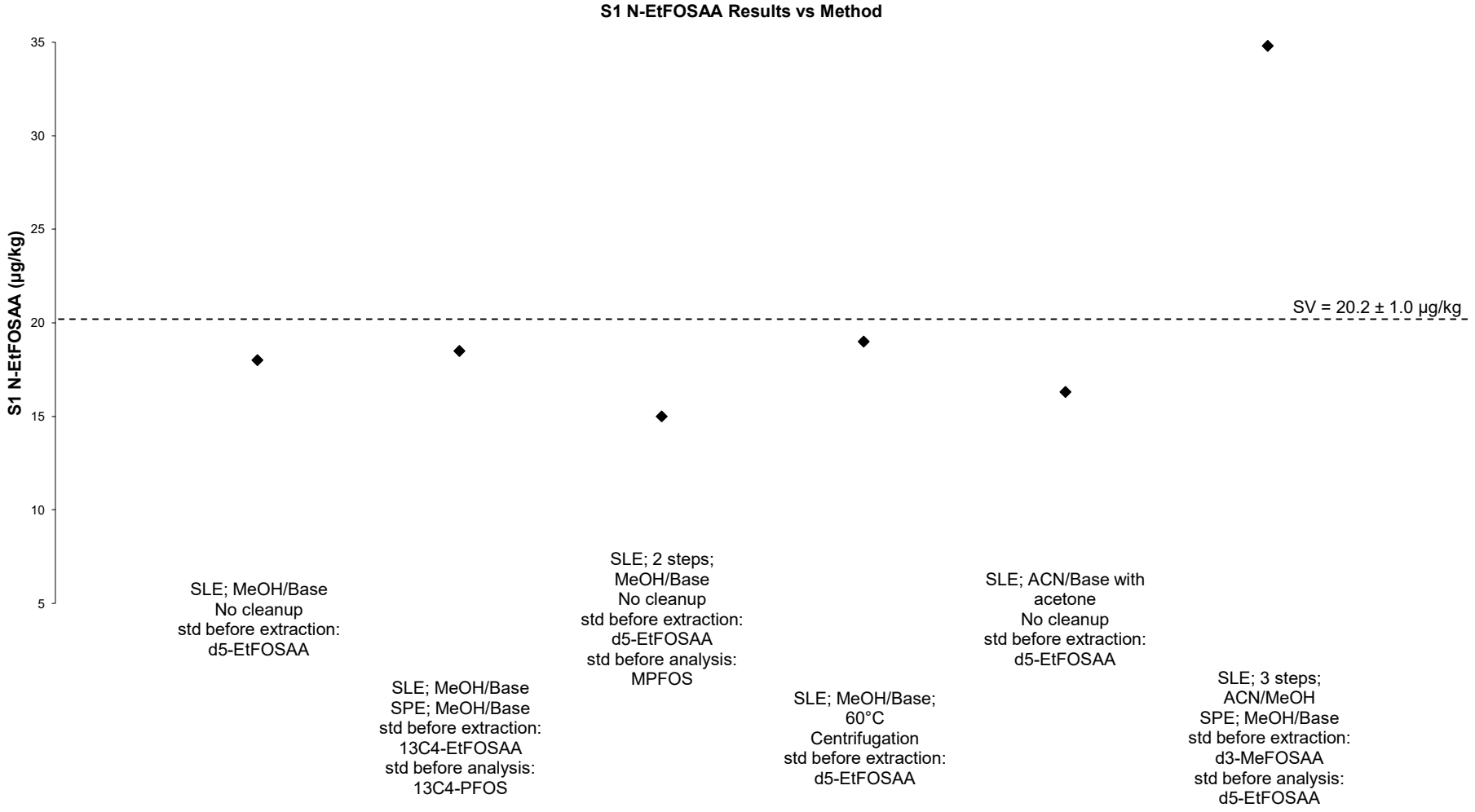


Figure 50 S1 N-EtFOSAA Results vs Method

6.7 Participants' Results and Analytical Methods for Extractable Organic Fluorine in Cardboard

Pilot Sample S2 was the same cardboard packaging material used for Sample S1 preparation. Participants were asked to report results for Extractable Organic Fluorine (EOF). No assessment was conducted for this sample as it was designed to assist laboratories to evaluate their methods for this test in cardboard material. Participants may compare their results with those reported by other participants, as well as the calculated spiked value (Table 24). The expected calculated value for EOF based on the individual PFAS compounds spiked into the sample was 5200 µg/kg.

EOF is not yet routinely tested by analytical laboratories: only 4 participants reported numeric results. One laboratory reported a result as less than their reporting limit at 256 µg/kg. The method descriptions provided by participants is presented in Appendix 6.

All participants who reported numeric results for EOF used the entire container for analysis as instructed.

One laboratory added reagent water before extraction. According to USEPA Method 1633, small amounts of reagent water can be added to unusually dry samples to facilitate extraction.[20] Their result was in good agreement with the calculated spiked value.

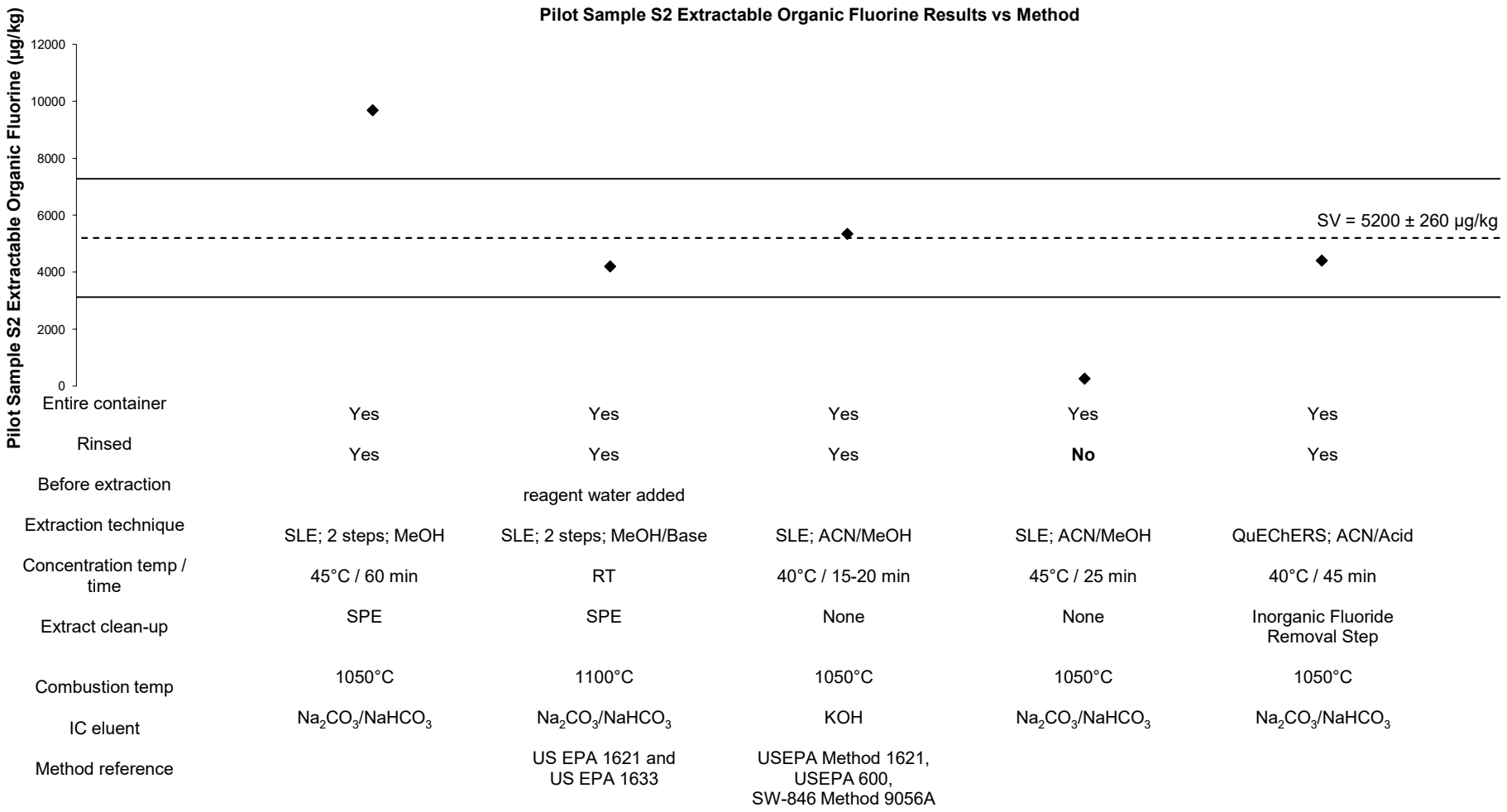
All laboratories used SLE for extraction except one who used QuEChERS. This laboratory reported using an acidified ACN solution as an extraction reagent. Their result was also in good agreement with the calculated spiked value.

All but one laboratory reported concentrating their sample extract at temperatures between 40°C and 45°C. One laboratory concentrated its extract at room temperature. No relationship was evident between participants results and the extract concentration temperature.

One laboratory reported using potassium hydroxide as the eluent. The result reported by this laboratory (5340 µg/kg) was in excellent agreement with the calculated spiked value of 5200 µg/kg. All the other laboratories used a sodium carbonate and sodium bicarbonate eluent.

One laboratory reported a result approximately double the calculated spiked value, an indication of possible calculation errors. This laboratory may want to review their calculation procedure.

Figure 51 presents participants' results versus the method employed. All laboratories used different method parameters. However, 3 of the 4 reported numeric results were in relatively good agreement with each other and with the calculated spiked value



Horizontal lines correspond to good agreement with the spiked value. The result of less than 256 plotted as 256.

Figure 51 Pilot Sample S2 Extractable Organic Fluorine Results vs Method

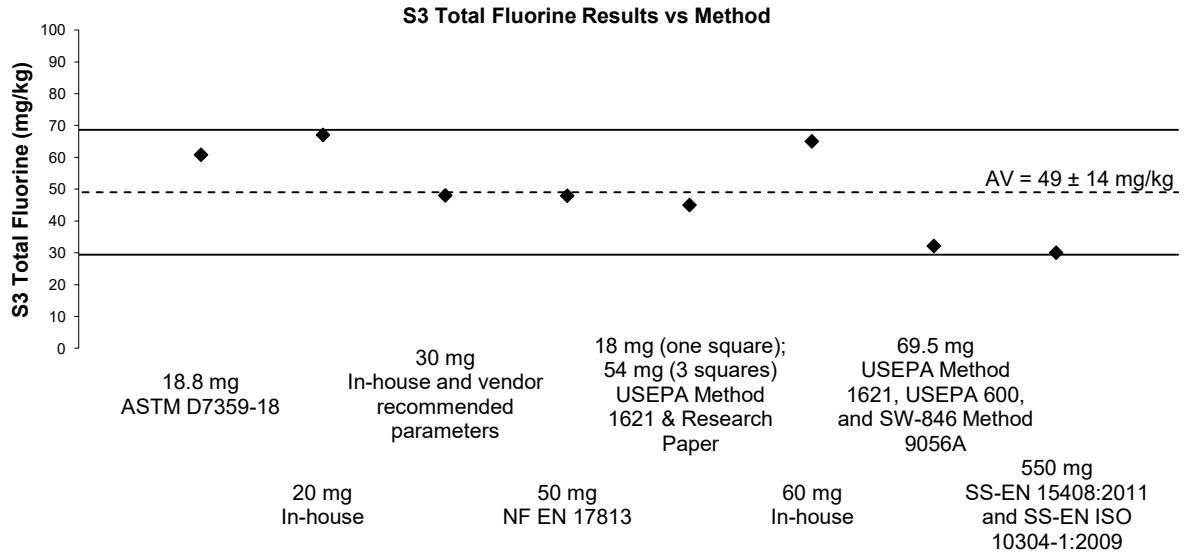
6.8 Participants' Results and Analytical Methods for Total Fluorine in Cardboard

Sample S3 was a cardboard packaging material. The Total Fluorine (TF) in this material was incurred. Participants were requested to analyse the sample using their normal test method and to report a single result as they would normally report to a client.

Eight laboratories reported results for TF in Sample S3. The method descriptions provided by participants are presented in Appendix 6.

Participants used a variety of sample sizes for analysis from 18 mg to 550 mg. One laboratory reported: "Squares cut into thin strips to lie in the bottom of the combustion boats. Three squares was the maximum that would fit. Both weights run in triplicate. Both were greater than reporting limit, so the reported value is the average of n=6". Their reported result of 45 mg/kg was in good agreement with the assigned value (49 mg/kg).

Figure 55 presents participants' performance versus the method employed. All reported results were in good agreement with each other and the assigned value.



Horizontal lines are equivalent to z-scores of 2 and -2 respectively.

Figure 52 S3 Total Fluorine Results vs Method

6.9 Participants' Results and Analytical Methods for PFAS in Cosmetics (Foundation)

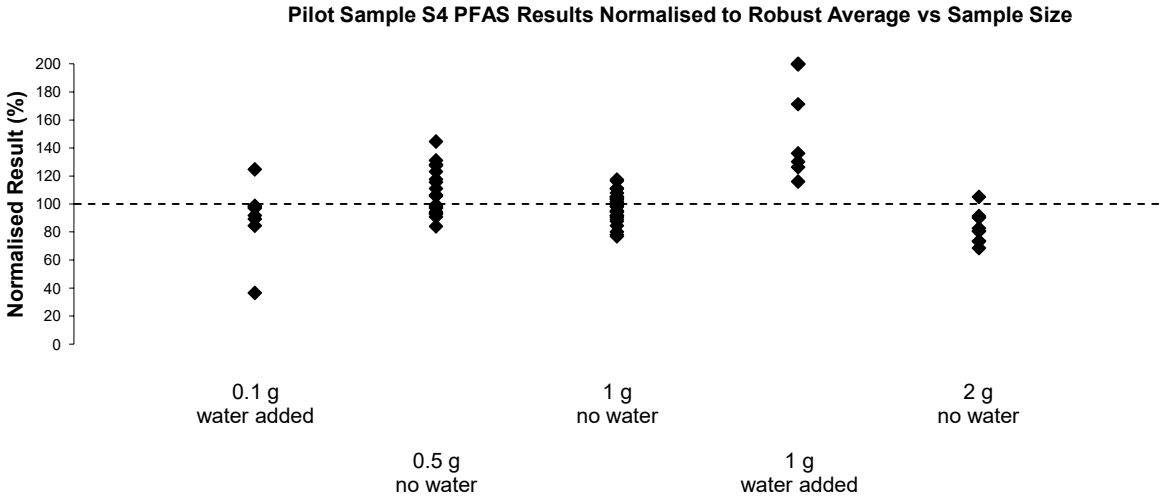
This is the first time that cosmetics (foundation) have been introduced in a NMIA PFAS PT study. This was a pilot sample aimed at assisting laboratories to develop their methods for PFAS in this matrix, and no assessment was conducted. Participants can evaluate their results against other participants' results and/or the spiked values (Tables 26 to 37). Where applicable they may also evaluate whether their reported results fall within the compatibility range (determined as the robust average excluding outliers, with a PCV of 20%) as suggested by the study coordinator in Appendix 5. Most reported results were in good agreement with each other and spiked value.

The sample was spiked for 10 individual PFAS components. Participating laboratories were asked to report results for individual PFAS using their routine method. The method descriptions provided by them is presented in Appendix 6.

Nine laboratories reported results for PFAS analytes in pilot Sample S4. Three of them reported using an analytical method similar to the one used for the cardboard packaging

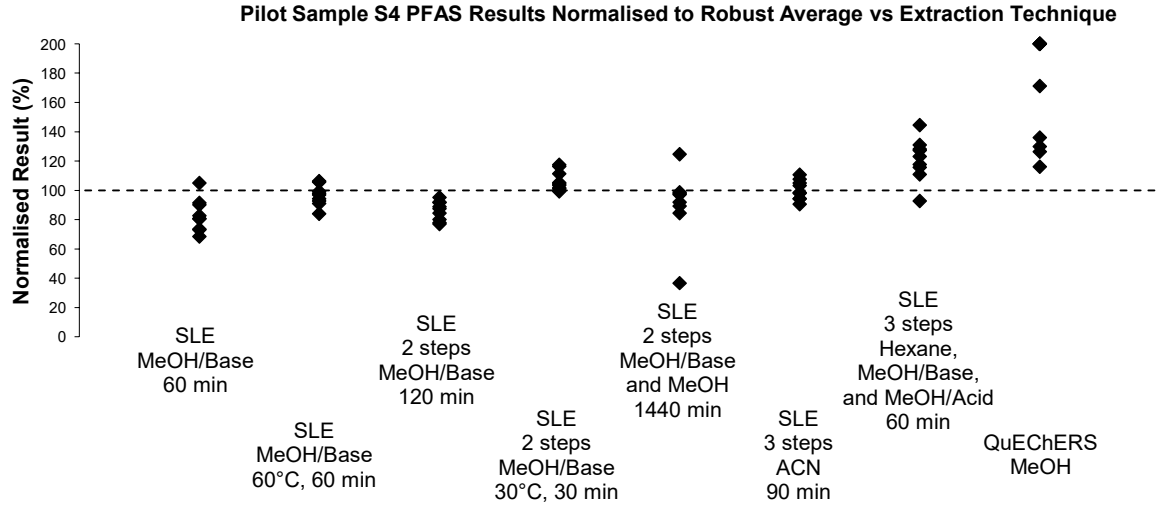
material. For the other 6 laboratories, changes included: QuEChERS as extraction method instead of SLE, different extraction solvents, extra extraction steps, or cleanup of crude extract.

As no performance was assessed, plots of participants' results normalised to robust average (outliers excluded) were used to evaluate the methods. Figure 53 presents plots of results normalised to robust average versus sample size.



Normalised results greater than 200% plotted as 200%.

Figure 53 Pilot Sample S4 PFAS Results Normalised to Robust Average vs Sample Size



Normalised results greater than 200% plotted as 200%.

Figure 54 Pilot Sample S4 PFAS Results Normalised to Robust Average vs Extraction Technique

Of 9 participants who reported results for PFAS in pilot Sample S4, 2 reported adding reagent water to facilitate extraction.[20] No significant differences between laboratories who did and did not add water were observed.

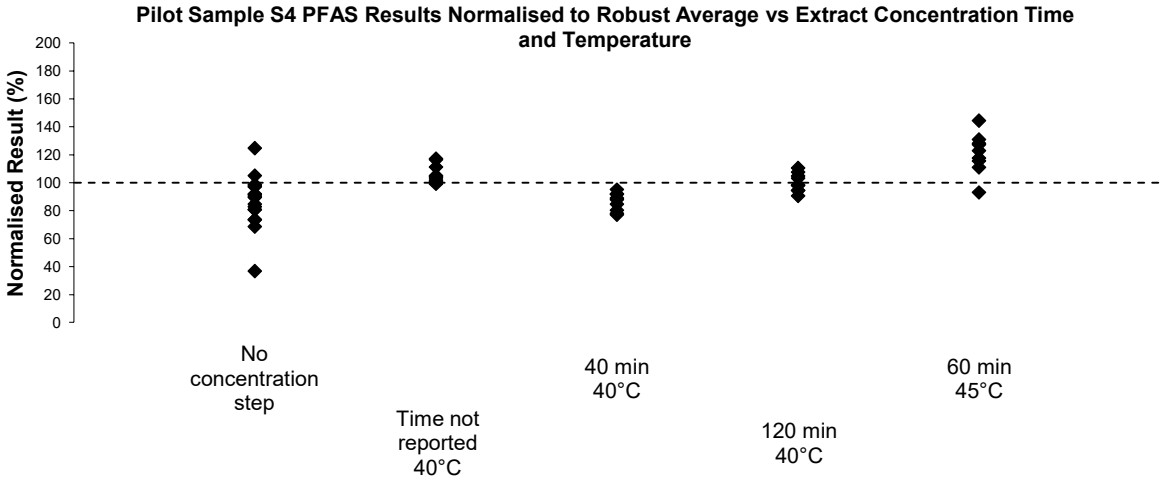
Figure 54 presents an overview of the methods employed by laboratories for PFAS extraction in cosmetics (foundation). Similarly to packaging material, all participants used the Solid Liquid Extraction (SLE) technique except one. One laboratory reported using QuEChERS,

Results obtained using the QuEChERS approach were generally biased high; however, these results were reported by a single laboratory only. Therefore, it is unclear whether the observed bias reflects a method-related effect or is attributable to laboratory-specific bias.

Most laboratories used basified methanol as extraction reagent. One participant reported conducting their extraction over 3 steps, each time using a different reagent: hexane, base modified methanol, and acid modified methanol. The USEPA's Method for Analysis of PFAS in Oily Matrix recommends the use of hexane to wash away the oily matrix.[29] The results from this laboratory were biased higher than the robust averages and spiked values.

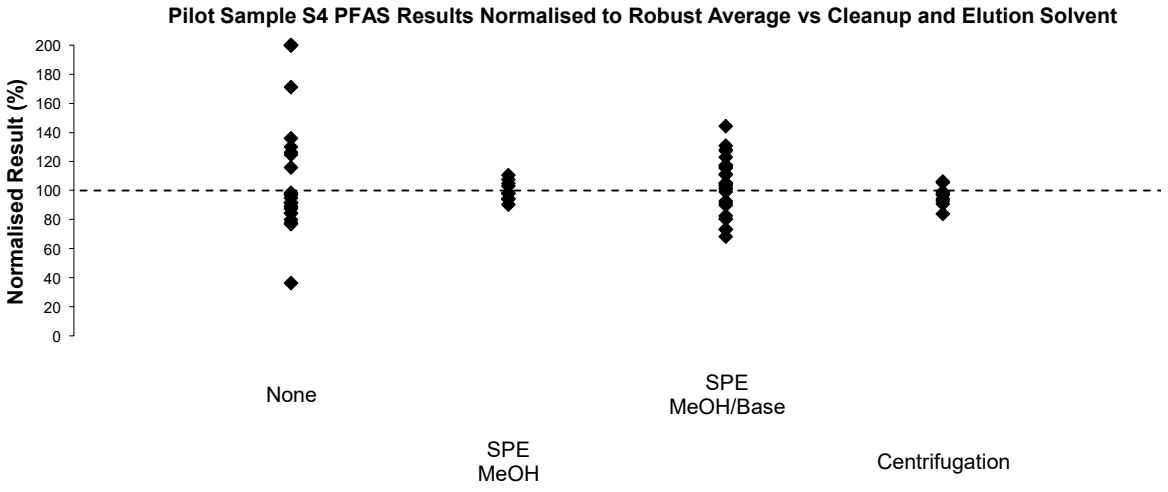
Laboratories extracted the samples from 30 minutes to 1440 minutes, at room temperature, 30°C or 60°C. No trends were evident between the reported results and these parameters. Five participants reported adding loose carbon to the sample extract to facilitate better adsorption of interferent organics.[20]

All but 2 laboratories reported concentrating their sample extracts using temperatures between 40°C and 45°C (Figure 55).



Normalised results greater than 200% plotted as 200%.

Figure 55 Pilot Sample S4 PFAS Results Normalised to Robust Average vs Extract Concentration Time and Temperature



Normalised results greater than 200% plotted as 200%.

Figure 56 Pilot Sample S4 PFAS Results Normalised to Robust Average vs Extract Cleanup Procedure

Of 9 laboratories who reported results, 5 performed a cleanup step. The results from laboratories who did not perform a cleanup step were more variable Figure 56. Cleanup of the crude extracts is an important step in the removal of matrix constituents that may interfere in instrumental determination.

The most popular method used for cosmetic material sample analysis involved SLE with a 1 g sample size, base modified methanol as extraction solvent, 40°C for concentration of extract, and SPE cleanup.

All participants reported using LC-MS/MS.

8:2diPAP A limited number of laboratories reported results for 8:2diPAP in pilot Sample S4. Of 5 reported results 4 were in relatively good agreement with each other and with the spiked value. Plots of participants' results versus method used for analyses are presented in Figure 57

Individual performance assessments for PFAS analytes in cosmetic samples will be conducted in the next PFAS in Consumer Goods PT study.

6.10 Participants' Results and Analytical Methods for Extractable Organic Fluorine in Cosmetics (Foundation)

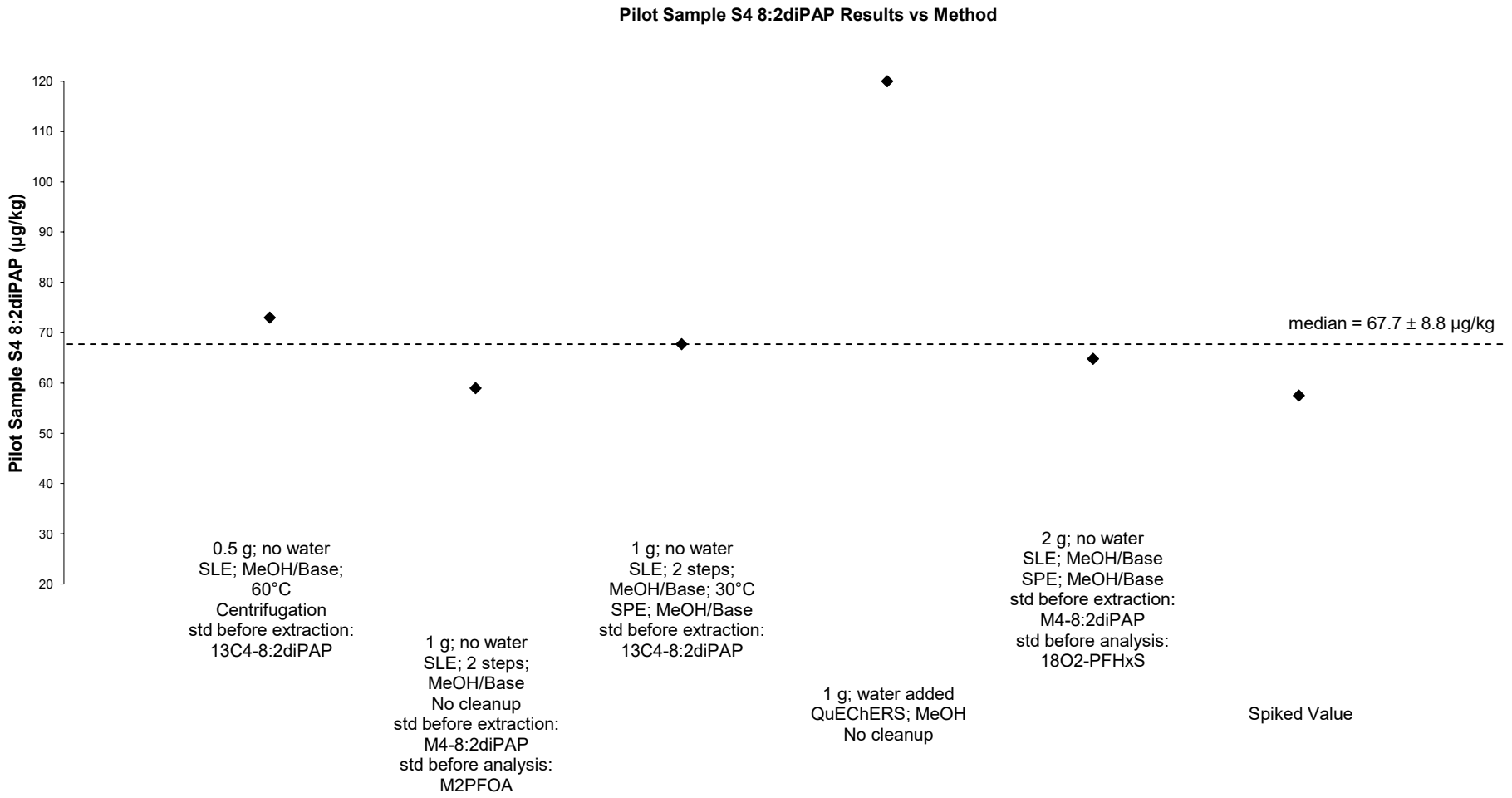
Participants were also asked to report Extractable Organic Fluorine (EOF) in the pilot Sample S4. No assessment was conducted for this test either as it was aimed at assisting laboratories in developing their method for EOF in cosmetics (foundation). Participants may assess their results against other participants' results and/or the calculated spiked value (Table 36). The expected EOF value for pilot Sample S4, based on the calculated PFAS spiked into the sample, was 5020 µg/kg, excluding any contribution from incurred PFAS.

The method descriptions provided by participants is presented in Appendix 6. Only 5 laboratories reported results, and four of them completed the questionnaire on the methods used.

Laboratory 9 commented: "For EOF, we originally tried processing 2 g of foundation, but the SPE cartridges clogged quickly. The method is set up for 5 g of solids/soil. The 0.5 g samples went through the SPE cartridges and the EOF result ended up just below the raised RL of 2,000 µg/kg but above the raised MDL of 540 µg/kg."

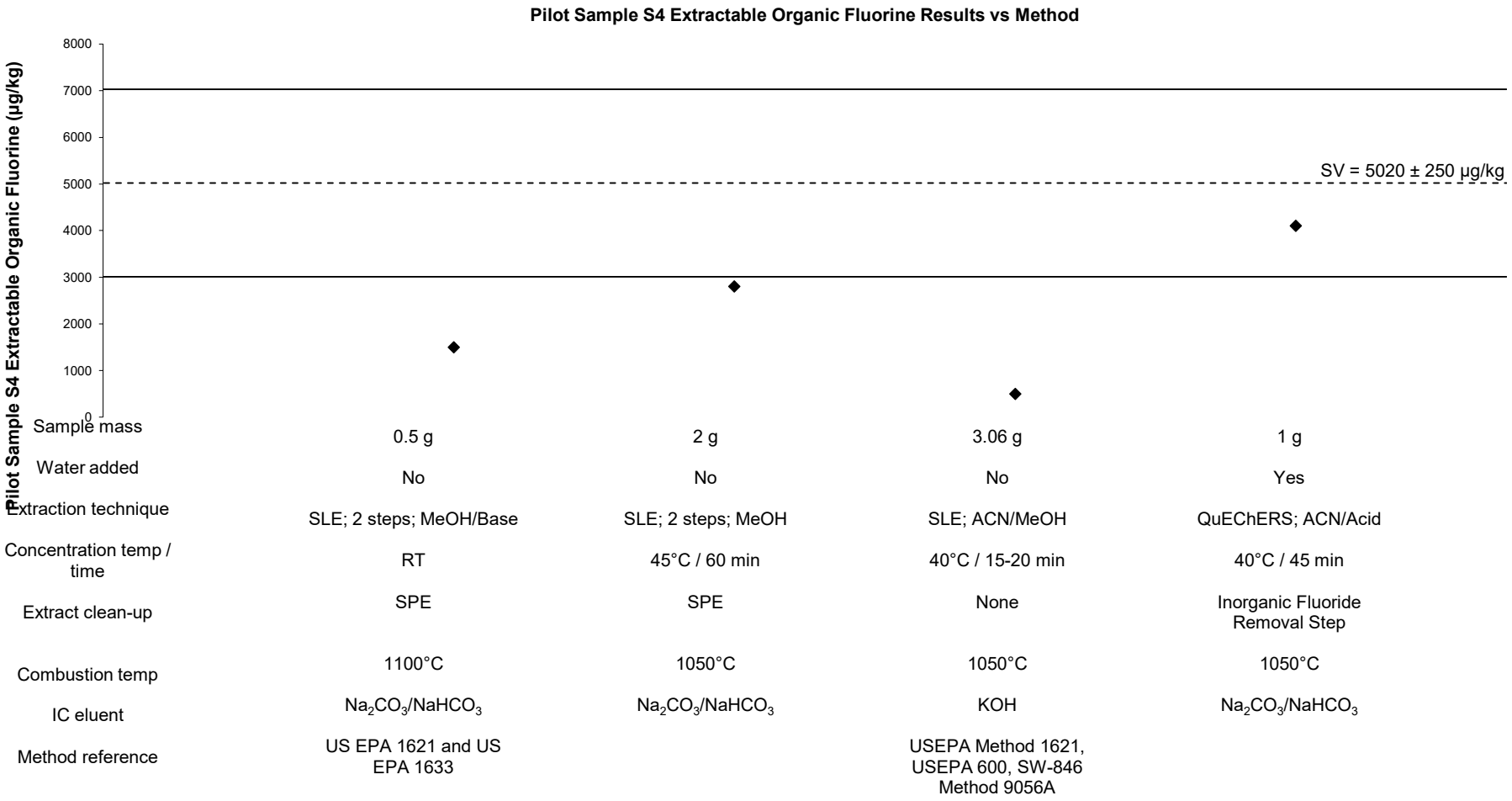
The result reported by Laboratory 5 of 4100 µg/kg was in good agreement with the EOF calculated spiked value of 5020 µg/kg. Laboratory 5 used a QuEChERS extraction method with a 1 g sample size, concentrated the extract at 40°C for 45 minutes, and used a sodium carbonate/sodium bicarbonate eluent. (Figure 58).

All reported EOF results were lower than the expected value calculated from the PFAS spiked into the sample. As EOF is not routinely measured by many analytical laboratories, this analyte will continue to be included in future NMIA PT studies to support method development.



Results greater than 120 µg/kg plotted as 120 µg/kg.

Figure 57 Pilot Sample S4 8:2diPAP Results vs Method



Horizontal lines correspond to good agreement with the spiked value.

Figure 58 Pilot Sample S4 Extractable Organic Fluorine Results vs Method

6.11 Participants' Results and Analytical Methods for TF in Cosmetics (Foundation)

Total Fluorine (TF) in the cosmetic (foundation) material was incurred. As for the other tests included in pilot Sample S4, no performance assessment was conducted for this test either. However, laboratories can evaluate whether their reported results fall within the compatible range (determined as the robust average excluding outliers, with a PCV of 20%) as suggested by the study coordinator in Appendix 5. Most reported results were in good agreement with each other.

Seven laboratories reported results for TF in pilot Sample S4, and 6 of them completed the questionnaire on the methods used. The method descriptions provided by these laboratories are presented in Appendix 6 and Figure 59.

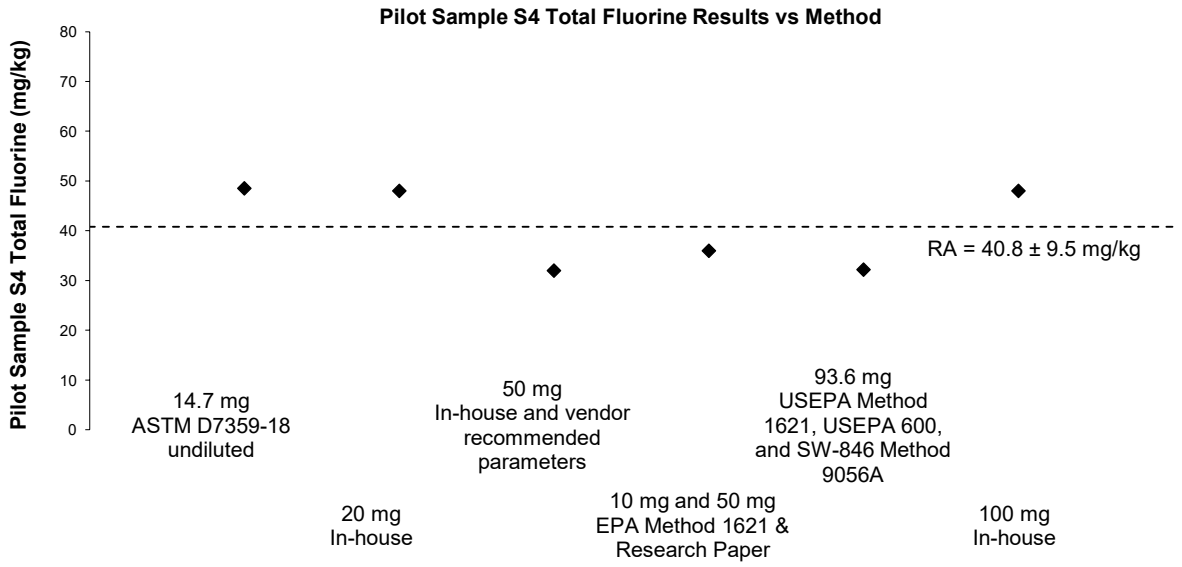


Figure 59 Pilot Sample S4 Total Fluorine Results vs Method

6.12 False Negatives

Appendix 4 presents false negative results. These are analytes present in the samples which a participant tested for but did not report a numeric result. This includes 'Not Reported' results, or those that were reported as a 'less than' result ($< x$) when the assigned value, less its expanded uncertainty, was greater than the participant's limit of reporting (LOR).

For analytes where no assigned value was set, results have only been considered a false negative if no value was reported, or if the consensus value and spiked value, less their respective expanded uncertainties, were both greater than the participants' LOR.

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

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[28] Zenobio, J.E., Salawu, O. A., Han, Z. & Adeleye, A.S., 2022, ‘Adsorption of per- and polyfluoroalkyl substances (PFAS) to containers’, *Journal of Hazardous Materials Advances*, vol. 7, 100130.

[29] USEPA, 2025, ‘Per- and Polyfluoroalkyl Substances (PFAS) in Pesticide and Other Packaging’, viewed September 2026, <https://www.epa.gov/pesticides/pfas-packaging>.

[30] NMI Organics Method NR70, Determination of PER-and Poly-fluorinated alkyl substances (PFAS) in water and solid matrices by LCMSMS.

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

APPENDIX 1 – SAMPLE PREPARATION, SAMPLE ANALYSIS and HOMOGENEITY ASSESSMENT, and STABILITY ASESSMENT

A1.1 Sample Preparation

Sample S1 and pilot Sample S2 consisted of cardboard packaging material, that was cut into approximately equal-sized pieces, fortified with 19 analytes, and dispensed into 3 g test portions.

Sample S3 consisted of cardboard packaging material, that was cut into approximately equal-sized pieces and dispensed into 3 g test portions.

Pilot Sample S4 was cosmetic (foundation) material fortified with 10 analytes and dispensed into portions of 20 g each.

Sample S1 and pilot Samples S2 and S4 were stored at 4°C prior to dispatch. Sample S3 was stored at room temperature

A1.2 Sample Analysis and Homogeneity Assessment

Sample Analysis

No homogeneity testing was completed for Sample S1, pilot Sample S2, and Sample S3 as the samples were prepared using a process previously demonstrated to produce sufficiently homogeneous samples. Participants' results in this study also gave no reason to question the samples' homogeneity.

A full homogeneity test was conducted for PFAS analytes pilot Sample S4. Measurements were conducted by NMIA Organic section using the method NR70.[30]

The method involved SLE of a 1 g sample using KOH and methanol for 120 minutes, followed by extract concentration at 40°C and analysis by LC-MS/MS. Although NMIA holds ISO/IEC 17025 accreditation for this method, cosmetic matrices are outside the scope of the accreditation.

Homogeneity Assessment

Homogeneity testing for the sample was based on that described by Thompson and Fearn,[31] which is also the procedure described in the International Harmonised Protocol for Proficiency Testing.[15]

A minimum of 6 containers of pilot Sample S4 were selected at random. Duplicate test portions were taken from each container, and the concentrations of the PFAS analytes were determined. Measurements were performed under repeatability conditions in a randomised order. The results of the homogeneity assessment are presented in Tables 43 to 52. Pilot Sample S4 was found to be sufficiently homogeneous for all PFAS analytes of interest.

Table 43 Pilot Sample S4 PFBA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	82	82
9	80	81
10	79	86
21	84	80
27	78	82
36	82	78
39	84	81
Mean	81	
CV	6.8%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.46	0.73	**Pass**
s_{an}/σ	0.17	0.50	**Pass**
s^2_{sam}	0.00001	61	**Pass**

Table 44 Pilot Sample S4 PFHxA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	200	200
9	200	190
10	190	200
21	200	190
27	180	190
36	200	190
39	200	200
Mean	200	
CV	6.1%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.20	0.73	**Pass**
s_{an}/σ	0.15	0.50	**Pass**
s^2_{sam}	7.1	340	**Pass**

Table 45 Pilot Sample S4 PFOA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	1.8	1.9
9	1.9	1.8
10	1.9	2.0
21	1.8	1.8
27	1.8	1.9
36	1.9	1.9
39	1.9	1.8
Mean	1.9	
CV	6.4%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.20	0.73	**Pass**
s_{an}/σ	0.16	0.50	**Pass**
s^2_{sam}	0.00048	0.031	**Pass**

Table 46 Pilot Sample S4 PFDoA Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	32	31
9	36	30
10	33	35
21	33	33
27	34	34
36	34	29
39	32	32
Mean	33	
CV	13%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.55	0.73	**Pass**
s_{an}/σ	0.33	0.50	**Pass**
s^2_{sam}	0.00001	15	**Pass**

Table 47 Pilot Sample S4 PFBS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	4.5	4.8
9	4.7	4.4
10	4.5	4.5
21	4.8	4.6
27	4.3	4.4
36	4.3	3.9
39	4.8	4.3
Mean	4.5	
CV	9.5%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.39	0.73	**Pass**
s_{an}/σ	0.24	0.50	**Pass**
s^2_{sam}	0.019	0.22	**Pass**

Table 48 Pilot Sample S4 PFHxS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	13	12
9	12	11
10	12	12
21	13	13
27	13	12
36	14	14
39	13	13
Mean	13	
CV	7.3%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.33	0.73	**Pass**
s_{an}/σ	0.18	0.50	**Pass**
s^2_{sam}	0.54	1.5	**Pass**

Table 49 Pilot Sample S4 PFHpS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	2.2	2.4
9	2.4	2.2
10	2.5	2.2
21	2.4	2.4
27	2.4	2.3
36	2.4	2.5
39	2.6	2.2
Mean	2.4	
CV	13%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.46	0.73	**Pass**
s_{an}/σ	0.33	0.50	**Pass**
s^2_{sam}	0.00001	0.078	**Pass**

Table 50 Pilot Sample S4 PFOS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	18	18
9	16	17
10	15	17
21	16	15
27	16	17
36	18	17
39	20	17
Mean	17	
CV	13%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.53	0.73	**Pass**
s_{an}/σ	0.33	0.50	**Pass**
s^2_{sam}	0.60	3.9	**Pass**

Table 51 Pilot Sample S4 6:2FTS Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	37	36
9	33	41
10	31	39
21	34	32
27	27	30
36	32	30
39	36	33
Mean	34	
CV	20%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.41	0.73	**Pass**
s_{an}/σ	0.49	0.50	**Pass**
s^2_{sam}	3.8	24	**Pass**

Table 52 Pilot Sample S4 8:2diPAP Homogeneity Testing

Container Number	Result (µg/kg)	
	Replicate 1	Replicate 2
1	62	56
9	57	58
10	52	57
21	62	53
27	54	61
36	60	51
39	58	64
Mean	58	
CV	16%	

Thompson and Fearn Homogeneity Tests

Test	Value	Critical	Result
Cochran	0.26	0.73	**Pass**
s_{an}/σ	0.41	0.50	**Pass**
s^2_{sam}	0.00001	57	**Pass**

A1.3 Stability Assessment

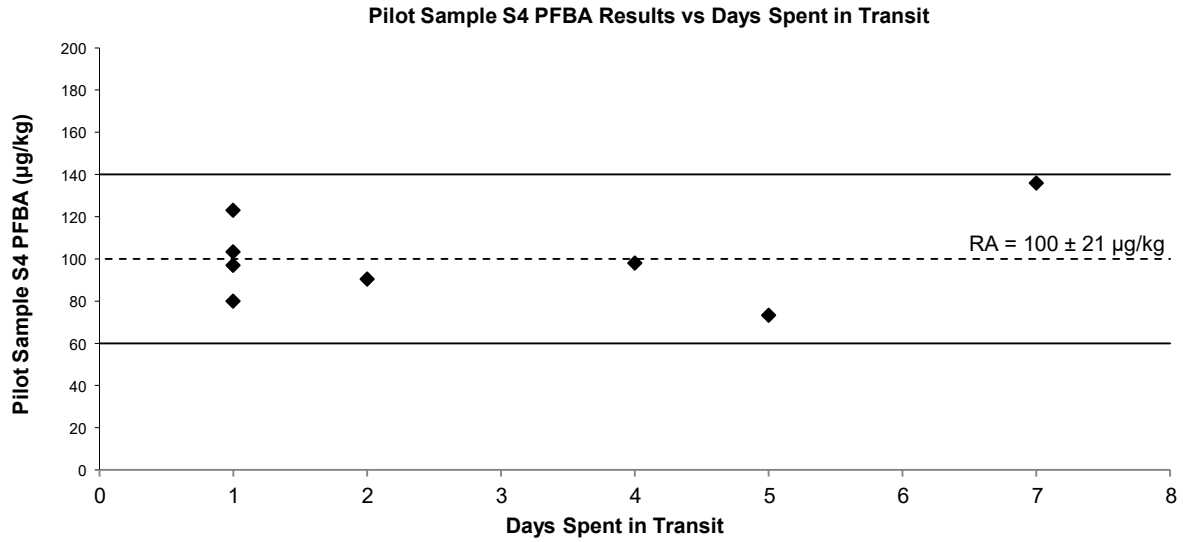
The test samples were dispatched on 1 June 2026. Participants were advised to store pilot Sample S4 refrigerated if unable to commence analysis on the day of receipt. Condition of samples on receipt, the date when they were received, and the date of analysis reported by participants are presented in Table 53.

Table 53 Pilot Sample S4 Condition on Receipt and Dates When Samples were Received and Analysed

Lab Code	Received Date	Pilot Sample S4	
		Condition on Receipt	Date of Analysis
1	3/06/2026	Good, room temperature	8/06/2026
2	9/06/2026	Good	10/07/2026
3	2/06/2026	satisfactory	3/06/2026
4	8/06/2026	18°C	25/06/2026
5	2/06/2026	Good	11/06/2026
6	2/06/2026	Good	1/07/2026
7	5/06/2026	Fit for analysis	15/06/2026
8	2/06/2026	Fit for Analysis	12/06/2026
9	11/06/2026	good	25/06/2026 (TF) 03/07/2026 (EOF)
11	2/06/2026	Good	18/06/2026
13*	15/06/2026		18/06/2026
14	2/06/2026	good	6/07/2026

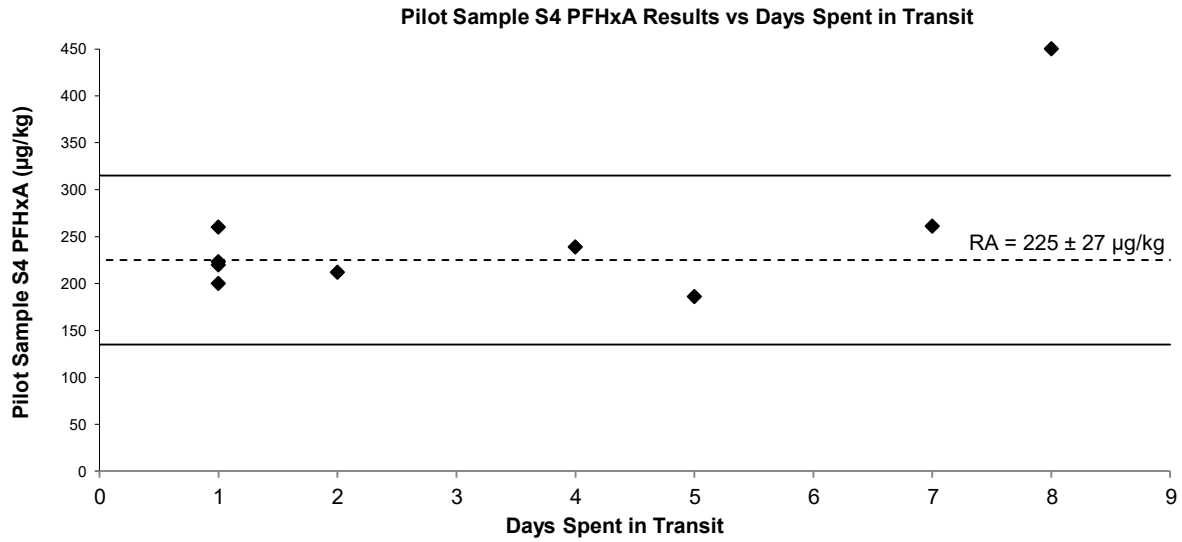
*Laboratory 13 dispatched 10/06/2026.

No significant trends between participant's results and the number of days spent in transit were evident (Figure 60).

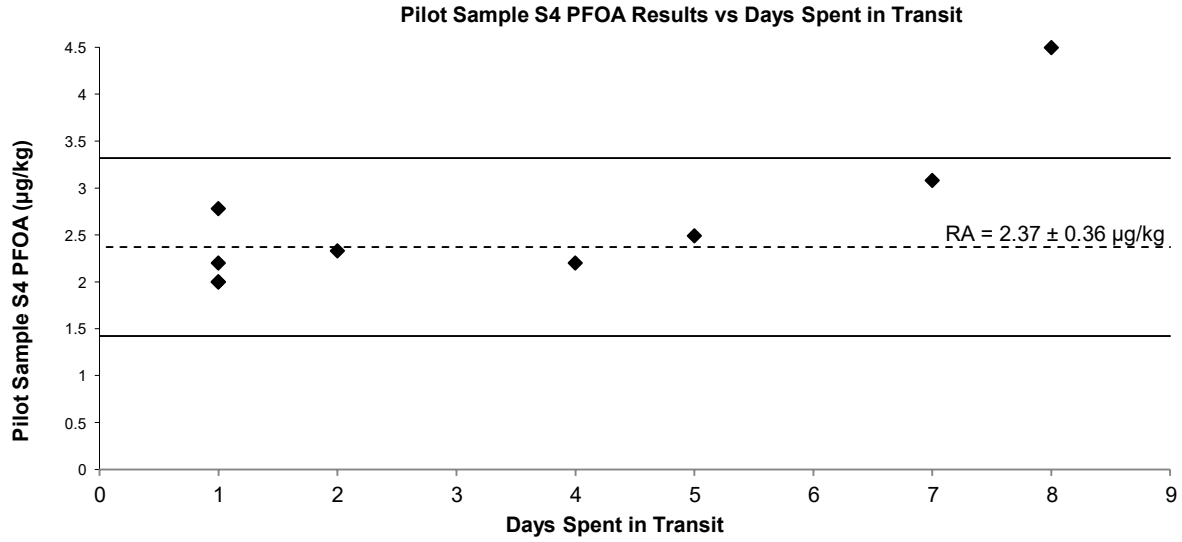


Horizontal lines are equivalent to the acceptable range outlined in Appendix 5.

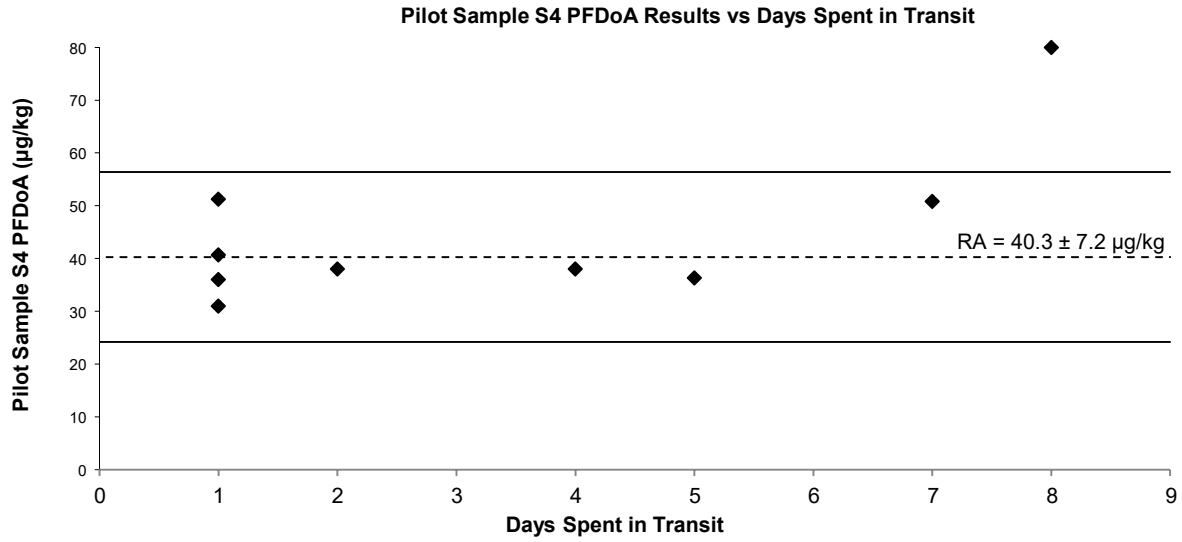
Figure 60 Pilot Sample S4 Results vs Days Spent in Transit



Horizontal lines are equivalent to the acceptable range outlined in Appendix 5. Results greater than 450 µg/kg plotted as 450 µg/kg.

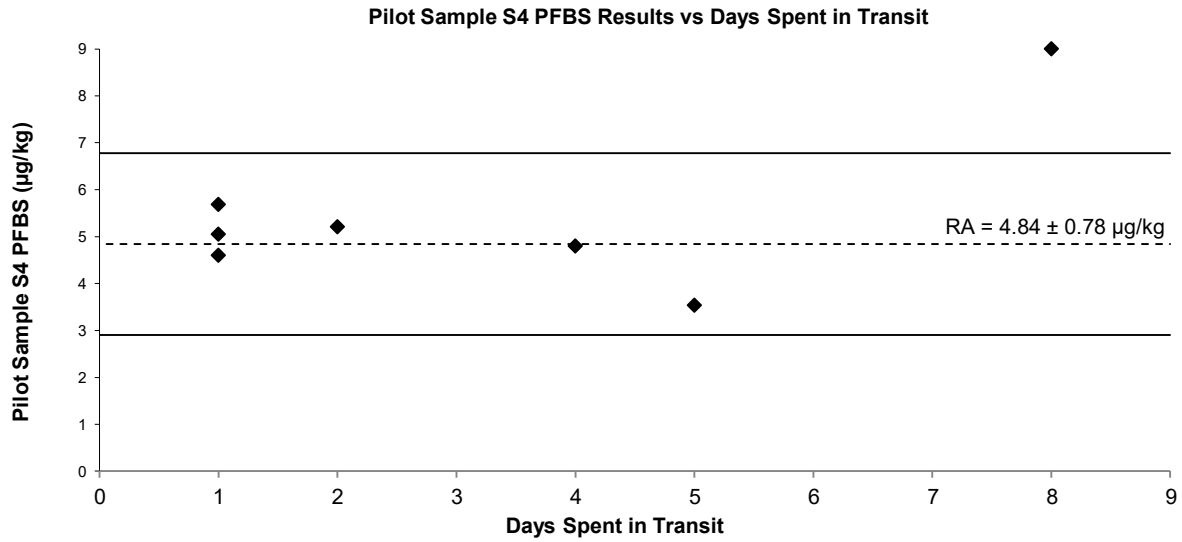


Horizontal lines are equivalent to the acceptable range outlined in Appendix 5. Results greater than 4.5 µg/kg plotted as 4.5 µg/kg.

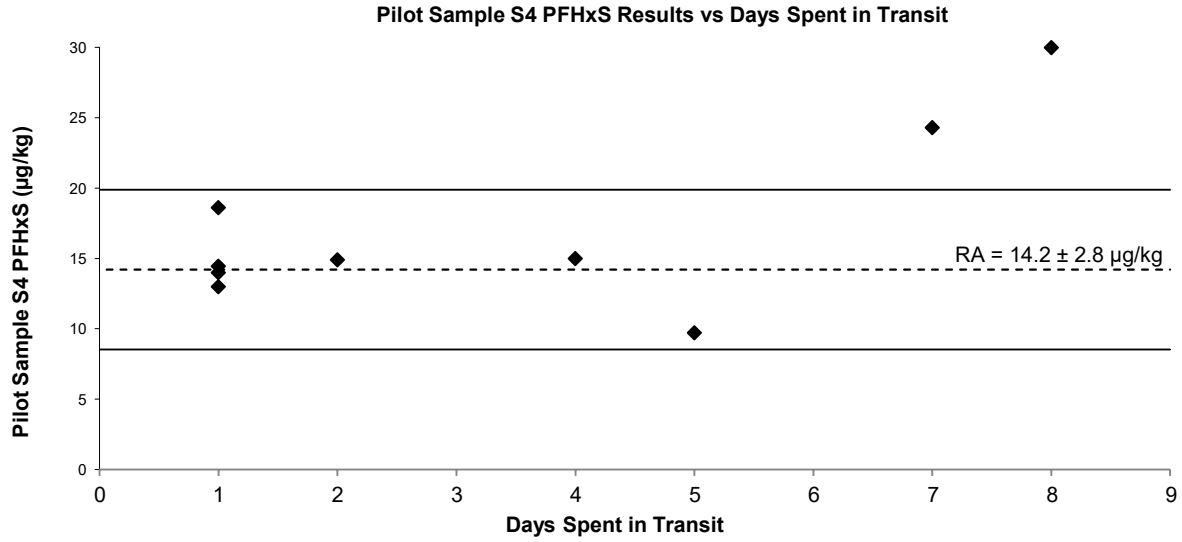


Horizontal lines are equivalent to the acceptable range outlined in Appendix 5. Results greater than 80 µg/kg plotted as 80 µg/kg.

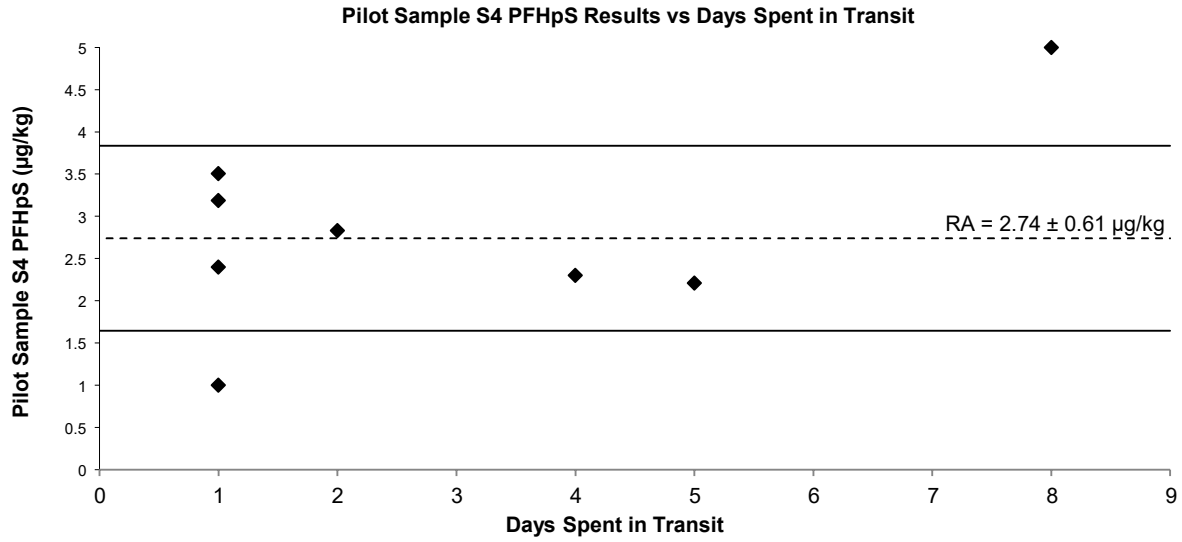
Figure 60 Pilot Sample S4 Results vs Days Spent in Transit (Continued)



Horizontal lines are equivalent to the acceptable range outlined in Appendix 5. Results greater than 9 µg/kg plotted as 9 µg/kg.

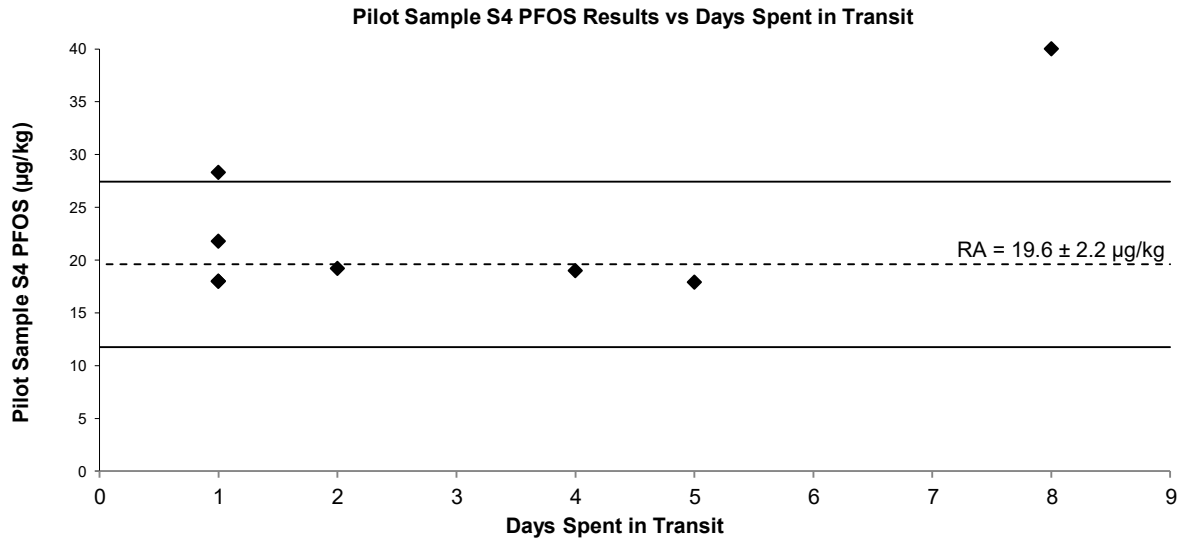


Horizontal lines are equivalent to the acceptable range outlined in Appendix 5. Results greater than 30 µg/kg plotted as 30 µg/kg.

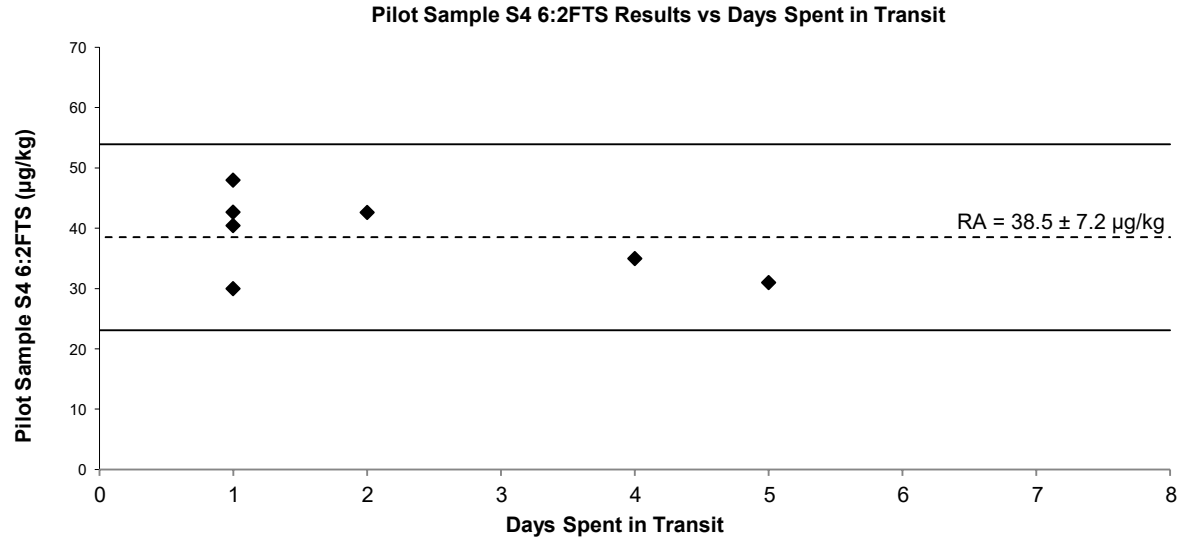


Horizontal lines are equivalent to the acceptable range outlined in Appendix 5. Results greater than 5 µg/kg plotted as 5 µg/kg.

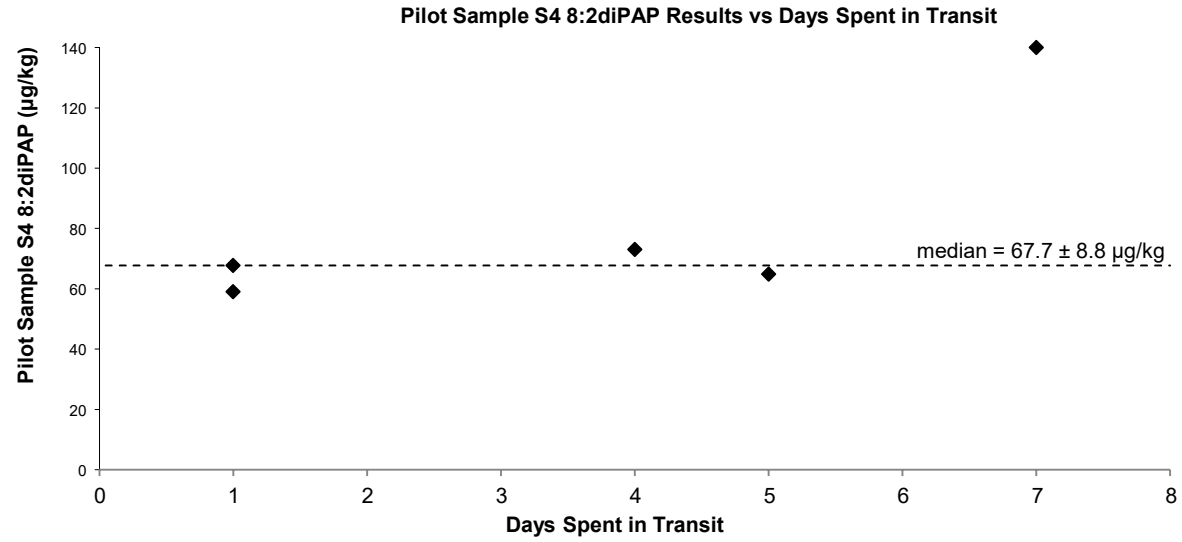
Figure 60 Pilot Sample S4 Results vs Days Spent in Transit (Continued)



Horizontal lines are equivalent to the acceptable range outlined in Appendix 5. Results greater than 40 µg/kg plotted as 40 µg/kg.



Horizontal lines are equivalent to the acceptable range outlined in Appendix 5.



Horizontal lines are equivalent to the acceptable range outlined in Appendix 5. Results greater than 140 µg/kg plotted as 140 µg/kg.

Figure 60 Pilot Sample S4 Results vs Days Spent in Transit (Continued)

A1.3.1 Storage and Transport Stability Study

A stability study was conducted for individual PFAS analytes in pilot Sample S4. Testing was conducted by the NMIA CRV section using the same procedure outlined in section A1.2. Two main factors were considered to affect the stability of these analytes in cosmetics (foundation): storage condition and time.

To assess stability, results from 2 samples were compared. One sample (Initial) was stored at 4°C and analysed before dispatch. The second sample (End of Study) was packaged as for dispatch and stored at ambient conditions for the same duration as the longest delivery time. After this period, the "End of Study"sample was stored at 4°C and analysed after results submission date.

The results from the 2 samples were in good agreement with each other and the robust average or median of participants results within their respective uncertainties for all analytes (Figures 61 to 70).

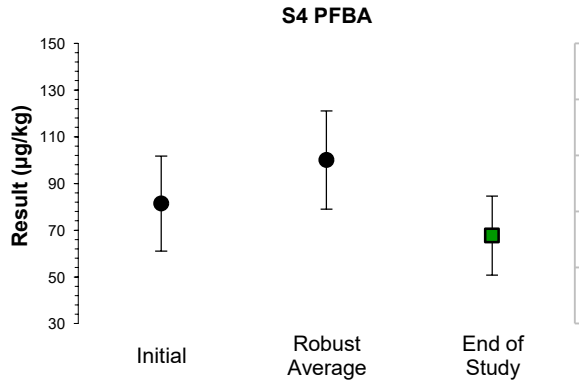


Figure 61 Pilot Sample S4 PFBA Stability Testing

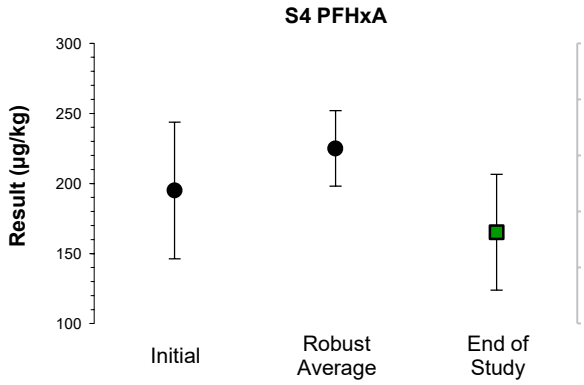


Figure 62 Pilot Sample S4 PFHxA Stability Testing

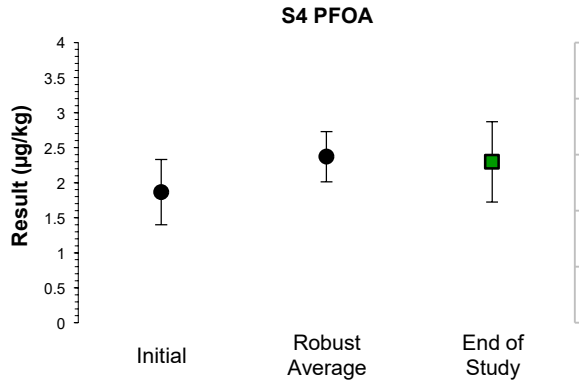


Figure 63 Pilot Sample S4 PFOA Stability Testing

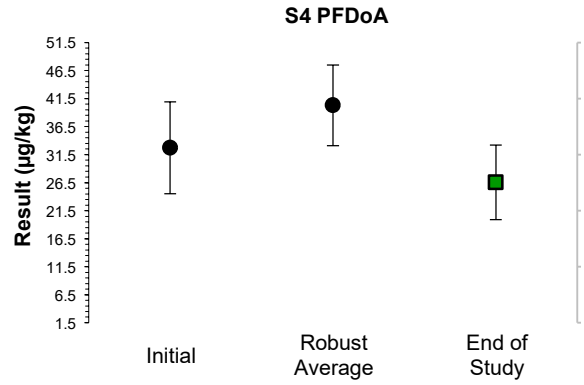


Figure 64 Pilot Sample S4 PFDoA Stability Testing

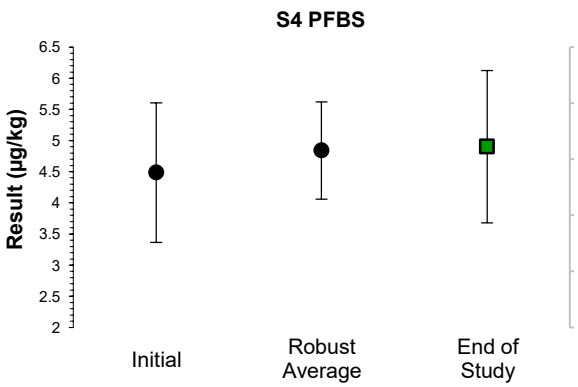


Figure 65 Pilot Sample S4 PFBS Stability Testing

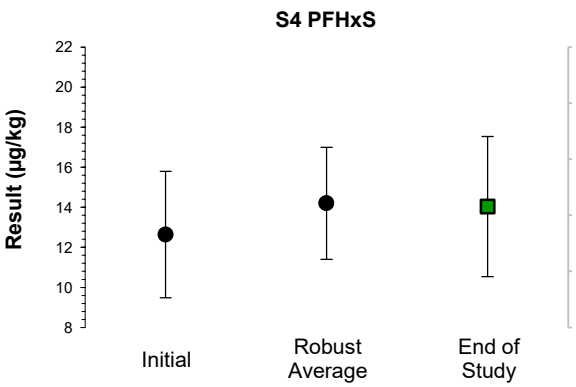


Figure 66 Pilot Sample S4 PFHxS Stability Testing

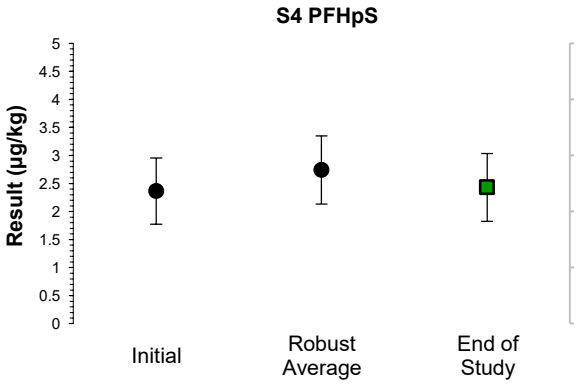


Figure 67 Pilot Sample S4 PFHpS Stability Testing

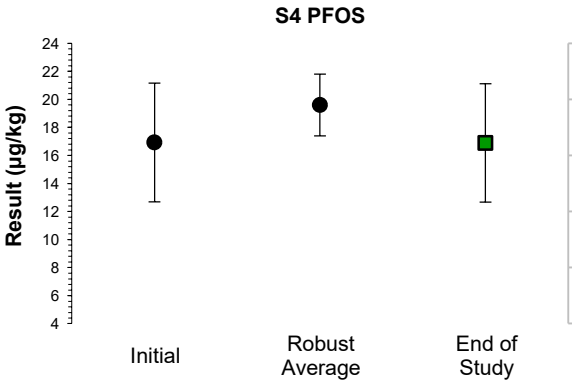


Figure 68 Pilot Sample S4 PFOS Stability Testing

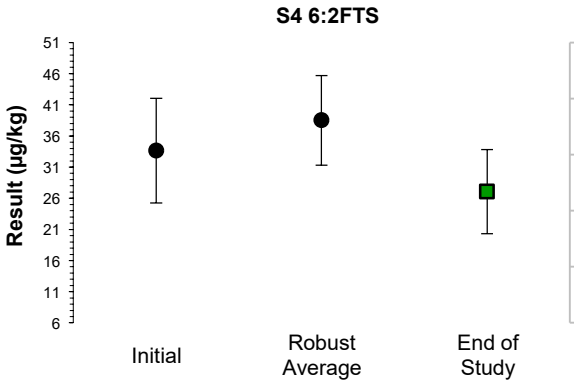


Figure 69 Pilot Sample S4 6:2FTS Stability Testing

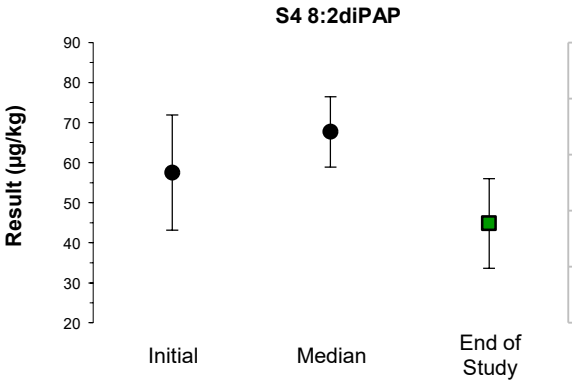


Figure 70 Pilot Sample S4 8:2diPAP Stability Testing

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

A2.1 Robust Average and Associated Uncertainty

The robust averages were calculated using the procedure described in ISO 13528.[16] The associated uncertainties were evaluated as according to Equation 4.

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

where:

$u_{rob\ av}$	is the standard uncertainty of the robust average
$S_{rob\ av}$	is the standard deviation of the robust average
p	is the number of results

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

A worked example is set out below in Table 54.

Table 54 Uncertainty Evaluation for PFBA in Sample S1

No. results (p)	9
Robust Average	19.9 µg/kg
$S_{rob\ av}$	2.9 µg/kg
$u_{rob\ av}$	1.2 µg/kg
k	2
$U_{rob\ av}$	2.4 µg/kg

Therefore, the robust average for PFBA in Sample S1 is 19.9 ± 2.4 µg/kg.

A2.2 z-Score and E_n-Score Calculations

For each participant's result, a z-score and E_n-score are calculated according to Equations 2 and 3 respectively (see Section 4).

A worked example is set out below in Table 55.

Table 55 z-Score and E_n-Score Calculation for Sample S1 PFBA Result Reported by Laboratory 5

Participant Result (µg/kg)	Assigned Value (µg/kg)	Standard Deviation for Proficiency Assessment	z-Score	E_n-Score
20 ± 4.9	19.9 ± 2.4	20% as PCV, or: 0.2 × 19.9 = 3.98 µg/kg	$z = \frac{20 - 19.9}{3.98}$ = 0.03	$E_n = \frac{20 - 19.9}{\sqrt{4.9^2 + 2.4^2}}$ = 0.02

APPENDIX 3 – ADDITIONAL ANALYTES

Table 56 Additional Analytes

Lab. Code	Sample	Analyte	Result (µg/kg)	Uncertainty (µg/kg)	Recovery (%)
1	S1	PFHpA	0.029	0.007	100
		PFHxDA	0.028	0.007	22.1
		PFODA	0.016	0.008	22.1
		N-EtFOSA	9.39	1.98	18.6
		N-MeFOSAA	0.029	0.003	32.1
	S4	PFNS	0.025	0.01	57.4
		PFDS	0.029	0.012	57.4
		N-MeFOSAA	0.02	0.002	42.4
2	S4	PFHpA	10.9	2.7	NR
4	S1	PFHpA	2.07	0.932	NR
		PFPeS	1.34	0.603	NR
		PFHpS	4.39	1.976	NR
		PFNS	2.77	1.246	NR
		PFOSA	3.54	1.593	NR
		6:2diPAP	1.05	0.473	NR
	S4	PFHxDA	353	159	NR
		PFOSA	271	122	NR
		N-EtFOSAA	280	126	NR
		4:2FTS	322	145	NR
		8:2FTS	359	162	NR
		6:2diPAP	189	85	NR
		GenX	12.7	5.71	NR
		FHxSA	0.22		
5	S1	6:2diPAP	1.9	0.48	NR
	S4	FHxSA	0.22	NR	NR
6	S4	PFPeA	0.175	0.021	80
		6:2diPAP	0.413	0.21	145
7	S1	6:2diPAP	2.0	0.6	NR
12	S1	10:2FTS	0.043	0.013	NR
13	S1	N-MeFOSE	1.1	0.3124	21.6
		6:2diPAP	2.07	0.36846	52.7
	S4	PFPeA	0.672	0.152544	45.3

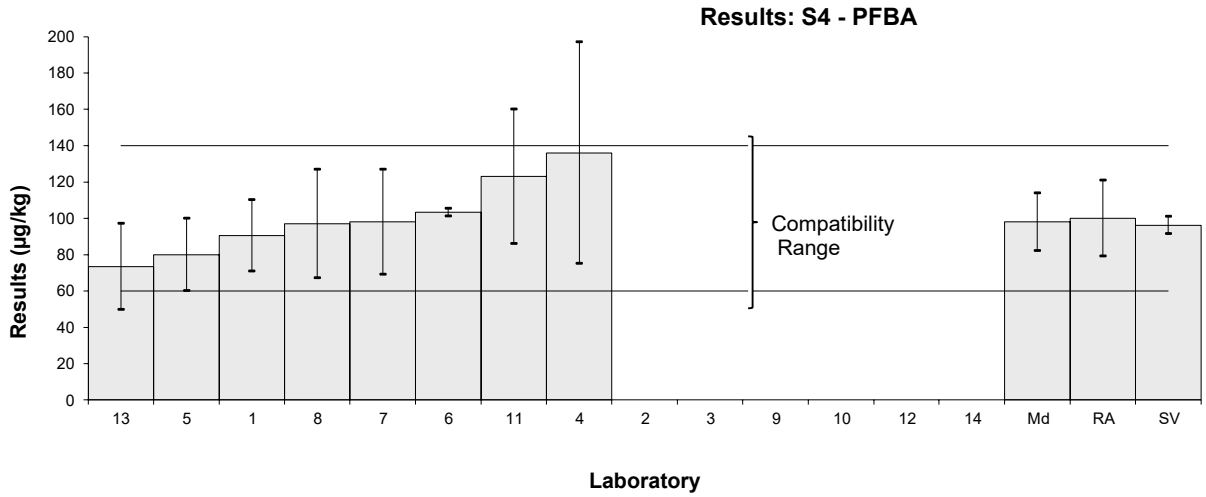
APPENDIX 4 – FALSE NEGATIVES

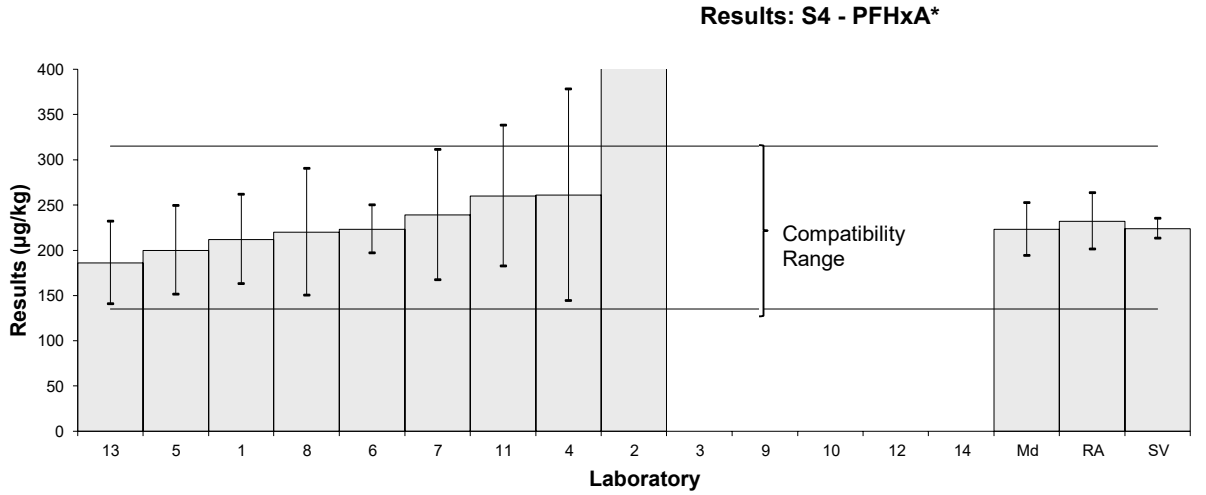
Table 57 False Negatives

Lab. Code	Sample	Analyte	Assigned Value (µg/kg)	Consensus Value* (µg/kg)	Spiked Value (µg/kg)	Result (µg/kg)
4	S1	PFDoA	10.1	NA	15.2	<0.50
		PFBS	2.75	NA	3.04	<0.50
		N-EtFOSAA	Not Set	18.3	20.2	<0.50
		8:2FTS	39.6	NA	47.0	<0.50
	S2	Extractable Organic Fluorine	Not Set	4900	5200	<256

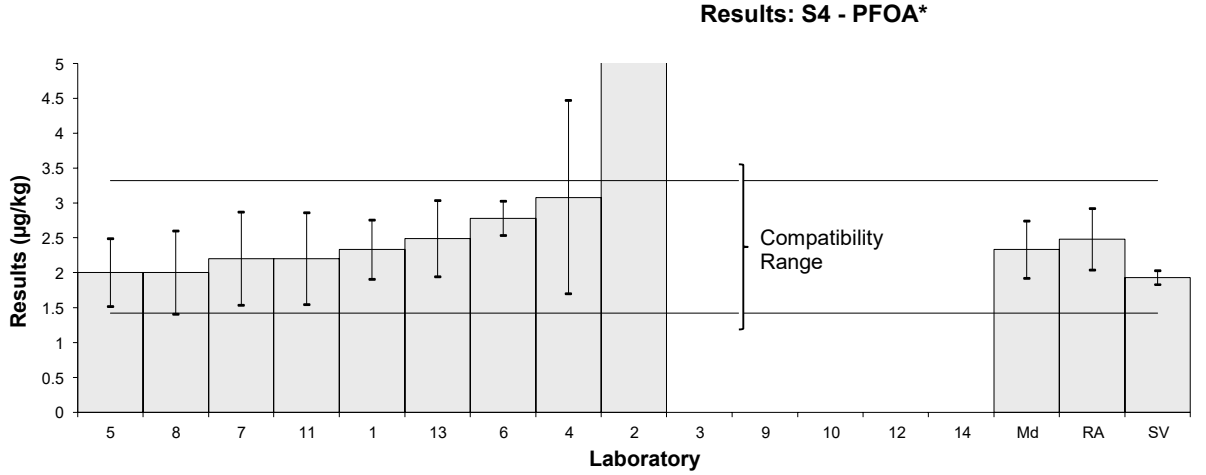
*Robust average or median as applicable (assigned value not set).

APPENDIX 5 – ASSESSMENT OF RESULTS COMPATIBILITY FOR PFAS IN PILOT SAMPLE S4



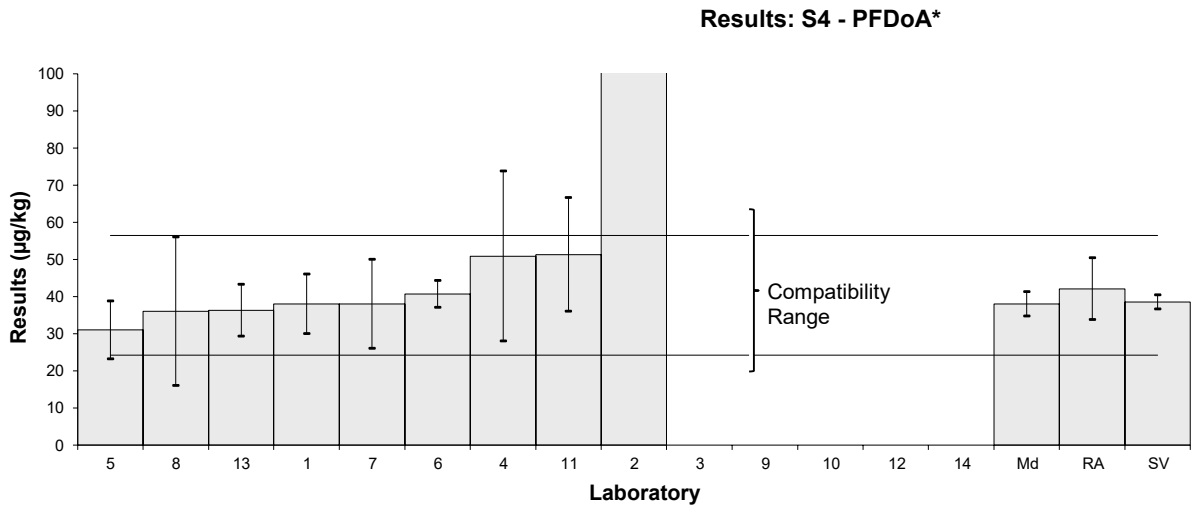


*Laboratory 2 submitted results after the release of the Preliminary Report.

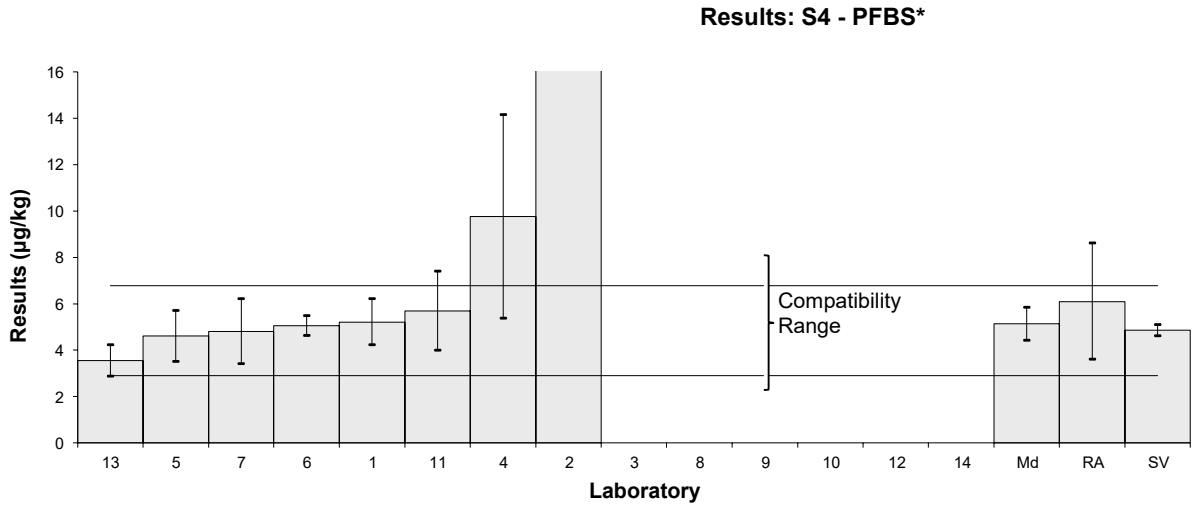


*Laboratory 2 submitted results after the release of the Preliminary Report.

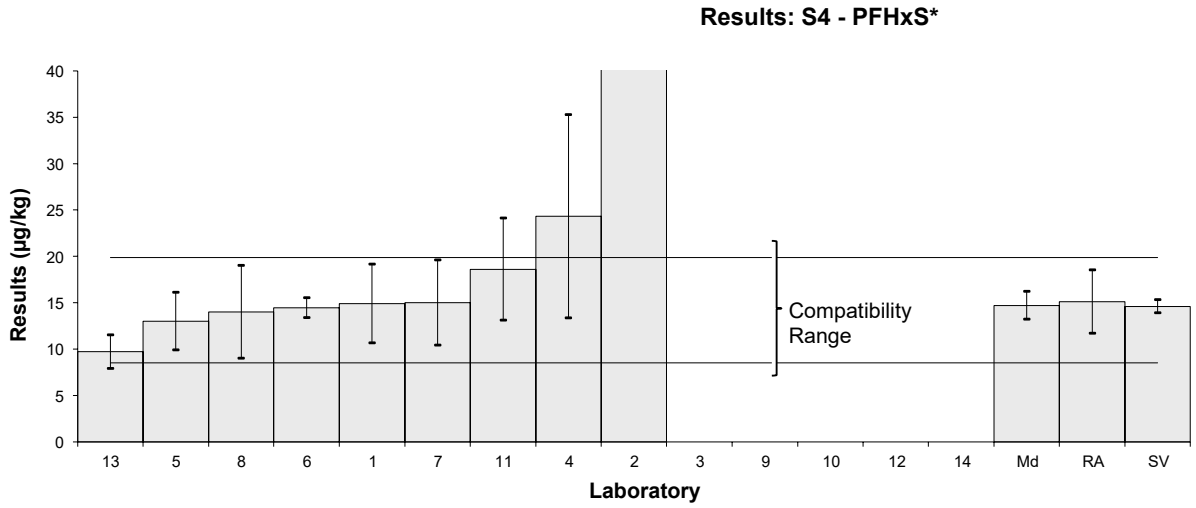
Figure 71 Assessment of Results Compatibility for PFAS in Pilot Sample S4



*Laboratory 2 submitted results after the release of the Preliminary Report.

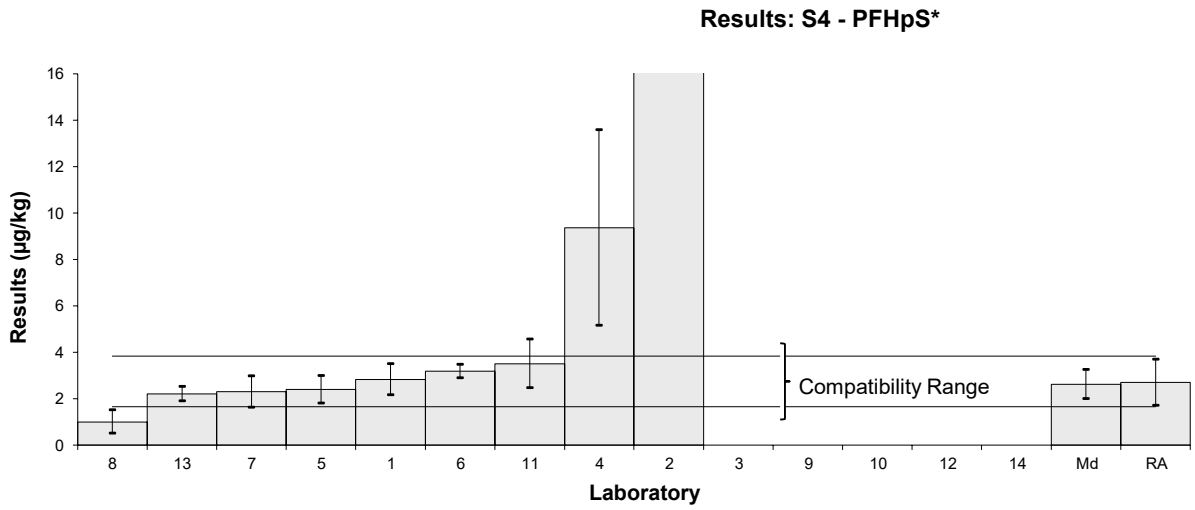


*Laboratory 2 submitted results after the release of the Preliminary Report.

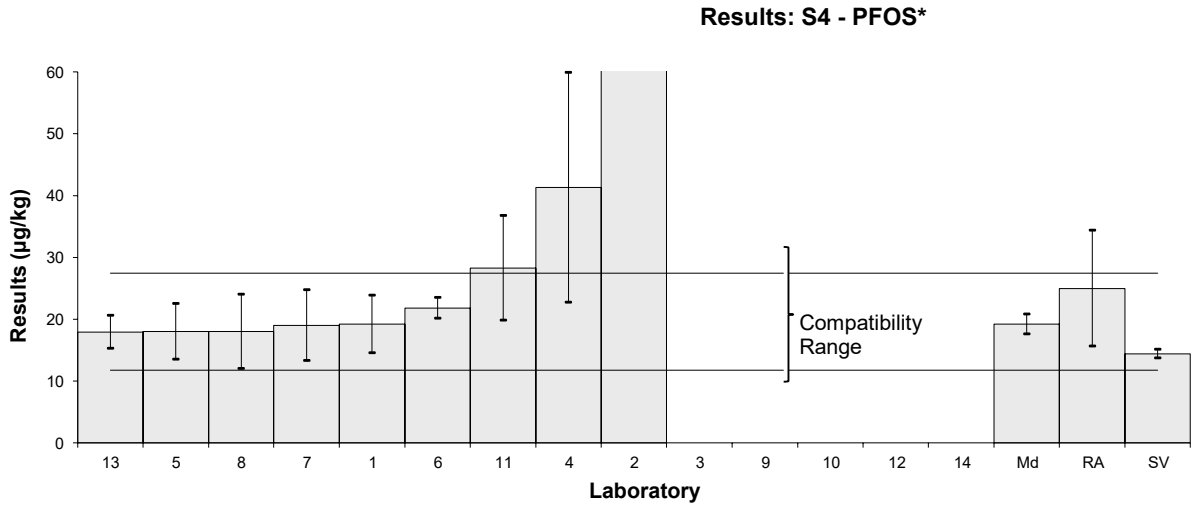


*Laboratory 2 submitted results after the release of the Preliminary Report.

Figure 71 Assessment of Results Compatibility for PFAS in Pilot Sample S4 (Continued)



*Laboratory 2 submitted results after the release of the Preliminary Report.



*Laboratory 2 submitted results after the release of the Preliminary Report.

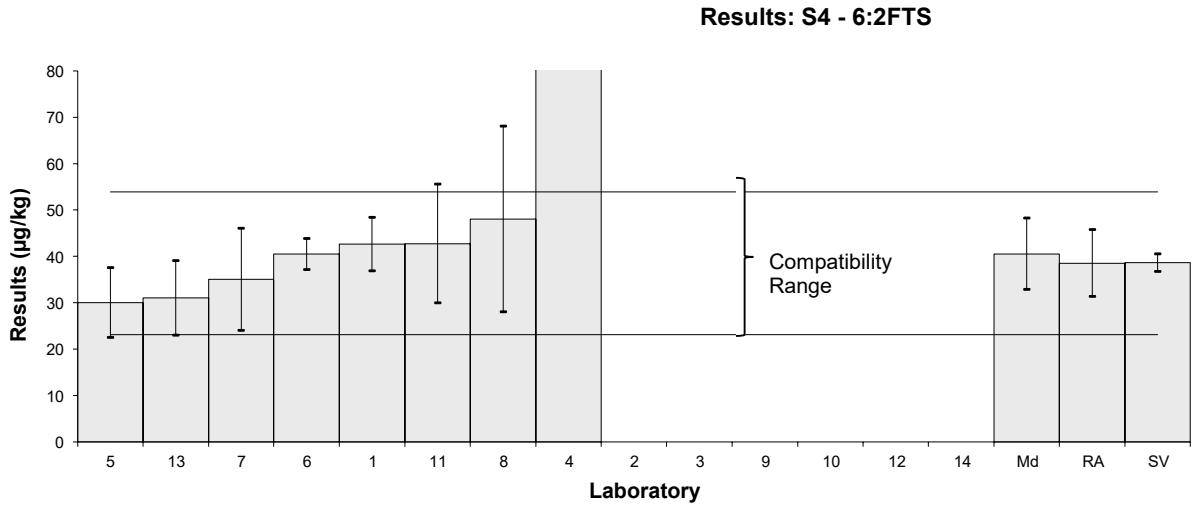
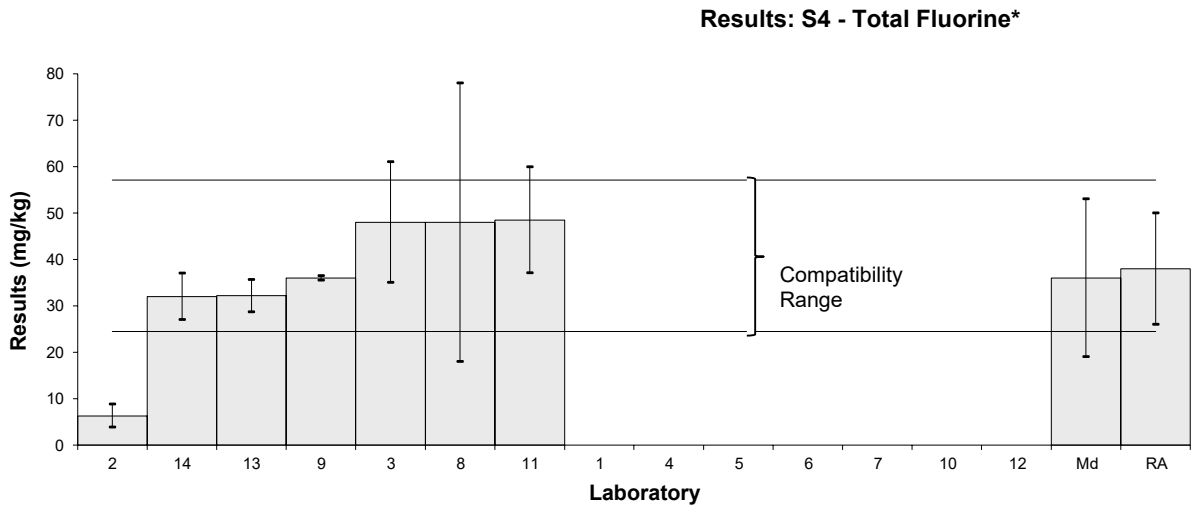


Figure 71 Assessment of Results Compatibility for PFAS in Pilot Sample S4 (Continued)



*Laboratory 2 submitted results after the release of the Preliminary Report.

Figure 71 Assessment of Results Compatibility for PFAS in Pilot Sample S4 (Continued)

APPENDIX 6 – PARTICIPANTS' TEST METHODS

A6.1 Participants' Test Methods for Total Fluorine

Participants' methods for analysis of Total Fluorine in Sample S3 and pilot Sample S4 are presented in Tables 58 to 60.

Table 58 Measurement Methods and Instrument Techniques for Total Fluorine in S3

Lab. Code	Sample amount used (g)	Instrument Technique	Method Reference
3	0.02	Combustion Ion Chromatography	In-House
4	50 mg	Combustion Ion Chromatography	NF EN 17813
7	0.55	Combustion Ion Chromatography	SS-EN 15408:2011, SS-EN ISO 10304-1:2009
8	0.06	Combustion Ion Chromatography	No. In-house
9*	1 square (~18 mg) and 3 squares (~54 mg)	CIC	Luc T. Miaz et la., Environ. Sci.: Processes Impacts,2020,22, 1071–1083 and EPA Method 1621
11	0.0188	Combustion Ion Chromatography	ASTM D7359-18 Standard Test Method for Total Fluorine, Chlorine and Sulfur in Aromatic Hydrocarbons and their Mixtures by Oxidative Pyrohydrolytic combustion followed by Ion Chromatography Detection (Combustion Ion Chromatography-CIC)
13	0.0695	Combustion Ion Chromatography	USEPA Method 1621, USEPA 600, SW-846 Method 9056A

*Additional Information in Table 60.

Table 59 Measurement Methods and Instrument Techniques for Total Fluorine in Pilot Sample S4

Lab. Code	Sample amount used (g)	Instrument Technique	Method Reference
3	0.02	Combustion Ion Chromatography	In-House
4	50 mg	Combustion Ion Chromatography	NF EN 17813
8	0.1	Combustion Ion Chromatography	No. In-house
9*	~10 mg and ~50 mg	CIC	Luc T. Miaz et la., Environ. Sci.: Processes Impacts,2020,22, 1071–1083 and EPA Method 1621
11*	0.0147	Combustion Ion Chromatography	ASTM D7359-18 Standard Test Method for Total Fluorine, Chlorine and Sulfur in Aromatic Hydrocarbons and their Mixtures by Oxidative Pyrohydrolytic combustion followed by Ion Chromatography Detection (Combustion Ion Chromatography-CIC)
13	0.0936	Combustion Ion Chromatography	USEPA Method 1621, USEPA 600, SW-846 Method 9056A

*Additional Information in Table 60.

Table 60 Participants' Total Fluorine Methodology – Additional Information

Lab. Code	Total Fluorine Additional Information
9	S3: Squares cut into thin strips to lie in the bottom of the combustion boats. Three squares was the maximum that would fit. Both weights run in triplicate. Both were >> RL so the reported value is the average of n=6. S4: Both weights run in triplicate. 50 mg was >> RL and 10 mg was at RL so the reported value is the average of n=3 from the 50 mg samples.
11	S4: Sample run without dilution
5	S4: FHxSA detected at 0.22ug/kg

A6.2 Participants' Test Methods for Extractable Organic Fluorine

Participants' methods for the analysis of Extractable Organic Fluorine in pilot Samples S2 and S4 are presented in Tables 61 to 63.

Table 61 Measurement Methods for Extractable Organic Fluorine by Combustion Ion Chromatography in Pilot Sample S2

Lab. Code	Entire Container Used?	Container Rinsed?	If Not Entire Container, Sample Amount Used (g)	Extraction Technique	If Heat Applied, Temperature Used (ºC)	Extraction Solvent	Extraction Time (min)	If Repeated Extractions, Number of Repeats	Extract Concentration Temperature(°C)	Extract Concentration Time(min)	Extract Clean-Up	Combustion Temperature (°C)	Buffer Solution	IC Eluent	Method Reference
4	Yes	No		Solid-Liquid Extraction (vortexed/sonicated/shake and centrifuged)	no	ACN/MeOH	60 min		45°C	25 min	None	1050°C	water	Na2CO3/ NaHCO3 mixture	No reference method
5	Yes	Yes		QuEChERS		Acidic MeCN	30		40	45	Inorganic fluoride removal step	1050	Ultra pure water	Na2CO3/ NaHCO3 mixture	
9*	Yes	Yes	n/a	Solid-Liquid Extraction (vortexed/sonicated/shake and centrifuged)	no heat	MeOH, 0.3% NH3	20 min + 10 min + final rinse/vortex	2 extractions + final rins/vortex	room temp	n/a	Solid-Phase Extraction	1100 °C	water	Na2CO3/ NaHCO3 mixture	US EPA 1621 and US EPA 1633
11	Yes	Yes		Solid-Liquid Extraction (vortexed/sonicated/shake and centrifuged)		MeOH	90	2	45	60	Solid-Phase Extraction	1050	H2O	Na2CO3/ NaHCO3 mixture	
13	Yes	Yes		Solid-Liquid Extraction (vortexed/sonicated/shake and centrifuged)		ACN/MeOH	60		40	~15-20	None	1050	Oxygen and Argon	KOH	USEPA Method 1621, USEPA 600, SW-846 Method 9056A

*Additional Information in Table 63.

Table 62 Measurement Methods for Extractable Organic Fluorine by Combustion Ion Chromatography in Pilot Sample S4

Lab. Code	Sample Weight (g)	Reagent Water Added?	Extraction Technique	If Heat Applied, Temperature Used (°C)	Extraction Solvent	Extraction Time (min)	If Repeated Extractions, Number of Repeats	Extract Concentration Temperature(°C)	Extract Concentration Time(min)	Extract Clean-Up	Combustion Temperature (°C)	IC Eluent	Buffer Solution	Method Reference
4	3.64g	No	Yes	No	ACN/MeOH	60 min		45°C	25 min	None	1050 °C	Na2CO3/ NaHCO3 mixture	Water	
5	1	Yes	QuEChERS		Acidic MeCN	30		40	45	Inorganic fluoride removal step	1050	Na2CO3/ NaHCO3 mixture	Ultra pure water	
9*	0.5 g	No	Solid-Liquid Extraction (vortexed/ sonicated/shake and centrifuged)	no heat	MeOH, 0.3% NH3	20 min + 10 min + final rinse/ vortex	2 extractions + final rins/ vortex	room temp	n/a	Solid-Phase Extraction	1100 °C	water	Na2CO3/ NaHCO3 mixture	US EPA 1621 (CIC) and US EPA 1633 (extraction & clean-up)
11	2	No	Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)		MeOH	90	2	45	60	Solid-Phase Extraction	1050	H2O	Na2CO3/ NaHCO3 mixture	
13	3.06	No	Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)		ACN/MeOH	60		40	~15-20	None	1050	KOH	Oxygen and Argon	USEPA Method 1621, USEPA 600, SW-846 Method 9056A

*Additional Information in Table 63.

Table 63 Participants' Extractable Organic Fluorine Methodology – Additional Information

Lab. Code	Extractable Organic Fluorine Additional Information
9	S3: 10% reagent water added to cardboard before extraction as per EPA method 1633. NittoSeiko furnace, Shimadzu IC. S4: NittoSeiko furnace
9	S4: For EOF, we originally tried processing 2 g of foundation but the SPE cartridges clogged quickly. The method is set up for 5 g of solids/soil. The 0.5 g samples went through the SPE cartridges and the EOF result ended up just below the raised RL of 2,000 µg/kg but above the raised MDL of 540 µg/kg..

A6.3 Participants' Test Methods for PFAS in Sample S1

Participants' methods for Sample S1 cardboard are presented in Tables 64 to 86.

Table 64 Sample Preparation and Extraction Methods for PFAS Analytes in S1

Lab. Code	Entire Container Used?	Container Rinsed?	If Not Entire Container, Sample Amount Used (g)	Labelled Standards Added Before Extraction?	Labelled Standards Directly into Container?	Equilibration Time (min)	Extraction Technique	If Heat Applied, Temperature Used (°C)	Extraction Solvent	Extraction Time (min)	If Repeated Extractions, Number of Repeats	Extract Carbon Clean-Up?	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Extract Clean-Up	Elution Solvent	Final pH Adjustment?	Labelled Standards Added Before Instrumental Analysis?
1	Yes	Yes	3	Yes	No	30	Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)	NA	ACN/ MeOH	90	3	No	40	120	Solid-Phase Extraction	NH4OH/ MeOH	No	Yes
4*	Yes	Yes		Yes	No	30	Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)	No	KOH/ MeOH	60	no	No	45°C	25 min	Solid-Phase Extraction	NH4OH/ MeOH	No	
5	Yes	Yes		Yes	No		Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)		KOH/ MeOH	120	2	No	40	40	None	NA	No	Yes
6	Yes	Yes		Yes	No	5	Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)	30	NH4OH/ MeOH	20	2	Yes	40		None		No	Yes
7	Yes	Yes		Yes	No	1	Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)	60	NaOH/ MeOH	60	No	Yes			Centrifugation	NA	Yes	No

Lab. Code	Entire Container Used?	Container Rinsed?	If Not Entire Container, Sample Amount Used (g)	Labelled Standards Added Before Extraction?	Labelled Standards Directly into Container?	Equilibration Time (min)	Extraction Technique	If Heat Applied, Temperature Used (°C)	Extraction Solvent	Extraction Time (min)	If Repeated Extractions, Number of Repeats	Extract Carbon Clean-Up?	Extract Concentration Temperature (°C)	Extract Concentration Time (min)	Extract Clean-Up	Elution Solvent	Final pH Adjustment?	Labelled Standards Added Before Instrumental Analysis?
8	Yes	Yes	NA	Yes	Yes	0	Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)	NA	NH4OH/ MeOH	1440	NA	No	NA	NA	None	NA	Yes	No
10	Yes	Yes		Yes	Yes	30	Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)		MeOH	30	2	No	60	60	None		No	No
11	Yes	Yes	3	Yes	No	30	Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)		Basic ACN and Acetone	30		Yes	45	35	None	NA	No	No
12	Yes	Yes		Yes	Yes	0.5	Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)	room temperature	ACN in 0.1% NH3	120					Solid-Phase Extraction	MeOH, 0.1% NH3	No	Yes
13	Yes	Yes	N/A	Yes	Yes	30	Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)	N/A	KOH/ MeOH	60	N/A	Yes	N/A	N/A	Solid-Phase Extraction	NH4OH/ MeOH	Yes	Yes

*Additional Information in Table 65.

Table 65 Sample Preparation and Extraction Methods for PFAS Analytes in S1 - Additional Information

Lab. Code	Additional Information
4	Before SPE purification (with WAX/CBG) pH is adjusted
2	S1: TF= 58.05 ug/kg
10	S1: NR value <1µg/kg. The analytical method used does not resolve linear and branched isomers

Table 66 Instrument Techniques for PFAS Analytes in S1

Lab. Code	Instrument	Dilution Factor (if Applicable)	Mobile Phase	Was a blank correction applied?
1	LC-MS/MS or LC-QQQ	no	MeOH/ACN 50/50 v/v	Yes
4	LC-MSMS or LC-QQQ	No	Methanol	No
5	LC-MSMS or LC-QQQ	20	Methanol	No
6	LC-MSMS or LC-QQQ	No	Methanol	No
7	LC-MSMS or LC-QQQ		Methanol & 5mM ammonium acetate in ultrapure water	No
8	LC-MSMS or LC-QQQ	5	Methanol	No
10	LC-MSMS or LC-QQQ	10	5mM di ammonium acetate in water/acetonitrile	No
11	LC-MSMS or LC-QQQ	0.4166	ACN	No
12	LC-MSMS or LC-QQQ	No	MeOH/ACN//	No
13	LC-MSMS or LC-QQQ	Undiluted and DL10	ACN	No

Table 67 Labelled Standards Methodology for PFAS Analytes in S1

Lab. Code	Labelled Internal Standard Source	Recovery Correction?	Standard Method?	Secondary Source Used to Check Standard?
1	Wellington	Yes	no	
5	Wellington	Yes	USEPA 1633	
6	Wellington	Yes	Isotopic Dilution	No
7	Greyhound, Wellington, TLC, LGC	No	Internal Method	
8	Wellington	Yes	No. In-house	Standards with a alternate lot number are used as check standards
10	Wellington	Yes	No	yes
11	Wellington Labs	Yes		Yes, ICC is used.
12	Wellington, CIL, LGC	No		no

Lab. Code	Labelled Internal Standard Source	Recovery Correction?	Standard Method?	Secondary Source Used to Check Standard?
13	Wellington			

Table 68 Labelled Standards for PFBA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C4-PFBA	13C8-PFOA
5	MPFBA	M3PFBA
6	13C4-PFBA	13C3-PFBA
7	13C4-PFBA	NA
8	13C4-PFBA	NA
11	13C4-PFBA	
12	13C4-PFBA	13C3-PFBA
13	13C4-PFBA	13C3-PFBA

Table 69 Labelled Standards for PFPeA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C5-PFPeA	13C8-PFOA
5	M5PFPeA	M3PFBA
6	13C4-PFPeA	13C5-PFPeA
7	13C5-PFPeA	NA
8	13C3-PFPeA	NA
10	13C5-PFPeA	None
11	13C5-PFPeA	
12	13C5-PFPA	13C2-PFHxA
13	13C5-PFPeA	13C2-PFHxA

Table 70 Labelled Standards for PFHxA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C5-PFHxA	13C8-PFOA
5	M5PFHxA	M3PFBA
6	13C2-PFHxA	13C5-PFPeA
7	13C5-PFHxA	NA
8	13C2-PFHxA	NA
10	13C5-PFHxA	None
11	13C2-PFHxA	
12	13C5-PFHxA	13C2-PFHxA
13	13C5-PFHxA	13C2-PFHxA

Table 71 Labelled Standards for PFOA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C4-PFOA	13C8-PFOA
5	M8PFOA	M2PFOA
6	13C4-PFOA	13C8-PFOA
7	13C4-PFOA	NA
8	13C4-PFOA	NA
10	13C8-PFOA	None
11	13C8-PFOA	
12	13C8-PFOA	13C2-PFOA
13	13C8-PFOA	13C4-PFOA

Table 72 Labelled Standards for PFNA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C9-PFNA	13C5-PFNA
5	M9PFNA	M2PFOA
6	13C5-PFNA	13C8-PFOA
7	13C5-PFNA	NA
8	13C5-PFNA	NA
10	13C9-PFNA	None
11	13C5-PFNA	
12	13C9-PFNA	13C5-PFNA
13	13C9-PFNA	13C5-PFNA

Table 73 Labelled Standards for PFDA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C2-PFDA	13C5-PFNA
5	M6PFDA	MPFDA
6	13C2-PFDA	13C8-PFOA
7	13C6-PFDA	NA
8	13C2-PFDA	NA
10	13C6-PFDA	None
11	13C6-PFDA	
12	13C6-PFDA	13C2-PFDA
13	13C6-PFDA	13C2-PFDA

Table 74 Labelled Standards for PFUdA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C2-PFUdA	13C5-PFNA
5	M7PFUdA	MPFDA
6	13C2-PFUdA	13C8-PFOA
7	13C7-PFUdA	NA
8	13C2-PFUdA	NA
10	13C7-PFUdA	None
11	13C2-PFUdA	
12	13C7-PFUnA	13C5-PFNA
13	13C7-PFUdA	13C2-PFDA

Table 75 Labelled Standards for PFDoA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C2-PFDoA	13C5-PFNA
5	MPFDoA	MPFDA
7	13C2-PFDoA	NA
8	13C2-PFDoDA	NA
10	13C2-PFDoA	None
11	13C2-PFDoA	
12	13C2-PFDoA	13C2-PFDA
13	13C2-PFDoA	13C2-PFDA

Table 76 Labelled Standards for PFTrDA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C2-PFTrDA	13C2-PFTeDA
5	MPFDoA	MPFDA
7	13C2-PFTrDA	NA
8	13C2-PFTeDA	NA
10	13C2-PFTeDA	None
11	13C2-PFDoA	
12	13C2-PFTrDA	13C2-PFDA
13	13C2-PFTrDA	13C2-PFDA

Table 77 Labelled Standards for PFTeDA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C2-PFHxDA	13C2-PFTeDA
5	M2PFTeDA	MPFDA
7	13C2-PFTeDA	NA
8	13C2-PFTeDA	NA
10	13C2-PFTeDA	None
11	13C2-PFTeDA	
12	13C2-PFTeDA	13C2-PFDA
13	13C2-PFTeDA	13C2-PFDA

Table 78 Labelled Standards for PFBS

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	18O2-PFHxS	18O2-PFOS
5	M3PFBS	MPFDA
6	13C3-PFBS	13C3-PFHxS
7	13C3-PFBS	NA
8	13C3-PFBS	NA
10	13C3-PFBS	None
11	13C3-PFBS	
12	13C3-PFBS	13C4-PFOS
13	13C3-PFBS	18O2-PFHxS

Table 79 Labelled Standards for PFHxS

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	18O2-PFHxS	18O2-PFOS
5	M3PFHxS	MPFDA
6	18O2-PFHxS	13C3-PFHxS
7	18O2-PFHxS	NA
8	18O2-PFHxS	NA
10	13C3-PFHxS	None
11	18O2-PFHxS	
12	13C3-PFHxS	13C4-PFOS
13	13C3-PFHxS	18O2-PFHxS

Table 80 Labelled Standards for PFOS

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C4-PFOS	18O2-PFOS
5	M8PFOS	MPFOS
6	13C4-PFOS	13C8-PFOS
7	13C8-PFOS	NA
8	13C4-PFOS	NA
10	13C8-PFOS	None
11	13C8-PFOS	
12	13C8-PFOS	13C4-PFOS
13	13C8-PFOS	13C4-PFOS

Table 81 Labelled Standards for N-EtFOSAA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	d3-MeFOSAA	d5-EtFOSAA
5	d5-N-EtFOSAA	MPFOS
7	d5-EtFOSAA	NA
8	D5-Et-FOSAA	NA
11	d5-NEtFOSAA	
13	13C4-EtFOSAA	13C4-PFOS

Table 82 Labelled Standards for 6:2FTS

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C2-6:2 FTS	13C2-8:2 FTS
5	M2-6:2 FTS	MPFOS
6	13C2-6:2 FTS	
7	13C2-6:2FTS	NA
8	13C2,12C6 6:2-FTS	NA
10	13C2-6:2 FTS	None
11	13C2-6-2 FTS	
12	13C2-6-2 FTS	
13	13C2-6:2-FTS	18O2-PFHxS

Table 83 Labelled Standards for 8:2FTS

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C2-6:2 FTS	13C2-8:2 FTS
5	M2-8:2 FTS	MPFOS
6	13C2-8:2 FTS	
7	13C2-8:2FTS	NA
8	13C2 8:2-FTS	NA
10	13C2-8:2 FTS	None
11	13C2-8-2 FTS	
12	13C2-8-2 FTS	
13	13C2-8:2-FTS	18O2-PFHxS

Table 84 Labelled Standards for 8:2diPAP

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
5	M4-8:2diPAP	M2PFOA
7	13C4-8:2diPAP	NA
13	M4-8:2diPAP	18O2-PFHxS

Table 85 Labelled Standards for GenX

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C2-GenX	13C8-PFOA
5	M3-HFPO-DA	MPFDA
6	13C3-GenX	
7	13C3-GenX	NA
8	13C312C3HF11O3	NA
10	13C3-HFPO-DA	None
11	13C3-GenX	
12	13C3-HFPO-DA	13C5-PFNA
13	13C3-HFPO-DA	13C2-PFHxA

Table 86 Labelled Standards for ADONA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
5	M3-HFPO-DA	MPFDA
6	13C3-PFHpA	13C8-PFOA
7	13C4-PFHpA	NA
8	13C4-PFHpA	NA
10	13C2-PFDoA	None
11	13C8-PFOS	
12	13C8-PFOA	
13	13C3-HFPO-DA	13C2-PFHxA

A6.4 Participants' Test Methods for PFAS in Pilot Sample S4

Participants' methods for pilot Sample S4 cosmetics (foundation) are presented in Tables 87 to 00.

Table 87 Sample Preparation and Extraction Methods for PFAS Analytes in Pilot Sample S4

Lab. Code	Sample Weight (g)	Reagent Water Added?	Labelled Standard Added Before Extraction?	Equilibration Time (min)	Other Sample Pretreatment	Extraction Technique	If Heat Applied, Temperature Used (ºC)?	Extraction Solvent	Extraction Time (min)	If Repeated Extractions, Number of Repeats	Extract Carbon Clean-Up?	Extract Concentration Temperature (°C)	Total Extract Concentration Time (min)	Extract Clean-Up	Elution Solvent	Final pH Adjustment?	Labelled Standard Added Before Instrumental Analysis?
1	1.0417	No	Yes	30	No	Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)	No	ACN	90 minutes	3	No	40	120	Solid-Phase Extraction	MeOH	No	Yes
4	1g	Yes	Yes	30	No	QuEChERS	No	MeOH									
5	1.0	No	Yes			Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)		KOH/ MeOH	120	2	No	40	40	None	NA	No	Yes
6	1g	No	Yes	20		Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)	30	NaOH/ MeOH	30	2	Yes	40		Solid-Phase Extraction	NH4OH /MeOH	No	Yes
7	0.5 g	No	Yes	1		Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)	60	NaOH/ MeOH	60 min		Yes			Centrifugation	NA	Yes	No

Lab. Code	Sample Weight (g)	Reagent Water Added?	Labelled Standard Added Before Extraction?	Equilibration Time (min)	Other Sample Pretreatment	Extraction Technique	If Heat Applied, Temperature Used (°C)?	Extraction Solvent	Extraction Time (min)	If Repeated Extractions, Number of Repeats	Extract Carbon Clean-Up?	Extract Concentration Temperature (°C)	Total Extract Concentration Time (min)	Extract Clean-Up	Elution Solvent	Final pH Adjustment?	Labelled Standard Added Before Instrumental Analysis?
8	0.1	Yes	Yes	30	NA	Solid-Liquid Extraction (vortexed/ sonicated/ shake and centrifuged)	NA	NH4OH/ MeOH and MeOH	1440	2	Yes	NA	NA	NA	NA	Yes	No
11	0.5	No	Yes	30		Solid-Liquid Extraction (vortexed/soni cated/shake and centrifuged)		Hexane, Basic Methanol, Acidic Methnaol	60	3	Yes	45	60	Solid-Phase Extraction	NH4OH /MeOH	No	No
13	2.00 grams	No	Yes	30	N/A	Solid-Liquid Extraction (vortexed/soni cated/shake and centrifuged)	N/A	KOH/ MeOH	60 min	N/A	Yes	N/A	N/A	Solid-Phase Extraction	NH4OH /MeOH	Yes	Yes

Table 88 Participants 'Additional Information for Pilot Sample S4

Lab. Code	Additional I
5	S4: FHxSA detected at 0.22ug/kg

Table 89 Instrument Techniques for PFAS Analytes in Pilot Sample S4

Lab. Code	Instrument	Dilution Factor (if Applicable)	Mobile Phase	Was a blank correction applied?
1	LC-MSMS or LC-QQQ	no	MeOH:ACN 50/50 v/v	Yes
4	LC-MSMS or LC-QQQ	No	Methanol	No
5	LC-MSMS or LC-QQQ	20	Methanol	No
6	LC-MSMS or LC-QQQ	No	Methanol	No
7	LC-MSMS or LC-QQQ		Methanol & 5mM ammonium acetate in ultrapure water	No
8	LC-MSMS or LC-QQQ	2	Methanol	No
11	LC-MSMS or LC-QQQ		ACN	No
13	LC-MSMS or LC-QQQ	Undiluted and DL10	ACN	No

Table 90 Labelled Standards Methodology for PFAS Analytes in Pilot Sample S4

Lab. Code	Labelled Internal Standard Source	Recovery Correction?	Standard Method?	Secondary Source Used to Check Standard?
1	Wellington	Yes	no	
5	Wellington	Yes	USEPA 1633	
6	Wellington	Yes	Isotopic Dilution	No
7	Greyhound, Wellington, TLC, LGC	No	Internal Method	
8	Wellington	Yes	No. In-house	Standards with a alternate lot number are used as check standards
11	Wellington,CIL	Yes		Yes, ICC is used.
13	Wellington			

Table 91 Labelled Standards for PFBA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C4-PFBA	13C8-PFOA
5	MPFBA	M3PFBA
6	13C4-PFBA	13C3-PFBA
7	13C4-PFBA	NA
8	13C4-PFBA	NA
11	13C4-PFBA	
13	13C4-PFBA	13C3-PFBA

Table 92 Labelled Standards for PFHxA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C5-PFHxA	13C8-PFOA
5	M5PFHxA	M3PFBA
6	13C2-PFHxA	13C5-PFPeA
7	13C5-PFHxA	NA
8	13C2-PFHxA	NA
11	13C2-PFHxA	
13	13C5-PFHxA	13C2-PFHxA

Table 93 Labelled Standards for PFOA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C4-PFOA	13C8-PFOA
5	M8PFOA	M2PFOA
6	13C4-PFOA	13C8-PFOA
7	13C4-PFOA	NA
8	13C4-PFOA	NA
11	13C8-PFOA	
13	13C8-PFOA	13C4-PFOA

Table 94 Labelled Standards for PFDoA

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C2-PFDoA	13C5-PFNA
5	MPFDoA	MPFDA
6	13C2-PFDoA	13C8-PFOA
7	13C2-PFDoA	NA
8	13C2-PFDoDA	NA
11	13C2-PFDoA	
13	13C2-PFDoA	13C2-PFDA

Table 95 Labelled Standards for PFBS

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	18O2-PFHxS	18O2-PFOS
5	M3PFBS	MPFDA
6	13C3-PFBS	13C3-PFHxS
7	13C3-PFBS	NA
8	13C3-PFBS	NA
11	13C3-PFBS	
13	13C3-PFBS	18O2-PFHxS

Table 96 Labelled Standards for PFHxS

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	18O2-PFHxS	18O2-PFOS
5	M3PFHxS	MPFDA
6	18O2-PFHxS	13C3-PFHxS
7	18O2-PFHxS	NA
8	18O2-PFHxS	NA
11	18O2-PFHxS	
13	13C3-PFHxS	18O2-PFHxS

Table 97 Labelled Standards for PFHpS

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	18O2-PFHxS	18O2-PFOS
5	M3PFHxS	MPFDA
6	18O2-PFHxS	13C3-PFHxS
7	18O2-PFHxS	NA
8	18O2-PFHxS	NA
13	13C8-PFOS	13C4-PFOS

Table 98 Labelled Standards for PFOS

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C4-PFOS	18O2-PFOS
5	M8PFOS	MPFOS
6	13C4-PFOS	13C8-PFOS
7	13C8-PFOS	NA
8	13C4-PFOS	NA
11	13C8-PFOS	
13	13C8-PFOS	13C4-PFOS

Table 99 Labelled Standards for 6:2FTS

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
1	13C2-6:2 FTS	13C2-8:2 FTS
5	M2-6:2 FTS	MPFOS
6	13C2-6:2 FTS	
7	13C2-6:2FTS	NA
8	13C2,12C6 6:2-FTS	NA
11	13C2-6-2 FTS	
13	13C2-6:2-FTS	18O2-PFHxS

Table 100 Labelled Standards for 8:2diPAP

Lab. Code	Labelled Standards added before extraction	Labelled Standards added before instrument analysis
5	M4-8:2diPAP	M2PFOA
6	13C4-8:2 DiPAP	
7	13C4-8:2diPAP	NA
13	M4-8:2diPAP	18O2-PFHxS

APPENDIX 7 – ACRONYMS AND ABBREVIATIONS

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

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

END OF REPORT