Australian Government
Department of Industry, Innovation and Science

National Measurement Institute

Proficiency Test Report
AQA 19-20
PFAS in Fish and Tomato

December 2019

ACKNOWLEDGMENTS

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

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

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SUMMARY

AQA 19-20 was conducted in June 2019. Twenty-five laboratories participated and twenty-two submitted results by the due date.

This study focused on the measurement of twenty six per-and polyfluorinated alkyl substances (PFAS) - PFOS, PFOA, PFHxS, PFOSA, PFBS, PFHpS, PFNS, PFDS, PFBA, PFPeA, PFHxA, PFHpA, PFNA, PFDA, PFDoA, PFTeDA, PFTrA, PFUnA, ETFOSA, ETFOSAA, MEFOSE, ADONA, GenX, 6:2 FTS, 8:2 FTS and 10:2 FTS, in fish and tomato. Two test samples were prepared at the NMI North Ryde laboratory and consisted of:

- A fish sample containing incurred PFAS contaminants and spiked with additional 11 PFAS components (S1); and
- A tomato sample spiked with 14 PFAS analytes (S2).

The samples were sufficiently homogeneous and stable for evaluation of participants' performance.

Of a possible 735 expected numeric results a total of 536 (73%) were submitted.

The assigned values were the robust average of participants results. The associated uncertainties were estimated from the robust standard deviation of the participants' results.

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

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

- *to compare the performances of participant laboratories and to assess their accuracy in the measurement of PFAS in fish and tomato matrices;*

  Laboratory performance was assessed using both z-scores and E_n-scores.
  Of 522 z-scores, 451 (86%) were satisfactory with $|z| \leq 2$.
  Of 522 E_n-scores, 390 (75%) were satisfactory with $|E_n| \leq 1$.
  Seventeen laboratories analysed all matrices, five laboratories analysed one matrix only.
  Laboratories **3** and **11** returned satisfactory z-scores for all analytes reported for which z-scores were calculated (35). No laboratory returned satisfactory E_n-scores for all analytes for which E_n-scores were calculated.
  Laboratory **9** requested only Sample S1 and returned satisfactory z and E_n-scores for all analytes reported for which scores were calculated (21).

- *evaluate the laboratories' methods;*

  Participants used a variety of methods for extraction. No correlation between results and method was evident. The analytical detection method of choice was LC-MS/MS.

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

  Five hundred and one of five hundred and thirty-six numeric results (93%) were reported with an associated estimate of expanded measurement uncertainty.
  The magnitude of the reported expanded uncertainties was within the range 1.7% to 100% of the reported value. Laboratory **10** did not report an estimate of measurement uncertainty, while laboratories **15**, **18** and **22** reported expanded measurement uncertainties for some analytes only.

1 INTRODUCTION

1.1 NMI Proficiency Testing Program

The National Measurement Institute (NMI) is responsible for Australia's national measurement infrastructure, providing a range of services including a chemical proficiency testing program. Proficiency testing (PT) is: 'evaluation of participant performance against pre-established criteria by means of interlaboratory comparison.'[1] NMI PT studies target chemical testing in areas of high public significance such as trade, environment, law enforcement and food safety. NMI offers studies in:

- pesticide residues in fruit and vegetables, soil and water;
- per- and polyfluorinated alkyl substances (PFAS);
- petroleum hydrocarbons in soil and water;
- inorganic analytes in soil, water, food and pharmaceuticals;
- controlled drug assay;
- folic acid in flour; and
- allergens in food.

1.2 Study Background

Per- and polyfluorinated alkyl substances (PFAS) are chemicals found in industrial products such as fire-fighting foams and non-stick coatings. Their resistance to degradation and potential toxicity makes them a growing global environmental concern. These complex contaminants are challenging to measure. PFOS the most commonly reported PFAS was added in 2010 to the list of chemicals regulated under the international Stockholm Convention for Persistent Organic Pollutants, to which Australia is a signatory.

1.3 Study Aims

The aims of the study were to:

- compare the performances of participant laboratories and assess their accuracy in the measurement of PFAS in fish and tomato matrices;
- evaluate the laboratories' test methods; and
- develop the practical application of traceability and measurement uncertainty and provide participants with information that will be useful in assessing their uncertainty estimates.

1.4 Study Conduct

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

The study falls within the scope of NMI's accreditation as a proficiency testing provider.

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of the study was:

Invitation issued	30 June 2019
Samples dispatched	23 July 2019
Results due	20 September 2019
Interim report issued	14 October 2019

2.2 Participation

One hundred and twenty-six Australian and international laboratories were invited to participate. Twenty-five laboratories participated and twenty-two submitted results by the due date.

2.3 Test Material Preparation

Two test samples were prepared in May/June 2019. Care was taken to avoid any Teflon contamination during sample preparation. Two samples were prepared:

- One fish sample S1 (5 g) with incurred PFAS contaminants and spiked with additional 11 PFAS components; and
- One tomato sample S2 (40 g) spiked with 11 individual PFAS components.

Adona, GenX, EtFOSA, EtFOSAA and MeFOSE were introduced for the first time in this PT study.

Seventeen analytical standards used for spiking samples in AQA 19-20 were purchased from HPC Standards GmbH, Toronto Research Chemicals, Sigma-Aldrich and Wellington Laboratories Canada.

Details of the spiked analytes and levels are presented in Table 1.

Table 1 Formulated concentrations of test samples

PFAS	S1 Fish µg/kg incurred + spiked	S2 Tomato µg/kg spiked
PFBA	-	50
PFPeA	3.01	47.2
PFHxA	-	25
PFHpA	-	13
PFOA	5.55	4.99
PFNA	2.51	1.00
PFDA	incurred	-
PFUnA	incurred	-
PFDoA	incurred	-
PFTrA	incurred	-
PFTeDA	incurred	-
PFBS	2.98	30.1
PFHxS	incurred	11
PFHpS	incurred	-
PFOS	incurred	2.77

PFAS	S1 Fish µg/kg incurred + spiked	S2 Tomato µg/kg spiked
PFNS	incurred	-
PFDS	incurred	-
PFOSA	4.94	5.01
EtFOSA	9.51	-
EtFOSAA	12.2	-
MeFOSE	15.0	-
6:2 FTS	3.87	9.10
8:2 FTS	incurred	9.19
10:2 FTS	incurred	-
GenX	20.4	19.2
ADONA	33.4	31.6

2.4 Test Material Homogeneity and Stability Testing

Tomato sample was introduced for the first time in this PT study. The preparation, homogeneity testing and stability check is presented in Appendix 1. No homogeneity or stability testing was conducted on the fish sample. This sample was prepared and packaged using a process that has been demonstrated to produce homogeneous and stable samples for previous NMI PFAS PT studies. The results of the study gave no reason to question the homogeneity or stability.

Tomato sample was demonstrated to be sufficiently homogeneous and stable for use in this PT study.

2.5 Laboratory Code

All laboratories that agreed to participate were assigned a confidential code number.

2.6 Sample Storage, Dispatch and Receipt

Prior to dispatch, fish and tomato samples were kept frozen, fish samples at -80°C and tomato at -20°C.

Participants were sent 5 g fish in Greiner tubes for Sample S1 and 40 g tomato puree also in Greiner tubes for Sample S2. The samples were packed in a foam box with a cooler brick and sent by courier on 23 July 2019.

The following items were packaged with the samples:

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

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

2.7 Instructions to Participants

Participants were instructed as follows:

- Quantitatively analyse the samples using your normal test method.
- Report results in units of **µg/kg** on **as received basis** for both samples.

- For each analyte in each sample report a single result expressed as if reporting to a client (i.e. corrected for recovery or not, according to your standard procedure). This figure will be used in all statistical analysis in the study report.
- For each analyte in each sample report the associated expanded measurement uncertainty (e.g. 0.50 ± 0.02 µg/kg).
- Report any analyte not tested as NT.
- No limit of reporting has been set for this study. Report results as you would to a client, applying the limit of reporting of the method used for analysis.
- Report the basis of your uncertainty estimates (e.g. uncertainty budget, repeatability precision, long term result variability).
- Return the completed results sheet by e-mail (proficiency@measurement.gov.au),
- **Please return completed result sheet by 20 September 2019. Late results cannot be included in the study report.**

2.8 Interim Report

An interim report was emailed to participants on 14 October 2019.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Methods Reported by Participants

Table 2 Test Methods – Samples S1 Fish

Lab Code	Sample Weight (g)	Sample pretreatment	Extraction Technique	Extraction Solvent(s):	Extraction Process	Extraction Temperature	Extraction Clean Up	Instrument	Column Type	Column Specifications	Delay Column
1	2	homogenisation	Alkaline digestion	Basified MeOH	sonication/shaking/SPE	Room	Envicarb	LC-MSMS or LCQQQ	C18	50 mm x 2.1mm x 1.8um	Yes
2	1	homogenisation	Alkaline digestion	KOH/methanol	Shaker/Sonication	Room (3 hour shake, 12 hour sonication bath)	SPE (WAX 150mg/6cc)	LC-MSMS or LCQQQ	C18	10cm x 3.0mm x 3um	No
3	1	homogenisation	Solid Liquid (SLE)	Acetonitrile	Shaking	Ambient 15mins	SPE	Orbitrap	C18	50 cm x 2.1 cm x 2.7 um	Yes
4	2	homogenisation	Solid Liquid (SLE)	Acetonitrile	Sonication	room	C18 d-SPE clean up	LC-MSMS or LCQQQ	C18	2.0mmx50mm(1.6um)	Yes
5		homogenisation	Alkaline digestion	methanol	Sonication		SPE (Oasis WAX)	LC-MSMS or LCQQQ validation by LC-QTOF	C 18	150 x 2 mm 3 µm	Yes
6	1	homogenisation	QuEChERS extraction	Acetonitrile	Vortex, shaking, centrifuge	Sonicate 30 min at 30-35 degrees	dSPE	Orbitrap	Kinetex C18	100x3mm 2.6 um	No
7		homogenisation	Solid Phase (SPE)	Acetonitrile	Sonication + Quechers	30min / 40°C	active carbon / SPE	LC-MSMS or LCQQQ	Nucleodur Shinx RP 3µm		Yes
8	0.25		Solid Phase (SPE)	NaOH-Methanol	Vortex, Sonication, Shaking	Room (60 min)	SPE WAX	LC-MSMS or LCQQQ	C18	10 cm x 3 mm x 3.5 µm	No
9		pH adjustment	Solid Liquid (SLE)	KOH-Methanol	Shaking	Room (16 hours)	Ultracentrifugation, SPE (Oasis, Wax)	LC-MSMS or LCQQQ	C18	5 cm x 2.1 mm x 1.7µm	Yes

Lab Code	Sample Weight (g)	Sample pretreatment	Extraction Technique	Extraction Solvent(s):	Extraction Process	Extraction Temperature	Extraction Clean Up	Instrument	Column Type	Column Specifications	Delay Column
10	0.5471 and 0.5053	pH adjustment	Solid Liquid (SLE)	Acetonitrile	Sonication & Shaking	Room (15+15mins x 2)	SPE (Oasis WAX), active carbon (Strata GCB cartridge)	LC-MSMS or LCQQQ	CSH Phenyl-Hexyl	1.7um, 2.1 x 100mm	Yes
11	1	homogenisation	Solid Liquid (SLE)	Acetonitrile	Shaking	Ambient 15mins	SPE	Orbitrap	C18	50 cm x 2.1 cm x 2.7 um	Yes
14		homogenisation	Solid Phase (SPE)	Methanol	tumbling/sonication	Room		LC-MSMS or LCQQQ	C18	2.1mm x 50mm x 2.7um	No
15		homogenisation	Alkaline digestion	KOH-methanol	Tumbling	Room (8 hrs)	active carbon SPE	LC-MSMS or LCQQQ	C18		No
16	2	NA	Solid Liquid (SLE)	2% formic acid in acetonitrile	Shaking Merris-Minimix shaker	Room temperature (8min)	dSPE (C18, Envicarb, MgSO4)	LC-MSMS or LCQQQ	Zorbax XDB-C18	100 mm x 2.1 mm, 1.8µm	Yes
17	1.02	homogenisation	Solid Phase (SPE)		Shaking	room	SPE cartridge	LC-MSMS or LCQQQ	BEH C18	100mm x 2.1 mm	No
18		homogenisation	QuEChERS	Acetonitrile	Shaking	Room (60 Mins)	C18 & active carbon	LC-MSMS or LCQQQ	C18	1.6µm, 2.0mm x 50mm	Yes
19		no	Solid Liquid (SLE)	acetonitrile	vortex, polytron, sonication, tumbling	Room (20 min)	SPE-WAX (Oasis); ultracentrifugation	LC-MSMS or LCQQQ	NX-C18	15 cm x 2 mm x 3 um	Yes
20		Digestion with 200 mM NaOH	Sonication	Acetonitrile	Sonication	Room Temperature (15minx2)	liquid liquid extraction(n-hexane) followed by active carbon	HPLC-MS/MS SCIEX Triple Quad 6500+	EVO C18	10cm x 2mm x 2.6µm	Yes

Lab Code	Sample Weight (g)	Sample pretreatment	Extraction Technique	Extraction Solvent(s):	Extraction Process	Extraction Temperature	Extraction Clean Up	Instrument	Column Type	Column Specifications	Delay Column
21	0.2	homogenisation	Alkaline digestion	NaOH-methanol	Tumbling	Room(16h)	SPE	LC-MSMS or LCQQQ	PFP	15cm×2.1mm× 1.8µm	Yes
22	1.05	homogenisation	Solid Phase (SPE)	0.2 M sodium hydroxide solution and Acetonitrile	Sonication and shaking	Room 20 mins	SPE	LC-MSMS or LCQQQ	C18	50 x 2.1mm, 1.9 µm	Yes
23		homogenisation	Alkaline digestion	acetonitrile	Sonication	Room temp (2 x 20min)	SPE (WAX)	LC-MSMS or LCQQQ	C18	1.6 µm, 50 mm x 2.0 mm	Yes

Table 3 Test Methods – Samples S1 Fish (continued)

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
1	PFBA: MPFBA; PFPeA: M3PFPeA; PFHxA: MPFHxA; PFHpA: M4PFHpA; PFOA: MPFOA; PFNA: MPFNA; PFDA: MPFDA; PFUnA: MPFUdA; PFDoA: MPFDoA; PFTrA: MPFDoA; PFTeDA: M2PFTeDA; PFBS: M3PFBS; PFPeS: MPFHxS; PFHxS: MPFHxS; PFHpS: MPFOS; PFOS: MPFOS; PFDS: MPFOS; PFOSA: M8FOSA; MeFOSA: d-N-MeFOSA-M; EtFOSA: d-N-EtFOSA-M; MeFOSAA: d3-N-MeFOSAA; EtFOSAA: d5-N-EtFOSAA; MeFOSE: d7-N-MeFOSE; EtFOSE: d9-N-EtFOSE-M; 6:2 FTS: M2-6:2FTS; 8:2 FTS: M2-6:2FTS; 10:2 FTS: M2-8:2FTS	Wellington	Yes	All added before extraction	No In house	No
2	PFBA: 13C4 PFBA; PFPeA: 13C5 PFPeA; PFHxA: 13C2 PFHxA; PFHpA: 13C4 PFHpA; PFOA: 13C4 PFOA; PFNA: 13C5 PFNA; PFDA: 13C2 PFDA; PFUnA: 13C2 PFUnA; PFDoA: 13C2 PFDoA; PFTrA: 13C2 PFDoA; PFTeDA: 13C2 PFTeDA; PFBS: 18O2 PFHxS; PFPeS: 18O2 PFHxS; PFHxS: 18O2 PFHxS; PFHpS: 13C4 PFOS; PFOS: 13C4 PFOS; PFNS: 13C4 PFOS; PFDS: 13C4 PFOS; PFOSA: 13C8 PFOSA; MeFOSA: d-N-MeFOSA-	Wellington	Yes		No	No

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
	M; EtFOSA: d-N-EtFOSA-M; MeFOSAA: d3-NMeFOSAA; EtFOSAA: d7-NEtFOSAA; MeFOSE: d7-N-MeFOSE-M; EtFOSE: d9-N-EtFOSA-M; 6:2 FTS: M2-6:2 FTS; 8:2 FTS: M2-8:2 FTS; 10:2 FTS: M2-8:2 FTS; GenX: 13C3 HFPO-DA; ADONA: 13C4 PFOS					
3	PFBA: PFBA-13C4; PFPeA: PFPeA-13C3; PFHxA: PFHxA-13C2; PFHpA: PFHpA-13C4; PFOA: PFOA-13C4; PFNA: PFNA-13C5; PFDA: PFDA-13C2; PFUnA: PFUnDA-13C2; PFDoA: PFDoDA-13C2; PFTrA: PFDoDA-13C2; PFTeDA: PFTeDA-13C2; PFBS: PFBS-13C3; PFPeS: PFHxS-18O2; PFHxS: PFHxS-18O2; PFHpS: PFOS-13C4; PFOS: PFOS-13C4; PFNS: PFOS-13C4; PFDS: 8:2 FTS-13C2; PFOSA: FOSA-13C8; MeFOSA: MeFOSA-D3; EtFOSA: EtFOSA-D5; MeFOSAA: MeFOSAA-D3; EtFOSAA: EtFOSAA-D5; MeFOSE: MeFOSE-D7; EtFOSE: EtFOSE-D9; 6:2 FTS: 6:2 FTS-13C2; 8:2 FTS: 8:2 FTS-13C2; 10:2 FTS: 8:2 FTS-13C2; GenX: HFPO-DA-13C3; ADONA: PFHxS-18O2	Wellington Laboratories	Yes	All: PFOA-13C8	No	No
4	PFBA: 13C4 PFBA ISTD; PFPeA: 13C4 PFBA ISTD; PFHxA: 13C2 PFHxA ISTD; PFHpA: 13C2 PFHxA ISTD; PFOA: 13C4 PFOA ISTD; PFBS: 18O2 PFHxS ISTD; PFHxS: 18O2 PFHxS ISTD; PFOS: 13C4 PFOS ISTD; 6:2 FTS: 13C2 12C6 62FTS ISTD; 8:2 FTS: 13C2 12C6 62FTS ISTD	Wellington	Yes	PFBA: 13C4 PFBA ISTD; PFPeA: 13C4 PFBA ISTD; PFHxA: 13C2 PFHxA ISTD; PFHpA: 13C2 PFHxA ISTD; PFOA: 13C4 PFOA ISTD; PFBS: 18O2 PFHxS ISTD; PFHxS: 18O2 PFHxS ISTD; PFOS: 13C4 PFOS ISTD; 6:2 FTS: 13C2 12C6 62FTS ISTD; 8:2 FTS: 13C2 12C6 62FTS ISTD	Yes	Yes
5	PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C8-PFOA; PFNA: 13C9-PFNA; PFDA: 13C6-PFDA; PFUnA: 13C7-PFUnA; PFDoA: 13C2-PFDoA; PFBS: 13C3-PFBS; PFHxS: 13C3-PFHxS; PFOS: 13C8-PFOS; PFOSA: 13C8-PFOSA; 6:2 FTS: 13C2-6:2 FTS; 8:2 FTS: 13C2-8:2 FTS	Wellington	Yes		No	No
6	PFBA: Perfluoro-n-[13C4]butanoic acid MPFBA; PFPeA: Perfluoro-n-[13C5]pentanoic acid M5PFPeA; PFHxA: Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid M5PFHxA; PFHpA: Perfluoro-n-[1,2,3,4-13C4]heptanoic acid	Wellington	Yes		No	Yes

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
	M4PFHpA; PFOA: Perfluoro-n-[13C8]octanoic acid M8PFOA; PFNA: Perfluoro-n-[13C9]nonanoic acid M9PFNA; PFDA: Perfluoro-n-[1,2,3,4,6-13C6]decanoic acid M6PFDA; PFUnA: Perfluoro-n-[1,2,3,4,6,7-13C7]undecanoic acid M7PFUdA; PFDoA: Perfluoro-n-[1,2 13C2]dodecanoic acid MPFDoA; PFTeDA: Perfluoro-n-[1,2 13C2]tetradecanoic acid M2PFTeDA; PFBS: Sodium perfluoro-1-[2,3,4 13C3] butanesulfonate M3PFBS; PFHxS: Sodium perfluoro-1-[1,2,3 13C3] hexanesulfonate M3PFHxS; PFOS: Sodium perfluoro-1-[13C8] ocatanesulfonate M8PFOS; PFOSA: Perfluoro-1-[13C8]otanesulfonamide; MeFOSA: N-methyl-d3-perfluoro-1-octancesulfonamide; EtFOSA: N-ethyl-d5-perfluoro-1-octanesulfonamide; EtFOSAA: N-ethyl-d5-perfluoro-1-octanesulfonamide					
7	PFBA: 13C4 PFBA; PFPeA: 13C5 PFPeA; PFHxA: 13C12 PFHxA; PFHpA: 13C4 PFHpA; PFOA: 13C8 PFOA; PFNA: 13C5 PFNA; PFDA: 13C2 PFDA; PFUnA: 13C2 PFUnA; PFDoA: 13C2 PFDoA; PFTrA: 13C2 PFTeDA; PFTeDA: 13C2 PFTeDA; PFBS: 13C3 PFBS; PFPeS: 13C3 PFBS; PFHxS: 18O2 PFHxS; PFHpS: 18O2 PFHxS; PFOS: 13C4 PFOS; PFDS: 13C2 PFUnA; PFOSA: 13C8 PFOSA; MeFOSA: d3-MeFOSA; EtFOSA: d5-EtFOSA; MeFOSAA: d3-N-MeFOSAA; EtFOSAA: d5-N-EtFOSAA; MeFOSE: d7-MeFOSE; EtFOSE: d9-EtFOSE; 6:2 FTS: 13C2 6:2 FTS; 8:2 FTS: 13C 8:2 FTS; 10:2 FTS: 13C 8:2 FTS	Wellington		For all PFAS tested: 13C4 PFOA		No
8	PFOA: 13C8-PFOA; PFOS: 13C8-PFOS	Cambridge Isotope Laboratories	Yes	PFOA: yes; PFOS: yes	No	No

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
9	PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C8-PFOA; PFNA: 13C9-PFNA; PFDA: 13C6-PFDA; PFUnA: 13C7-PFUnA; PFDoA: 13C2-PFDoA; PFTrA: 13C2-PFTeDA; PFTeDA: 13C2-PFTeDA; PFBS: 13C3-PFBS; PFPeS: 13C3-PFHxS; PFHxS: 13C3-PFHxS; PFHpS: 13C8-PFOS; PFOS: 13C8-PFOS; PFNS: 13C8-PFOS; PFDS: 13C8-PFOS; PFOSA: 13C8-PFOSA; MeFOSA: D3-N-MeFOSA; EtFOSA: D5-N-EtFOSA; MeFOSAA: D3-MeFOSAA; EtFOSAA: D5-EtFOSAA; MeFOSE: D7-N-MeFOSE; EtFOSE: D9-N-EtFOSE; 6:2 FTS: 13C2-6:2 FTS; 8:2 FTS: 13C2-8:2 FTS; GenX: 13C3-HFPO-DA; ADONA: 13C3-HFPO-DA		Yes	PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C8-PFOA; PFNA: 13C9-PFNA; PFDA: 13C6-PFDA; PFUnA: 13C7-PFUnA; PFDoA: 13C2-PFDoA; PFTrA: 13C2-PFTeDA; PFTeDA: 13C2-PFTeDA; PFBS: 13C3-PFBS; PFPeS: 13C3-PFHxS; PFHxS: 13C3-PFHxS; PFHpS: 13C8-PFOS; PFOS: 13C8-PFOS; PFNS: 13C8-PFOS; PFDS: 13C8-PFOS; PFOSA: 13C8-PFOSA; MeFOSA: D3-N-MeFOSA; EtFOSA: D5-N-EtFOSA; MeFOSAA: D3-MeFOSAA; EtFOSAA: D5-EtFOSAA; MeFOSE: D7-N-MeFOSE; EtFOSE: D9-N-EtFOSE; 6:2 FTS: 13C2-6:2 FTS; 8:2 FTS: 13C2-8:2 FTS; GenX: 13C3-HFPO-DA; ADONA: 13C3-HFPO-DA	No	No
10	PFBA: MPFBA; PFPeA: M5PFPeA; PFHxA: M5PFHxA; PFHpA: M4PFHpA; PFOA: M8PFOA; PFNA: M9PFNA; PFDA: M6PFDA; PFUnA: M7PFUnA; PFDoA: MPFDoA; PFTrA: M2PFTeDA; PFTeDA: M2PFTeDA; PFHxS: M3PFHxS; PFHpS: M8PFOS; PFOS: M8PFOS	Chiron & Wellington	Yes	PFOA: M2PFOA; PFOS: MPFOS	No	No
11	PFBA: PFBA-13C4; PFPeA: PFPeA-13C3; PFHxA: PFHxA-13C2; PFHpA: PFHpA-13C4; PFOA: PFOA-13C4; PFNA: PFNA-13C5; PFDA: PFDA-13C2; PFUnA: PFUnDA-13C2; PFDoA: PFDoDA-13C2; PFTrA: PFDoDA-13C2; PFTeDA: PFTeDA-13C2; PFBS: PFBS-13C3; PFPeS: PFHxS-18O2; PFHxS: PFHxS-18O2; PFHpS: PFOS-13C4; PFOS: PFOS-13C4; PFNS: PFOS-13C4; PFDS: 8:2 FTS-13C2; PFOSA: FOSA-13C8; MeFOSA: MeFOSA-D3; EtFOSA: EtFOSA-D5; MeFOSAA: MeFOSAA-D3; EtFOSAA: EtFOSAA-D5; MeFOSE: MeFOSE-D7; EtFOSE: EtFOSE-D9; 6:2 FTS: 6:2 FTS-13C2; 8:2 FTS: 8:2 FTS-13C2; 10:2 FTS: 8:2	Wellington Laboratories	Yes	All: PFOA-13C8	No	No

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
	FTS-13C2; GenX: HFPO-DA-13C3; ADONA: PFHxS-18O2					
14	PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C2-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C8-PFOA; PFNA: 13C5-PFNA; PFDA: 13C6-PFDA; PFUnA: 13C2-PFUnA; PFDoA: 13C2-PFDoA; PFBS: 13C3-PFBS; PFHxS: 18O2-PFHxS; PFOS: 13C8-PFOS; PFOSA: 13C8-FOSA; MeFOSA: d3-N-MeFOSA; EtFOSA: d5-N-EtFOSA; MeFOSAA: d3-N-MeFOSAA; EtFOSAA: d5-N-EtFOSAA; MeFOSE: d7-N-MeFOSE; EtFOSE: d9-N-EtFOSE; 6:2 FTS: 13C2-6-2 FTS; 8:2 FTS: 13C2-8-2 FTS; GenX: 13C3-GenX	Wellington	Yes		No	No
15	PFBA: 13C4 PFBA; PFPeA: 13C5 PFPeA; PFHxA: 13C5 PFHxA; PFHpA: 13C4 PFHpA; PFOA: 13C8 PFOA; PFNA: 13C9 PFNA; PFDA: 13C6 PFDA; PFUnA: 13C7 PFUnA; PFDoA: 13C2 PFDoA; PFTrA: 13C2 PFDoA; PFTeDA: 13C2 PFTeDA; PFBS: 13C3 PFBS; PFPeS: 13C3 PFBS; PFHxS: 13C3 PFHxS; PFHpS: 13C3 PFHxS; PFOS: 13C6 PFOS; PFNS: 13C6 PFOS; PFDS: 13C6 PFOS; PFOSA: 13C8 PFOSA; MeFOSAA: d3-N-MeFOSAA; EtFOSAA: d5-N-EtFOSAA; 6:2 FTS: 13C2 6:2 FTS; 8:2 FTS: 13C2 8:2 FTS; 10:2 FTS: 13C2 8:2 FTS	Wellington	Yes	PFBA: 13C3 PFBA; PFOA: 13C2 PFOA; PFDA: 13C2 PFDA; PFOS: 13C4 PFOS		No
16	PFBA: M4PFBA; PFPeA: M5PFPeA; PFHxA: M5PFHxA; PFHpA: MPFHpA; PFOA: M8PFOA; PFNA: M9PFNA; PFDA: M6PFDA; PFUnA: M7PFUnDA; PFDoA: MPFDoDA; PFTrA: MPFDoDA; PFTeDA: MPFTeDA; PFBS: M3PFBS; PFPeS: M5PFHxA; PFHxS: M3PFHxS; PFHpS: M3PFHxS; PFOS: M8PFOS; PFNS: M8PFOS; PFDS: M8PFOS; PFOSA: MPFOSA; MeFOSA: d-NMeFOSA-M; EtFOSA: d-NEtFOSA-M; MeFOSAA: d3-NMeFOSAA; EtFOSAA: d5-NEtFOSAA; MeFOSE: d7-NMeFOSE-M; EtFOSE: d9-NEtFOSE-M; 6:2 FTS: M6:2 FTS; 8:2 FTS: M8:2 FTS	Wellington Laboratory	Yes		No	No

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
17	PFBA: 13c3-PFBA; PFPeA: 13C3-PFPeA; PFHxA: 13C2-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C2-PFOA; PFNA: 13C5-PFNA; PFDA: 13C2-PFDA; PFUnA: 13C2-PFUdA; PFDoA: 13C2-PFDoA; PFTrA: 13C2-PFDoA; PFTeDA: 13C2-PFTeDA; PFBS: 13C3-PFBS; PFPeS: 13C3-PFBS; PFHxS: 13C3-PFHxS; PFHpS: 13C2-PFOA; PFOS: 13C8-PFOS; PFNS: 13C8-PFOS; PFDS: 13C8-PFOS; PFOSA: 13C8-PFOSA; MeFOSA: d3-N-MeFOSA; EtFOSA: d5-N-EtFOSA; MeFOSAA: d3-N-MeFOSAA; EtFOSAA: d5-N-EtFOSAA; MeFOSE: d7-N-MeFOSE; EtFOSE: d9-N-EtFOSE; 6:2 FTS: 13C2-6:2 FTS; 8:2 FTS: 13C2-8:2 FTS; 10:2 FTS: 13C2-PFDoA; GenX: 13C3-HFPO-DA; ADONA: 13C4-PFHpA	Wellington	Yes	PFBA: 13C8-PFBA; PFHxA: 13C5-PFHxA; PFOA: 13C8-PFOA; PFNA: 13C9-PFNA; PFDA: 13C6-PFDA; PFUnA: 13C7-PFUdA; PFHxS: 18O2-PFHxS; PFOS: 13C4-PFOS	No	No
18	PFOA: 13C8-PFOA; PFOS: 13C4-PFOS	Wellington Laboratories	No	PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C4-PFOA; PFNA: 13C5-PFNA; PFDA: 13C6-PFDA; PFUnA: 13C2-PFUnDA; PFDoA: 13C2-PFDoDA; PFTrA: 13C2-PFTeDA; PFTeDA: 13C2-PFTeDA; PFBS: 13C3-PFBS; PFPeS: 16O2-PFHxS; PFHxS: 16O2-PFHxS; PFHpS: 13C8-PFOS; PFOS: 13C8-PFOS; PFDS: 13C8-PFOS; PFOSA: 13C8-FOSA; MeFOSA: d3-MeFOSA; EtFOSA: d5-EtFOSA; MeFOSAA: d7-MeFOSE; EtFOSAA: d3EtFOSE; MeFOSE: d3-MeFOSAA; EtFOSE: d5-EtFOSAA; 6:2 FTS: 13C2-6:2 FTS; 8:2 FTS: 13C2-8:2 FTS; 10:2 FTS: 13C2-8:2 FTS	No	No
19	PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C4-PFOA; PFNA: 13C9-PFNA; PFDA: 13C2-PFDA; PFUnA: 13C2-PFUnA; PFDoA: 13C2-PFDoA; PFTrA: 13C2-PFHxDA; PFTeDA: 13C2-PFHxDA; PFBS: 18O2-PFHxS; PFPeS: 18O2-PFHxS; PFHxS: 18O2-PFHxS; PFHpS: 18O2-PFHxS; PFOS: 13C4-PFOS; PFNS: 13C4-PFOS; PFDS: 13C4-PFOS; PFOSA: 13C8-PFOSA	Wellington	Yes	PFBA: 13C8-PFOA; PFPeA: 13C8-PFOA; PFHxA: 13C8-PFOA; PFHpA: 13C8-PFOA; PFOA: 13C8-PFOA; PFNA: 13C5-PFNA; PFDA: 13C5-PFNA; PFUnA: 13C5-PFNA; PFDoA: 13C5-PFNA; PFTrA: 13C2-PFTeDA; PFTeDA: 13C2-PFTeDA; PFBS: 18O2-PFOS; PFPeS: 18O2-PFOS; PFHxS: 18O2-PFOS; PFHpS: 18O2-PFOS; PFOS:	No	Yes

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
				18O2-PFOS; PFNS: 18O2-PFOS; PFDS: 18O2-PFOS; PFOSA: 13C2-PFTeDA		
20	PFBA: 13C4-PFBA; PFPeA: 13C3 PFPeA; PFHxA: 13C2-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C4-PFOA; PFNA: 13C5-PFNA; PFDA: 13C2-PFDA; PFUnA: 13C2PFUdA; PFDoA: 13C2PFDoA; PFTrA: 13C2PFDoA; PFTeDA: 13C2PFTeDA; PFBS: 13C3 PFBS; PFPeS: 13C3 PFBS; PFHxS: 18O2-PFHxS; PFHpS: 18O2-PFHxS; PFOS: 13C4-PFOS; PFNS: 13C4-PFOS; PFDS: 13C4-PFOS; PFOSA: 13C8-FOSA; MeFOSA: D3-N-Me FOSA; EtFOSA: D5-N-Et FOSA; MeFOSAA: D3-N-Me FOSAA; EtFOSAA: D5-N-Et FOSAA; MeFOSE: D7-N-Me FOSE; EtFOSE: D9-N-Et FOSE; 6:2 FTS: 13C26:2 FTS; 8:2 FTS: 13C2-8:2 FTS; 10:2 FTS: 13C2-8:2 FTS	Wellington	Yes	PFBA: 13C3-PFBA; PFPeA: 13C5 -PFPeA; PFHxA: 13C5 -PFPeA; PFHpA: 13C8-PFOA; PFOA: 13C8-PFOA; PFNA: 13C8-PFOA; PFDA: 13C8-PFOA; PFUnA: 13C8-PFOA; PFDoA: 13C8-PFOA; PFBS: 18O3-PFHxS; PFPeS: 18O3-PFHxS; PFHxS: 18O3-PFHxS; PFHpS: 18O3-PFHxS; PFOS: 13C8-PFOS	No	No
21	PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C8-PFOA; PFNA: 13C9-PFNA; PFDA: 13C6-PFDA; PFUnA: 13C7-PFUnA; PFDoA: 13C2-PFDoA; PFTrA: 13C2-PFDoA; PFTeDA: 13C2-PFTeDA; PFBS: 13C3-PFBS; PFPeS: 13C3-PFHxS; PFHxS: 13C3-PFHxS; PFHpS: 13C3-PFHxS; PFOS: 13C8-PFOS; PFNS: 13C8-PFOS; PFDS: 13C8-PFOS; PFOSA: 13C8-FOSA; 6:2 FTS: 13C2-6:2FTS; 8:2 FTS: 13C2-8:2FTS; 10:2 FTS: 13C2-8:2FTS; GenX: 13C3-GenX; ADONA: 13C8-PFOA	Wellington	No		No	Yes
22	All: C8-PFOS	Wellington	No	PFBA: 13C4-PFBA; PFPeA: 13-C4 PFOS; PFHxA: 13-C4 PFOS; PFHpA: 13-C4 PFOS; PFOA: 13-C4 PFOS; PFNA: 13-C4 PFOS; PFDA: 13-C4 PFOS; PFUnA: 13C2-PFDOA; PFDoA: 13C2-PFDOA; PFTrA: 13C2-PFDOA; PFTeDA: 13C2-PFDOA; PFBS: 13-C4 PFOS; PFPeS: 13-C4 PFOS; PFHxS: 13-C4 PFOS; PFHpS: 13-C4 PFOS; PFOS: 13-C4 PFOS; PFNS: 13C2-PFDOA; PFDS: 13-C4 PFOS; PFOSA: 13-C4 PFOS; MeFOSA: d5-EtFOSA; EtFOSA: d5-EtFOSA; MeFOSAA: d5-EtFOSA; EtFOSAA: d5-	Yes MOECC E3506	No

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
				EtFOSA; MeFOSE: d5-EtFOSA; EtFOSE: d5-EtFOSA; 6:2 FTS: 13C2 4:2-FTS; 8:2 FTS: 13C2 4:2-FTS; 10:2 FTS: 13C2 4:2-FTS; GenX: M3-HFPO-DA; ADONA: 13C2-PFDOA		
23	PFHxA: M2PFHxA; PFHpA: M4PFHpA; PFOA: M4PFOA; PFNA: M5PFNA; PFDA: M2PFDA; PFBS: M3PFBS; PFHxS: MPFHxS; PFOS: M4PFOS; 6:2 FTS: M2-6:2 FTS; 8:2 FTS: M2-8:2 FTS	Wellington	Yes	PFHxA: M8PFOA; PFHpA: M8PFOA; PFOA: M8PFOA; PFNA: M8PFOA; PFDA: M8PFOA; PFBS: M8PFOS; PFHxS: M8PFOS; PFOS: M8PFOS; 6:2 FTS: M8PFOS; 8:2 FTS: M8PFOS	No	No

Table 4 Test Methods – Samples S2 Tomato

Lab Code	Sample Weight (g)	Sample pretreatment	Extraction Technique	Extraction Solvent(s):	Extraction Process	Extraction Temperature	Extraction Clean Up	Instrument	Column Type	Column Specifications	Delay Column
1	2	homogenisation	Alkaline digestion	Basified MeOH	sonication/shaking/SPE	Room	Envicarb	LC-MSMS or LCQQQ	C18	50 mm x 2.1mm x 1.8um	No
2	1.01	None	Alkaline digestion	KOH/methanol	Shaker/Sonication	Room (3 hour shake, 12 hour sonication bath)	SPE (WAX 150mg/6cc)	LC-MSMS or LCQQQ	C18	10cm x 3.0mm x 3um	No
3	1	homogenisation	Solid Liquid (SLE)	Acetonitrile	Shaking	Ambient 15mins	SPE	Orbitrap	C18	50 cm x 2.1 cm x 2.7 um	Yes
5		homogenisation	Alkaline digestion	methanol	Sonication		SPE (Oasis WAX)	LC-MSMS or LCQQQ validation by LC-QTOF	C 18	150 x 2 mm 3 µm	Yes
6	10	homogenisation	QuEChERS extraction	Acetonitrile	vortex, shaking, centrifuge	Sonicate 30 min at 30-35 degrees	envicarb	Orbitrap	Kinetex C18	100x3mm 2.6 um	No

Lab Code	Sample Weight (g)	Sample pretreatment	Extraction Technique	Extraction Solvent(s):	Extraction Process	Extraction Temperature	Extraction Clean Up	Instrument	Column Type	Column Specifications	Delay Column
7		homogenisation	Solid Phase (SPE)	Acetonitrile	Sonication + Quechers	30min / 40° C	active carbon / SPE	LC-MSMS or LCQQQ	Nucleodur Shinx RP 3µm		Yes
8	1		Solid Phase (SPE)	NaOH-Methanol	Vortex, Sonication, Shaking	Room (60 min)	SPE WAX	LC-MSMS or LCQQQ	C18	10 cm x 3 mm x 3.5 µm	No
11	2	homogenisation	Solid Liquid (SLE)	Acetonitrile	Shaking	Ambient 15mins	SPE	Orbitrap	C18	50 cm x 2.1 cm x 2.7 um	Yes
14		homogenisation	Solid Phase (SPE)	Methanol	tumbling/sonication	Room		LC-MSMS or LCQQQ	C18	2.1mm x 50mm x 2.7um	No
15		homogenisation	Alkaline digestion	KOH-methanol	Tumbling	Room (8 hrs)	active carbon SPE	LC-MSMS or LCQQQ	C18		No
16	2	NA	Solid Liquid (SLE)	2% formic acid in acetonitrile	Shaking Merris-Minimix shaker	Room temperature (8min)	dSPE (C18, Envicarb, MgSO4)	LC-MSMS or LCQQQ	Zorbax XDB-C18	100 mm x 2.1 mm, 1.8µm	Yes
17	24.2	homogenisation	Solid Phase (SPE)		Shaking	room	SPE cartridge	LC-MSMS or LCQQQ	BEH C18	100mm x 2.1 mm	No
18		homogenisation	QuEChERS	Acetonitrile	Shaking	Room (60 Mins)	C18 & active carbon	LC-MSMS or LCQQQ	C18	1.6µm, 2.0mm x 50mm	Yes
19		no	Solid Liquid (SLE)	acetonitrile	vortex, sonication, tumbling	Room (20 min)	SPE-WAX (Oasis); ultracentrifugation	LC-MSMS or LCQQQ	NX-C18	15 cm x 2 mm x 3 um	Yes
20		Base digestion NaOH	Quechers	Acetonitrile		Room Temp	SPE (W-AX)	HPLC-MS/MS SCIEX Triple Quad 6500+	EVO C18	10cm x 2mm x 2.6µm	Yes

Lab Code	Sample Weight (g)	Sample pretreatment	Extraction Technique	Extraction Solvent(s):	Extraction Process	Extraction Temperature	Extraction Clean Up	Instrument	Column Type	Column Specifications	Delay Column
21	0.4	homogenisation	Alkaline digestion	NaOH-methanol	Tumbling	Room(16h)	SPE	LC-MSMS or LCQQQ	PFP	15cm×2.1mm ×1.8µm	Yes
22	1.06	homogenisation	Solid Phase (SPE)	0.2 M sodium hydroxide solution and Acetonitrile	Sonication and shaking	Room 20 mins	SPE	LC-MSMS or LCQQQ	C18	50 x 2.1mm, 1.9 µm	Yes
24	11.5 dried to 0.505	Homogenisation desiccation	Soxhlet	Methanol	hot extraction	4h	centrifugation	LC-MSMS or LCQQQ	Biphenyl	15cmx2.1mm x3.5µm	Yes

Table 5 Test Methods – Samples S2 Tomato (Continued)

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
1	PFBA: MPFBA; PFPeA: M3PFPeA; PFHxA: MPFHxA; PFHpA: M4PFHpA; PFOA: MPFOA; PFNA: MPFNA; PFDA: MPFDA; PFUnA: MPFUdA; PFDoA: MPFDoA; PFTrA: MPFDoA; PFTeDA: M2PFTeDA; PFBS: M3PFBS; PFPeS: MPFHxS; PFHxS: MPFHxS; PFHpS: MPFOS; PFOS: MPFOS; PFDS: MPFOS; PFOSA: M8FOSA; MeFOSA: d-N-MeFOSA-M; EtFOSA: d-N-EtFOSA-M; MeFOSAA: d3-N-MeFOSAA; EtFOSAA: d5-N-EtFOSAA; MeFOSE: d7-N-MeFOSE; EtFOSE: d9-N-EtFOSE-M; 6:2 FTS: M2-6:2FTS; 8:2 FTS: M2-6:2FTS; 10:2 FTS: M2-8:2FTS	Wellington	Yes	All added before extraction	No In house	No
2	PFBA: 13C4 PFBA; PFPeA: 13C5 PFPeA; PFHxA: 13C2 PFHxA; PFHpA: 13C4 PFHpA; PFOA: 13C4 PFOA; PFNA: 13C5 PFNA; PFDA: 13C2 PFDA; PFUnA: 13C2 PFUnA; PFDoA: 13C2 PFDoA; PFTrA: 13C2 PFDoA;	Wellington	Yes		No	No

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
	PFTeDA: 13C2 PFTeDA; PFBS: 18O2 PFHxS; PFPeS: 18O2 PFHxS; PFHxS: 18O2 PFHxS; PFHpS: 13C4 PFOS; PFOS: 13C4 PFOS; PFNS: 13C4 PFOS; PFDS: 13C4 PFOS; PFOSA: 13C8 PFOSA; MeFOSA: d-N-MeFOSA-M; EtFOSA: d-N-EtFOSA-M; MeFOSAA: d3-NMeFOSAA; EtFOSAA: d7-NEtFOSAA; MeFOSE: d7-N-MeFOSE-M; EtFOSE: d9-N-EtFOSA-M; 6:2 FTS: M2-6:2 FTS; 8:2 FTS: M2-8:2 FTS; 10:2 FTS: M2-8:2 FTS; GenX: 13C3 HFPO-DA; ADONA: 13C4 PFOS					
3	PFBA: PFBA-13C4; PFPeA: PFPeA-13C3; PFHxA: PFHxA-13C2; PFHpA: PFHpA-13C4; PFOA: PFOA-13C4; PFNA: PFNA-13C5; PFDA: PFDA-13C2; PFUnA: PFUnDA-13C2; PFDoA: PFDoDA-13C2; PFTrA: PFDoDA-13C2; PFTeDA: PFTeDA-13C2; PFBS: PFBS-13C3; PFPeS: PFHxS-18O2; PFHxS: PFHxS-18O2; PFHpS: PFOS-13C4; PFOS: PFOS-13C4; PFNS: PFOS-13C4; PFDS: 8:2 FTS-13C2; PFOSA: FOSA-13C8; MeFOSA: MeFOSA-D3; EtFOSA: EtFOSA-D5; MeFOSAA: MeFOSAA-D3; EtFOSAA: EtFOSAA-D5; MeFOSE: MeFOSE-D7; EtFOSE: EtFOSE-D9; 6:2 FTS: 6:2 FTS-13C2; 8:2 FTS: 8:2 FTS-13C2; 10:2 FTS: 8:2 FTS-13C2; GenX: HFPO-DA-13C3; ADONA: PFHxS-18O2	Wellington Laboratories	Yes	All: PFOA-13C8	No	No
5	PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C8-PFOA; PFNA: 13C9-PFNA; PFDA: 13C6-PFDA; PFUnA: 13C7-PFUnA; PFDoA: 13C2-PFDoA; PFBS: 13C3-PFBS; PFHxS: 13C3-PFHxS; PFOS: 13C8-PFOS; PFOSA: 13C8-PFOSA; 6:2 FTS: 13C2-6:2 FTS; 8:2 FTS: 13C2-8:2 FTS	Wellington	Yes		No	No
6	PFBA: Perfluoro-n-[13C4]butanoic acid MPFBA; PFPeA: Perfluoro-n-[13C5]pentanoic acid M5PFPeA; PFHxA: Perfluoro-n-[1,2,3,4,6-13C5]hexanoic acid M5PFHxA; PFHpA: Perfluoro-n-[1,2,3,4-13C4]heptanoic acid M4PFHpA; PFOA: Perfluoro-n-[13C8]octanoic acid M8PFOA; PFNA: Perfluoro-n-[13C9]nonanoic acid	Wellington	Yes		No	Yes

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
	M9PFNA; PFDA: Perfluoro-n-[1,2,3,4,6-13C6]decanoic acid M6PFDA; PFUnA: Perfluoro-n-[1,2,3,4,6,7-13C7]undecanoic acid M7PFUdA; PFDoA: Perfluoro-n-[1,2 13C2]dodecanoic acid MPFDoA; PFTeDA: Perfluoro-n-[1,2 13C2]tetradecanoic acid M2PFTeDA; PFBS: Sodium perfluoro-1-[2,3,4 13C3] butanesulfonate M3PFBS; PFHxS: Sodium perfluoro-1-[1,2,3 13C3] hexanesulfonate M3PFHxS; PFOS: Sodium perfluoro-1-[13C8] ocatanesulfonate M8PFOS; PFOSA: Perfluoro-1-[13C8]otanesulfonamide; MeFOSA: N-methyl-d3-perfluoro-1-octancesulfonamide; EtFOSA: N-ethyl-d5-perfluoro-1-octanesulfonamide; EtFOSAA: N-ethyl-d5-perfluoro-1-octanesulfonamide					
7	PFBA: 13C4 PFBA; PFPeA: 13C5 PFPeA; PFHxA: 13C12 PFHxA; PFHpA: 13C4 PFHpA; PFOA: 13C8 PFOA; PFNA: 13C5 PFNA; PFDA: 13C2 PFDA; PFUnA: 13C2 PFUnA; PFDoA: 13C2 PFDoA; PFTrA: 13C2 PFTeDA; PFTeDA: 13C2 PFTeDA; PFBS: 13C3 PFBS; PFPeS: 13C3 PFBS; PFHxS: 18O2 PFHxS; PFHpS: 18O2 PFHxS; PFOS: 13C4 PFOS; PFDS: 13C2 PFUnA; PFOSA: 13C8 PFOSA; MeFOSA: d3-MeFOSA; EtFOSA: d5-EtFOSA; MeFOSAA: d3-N-MeFOSAA; EtFOSAA: d5-N-EtFOSAA; MeFOSE: d7-MeFOSE; EtFOSE: d9-EtFOSE; 6:2 FTS: 13C2 6:2 FTS; 8:2 FTS: 13C 8:2 FTS; 10:2 FTS: 13C 8:2 FTS	Wellington		For all PFAS tested: 13C4 PFOA		No
8	PFOA: 13C8-PFOA; PFOS: 13C8-PFOS	Cambridge Isotope Laboratories	Yes	PFOA: yes; PFOS: yes	No	No
11	PFBA: PFBA-13C4; PFPeA: PFPeA-13C3; PFHxA: PFHxA-13C2; PFHpA: PFHpA-13C4; PFOA: PFOA-13C4; PFNA: PFNA-13C5; PFDA: PFDA-13C2; PFUnA: PFUnDA-13C2; PFDoA: PFDoDA-13C2; PFTrA: PFDoDA-13C2; PFTeDA: PFTeDA-13C2; PFBS: PFBS-13C3; PFPeS: PFHxS-18O2; PFHxS: PFHxS-18O2; PFHpS: PFOS-13C4; PFOS: PFOS-13C4; PFNS: PFOS-13C4; PFDS: 8:2 FTS-13C2; PFOSA: FOSA-13C8;	Wellington Laboratories	Yes	All: PFOA-13C8	No	No

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
	MeFOSA: MeFOSA-D3; EtFOSA: EtFOSA-D5; MeFOSAA: MeFOSAA-D3; EtFOSAA: EtFOSAA-D5; MeFOSE: MeFOSE-D7; EtFOSE: EtFOSE-D9; 6:2 FTS: 6:2 FTS-13C2; 8:2 FTS: 8:2 FTS-13C2; 10:2 FTS: 8:2 FTS-13C2; GenX: HFPO-DA-13C3; ADONA: PFHxS-18O2					
14	PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C2-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C8-PFOA; PFNA: 13C5-PFNA; PFDA: 13C6-PFDA; PFUnA: 13C2-PFUnA; PFDoA: 13C2-PFDoA; PFBS: 13C3-PFBS; PFHxS: 18O2-PFHxS; PFOS: 13C8-PFOS; PFOSA: 13C8-FOSA; MeFOSA: d3-N-MeFOSA; EtFOSA: d5-N-EtFOSA; MeFOSAA: d3-N-MeFOSAA; EtFOSAA: d5-N-EtFOSAA; MeFOSE: d7-N-MeFOSE; EtFOSE: d9-N-EtFOSE; 6:2 FTS: 13C2-6-2 FTS; 8:2 FTS: 13C2-8-2 FTS; GenX: 13C3-GenX	Wellington	Yes		No	No
15	PFBA: 13C4 PFBA; PFPeA: 13C5 PFPeA; PFHxA: 13C5 PFHxA; PFHpA: 13C4 PFHpA; PFOA: 13C8 PFOA; PFNA: 13C9 PFNA; PFDA: 13C6 PFDA; PFUnA: 13C7 PFUnA; PFDoA: 13C2 PFDoA; PFTrA: 13C2 PFDoA; PFTeDA: 13C2 PFTeDA; PFBS: 13C3 PFBS; PFPeS: 13C3 PFBS; PFHxS: 13C3 PFHxS; PFHpS: 13C3 PFHxS; PFOS: 13C6 PFOS; PFNS: 13C6 PFOS; PFDS: 13C6 PFOS; PFOSA: 13C8 PFOSA; MeFOSAA: d3-N-MeFOSAA; EtFOSAA: d5-N-EtFOSAA; 6:2 FTS: 13C2 6:2 FTS; 8:2 FTS: 13C2 8:2 FTS; 10:2 FTS: 13C2 8:2 FTS	Wellington	Yes	PFBA: 13C3 PFBA; PFOA: 13C2 PFOA; PFDA: 13C2 PFDA; PFOS: 13C4 PFOS		No
16	PFBA: M4PFBA; PFPeA: M5PFPeA; PFHxA: M5PFHxA; PFHpA: MPFHpA; PFOA: M8PFOA; PFNA: M9PFNA; PFDA: M6PFDA; PFUnA: M7PFUnDA; PFDoA: MPFDoDA; PFTrA: MPFDoDA; PFTeDA: MPFTeDA; PFBS: M3PFBS; PFPeS: M5PFHxA; PFHxS: M3PFHxS; PFHpS: M3PFHxS; PFOS: M8PFOS; PFNS: M8PFOS; PFDS: M8PFOS; PFOSA: MPFOSA; MeFOSA: d-NMeFOSA-M; EtFOSA: d-NEtFOSA-M; MeFOSAA: d3-NMeFOSAA; EtFOSAA: d5-	Wellington Laboratory	Yes		No	No

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
	NEtFOSAA; MeFOSE: d7-NMeFOSE-M; EtFOSE: d9-NEtFOSE-M; 6:2 FTS: M6:2 FTS; 8:2 FTS: M8:2 FTS					
17	PFBA: 13C3-PFBA; PFPeA: 13C3-PFPeA; PFHxA: 13C2-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C2-PFOA; PFNA: 13C5-PFNA; PFDA: 13C2-PFDA; PFUnA: 13C2-PFUdA; PFDoA: 13C2-PFDoA; PFTrA: 13C2-PFDoA; PFTeDA: 13C2-PFTeDA; PFBS: 13C3-PFBS; PFPeS: 13C3-PFBS; PFHxS: 13C3-PFHxS; PFHpS: 13C2-PFOA; PFOS: 13C8-PFOS; PFNS: 13C8-PFOS; PFDS: 13C8-PFOS; PFOSA: 13C8-PFOSA; MeFOSA: d3-N-MeFOSA; EtFOSA: d5-N-EtFOSA; MeFOSAA: d3-N-MeFOSAA; EtFOSAA: d5-N-EtFOSAA; MeFOSE: d7-N-MeFOSE; EtFOSE: d9-N-EtFOSE; 6:2 FTS: 13C2-6:2 FTS; 8:2 FTS: 13C2-8:2 FTS; 10:2 FTS: 13C2-PFDoA; GenX: 13C3-HFPO-DA; ADONA: 13C4-PFHpA	Wellington	Yes	PFBA: 13C8-PFBA; PFHxA: 13C5-PFHxA; PFOA: 13C8-PFOA; PFNA: 13C9-PFNA; PFDA: 13C6-PFDA; PFUnA: 13C7-PFUdA; PFHxS: 18O2-PFHxS; PFOS: 13C4-PFOS	No	No
18	PFOA: 13C8-PFOA; PFOS: 13C4-PFOS	Wellington Laboratories	No	PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C4-PFOA; PFNA: 13C5-PFNA; PFDA: 13C6-PFDA; PFUnA: 13C2-PFUnDA; PFDoA: 13C2-PFDoDA; PFTrA: 13C2-PFTeDA; PFTeDA: 13C2-PFTeDA; PFBS: 13C3-PFBS; PFPeS: 16O2-PFHxS; PFHxS: 16O2-PFHxS; PFHpS: 13C8-PFOS; PFOS: 13C8-PFOS; PFDS: 13C8-PFOS; PFOSA: 13C8-FOSA; MeFOSA: d3-MeFOSA; EtFOSA: d5-EtFOSA; MeFOSAA: d7-MeFOSE; EtFOSAA: d3EtFOSE; MeFOSE: d3-MeFOSAA; EtFOSE: d5-EtFOSAA; 6:2 FTS: 13C2-6:2 FTS; 8:2 FTS: 13C2-8:2 FTS; 10:2 FTS: 13C2-8:2 FTS	No	No
19	PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C4-PFOA; PFNA: 13C9-PFNA; PFDA: 13C2-PFDA; PFUnA: 13C2-PFUnA; PFDoA: 13C2-PFDoA; PFTrA: 13C2-PFHxDA; PFTeDA: 13C2-PFHxDA; PFBS: 18O2-	Wellington	Yes	PFBA: 13C8-PFOA; PFPeA: 13C8-PFOA; PFHxA: 13C8-PFOA; PFHpA: 13C8-PFOA; PFOA: 13C8-PFOA; PFNA: 13C5-PFNA; PFDA: 13C5-PFNA; PFUnA: 13C5-PFNA; PFDoA: 13C5-PFNA; PFTrA: 13C2-	No	Yes

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
	PFHxS; PFPeS: 18O2-PFHxS; PFHxS: 18O2-PFHxS; PFHpS: 18O2-PFHxS; PFOS: 13C4-PFOS; PFNS: 13C4-PFOS; PFDS: 13C4-PFOS; PFOSA: 13C8-PFOSA			PFTeDA; PFTeDA: 13C2-PFTeDA; PFBS: 18O2-PFOS; PFPeS: 18O2-PFOS; PFHxS: 18O2-PFOS; PFHpS: 18O2-PFOS; PFOS: 18O2-PFOS; PFNS: 18O2-PFOS; PFDS: 18O2-PFOS; PFOSA: 13C2-PFTeDA		
20	PFBA: 13C4-PFBA; PFPeA: 13C3 PFPeA; PFHxA: 13C2-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C4-PFOA; PFNA: 13C5-PFNA; PFDA: 13C2-PFDA; PFUnA: 13C2PFUdA; PFDoA: 13C2PFDoA; PFTrA: 13C2PFDoA; PFTeDA: 13C2PFTeDA; PFBS: 13C3 PFBS; PFPeS: 13C3 PFBS; PFHxS: 18O2-PFHxS; PFHpS: 18O2-PFHxS; PFOS: 13C4-PFOS; PFNS: 13C4-PFOS; PFDS: 13C4-PFOS; PFOSA: 13C8-FOSA; MeFOSA: D3-N-Me FOSA; EtFOSA: D5-N-Et FOSA; MeFOSAA: D3-N-Me FOSAA; EtFOSAA: D5-N-Et FOSAA; MeFOSE: D7-N-Me FOSE; EtFOSE: D9-N-Et FOSE; 6:2 FTS: 13C26:2 FTS; 8:2 FTS: 13C2-8:2 FTS; 10:2 FTS: 13C2-8:2 FTS	Wellington	Yes	PFBA: 13C3-PFBA; PFPeA: 13C5 -PFPeA; PFHxA: 13C5 -PFPeA; PFHpA: 13C8-PFOA; PFOA: 13C8-PFOA; PFNA: 13C8-PFOA; PFDA: 13C8-PFOA; PFUnA: 13C8-PFOA; PFDoA: 13C8-PFOA; PFBS: 18O3-PFHxS; PFPeS: 18O3-PFHxS; PFHxS: 18O3-PFHxS; PFHpS: 18O3-PFHxS; PFOS: 13C8-PFOS	No	No
21	PFBA: 13C4-PFBA; PFPeA: 13C5-PFPeA; PFHxA: 13C5-PFHxA; PFHpA: 13C4-PFHpA; PFOA: 13C8-PFOA; PFNA: 13C9-PFNA; PFDA: 13C6-PFDA; PFUnA: 13C7-PFUnA; PFDoA: 13C2-PFDoA; PFTrA: 13C2-PFDoA; PFTeDA: 13C2-PFTeDA; PFBS: 13C3-PFBS; PFPeS: 13C3-PFHxS; PFHxS: 13C3-PFHxS; PFHpS: 13C3-PFHxS; PFOS: 13C8-PFOS; PFNS: 13C8-PFOS; PFDS: 13C8-PFOS; PFOSA: 13C8-FOSA; 6:2 FTS: 13C2-6:2FTS; 8:2 FTS: 13C2-8:2FTS; 10:2 FTS: 13C2-8:2FTS; GenX: 13C3-GenX; ADONA: 13C8-PFOA	Wellington	No		No	Yes
22	All: C8-PFOS	Wellington	No	PFBA: 13C4-PFBA; PFPeA: 13-C4 PFOS; PFHxA: 13-C4 PFOS; PFHpA: 13-C4 PFOS; PFOA: 13-C4 PFOS; PFNA: 13-C4 PFOS; PFDA: 13-C4 PFOS; PFUnA: 13C2-PFDOA; PFDoA: 13C2-PFDOA; PFTrA: 13C2-PFDOA; PFTeDA: 13C2-PFDOA; PFBS: 13-C4 PFOS; PFPeS: 13-C4 PFOS; PFHxS: 13-	Yes MOECC E3506	No

Lab code	Labelled standard added before extraction	Labelled standard source	Recovery correction	Labelled standard added before instrument analysis	Standard Method used	Blank corrected
				C4 PFOS; PFHpS: 13-C4 PFOS; PFOS: 13-C4 PFOS; PFNS: 13C2-PFDOA; PFDS: 13-C4 PFOS; PFOSA: 13-C4 PFOS; MeFOSA: d5-EtFOSA; EtFOSA: d5-EtFOSA; MeFOSAA: d5-EtFOSA; EtFOSAA: d5-EtFOSA; MeFOSE: d5-EtFOSA; EtFOSE: d5-EtFOSA; 6:2 FTS: 13C2 4:2-FTS; 8:2 FTS: 13C2 4:2-FTS; 10:2 FTS: 13C2 4:2-FTS; GenX: M3-HFPO-DA; ADONA: 13C2-PFDOA		
24	PFBA: Perfluoro-[13C4] butanoic acid; PFHxA: Perfluoro-[1,2-13C2] hexanoic acid; PFOA: Perfluoro-[1,2,3,4-13C4] octanoic acid; PFNA: Perfluoro-[1,2,3,4,5-13C5] nonanoic acid; PFDA: Perfluoro-[1,2-13C2] decanoic acid; PFUnA: Perfluoro-[1,2-13C2] undecanoic acid; PFDoA: Perfluoro-[1,2-13C2] dodecanoic acid; PFHxS: Sodium-perfluoro-1-hexane-[18O2] sulfonate; PFOS: Sodium-perfluoro-1-[1,2,3,4-13C4] octanesulfonate	Wellington	Yes		Yes DIN 38414 S14	Yes

3.2 Basis of Participants' Measurement Uncertainty Estimates

Table 6 Basis of Participants' Uncertainty Estimate

Lab Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
1	Top Down - precision and estimates of the method and laboratory bias	Control samples – SS	Recoveries of SS	NATA - Estimating and reporting MU of chemical test results
2	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples – SS	Recoveries of SS	USEPA SW-846
3	Standard deviation of replicate analyses multiplied by 2 or 3	Standard deviation from PT studies		Nata Technical Note 33
		Control samples – SS	CRM Recoveries of SS Standard purity	
4	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples – SS	Instrument calibration Recoveries of SS	NATA - Estimating and reporting MU of chemical test results
5	Top Down - reproducibility (standard deviation) from PT studies used directly	Control samples – RM	Laboratory bias from PT studies	Nordtest Report TR537
6	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples – SS Duplicate analysis Instrument calibration	Laboratory bias from PT studies Recoveries of SS	NATA GAG Estimating and reporting measurement uncertainty of chemical test results January 2018
7	Bottom Up (ISO/GUM, fish bone/ cause and effect diagram)	Control samples – RM	Laboratory bias from PT studies	ISO/GUM
8	Standard deviation of replicate analyses multiplied by 2 or 3	Duplicate analysis Instrument calibration	Recoveries of SS	ÁM-162:2019
9	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples – SS		ISO/GUM
10				
11	Standard deviation of replicate analyses multiplied by 2 or 3	Standard deviation from PT studies		Nata Technical Note 33
		Control samples – SS	CRM Recoveries of SS Standard purity	
14	Top Down - precision and estimates of the method and laboratory bias	Control samples – RM	Instrument calibration Recoveries of SS Standard purity	ISO/GUM
15	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples – CRM	CRM Laboratory bias from PT studies Recoveries of SS	NMI Uncertainty Course

Lab Code	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
		Precision	Method Bias	
16	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples – SS	Recoveries of SS	Statistics and Chemometrics for Analytical Chemistry, Miller and Miller, 5th Edition.
17	Professional judgment	Instrument calibration	CRM Instrument calibration Recoveries of SS Standard purity	
18	Top Down - precision and estimates of the method and laboratory bias	Control samples – SS Duplicate analysis	Recoveries of SS	Eurachem/CITAC Guide
19	Top Down - precision and estimates of the method and laboratory bias	Control samples Duplicate analysis	CRM Laboratory bias from PT studies Recoveries of SS	Nordtest Report TR537
20	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples – SS Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS	Eurachem/CITAC Guide
21	Bottom Up (ISO/GUM, fish bone/ cause and effect diagram)	Duplicate analysis Instrument calibration	Instrument calibration	ISO/GUM
22	Top Down - precision and estimates of the method and laboratory bias	Duplicate analysis	Recoveries of SS	Eurachem/CITAC Guide
23	Top Down - precision and estimates of the method and laboratory bias	Control samples – SS Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS Standard purity	NATA - Estimating and reporting MU of chemical test results
24	Top Down - reproducibility (standard deviation) from PT studies used directly	Standard deviation from PT studies		ISO/GUM
		Control samples – RM Duplicate analysis	Recoveries of SS	

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

3.3 Participants' Comments

The study co-ordinator welcomes comments or suggestions from participants about this study or possible future studies. Participants' comments are reproduced in Table 7.

Table 7 Participants' Comments

Lab Code	Sample	Participants' Comments	Study manager response
1	All	Note, all branched and linear present have been reported even though some branched peaks aren't confirmed by a traceable standard.	
2	S1	PFUnA transition mass ratio was outside established limits, indicating some interference with the qualitative identification. PFOS reported from a simple (no refortification of extracted internal standard) dilution due to the magnitude of the detection.	
	S2	6:2 and 8:2 FTS reported from a simple (no refortification of extracted internal standard) dilution due to the magnitude of the detection.	
	All	Uncertainty: Standard practice for laboratories utilizing US EPA's SW-846 document.	
3	All	Quantitative result of PFDA, PFOA, PFOS, PFHxS, EtFOSAA and MeFOSAA are reported as sum of linear and branched isomers. All other PFAS are reported as linear only.	
5	S1	PFBA: Interfering compound separted with HRMS; MS/MS Result 2.0 µg/kg	
6	S1	The method is not validated for the following compounds but it was detected in the fish sample: PFHpS 1.0 ug/kg, PFNS 1.7 ug/kg, PFDoS 4.7 ug/kg, PFOSA 4.3 ug/kg, N-EtFOSAA 13 ug/kg. PFDoA was detected at 3.6 ug/kg (below LOR)	
	All	Uncertainty: Recovery and uncertainty data given for analytes at method limit of reporting.	
9	S1	Sample was received at ambient temperature and above method recommended maximum sample storage temperature (< or equal to 4ºC). Uncertainty: The expanded measurement uncertainty value estimates were calculated using a coverage factor (K) of 2.58.	A stability study has been conducted for all analytes and matrices. Samples have been confirmed stable at room temperature for at least three months.
10	S1	The results supplied are the average of two determinations. Calibration curves for PFTrA is based on 6 points & PFTeDA are based on 2 points as these analytes were only added recently & it was discovered that the lower points were not sufficient for quantitation. Larger sample size to be sent in the future. Uncertainty: Measurement of uncertainty has not yet been determined.	NMI has a limited amount of total sample to be distributed. Most participants used 2 g or less of Sample S1 for each analysis; participants were provided with 5 g of sample.
11	All	PFDA, PFOA, PFOS, PFHxS, EtFOSAA and MeFOSAA are reported as sum of linear and branched isomers. All other PFASs are reported as linear structure only.	
16	All	Uncertainty: Measurement Uncertainty (U) estimated from the standard deviation (u) of replicate recovery samples using the expression U = 2 x u. Procedure as set out in Statistics and	

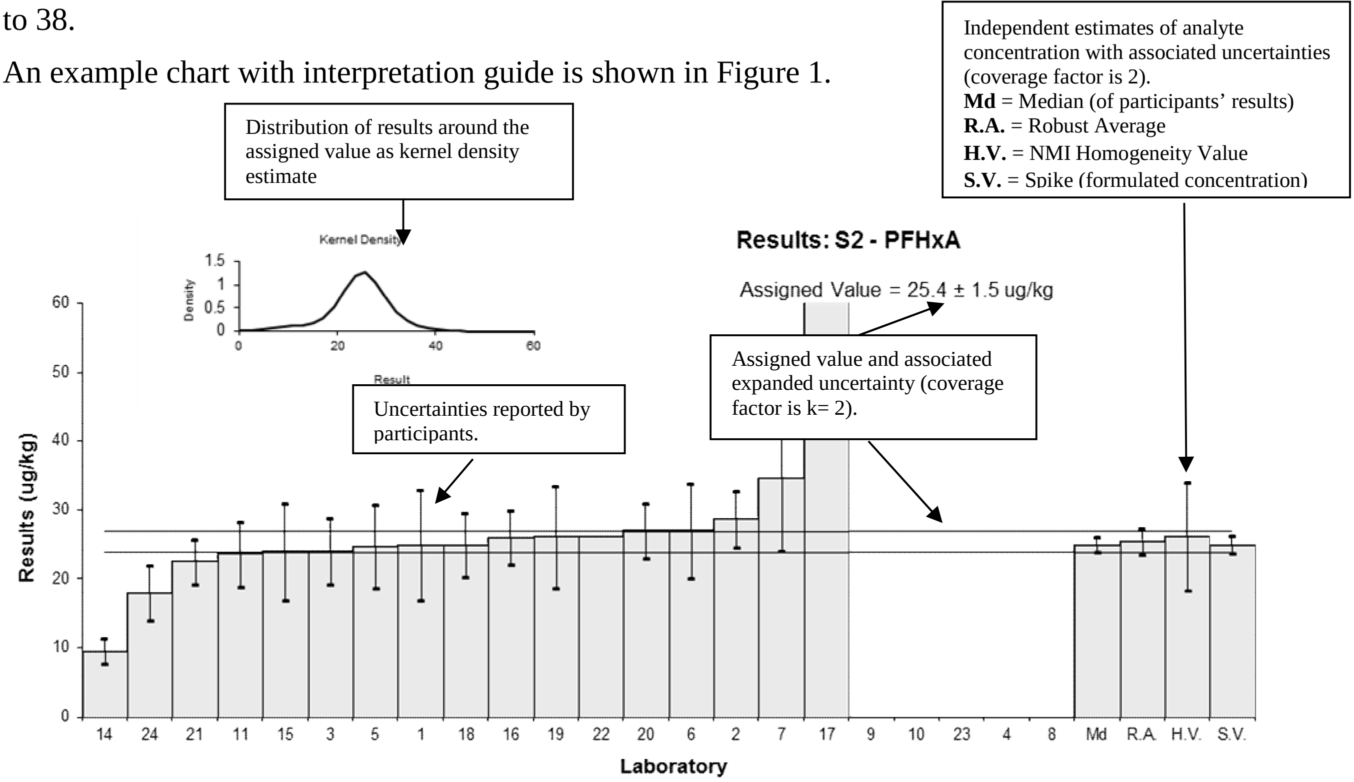
Lab Code	Sample	Participants’ Comments	Study manager response
		Chemometrics for Analytical Chemistry, Miller and Miller, 5th Edition.	
17	All	Better communication during the study, including deadline reminders and check-in.	Deadline reminders are sent with every PT study.
23	S1	PFHxA reporting limit increased due to matrix interference	
24	S2	Extraction was performed with dried samples. Desiccation may have an influence on the result.	

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 8 to 44 with the summary statistics robust average, mean, median, maximum, minimum, robust standard deviation (SD_{rob}) and robust coefficient of variation (CV_{rob}). Bar charts of results and performance scores are presented in Figures 2 to 38.

An example chart with interpretation guide is shown in Figure 1.



Independent estimates of analyte concentration with associated uncertainties (coverage factor is 2).
Md = Median (of participants' results)
R.A. = Robust Average
H.V. = NMI Homogeneity Value
S.V. = Spike (formulated concentration)

Distribution of results around the assigned value as kernel density estimate

Assigned value and associated expanded uncertainty (coverage factor is k= 2).

Uncertainties reported by participants.

Figure 1 Guide to Presentation of Results

4.2 Assigned Value

The assigned value is defined as: 'value attributed to a particular property of a proficiency test item.'[1] In this study property is the mass fraction of analyte. Assigned values were the robust average of participants' results; the expanded uncertainties were estimated from the associated robust standard deviations.

4.3 Performance Coefficient of Variation (PCV)

The performance coefficient of variation (PCV) is a measure of the between laboratory variation that in the judgement of the study coordinator would be expected from participants given the analyte concentration. It is important to note that this is a performance measure set by the study coordinator; it is not the coefficient of variation of participant results.

4.4 Target Standard Deviation

The target standard deviation (σ) is the product of the assigned value (*X*) and the performance coefficient of variation (PCV). This value is used for calculation of each participant z-score.

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

4.5 z-Score

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

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

where:

z is z-score
χ is a participant's result
X is the assigned value
σ is the target standard deviation from equation 1

A z-score with absolute value (|z|):

- $|z| \leq 2$ is satisfactory;
- $2 < |z| < 3$ is questionable;
- $|z| \geq 3$ is unsatisfactory.

4.6 E_n-Score

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

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

where:

E_n is E_n-score
χ is a participant's result
X is the assigned value
U_χ is the expanded uncertainty of the participant's result
U_X is the expanded uncertainty of the assigned value

An E_n-score with absolute value ($|E_n|$):

- $|E_n| \leq 1$ is satisfactory;
- $|E_n| > 1$ is unsatisfactory.

4.7 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025:2017[5] must establish and demonstrate the traceability and measurement uncertainty associated with their test results.

Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.[6]

4.8 Robust Average

The robust averages and associated expanded measurement uncertainties were calculated using the procedure described in 'ISO13528:2015(E), Statistical methods for use in proficiency testing by interlaboratory comparisons'.[7]

5 TABLES AND FIGURES

Table 8

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	10:2 FTS
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 1	NR	181
2	0.368	0.11	374
3	<0.5	NR	81
4	NT	NT	NT
5	NT	NT	NT
6	NT	NT	NT
7	< 0.500	0.15	21.8
8	NT	NT	NT
9	NT	NT	NT
10	NT	NT	NT
11	0.37	0.073	81
14	<0.5	0.1	NR
15	<0.5	NR	NR
16	NT	NT	NT
17	NR	NR	NR
18	<2	NR	NR
19	NT	NT	NT
20	0.3	0.01	157
21	0.282	0.083	NR
22	0.943	NR	93.2
23	NT	NT	NT
24	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Robust Average	0.38	0.16
Median	0.37	0.13
Mean	0.45	
N	5	
Max.	0.943	
Min.	0.282	
Robust SD	0.139	
Robust CV	37%	

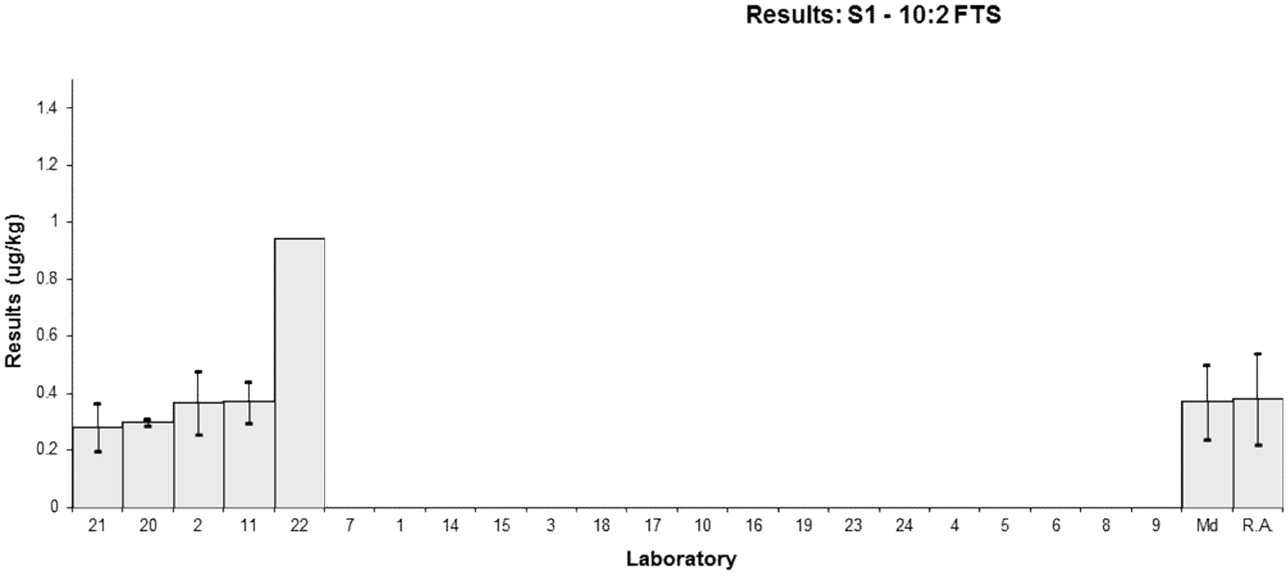

Results: S1 - 10:2 FTS
Results (ug/kg)
1.4
1.2
1
0.8
0.6
0.4
0.2
0
21
20
2
11
22
7
1
14
15
3
18
17
10
16
19
23
24
4
5
6
8
9
Md
R.A.
Laboratory

Figure 2

Table 9

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	6:2 FTS
Units	ug/kg

Participant Results

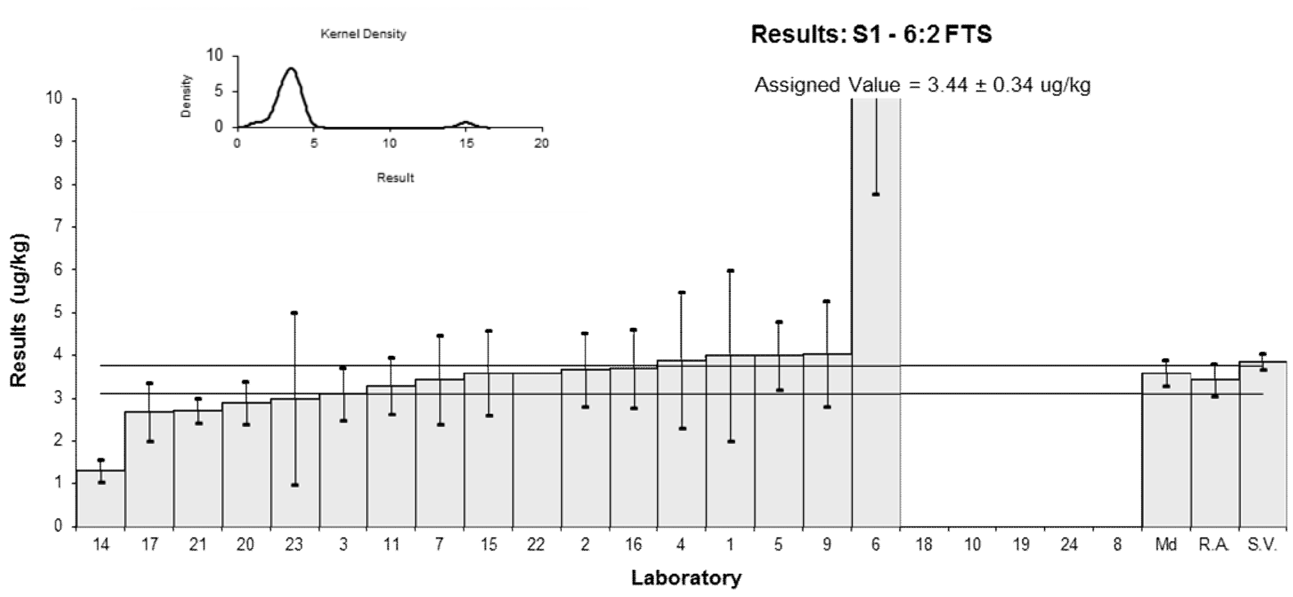
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	4	2	145	0.81	0.28
2	3.67	0.85	256	0.33	0.25
3	3.11	0.622	88	-0.48	-0.47
4	3.9	1.6	NR	0.67	0.28
5	4.0	0.8	NR	0.81	0.64
6	15	7.2	NR	16.80	1.60
7	3.43	1.03	110	-0.01	-0.01
8	NT	NT	NT		
9	4.05	1.22	208	0.89	0.48
10	NT	NT	NT		
11	3.30	0.661	88	-0.20	-0.19
14*	1.3	0.26	62	-3.11	-5.00
15	3.6	1	102	0.23	0.15
16	3.7	0.91	181	0.38	0.27
17	2.68	0.67	122	-1.10	-1.01
18	<2	NR	NR		
19	NT	NT	NT		
20	2.9	0.5	130	-0.78	-0.89
21	2.710	0.288	NR	-1.06	-1.64
22	3.6	NR	93.2	0.23	0.47
23	3	2	379	-0.64	-0.22
24	NT	NT	NT		

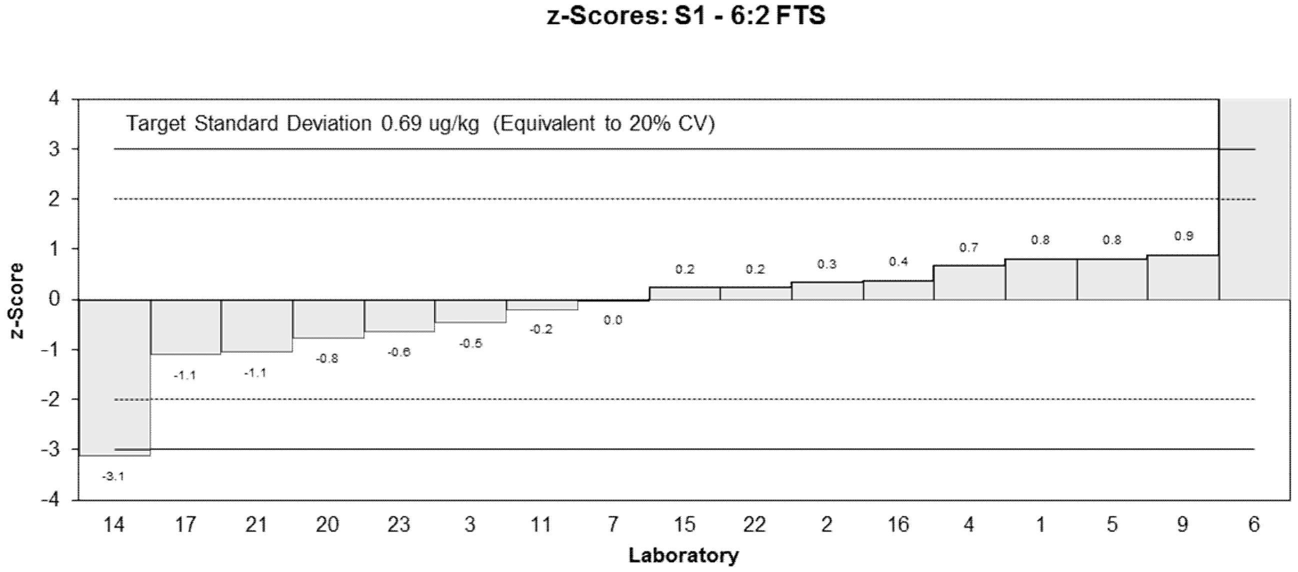
Statistics

Assigned Value**	3.44	0.34
Spike	3.87	0.19
Robust Average	3.44	0.38
Median	3.60	0.30
Mean	4.00	
N	17	
Max.	15	
Min.	1.3	
Robust SD	0.53	
Robust CV	15%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratories 6 and 14.


Results: S1 - 6:2 FTS
Assigned Value = 3.44 ± 0.34 ug/kg
Kernel Density
Density
Result
Results (ug/kg)
Laboratory
14
17
21
20
23
3
11
7
15
22
2
16
4
1
5
9
6
18
10
19
24
8
Md
R.A.
S.V.


z-Scores: S1 - 6:2 FTS
Target Standard Deviation 0.69 ug/kg (Equivalent to 20% CV)
z-Score
Laboratory
-3.1
-1.1
-1.1
-0.8
-0.6
-0.5
-0.2
0.0
0.2
0.2
0.3
0.4
0.7
0.8
0.8
0.9
14
17
21
20
23
3
11
7
15
22
2
16
4
1
5
9
6

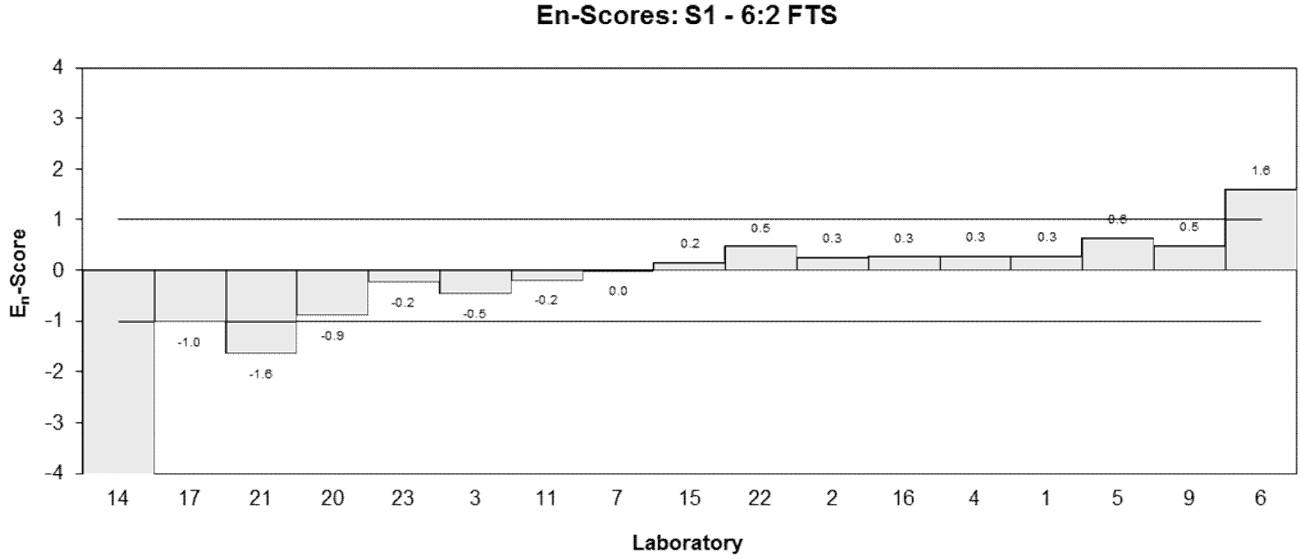

En-Scores: S1 - 6:2 FTS
E_n-Score
Laboratory
-1.0
-1.6
-0.9
-0.2
-0.5
-0.2
0.0
0.2
0.5
0.3
0.3
0.3
0.3
0.6
0.5
1.6
14
17
21
20
23
3
11
7
15
22
2
16
4
1
5
9
6

Figure 3

Table 10

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	8:2 FTS
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery
1	< 1	NR	181
2	< 10.0	NR	374
3	0.44	0.0880	81
4	< 1	NR	NR
5	0.45	0.11	NR
6	< 2.0	0.84	NR
7	< 0.300	0.09	21.8
8	NT	NT	NT
9	1.57	0.47	227
10	NT	NT	NT
11	0.493	0.0981	81
14	<0.5	0.1	62
15	0.66	0.2	92
16	0.44	0.0096	260
17	NR	NR	68.7
18	<2	NR	NR
19	NT	NT	NT
20	0.3	0.04	157
21	0.537	0.057	NR
22	1.36	NR	93.2
23	<3	NR	204
24	NT	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Robust Average	0.64	0.33
Median	0.49	0.06
Mean	0.69	
N	9	
Max.	1.57	
Min.	0.3	
Robust SD	0.398	
Robust CV	62%	

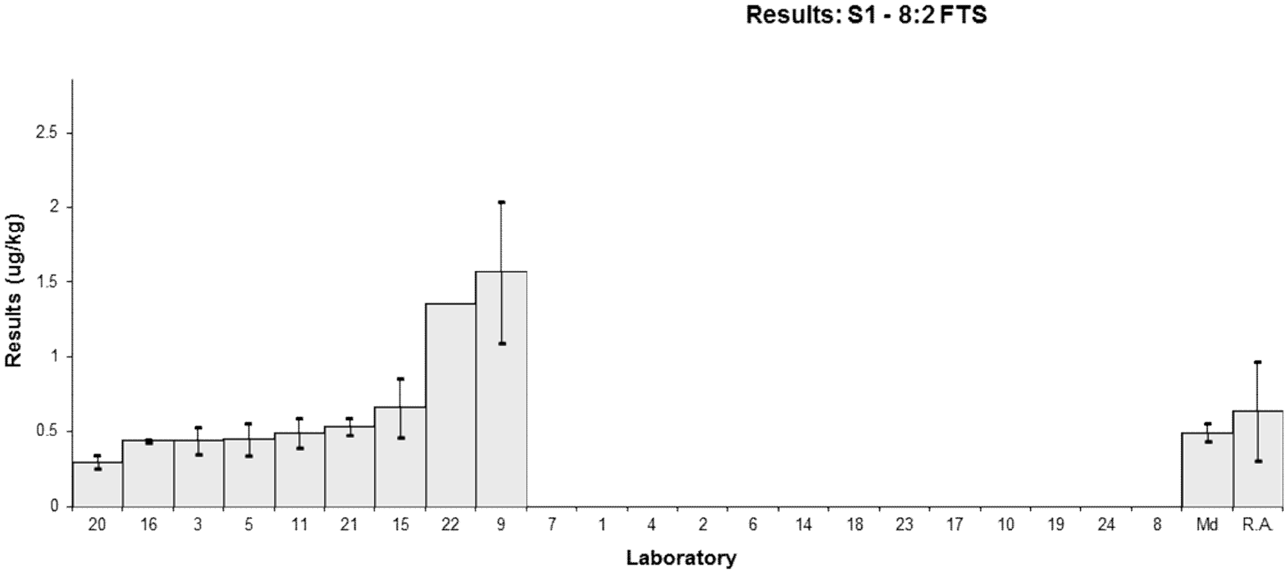

Results: S1 - 8:2 FTS
Results (ug/kg)
2.5
2
1.5
1
0.5
0
20
16
3
5
11
21
15
22
9
7
1
4
2
6
14
18
23
17
10
19
24
8
Md
R.A.
Laboratory

Figure 4

Table 11

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	ADONA
Units	ug/kg

Participant Results

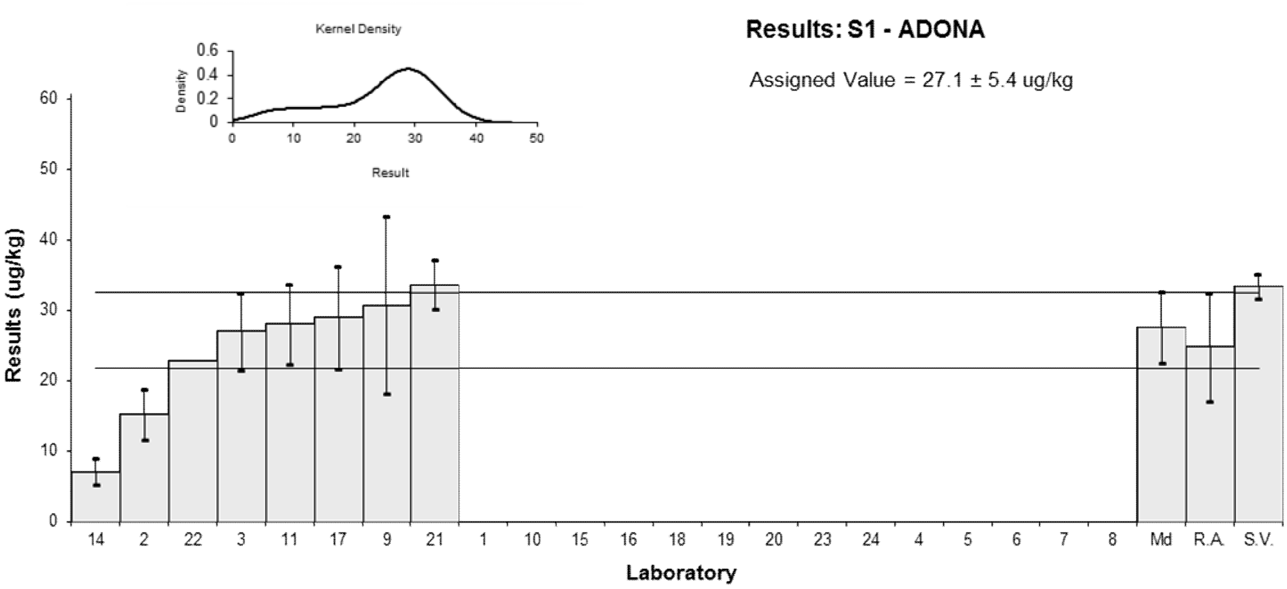
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	NT	NT	NT		
2	15.2	3.51	114	-2.20	-1.85
3	27.0	5.40	71	-0.02	-0.01
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	NT	NT	NT		
8	NT	NT	NT		
9	30.8	12.6	125	0.68	0.27
10	NT	NT	NT		
11	28.1	5.62	71	0.18	0.13
14*	7.1	1.8	NR	-3.69	-3.51
15	NT	NT	NT		
16	NT	NT	NT		
17	29	7.25	NT	0.35	0.21
18	NT	NT	NT		
19	NT	NT	NT		
20	NT	NT	NT		
21	33.642	3.470	NR	1.21	1.02
22	22.8	NR	93.2	-0.79	-0.80
23	NT	NT	NT		
24	NT	NT	NT		

Statistics

Assigned Value**	27.1	5.4
Spike	33.4	1.7
Robust Average	24.8	7.7
Median	27.6	5.0
Mean	24.2	
N	8	
Max.	33.642	
Min.	7.1	
Robust SD	5.7	
Robust CV	21%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

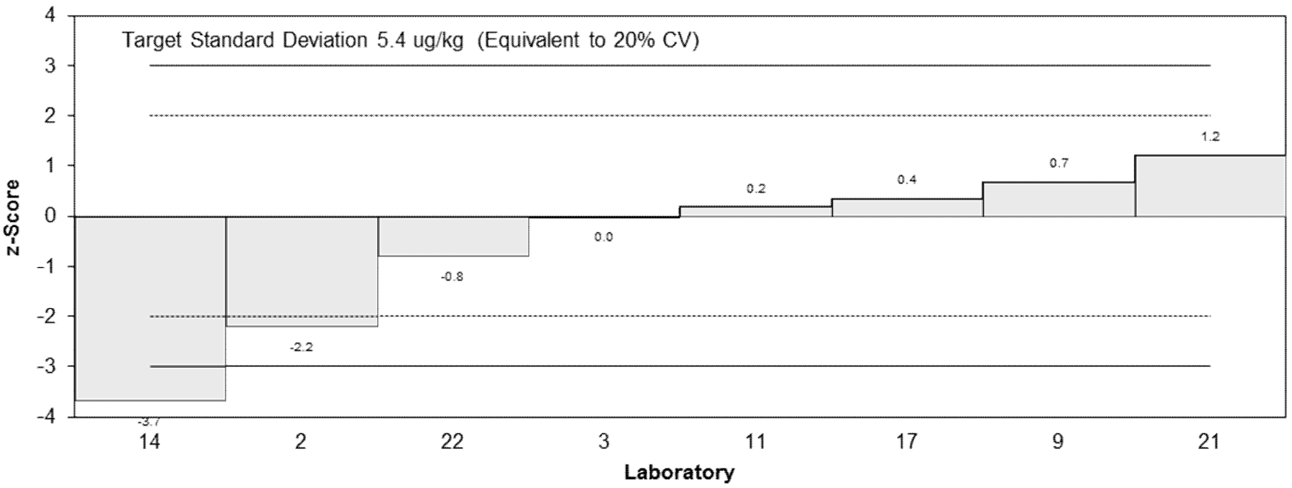
**Robust average excluding laboratory 14.



Results: S1 - ADONA

Assigned Value = 27.1 ± 5.4 ug/kg

z-Scores: S1 - ADONA



En-Scores: S1 - ADONA

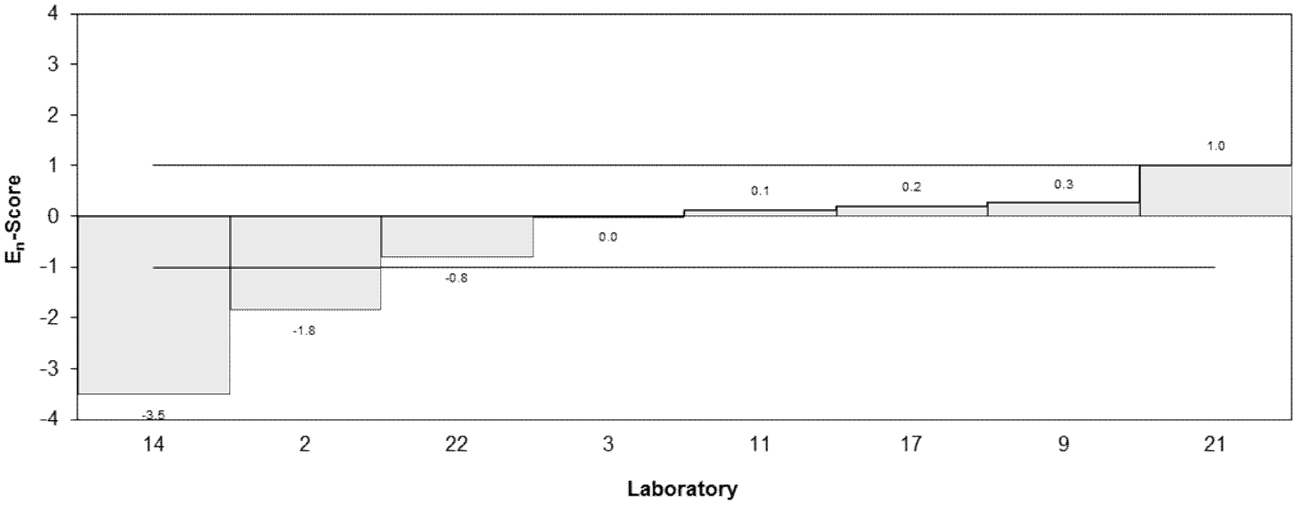


Figure 5

Table 12

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	EtFOSA
Units	ug/kg

Participant Results

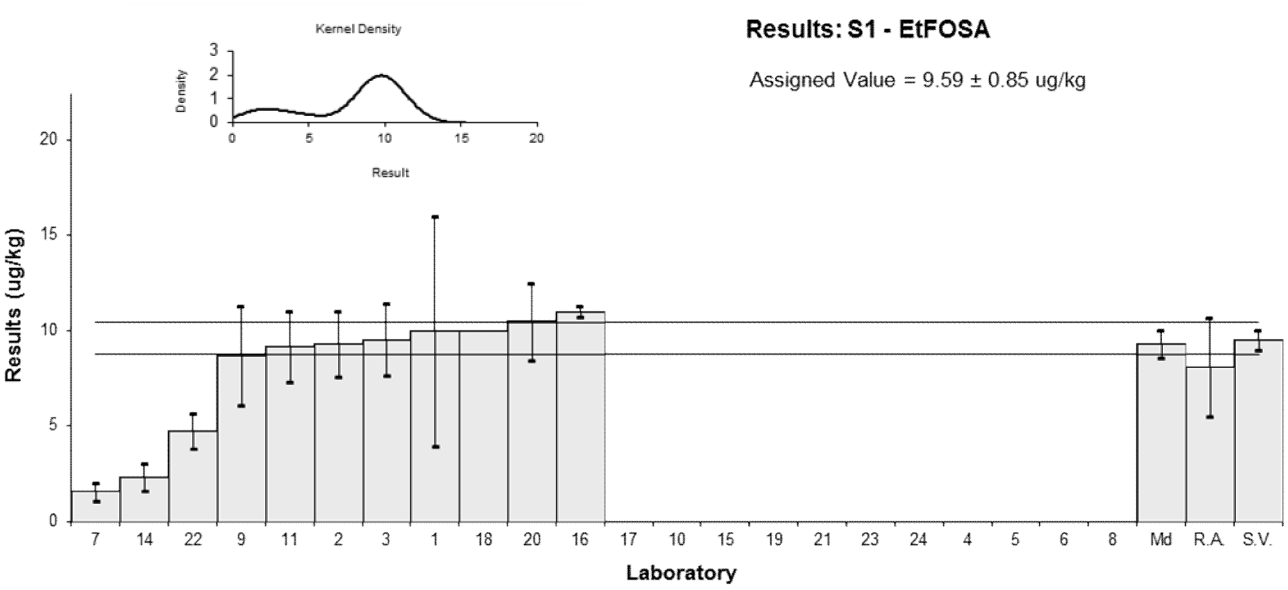
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	10	6	81	0.21	0.07
2	9.31	1.7	28	-0.15	-0.15
3	9.54	1.91	80	-0.03	-0.02
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	1.56	0.468	7.7	-4.19	-8.28
8	NT	NT	NT		
9	8.71	2.61	53.8	-0.46	-0.32
10	NT	NT	NT		
11	9.19	1.840	80	-0.21	-0.20
14*	2.3	0.70	77	-3.80	-6.62
15	NT	NT	NT		
16	11	0.28	69	0.74	1.58
17	NR	NR	11.4		
18	10	NR	NR	0.21	0.48
19	NT	NT	NT		
20	10.5	2.0	9	0.47	0.42
21	NT	NT	NT		
22	4.73	0.89	93.2	-2.53	-3.95
23	NT	NT	NT		
24	NT	NT	NT		

Statistics

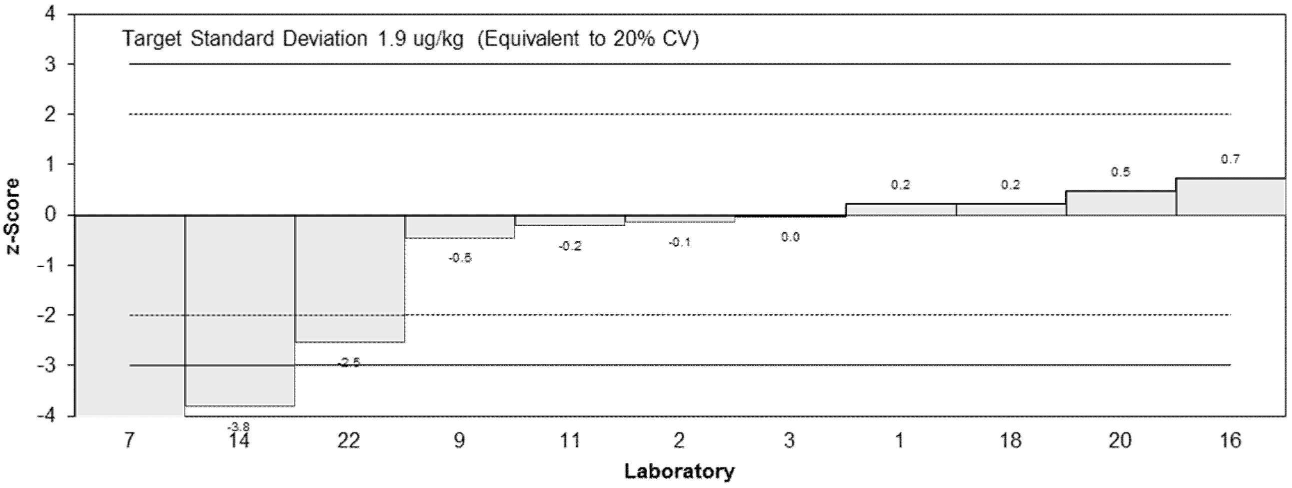
Assigned Value**	9.59	0.85
Spike	9.51	0.48
Robust Average	8.1	2.6
Median	9.31	0.69
Mean	7.90	
N	11	
Max.	11	
Min.	1.56	
Robust SD	1.02	
Robust CV	11%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratories 7 and 14.



z-Scores: S1 - EtFOSA



En-Scores: S1 - EtFOSA

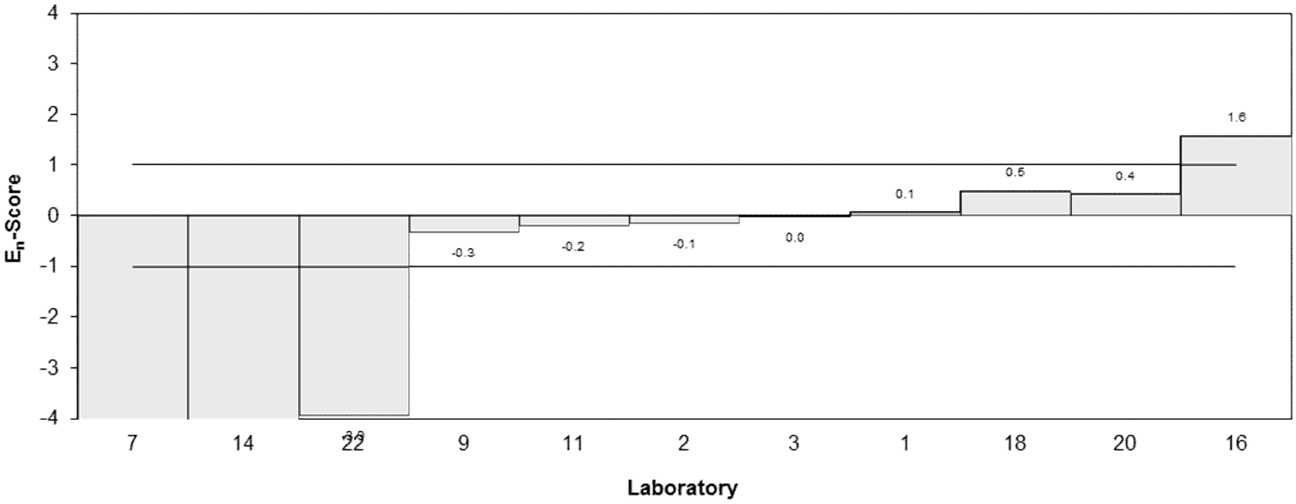


Figure 6

Table 13

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	EtFOSAA
Units	ug/kg

Participant Results

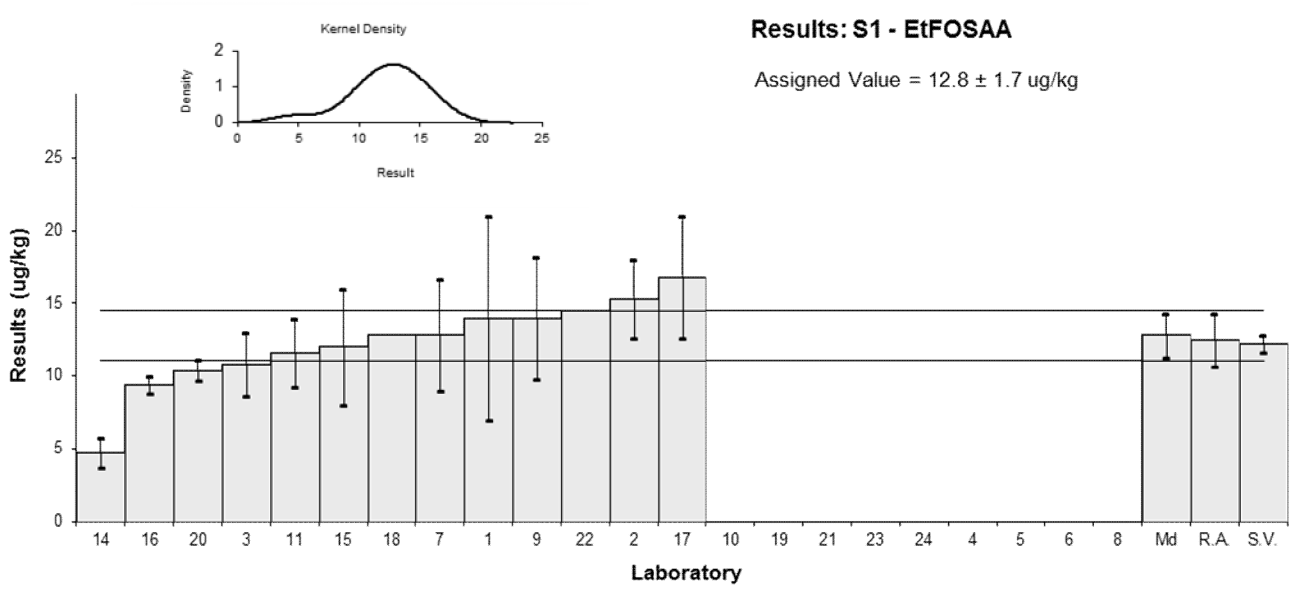
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	14	7	77	0.47	0.17
2	15.3	2.7	99	0.98	0.78
3	10.8	2.16	88	-0.78	-0.73
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	12.8	3.84	113	0.00	0.00
8	NT	NT	NT		
9	14.0	4.2	165	0.47	0.26
10	NT	NT	NT		
11	11.6	2.33	88	-0.47	-0.42
14*	4.7	1	76	-3.16	-4.11
15	12	4	58	-0.31	-0.18
16	9.4	0.59	105	-1.33	-1.89
17	16.8	4.2	24.5	1.56	0.88
18	12.8	NR	NR	0.00	0.00
19	NT	NT	NT		
20	10.4	0.7	29	-0.94	-1.31
21	NT	NT	NT		
22	14.5	NR	93.2	0.66	1.00
23	NT	NT	NT		
24	NT	NT	NT		

Statistics

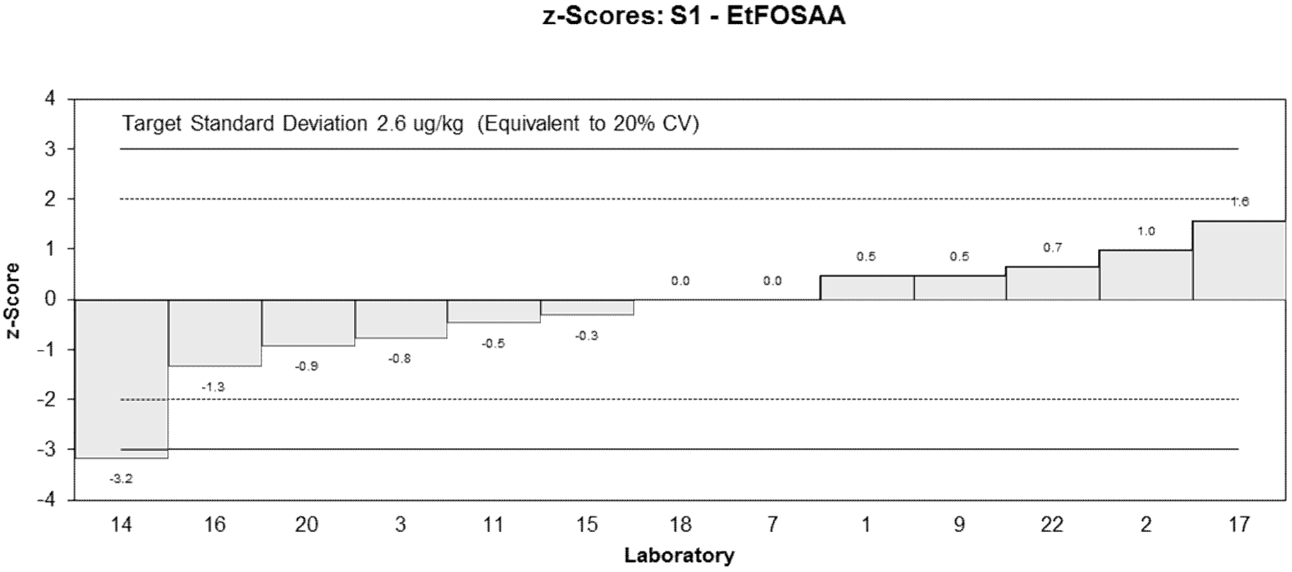
Assigned Value**	12.8	1.7
Spike	12.2	0.6
Robust Average	12.5	1.8
Median	12.8	1.5
Mean	12.2	
N	13	
Max.	16.8	
Min.	4.7	
Robust SD	2.4	
Robust CV	19%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratory 14.


Results: S1 - EtFOSAA

Assigned Value = 12.8 ± 1.7 ug/kg


z-Scores: S1 - EtFOSAA

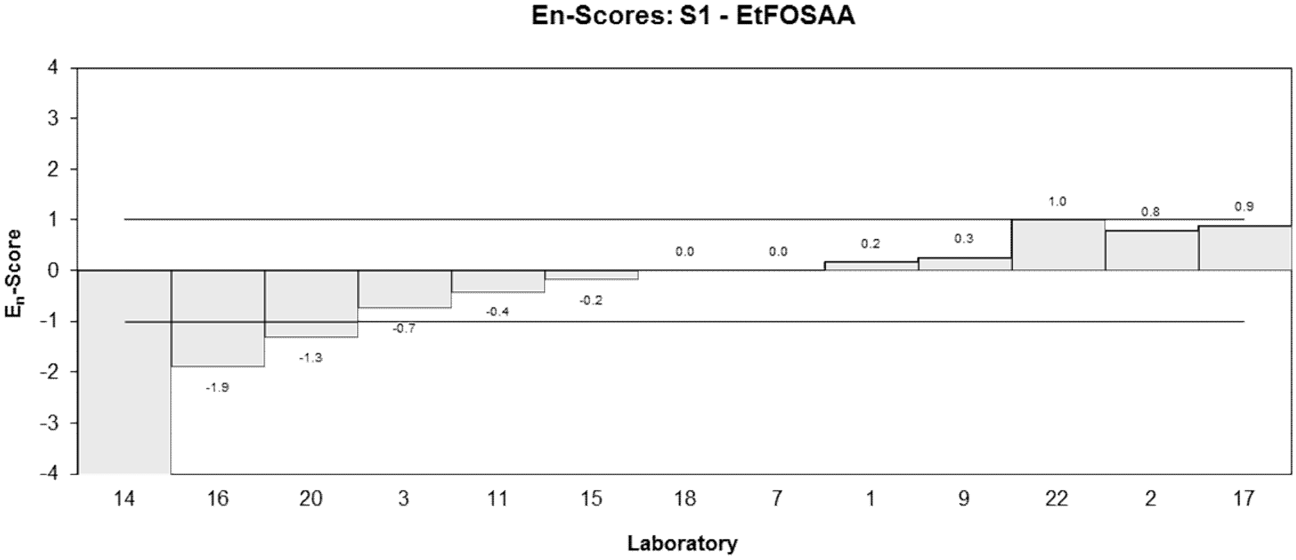

En-Scores: S1 - EtFOSAA

Figure 7

Table 14

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	GenX
Units	ug/kg

Participant Results

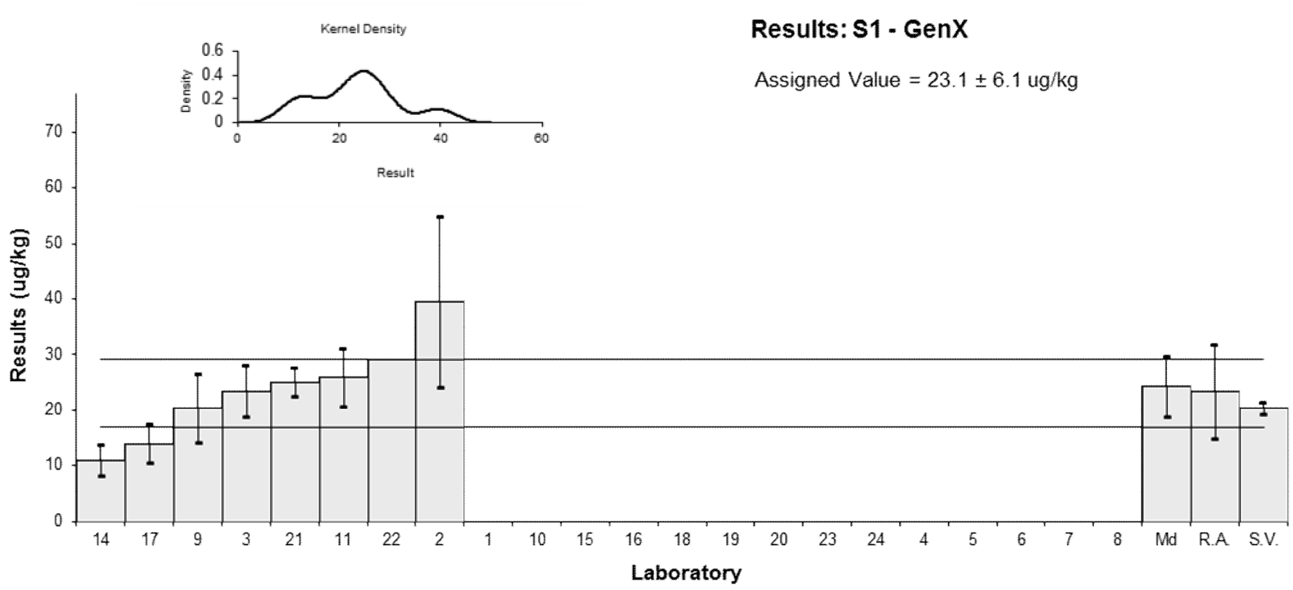
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	NT	NT	NT		
2	39.6	15.3	25	3.57	1.00
3	23.5	4.69	86	0.09	0.05
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	NT	NT	NT		
8	NT	NT	NT		
9	20.4	6.1	125	-0.58	-0.31
10	NT	NT	NT		
11	26.0	5.20	86	0.63	0.36
14*	11	2.8	60	-2.62	-1.80
15	NT	NT	NT		
16	NT	NT	NT		
17	14	3.5	92.2	-1.97	-1.29
18	NT	NT	NT		
19	NT	NT	NT		
20	NT	NT	NT		
21	25.075	2.542	NR	0.43	0.30
22	29.2	NR	93.2	1.32	1.00
23	NT	NT	NT		
24	NT	NT	NT		

Statistics

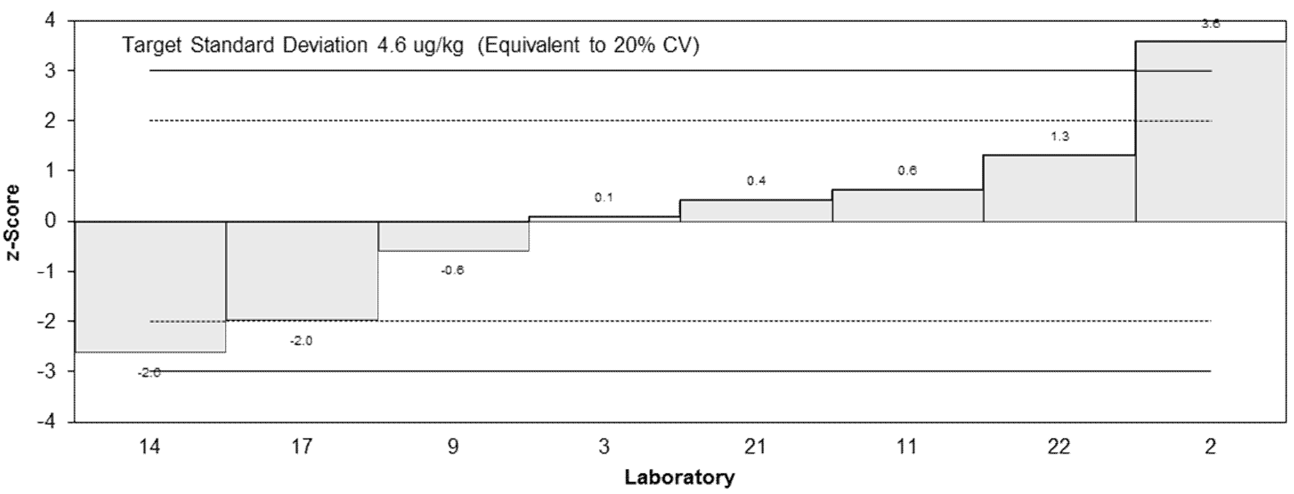
Assigned Value**	23.1	6.1
Spike	20.4	1.0
Robust Average	23.4	8.5
Median	24.3	5.5
Mean	23.6	
N	8	
Max.	39.6	
Min.	11	
Robust SD	5.9	
Robust CV	26%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratories 2 and 14.


Results: S1 - GenX

Assigned Value = 23.1 ± 6.1 ug/kg


z-Scores: S1 - GenX

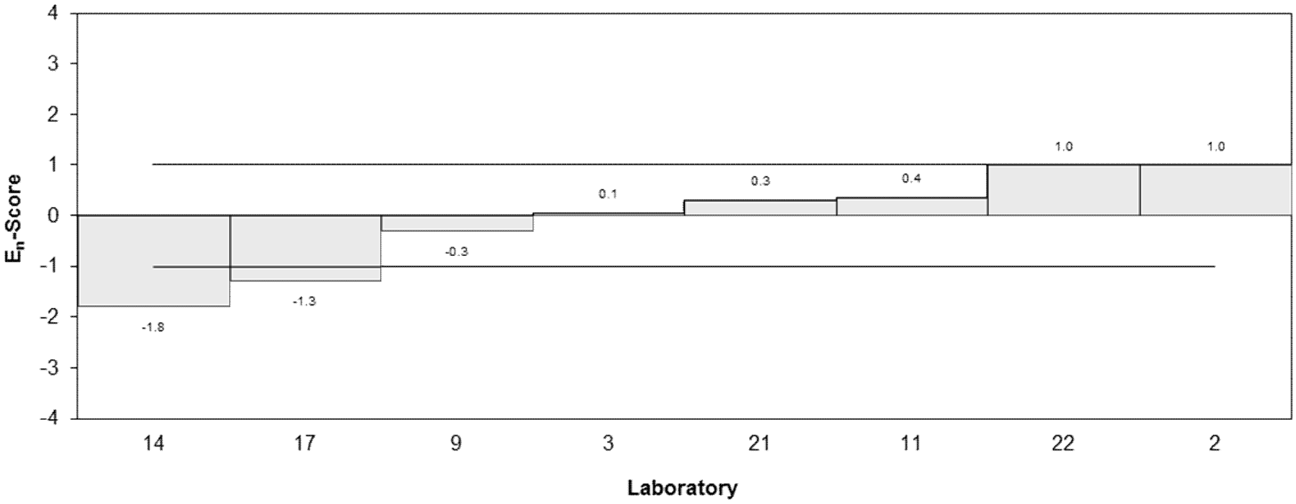

En-Scores: S1 - GenX

Figure 8

Table 15

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	MeFOSE
Units	ug/kg

Participant Results

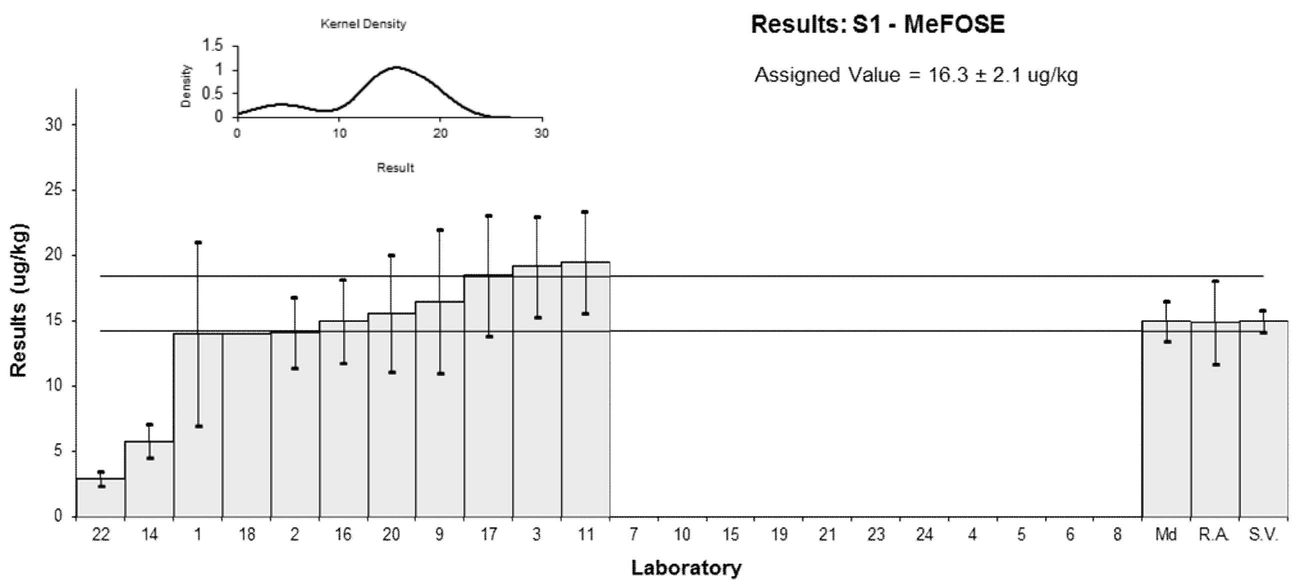
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	14	7	119	-0.71	-0.31
2	14.1	2.7	15	-0.67	-0.64
3	19.2	3.85	105	0.89	0.66
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	< 1.00	0.3	1.24		
8	NT	NT	NT		
9	16.5	5.5	50.8	0.06	0.03
10	NT	NT	NT		
11	19.5	3.91	105	0.98	0.72
14*	5.8	1.3	66	-3.22	-4.25
15	NT	NT	NT		
16	15	3.2	63	-0.40	-0.34
17	18.5	4.625	24.2	0.67	0.43
18	14	NR	NR	-0.71	-1.10
19	NT	NT	NT		
20	15.6	4.5	42	-0.21	-0.14
21	NT	NT	NT		
22	2.94	.54	93.2	-4.10	-6.16
23	NT	NT	NT		
24	NT	NT	NT		

Statistics

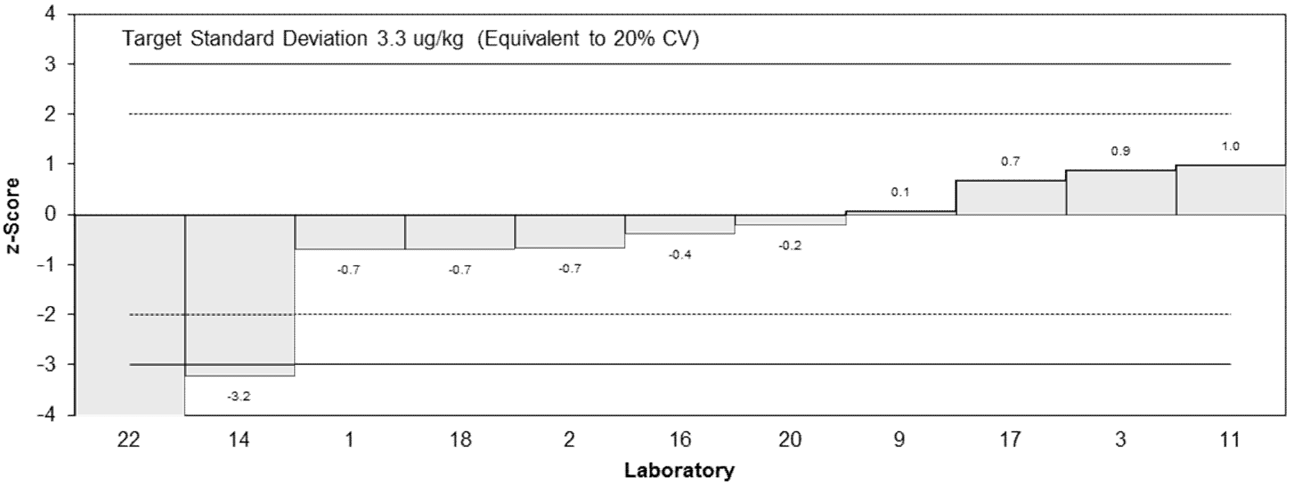
Assigned Value**	16.3	2.1
Spike	15.0	0.8
Robust Average	14.9	3.2
Median	15.0	1.5
Mean	14.1	
N	11	
Max.	19.5	
Min.	2.94	
Robust SD	2.6	
Robust CV	16%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratories 14 and 22.



z-Scores: S1 - MeFOSE



En-Scores: S1 - MeFOSE

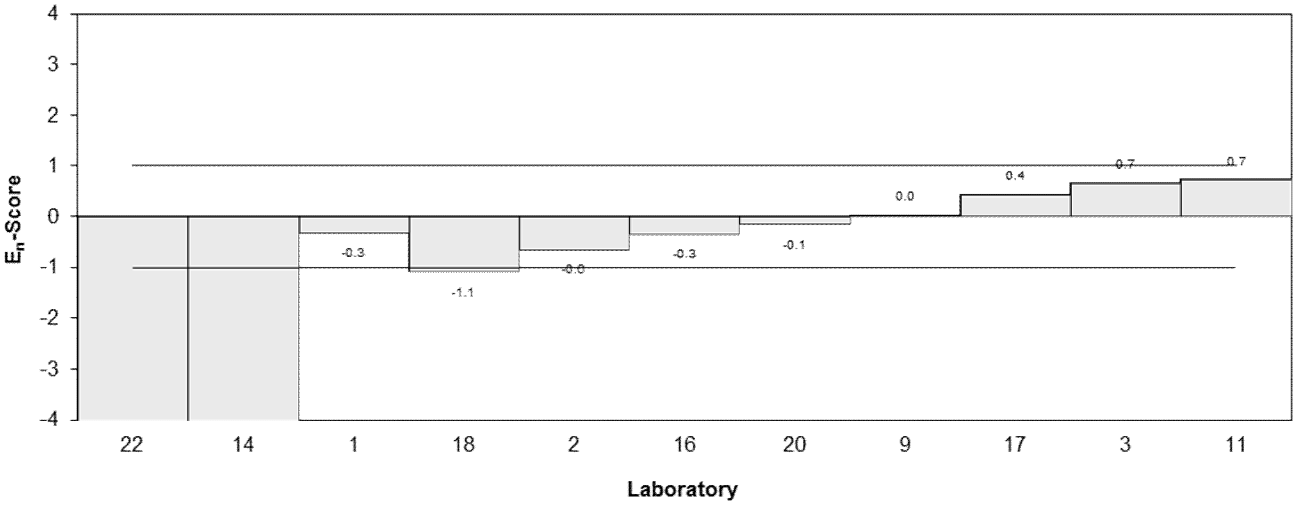


Figure 9

Table 16

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFBS
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	3	1	84	0.88	0.44
2	2.21	0.70	86	-0.67	-0.47
3	2.66	0.532	101	0.22	0.20
4	2.6	1.0	NR	0.10	0.05
5	2.70	0.54	NR	0.29	0.26
6	2.6	0.65	127	0.10	0.07
7	2.95	0.885	186	0.78	0.44
8	NT	NT	NT		
9	2.66	0.80	84.7	0.22	0.13
10	NT	NT	NT		
11	2.69	0.538	101	0.27	0.25
14*	0.92	0.17	91	-3.20	-6.78
15	2.3	0.7	106	-0.49	-0.35
16	2.4	0.041	92	-0.29	-0.86
17	2.36	0.59	75.8	-0.37	-0.31
18	2.8	0.9	NR	0.49	0.27
19	2.64	0.69	120.3	0.18	0.13
20	2.5	0.2	110	-0.10	-0.19
21	2.031	0.231	NR	-1.02	-1.81
22	2.0	0.3	93.2	-1.08	-1.60
23	2.6	0.7	72	0.10	0.07
24	NT	NT	NT		

Statistics

Assigned Value**	2.55	0.17
Spike	2.98	0.15
Robust Average	2.51	0.19
Median	2.60	0.14
Mean	2.45	
N	19	
Max.	3	
Min.	0.92	
Robust SD	0.29	
Robust CV	11%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratory 14.

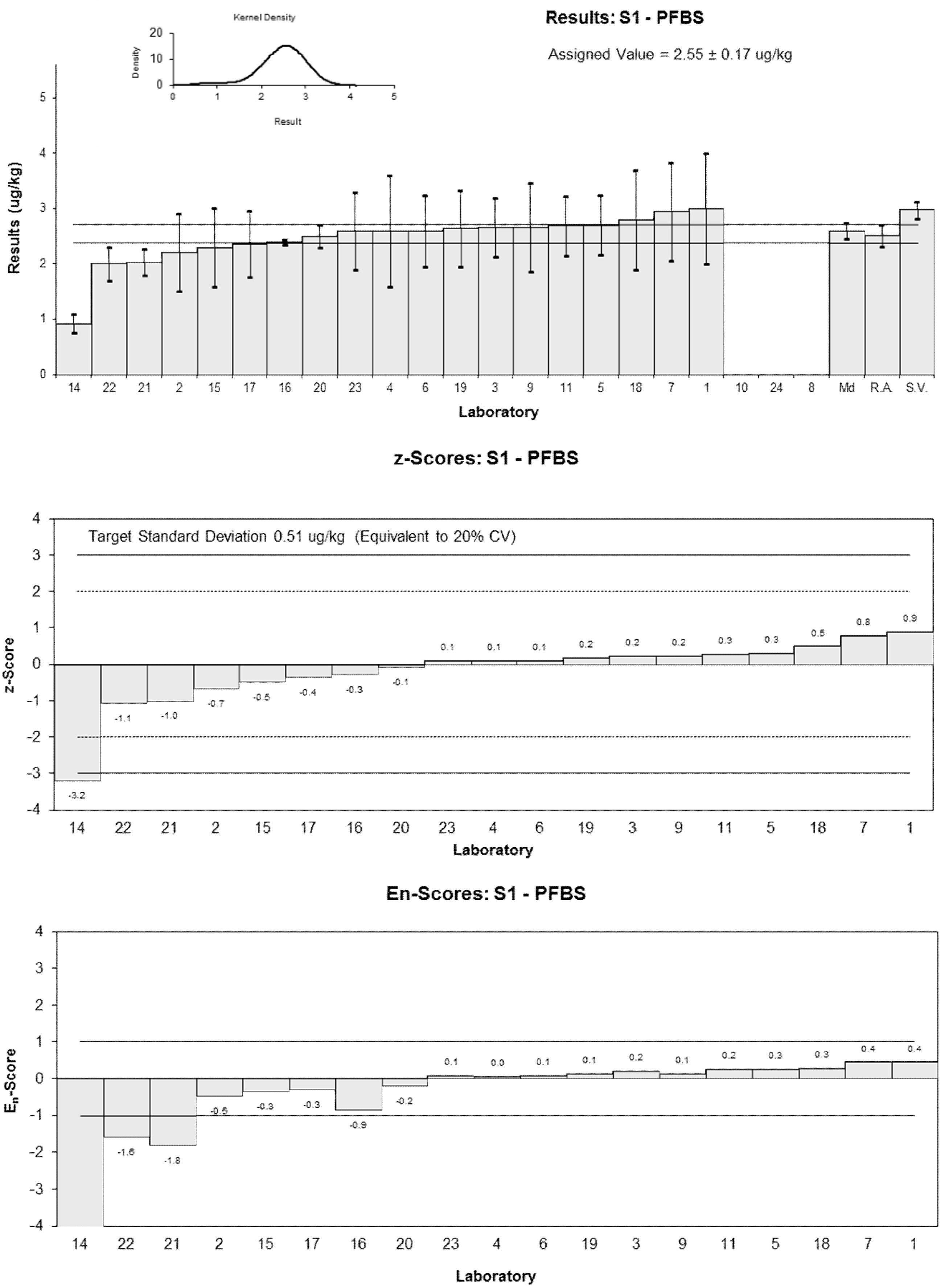

Results: S1 - PFBS
Assigned Value = 2.55 ± 0.17 ug/kg
Kernel Density
Density
Result
Results (ug/kg)
Laboratory
14 22 21 2 15 17 16 20 23 4 6 19 3 9 11 5 18 7 1 10 24 8 Md R.A. S.V.
z-Scores: S1 - PFBS
Target Standard Deviation 0.51 ug/kg (Equivalent to 20% CV)
z-Score
-3.2 -1.1 -1.0 -0.7 -0.5 -0.4 -0.3 -0.1 0.1 0.1 0.1 0.2 0.2 0.2 0.3 0.3 0.5 0.8 0.9
14 22 21 2 15 17 16 20 23 4 6 19 3 9 11 5 18 7 1
Laboratory
En-Scores: S1 - PFBS
En-Score
-1.6 -1.8 -0.5 -0.3 -0.3 -0.9 -0.2 0.1 0.0 0.1 0.1 0.2 0.1 0.2 0.3 0.3 0.4 0.4
14 22 21 2 15 17 16 20 23 4 6 19 3 9 11 5 18 7 1
Laboratory

Figure 10

Table 17

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFDA
Units	ug/kg

Participant Results

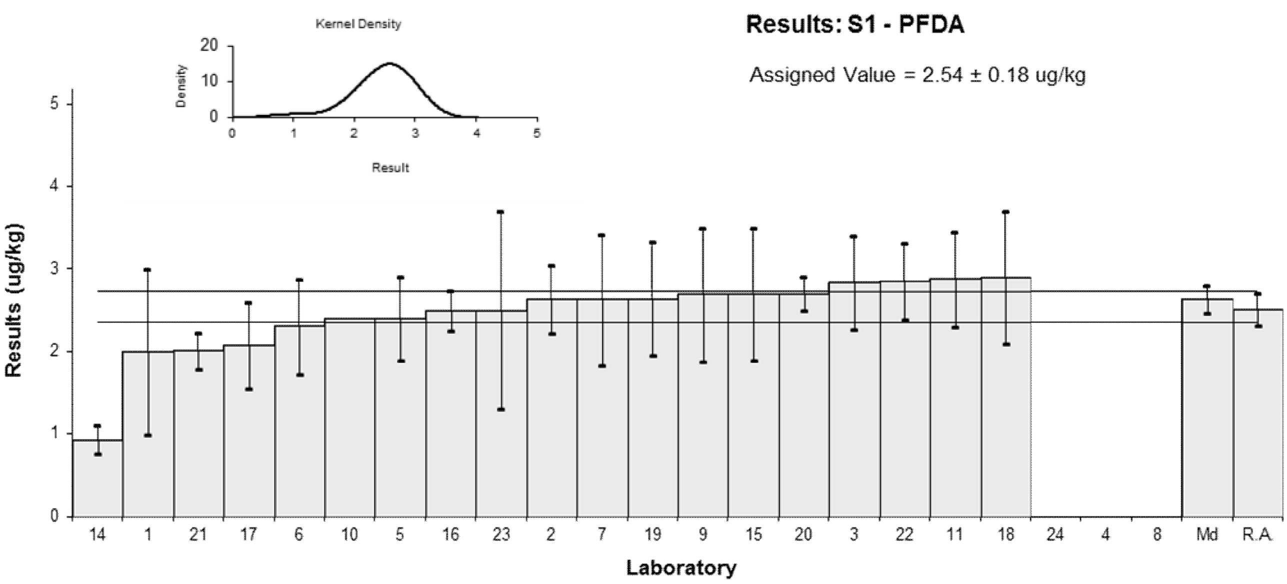
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	2	1	160	-1.06	-0.53
2	2.63	0.41	93	0.18	0.20
3	2.83	0.566	90	0.57	0.49
4	NT	NT	NT		
5	2.4	0.5	NR	-0.28	-0.26
6	2.3	0.58	113	-0.47	-0.40
7	2.63	0.789	55	0.18	0.11
8	NT	NT	NT		
9	2.69	0.81	93.3	0.30	0.18
10	2.395	NR	143	-0.29	-0.81
11	2.88	0.576	90	0.67	0.56
14*	0.93	0.17	74	-3.17	-6.50
15	2.7	0.8	60	0.31	0.20
16	2.5	0.24	78	-0.08	-0.13
17	2.08	0.52	37.7	-0.91	-0.84
18	2.9	0.8	NR	0.71	0.44
19	2.64	0.68	66.2	0.20	0.14
20	2.7	0.2	49	0.31	0.59
21	2.007	0.215	NR	-1.05	-1.90
22	2.85	0.46	93.2	0.61	0.63
23	2.5	1.2	63	-0.08	-0.03
24	NT	NT	NT		

Statistics

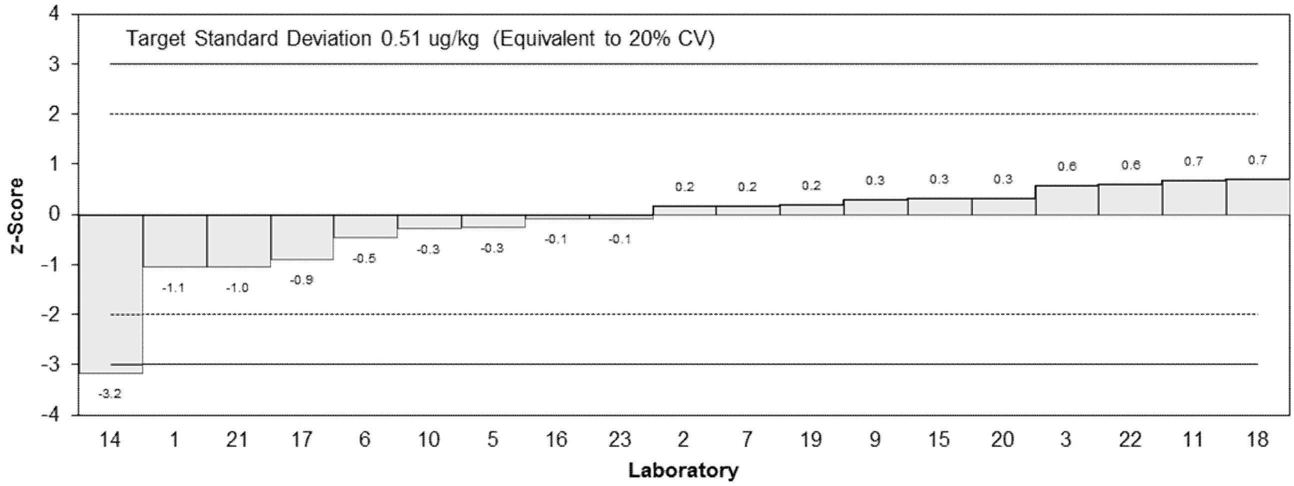
Assigned Value**	2.54	0.18
Spike	Not Spiked	
Robust Average	2.51	0.20
Median	2.63	0.16
Mean	2.45	
N	19	
Max.	2.9	
Min.	0.93	
Robust SD	0.31	
Robust CV	12%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratory 14.



z-Scores: S1 - PFDA



En-Scores: S1 - PFDA

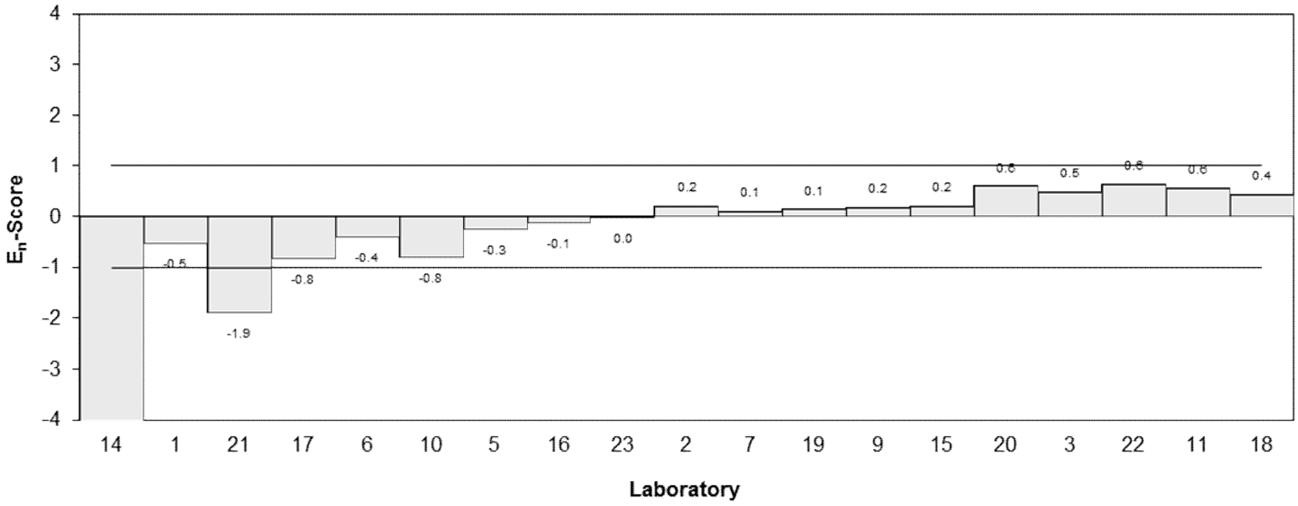


Figure 11

Table 18

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFDoA
Units	ug/kg

Participant Results

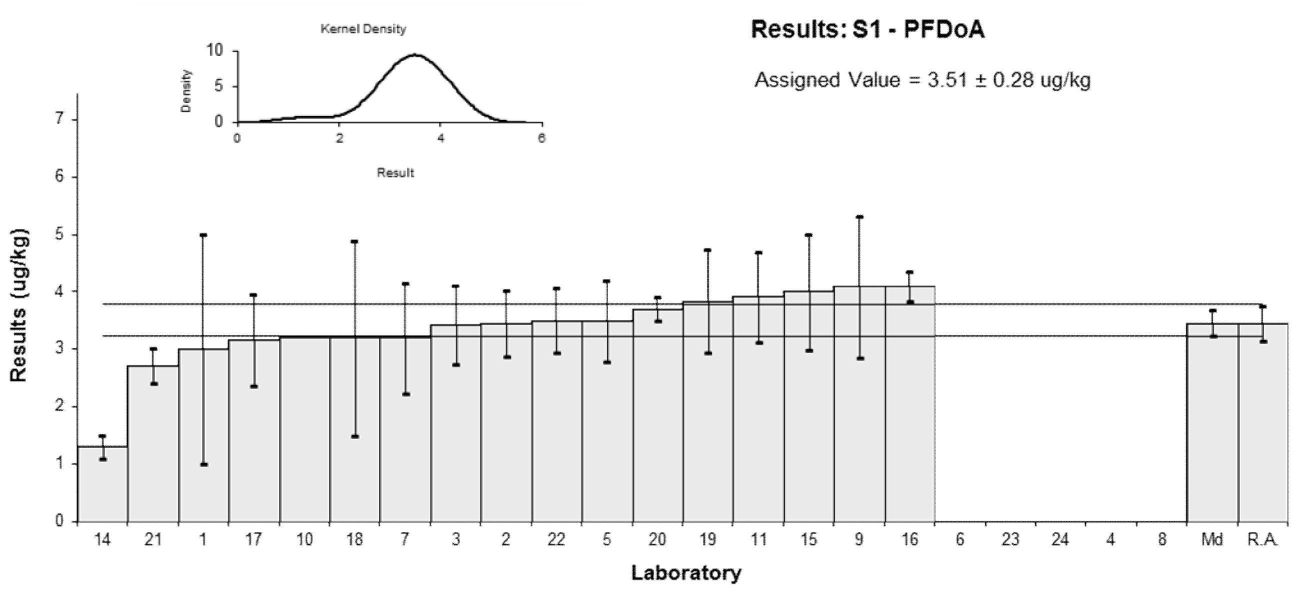
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	3	2	181	-0.73	-0.25
2	3.46	0.57	104	-0.07	-0.08
3	3.43	0.686	115	-0.11	-0.11
4	NT	NT	NT		
5	3.5	0.70	NR	-0.01	-0.01
6	< 5.0	1.8	127		
7	3.20	0.96	130	-0.44	-0.31
8	NT	NT	NT		
9	4.09	1.23	89.6	0.83	0.46
10	3.2	NR	251.95	-0.44	-1.11
11	3.91	0.782	115	0.57	0.48
14*	1.3	0.20	87	-3.15	-6.42
15	4.0	1	26	0.70	0.47
16	4.1	0.25	95	0.84	1.57
17	3.17	0.7925	20.8	-0.48	-0.40
18	3.2	1.7	NR	-0.44	-0.18
19	3.84	0.90	106.9	0.47	0.35
20	3.7	0.2	30	0.27	0.55
21	2.715	0.298	NR	-1.13	-1.94
22	3.50	0.56	93.2	-0.01	-0.02
23	NT	NT	NT		
24	NT	NT	NT		

Statistics

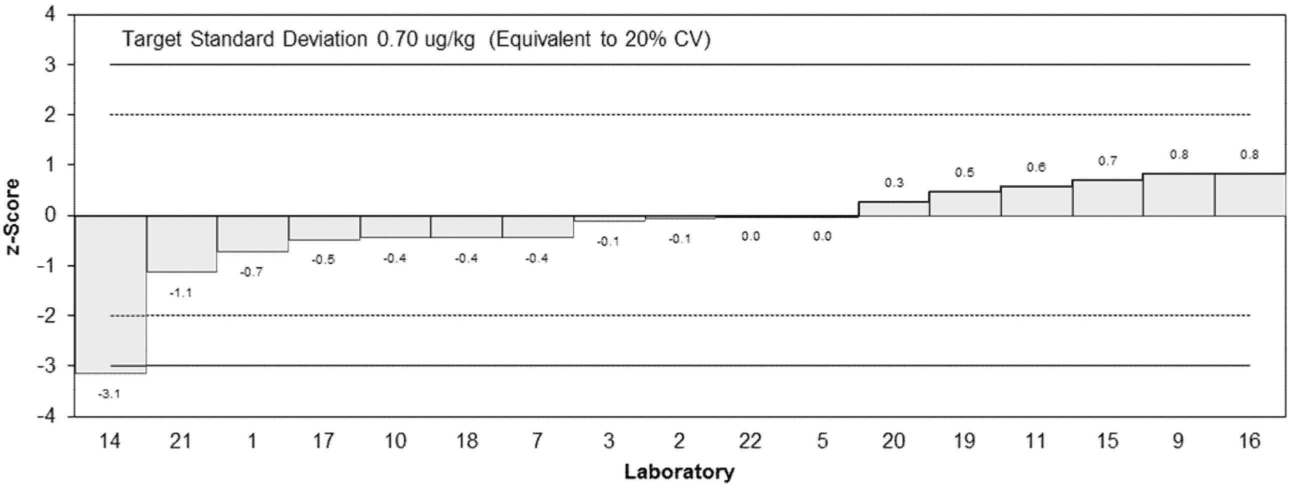
Assigned Value**	3.51	0.28
Spike	Not Spiked	
Robust Average	3.45	0.30
Median	3.46	0.22
Mean	3.37	
N	17	
Max.	4.1	
Min.	1.3	
Robust SD	0.45	
Robust CV	13%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratory 14.



z-Scores: S1 - PFDoA



En-Scores: S1 - PFDoA

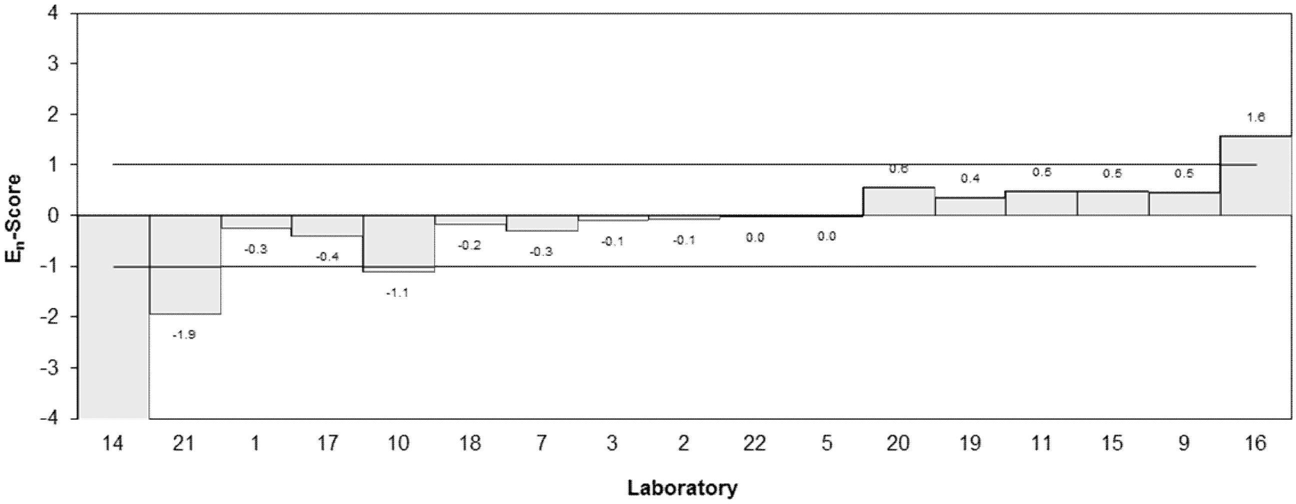


Figure 12

Table 19

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFDS
Units	ug/kg

Participant Results

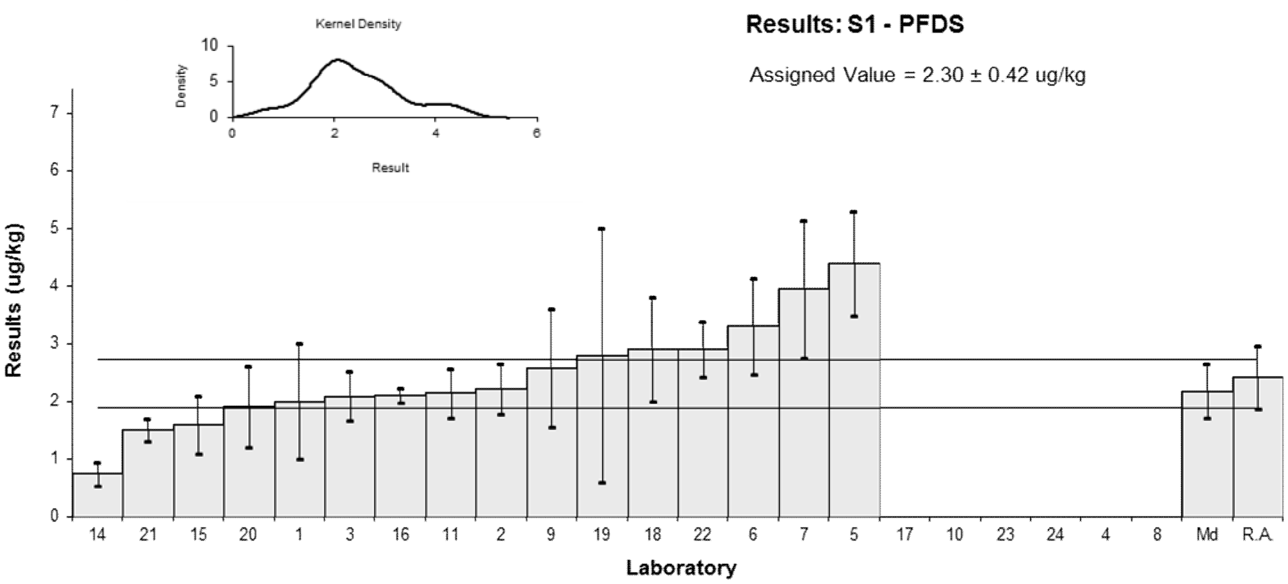
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	2	1	93	-0.65	-0.28
2	2.21	0.43	114	-0.20	-0.15
3	2.09	0.418	114	-0.46	-0.35
4	NT	NT	NT		
5	4.4	0.9	NR	4.57	2.11
6	3.3	0.83	NR	2.17	1.08
7	3.96	1.19	63	3.61	1.32
8	NT	NT	NT		
9	2.58	1.02	86.1	0.61	0.25
10	NT	NT	NT		
11	2.14	0.428	114	-0.35	-0.27
14*	0.74	0.20	NR	-3.39	-3.35
15	1.6	0.5	NR	-1.52	-1.07
16	2.1	0.12	74	-0.43	-0.46
17	NR	NR	NR		
18	2.9	0.9	NR	1.30	0.60
19	2.8	2.2	80.6	1.09	0.22
20	1.9	0.7	98	-0.87	-0.49
21	1.509	0.189	NR	-1.72	-1.72
22	2.91	0.48	93.2	1.33	0.96
23	NT	NT	NT		
24	NT	NT	NT		

Statistics

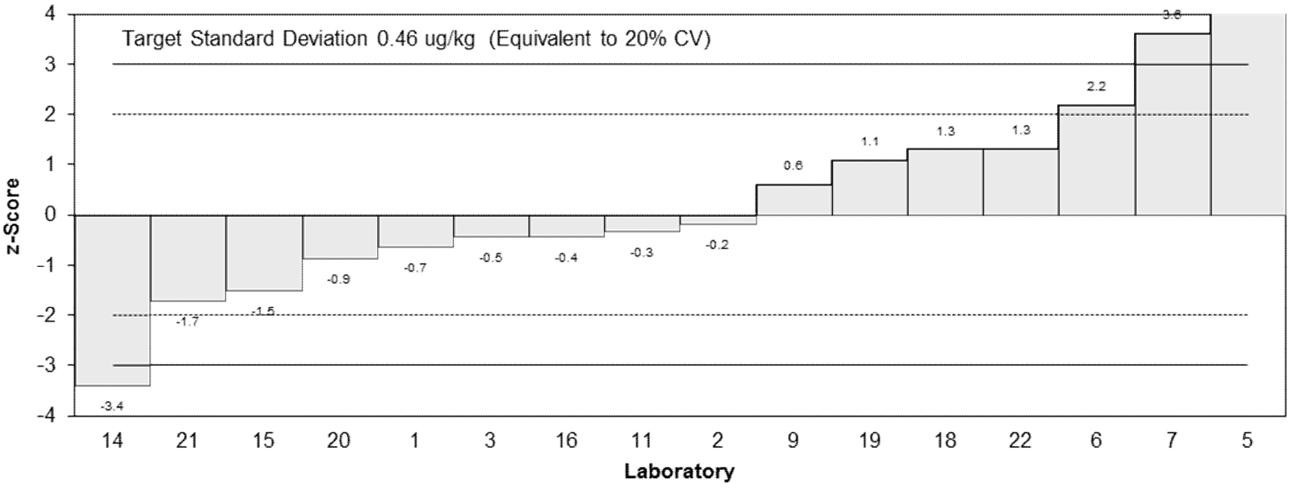
Assigned Value**	2.30	0.42
Spike	Not Spiked	
Robust Average	2.41	0.54
Median	2.18	0.47
Mean	2.45	
N	16	
Max.	4.4	
Min.	0.74	
Robust SD	0.60	
Robust CV	26%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratories 5, 7 and 14.



z-Scores: S1 - PFDS



En-Scores: S1 - PFDS

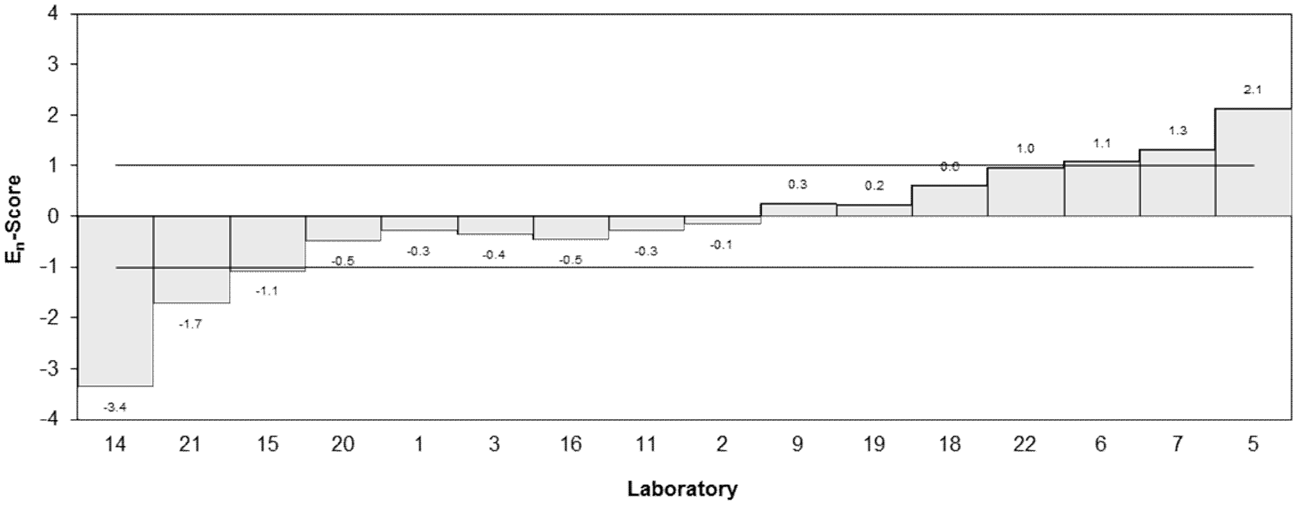


Figure 13

Table 20

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFHpS
Units	ug/kg

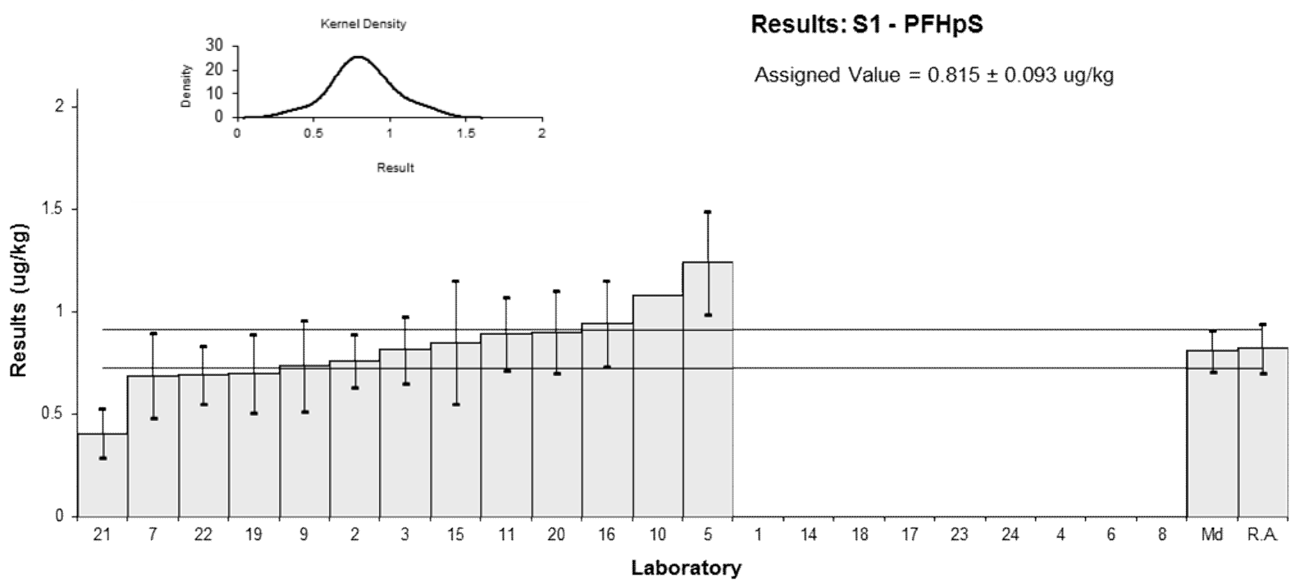
Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	< 1	NR	93		
2	0.760	0.13	86	-0.34	-0.34
3	0.814	0.163	76	-0.01	-0.01
4	NT	NT	NT		
5	1.24	0.25	NR	2.61	1.59
6	NT	NT	NT		
7	0.688	0.206	180	-0.78	-0.56
8	NT	NT	NT		
9	0.737	0.221	86.1	-0.48	-0.33
10	1.082	NR	134.75	1.64	2.87
11	0.89	0.179	76	0.46	0.37
14	<0.5	0.1	NR		
15	0.85	0.3	NR	0.21	0.11
16	0.94	0.21	73	0.77	0.54
17	NR	NR	NR		
18	<1	NR	NR		
19	0.70	0.19	120.3	-0.71	-0.54
20	0.9	0.2	84	0.52	0.39
21	0.405	0.119	NR	-2.52	-2.71
22	0.69	0.14	93.2	-0.77	-0.74
23	NT	NT	NT		
24	NT	NT	NT		

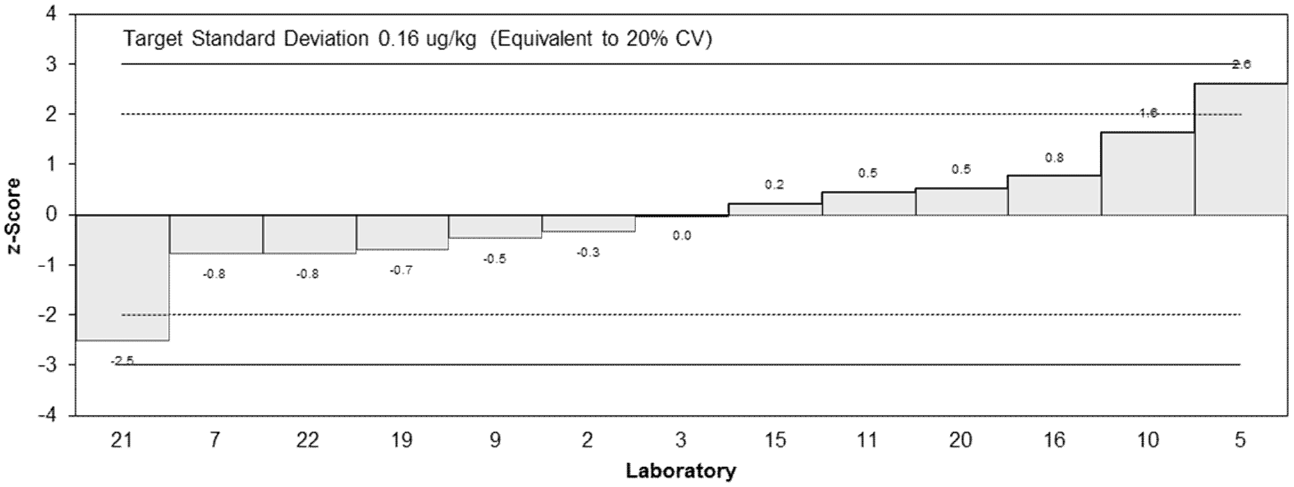
Statistics

Assigned Value*	0.815	0.093
Spike	Not Spiked	
Robust Average	0.82	0.12
Median	0.81	0.10
Mean	0.82	
N	13	
Max.	1.24	
Min.	0.405	
Robust SD	0.12	
Robust CV	15%	

*Robust average excluding laboratories 5 and 21.



z-Scores: S1 - PFHpS



En-Scores: S1 - PFHpS

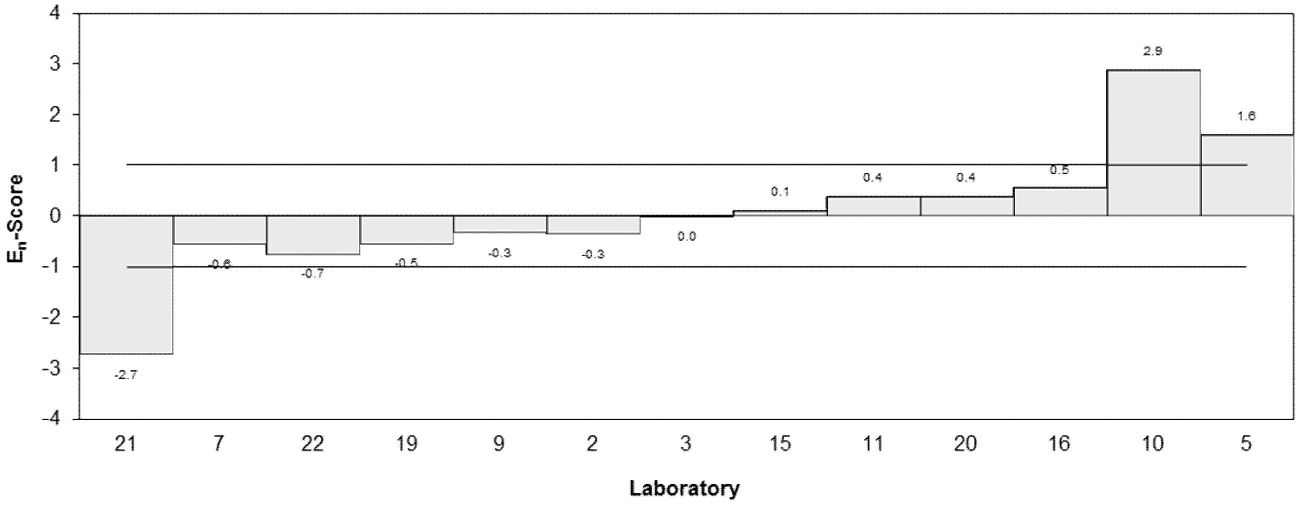


Figure 14

Table 21

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFHxS
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	6	2	95	1.52	0.67
2	4.09	0.63	86	-0.55	-0.60
3	4.98	0.996	71	0.41	0.33
4	2.3	0.91	NR	-2.50	-2.14
5	5.5	1.1	NR	0.98	0.73
6	4.8	1.2	97	0.22	0.15
7	4.25	1.28	180	-0.38	-0.25
8	NT	NT	NT		
9	4.55	1.37	94.0	-0.05	-0.03
10	4.8485	NR	216.35	0.27	0.44
11	5.68	1.14	71	1.17	0.85
14*	2.2	0.33	76	-2.61	-3.64
15	4.6	1	81	0.00	0.00
16	5.1	0.71	73	0.54	0.55
17	3.37	0.8425	75.4	-1.34	-1.21
18	4.8	1.8	NR	0.22	0.11
19	4.8	1.0	120.3	0.22	0.17
20	4.6	0.5	84	0.00	0.00
21	2.291	0.241	NR	-2.51	-3.73
22	3.34	0.43	93.2	-1.37	-1.76
23	5.8	2.6	89	1.30	0.45
24	NT	NT	NT		

Statistics

Assigned Value**	4.60	0.57
Spike	Not Spiked	
Robust Average	4.47	0.64
Median	4.70	0.37
Mean	4.40	
N	20	
Max.	6	
Min.	2.2	
Robust SD	1.00	
Robust CV	22%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratory 14.

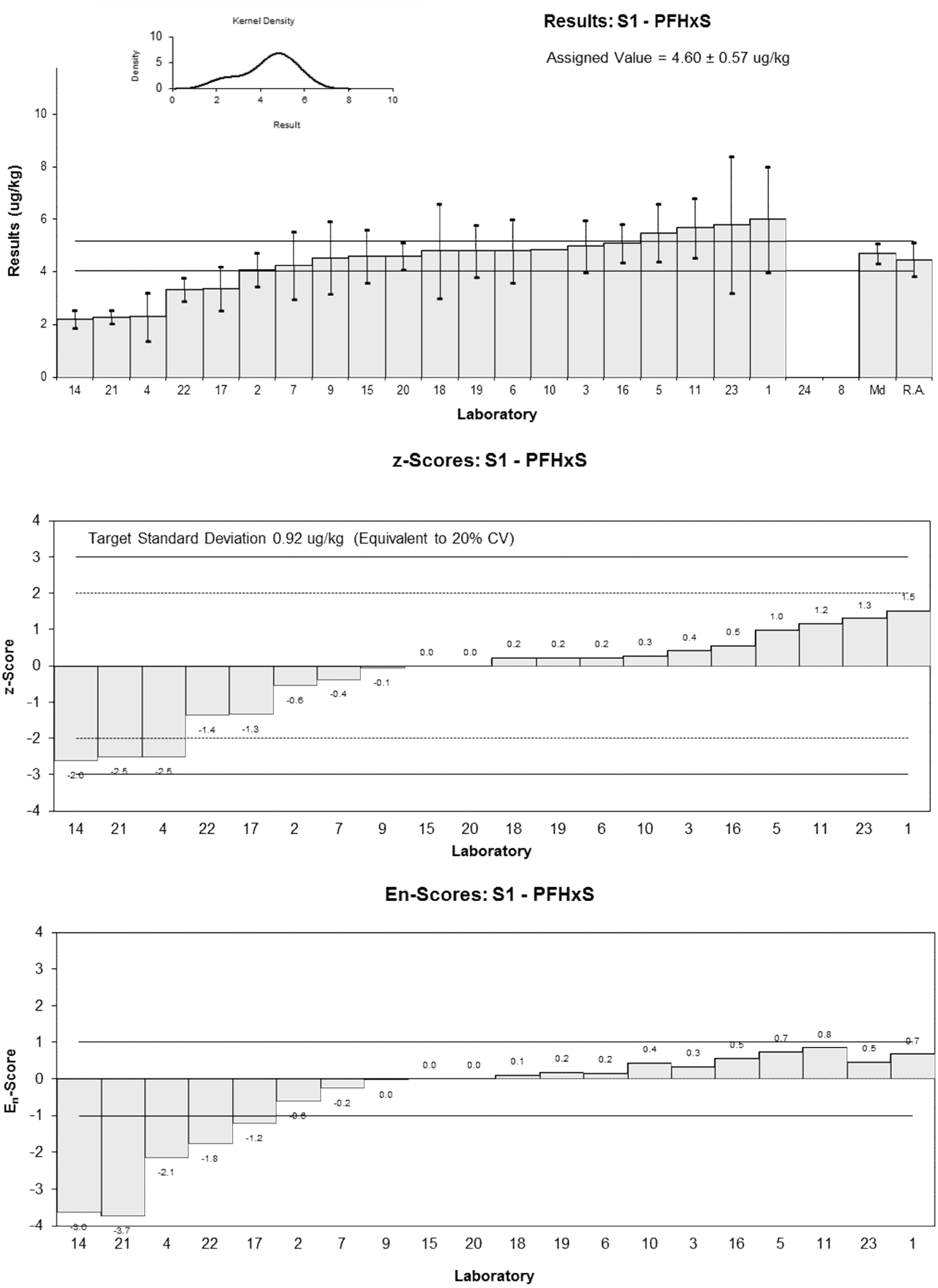

Results: S1 - PFHxS
Assigned Value = 4.60 ± 0.57 ug/kg
Kernel Density
Density
10
5
0
0 2 4 6 8 10
Result
Results (ug/kg)
0 2 4 6 8 10
14 21 4 22 17 2 7 9 15 20 18 19 6 10 3 16 5 11 23 1 24 8 Md R.A.
Laboratory
z-Scores: S1 - PFHxS
Target Standard Deviation 0.92 ug/kg (Equivalent to 20% CV)
z-Score
4 3 2 1 0 -1 -2 -3 -4
-2.6 -2.5 -2.5 -1.4 -1.3 -0.6 -0.4 -0.1 0.0 0.0 0.2 0.2 0.2 0.3 0.4 0.5 1.0 1.2 1.3 1.5
14 21 4 22 17 2 7 9 15 20 18 19 6 10 3 16 5 11 23 1
Laboratory
En-Scores: S1 - PFHxS
En-Score
4 3 2 1 0 -1 -2 -3 -4
-3.6 -3.7 -2.1 -1.8 -1.2 -0.6 -0.2 0.0 0.0 0.0 0.1 0.2 0.2 0.4 0.3 0.5 0.7 0.8 0.5 0.7
14 21 4 22 17 2 7 9 15 20 18 19 6 10 3 16 5 11 23 1
Laboratory

Figure 15

Table 22

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFNA
Units	ug/kg

Participant Results

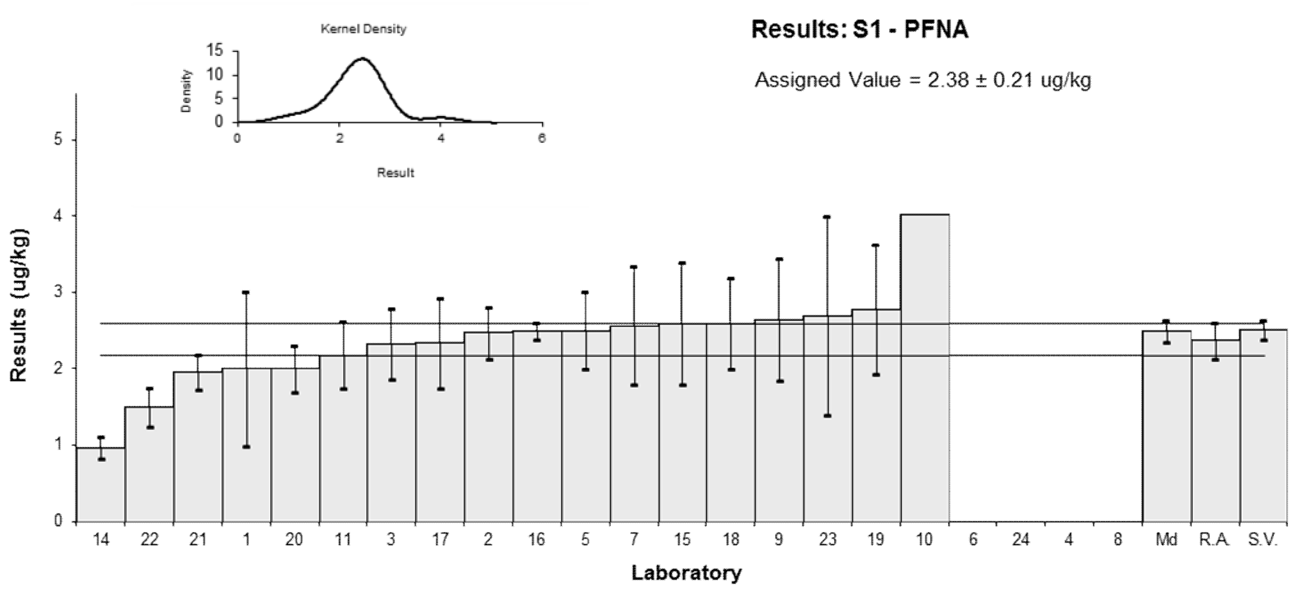
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	2	1	148	-0.80	-0.37
2	2.47	0.34	65	0.19	0.23
3	2.33	0.466	77	-0.11	-0.10
4	NT	NT	NT		
5	2.5	0.5	NR	0.25	0.22
6	< 1.0	0.25	77		
7	2.57	0.771	29	0.40	0.24
8	NT	NT	NT		
9	2.65	0.80	93.9	0.57	0.33
10	4.0165	NR	41.95	3.44	7.79
11	2.18	0.436	77	-0.42	-0.41
14*	0.97	0.14	71	-2.96	-5.59
15	2.6	0.8	63	0.46	0.27
16	2.5	0.11	70	0.25	0.51
17	2.34	0.585	61.8	-0.08	-0.06
18	2.6	0.6	NR	0.46	0.35
19	2.78	0.84	81.0	0.84	0.46
20	2.0	0.3	65	-0.80	-1.04
21	1.956	0.229	NR	-0.89	-1.36
22	1.50	0.25	93.2	-1.85	-2.70
23	2.7	1.3	57	0.67	0.24
24	NT	NT	NT		

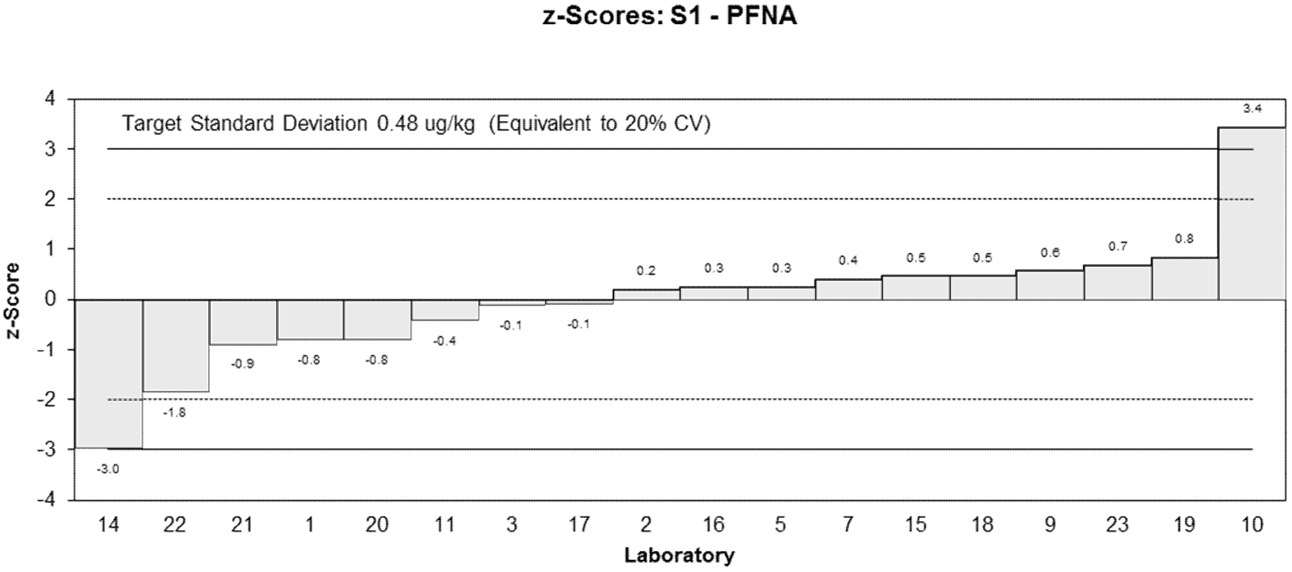
Statistics

Assigned Value**	2.38	0.21
Spike	2.51	0.13
Robust Average	2.37	0.23
Median	2.49	0.14
Mean	2.37	
N	18	
Max.	4.0165	
Min.	0.97	
Robust SD	0.33	
Robust CV	14%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratories 10 and 14.


Results: S1 - PFNA
Assigned Value = 2.38 ± 0.21 ug/kg
Kernel Density
Density
15
10
5
0
0
2
4
6
Result
Results (ug/kg)
0
1
2
3
4
5
14
22
21
1
20
11
3
17
2
16
5
7
15
18
9
23
19
10
6
24
4
8
Md
R.A.
S.V.
Laboratory


z-Scores: S1 - PFNA
Target Standard Deviation 0.48 ug/kg (Equivalent to 20% CV)
z-Score
4
3
2
1
0
-1
-2
-3
-4
-3.0
-1.8
-0.9
-0.8
-0.8
-0.4
-0.1
-0.1
0.2
0.3
0.3
0.4
0.5
0.5
0.6
0.7
0.8
3.4
14
22
21
1
20
11
3
17
2
16
5
7
15
18
9
23
19
10
Laboratory

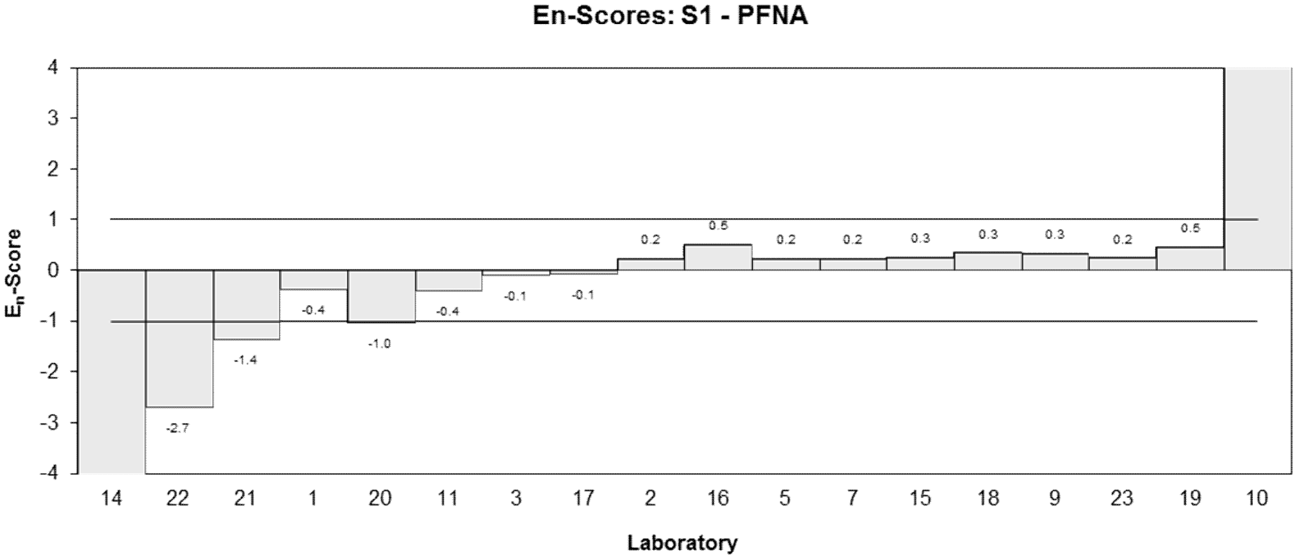

En-Scores: S1 - PFNA
E_n-Score
4
3
2
1
0
-1
-2
-3
-4
-2.7
-1.4
-0.4
-1.0
-0.4
-0.1
-0.1
0.2
0.5
0.2
0.2
0.3
0.3
0.3
0.2
0.5
14
22
21
1
20
11
3
17
2
16
5
7
15
18
9
23
19
10
Laboratory

Figure 16

Table 23

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFNS
Units	ug/kg

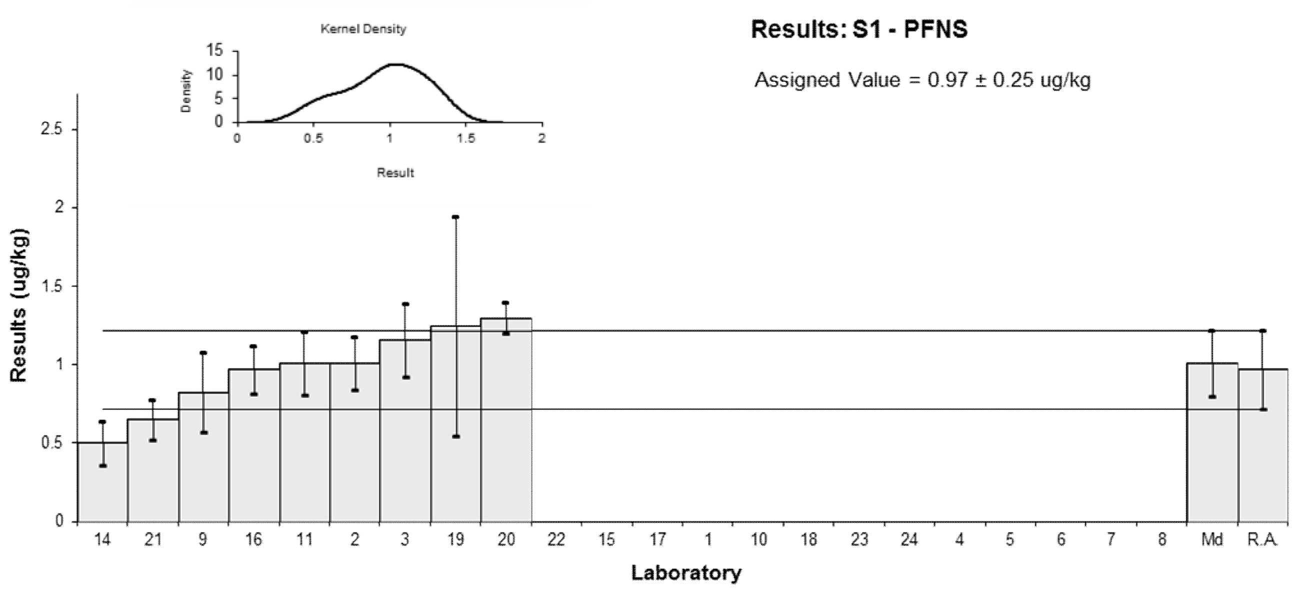
Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	NT	NT	NT		
2	1.01	0.17	114	0.21	0.13
3	1.16	0.232	76	0.98	0.56
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	NT	NT	NT		
8	NT	NT	NT		
9	0.827	0.257	86.1	-0.74	-0.40
10	NT	NT	NT		
11	1.01	0.202	76	0.21	0.12
14*	0.5	0.14	NR	-2.42	-1.64
15	<1	NR	NR		
16	0.97	0.15	74	0.00	0.00
17	NR	NR	NR		
18	NT	NT	NT		
19	1.25	0.70	80.6	1.44	0.38
20	1.3	0.1	98	1.70	1.23
21	0.652	0.129	NR	-1.64	-1.13
22	<0.1	NR	93.2		
23	NT	NT	NT		
24	NT	NT	NT		

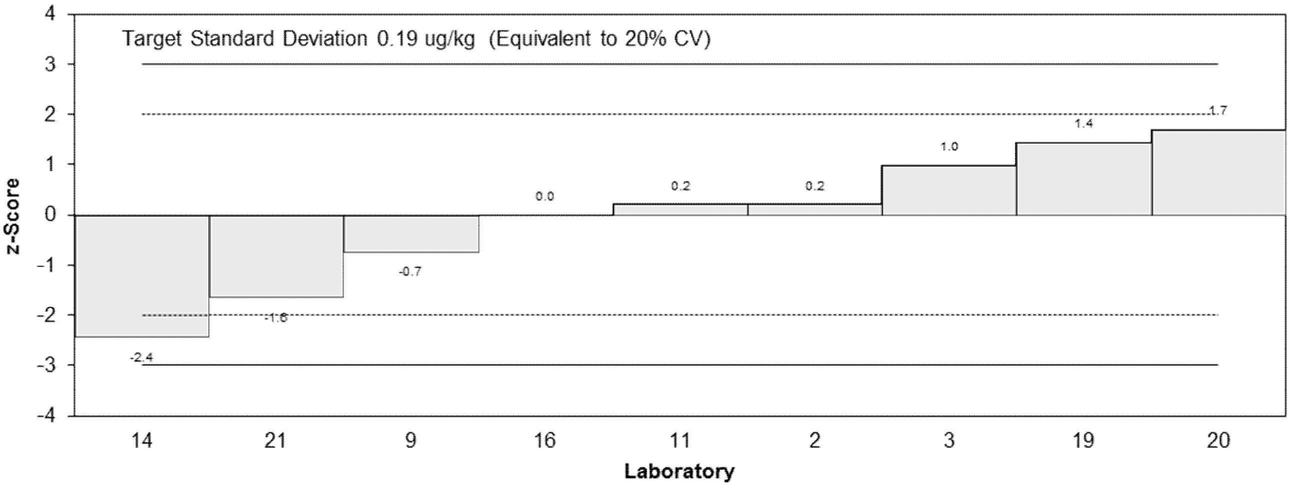
Statistics

Assigned Value	0.97	0.25
Spike	Not Spiked	
Robust Average	0.97	0.25
Median	1.01	0.21
Mean	0.96	
N	9	
Max.	1.3	
Min.	0.5	
Robust SD	0.30	
Robust CV	31%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.



z-Scores: S1 - PFNS



En-Scores: S1 - PFNS

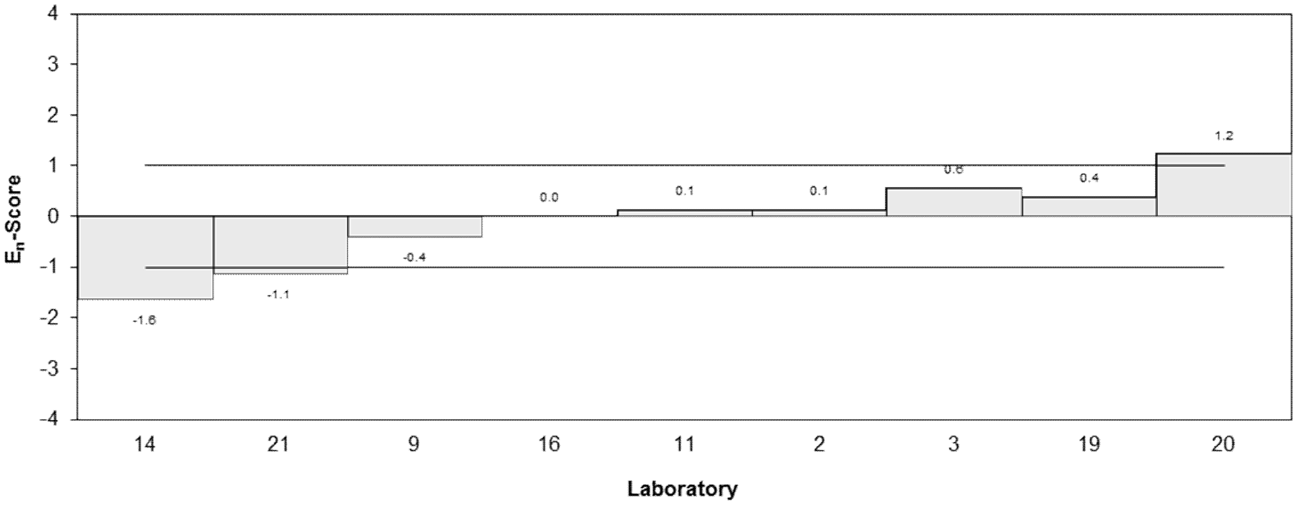


Figure 17

Table 24

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFOA
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	5	2	116	0.06	0.03
2	5.25	0.89	58	0.31	0.32
3	4.61	0.923	80	-0.33	-0.33
4	3.3	1.3	NR	-1.66	-1.21
5	5.0	1.0	NR	0.06	0.06
6	5.7	1.5	85	0.77	0.49
7	4.63	1.39	78	-0.31	-0.22
8	9.45	0.945	NR	4.56	4.43
9	5.33	1.60	86.6	0.39	0.24
10	6.1755	NR	38.3	1.25	3.25
11	5.56	1.11	80	0.63	0.53
14*	1.9	0.30	65	-3.08	-6.28
15	5.2	2	63	0.26	0.13
16	4.7	1.1	60	-0.24	-0.21
17	4.32	1.08	78	-0.63	-0.54
18	5.3	1.6	123	0.36	0.22
19	5.5	1.2	82.9	0.57	0.44
20	4.9	0.7	56	-0.04	-0.05
21	4.040	0.421	NR	-0.91	-1.59
22	3.35	0.52	93.2	-1.61	-2.47
23	5.0	1.4	84	0.06	0.04
24	NT	NT	NT		

Statistics

Assigned Value**	4.94	0.38
Spike	5.55	0.28
Robust Average	4.92	0.47
Median	5.00	0.26
Mean	4.96	
N	21	
Max.	9.45	
Min.	1.9	
Robust SD	0.66	
Robust CV	13%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratories 8 and 14.

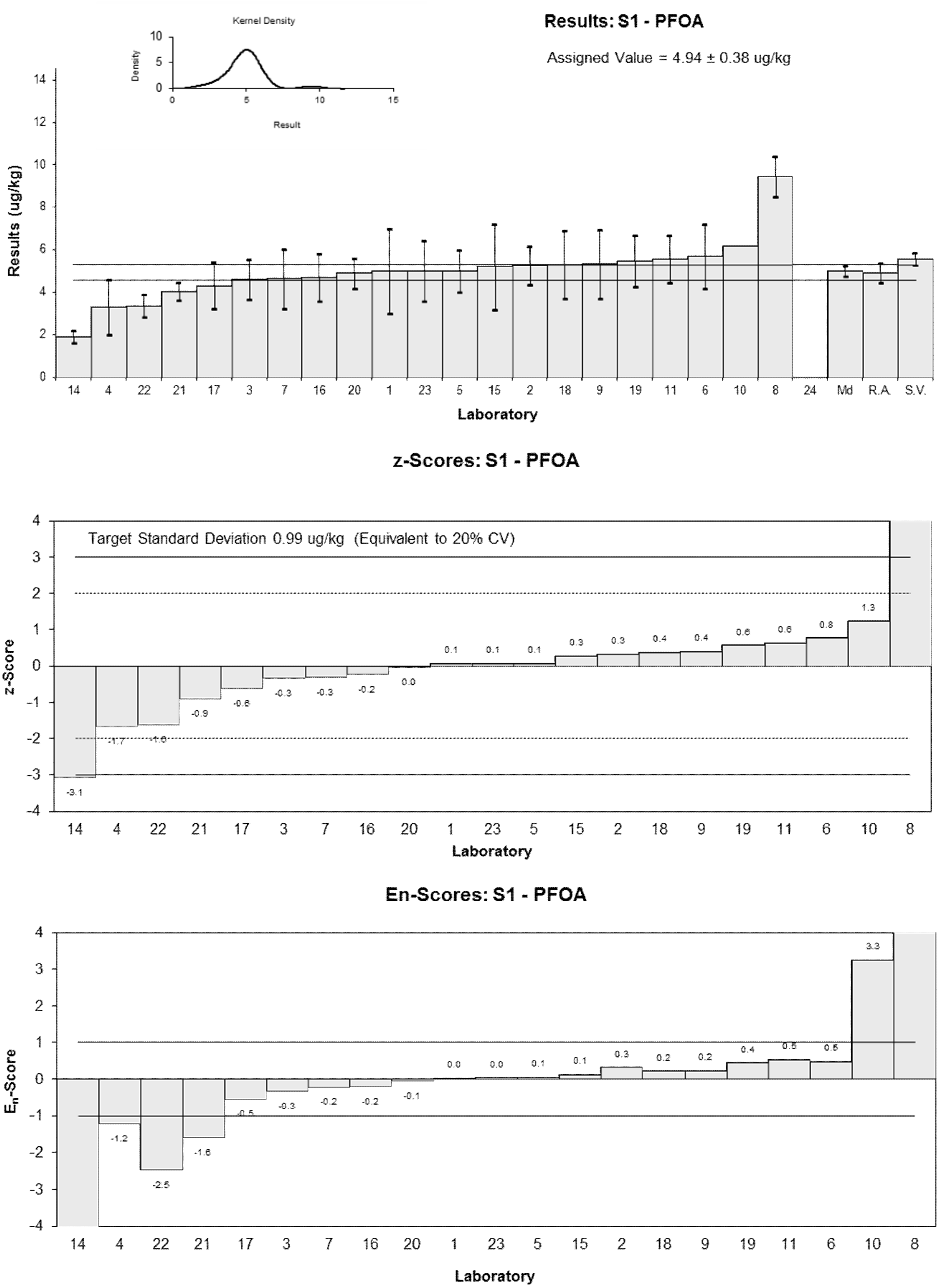

Results: S1 - PFOA
Assigned Value = 4.94 ± 0.38 ug/kg
Kernel Density
Density
10
5
0
0
5
10
15
Result
Results (ug/kg)
14
12
10
8
6
4
2
0
14
4
22
21
17
3
7
16
20
1
23
5
15
2
18
9
19
11
6
10
8
24
Md
R.A.
S.V.
Laboratory
z-Scores: S1 - PFOA
Target Standard Deviation 0.99 ug/kg (Equivalent to 20% CV)
z-Score
4
3
2
1
0
-1
-2
-3
-4
-3.1
-1.7
-1.6
-0.9
-0.6
-0.3
-0.3
-0.2
0.0
0.1
0.1
0.1
0.3
0.3
0.4
0.4
0.6
0.6
0.8
1.3
14
4
22
21
17
3
7
16
20
1
23
5
15
2
18
9
19
11
6
10
8
Laboratory
En-Scores: S1 - PFOA
E_n-Score
4
3
2
1
0
-1
-2
-3
-4
-1.2
-2.5
-1.6
-0.5
-0.3
-0.2
-0.2
-0.1
0.0
0.0
0.1
0.1
0.3
0.2
0.2
0.4
0.5
0.5
3.3
14
4
22
21
17
3
7
16
20
1
23
5
15
2
18
9
19
11
6
10
8
Laboratory

Figure 18

Table 25

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFOS
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	190	60	93	-0.05	-0.03
2	182	57	114	-0.26	-0.17
3	185	37.0	76	-0.18	-0.18
4	110	43	NR	-2.14	-1.86
5	221	55	NR	0.76	0.52
6	190	48	105	-0.05	-0.04
7	297	89.1	77.3	2.73	1.17
8	251	25.1	NR	1.54	2.18
9	204	61	86.1	0.31	0.19
10	206.503	NR	134.75	0.38	1.45
11	190	38.1	76	-0.05	-0.05
14*	84	15	78	-2.81	-5.99
15	179	50	67	-0.34	-0.25
16	196	10	74	0.10	0.28
17	186	46.5	50.2	-0.16	-0.13
18	178	63.8	128	-0.36	-0.22
19	185	42	80.6	-0.18	-0.16
20	197	22	98	0.13	0.21
21	213.785	21.536	NR	0.57	0.92
22	149	22	93.2	-1.12	-1.78
23	200	80	42	0.21	0.10
24	NT	NT	NT		

Statistics

Assigned Value**	192	10
Spike	Not Spiked	
Robust Average	192	13
Median	190	7
Mean	190	
N	21	
Max.	297	
Min.	84	
Robust SD	18	
Robust CV	9.4%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratories 7 and 14.

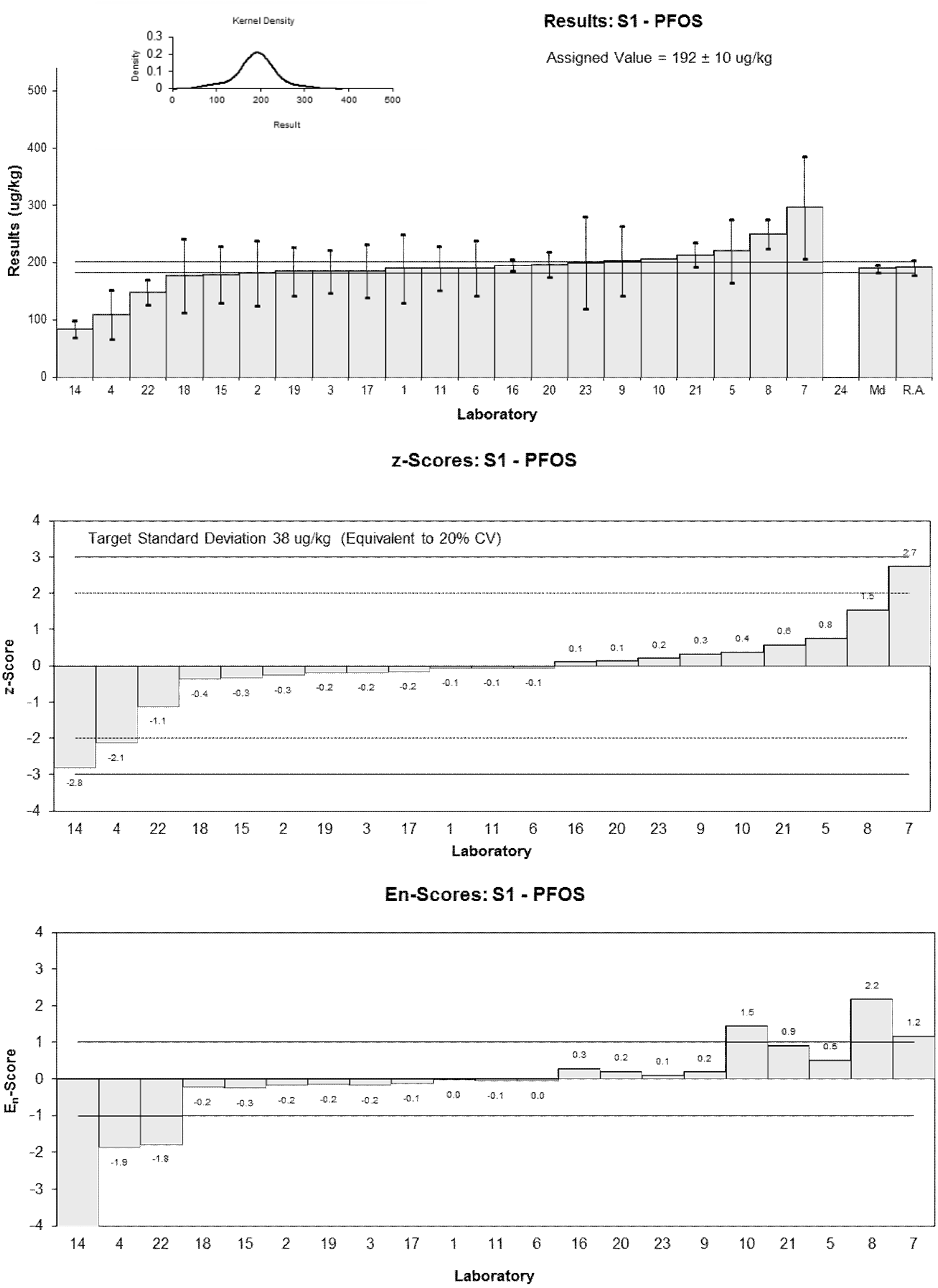

Results: S1 - PFOS
Assigned Value = 192 ± 10 ug/kg
Kernel Density
Density
0.3
0.2
0.1
0
0
100
200
300
400
500
Result
Results (ug/kg)
500
400
300
200
100
0
14
4
22
18
15
2
19
3
17
1
11
6
16
20
23
9
10
21
5
8
7
24
Md
R.A.
Laboratory
z-Scores: S1 - PFOS
Target Standard Deviation 38 ug/kg (Equivalent to 20% CV)
z-Score
4
3
2
1
0
-1
-2
-3
-4
-2.8
-2.1
-1.1
-0.4
-0.3
-0.3
-0.2
-0.2
-0.2
-0.1
-0.1
-0.1
0.1
0.1
0.2
0.3
0.4
0.6
0.8
1.5
2.7
14
4
22
18
15
2
19
3
17
1
11
6
16
20
23
9
10
21
5
8
7
Laboratory
En-Scores: S1 - PFOS
En-Score
4
3
2
1
0
-1
-2
-3
-4
-1.9
-1.8
-0.2
-0.3
-0.2
-0.2
-0.2
-0.1
0.0
-0.1
0.0
0.3
0.2
0.1
0.2
1.5
0.9
0.5
2.2
1.2
14
4
22
18
15
2
19
3
17
1
11
6
16
20
23
9
10
21
5
8
7
Laboratory

Figure 19

Table 26

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFOSA
Units	ug/kg

Participant Results

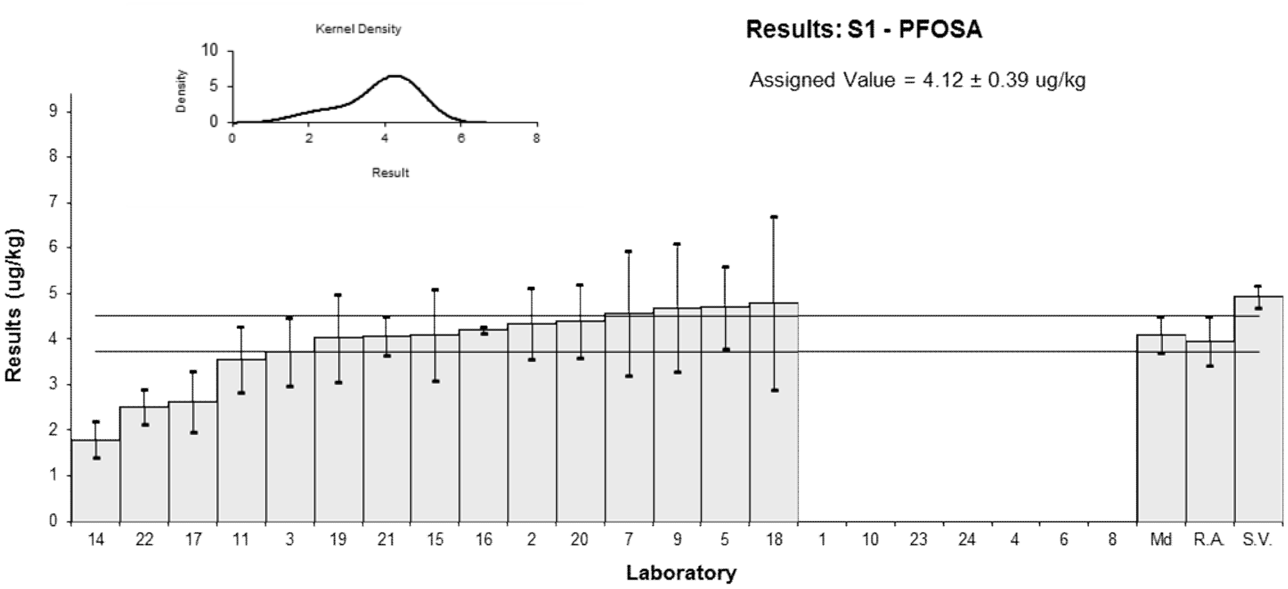
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	<5	NR	102		
2	4.34	0.78	50	0.27	0.25
3	3.74	0.748	62	-0.46	-0.45
4	NT	NT	NT		
5	4.7	0.9	NR	0.70	0.59
6	NT	NT	NT		
7	4.57	1.37	13.2	0.55	0.32
8	NT	NT	NT		
9	4.69	1.41	127	0.69	0.39
10	NT	NT	NT		
11	3.57	0.714	62	-0.67	-0.68
14*	1.8	0.40	67	-2.82	-4.15
15	4.1	1	36	-0.02	-0.02
16	4.2	0.074	75	0.10	0.20
17	2.64	0.66	28.1	-1.80	-1.93
18	4.8	1.9	NR	0.83	0.35
19	4.03	0.95	11.7	-0.11	-0.09
20	4.4	0.8	36	0.34	0.31
21	4.080	0.433	NR	-0.05	-0.07
22	2.51	0.38	93.2	-1.95	-2.96
23	NT	NT	NT		
24	NT	NT	NT		

Statistics

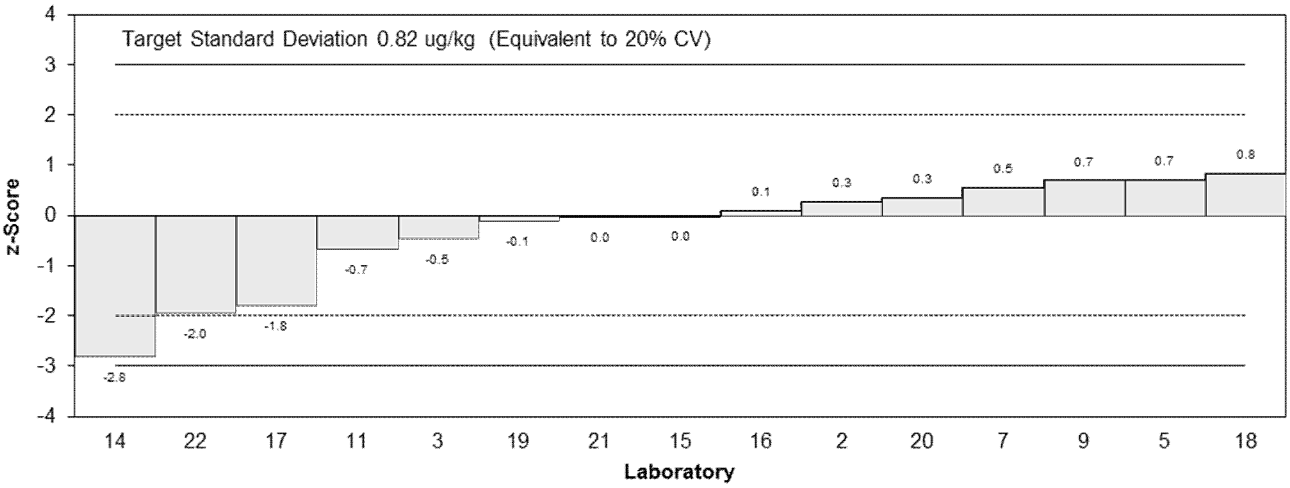
Assigned Value**	4.12	0.39
Spike	4.94	0.25
Robust Average	3.96	0.53
Median	4.10	0.39
Mean	3.88	
N	15	
Max.	4.8	
Min.	1.8	
Robust SD	0.58	
Robust CV	14%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratory 14.


Results: S1 - PFOSA

Assigned Value = 4.12 ± 0.39 ug/kg


z-Scores: S1 - PFOSA

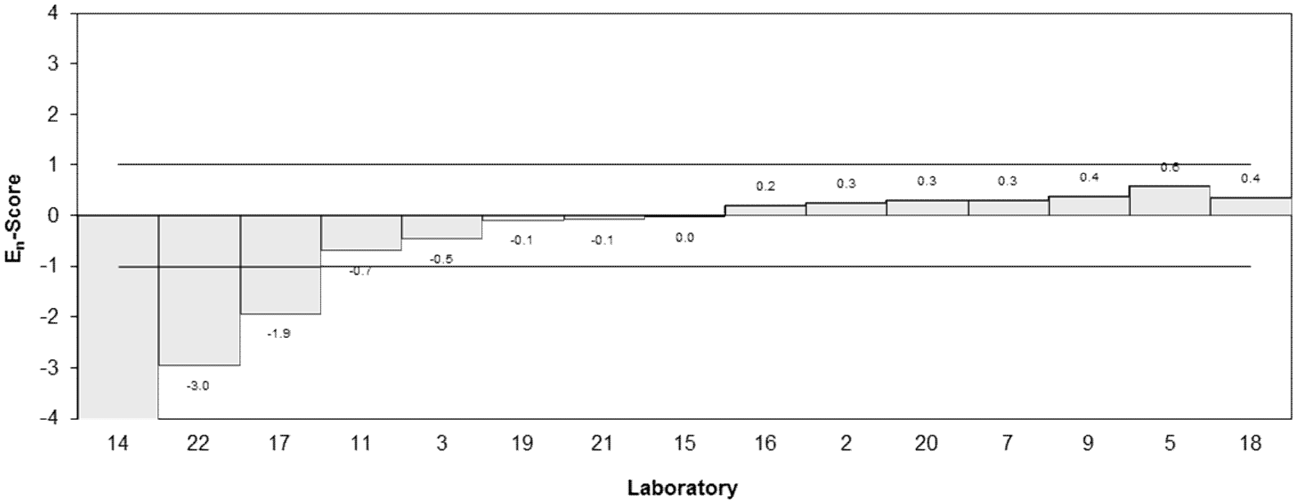

En-Scores: S1 - PFOSA

Figure 20

Table 27

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFPeA
Units	ug/kg

Participant Results

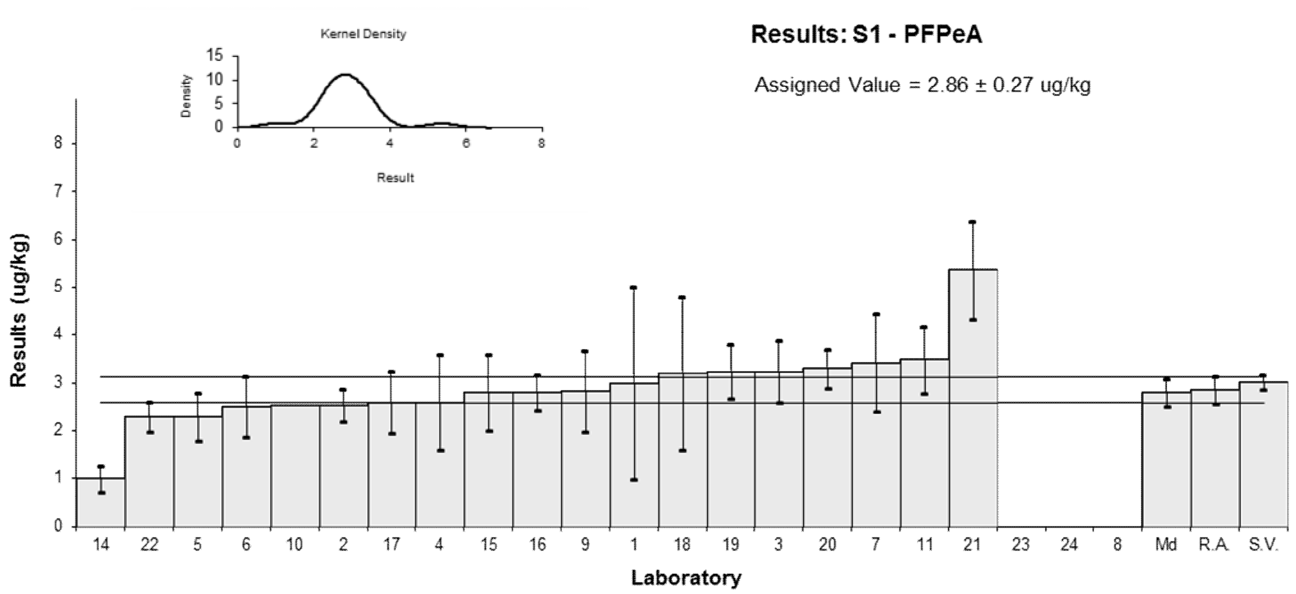
Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	3	2	76	0.24	0.07
2	2.54	0.33	60	-0.56	-0.75
3	3.24	0.648	90	0.66	0.54
4	2.6	1.0	NR	-0.45	-0.25
5	2.3	0.5	NR	-0.98	-0.99
6	2.5	0.63	78	-0.63	-0.53
7	3.43	1.03	138	1.00	0.54
8	NT	NT	NT		
9	2.83	0.85	119	-0.05	-0.03
10	2.5215	NR	13.85	-0.59	-1.25
11	3.49	0.699	90	1.10	0.84
14*	1.0	0.27	88	-3.25	-4.87
15	2.8	0.8	46	-0.10	-0.07
16	2.8	0.36	81	-0.10	-0.13
17	2.6	0.65	103	-0.45	-0.37
18	3.2	1.6	NR	0.59	0.21
19	3.24	0.57	70.7	0.66	0.60
20	3.3	0.4	46	0.77	0.91
21	5.368	1.015	NR	4.38	2.39
22	2.29	0.31	93.2	-1.00	-1.39
23	NT	NT	NT		
24	NT	NT	NT		

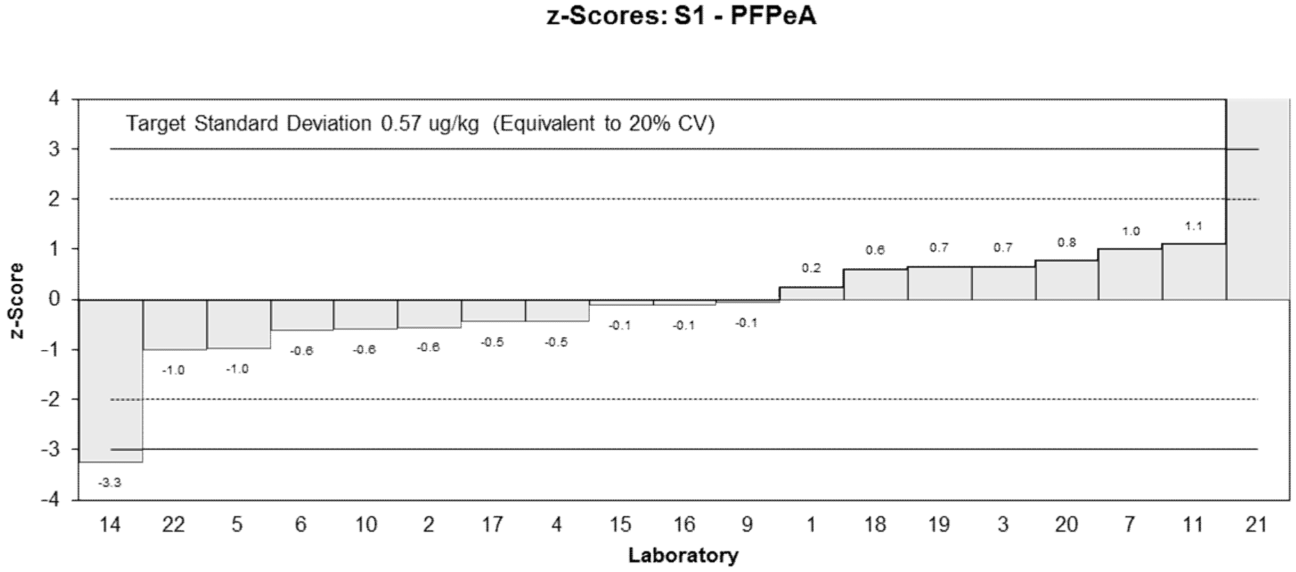
Statistics

Assigned Value**	2.86	0.27
Spike	3.01	0.15
Robust Average	2.86	0.29
Median	2.80	0.29
Mean	2.90	
N	19	
Max.	5.368	
Min.	1	
Robust SD	0.45	
Robust CV	16%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Robust average excluding laboratories 14 and 21.


Results: S1 - PFPeA
Assigned Value = 2.86 ± 0.27 ug/kg
Kernel Density
Density
15
10
5
0
0
2
4
6
8
Result
Results (ug/kg)
0
1
2
3
4
5
6
7
8
14
22
5
6
10
2
17
4
15
16
9
1
18
19
3
20
7
11
21
23
24
8
Md
R.A.
S.V.
Laboratory


z-Scores: S1 - PFPeA
Target Standard Deviation 0.57 ug/kg (Equivalent to 20% CV)
z-Score
4
3
2
1
0
-1
-2
-3
-4
-3.3
-1.0
-1.0
-0.6
-0.6
-0.6
-0.5
-0.5
-0.1
-0.1
-0.1
0.2
0.6
0.7
0.7
0.8
1.0
1.1
14
22
5
6
10
2
17
4
15
16
9
1
18
19
3
20
7
11
21
Laboratory

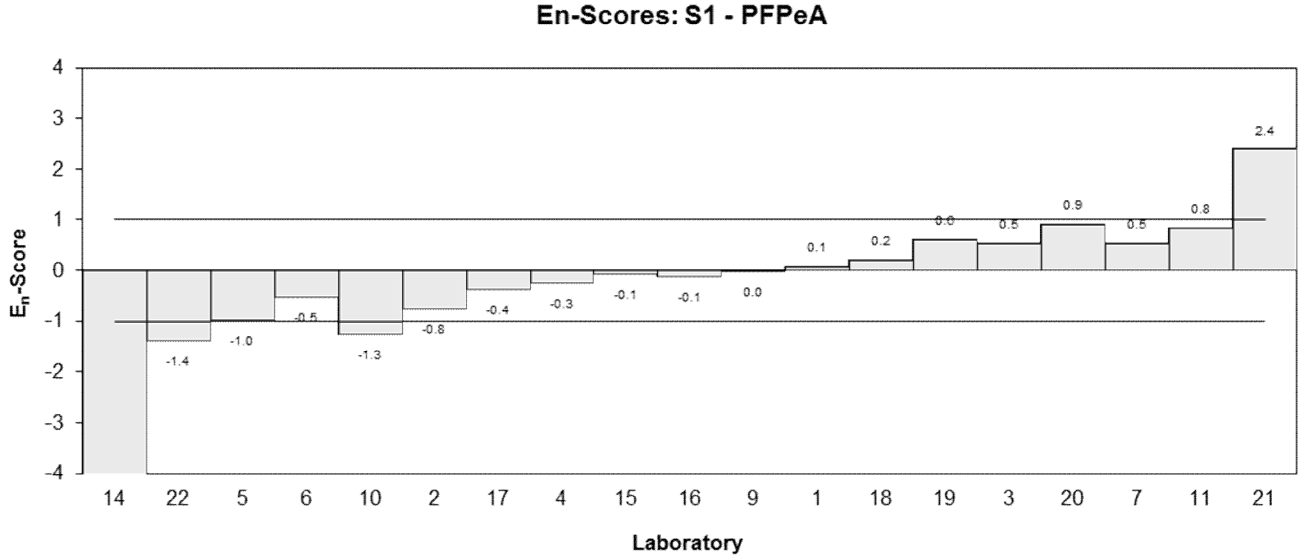

En-Scores: S1 - PFPeA
En-Score
4
3
2
1
0
-1
-2
-3
-4
-1.4
-1.0
-0.5
-1.3
-0.8
-0.4
-0.3
-0.1
-0.1
0.0
0.1
0.2
0.6
0.5
0.9
0.5
0.8
2.4
14
22
5
6
10
2
17
4
15
16
9
1
18
19
3
20
7
11
21
Laboratory

Figure 21

Table 28

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFTeDA
Units	ug/kg

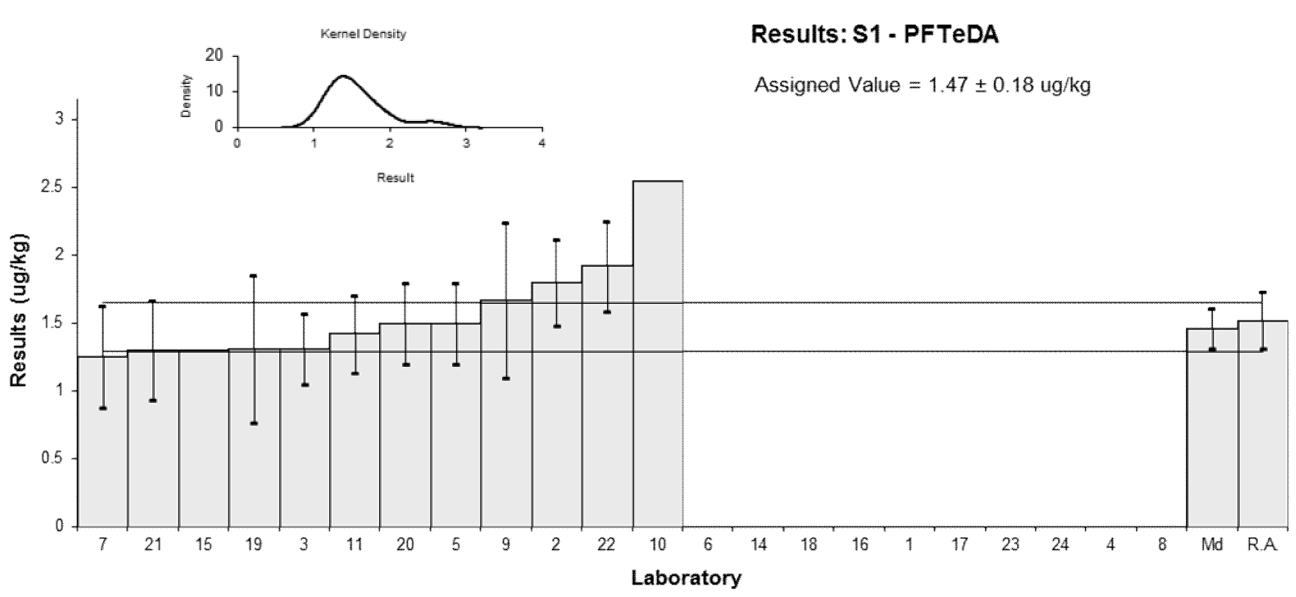
Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	<5	NR	95		
2	1.80	0.32	30	1.12	0.90
3	1.31	0.262	95	-0.54	-0.50
4	NT	NT	NT		
5	1.50	0.30	NR	0.10	0.09
6	< 5.0	1.9	116		
7	1.25	0.375	122	-0.75	-0.53
8	NT	NT	NT		
9	1.67	0.57	75.3	0.68	0.33
10	2.5493	NR	5242.1	3.67	6.00
11	1.42	0.284	95	-0.17	-0.15
14	<0.5	0.1	71		
15	1.3	NR	68	-0.58	-0.94
16	<2.5	NR	72		
17	NR	NR	16.1		
18	<2	NR	NR		
19	1.31	0.54	99.5	-0.54	-0.28
20	1.5	0.3	20	0.10	0.09
21	1.299	0.363	NR	-0.58	-0.42
22	1.92	0.33	93.2	1.53	1.20
23	NT	NT	NT		
24	NT	NT	NT		

Statistics

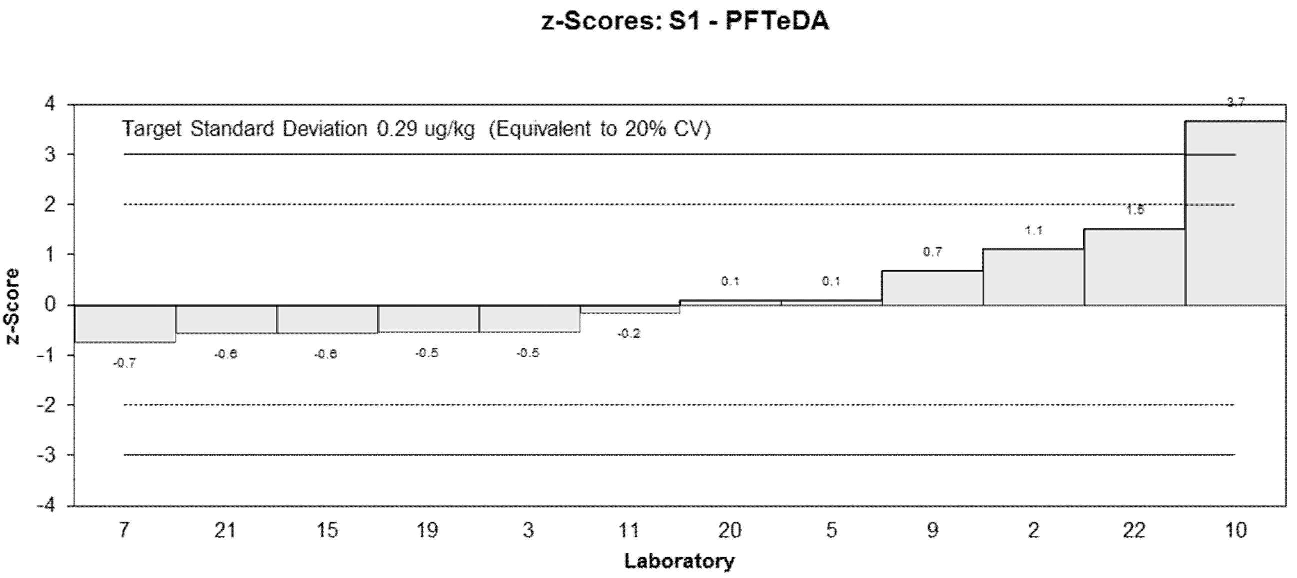
Assigned Value*	1.47	0.18
Spike	Not Spiked	
Robust Average	1.52	0.21
Median	1.46	0.15
Mean	1.57	
N	12	
Max.	2.5493	
Min.	1.25	
Robust SD	0.24	
Robust CV	16%	

*Robust average excluding laboratory 10.



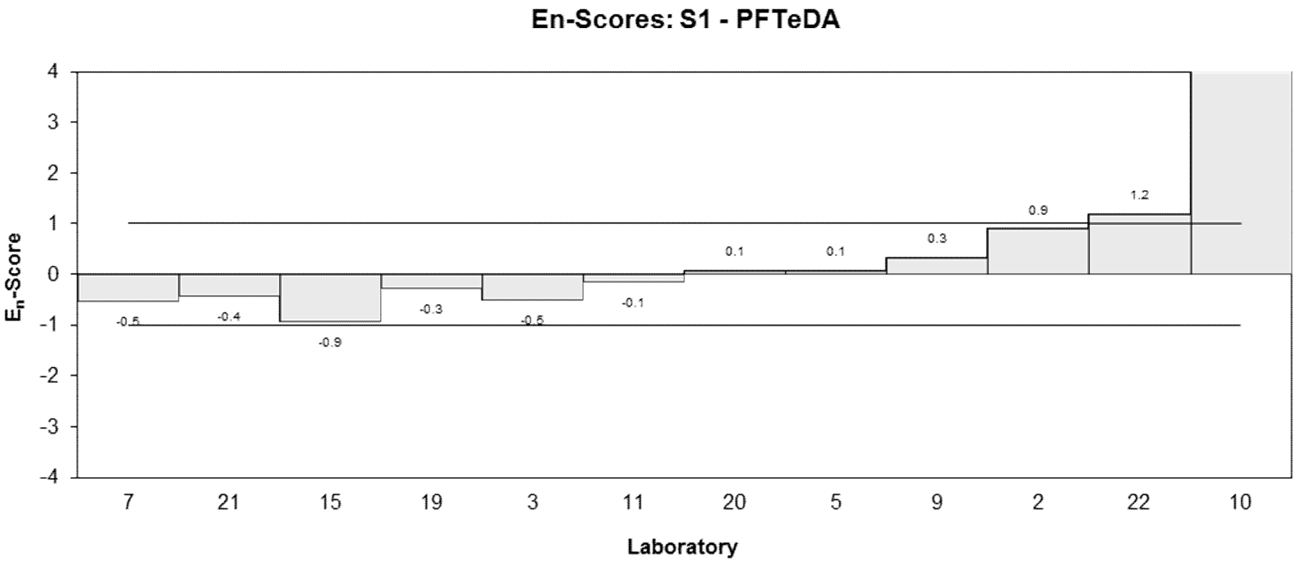
Results: S1 - PFTeDA

Assigned Value = 1.47 ± 0.18 ug/kg



z-Scores: S1 - PFTeDA

Target Standard Deviation 0.29 ug/kg (Equivalent to 20% CV)



En-Scores: S1 - PFTeDA

Figure 22

Table 29

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFTrA
Units	ug/kg

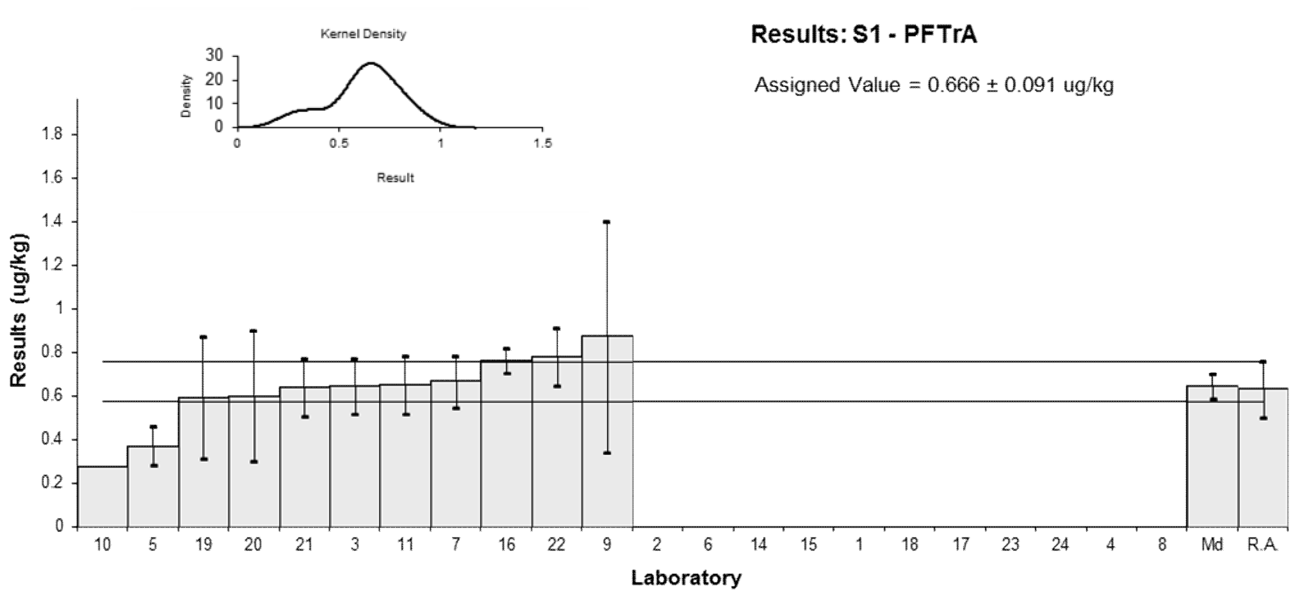
Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	<2	NR	95		
2	< 1.00	NR	104		
3	0.644	0.129	115	-0.17	-0.14
4	NT	NT	NT		
5	0.37	0.09	NR	-2.22	-2.31
6	< 2.0	0.96	NR		
7	0.665	0.120	122	-0.01	-0.01
8	NT	NT	NT		
9	0.871	0.530	75.3	1.54	0.38
10	0.2766	NR	5242.1	-2.92	-4.28
11	0.65	0.130	115	-0.12	-0.10
14	<0.5	0.1	NR		
15	<1	NR	NR		
16	0.76	0.056	95	0.71	0.88
17	NR	NR	NR		
18	<2	NR	NR		
19	0.59	0.28	99.5	-0.57	-0.26
20	0.6	0.3	30	-0.50	-0.21
21	0.637	0.132	NR	-0.22	-0.18
22	0.78	0.13	93.2	0.86	0.72
23	NT	NT	NT		
24	NT	NT	NT		

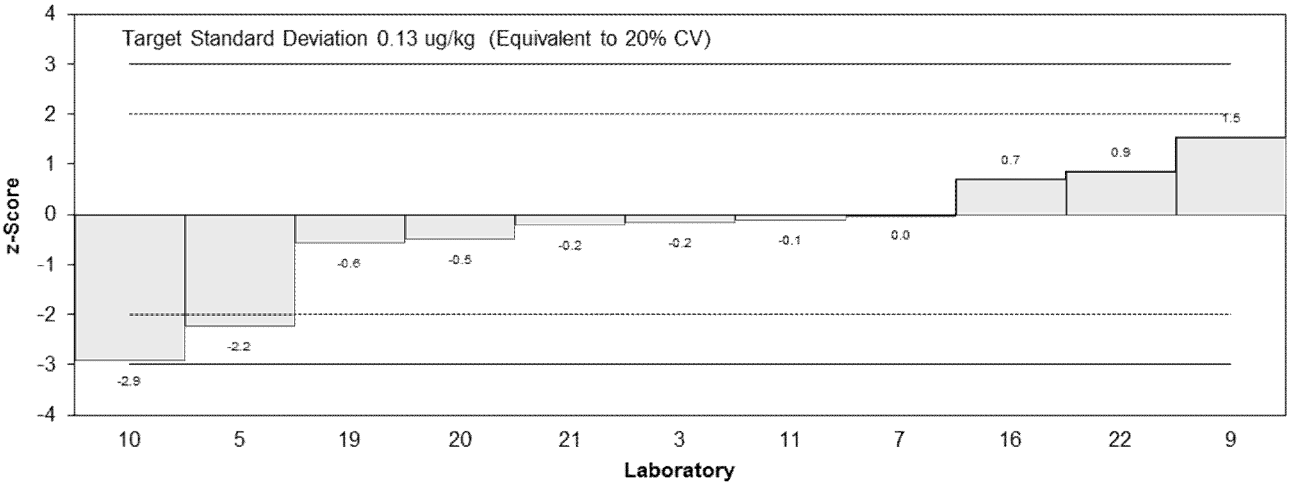
Statistics

Assigned Value*	0.666	0.091
Spike	Not Spiked	
Robust Average	0.63	0.13
Median	0.644	0.054
Mean	0.622	
N	11	
Max.	0.871	
Min.	0.2766	
Robust SD	0.120	
Robust CV	18%	

*Robust average excluding laboratory 10.



z-Scores: S1 - PFTrA



En-Scores: S1 - PFTrA

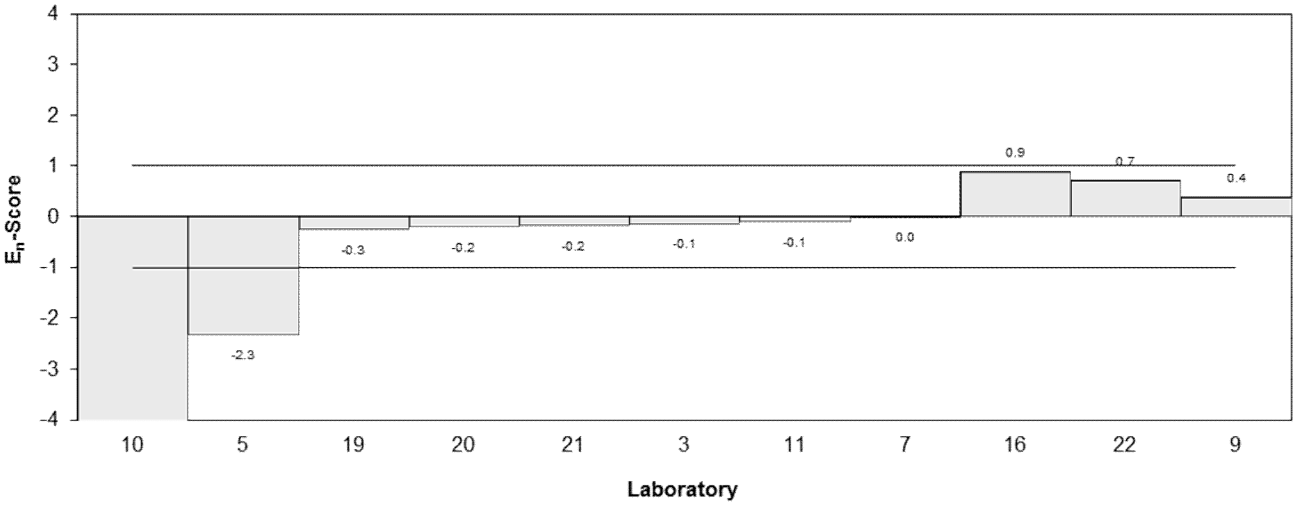


Figure 23

Table 30

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	PFUnA
Units	ug/kg

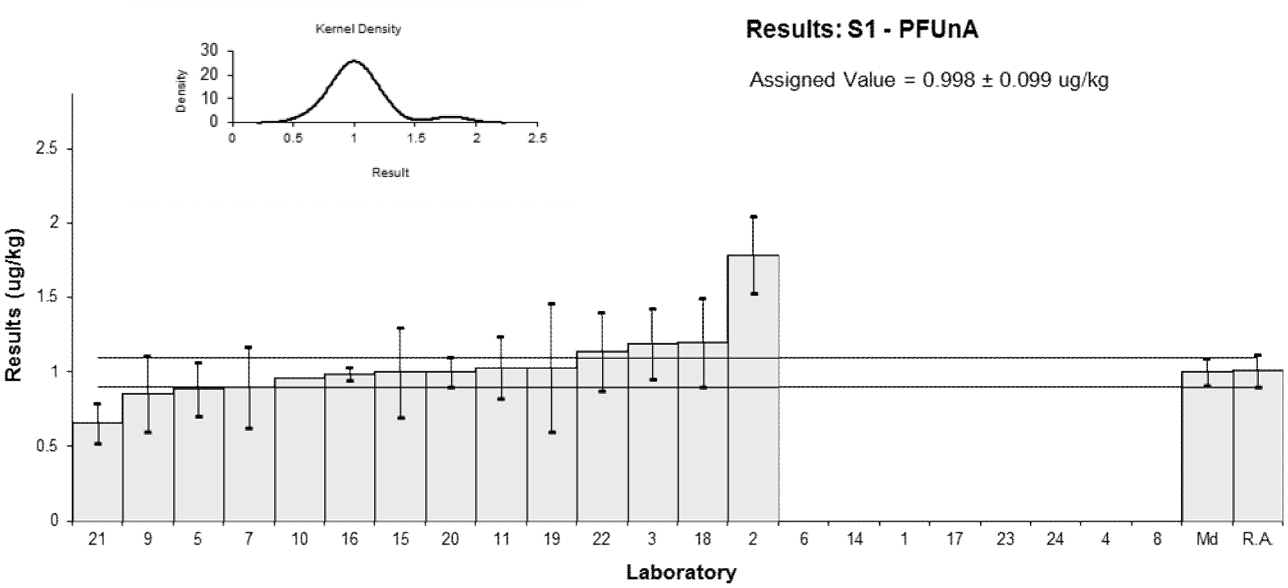
Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	<2	NR	173		
2	1.79	0.26	109	3.97	2.85
3	1.19	0.238	73	0.96	0.74
4	NT	NT	NT		
5	0.89	0.18	NR	-0.54	-0.53
6	< 2.0	0.50	116		
7	0.898	0.269	63	-0.50	-0.35
8	NT	NT	NT		
9	0.856	0.257	106	-0.71	-0.52
10	0.964	NR	257.9	-0.17	-0.34
11	1.03	0.206	73	0.16	0.14
14	<0.5	0.1	74		
15	1.0	0.3	31	0.01	0.01
16	0.99	0.047	87	-0.04	-0.07
17	NR	NR	NR		
18	1.2	0.3	NR	1.01	0.64
19	1.03	0.43	58.9	0.16	0.07
20	1.0	0.1	42	0.01	0.01
21	0.658	0.132	NR	-1.70	-2.06
22	1.14	0.26	93.2	0.71	0.51
23	NT	NT	NT		
24	NT	NT	NT		

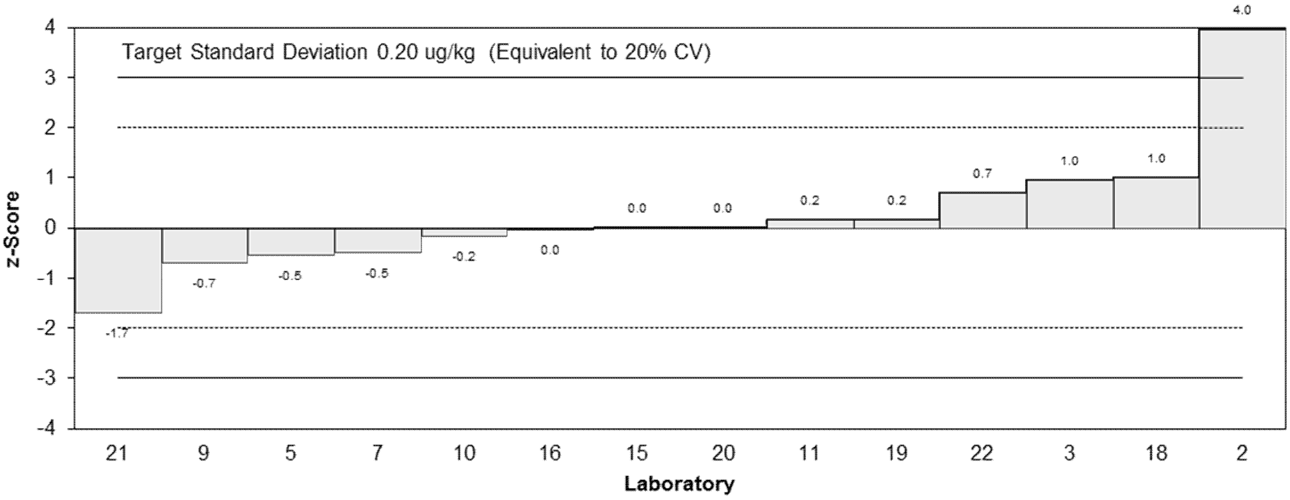
Statistics

Assigned Value*	0.998	0.099
Spike	Not Spiked	
Robust Average	1.01	0.11
Median	1.000	0.090
Mean	1.045	
N	14	
Max.	1.79	
Min.	0.658	
Robust SD	0.140	
Robust CV	14%	

*Robust average excluding laboratory 2.



z-Scores: S1 - PFUnA



En-Scores: S1 - PFUnA

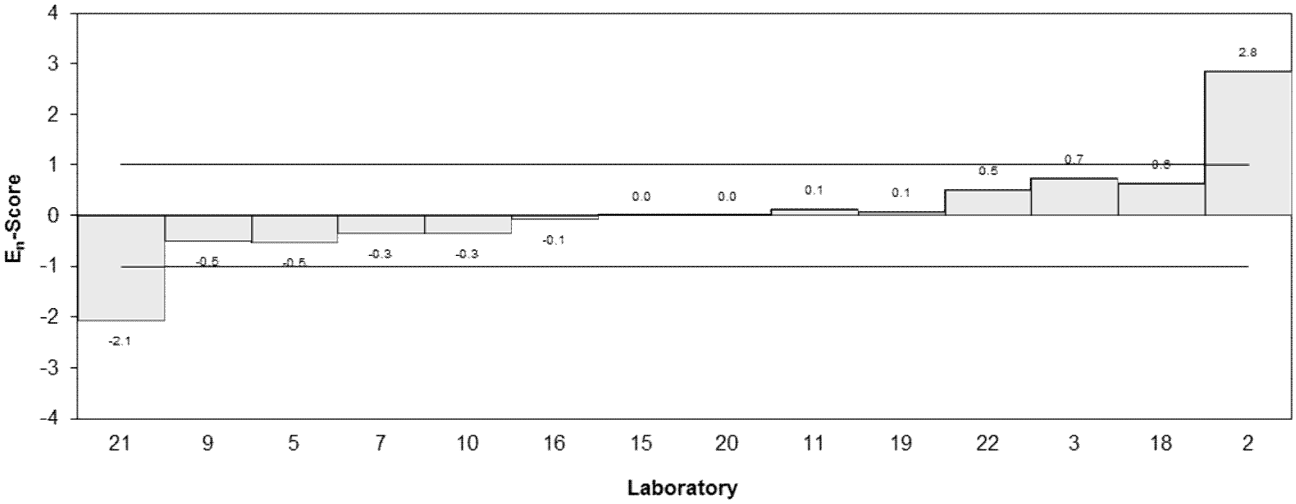


Figure 24

Table 31

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	6:2 FTS
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	9	4	113	0.00	0.00
2	10.5	2.4	155	0.83	0.56
3	8.85	1.77	83	-0.08	-0.07
4	NT	NT	NT		
5	10.1	2.0	NR	0.61	0.47
6	NT	NT	NT		
7	11.2	3.36	205	1.22	0.62
8	NT	NT	NT		
9	NT	NT	NT		
10	NT	NT	NT		
11	8.63	1.73	110	-0.21	-0.18
14*	3.1	0.62	87	-3.28	-4.37
15	8.9	3	71	-0.06	-0.03
16	9.6	0.70	105	0.33	0.43
17**	168	42	117	88.33	3.78
18	8.4	2.77	NR	-0.33	-0.20
19	NT	NT	NT		
20	<0.5	NR	138		
21	6.741	0.716	NR	-1.26	-1.62
22	6.63	NR	74	-1.32	-1.98
23	NT	NT	NT		
24	NT	NT	NT		

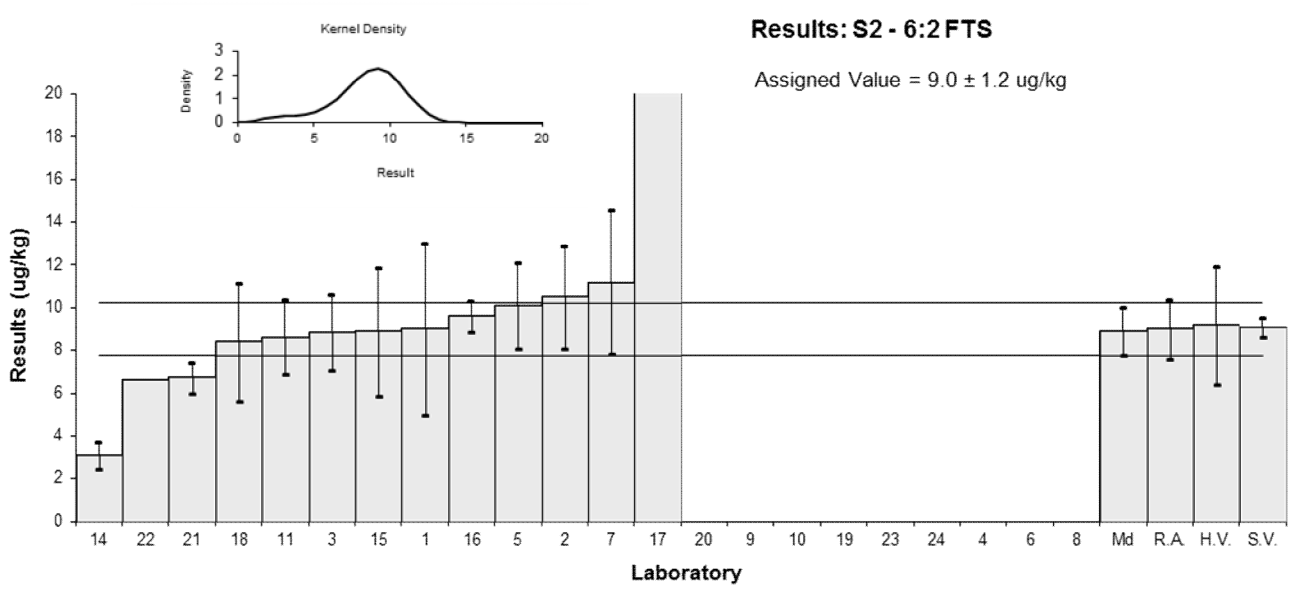
Statistics

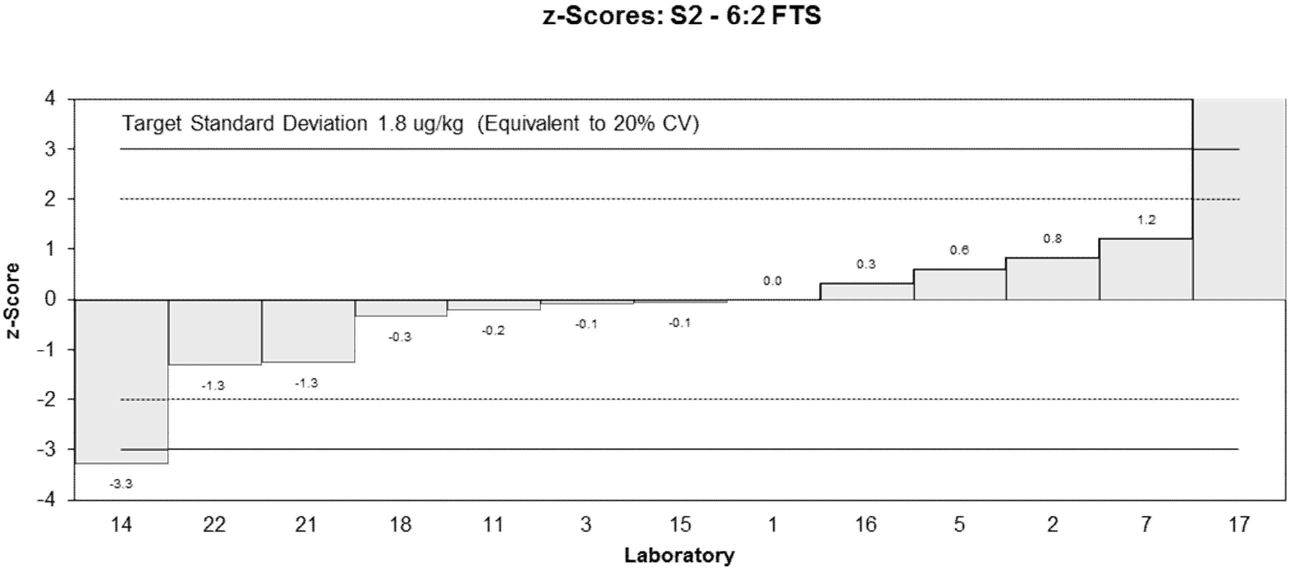
Assigned Value***	9.0	1.2
Spike	9.10	0.45
Homogeneity Value	9.2	2.8
Robust Average**	8.7	1.3
Median**	8.88	0.92
Mean**	8.5	
N	13	
Max.	168	
Min.	3.1	
Robust SD**	1.6	
Robust CV**	18%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Results from laboratory 17 were omitted from the statistical calculations.

***Robust average excluding laboratory 14.


Results: S2 - 6:2 FTS
Assigned Value = 9.0 ± 1.2 ug/kg
Kernel Density
Density
Result
Results (ug/kg)
Laboratory
14
22
21
18
11
3
15
1
16
5
2
7
17
20
9
10
19
23
24
4
6
8
Md
R.A.
H.V.
S.V.


z-Scores: S2 - 6:2 FTS
Target Standard Deviation 1.8 ug/kg (Equivalent to 20% CV)
z-Score
Laboratory
-3.3
-1.3
-1.3
-0.3
-0.2
-0.1
-0.1
0.0
0.3
0.6
0.8
1.2
14
22
21
18
11
3
15
1
16
5
2
7
17

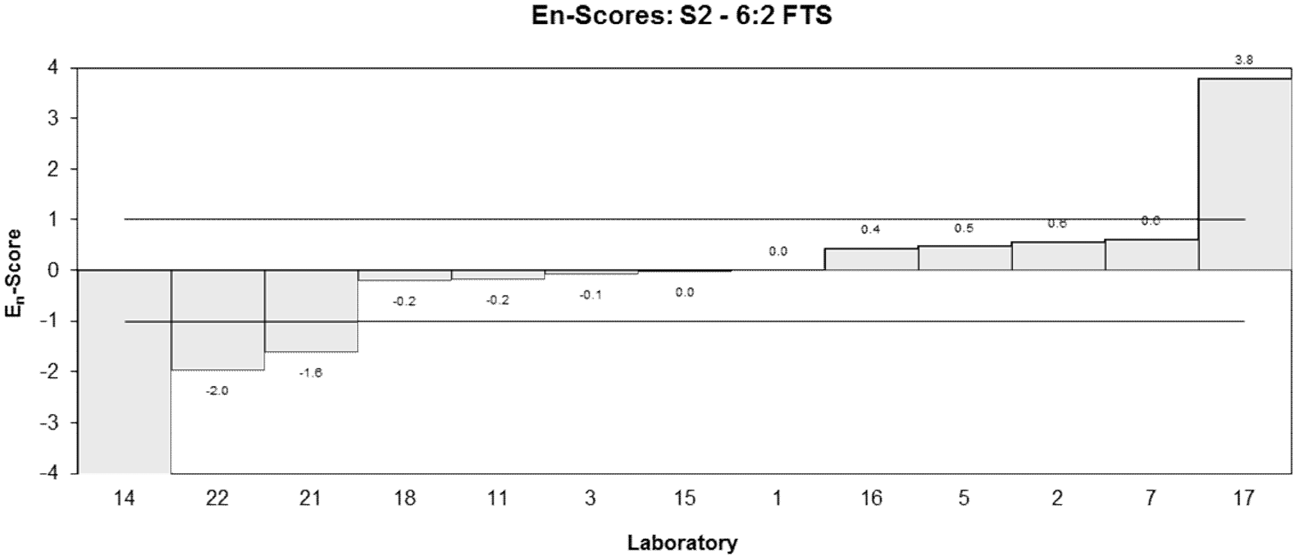

En-Scores: S2 - 6:2 FTS
En-Score
Laboratory
-2.0
-1.6
-0.2
-0.2
-0.1
0.0
0.0
0.4
0.5
0.6
0.6
3.8
14
22
21
18
11
3
15
1
16
5
2
7
17

Figure 25

Table 32

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	8:2 FTS
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	9	4	136	0.07	0.03
2	< 99.0	NR	149		
3	8.63	1.73	96	-0.14	-0.13
4	NT	NT	NT		
5	9.9	2.0	NR	0.58	0.50
6	8.3	2.1	NR	-0.32	-0.26
7	8.95	2.69	399	0.05	0.03
8	NT	NT	NT		
9	NT	NT	NT		
10	NT	NT	NT		
11	8.59	1.72	108	-0.16	-0.16
14*	3.3	0.84	90	-3.14	-5.67
15	9.3	3	58	0.24	0.14
16	10	1.7	83	0.64	0.64
17**	200	50	101	107.74	3.82
18	8.6	2.29	NR	-0.15	-0.12
19	NT	NT	NT		
20	8.0	0.9	105	-0.49	-0.84
21	9.058	0.955	NR	0.11	0.17
22	8.14	NR	74	-0.41	-1.43
23	NT	NT	NT		
24	NT	NT	NT		

Statistics

Assigned Value***	8.87	0.51
Spike	9.19	0.46
Homogeneity Value	10.1	3.0
Robust Average**	8.77	0.55
Median**	8.63	0.38
Mean**	8.44	
N	14	
Max.	200	
Min.	3.3	
Robust SD**	0.70	
Robust CV**	7.9%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Results from laboratory 17 were omitted from the statistical calculations.

***Robust average excluding laboratory 14.

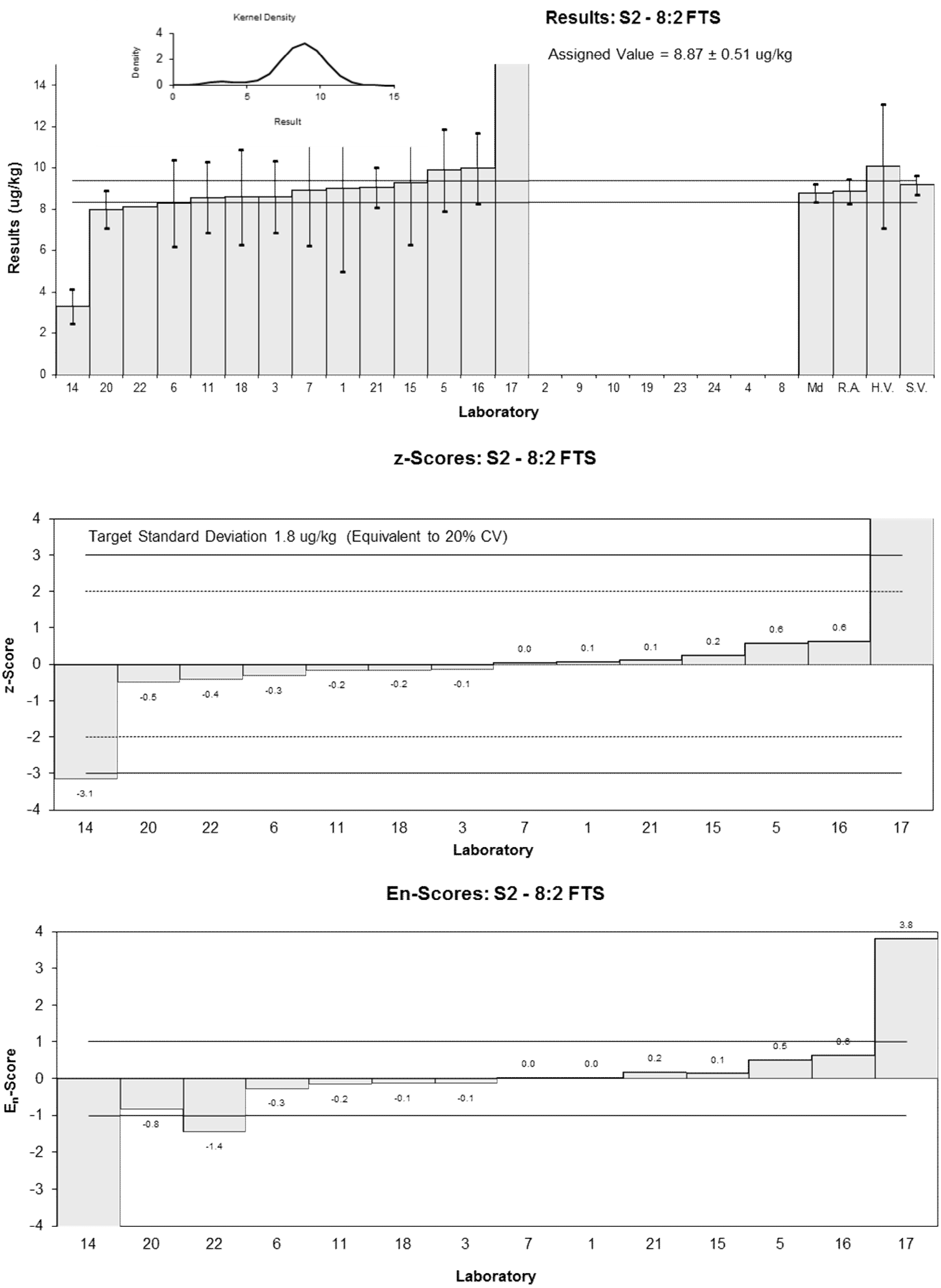

Kernel Density
Density
Result
Results: S2 - 8:2 FTS
Assigned Value = 8.87 ± 0.51 ug/kg
Results (ug/kg)
Laboratory
14 20 22 6 11 18 3 7 1 21 15 5 16 17 2 9 10 19 23 24 4 8 Md R.A. H.V. S.V.
z-Scores: S2 - 8:2 FTS
Target Standard Deviation 1.8 ug/kg (Equivalent to 20% CV)
z-Score
-3.1
-0.5
-0.4
-0.3
-0.2
-0.2
-0.1
0.0
0.1
0.1
0.2
0.6
0.6
14 20 22 6 11 18 3 7 1 21 15 5 16 17
Laboratory
En-Scores: S2 - 8:2 FTS
En-Score
-0.8
-1.4
-0.3
-0.2
-0.1
-0.1
0.0
0.0
0.2
0.1
0.5
0.6
3.8
14 20 22 6 11 18 3 7 1 21 15 5 16 17
Laboratory

Figure 26

Table 33

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	ADONA
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	NT	NT	NT		
2	31.5	7.3	102	1.06	0.61
3	24.0	4.80	81	-0.38	-0.28
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	NT	NT	NT		
8	NT	NT	NT		
9	NT	NT	NT		
10	NT	NT	NT		
11	22.9	4.59	75	-0.60	-0.45
14*	9.1	2.3	NR	-3.25	-2.97
15	NT	NT	NT		
16	NT	NT	NT		
17**	411	102.75	NT	74.04	3.74
18	NT	NT	NT		
19	NT	NT	NT		
20	NT	NT	NT		
21	29.140	2.959	NR	0.60	0.52
22	22.4	NR	74	-0.69	-0.69
23	NT	NT	NT		
24	NT	NT	NT		

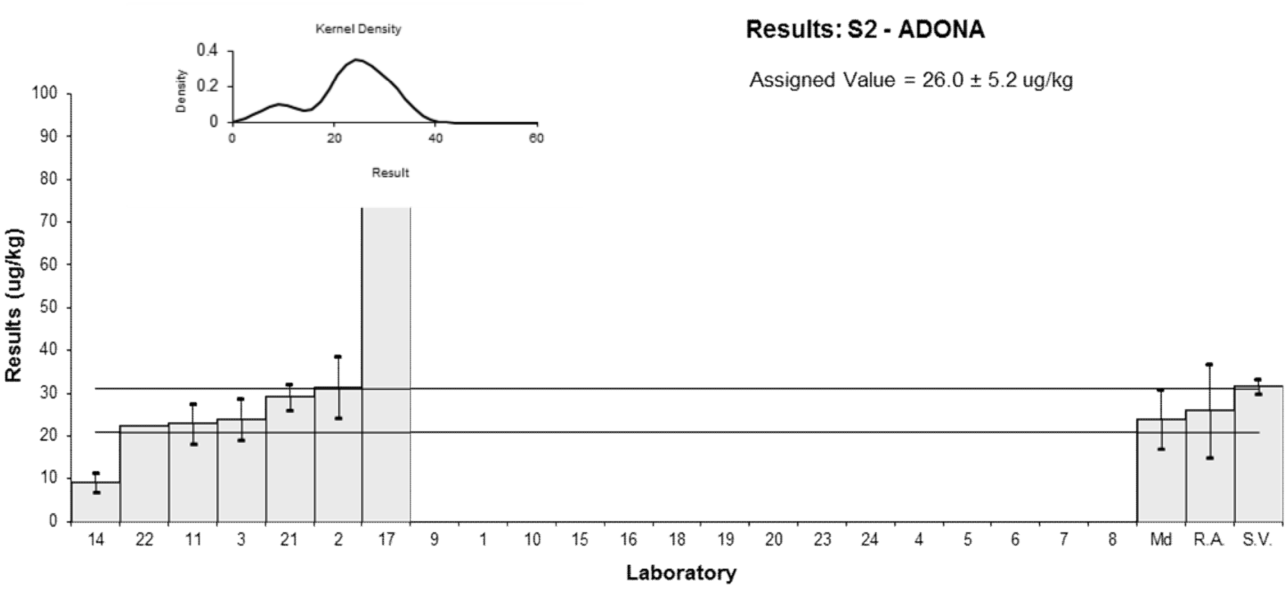
Statistics

Assigned Value***	26.0	5.2
Spike	31.6	1.6
Robust Average**	23.7	7.6
Median**	23.5	5.2
Mean**	23.2	
N	7	
Max.	411	
Min.	9.1	
Robust SD**	4.6	
Robust CV**	18%	

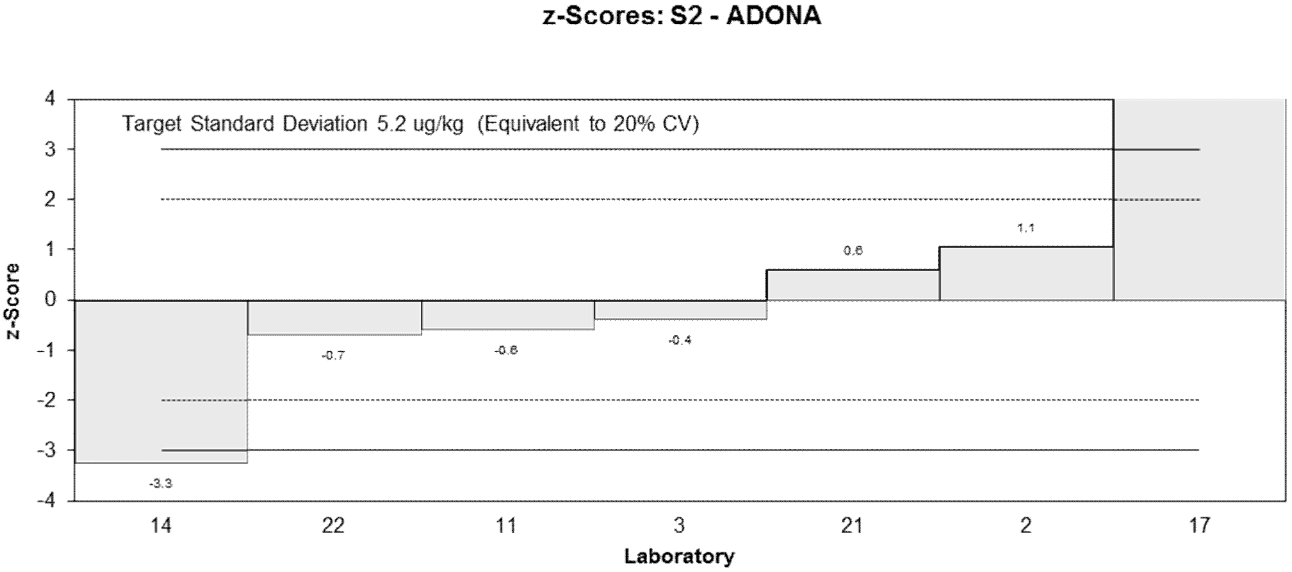
*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Results from laboratory 17 were omitted from the statistical calculations.

***Robust average excluding laboratory 14.


Results: S2 - ADONA

Assigned Value = 26.0 ± 5.2 ug/kg


z-Scores: S2 - ADONA

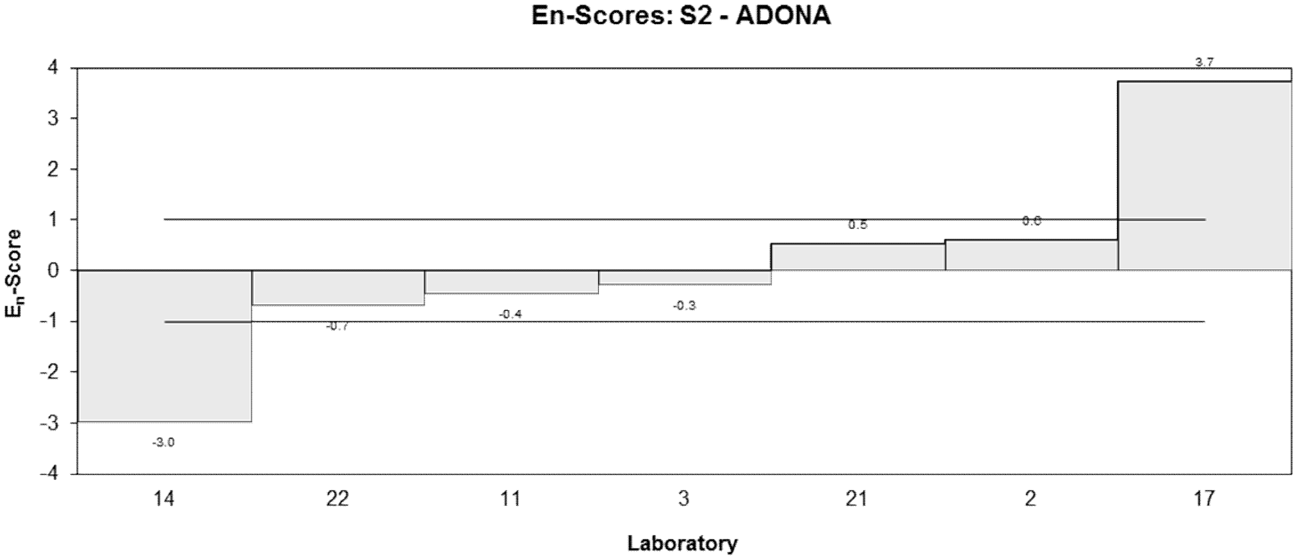

En-Scores: S2 - ADONA

Figure 27

Table 34

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	GenX
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	NT	NT	NT		
2	27.4	11	62	1.46	0.51
3	19.4	3.89	81	-0.42	-0.29
4	NT	NT	NT		
5	NT	NT	NT		
6	NT	NT	NT		
7	NT	NT	NT		
8	NT	NT	NT		
9	NT	NT	NT		
10	NT	NT	NT		
11	19.5	3.91	86	-0.40	-0.27
14*	7.0	1.8	95	-3.35	-2.72
15	NT	NT	NT		
16	NT	NT	NT		
17**	488	122	115	110.09	3.82
18	NT	NT	NT		
19	NT	NT	NT		
20	NT	NT	NT		
21	22.068	2.225	NR	0.20	0.16
22	17.5	NR	74	-0.87	-0.76
23	NT	NT	NT		
24	NT	NT	NT		

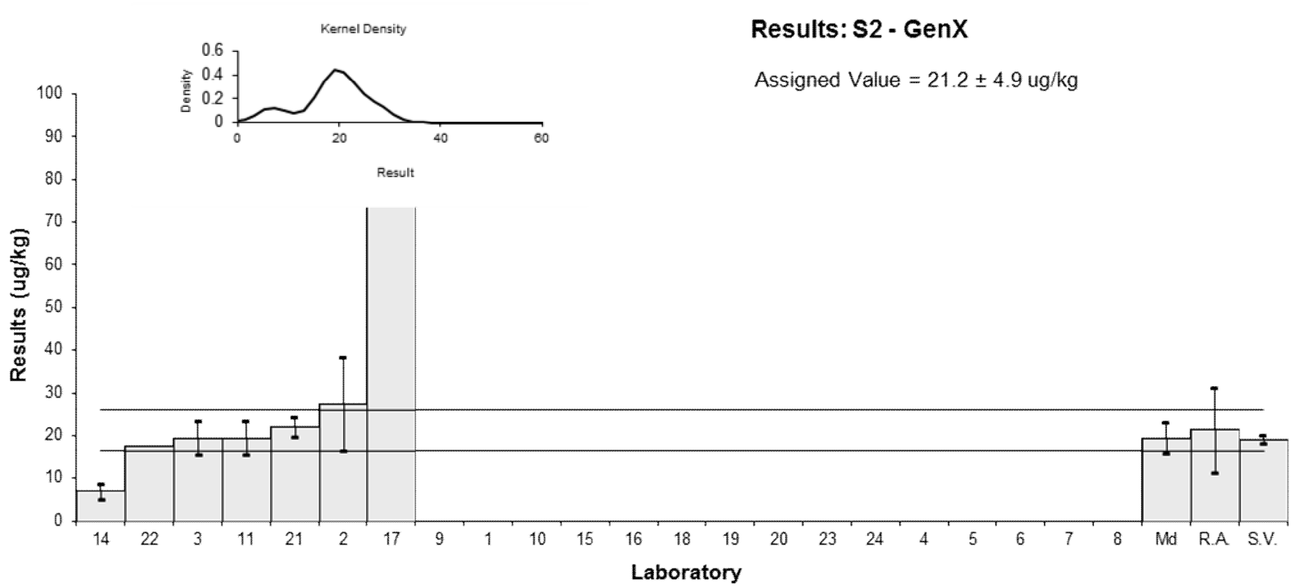
Statistics

Assigned Value***	21.2	4.9
Spike	19.2	1.0
Robust Average**	19.1	7.2
Median**	19.5	3.6
Mean**	18.8	
N	7	
Max.	488	
Min.	7	
Robust SD**	4.4	
Robust CV**	21%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Results from laboratory 17 were omitted from the statistical calculations.

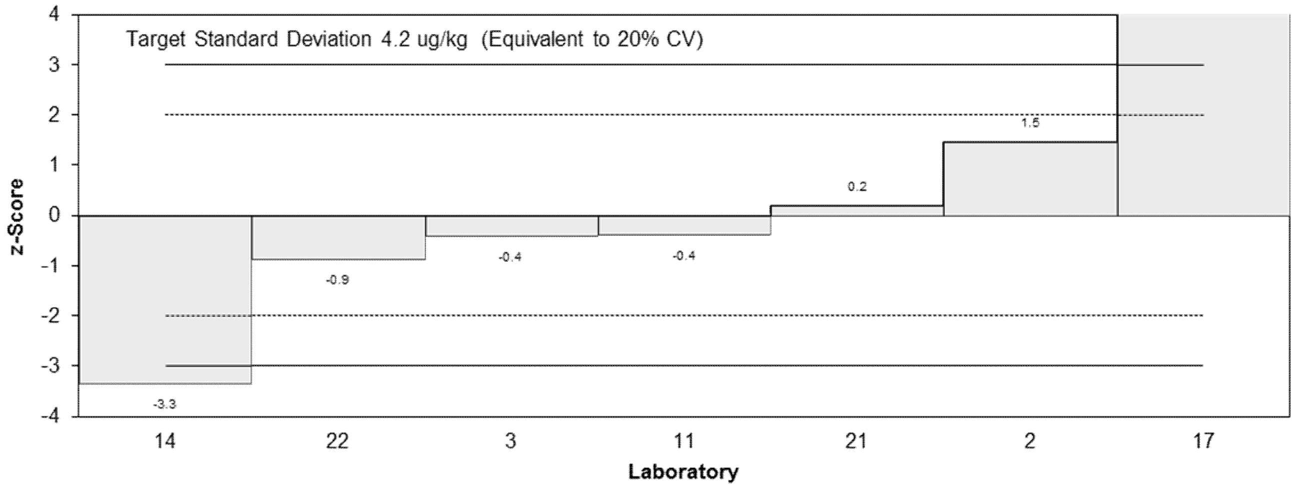
***Robust average excluding laboratory 14.



Results: S2 - GenX

Assigned Value = 21.2 ± 4.9 ug/kg

z-Scores: S2 - GenX



En-Scores: S2 - GenX

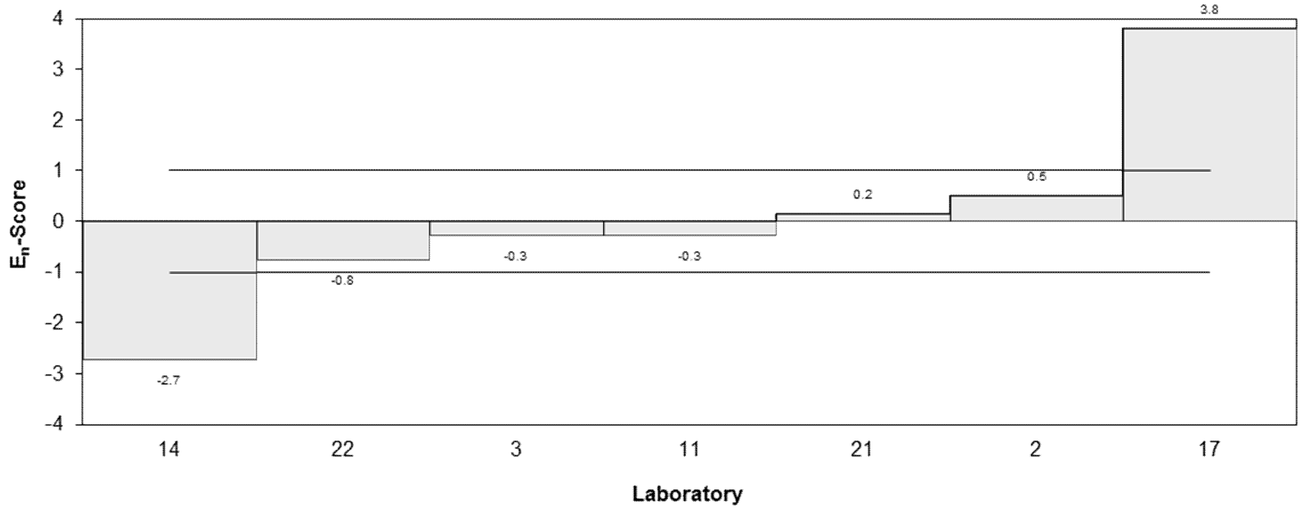


Figure 28

Table 35

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	PFBA
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	48	10	99	-0.17	-0.14
2	57.9	8.6	61	0.82	0.77
3	38.0	7.60	88	-1.18	-1.19
4	NT	NT	NT		
5	63.8	16	NR	1.42	0.82
6	48	12	128	-0.17	-0.13
7	77.4	23.2	46	2.79	1.15
8	NT	NT	NT		
9	NT	NT	NT		
10	NT	NT	NT		
11	37.4	7.49	73	-1.24	-1.27
14*	18	2.6	90	-3.19	-4.72
15	47	10	73	-0.27	-0.23
16	45	1.3	89	-0.47	-0.74
17**	933	233.25	88.7	88.86	3.79
18	45.6	30.99	NR	-0.41	-0.13
19	56	14	8.3	0.63	0.41
20	43.4	1.2	60	-0.63	-1.00
21	51.849	5.474	NR	0.22	0.26
22	50	NR	74	0.03	0.05
23	NT	NT	NT		
24	67	13	60	1.74	1.20

Statistics

Assigned Value***	49.7	6.2
Spike	50.0	2.5
Homogeneity Value	47	14
Robust Average**	49.9	7.3
Median**	48.0	5.0
Mean**	49.6	
N	17	
Max.	933	
Min.	18	
Robust SD**	9.3	
Robust CV**	19%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Results from laboratory 17 were omitted from the statistical calculations.

***Robust average excluding laboratories 7 and 14.

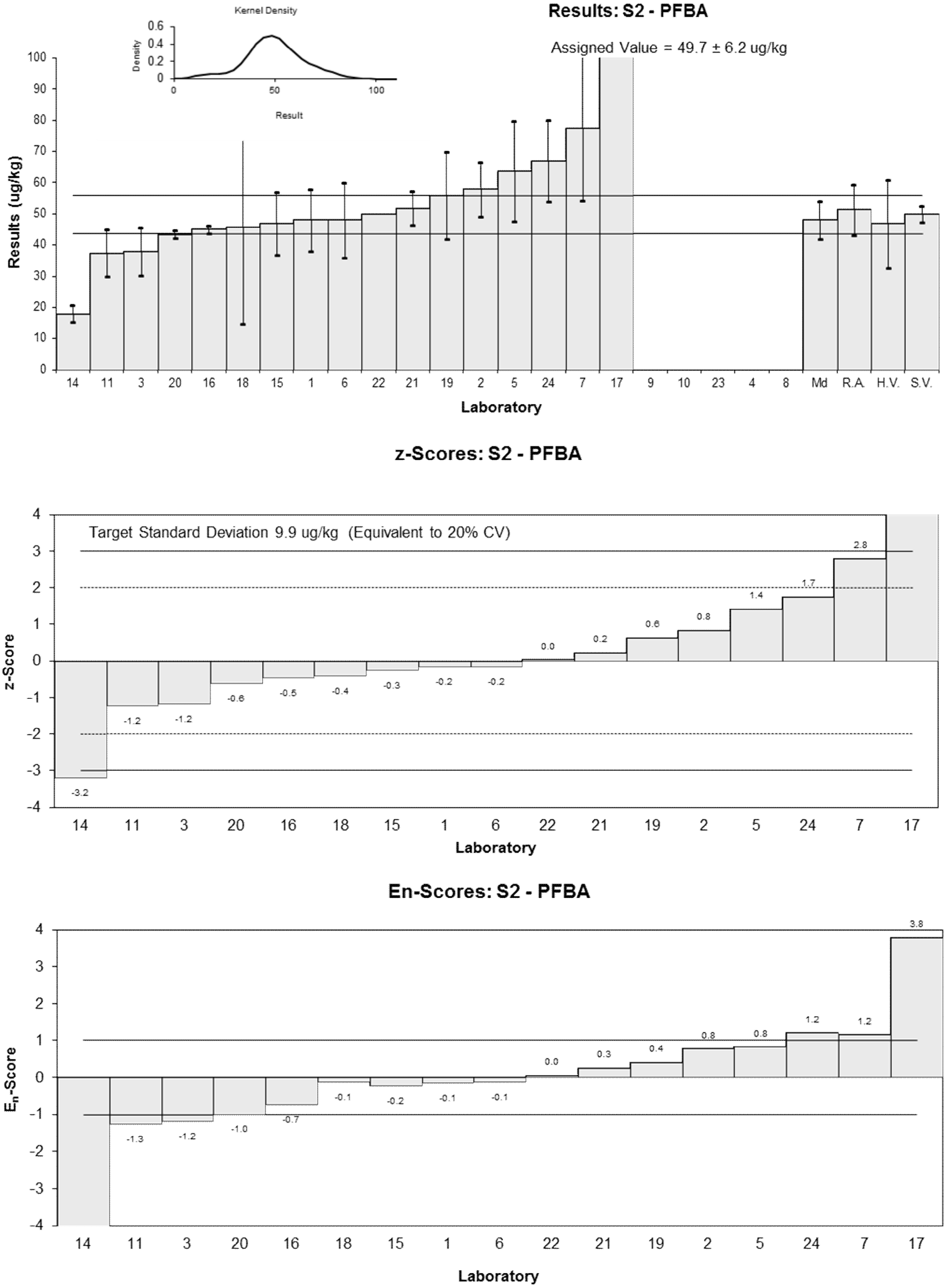

Results: S2 - PFBA
Assigned Value = 49.7 ± 6.2 ug/kg
Kernel Density
Density
Result
0.6
0.4
0.2
0
0
50
100
Results (ug/kg)
100
90
80
70
60
50
40
30
20
10
0
14
11
3
20
16
18
15
1
6
22
21
19
2
5
24
7
17
9
10
23
4
8
Md
R.A.
H.V.
S.V.
Laboratory
z-Scores: S2 - PFBA
Target Standard Deviation 9.9 ug/kg (Equivalent to 20% CV)
z-Score
4
3
2
1
0
-1
-2
-3
-4
-3.2
-1.2
-1.2
-0.6
-0.5
-0.4
-0.3
-0.2
-0.2
0.0
0.2
0.6
0.8
1.4
1.7
2.8
14
11
3
20
16
18
15
1
6
22
21
19
2
5
24
7
17
Laboratory
En-Scores: S2 - PFBA
E_n-Score
4
3
2
1
0
-1
-2
-3
-4
-1.3
-1.2
-1.0
-0.7
-0.1
-0.2
-0.1
-0.1
0.0
0.3
0.4
0.8
0.8
1.2
1.2
3.8
14
11
3
20
16
18
15
1
6
22
21
19
2
5
24
7
17
Laboratory

Figure 29

Table 36

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	PFBS
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	24	7	95	-0.06	-0.04
2	33.4	11	106	1.87	0.81
3	20.1	4.03	94	-0.86	-0.91
4	NT	NT	NT		
5	25.5	6.4	NR	0.25	0.18
6	25	6.3	114	0.14	0.10
7	36.9	11.07	60	2.59	1.11
8	NT	NT	NT		
9	NT	NT	NT		
10	NT	NT	NT		
11	18.8	3.76	71	-1.13	-1.25
14*	8.8	1.7	114	-3.19	-5.42
15	23	7	68	-0.27	-0.18
16	25	2.9	89	0.14	0.19
17**	478	119.5	NT	93.35	3.80
18	26.6	4.86	NR	0.47	0.43
19	23.4	6.1	13.5	-0.19	-0.14
20	22.2	1.1	83	-0.43	-0.82
21	23.747	2.843	NR	-0.11	-0.15
22	20.5	NR	74	-0.78	-1.65
23	NT	NT	NT		
24	27	5	84	0.56	0.49

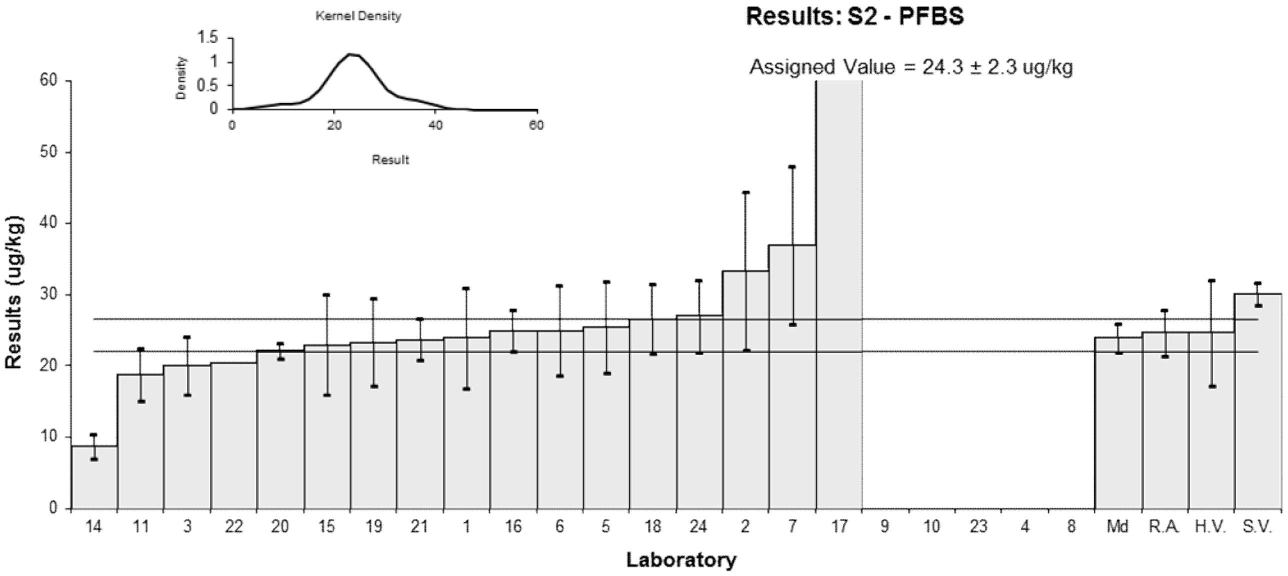
Statistics

Assigned Value***	24.3	2.3
Spike	30.1	1.5
Homogeneity Value	24.7	7.4
Robust Average**	23.9	2.5
Median**	23.9	1.7
Mean**	24.0	
N	17	
Max.	478	
Min.	8.8	
Robust SD**	3.6	
Robust CV**	15%	

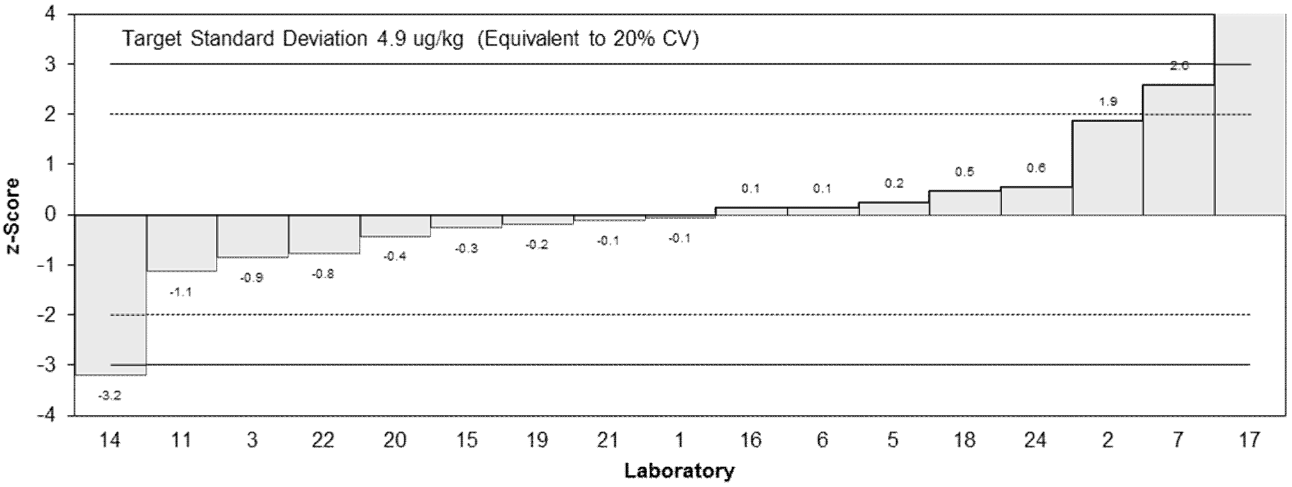
*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Results from laboratory 17 were omitted from the statistical calculations.

***Robust average excluding laboratory 14.



z-Scores: S2 - PFBS



En-Scores: S2 - PFBS

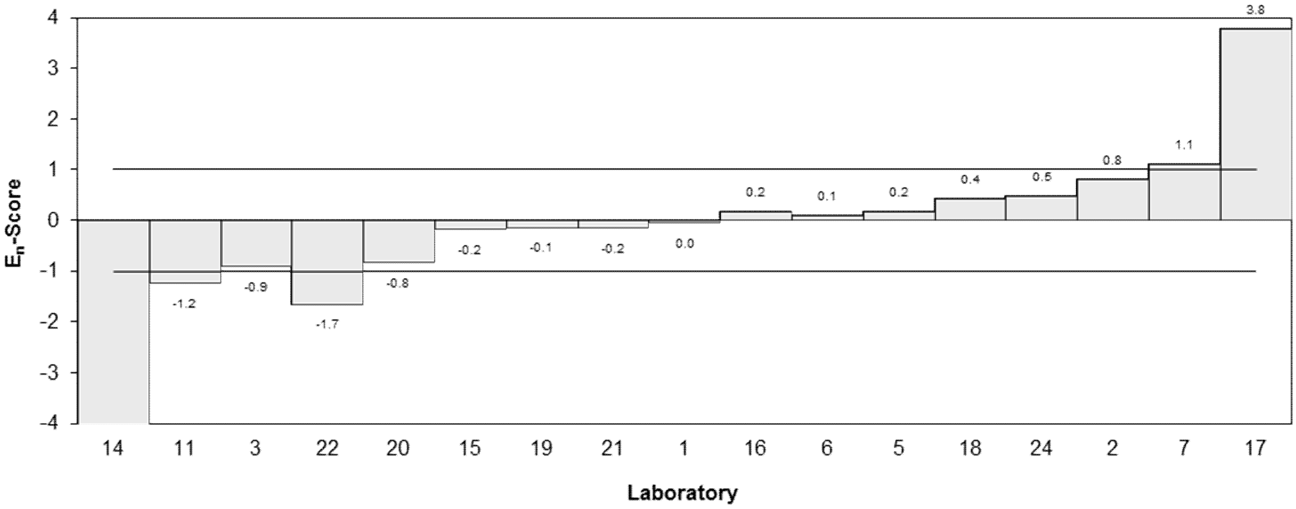


Figure 30

Table 37

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	PFHpA
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	12	4	106	-0.04	-0.02
2	14.3	2.0	94	0.91	0.98
3	11.1	2.22	78	-0.41	-0.41
4	NT	NT	NT		
5	12.3	2.5	NR	0.08	0.07
6	13	3.3	122	0.37	0.26
7	18.9	5.67	78	2.81	1.18
8	NT	NT	NT		
9	NT	NT	NT		
10	NT	NT	NT		
11	10.8	2.17	83	-0.54	-0.54
14*	4.4	0.80	117	-3.18	-6.01
15	12	4	81	-0.04	-0.02
16	13	1.7	88	0.37	0.46
17**	244	61	132	95.83	3.80
18	12.2	2.35	NR	0.04	0.04
19	13.4	3.8	12.6	0.54	0.33
20	13.9	1.7	88	0.74	0.91
21	10.968	1.574	NR	-0.47	-0.61
22	10.3	NR	74	-0.74	-1.80
23	NT	NT	NT		
24	8.6	1.7	79	-1.45	-1.77

Statistics

Assigned Value***	12.1	1.0
Spike	13.0	0.7
Homogeneity Value	12.1	3.6
Robust Average**	12.0	1.2
Median**	12.10	0.96
Mean**	11.9	
N	17	
Max.	244	
Min.	4.4	
Robust SD**	1.5	
Robust CV**	12%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Results from laboratory 17 were omitted from the statistical calculations.

***Robust average excluding laboratories 7 and 14.

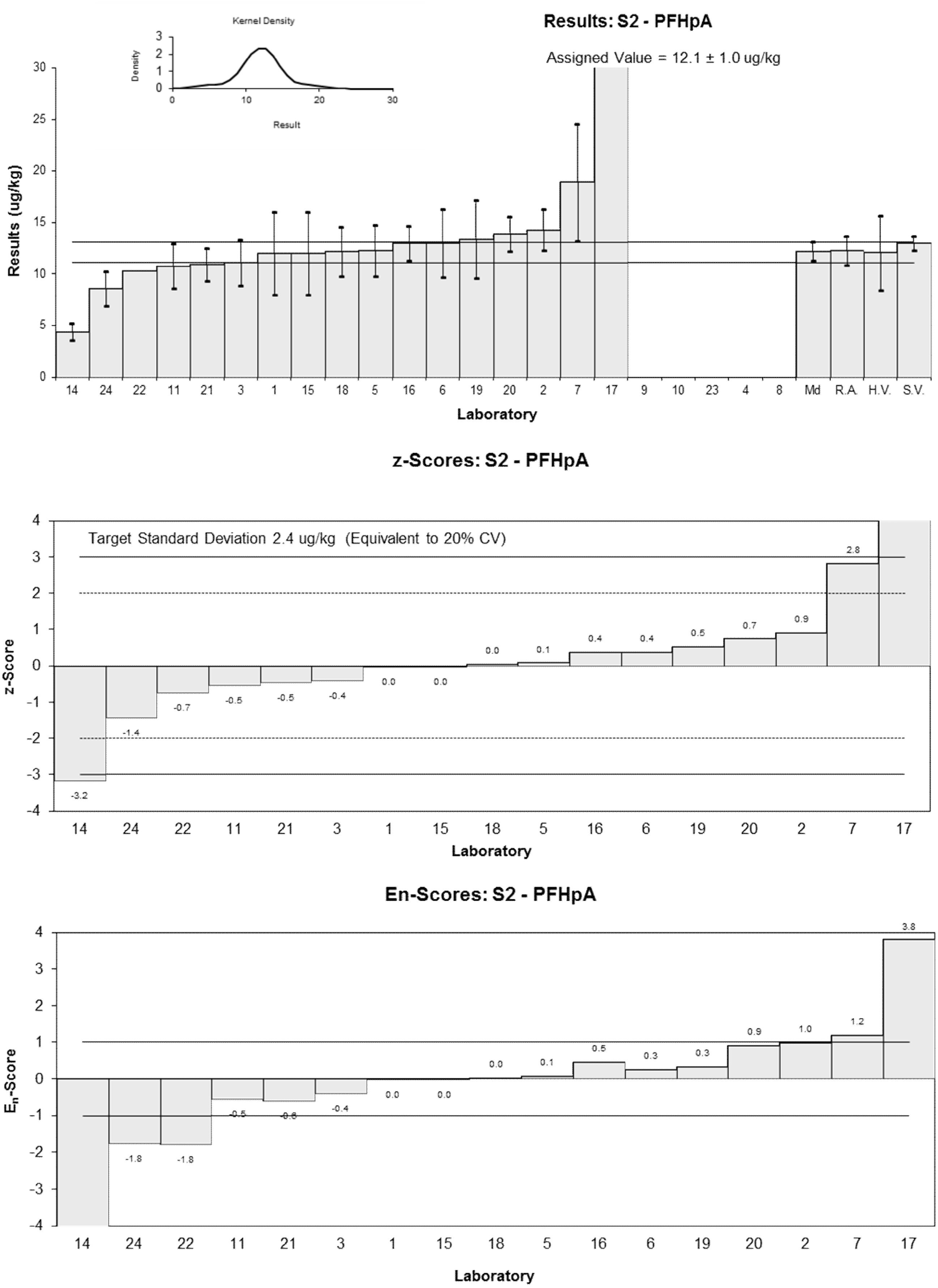

Results: S2 - PFHpA
Assigned Value = 12.1 ± 1.0 ug/kg
Kernel Density
Density
Result
Results (ug/kg)
Laboratory
14 24 22 11 21 3 1 15 18 5 16 6 19 20 2 7 17 9 10 23 4 8 Md R.A. H.V. S.V.
z-Scores: S2 - PFHpA
Target Standard Deviation 2.4 ug/kg (Equivalent to 20% CV)
z-Score
-3.2 -1.4 -0.7 -0.5 -0.5 -0.4 0.0 0.0 0.0 0.1 0.4 0.4 0.5 0.7 0.9 2.8
14 24 22 11 21 3 1 15 18 5 16 6 19 20 2 7 17
Laboratory
En-Scores: S2 - PFHpA
En-Score
-1.8 -1.8 -0.5 -0.6 -0.4 0.0 0.0 0.0 0.1 0.5 0.3 0.3 0.9 1.0 1.2 3.8
14 24 22 11 21 3 1 15 18 5 16 6 19 20 2 7 17
Laboratory

Figure 31

Table 38

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	PFHxA
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	25	8	101	-0.08	-0.05
2	28.7	4.1	85	0.65	0.76
3	24.0	4.80	79	-0.28	-0.28
4	NT	NT	NT		
5	24.8	6.0	NR	-0.12	-0.10
6	27	6.8	118	0.31	0.23
7	34.6	10.4	69	1.81	0.88
8	NT	NT	NT		
9	NT	NT	NT		
10	NT	NT	NT		
11	23.6	4.73	90	-0.35	-0.36
14*	9.5	1.8	111	-3.13	-6.79
15	24	7	55	-0.28	-0.20
16	26	3.9	88	0.12	0.14
17**	529	132.25	87.1	99.13	3.81
18	25.0	4.60	NR	-0.08	-0.08
19	26.1	7.3	13.4	0.14	0.09
20	27.0	4.0	88	0.31	0.37
21	22.523	3.222	NR	-0.57	-0.81
22	26.1	NR	74	0.14	0.47
23	NT	NT	NT		
24	18	4	79	-1.46	-1.73

Statistics

Assigned Value***	25.4	1.5
Spike	25.0	1.2
Homogeneity Value	26.2	7.9
Robust Average**	25.1	1.7
Median**	25.0	1.0
Mean**	24.5	
N	17	
Max.	529	
Min.	9.5	
Robust SD**	2.3	
Robust CV**	9.1%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Results from laboratory 17 were omitted from the statistical calculations.

***Robust average excluding laboratory 14.

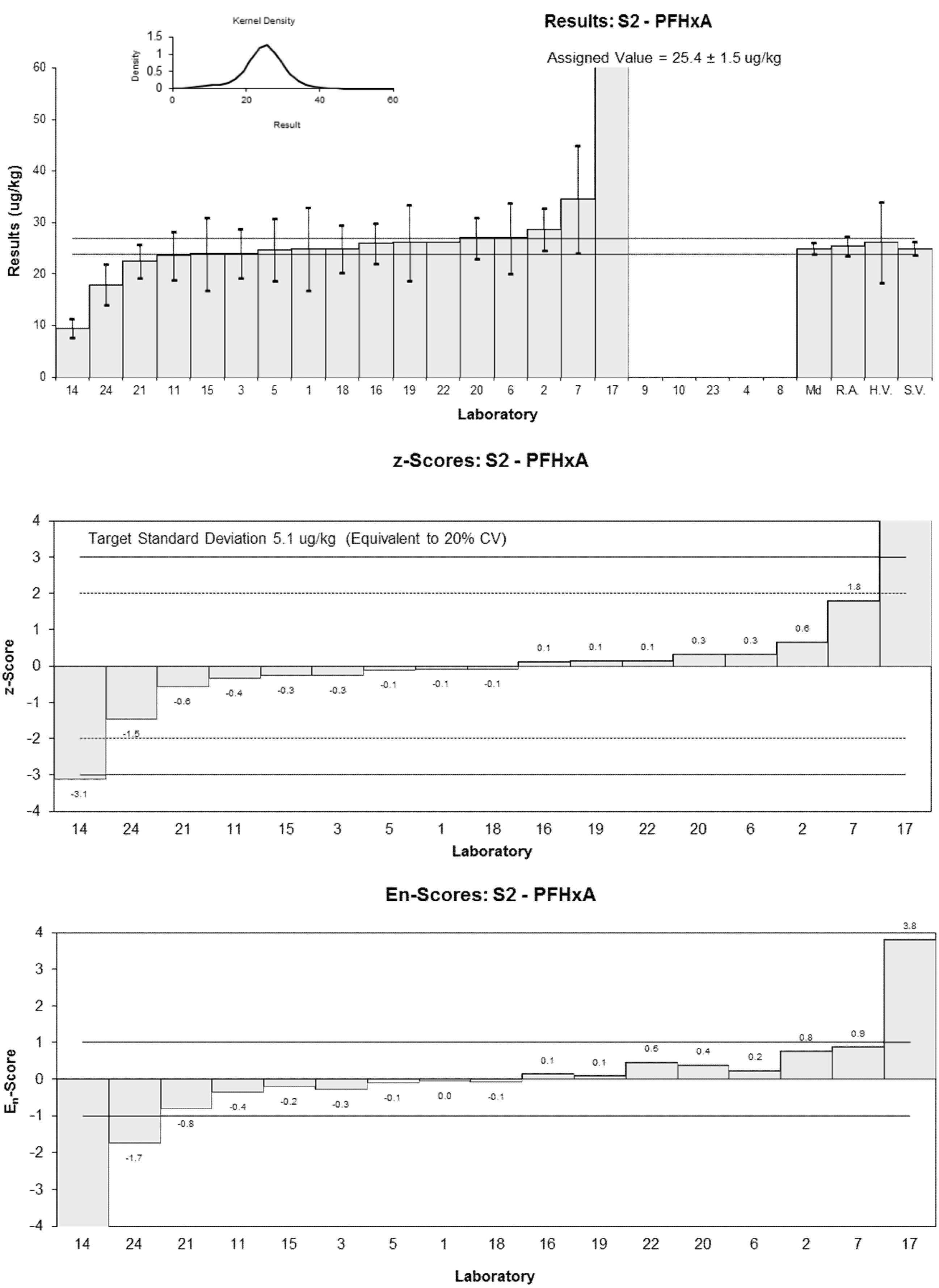

Results: S2 - PFHxA
Assigned Value = 25.4 ± 1.5 ug/kg
Kernel Density
Density
1.5
1
0.5
0
0
20
40
60
Result
Results (ug/kg)
60
50
40
30
20
10
0
14
24
21
11
15
3
5
1
18
16
19
22
20
6
2
7
17
9
10
23
4
8
Md
R.A.
H.V.
S.V.
Laboratory
z-Scores: S2 - PFHxA
Target Standard Deviation 5.1 ug/kg (Equivalent to 20% CV)
z-Score
4
3
2
1
0
-1
-2
-3
-4
-3.1
-1.5
-0.6
-0.4
-0.3
-0.3
-0.1
-0.1
-0.1
0.1
0.1
0.1
0.3
0.3
0.6
1.8
14
24
21
11
15
3
5
1
18
16
19
22
20
6
2
7
17
Laboratory
En-Scores: S2 - PFHxA
E_n-Score
4
3
2
1
0
-1
-2
-3
-4
-1.7
-0.8
-0.4
-0.2
-0.3
-0.1
0.0
-0.1
0.1
0.1
0.5
0.4
0.2
0.8
0.9
3.8
14
24
21
11
15
3
5
1
18
16
19
22
20
6
2
7
17
Laboratory

Figure 32

Table 39

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	PFHxS
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	11	4	104	0.19	0.10
2	10.8	1.7	106	0.09	0.11
3	10.6	2.12	81	0.00	0.00
4	NT	NT	NT		
5	11.6	2.3	NR	0.47	0.41
6	12	3.0	108	0.66	0.45
7	13.9	4.17	66	1.56	0.78
8	NT	NT	NT		
9	NT	NT	NT		
10	NT	NT	NT		
11	10.5	2.10	85	-0.05	-0.04
14*	4.5	0.67	116	-2.88	-5.85
15	10	3	73	-0.28	-0.19
16	11	0.35	99	0.19	0.46
17**	226	56.5	81.4	101.60	3.81
18	10.6	1.71	NR	0.00	0.00
19	9.9	2.1	13.5	-0.33	-0.31
20	11.6	2.6	80	0.47	0.37
21	7.736	0.786	NR	-1.35	-2.55
22	9.1	NR	74	-0.71	-1.88
23	NT	NT	NT		
24	9.0	2	84	-0.75	-0.74

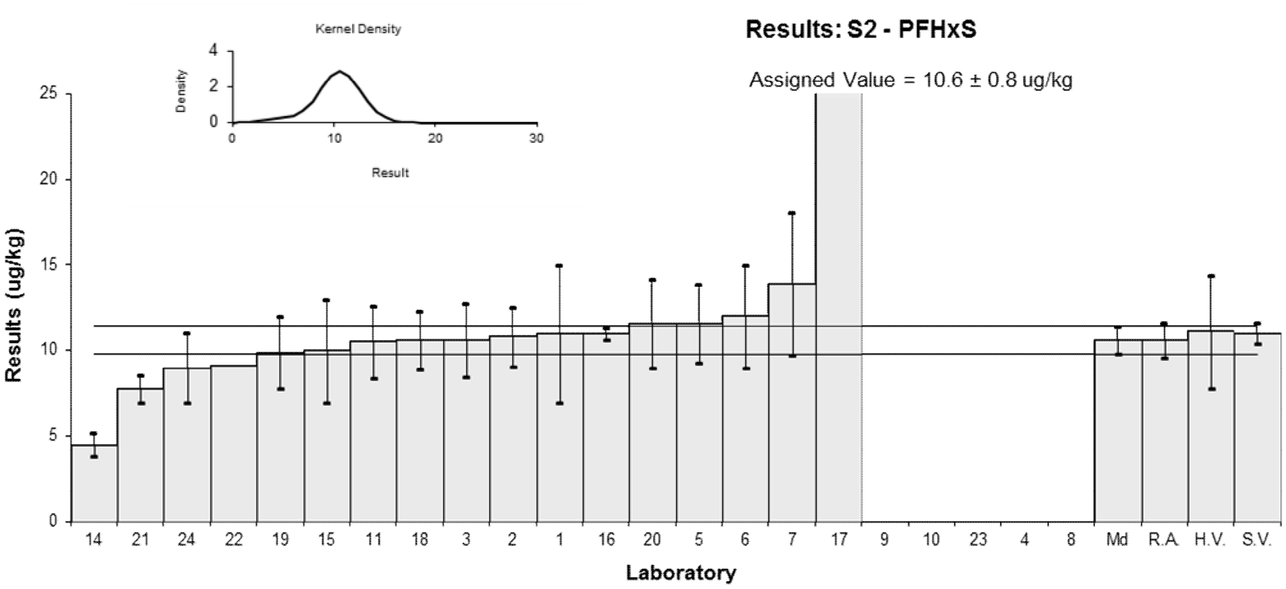
Statistics

Assigned Value***	10.6	0.8
Spike	11.0	0.6
Homogeneity Value	11.1	3.3
Robust Average**	10.4	0.9
Median**	10.6	0.7
Mean**	10.24	
N	17	
Max.	226	
Min.	4.5	
Robust SD**	1.2	
Robust CV**	11%	

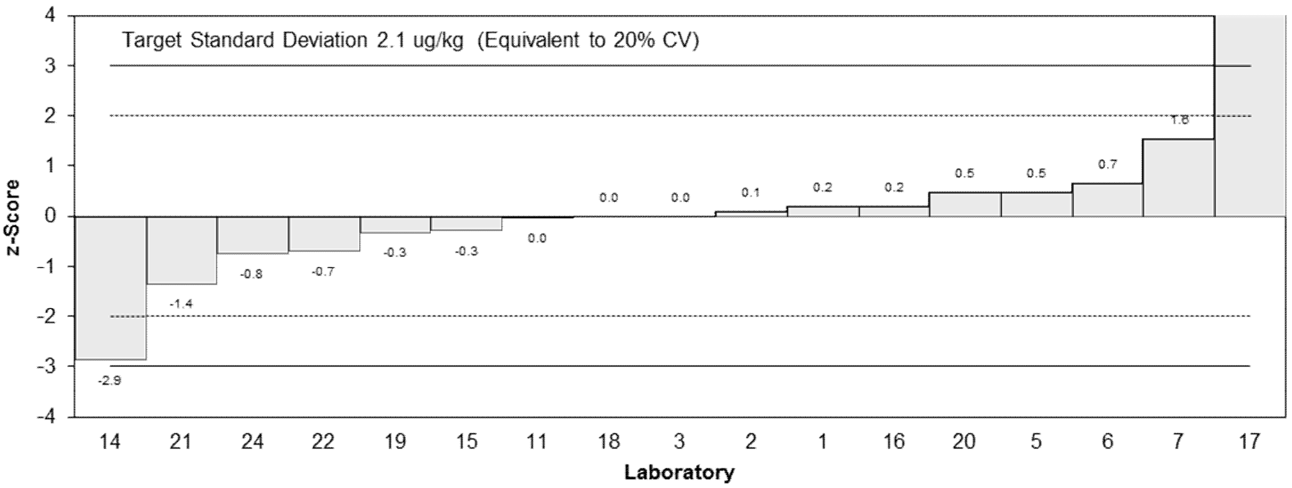
*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Results from laboratory 17 were omitted from the statistical calculations.

***Robust average excluding laboratory 14.



z-Scores: S2 - PFHxS



En-Scores: S2 - PFHxS

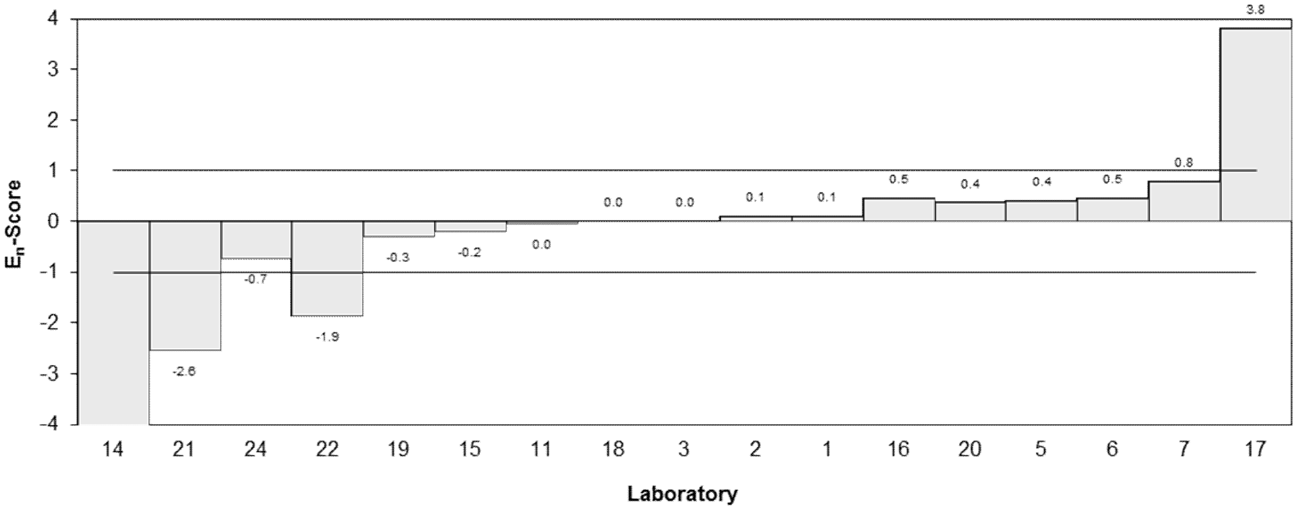


Figure 33

Table 40

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	PFNA
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	1	1	108	0.05	0.01
2	1.17	0.16	99	0.91	0.93
3	0.943	0.189	102	-0.24	-0.21
4	NT	NT	NT		
5	0.91	0.18	NR	-0.40	-0.38
6	1.1	0.28	143	0.56	0.37
7	1.32	0.396	132	1.67	0.80
8	NT	NT	NT		
9	NT	NT	NT		
10	NT	NT	NT		
11	1.01	0.201	95	0.10	0.09
14	<0.5	0.1	107		
15	1.0	0.3	71	0.05	0.03
16	0.96	0.11	87	-0.15	-0.19
17*	22	5.5	76.1	106.11	3.82
18	1.2	0.25	NR	1.06	0.77
19	1.02	0.31	14.6	0.15	0.09
20	1.0	0.3	80	0.05	0.03
21	0.720	0.093	NR	-1.36	-1.87
22	0.77	NR	74	-1.11	-2.00
23	NT	NT	NT		
24	0.83	0.2	101	-0.81	-0.70

Statistics

Assigned Value	0.99	0.11
Spike	1.00	0.05
Homogeneity Value	1.02	0.31
Robust Average*	0.99	0.11
Median*	1.00	0.01
Mean*	1.00	
N	16	
Max.	22	
Min.	0.72	
Robust SD*	0.16	
Robust CV*	16%	

*Results from laboratory 17 were omitted from the statistical calculations.

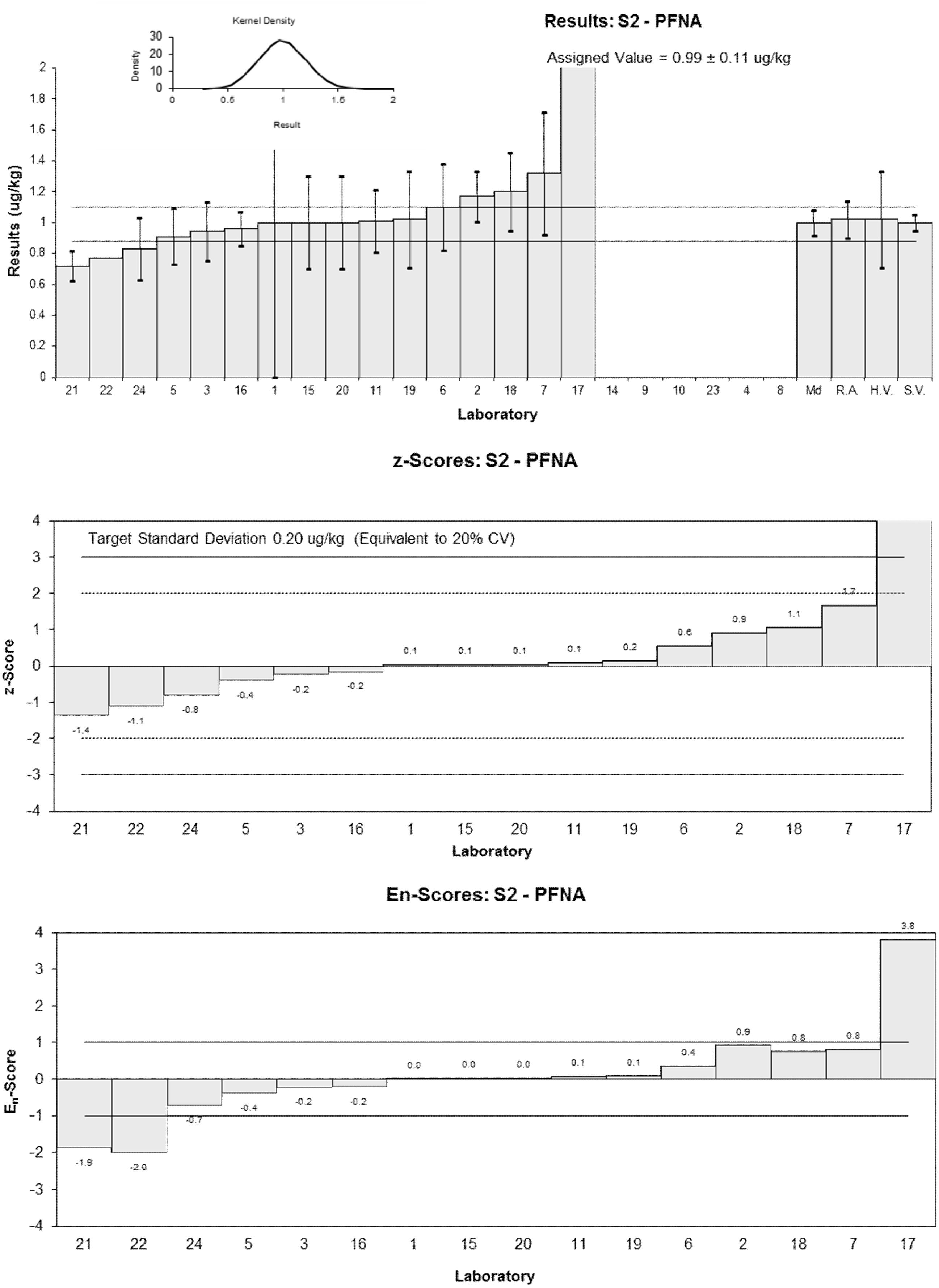

Results: S2 - PFNA
Assigned Value = 0.99 ± 0.11 ug/kg
Kernel Density
Density
30
20
10
0
0
0.5
1
1.5
2
Result
Results (ug/kg)
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
21
22
24
5
3
16
1
15
20
11
19
6
2
18
7
17
14
9
10
23
4
8
Md
R.A.
H.V.
S.V.
Laboratory
z-Scores: S2 - PFNA
Target Standard Deviation 0.20 ug/kg (Equivalent to 20% CV)
z-Score
4
3
2
1
0
-1
-2
-3
-4
-1.4
-1.1
-0.8
-0.4
-0.2
-0.2
0.1
0.1
0.1
0.1
0.2
0.6
0.9
1.1
1.7
21
22
24
5
3
16
1
15
20
11
19
6
2
18
7
17
Laboratory
En-Scores: S2 - PFNA
En-Score
4
3
2
1
0
-1
-2
-3
-4
-1.9
-2.0
-0.7
-0.4
-0.2
-0.2
0.0
0.0
0.0
0.1
0.1
0.4
0.9
0.8
0.8
3.8
21
22
24
5
3
16
1
15
20
11
19
6
2
18
7
17
Laboratory

Figure 34

Table 41

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	PFOA
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	4	2	109	-0.33	-0.14
2	5.24	0.89	98	1.12	0.96
3	4.13	0.825	86	-0.18	-0.16
4	NT	NT	NT		
5	4.4	0.9	NR	0.14	0.12
6	4.8	1.2	135	0.61	0.41
7	5.69	1.71	108	1.65	0.80
8	7.78	0.778	NR	4.09	3.89
9	NT	NT	NT		
10	NT	NT	NT		
11	4.25	0.851	90	-0.04	-0.03
14*	1.7	0.27	101	-3.01	-4.92
15	4.2	1	73	-0.09	-0.07
16	4.2	0.18	97	-0.09	-0.17
17**	91.6	22.9	88.6	102.01	3.81
18	4.5	0.75	115	0.26	0.25
19	4.31	0.94	15.2	0.04	0.03
20	4.8	1.4	83	0.61	0.35
21	3.308	0.347	NR	-1.14	-1.71
22	3.40	NR	74	-1.03	-1.96
23	NT	NT	NT		
24	3.4	0.7	97	-1.03	-1.06

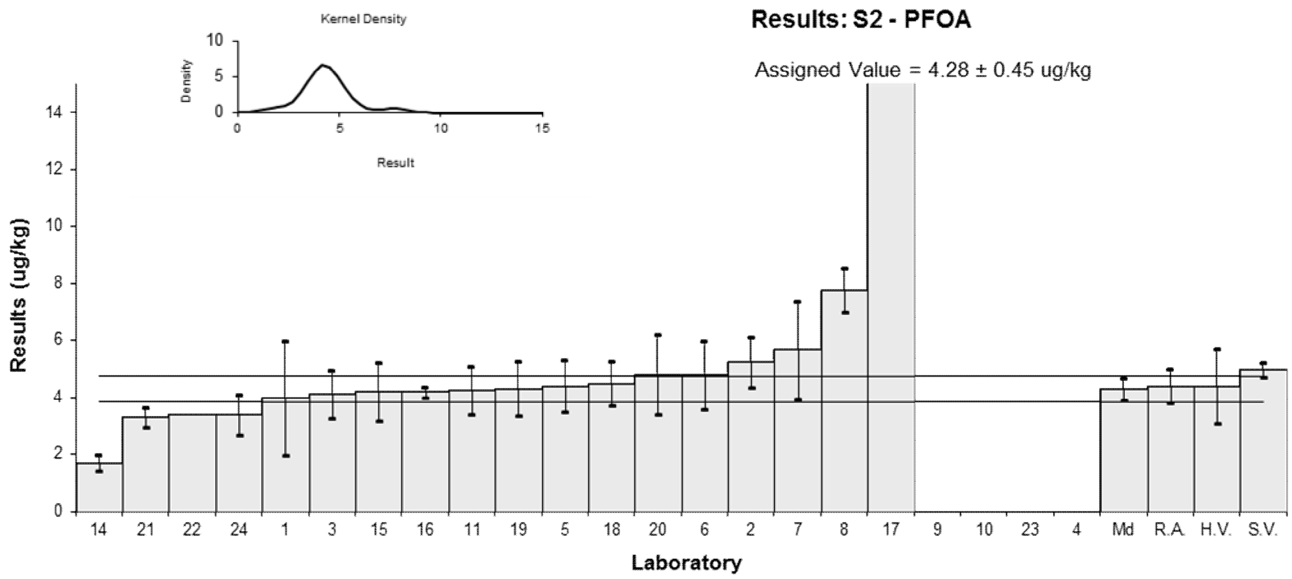
Statistics

Assigned Value***	4.28	0.45
Spike	4.99	0.25
Homogeneity Value	4.4	1.3
Robust Average**	4.30	0.52
Median**	4.50	0.42
Mean**	4.36	
N	18	
Max.	91.6	
Min.	1.7	
Robust SD**	0.69	
Robust CV**	16%	

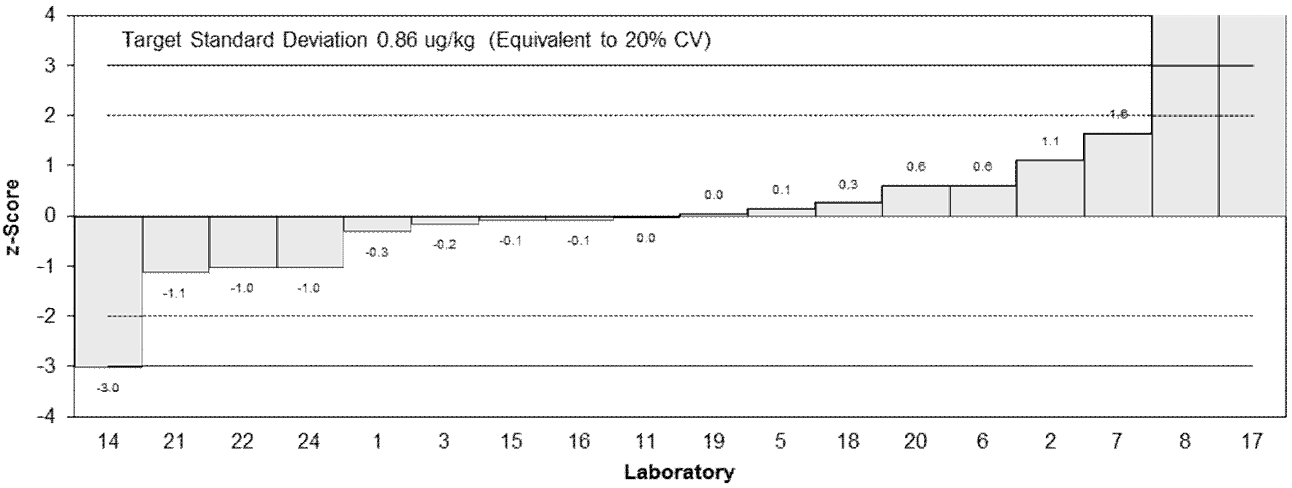
*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Results from laboratory 17 were omitted from the statistical calculations.

***Robust average excluding laboratories 8 and 14.



z-Scores: S2 - PFOA



En-Scores: S2 - PFOA

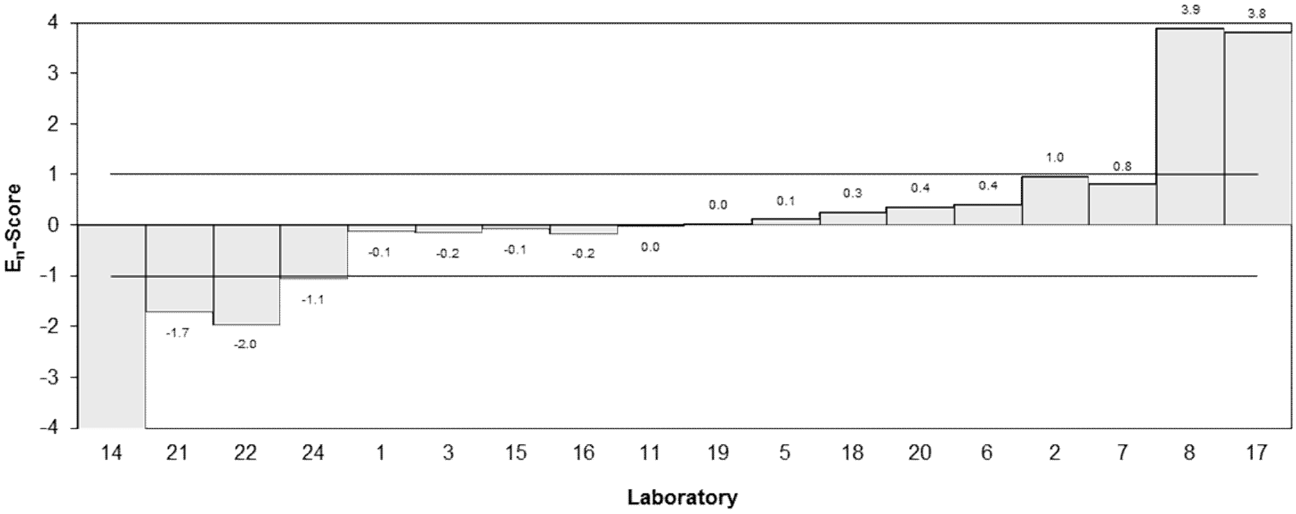


Figure 35

Table 42

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	PFOS
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	3	1	96	0.70	0.35
2	2.84	0.88	102	0.40	0.22
3	2.41	0.482	96	-0.42	-0.38
4	NT	NT	NT		
5	2.9	0.6	NR	0.51	0.40
6	2.8	0.70	115	0.32	0.22
7	3.34	1.00	135	1.35	0.68
8	3.16	0.316	NR	1.01	1.18
9	NT	NT	NT		
10	NT	NT	NT		
11	2.39	0.478	93	-0.46	-0.42
14*	1.0	0.17	85	-3.10	-4.50
15	2.3	0.7	75	-0.63	-0.43
16	2.5	0.14	95	-0.25	-0.37
17**	59.2	14.8	80.3	107.55	3.82
18	3.4	0.95	120	1.46	0.77
19	2.35	0.57	14.7	-0.53	-0.43
20	2.5	0.1	73	-0.25	-0.39
21	2.057	0.215	NR	-1.09	-1.49
22	1.82	NR	74	-1.54	-2.53
23	NT	NT	NT		
24	2.3	0.4	77	-0.63	-0.64

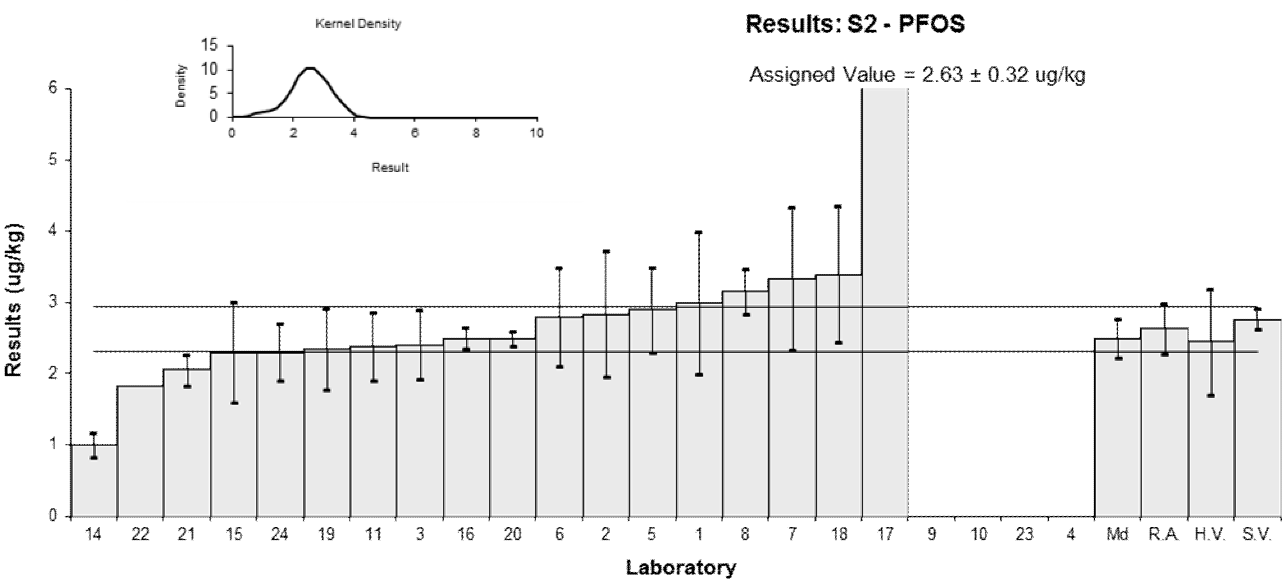
Statistics

Assigned Value***	2.63	0.32
Spike	2.77	0.14
Homogeneity Value	2.45	0.74
Robust Average**	2.58	0.34
Median**	2.50	0.26
Mean**	2.53	
N	18	
Max.	59.2	
Min.	1	
Robust SD**	0.51	
Robust CV**	19%	

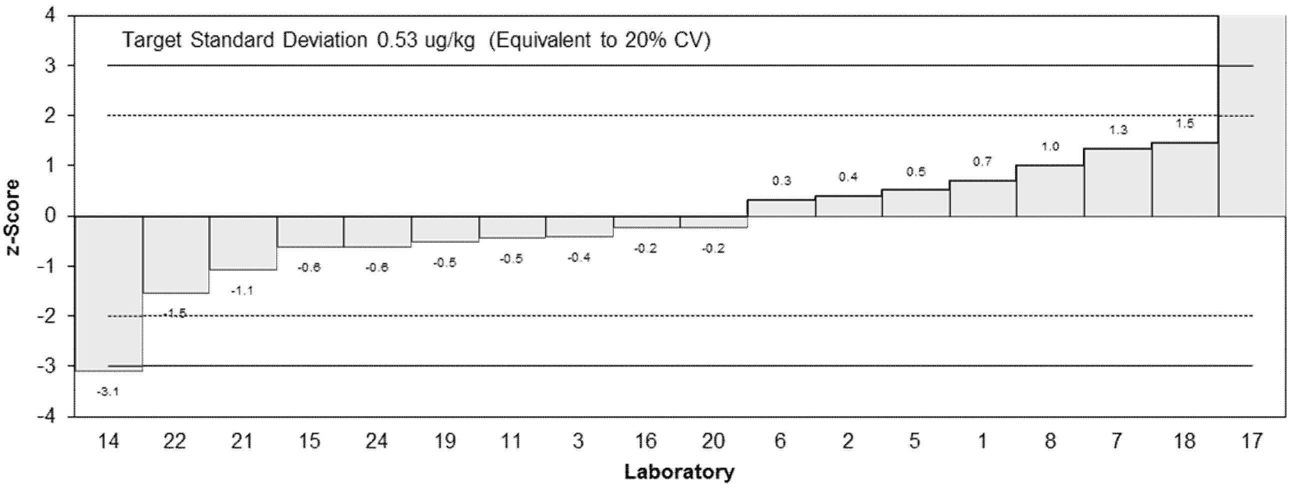
*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Results from laboratory 17 were omitted from the statistical calculations.

***Robust average excluding laboratory 14.



z-Scores: S2 - PFOS



En-Scores: S2 - PFOS

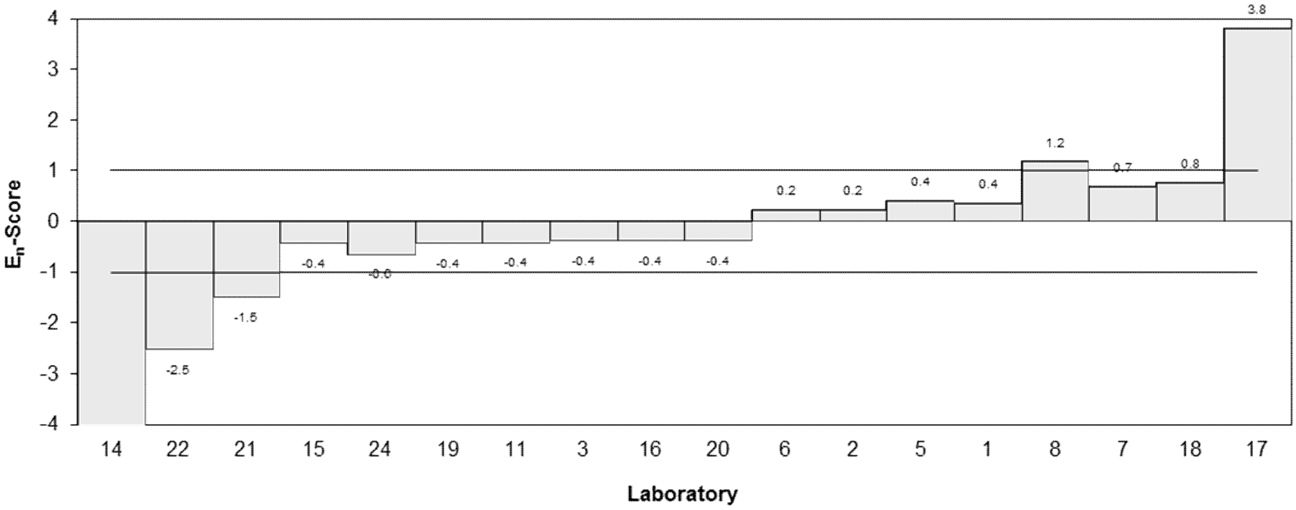


Figure 36

Table 43

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	PFOSA
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	<5	NR	109		
2	5.19	0.93	96	1.15	0.95
3	4.47	0.893	85	0.30	0.25
4	NT	NT	NT		
5	3.5	0.7	NR	-0.85	-0.89
6	4.6	1.6	71	0.45	0.23
7	5.68	1.70	35	1.73	0.83
8	NT	NT	NT		
9	NT	NT	NT		
10	NT	NT	NT		
11	4.22	0.845	78	0.00	0.00
14*	1.9	0.42	84	-2.75	-3.95
15	4.0	1	56	-0.26	-0.20
16	4.1	0.32	91	-0.14	-0.23
17**	95.2	23.8	22.5	107.80	3.82
18	4.1	1.04	NR	-0.14	-0.11
19	4.21	0.99	24.9	-0.01	-0.01
20	4.2	0.3	88	-0.02	-0.04
21	3.880	0.412	NR	-0.40	-0.58
22	2.98	NR	74	-1.47	-3.02
23	NT	NT	NT		
24	NT	NT	NT		

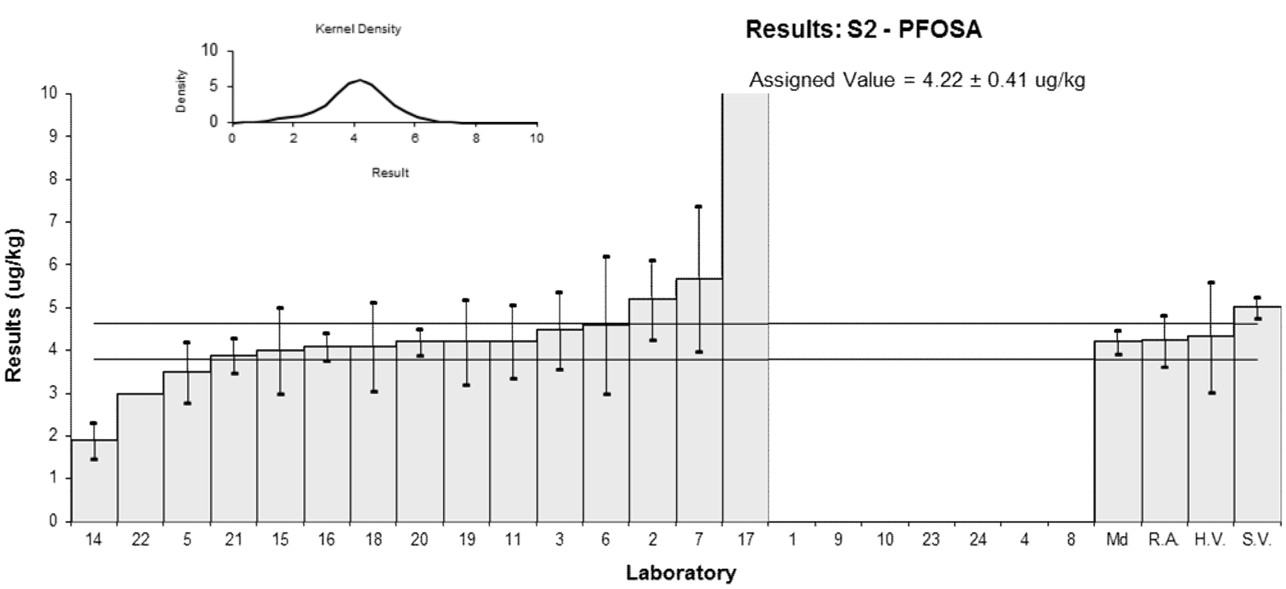
Statistics

Assigned Value***	4.22	0.41
Spike	5.01	0.25
Homogeneity Value	4.3	1.3
Robust Average**	4.12	0.51
Median**	4.15	0.25
Mean**	4.07	
N	15	
Max.	95.2	
Min.	1.9	
Robust SD**	0.59	
Robust CV**	14%	

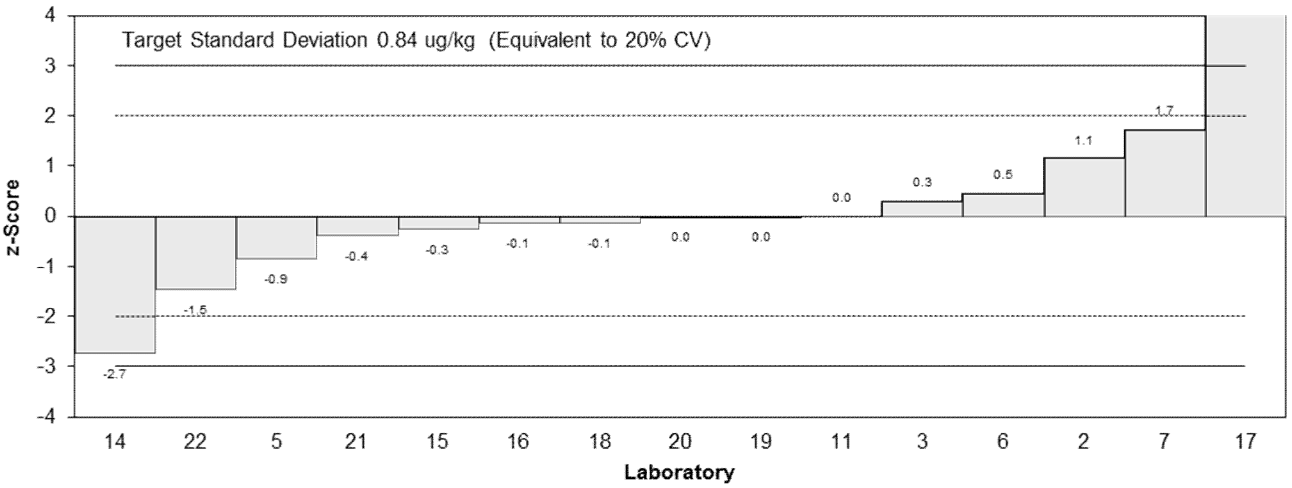
*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Results from laboratory 17 were omitted from the statistical calculations.

***Robust average excluding laboratory 14.



z-Scores: S2 - PFOSA



En-Scores: S2 - PFOSA

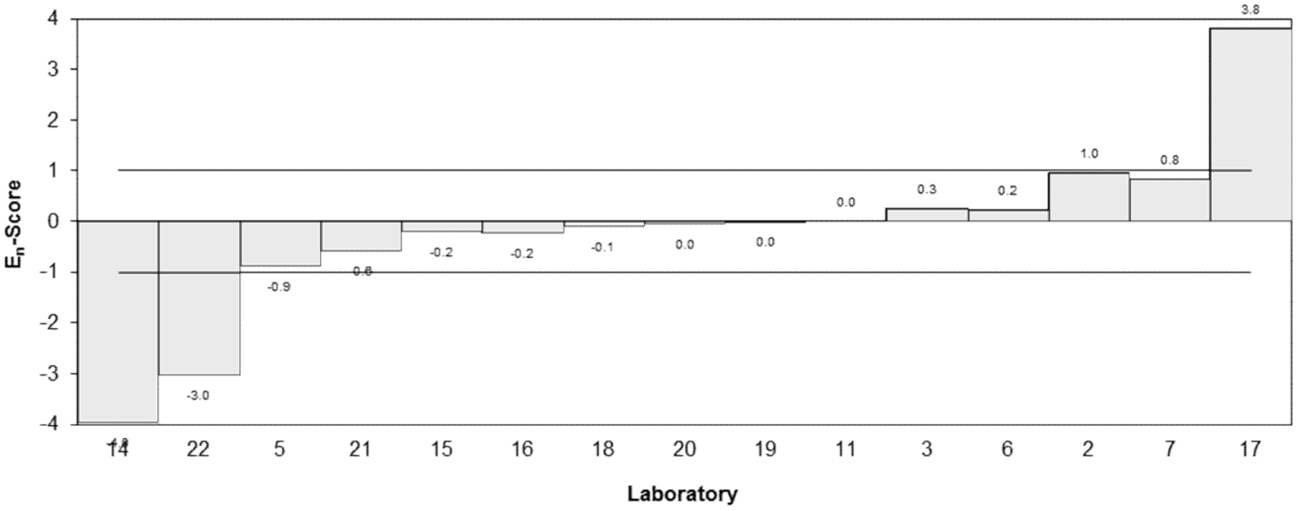


Figure 37

Table 44

Sample Details

Sample No.	S2
Matrix.	Tomato
Analyte.	PFPeA
Units	ug/kg

Participant Results

Lab Code	Result	Uncertainty	Recovery	z-Score	E_n-Score
1	47	10	101	0.16	0.14
2	50.8	6.6	84	0.58	0.68
3	38.0	7.61	102	-0.82	-0.87
4	NT	NT	NT		
5	46.7	12	NR	0.13	0.09
6	47	12	120	0.16	0.12
7	72.8	21.8	45	3.00	1.23
8	NT	NT	NT		
9	NT	NT	NT		
10	NT	NT	NT		
11	39.3	7.86	88	-0.68	-0.70
14*	18	4.8	97	-3.02	-4.36
15	46	10	60	0.05	0.05
16	44	2.3	87	-0.16	-0.32
17**	913	228.25	124	95.33	3.80
18	45.7	30.44	NR	0.02	0.01
19	54.8	9.7	11.6	1.02	0.88
20	44.2	4.0	88	-0.14	-0.23
21	61.904	7.157	NR	1.80	1.99
22	41.2	NR	74	-0.47	-1.05
23	NT	NT	NT		
24	38	7	79	-0.82	-0.92

Statistics

Assigned Value***	45.5	4.1
Spike	47.2	2.4
Homogeneity Value	51	15
Robust Average**	45.7	4.9
Median**	45.9	3.8
Mean**	46.0	
N	17	
Max.	913	
Min.	18	
Robust SD**	6.1	
Robust CV**	13%	

*After the interim report was published, laboratory 14 reported that an incorrect dilution factor was applied. All results are out by a factor of 2.5.

**Results from laboratory 17 were omitted from the statistical calculations.

***Robust average excluding laboratories 7 and 14.

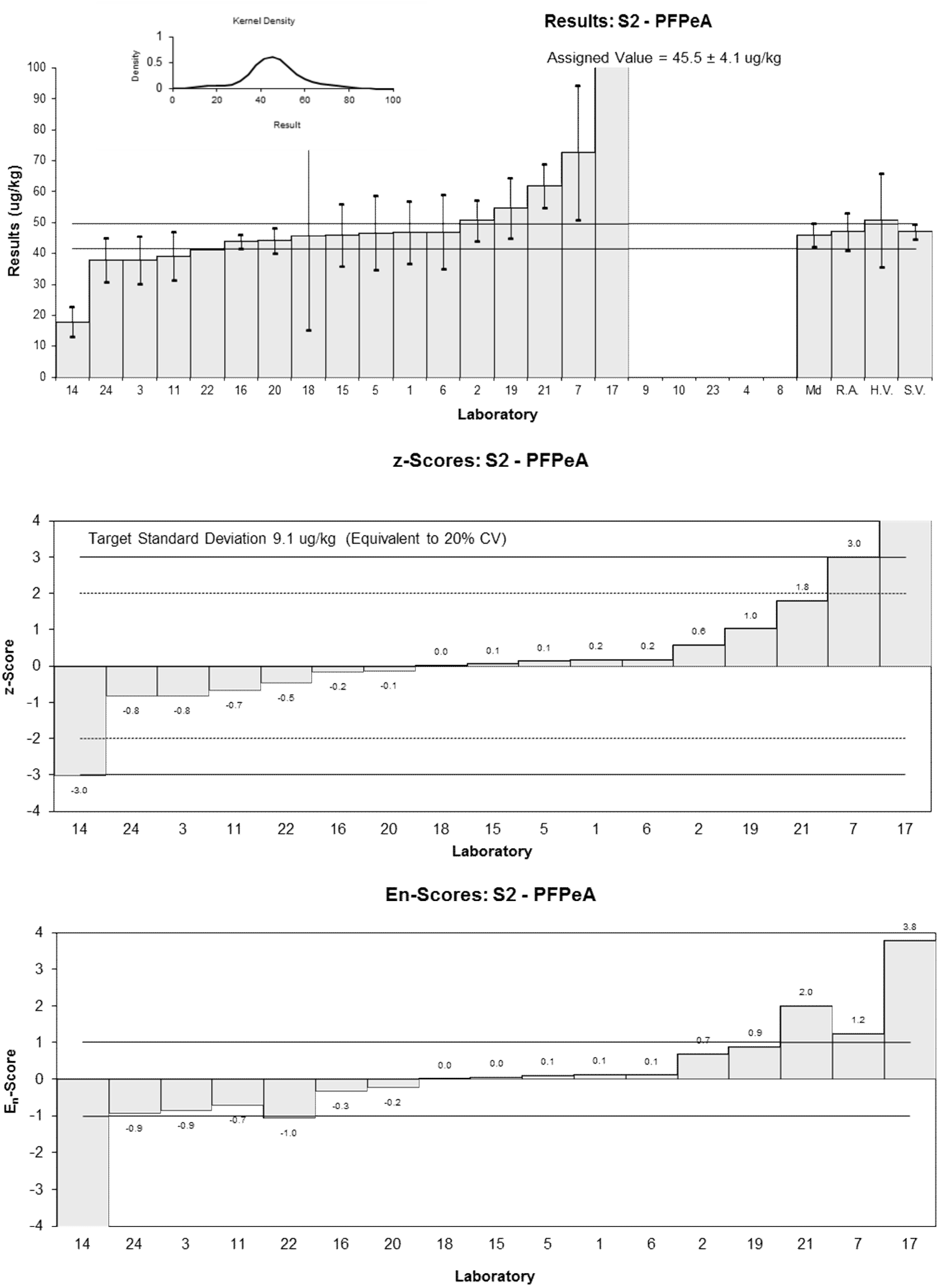

Kernel Density
Density
1
0.5
0
0
20
40
60
80
100
Result
Results: S2 - PFPeA
Assigned Value = 45.5 ± 4.1 ug/kg
Results (ug/kg)
100
90
80
70
60
50
40
30
20
10
0
14
24
3
11
22
16
20
18
15
5
1
6
2
19
21
7
17
9
10
23
4
8
Md
R.A.
H.V.
S.V.
Laboratory
z-Scores: S2 - PFPeA
Target Standard Deviation 9.1 ug/kg (Equivalent to 20% CV)
z-Score
4
3
2
1
0
-1
-2
-3
-4
-3.0
-0.8
-0.8
-0.7
-0.5
-0.2
-0.1
0.0
0.1
0.1
0.2
0.2
0.6
1.0
1.8
3.0
14
24
3
11
22
16
20
18
15
5
1
6
2
19
21
7
17
Laboratory
En-Scores: S2 - PFPeA
E_n-Score
4
3
2
1
0
-1
-2
-3
-4
-0.9
-0.9
-0.7
-1.0
-0.3
-0.2
0.0
0.0
0.1
0.1
0.1
0.7
0.9
2.0
1.2
3.8
14
24
3
11
22
16
20
18
15
5
1
6
2
19
21
7
17
Laboratory

Figure 38

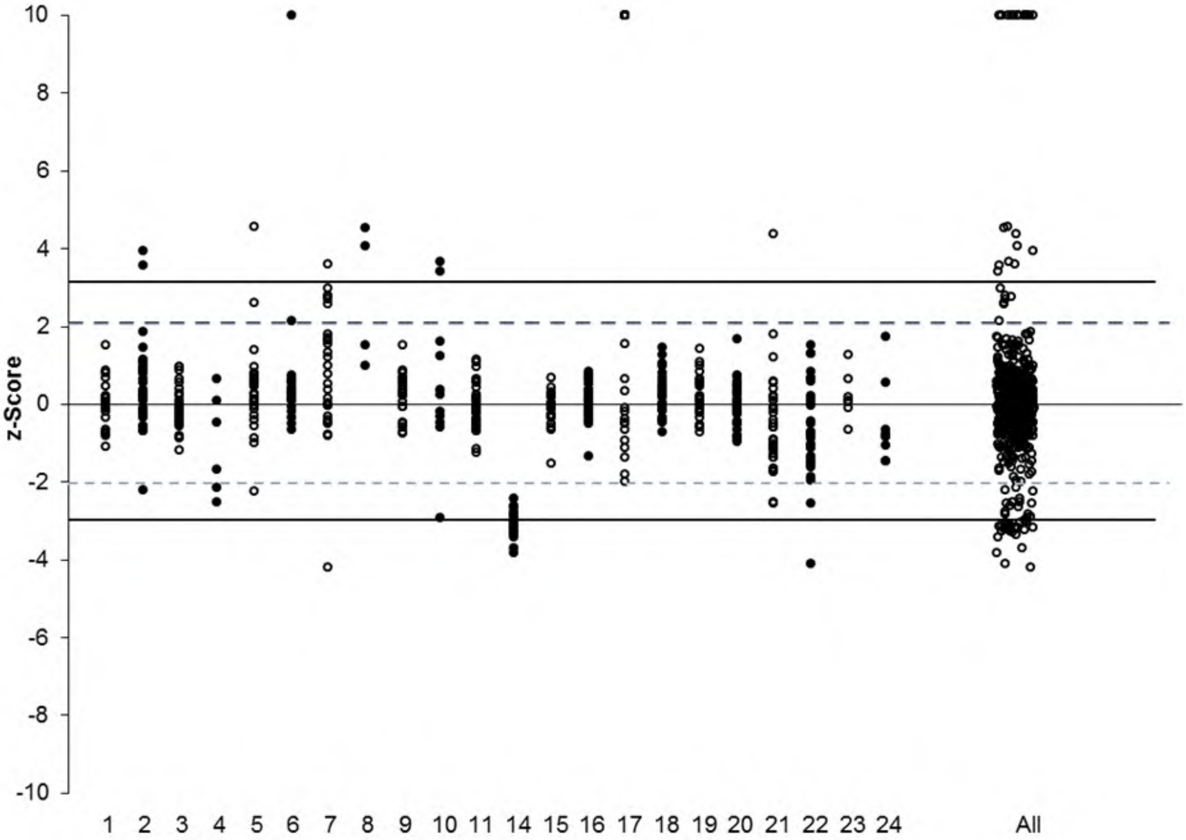


Figure 39 z-Score Dispersal by Laboratory*

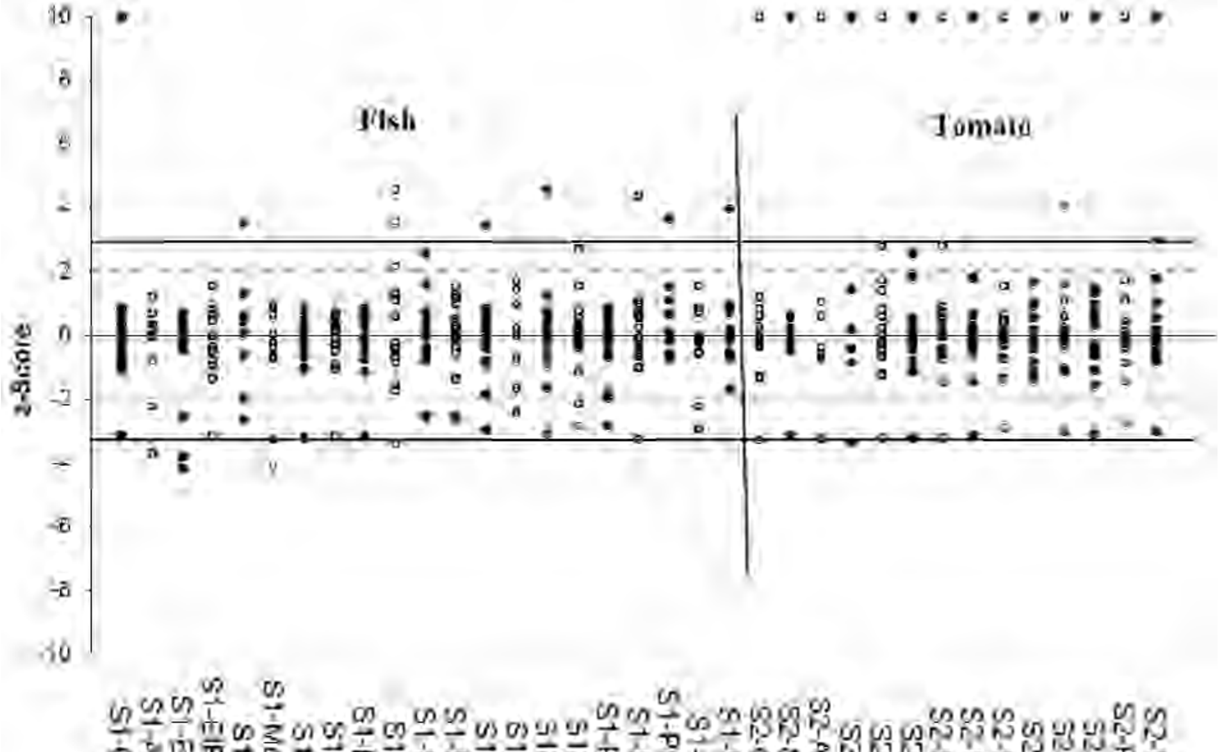


Figure 40 z-Score Dispersal by Analyte*

* Scores greater than 10 have been plotted as 10.

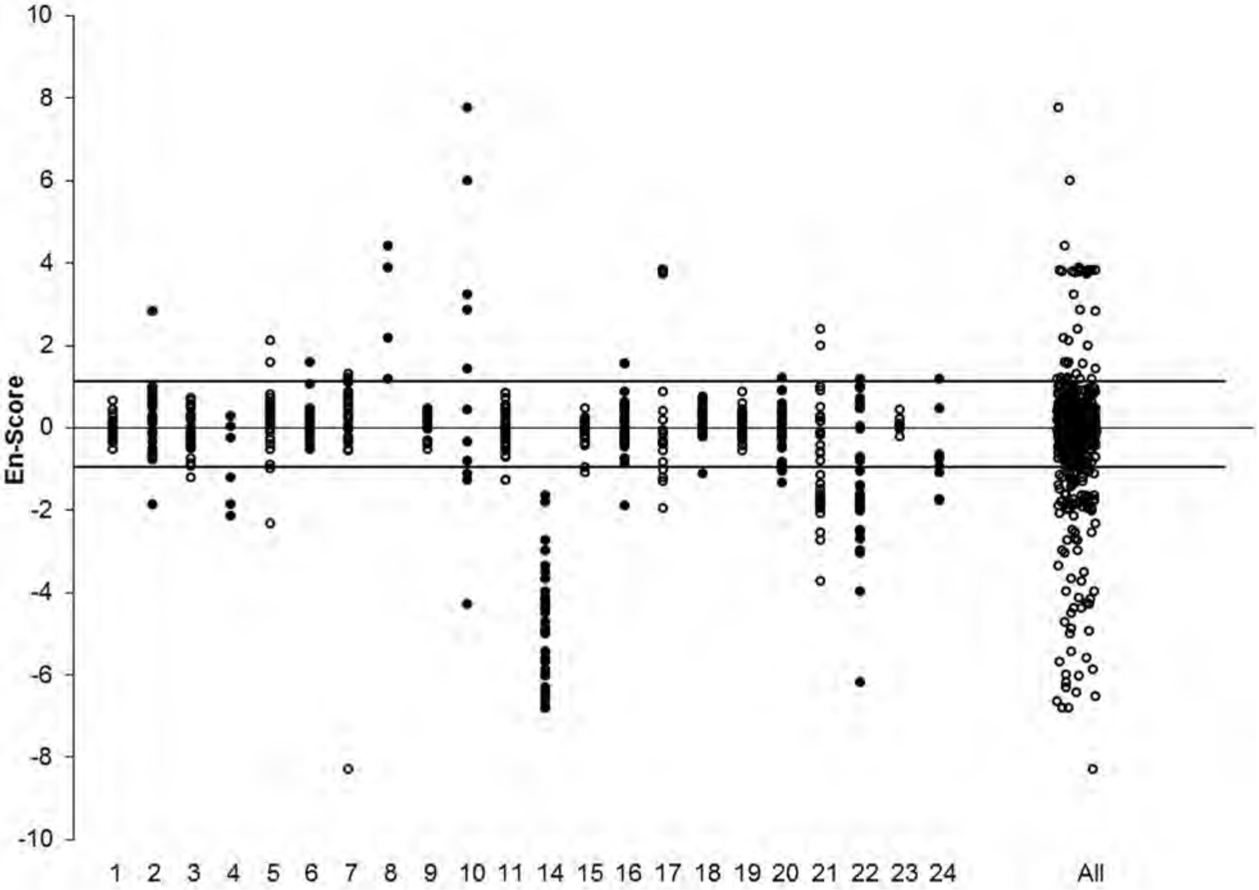


Figure 41 E_n-Score Dispersal by Laboratory

6 DISCUSSION OF RESULTS

6.1 Assigned Value

The robust average of participants' results was used as the assigned value for all samples. The robust averages and associated expanded uncertainties, were calculated using the procedure described in 'ISO13528:2015(E), Statistical methods for use in proficiency testing by interlaboratory comparisons'.[7] The calculation of the expanded uncertainty for the robust average of PFDA in Sample S1 is presented in Appendix 2.

Results less than 50% and greater than 150% of the robust average were removed before calculation of the assigned value.[3,4]

Assigned values for spiked Samples S1 and S2 were within the range 81 – 113% of the spiked concentration for that analyte (Table 45).

No assigned values were calculated for 8:2 FTS and 10:2 FTS in Sample S1 the levels in the incurred samples were low, few laboratories reported numeric results and these were too variable.

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 45 Comparison of Assigned Value and Spiked Concentration.

Sample	Matrix	Analyte	Units	Spiked Concentration	Assigned Value	Assigned/ Spike (%)
S1	Fish	6:2 FTS	µg/kg	3.87	3.44	89
S1	Fish	ADONA	µg/kg	33.4	27.1	81
S1	Fish	EtFOSA	µg/kg	9.51	9.59	101
S1	Fish	EtFOSAA	µg/kg	12.2	12.8	105
S1	Fish	GenX	µg/kg	20.4	23.1	113
S1	Fish	MeFOSE	µg/kg	15.0	16.3	109
S1	Fish	PFBS	µg/kg	2.98	2.55	86
S1	Fish	PFNA	µg/kg	2.51	2.38	95
S1	Fish	PFOA	µg/kg	5.55	4.94	89
S1	Fish	PFOSA	µg/kg	4.94	4.12	83
S1	Fish	PFPeA	µg/kg	3.01	2.86	95
S2	Tomato	6:2 FTS	µg/kg	9.10	9.0	99
S2	Tomato	8:2 FTS	µg/kg	9.19	8.87	97
S2	Tomato	ADONA	µg/kg	31.6	26.0	82
S2	Tomato	GenX	µg/kg	19.2	21.2	110
S2	Tomato	PFBA	µg/kg	50.0	49.7	99
S2	Tomato	PFBS	µg/kg	30.1	24.3	81
S2	Tomato	PFHpA	µg/kg	13.0	12.1	93
S2	Tomato	PFHxA	µg/kg	25.0	25.4	102
S2	Tomato	PFHxS	µg/kg	11.0	10.6	96

S2	Tomato	PFNA	µg/kg	1.00	0.99	99
S2	Tomato	PFOA	µg/kg	4.99	4.28	86
S2	Tomato	PFOS	µg/kg	2.77	2.63	95
S2	Tomato	PFOSA	µg/kg	5.01	4.22	84
S2	Tomato	PFPeA	µg/kg	47.2	45.5	96

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded uncertainty associated with their results and the basis of this uncertainty estimate (Table 6).

It is a requirement of the ISO Standard 17025 that laboratories have procedures to estimate the uncertainty of chemical measurements and to report this uncertainty in specific circumstances, including: when the client’s instruction so requires.

Five hundred and one of five hundred and thirty-six results (93%) were reported with an associated estimate of expanded measurement uncertainty. Laboratory **10** did not report an estimate of measurement uncertainty, while laboratories **15**, **18** and **22** reported expanded measurement uncertainties for some analytes only.

The magnitude of the reported expanded uncertainties was within the range 1.7% to 100% of the reported value. Thirty were less than 10% relative, which the study coordinator believes are unrealistically small for a routine PFAS measurement and eleven were larger than 50% relative.

Results returning a satisfactory z-score but an unsatisfactory E_n-score may have underestimated the uncertainty.

Some participants attached an estimate of the expanded measurement uncertainty to a result reported as less than their limit of reporting.

In some cases the results were reported with an inappropriate number of significant figures. The recommended format is to write uncertainty to no more than two significant figures and then to write the result with the corresponding number of decimal places (for example instead of 3.37 ± 0.8425 µg/kg better report 3.37 ± 0.84 µg/kg)[6].

6.3 z-Score

A target standard deviation equivalent to 20% coefficient of variation (CV) was used to calculate z-scores. The between-laboratory coefficient of variation predicted by the modified Horwitz equation[8] and the between laboratories CV are presented for comparison in Table 46.

Table 46 Target standard deviation, between laboratories CV and modified Horwitz values

Sample	Analyte	Assigned value	Unit	Target SD (as PCV, %)	Modified Horwitz CV (%)	Between laboratories’ CV (%)
S1	6:2 FTS	3.44	µg/kg	20	22	15
S1	ADONA	27.1	µg/kg	20	22	21
S1	EtFOSA	9.59	µg/kg	20	22	11
S1	EtFOSAA	12.8	µg/kg	20	22	19
S1	GenX	23.1	µg/kg	20	22	26
S1	MeFOSE	16.3	µg/kg	20	22	16

Sample	Analyte	Assigned value	Unit	Target SD (as PCV, %)	Modified Horwitz CV (%)	Between laboratories' CV (%)
S1	PFBS	2.55	µg/kg	20	22	11
S1	PFDA	2.54	µg/kg	20	22	12
S1	PFDoA	3.51	µg/kg	20	22	13
S1	PFDS	2.30	µg/kg	20	22	26
S1	PFHpS	0.815	µg/kg	20	22	15
S1	PFHxS	4.60	µg/kg	20	22	22
S1	PFNA	2.38	µg/kg	20	22	14
S1	PFNS	0.97	µg/kg	20	22	31
S1	PFOA	4.94	µg/kg	20	22	13
S1	PFOS	192	µg/kg	20	21	9.4
S1	PFOSA	4.12	µg/kg	20	22	14
S1	PFPeA	2.86	µg/kg	20	22	16
S1	PFTeDA	1.47	µg/kg	20	22	16
S1	PFTrA	0.666	µg/kg	20	22	18
S1	PFUnA	0.998	µg/kg	20	22	14
S2	6:2 FTS	9.0	µg/kg	20	22	18
S2	8:2 FTS	8.87	µg/kg	20	22	7.9
S2	ADONA	26.0	µg/kg	20	22	18
S2	GenX	21.2	µg/kg	20	22	21
S2	PFBA	49.7	µg/kg	20	22	19
S2	PFBS	24.3	µg/kg	20	22	15
S2	PFHpA	12.1	µg/kg	20	22	12
S2	PFHxA	25.4	µg/kg	20	22	9.1
S2	PFHxS	10.6	µg/kg	20	22	11
S2	PFNA	0.99	µg/kg	20	22	16
S2	PFOA	4.28	µg/kg	20	22	16
S2	PFOS	2.63	µg/kg	20	22	19
S2	PFOSA	4.22	µg/kg	20	22	14
S2	PFPeA	45.5	µg/kg	20	22	13

Note: Shaded cells are between participant laboratories' CV which were higher than the target SD established by the study coordinator and the coefficient of variation from predictive mathematical model (modified Horwitz equation).

The dispersal of participants' z-scores is graphically presented by laboratory in Figure 39 and by analyte in Figure 40.

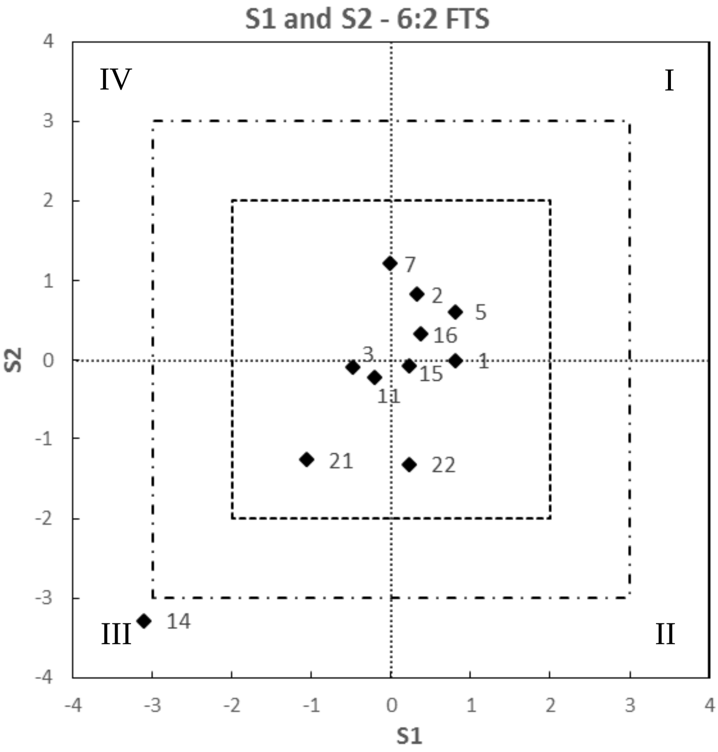
Of the five hundred and twenty-two results for which z-scores were calculated, 451 (86%) returned a satisfactory z-score of |z| ≤ 2.

Seventeen laboratories analysed both matrices. Laboratories **3** and **11** returned satisfactory z-scores for all analytes reported for which z-scores were calculated (35).

Five laboratories analysed only one matrix. Of these laboratories, laboratory **9** performed satisfactorily for all analytes reported for which z-scores were calculated (21).

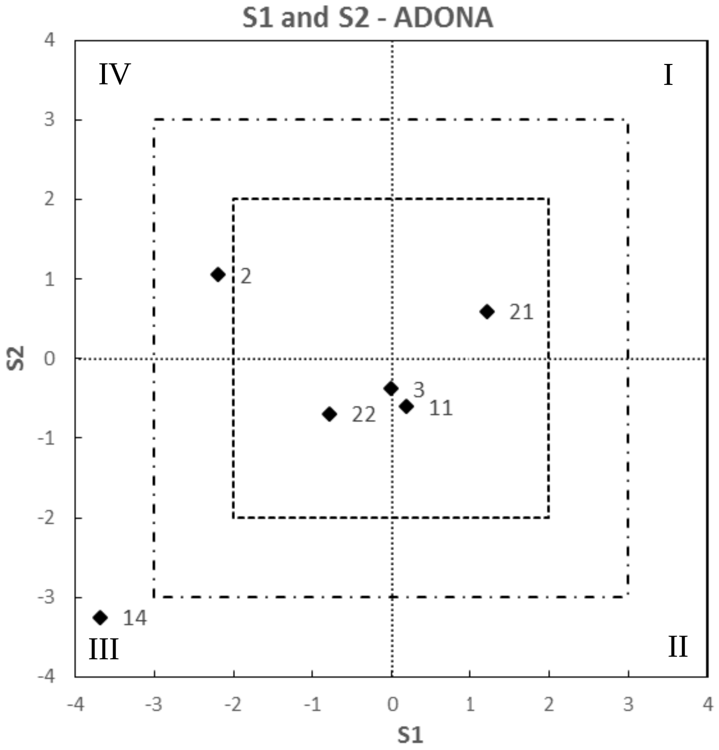
Laboratory **14** returned unsatisfactory z-scores for all analytes tested for which z-scores were calculated (30). After the interim report was published, this laboratory reported that an incorrect dilution factor was applied, with all results out by a factor of 2.5.

Scatter plots of z-scores for all analytes are presented in Figures 42 to 51. Scores are predominantly plotted in quadrants I and III, indicating laboratory bias is the major contributor to the variability of results.



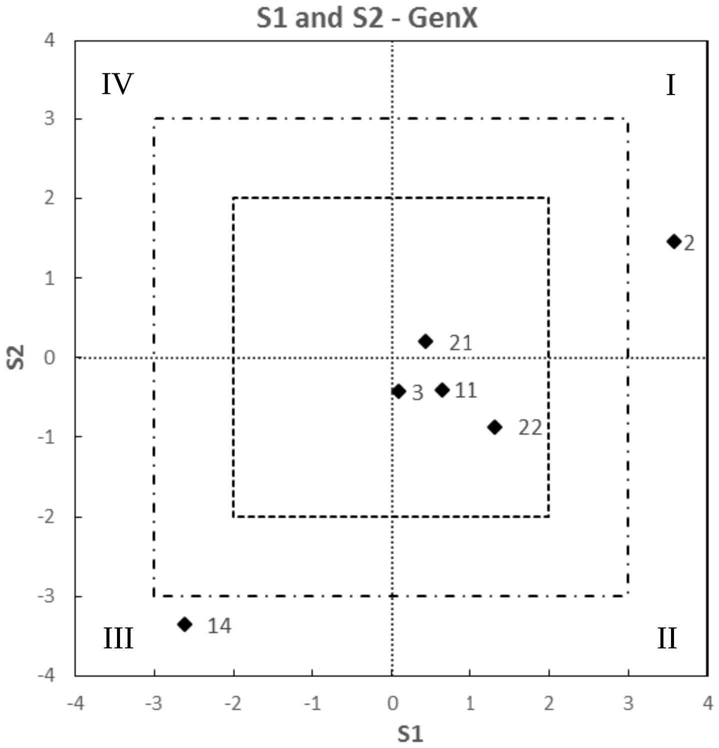
Laboratory 17 is off scale.

Figure 42 S1 (Fish) and S2 (Tomato) z-score scatter plot 6:2 FTS



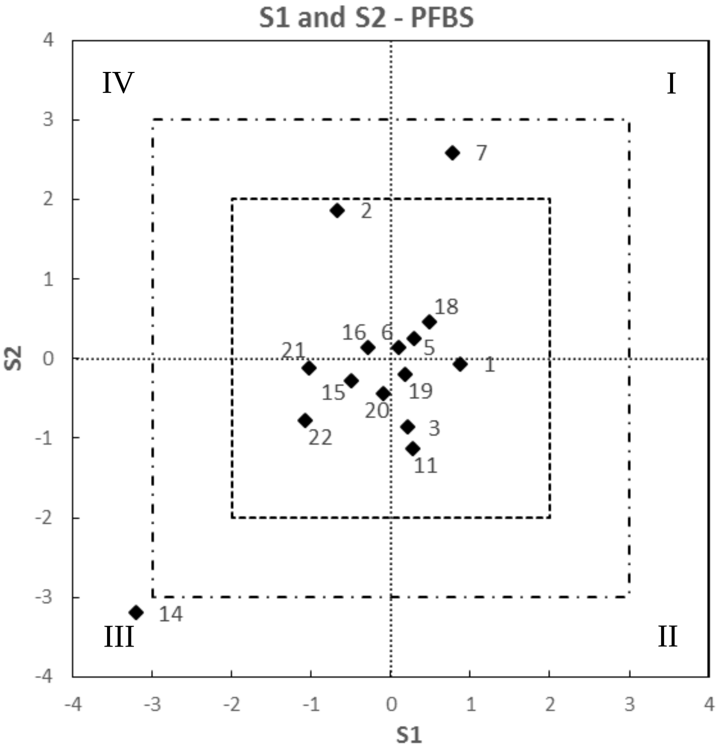
Laboratory 17 is off scale.

Figure 43 S1 (Fish) and S2 (Tomato) z-score scatter plot ADONA



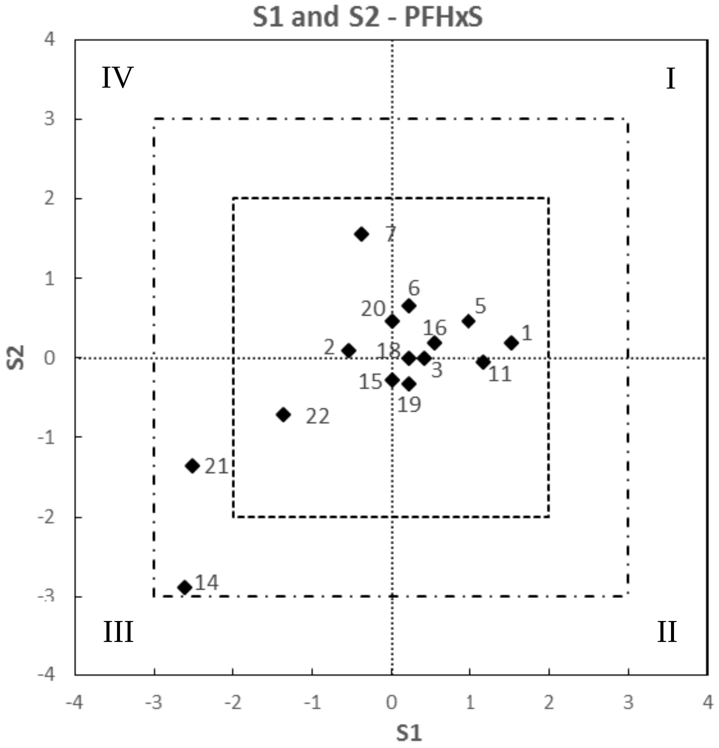
Laboratory 17 is off scale.

Figure 44 S1 (Fish) and S2 (Tomato) z-score scatter plot GenX



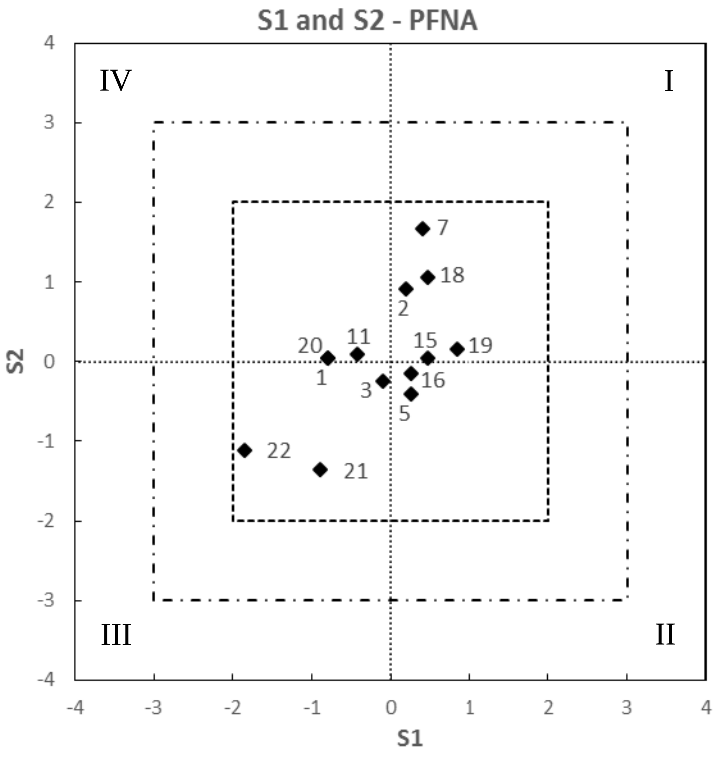
Laboratory 17 is off scale.

Figure 45 S1 (Fish) and S2 (Tomato) z-score scatter plot PFBS



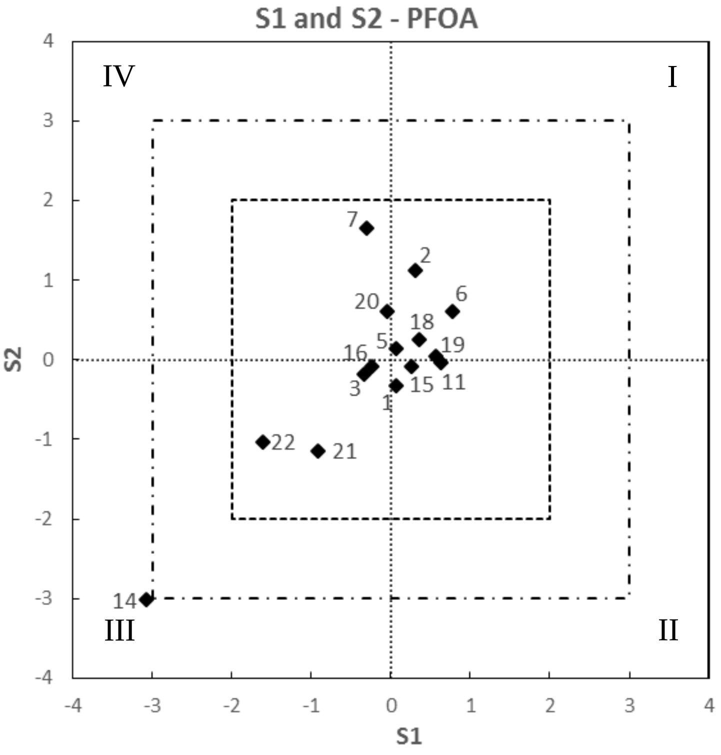
Laboratory 17 is off scale.

Figure 46 S1 (Fish) and S2 (Tomato) z-score scatter plot PFHxS



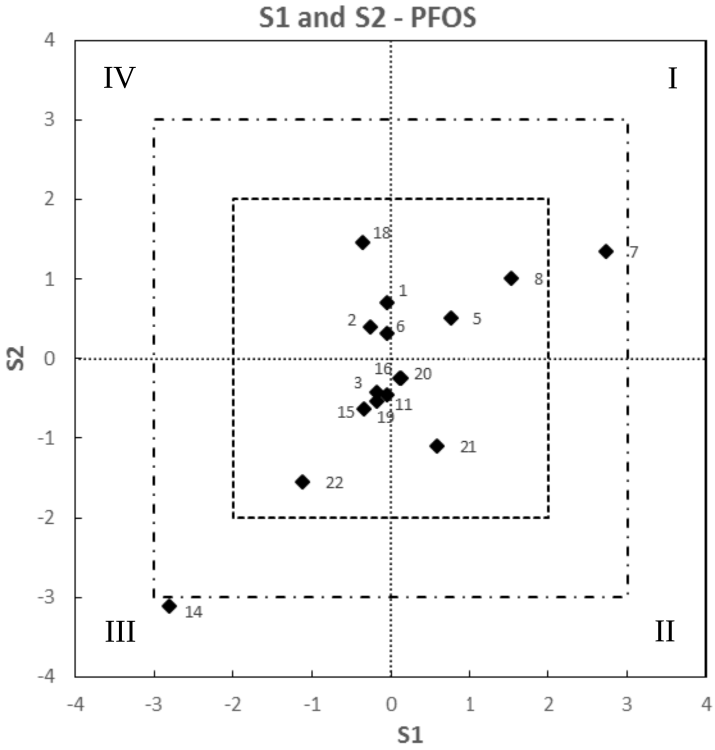
Laboratory 17 is off scale.

Figure 47 S1 (Fish) and S2 (Tomato) z-score scatter plot PFNA



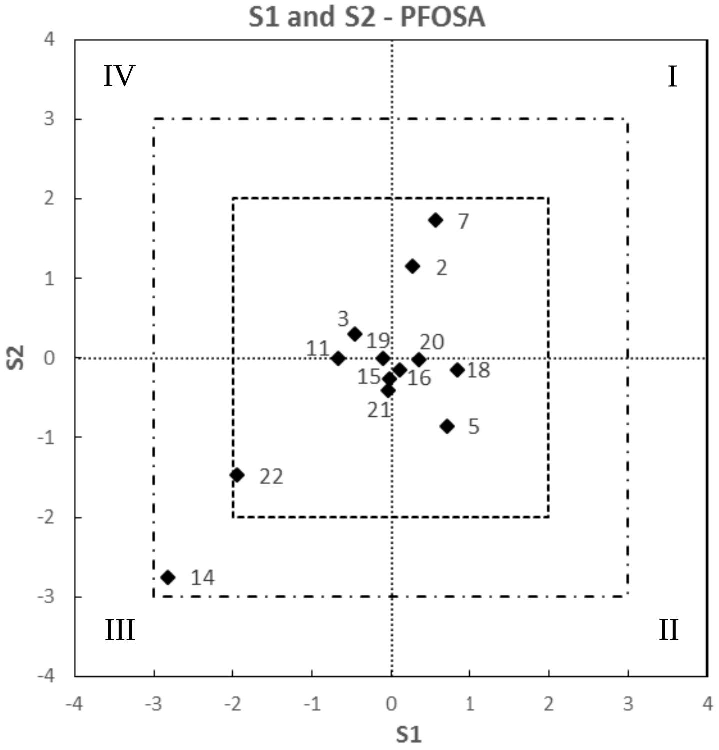
Laboratories 8 and 17 are off scale.

Figure 48 S1 (Fish) and S2 (Tomato) z-score scatter plot PFOA



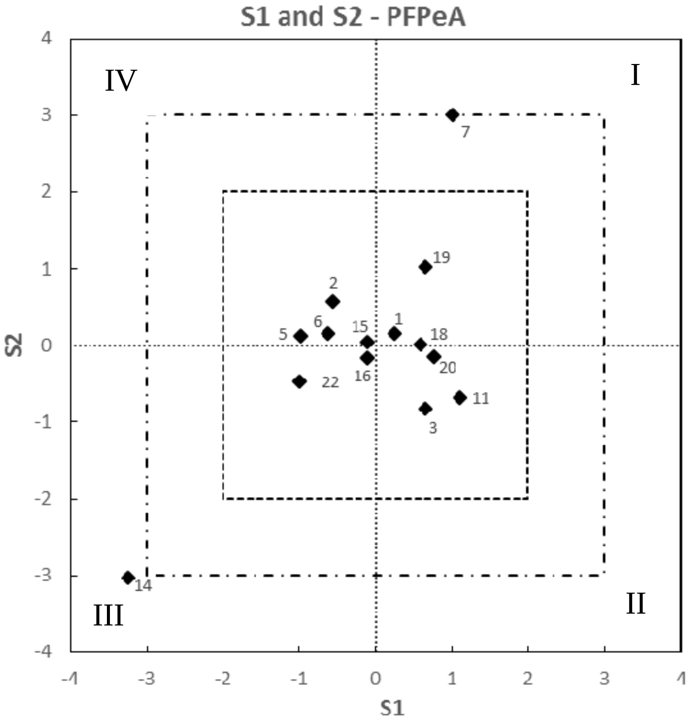
Laboratory 17 is off scale.

Figure 49 S1 (Fish) and S2 (Tomato) z-score scatter plot PFOS



Laboratory 17 is off scale.

Figure 50 S1 (Fish) and S2 (Tomato) z-score scatter plot PFOSA



Laboratories 17 and 21 are off scale.

Figure 51 S1 (Fish) and S2 (Tomato) z-score scatter plot PFPeA

6.4 E_n-Score

Where a laboratory did not report an uncertainty estimate an uncertainty of zero (0) was used to calculate the E_n-score.

Of five hundred and twenty-two E_n-scores, 390 (75%) were satisfactory with $|E_n| \leq 1$.

No laboratories which analysed two matrices returned satisfactory E_n-scores for all analytes reported (35) and for which E_n-scores were calculated. Laboratory **3** returned 34 satisfactory E_n-scores.

Laboratory **9** which analysed one matrix (S1 Fish) returned satisfactory E_n-score for all analytes reported (21) and for which E_n-scores were calculated.

Laboratory **14** returned unsatisfactory E_n-scores for all analytes tested (30) for which E_n-scores were calculated. After the interim report was published, this laboratory reported that an incorrect dilution factor was applied, with all results out by a factor of 2.5. Laboratory **8** returned unsatisfactory E_n-scores for all analytes tested (4) for which E_n-scores were calculated.

The dispersal of participants' E_n-scores is presented in Figure 41.

6.5 Participants' Methods

Participants were requested to analyse the samples using their normal test method and to report a single result as they would normally report to a client. Results reported in this way reflect the true variability of results reported to clients. The method descriptions provided by participants are presented in Tables 2 to 5. The study coordinator thanks all laboratories that completed the method questionnaire. A summary is presented below.

Fish sample
Pre-treatment: homogenisation (15), pH adjustment (2), alkaline digestion (1).

Extraction technique: alkaline digestion (6), solid-liquid extraction (7), QuEChERS (2), solid phase extraction (5), sonication (1).
Extraction process: sonication (12), shaking (12), tumbling (4), vortex (3), SPE (1), QuEChERS (1).
Clean-up: carbon (7), SPE (16), centrifugation (2), liquid-liquid extraction (1).
Extraction solvent: methanol/base e.g. KOH, NaOH (6), methanol (2), acetonitrile (10), acetonitrile/other (2).

Tomato sample
Pre-treatment: homogenisation (13), desiccation (1), alkaline digestion (1).
Extraction technique: alkaline digestion (5), solid-liquid extraction (4), solid phase extraction (5), QuEChERS (3), Soxhlet (1).
Extraction process: sonication (8), shaking (10), vortex (3), tumbling (4), QuEChERS (1), hot extraction (1), centrifuge (1), SPE (1).
Clean-up: carbon (6), SPE (12), centrifugation (2)
Extraction solvent: methanol/base e.g. KOH, NaOH (5), methanol (3), acetonitrile (7), acetonitrile/other (2).

For all matrices the analytical detection method of choice was LC-MS (with a variety of mass analysers QQQ and Orbitrap).

Due to the limited amount of data and the variety of analytical methods used no significant trend with extraction and sample preparation was identified.

6.6 Effects of Sample Matrix

Sample S1 was fish, and Sample S2 was tomato. The tomato sample had a higher percentage of results for which z-scores were calculated. The fish and tomato samples received similar number of percent satisfactory z-scores.

Table 47 Satisfactory z-scores for each matrix

Sample		Expected number of z-scores	Actual number of z-scores (% of expected no of z-scores)	Satisfactory	% satisfactory
S1 Fish	Spiked	483	312 (65%)	273	88
S2 Tomato	Spiked	252	210 (83%)	178	85

6.7 False Negatives

Table 48 lists false negative results – an analyte for which a laboratory tested but did not report a result (e.g. laboratories reporting as '<' result when the assigned value was significantly higher than the < figure).

Table 48 False Negatives

Lab Code	Sample	Analyte
6	S1	PFNA
7	S1	MeFOSE
14	S2	PFNA
17	S1	EtFOSA
18	S1	6:2 FTS
20	S2	6:2 FTS

7 REFERENCES

[1] ISO/IEC 17043 2010, *Conformity assessment – General requirements for proficiency testing*.

[2] *Chemical Proficiency Testing Study Protocol,* viewed 21 July 2019, <http://www.measurement.gov.au>.

[3] *Chemical Proficiency Testing Statistical Manual*, viewed 21 July 2019, <http://www.measurement.gov.au >.

[4] Thompson, M., Ellison, SLR. & Wood, R. 2005. 'The international harmonized protocol for proficiency testing of (chemical) analytical laboratories', *Pure Appl. Chem*, vol 78, pp 145-196

[5] ISO/IEC 17025 2017, *General requirements for the competence of testing and calibration laboratories.*

[6] Eurachem 2012, *Quantifying Uncertainty in Analytical Measurement*, 3rd edition, viewed 10 May 2017, <http://www.eurachem.org/images/stories/Guides/pdf/QUAM2012_P1.pdf>.

[7] ISO13528:2015(E), *Statistical methods for use in proficiency testing by interlaboratory comparisons*.

[8] Thompson, M. and Lowthian, P.J. 1995. 'A Horwitz-like function describes precision in a proficiency test', *Analyst*, vol 120, pp 271-272.

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

APPENDIX 1 - SAMPLE PREPARATION, HOMOGENEITY TESTING AND STABILITY CHECK

A1.1 Sample Preparation

Analytical standards used for spiking samples in AQA 19-20 were purchased from HPC Standards GmbH, Toronto Research Chemicals, Sigma-Aldrich and Wellington Laboratories Canada. On the analytical reports provided with the standards, all analytes have a stated purity of ≥95%.

Sample S1: Contaminated European Carp from a control site was composited and blended. Approximately 368 g of fish composite was sprayed with a spiking solution containing an 11 PFAS analytes using a commercial pump. This was repeated until all spiking solution was used. The fish was thoroughly mixed.

Sample S1 was placed on stainless steel trays as patties of no more than six centimeters square and frozen overnight in -80ºC freezer. The patties of fish were embrittled by immersion in liquid nitrogen and then passed through a Retsch SM2000 mill that was cooled with liquid nitrogen and dry ice. After grinding, samples were placed in a refrigerator in a covered Pyrex dish. The next day after all the carbon dioxide sublimed, 5 g portions of fish were packed into labelled 50 mL Greiner tubes.

Sample S2: Tomatoes were bought from a Sydney organic fruit and vegetable wholesaler. Samples were rinsed using tap water and allowed to air dry.

The whole tomato, including the peel, was chopped, pureed and passed through an 850 µm sieve. After blending the pureed was passed through an 850 µm sieve. Each sieved puree was continuously stirred while 40 mL aliquots were dispensed into 50 mL Greiner tubes to provide unspiked samples.

The remaining puree was spiked with 14 PFAS analytes, stirred for at least two hours and 40 mL dispensed in 50 mL Greiner tubes. Each sample was then labelled and shrink-wrapped.

Fish samples were stored at -80ºC and tomato at -20 ºC prior to dispatch to participants.

A1.2 Homogeneity Testing for Tomato Sample S2

Homogeneity testing was based on that described in the International Protocol.[4]

Tomato samples were prepared (see A2.1) and analysed at NMI North Ryde. A brief description of the analysis method is presented below. The measurements were made under repeatability conditions in random order. The mean results were used as the NMI homogeneity value.

Samples were prepared in duplicate by accurately weighing 1 g of the sample then spiking with 30 µL of labelled internal standard in methanol. The samples were extracted by overnight tumbling in alkaline methanol (0.01 N potassium hydroxide), then centrifuged and a portion was purified by passing through activated carbon (SUPLCLEAN ENVI-CARB, 500 mg, 120-400 Mesh) eluted using methanol. After evaporation under nitrogen, the extract was reconstituted to 600 µL in mobile phase and spiked with 10 µL labelled recovery standard in methanol. All chemicals are analytical reagents or LCMS grade solvents. Instrument analysis was performed using an Ultra Performance Liquid Chromatography (UPLC) coupled with a Liquid Chromatography Qtrap Mass spectrometer (ABSciex 6500+), operating in multiple reaction monitoring mode. 1 µL of extract was injected onto a Waters Acquity BEH C18 column (2.1 mm x 100 mm x 1.7 µm, 130 Å) with a mobile phase gradient consisting of water:methanol (2 mM ammonium acetate). Two mass transitions were monitored for each target analyte and labelled internal standard, and abundance ratios checked. The instrument mass accuracy is calibrated annually during preventative maintenance, and the six point calibration curve established for each analytical batch. A solvent batch blank is extracted and analysed with each batch, and sample results must be at least three times the level of any analyte detected in the batch blank to be reported. Quantification is based on the use of the labelled internal standards using relative retention factors from the multipoint calibration, and is corrected for internal standard recoveries. The analysis is based on USEPA Method 537 and used calibration, internal and recovery standards supplied by Wellington Laboratories, Canada.

Results of the tomato homogeneity testing are presented in tables 49 to 60. For each sample, the mean of 10 measurements were used as the homogeneity value. All samples were found to be sufficiently homogeneous for use in this PT study.

Table 49 Sample **S2**, PFBA homogeneity testing results

Bottle Fill Number	PFBA µg/kg	
	Replicate 1	Replicate 2
5	46.97	49.09
10	45.68	40.64
11*	55.82	41.36
14	45.59	51.13
23	49.63	42.33
30	48.76	46.50
46	48.92	47.94
Mean	46.9	
CV	6.5%	

*Results on bottle fill 11 were not included in the test for homogeneity being identified as analytical outliers (test Sa/σ) due to the difference between replicates.[9]

Thompson and Fearn Homogeneity Tests[9]

Test	Value	Critical	Result
Cochran	0.44	0.78	**Pass**
S_{an}/σ	0.34	0.5	**Pass**
s^2_{sam}	0.000	34.51	**Pass**

Table 50 Sample **S2**, PFBS homogeneity testing results

Bottle Fill Number	PFBS µg/kg	
	Replicate 1	Replicate 2
5	24.01	25.66
10	23.30	24.29
11	26.27	25.12
14	24.42	25.03
23	25.18	24.55
30	25.14	24.84
46	24.01	23.85
Mean	24.7	
CV	3.2%	

Thompson and Fearn Homogeneity Tests[9]

Test	Value	Critical	Result
Cochran	0.46	0.73	**Pass**
S_{an}/σ	0.13	0.5	**Pass**
s^4_{sam}	0.209	5.22	**Pass**

Table 51 Sample **S2**, PFHpA homogeneity testing results

Bottle Fill Number	PFHpA µg/kg	
	Replicate 1	Replicate 2
5	11.26	12.41
10	12.89	13.62
11	14.38	12.16
14	13.11	12.47
23	12.47	12.78
30	12.54	12.50
46	12.33	12.90
Mean	12.7	
CV	5.6%	

Thompson and Fearn Homogeneity Tests[9]

Test	Value	Critical	Result
Cochran	0.65	0.73	**Pass**
S_{an}/σ	0.29	0.5	**Pass**
s^2_{sam}	0.000	2.00	**Pass**

Table 52 Sample **S2**, PFHxA homogeneity testing results

Bottle Fill Number	PFHxA µg/kg	
	Replicate 1	Replicate 2
5	25.14	26.28
10	25.84	25.73
11	27.49	26.60
14	26.77	27.02
23	26.55	24.98
30	25.34	25.75
46	26.70	26.32
Mean	26.2	
CV	2.8%	

Thompson and Fearn Homogeneity Tests[9]

Test	Value	Critical	Result
Cochran	0.50	0.73	**Pass**
S_{an}/σ	0.11	0.5	**Pass**
s^4_{sam}	0.212	5.68	**Pass**

Table 53 Sample **S2**, PFHxS homogeneity testing results

Bottle Fill Number	PFHxS µg/kg	
	Replicate 1	Replicate 2
5	10.18	10.98
10	10.16	12.62
11	11.61	10.77
14	10.56	10.97
23	11.06	10.08
30	12.68	12.34
46	10.66	10.63
Mean	11.1	
CV	8.0%	

Thompson and Fearn Homogeneity Tests[9]

Test	Value	Critical	Result
Cochran	0.70	0.73	**Pass**
S_{an}/σ	0.35	0.5	**Pass**
s^4_{sam}	0.181	1.81	**Pass**

Table 54 Sample **S2**, PFNA homogeneity testing results

Bottle Fill Number	PFNA µg/kg	
	Replicate 1	Replicate 2
5	0.93	0.98
10	1.15	1.15
11	0.94	0.94
14*	1.26	0.96
23	1.14	1.06
30	1.03	1.05
46	0.99	0.90
Mean	1.0	
CV	8.9%	

*Results on bottle fill 14 were not included in the test for homogeneity being identified as analytical outliers (test Sa/σ) due to the difference between replicates.[9]

Thompson and Fearn Homogeneity Tests[9]

Test	Value	Critical	Result
Cochran	0.48	0.78	**Pass**
S_{an}/σ	0.18	0.5	**Pass**
s^4_{sam}	0.007	0.01	**Pass**

Table 55 Sample **S2**, PFOA homogeneity testing results

Bottle Fill Number	PFOA µg/kg	
	Replicate 1	Replicate 2
5	4.64	4.87
10	4.27	4.14
11	4.29	4.56
14	4.56	4.74
23	4.29	3.77
30	4.99	4.44
46	3.68	4.48
Mean	4.4	
CV	8.5%	

Thompson and Fearn Homogeneity Tests[9]

Test	Value	Critical	Result
Cochran	0.46	0.73	**Pass**
S_{an}/σ	0.36	0.5	**Pass**
s^2_{sam}	0.044	0.29	**Pass**

Table 56 Sample **S2**, PFOS homogeneity testing results

Bottle Fill Number	PFOS µg/kg	
	Replicate 1	Replicate 2
5	2.44	2.51
10	2.29	2.38
11	2.67	2.42
14	2.40	2.59
23	2.31	2.35
30	2.63	2.46
46	2.36	2.44
Mean	2.4	
CV	4.8%	

Thompson and Fearn Homogeneity Tests[9]

Test	Value	Critical	Result
Cochran	0.41	0.73	**Pass**
S_{an}/σ	0.21	0.5	**Pass**
s^2_{sam}	0.003	0.06	**Pass**

Table 57 Sample **S2**, PFOSA homogeneity testing results

Bottle Fill Number	PFOSA µg/kg	
	Replicate 1	Replicate 2
5	4.29	4.48
10	4.05	4.19
11	4.76	4.21
14	4.23	4.23
23	4.31	4.22
30	4.42	4.44
46	4.10	4.52
Mean	4.3	
CV	4.3%	

Thompson and Fearn Homogeneity Tests[9]

Test	Value	Critical	Result
Cochran	0.56	0.73	**Pass**
S_{an}/σ	0.23	0.5	**Pass**
s^2_{sam}	0.000	0.20	**Pass**

Table 58 Sample **S2**, PFPeA homogeneity testing results

Bottle Fill Number	PFPeA µg/kg	
	Replicate 1	Replicate 2
5	51.05	52.22
10	47.61	51.07
11	53.99	51.12
14	49.91	50.53
23	51.33	50.20
30	50.70	51.05
46	48.96	51.49
Mean	50.8	
CV	2.9%	

Thompson and Fearn Homogeneity Tests[9]

Test	Value	Critical	Result
Cochran	0.40	0.73	**Pass**
S_{an}/σ	0.14	0.5	**Pass**
s^2_{sam}	0.037	22.54	**Pass**

Table 59 Sample **S2**, 6:2 FTS homogeneity testing results

Bottle Fill Number	6:2 FTS µg/kg	
	Replicate 1	Replicate 2
5	8.49	9.99
10	8.94	9.75
11	9.41	9.89
14	9.49	10.08
23	8.53	8.72
30	8.86	8.88
46	8.92	8.78
Mean	9.2	
CV	6.0%	

Thompson and Fearn Homogeneity Tests[9]

Test	Value	Critical	Result
Cochran	0.63	0.73	**Pass**
S_{an}/σ	0.27	0.5	**Pass**
s^2_{sam}	0.060	1.00	**Pass**

Table 60 Sample **S2**, 8:2 FTS homogeneity testing results

Bottle Fill Number	8:2 FTS µg/kg	
	Replicate 1	Replicate 2
5	9.23	10.16
10	9.69	10.44
11	10.42	10.10
14	10.33	9.58
23	9.57	9.54
30	10.47	10.50
46	10.92	10.47
Mean	10.1	
CV	4.9%	

Thompson and Fearn Homogeneity Tests[9]

Test	Value	Critical	Result
Cochran	0.37	0.73	**Pass**
S_{an}/σ	0.20	0.5	**Pass**
s^2_{sam}	0.085	1.00	**Pass**

A1.3 Stability Testing for Tomato Sample S2

A stability study was conducted for tomato Sample S2. Short term stability and stability during transportation was assessed by comparing the average of duplicate measurements performed on each sample, kept at room temperature, starting July 2019 and ending September 2019. Results were in good agreement with each other, with the spiked level and the assigned value (Figure 52).

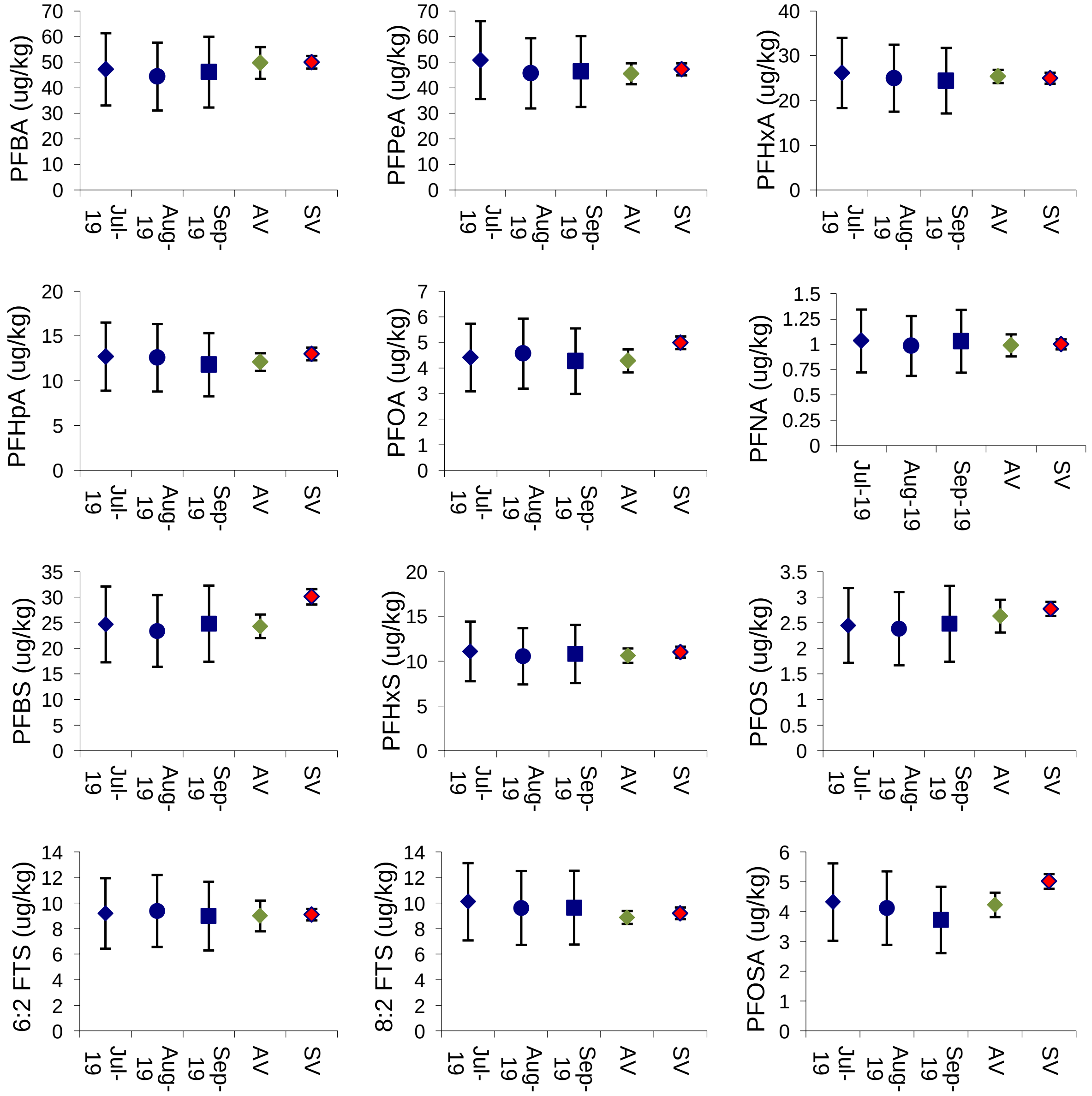


Figure 52 Stability results, assigned value (AV), and spiked value (SV) in spiked tomato Sample S2 (room temperature)

APPENDIX 2 - ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY

The robust average was calculated using the procedure described in 'ISO13528:2015(E), Statistical methods for use in proficiency testing by interlaboratory comparisons – Annex C'[7] the uncertainty was estimated as:

$$u_{\text{rob av}} = 1.25 * S_{rob\ av} / \sqrt{p} \qquad \text{Equation 4}$$

where:

$u_{\text{rob av}}$	robust average standard uncertainty
$S_{rob\ av}$	robust average standard deviation
p	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 61.

Table 61 Uncertainty Estimate for PFDA in Sample S1

No. results (p)	19
Robust Average	2.506 µg/kg
$S_{rob\ av}$	0.349 µg/kg
$u_{rob\ av}$	0.100 µg/kg
k	2
$U_{rob\ av}$	0.200 µg/kg

The robust average for PFDA in Sample S1 is 2.51 ± 0.20 µg/kg.

APPENDIX 3 - ACRONYMS AND ABBREVIATIONS

10:2 FTS	1H, 1H, 2H, 2H-perfluorododecane sulfonate
6:2 FTS	1H, 1H, 2H, 2H-perfluorooctane sulfonate
8:2 FTS	1H, 1H, 2H, 2H-perfluorodecane sulfonate
ADONA	Ammonium 4,8-dioxa-3H-perfluorononanoate
AV	Assigned Value
CRM	Certified Reference Material
CV	Coefficient of Variation
EPA	Environment Protection Authority
EtFOSA	N-Ethyl perfluorooctane sulfonamide
EtFOSAA	N-Ethyl perfluorooctane sulfonamido acetic acid
EtFOSE	N-Ethyl perfluorooctane sulfonamidoethanol
FOSA	Perfluoro-1-octanesulfonamide
GenX	Ammonium 2,3,3,3-tetrafluoro-2-(heptafluoropropoxy) propanoate
GUM	Guide for Uncertainty Measurement
HV	Homogeneity Value
ISO	International Standards Organisation
LC	Liquid Chromatography
Max	Maximum value in a set of results
Md	Median
MeFOSA	N-Methyl perfluorooctane sulfonamide
MeFOSAA	N-Methyl perfluorooctane sulfonamidoacetic acid
MeFOSE	N-Methyl perfluorooctane sulfonamidoethanol
Min	Minimum value in a set of results
MS	Mass Spectrometry
NMI	National Measurement Institute (of Australia)
NR	Not Reported
NT	Not Tested
PFAS	Per- and poly fluorinated alkyl substances
PFBA	Perfluoro-n-butanoic acid
PFBS	Potassium perfluoro-1-butanesulfonate
PFDA	Perfluoro-n-decanoic acid
PFDoA	Perfluorododecanoic acid
PFDS	Perfluorodecane sulfonate
PFHpA	Perfluoro-n-heptanoic acid

PFHpS	Perfluoroheptane sulfonate
PFHxA	Perfluoro-n-hexanoic acid
PFHxS	Potassium perfluorohexanesulfonate
PFNA	Perfluoro-n-nonanoic acid
PFNS	Perfluorononane sulfonate
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctane sulfonate
PFOSA	Perfluoro-1-octanesulfonamide
PFPeA	Perfluoro-n-pentanoic acid
PFPeS	Perfluoropentane sulfonate
PFTeDA	Perfluorotetradecanoic acid
PFTrA	Perfluorotridecanoic acid
PFUnA	Perfluoroundecanoic acid
PT	Proficiency Test
Q	Quadrupole mass analyser
QQQ	Triple Quadrupole (mass spectrometry)
QuEChERS	Quick, Easy, Cheap, Effective, Rugged and Safe extraction method
RA	Robust Average
Robust CV	Robust Coefficient of Variation
Robust SD	Robust Standard Deviation
SV	Spiked or formulated concentration of a PT sample (Spike Value)
SPE	Solid Phase Extraction
Target SD	Target standard deviation
σ	Target standard deviation

End of Report