Australian Government
Department of Industry, Science, Energy and Resources

National Measurement Institute

Proficiency Test Final Report AQA 21-17 Hydrocarbons in Water

March 2022

ACKNOWLEDGMENTS

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

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

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

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SUMMARY

AQA 21-17 Hydrocarbons in Water commenced in October 2021. Eighteen laboratories registered to participate and all participants submitted results.

The sample set consisted of four water samples. Samples were prepared in the Sydney NMI laboratory using surface water from Browns Waterhole in Sydney. Participants measured total recoverable hydrocarbons (TRH) in Sample S1, volatile hydrocarbons (C6 to C10), benzene, toluene, ethylbenzene and xylenes (BTEX) in Sample S2 and polycyclic aromatic hydrocarbons (PAHs) in Samples S3 and S4.

The assigned values for all scored analytes were the robust averages of participants' results. The associated uncertainties were estimated from the robust standard deviations of 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:

- *Compare the performances of participants and assess their accuracy in the identification and measurement of petroleum hydrocarbon pollutants in water.*

Laboratories **1**, **2**, **3**, **5**, **6**, **7**, **12**, **13**, **14**, **15**, **16** and **18** reported results for all 18 scored analytes. For Sample S1, some participants reported hydrocarbon ranges outside of the recommended National Environment Protection Measure (NEPM) ranges. Three laboratories did not report numeric results for analytes which they tested for and were present in the test samples (total of 14 results).

Of 288 z-scores, 252 (88%) returned a score of $|z| \leq 2.0$, indicating a satisfactory performance.

Of 288 E_n-scores, 231 (80%) returned a score of $|E_n| \leq 1.0$, indicating agreement of the participant's result with the assigned value within their respective expanded uncertainties.

Laboratories **1**, **5**, **7**, **12**, **14**, **15**, and **16** returned satisfactory z-scores and E_n-scores for all 18 scored analytes.

- *Evaluate the laboratories' test methods.*

For TRH analysis, participants used liquid-liquid extraction with various extraction solvents. GC-FID was the instrument of choice, though one participant reported using GC-MS.

For BTEX analysis, ten participants used purge-and-trap GC-MS(/MS) and five participants used headspace GC-MS(/MS). Two participants reported using liquid-liquid extraction also.

For PAHs analysis, one participants used solid phase extraction, while all other participants used liquid-liquid extraction. A variety of extraction solvents were used. All participants used GC-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.*

Of 327 numeric results, 296 (91%) were reported with an associated expanded measurement uncertainty. Reported expanded uncertainties were within the range of 3.2% to 100% relative.

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

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

1 INTRODUCTION

1.1 NMI Proficiency Testing Program

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

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

1.2 Study Aims

The aims of the study were to:

- compare the performances of participants and assess their accuracy in the identification and measurement of petroleum hydrocarbon pollutants in water;
- evaluate the laboratories' test methods;
- develop the practical application of traceability and measurement uncertainty, and provide participants with information that will be useful in assessing their uncertainty estimates; and
- produce materials that can be used in method validation and as control samples.

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

1.3 Study Conduct

The conduct of NMI proficiency tests is described in the NMI Study Protocol for Proficiency Testing.[2] The statistical methods used are described in the NMI Chemical Proficiency Testing Statistical Manual.[3] These documents have been prepared with reference to ISO/IEC 17043:2010,[1] and The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.[4]

NMI is accredited by the National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043 as a provider of proficiency testing schemes.[1] This study falls within the scope of NMI's accreditation.

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of this study was:

Invitation issued	5 October 2021
Samples dispatched	9 November 2021
Results due	15 December 2021
Interim report issued	5 January 2022

2.2 Participation

Eighteen laboratories registered to participate, and all participants were assigned a confidential laboratory code number for this study. All participants submitted results.

2.3 Selection of Hydrocarbon Analytes

The hydrocarbons and their concentrations in this study were typical of those encountered by environmental testing laboratories monitoring water to assess the impact of transport fuels in the environment, or the contamination from industry that entails the use of wood, petroleum or coal to generate heat and power.

Investigation levels for the hydrocarbons studied are set out in the National Environmental Protection (Assessment of Site Contamination) Measure, Schedule B1 *Guideline on Investigation Levels for Soil and Groundwater*.[5]

Participants were provided with a list of potential PAHs spiked into Samples S3 and S4 (Table 1).

Table 1 Possible Spiked PAHs in Samples S3 and S4

Naphthalene	Fluorene	Benz[a]anthracene	Benzo[a]pyrene
Acenaphthylene	Phenanthrene	Chrysene	Indeno[1,2,3-cd]pyrene
Acenaphthene	Fluoranthene	Benzo[b]fluoranthene	Dibenz[a,h]anthracene
Anthracene	Pyrene	Benzo[k]fluoranthene	Benzo[g,h,i]perylene

2.4 Test Material Preparation

Four test samples were prepared by spiking water from a local river. Sample S1 was spiked with diesel fuel, Sample S2 was spiked with unleaded petrol and diesel fuel, and Samples S3 and S4 were spiked with differing amounts of anthracene, benzo(a)pyrene, fluoranthene, fluorene, phenanthrene and pyrene.

The spiked concentrations in each sample is presented in Table 2.

Table 2 Spiked Concentrations of Samples

Sample	Analyte	Spiked Value (µg/L)	Uncertainty* (µg/L)
S1	TRH	1970	100
S2	Benzene	67.4	3.4
	Toluene	285	14
	Ethylbenzene	17.9	0.9
	Xylenes	134	7
	Total BTEX	504	25

Sample	Analyte	Spiked Value (µg/L)	Uncertainty* (µg/L)
S3	Anthracene	3.52	0.18
	Benzo(a)pyrene	8.48	0.42
	Fluoranthene	12.0	0.6
	Fluorene	6.07	0.30
	Phenanthrene	3.03	0.15
	Pyrene	8.02	0.40
S4	Anthracene	8.99	0.45
	Benzo(a)pyrene	0.921	0.046
	Fluoranthene	3.00	0.15
	Fluorene	11.0	0.5
	Phenanthrene	5.02	0.25
	Pyrene	7.02	0.35

* Estimated expanded uncertainty at approximately 95% confidence using a coverage factor of 2. Stability was not considered and so the expanded uncertainty is related to the concentration of the analyte at the time of spiking.

Additional information on sample preparation is given in Appendix 1.

2.5 Homogeneity and Stability of Test Materials

All samples were prepared and packaged using a process that has been demonstrated to produce homogeneous samples in previous NMI Hydrocarbons in Water PT studies. No homogeneity testing was conducted for this study, and participants' results gave no reason to question the homogeneity of these samples.

The storage stability of petroleum hydrocarbons spiked into water samples has been previously established.[6] No stability study was conducted for this study, and to assess possible instability, the results returned by participants were compared to the spiked concentration.

For Sample S1 TRH, the robust average was 68% of the spiked value. This is similar to values observed in previous Hydrocarbons in Water PT studies, and as there was a reasonable consensus between participants' results, an assigned value was set.

For Sample S2 BTEX, the robust averages were between 82% and 102% of the spiked values, providing good support for the stability of these analytes.

For Samples S3 and S4 PAHs, the robust averages of scored analytes were between 66% and 78% of the spiked values, and these were similar to values observed in previous Hydrocarbons in Water PT studies. Assigned values were set where there was a reasonable consensus between participants' results.

2.6 Sample Storage, Dispatch and Receipt

The test samples were stored in a cool room at approximately 4 °C prior to dispatch. Samples were dispatched on 9 November 2021.

The following items were also sent to participants:

- 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 test samples.

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

2.7 Instructions to Participants

Participants were instructed as follows:

- Quantitatively analyse the samples using your routine test method.
- Do not test for volatile hydrocarbons (C6-C10) or BTEX components in Sample S1.
- Participants need not test for all listed analytes.
- For each analyte in each sample, report a single result in units of µg/L expressed as if reporting to a client, applying the limit of reporting of the method used for analysis. This is the figure that will be used in all statistical analysis in the study report.
- For each analyte in each sample, report the associated expanded uncertainty in units of µg/L (e.g. 2000 ± 200 µg/L), if determined.
- Report results for the following:
    - Sample S1: Semi-volatile hydrocarbons (>C10-C40) and Total Recoverable Hydrocarbons (TRH). Use your laboratory's chosen quantitation range, and indicate what this range is. Australian NEPM fractions >C10-C16, >C16-C34, >C34-C40 are encouraged. The concentration range is between 200 – 10000 µg/L.
    - Sample S2: Volatile Hydrocarbons (C6-C10), Benzene, Toluene, Ethylbenzene, Total Xylenes and Total BTEX. Individual BTEX components concentration is between 0.2 – 800 µg/L.
    - Samples S3 and S4: PAHs. The concentration range is between 0.05 – 50 µg/L.
- Give details of your methodology and basis of uncertainty estimate as requested by the results sheet emailed to you.
- Return the completed results sheet by 6 December 2021 by email to proficiency@measurement.gov.au.

The results due date was extended to 15 December 2021 due to sample delivery delays to some participants.

2.8 Interim Report

An interim report was emailed to all participants on 5 January 2022.

The interim report was delayed due to an extension granted to a participant because of exceptional circumstances, as well as the NMI end-of-year shut down period.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Participants' Test Methods

Participants were requested to provide information about their test methods. Responses are presented in Appendix 2.

3.2 Basis of Participants' Measurement Uncertainty Estimates

Participants were requested to provide information about their basis of measurement uncertainty (MU). Responses are presented in Table 3.

Table 3 Basis of Uncertainty Estimate

Lab. Code	Analyte	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
			Precision	Method Bias	
1	All	Top Down - precision and estimates of the method and laboratory bias	Control samples Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS Standard purity	NMI Uncertainty Course
2	All	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS	Recoveries of SS	NATA General Accreditation Guidance Estimating and Reporting Measurement Uncertainty of Chemical Test Results
3	All	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM	CRM Instrument calibration Recoveries of SS	ISO/GUM
4	All	Replicate data during validation			
5	All	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM	CRM Instrument calibration Recoveries of SS	NMI Uncertainty Course
6	All	Bottom Up (ISO/GUM, fish bone/cause and effect diagram)	Duplicate analysis	Laboratory bias from PT studies Instrument calibration Recoveries of SS Standard purity	ISO/GUM
7	All	Top Down - precision and estimates of the method and laboratory bias	Control samples Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS	Eurachem/CITAC Guide
8	TRH / PAH	Standard deviation of replicate analyses multiplied by 2 or 3	Duplicate analysis	Recoveries of SS	ISO/GUM
9	All	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS Duplicate analysis Instrument calibration	CRM Instrument calibration Recoveries of SS Standard purity	Eurachem/CITAC Guide

Lab. Code	Analyte	Approach to Estimating MU	Information Sources for MU Estimation*		Guide Document for Estimating MU
			Precision	Method Bias	
10	All	Standard deviation of replicate analyses multiplied by 2 or 3	Duplicate analysis		Eurolab Technical Report No1/2007
11	All	Based on historical data	Duplicate analysis Instrument calibration	Instrument calibration Standard purity	Eurachem/CITAC Guide
12	All	SD of replicate analyses x 2 x 100/85 x 100/mean	Control samples - CRM	CRM Instrument calibration	
13	All	Top Down - precision and estimates of the method and laboratory bias	Instrument calibration	CRM	NATA General Accreditation Guidance Estimating and Reporting Measurement Uncertainty of Chemical Test Results
14	All	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM Duplicate analysis Instrument calibration	CRM Instrument calibration Recoveries of SS	NATA General Accreditation Guidance Estimating and Reporting Measurement Uncertainty of Chemical Test Results
15	All	Standard deviation of replicate analyses multiplied by 2 or 3	Control samples Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS	Eurachem/CITAC Guide
16	All	Top Down - precision and estimates of the method and laboratory bias	Control samples - SS		ISO/GUM
17	TRH				
18	All	Top Down - precision and estimates of the method and laboratory bias	Control samples - CRM	CRM	ISO/GUM

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

3.3 Participants' Comments

Participants were invited to make any comments or suggestions on the samples, this study, or possible future studies. Such feedback may be useful in improving future studies. Participants' comments are presented in Table 4.

Table 4 Participants' Comments

Lab. Code	Sample	Participant's Comments
11	S2	C6-C9 result reported above.
15	S3 and S4	PAH results are not corrected for recovery of analytes.
16	S3 and S4	Uncertainty measured using Control chart from 26/08/21 to 26/11/2021

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 5 to 27 with summary statistics: robust average, median, mean, number of numeric results (N), maximum (Max.), minimum (Min.), robust standard deviation (Robust SD) and robust coefficient of variation (Robust CV). Bar charts of results and performance scores are presented in Figures 2 to 22. An example chart with interpretation guide is shown in Figure 1.

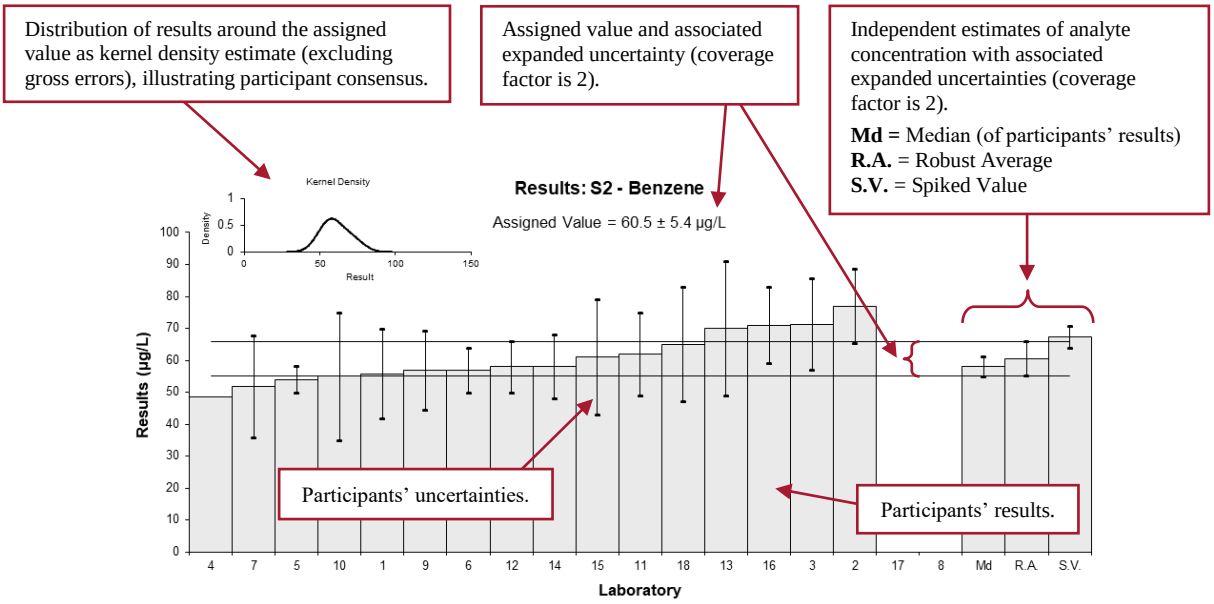


Figure 1 Guide to Presentation of Results

4.2 Outliers and Gross Errors

Outliers were results less than 50% and greater than 150% of the robust average, and these were removed before the calculation of the assigned value.[3,4] Gross errors were obvious blunders, e.g. results reported with incorrect units or basis, or for a different analyte or sample, and such results were removed for the calculation of all summary statistics.[3,4]

4.3 Assigned Value

The assigned value is defined as the 'value attributed to a particular property of a proficiency test item'.[1] In this PT study, the property is the concentration of the analytes in the samples. Assigned values were the robust averages of participants' results, and the expanded uncertainties were estimated from the associated robust SDs (Appendix 3).

4.4 Robust Average and Robust Between Laboratory Coefficient of Variation

The robust averages and associated expanded MUs, and robust CVs (a measure of the variability of participants' results) were calculated using the procedure described in ISO 13528:2015.[7]

4.5 Performance Coefficient of Variation

The performance coefficient of variation (PCV) is a fixed measure of the between-laboratory variation that in the judgement of the study coordinator would be expected from participants, given the analyte concentrations. The PCV is not the CV of participants' results; it is set by the study coordinator and is based on the concentration of the analytes and experience from previous studies, and is supported by mathematical models such as the Thompson-Horwitz equation.[8] By setting a fixed and realistic value for the PCV, a participant's performance does not depend on other participants' performances and can be compared from study to study.

4.6 Target Standard Deviation

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

$$\sigma = X \times PCV \qquad \textit{Equation 1}$$

4.7 z-Score

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

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

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

For the absolute value of a z-score (|z|):

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

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

4.8 E_n-Score

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

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

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

For the absolute value of an E_n-score ($|E_n|$):

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

4.9 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC 17025 must establish and demonstrate the traceability and measurement uncertainty associated with their test results.[9]

Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.[10]

5 TABLES AND FIGURES

Table 5

Sample Details

Sample No.	S1
Matrix	Water
Analyte	>C10-C16
Units	µg/L

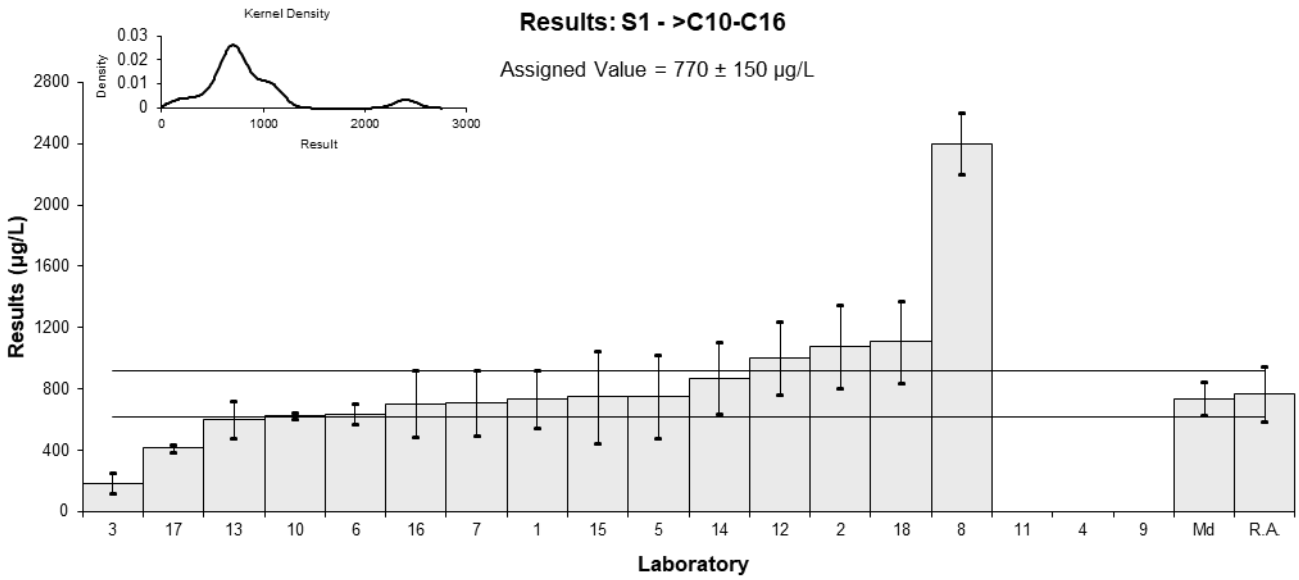
Participant Results

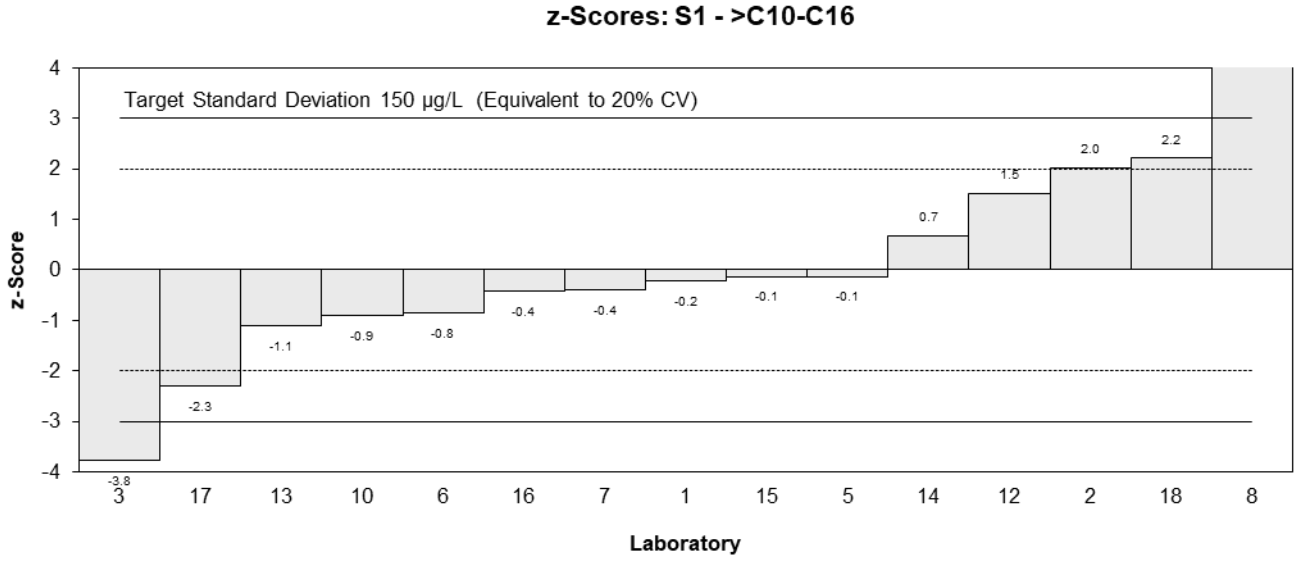
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	736	184	-0.22	-0.14
2	1081	270	2.02	1.01
3	188	65.8	-3.78	-3.55
4	NR	NR		
5	750	270	-0.13	-0.06
6	640	70	-0.84	-0.79
7	710	210	-0.39	-0.23
8	2400	200	10.58	6.52
9	NR	NR		
10	630	20	-0.91	-0.93
11	NR	NR		
12	1003	240	1.51	0.82
13	600	120	-1.10	-0.88
14	873	233	0.67	0.37
15	750	300	-0.13	-0.06
16	705	215	-0.42	-0.25
17	415.7	21.8	-2.30	-2.34
18	1110	266.5	2.21	1.11

Statistics

Assigned Value*	770	150
Robust Average	770	180
Median	740	110
Mean	840	
N	15	
Max.	2400	
Min.	188	
Robust SD	270	
Robust CV	36%	

* Robust average excluding Laboratories 3 and 8.


Kernel Density
Density
Result
Results: S1 - >C10-C16
Assigned Value = 770 ± 150 µg/L
Results (µg/L)
Laboratory
3 17 13 10 6 16 7 1 15 5 14 12 2 18 8 11 4 9 Md R.A.


z-Scores: S1 - >C10-C16
Target Standard Deviation 150 µg/L (Equivalent to 20% CV)
z-Score
Laboratory
3 17 13 10 6 16 7 1 15 5 14 12 2 18 8

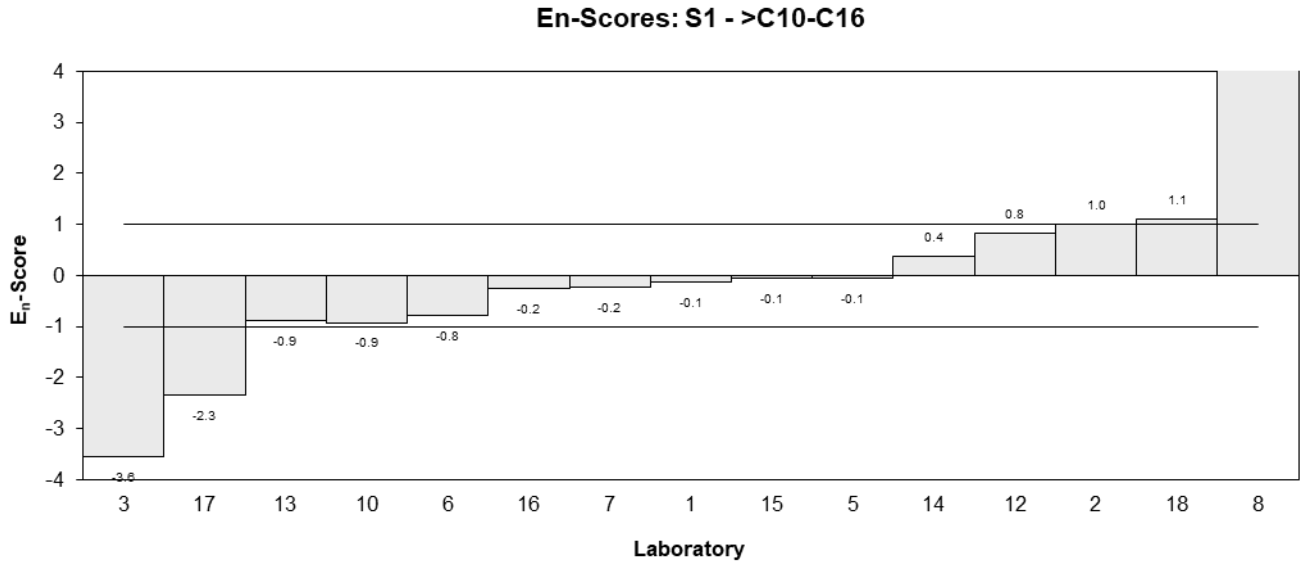

En-Scores: S1 - >C10-C16
En-Score
Laboratory
3 17 13 10 6 16 7 1 15 5 14 12 2 18 8

Figure 2

Table 6

Sample Details

Sample No.	S1
Matrix	Water
Analyte	>C16-C34
Units	µg/L

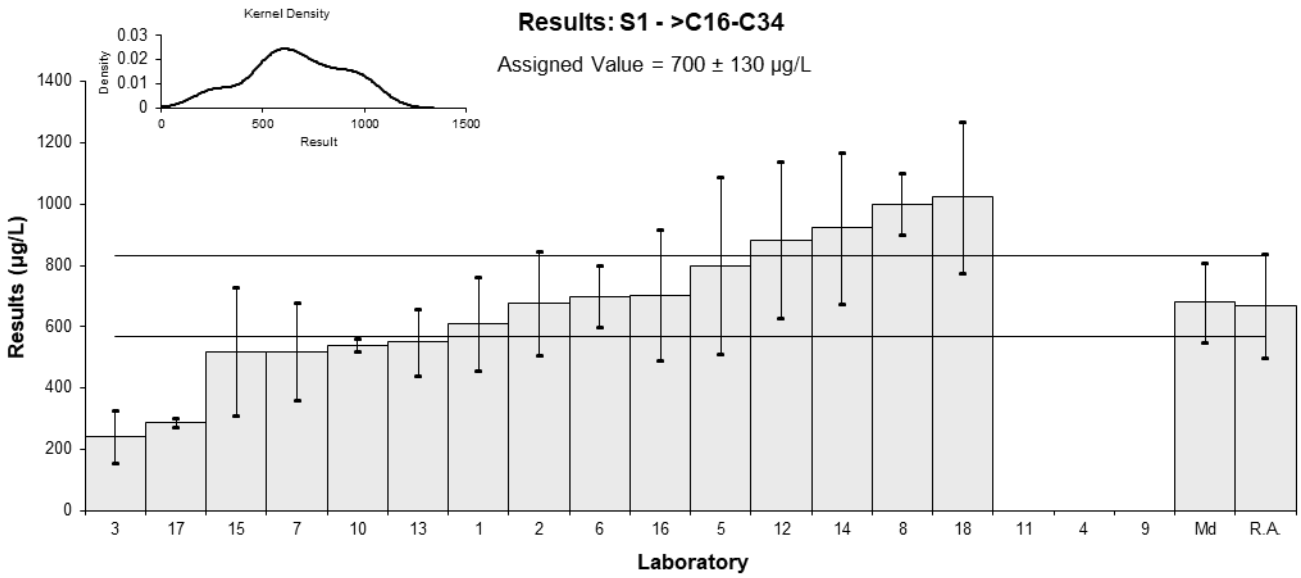
Participant Results

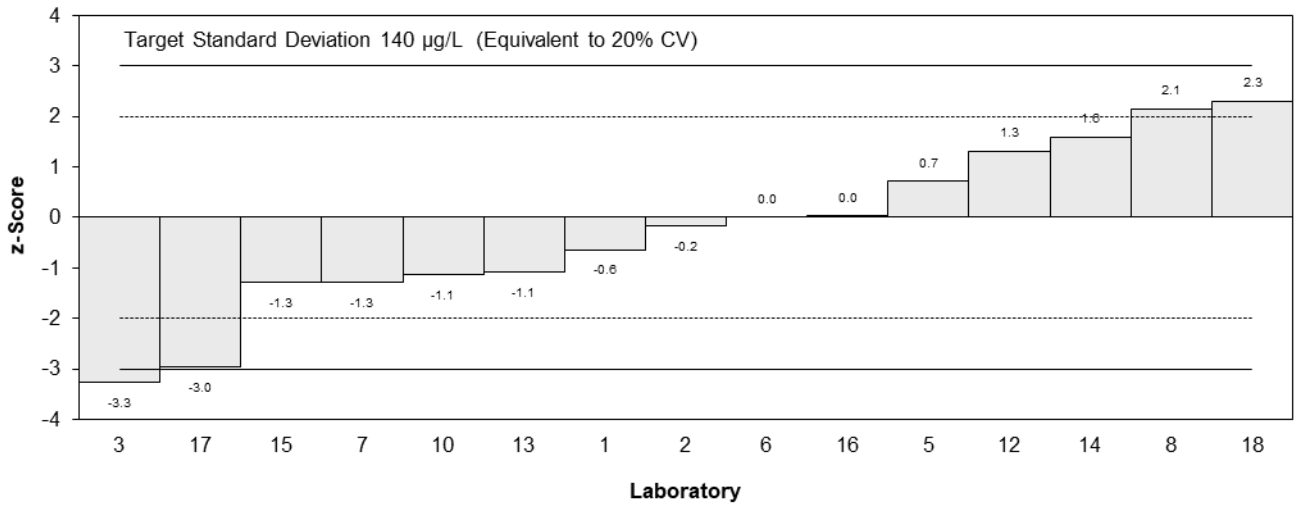
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	611	152	-0.64	-0.44
2	677	169	-0.16	-0.11
3	242	84.7	-3.27	-2.95
4	NR	NR		
5	800	290	0.71	0.31
6	700	100	0.00	0.00
7	520	160	-1.29	-0.87
8	1000	100	2.14	1.83
9	NR	NR		
10	540	20	-1.14	-1.22
11	NR	NR		
12	884	255	1.31	0.64
13	550	110	-1.07	-0.88
14	922	246	1.59	0.80
15	520	210	-1.29	-0.73
16	704	215	0.03	0.02
17	285.7	14.6	-2.96	-3.17
18	1023	245.5	2.31	1.16

Statistics

Assigned Value*	700	130
Robust Average	670	170
Median	680	130
Mean	670	
N	15	
Max.	1023	
Min.	242	
Robust SD	260	
Robust CV	39%	

* Robust average excluding Laboratories 3, 17 and 18.


Kernel Density
Density
Result
Results: S1 - >C16-C34
Assigned Value = 700 ± 130 µg/L
Results (µg/L)
Laboratory
3 17 15 7 10 13 1 2 6 16 5 12 14 8 18 11 4 9 Md R.A.


z-Scores: S1 - >C16-C34
Target Standard Deviation 140 µg/L (Equivalent to 20% CV)
z-Score
Laboratory
3 17 15 7 10 13 1 2 6 16 5 12 14 8 18
-3.3 -3.0 -1.3 -1.3 -1.1 -1.1 -0.6 -0.2 0.0 0.0 0.7 1.3 1.6 2.1 2.3

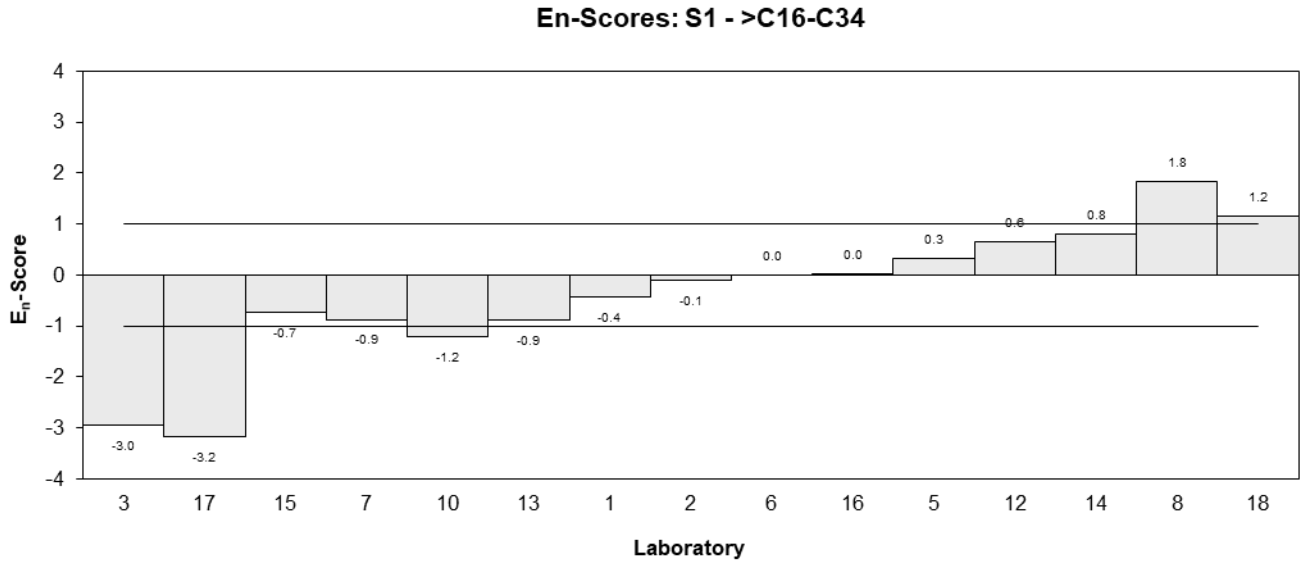

En-Scores: S1 - >C16-C34
En-Score
Laboratory
3 17 15 7 10 13 1 2 6 16 5 12 14 8 18
-3.0 -3.2 -0.7 -0.9 -1.2 -0.9 -0.4 -0.1 0.0 0.0 0.3 0.6 0.8 1.8 1.2

Figure 3

Table 7

Sample Details

Sample No.	S1
Matrix	Water
Analyte	>C34-C40
Units	µg/L

Participant Results

Lab. Code	Result	Uncertainty
1	34	8.5
2	NR	NR
3	<100	35
4	NR	NR
5	< 100	NR
6	<100	NR
7	<200	NR
8	<10	<10
9	NR	NR
10	<100	20
11	NR	NR
12	<100	39
13	<100	NR
14	< 100	30
15	<100	NR
16	< 100	30
17	NR	NR
18	0	0

Statistics

Insuffient data to calculate statistics.

Table 8 Additional hydrocarbon ranges to those defined in Schedule B3 of the NEPM,[5] reported by participants for Sample S1

Lab. Code	Range	Result (µg/L)	Uncertainty (µg/L)
2	C34-C36	<100	NR
4	C7-C9	0	NR
	C10-C14	400	NR
	C15-C36	600	NR
9	C7-C9	<0.2	NR
	C10-C14	270	81
	C15-C36	470	122
11	C7-C9	<100	67
	C10-C14	<200	110
	C15-C36	500	180
17	C34-C37	NR	NR

Table 9

Sample Details

Sample No.	S1
Matrix	Water
Analyte	TRH
Units	µg/L

Participant Results

Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	1381	345	0.53	0.23
2**	1758	440	2.00	0.92
3	430	151	-4.43	-2.67
4	1000	NR	-1.46	-1.00
5	1600	530	1.67	0.53
6	1340	170	0.31	0.18
7	1230	370	-0.26	-0.11
8	3400	300	11.04	5.17
9	740	222	-2.81	-1.51
10	1170	NR	-0.57	-0.39
11	710	220	-2.97	-1.60
12**	1887	435	2.00	1.00
13	1150	330	-0.68	-0.30
14**	1820	486	2.00	0.96
15	1270	510	-0.05	-0.02
16	1409	430	0.67	0.25
17	701.4	36.4	-3.01	-2.05
18**	2133	512	2.00	1.00

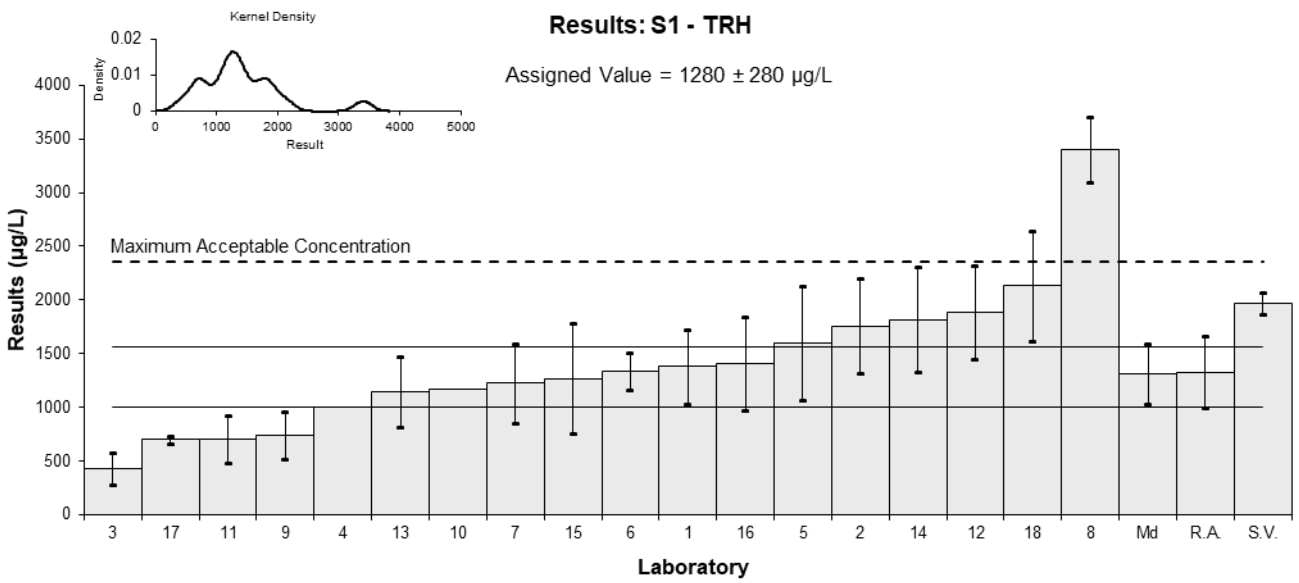
Statistics

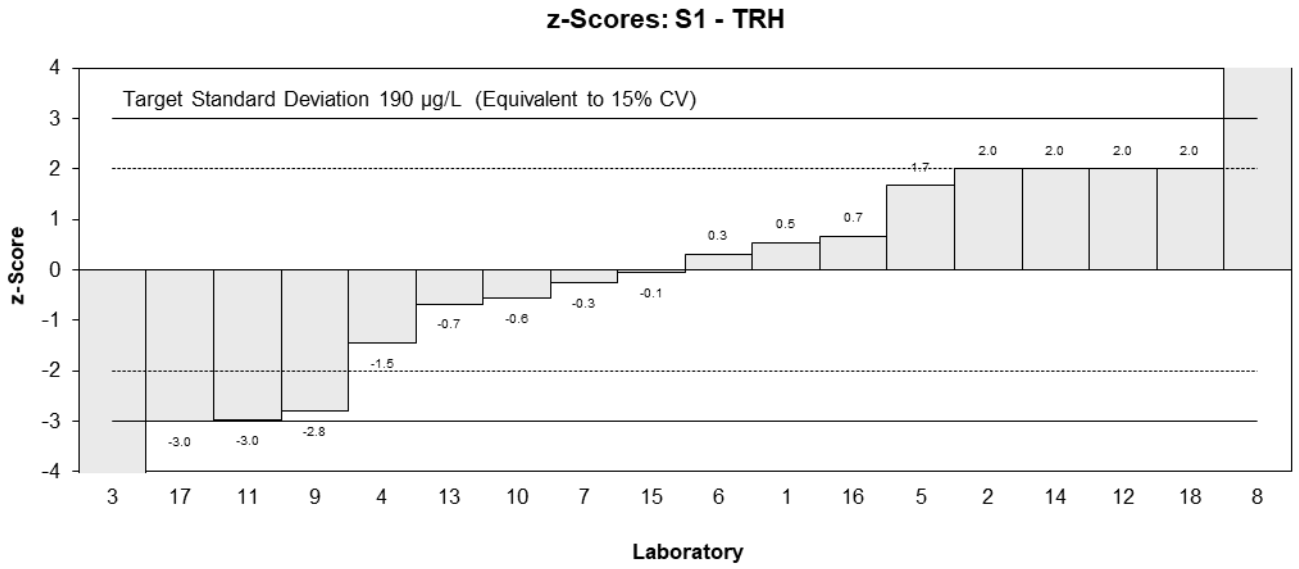
Assigned Value*	1280	280
Spike	1970	100
Max. Acceptable Concentration**	2350	
Robust Average	1330	330
Median	1310	280
Mean	1400	
N	18	
Max.	3400	
Min.	430	
Robust SD	560	
Robust CV	42%	

* Robust average excluding Laboratories 3, 8 and 18.

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

If a participant did not report a TRH value, the TRH result was calculated by the study coordinator by summing the individual hydrocarbon ranges reported, and no estimate of the uncertainty of the TRH result was made.


Kernel Density
Density
Result
Results: S1 - TRH
Assigned Value = 1280 ± 280 µg/L
Maximum Acceptable Concentration
Results (µg/L)
Laboratory
3 17 11 9 4 13 10 7 15 6 1 16 5 2 14 12 18 8 Md R.A. S.V.


z-Scores: S1 - TRH
Target Standard Deviation 190 µg/L (Equivalent to 15% CV)
z-Score
Laboratory
3 17 11 9 4 13 10 7 15 6 1 16 5 2 14 12 18 8
-3.0 -3.0 -2.8 -1.5 -0.7 -0.6 -0.3 -0.1 0.3 0.5 0.7 1.7 2.0 2.0 2.0 2.0

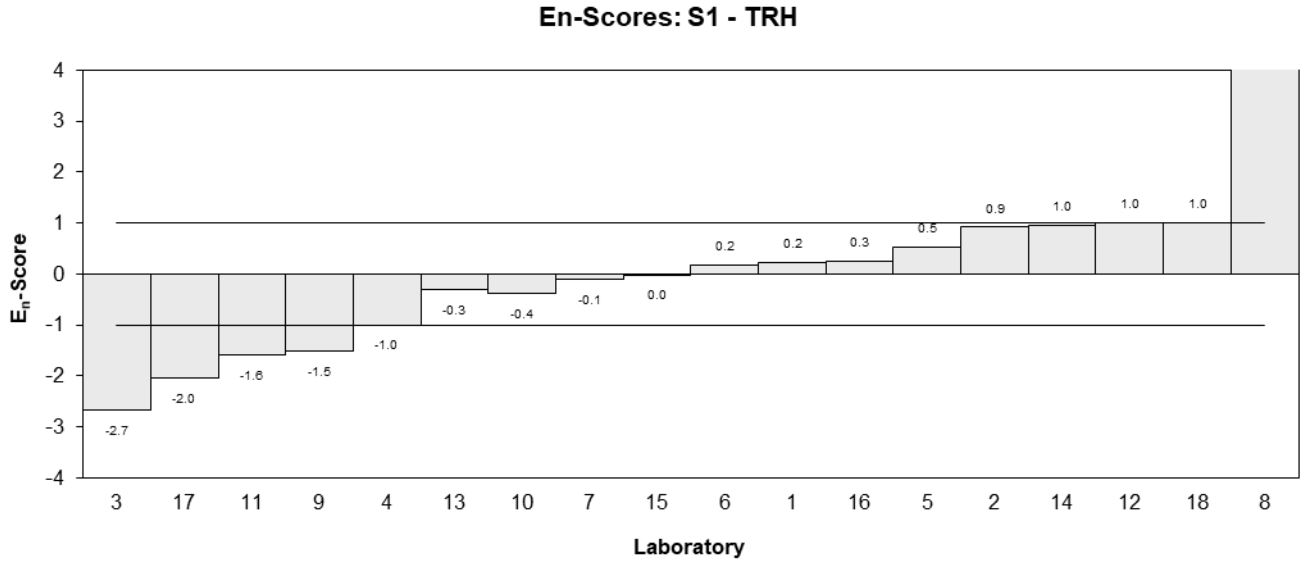

En-Scores: S1 - TRH
En-Score
Laboratory
3 17 11 9 4 13 10 7 15 6 1 16 5 2 14 12 18 8
-2.7 -2.0 -1.6 -1.5 -1.0 -0.3 -0.4 -0.1 0.0 0.2 0.2 0.3 0.5 0.9 1.0 1.0 1.0

Figure 4

Table 10

Sample Details

Sample No.	S2
Matrix	Water
Analyte	C6-C10
Units	µg/L

Participant Results

Lab. Code	Result	Uncertainty
1	601	180
2	NT	NT
3	690	173
4	NT	NT
5	590	95
6	710	120
7	543	162
8	NT	NT
9	NT	NT
10	200	20
11	730	210
12	605	85
13	900	270
14	694	167
15	600	180
16	610	160
17	NT	NT
18	802	280.7

Statistics*

Assigned Value	Not Set	
Robust Average	644	83
Median	608	69
Mean	629	
N	12	
Max.	900	
Min.	200	
Robust SD	110	
Robust CV	18%	

* Laboratory 11 reported for C6-C9; this result has been excluded from all statistical calculations.

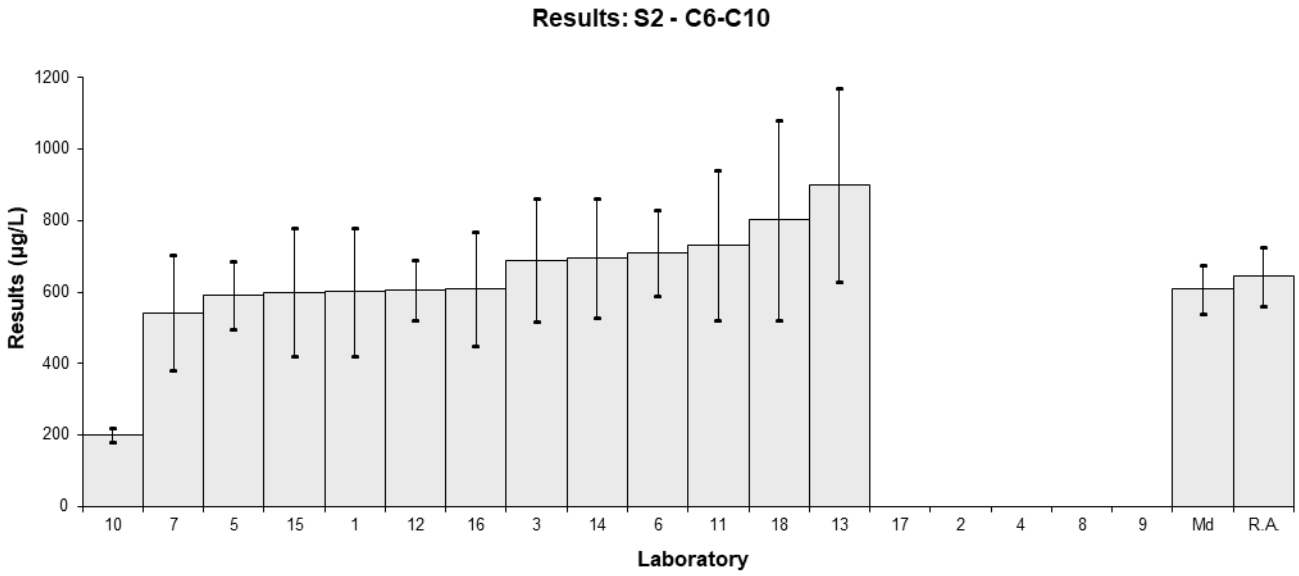

Results: S2 - C6-C10
Results (µg/L)
1200
1000
800
600
400
200
0
10
7
5
15
1
12
16
3
14
6
11
18
13
17
2
4
8
9
Md
R.A.
Laboratory

Figure 5

Table 11

Sample Details

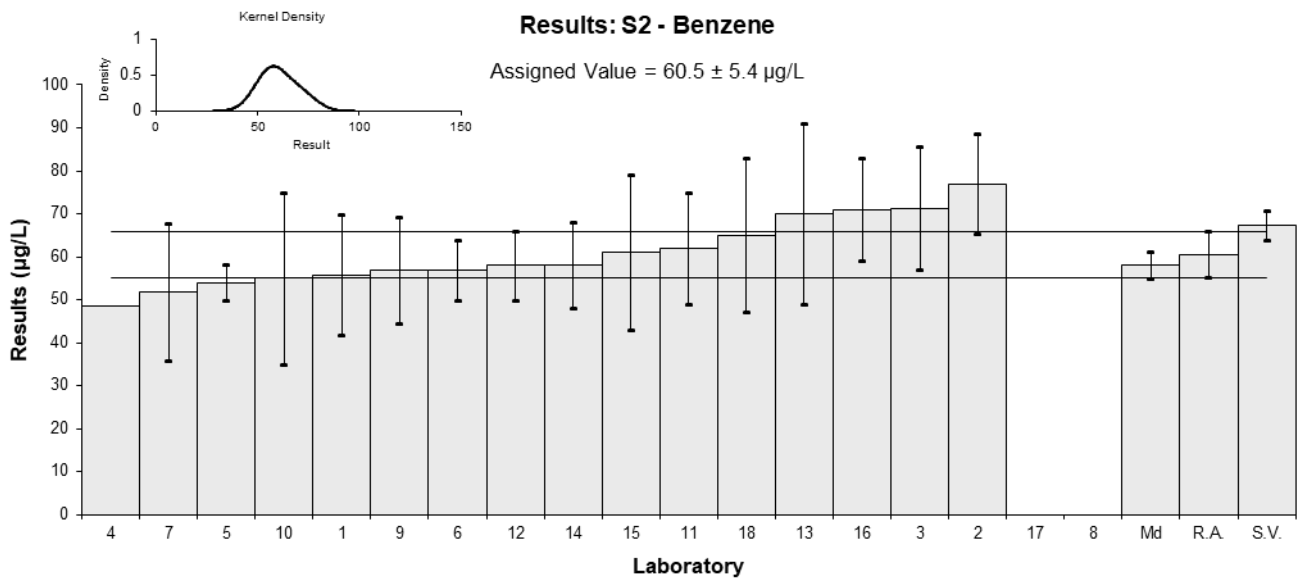
Sample No.	S2
Matrix	Water
Analyte	Benzene
Units	µg/L

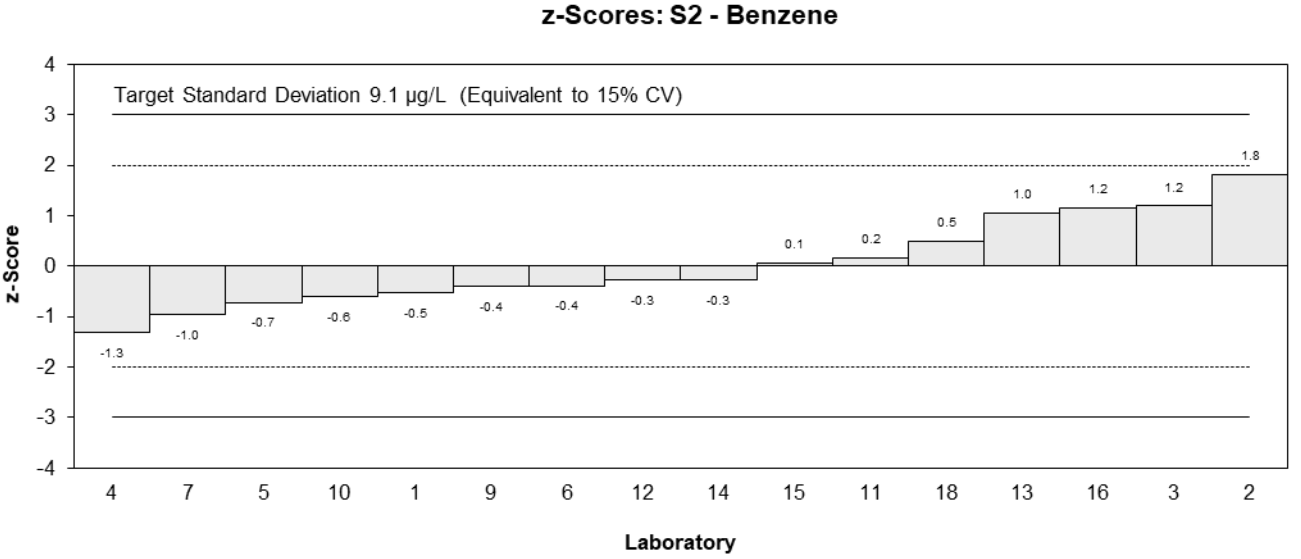
Participant Results

Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	55.8	14	-0.52	-0.31
2	77	11.7	1.82	1.28
3	71.3	14.3	1.19	0.71
4	48.5	NR	-1.32	-2.22
5	54	4.1	-0.72	-0.96
6	57	7	-0.39	-0.40
7	51.8	16	-0.96	-0.52
8	NT	NT		
9	56.9	12.5	-0.40	-0.26
10	55	20	-0.61	-0.27
11	62	13	0.17	0.11
12	58	8	-0.28	-0.26
13	70	21	1.05	0.44
14	58	10	-0.28	-0.22
15	61	18	0.06	0.03
16	71	12	1.16	0.80
17	NT	NT		
18	65	17.9	0.50	0.24

Statistics

Assigned Value	60.5	5.4
Spike	67.4	3.4
Robust Average	60.5	5.4
Median	58.0	3.2
Mean	60.8	
N	16	
Max.	77	
Min.	48.5	
Robust SD	8.6	
Robust CV	14%	


Results: S2 - Benzene
Assigned Value = 60.5 ± 5.4 µg/L
Kernel Density
Density
Result
Results (µg/L)
Laboratory
4 7 5 10 1 9 6 12 14 15 11 18 13 16 3 2 17 8 Md R.A. S.V.


z-Scores: S2 - Benzene
Target Standard Deviation 9.1 µg/L (Equivalent to 15% CV)
z-Score
Laboratory
4 7 5 10 1 9 6 12 14 15 11 18 13 16 3 2
-1.3 -1.0 -0.7 -0.6 -0.5 -0.4 -0.4 -0.3 -0.3 0.1 0.2 0.5 1.0 1.2 1.2 1.8

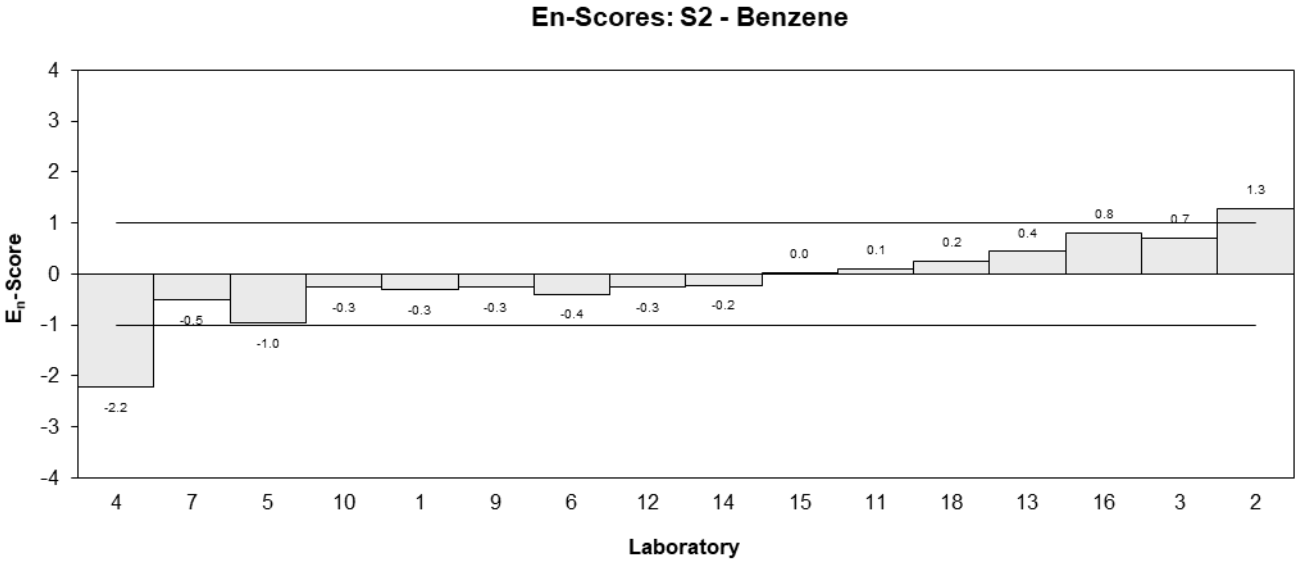

En-Scores: S2 - Benzene
En-Score
Laboratory
4 7 5 10 1 9 6 12 14 15 11 18 13 16 3 2
-2.2 -0.5 -1.0 -0.3 -0.3 -0.3 -0.4 -0.3 -0.2 0.0 0.1 0.2 0.4 0.8 0.7 1.3

Figure 6

Table 12

Sample Details

Sample No.	S2
Matrix	Water
Analyte	Toluene
Units	µg/L

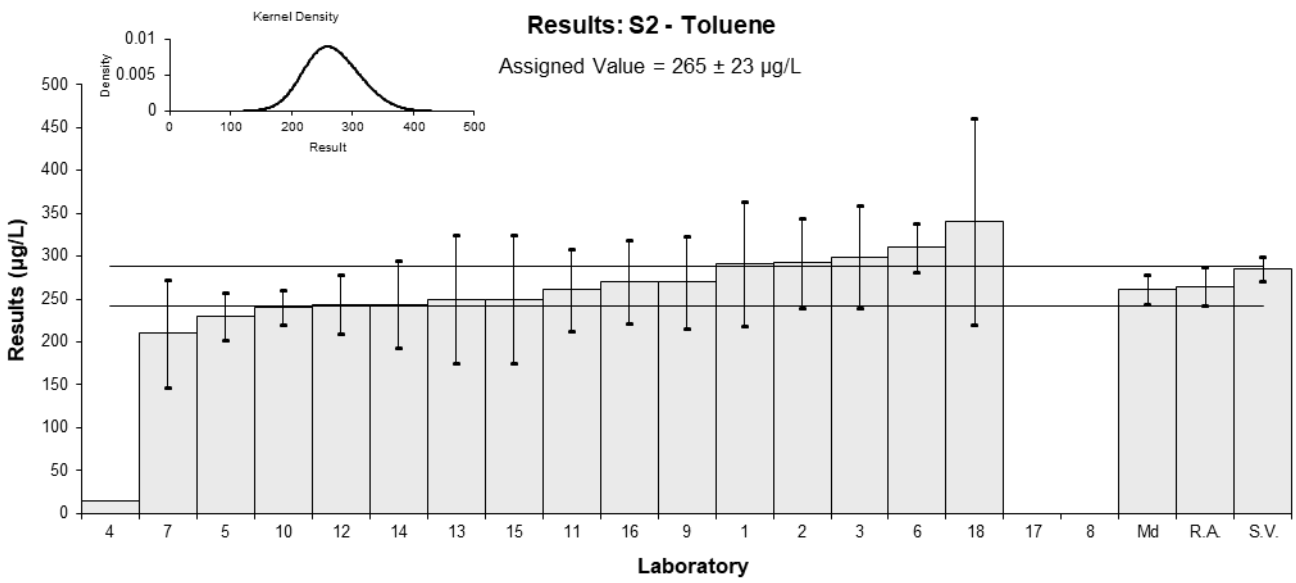
Participant Results

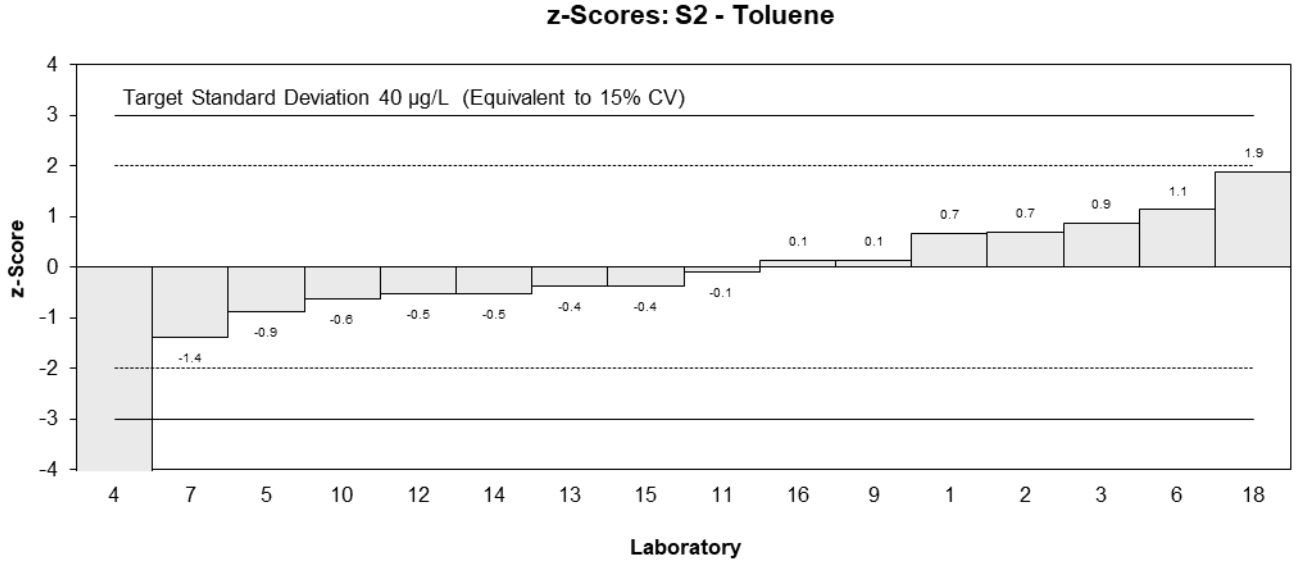
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	291	72	0.65	0.34
2	292	52.6	0.68	0.47
3	299	59.8	0.86	0.53
4	14.5	NR	-6.30	-10.89
5	230	27	-0.88	-0.99
6	310	28	1.13	1.24
7	210	63	-1.38	-0.82
8	NT	NT		
9	270	54	0.13	0.09
10	240	20	-0.63	-0.82
11	261	48	-0.10	-0.08
12	244	34	-0.53	-0.51
13	250	75	-0.38	-0.19
14	244	51	-0.53	-0.38
15	250	75	-0.38	-0.19
16	270	49	0.13	0.09
17	NT	NT		
18	340	120.4	1.89	0.61

Statistics*

Assigned Value	265	23
Spike	285	14
Robust Average	265	23
Median	261	17
Mean	267	
N	15	
Max.	340	
Min.	210	
Robust SD	35	
Robust CV	13%	

* Laboratory 4 excluded from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S2 - Toluene
Assigned Value = 265 ± 23 µg/L
Results (µg/L)
Laboratory
4 7 5 10 12 14 13 15 11 16 9 1 2 3 6 18 17 8 Md R.A. S.V.


z-Scores: S2 - Toluene
Target Standard Deviation 40 µg/L (Equivalent to 15% CV)
z-Score
Laboratory
4 7 5 10 12 14 13 15 11 16 9 1 2 3 6 18
-1.4 -0.9 -0.6 -0.5 -0.5 -0.4 -0.4 -0.1 0.1 0.1 0.7 0.7 0.9 1.1 1.9

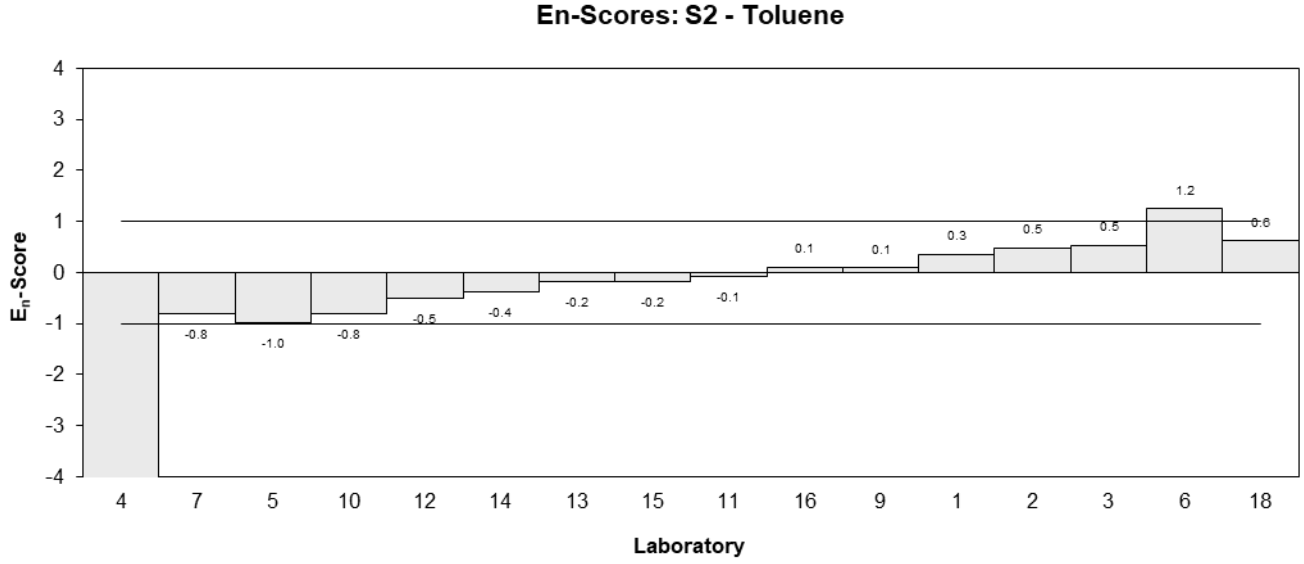

En-Scores: S2 - Toluene
En-Score
Laboratory
4 7 5 10 12 14 13 15 11 16 9 1 2 3 6 18
-0.8 -1.0 -0.8 -0.5 -0.4 -0.2 -0.2 -0.1 0.1 0.1 0.3 0.5 0.5 1.2 0.6

Figure 7

Table 13

Sample Details

Sample No.	S2
Matrix	Water
Analyte	Ethylbenzene
Units	µg/L

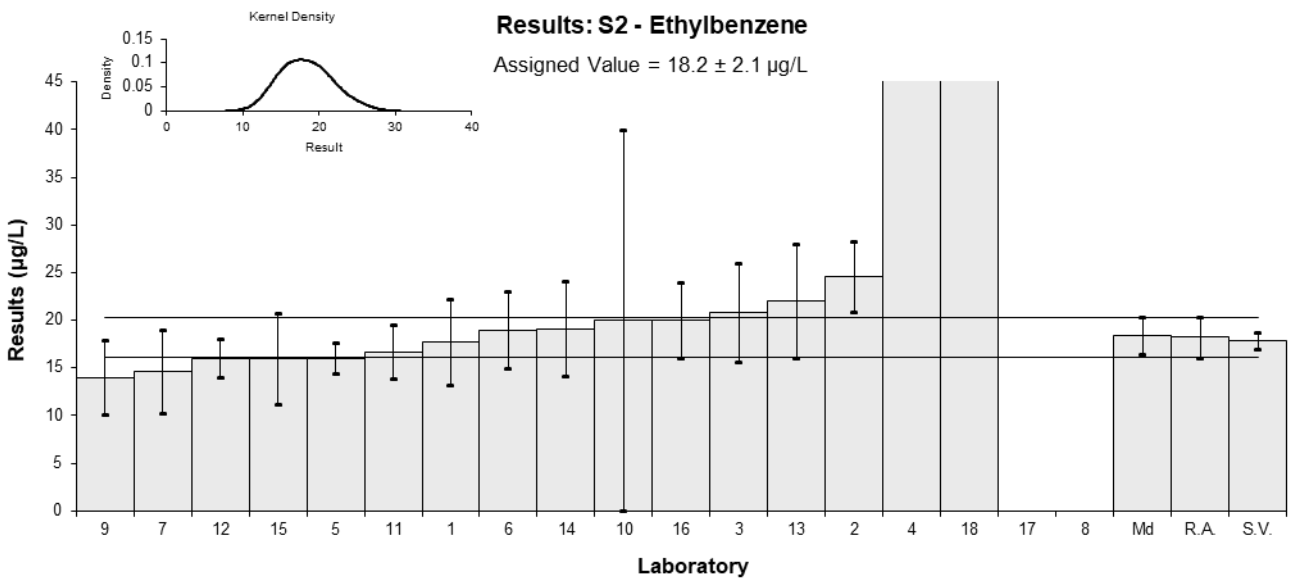
Participant Results

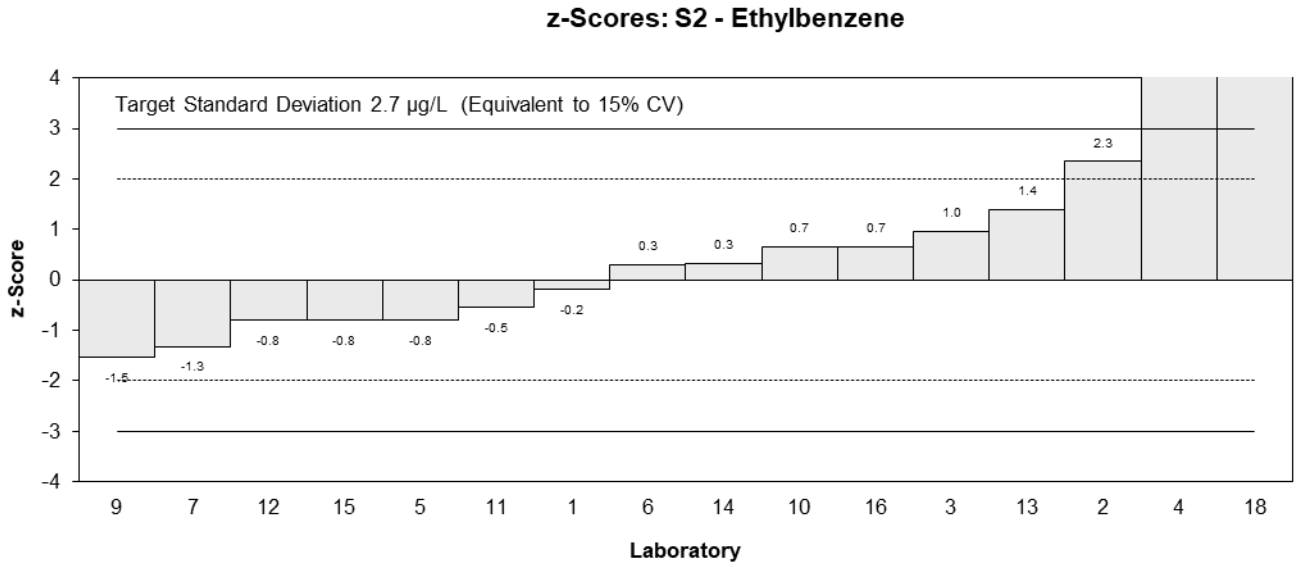
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	17.7	4.5	-0.18	-0.10
2	24.6	3.72	2.34	1.50
3	20.8	5.2	0.95	0.46
4	195	NR	64.76	84.19
5	16	1.6	-0.81	-0.83
6	19	4	0.29	0.18
7	14.6	4.4	-1.32	-0.74
8	NT	NT		
9	14	3.9	-1.54	-0.95
10	20	20	0.66	0.09
11	16.7	2.8	-0.55	-0.43
12	16	2	-0.81	-0.76
13	22	6	1.39	0.60
14	19.1	5	0.33	0.17
15	16	4.8	-0.81	-0.42
16	20	4	0.66	0.40
17	NT	NT		
18	220	83.8	73.92	2.41

Statistics*

Assigned Value	18.2	2.1
Spike	17.9	0.9
Robust Average	18.2	2.1
Median	18.4	2.0
Mean	18.3	
N	14	
Max.	24.6	
Min.	14	
Robust SD	3.1	
Robust CV	17%	

* Laboratories 4 and 18 excluded from all statistical calculations (gross error).


Kernel Density
Density
Result
Results: S2 - Ethylbenzene
Assigned Value = 18.2 ± 2.1 µg/L
Results (µg/L)
9 7 12 15 5 11 1 6 14 10 16 3 13 2 4 18 17 8 Md R.A. S.V.
Laboratory


z-Scores: S2 - Ethylbenzene
Target Standard Deviation 2.7 µg/L (Equivalent to 15% CV)
z-Score
-1.5 -1.3 -0.8 -0.8 -0.8 -0.5 -0.2 0.3 0.3 0.7 0.7 1.0 1.4 2.3
9 7 12 15 5 11 1 6 14 10 16 3 13 2 4 18
Laboratory

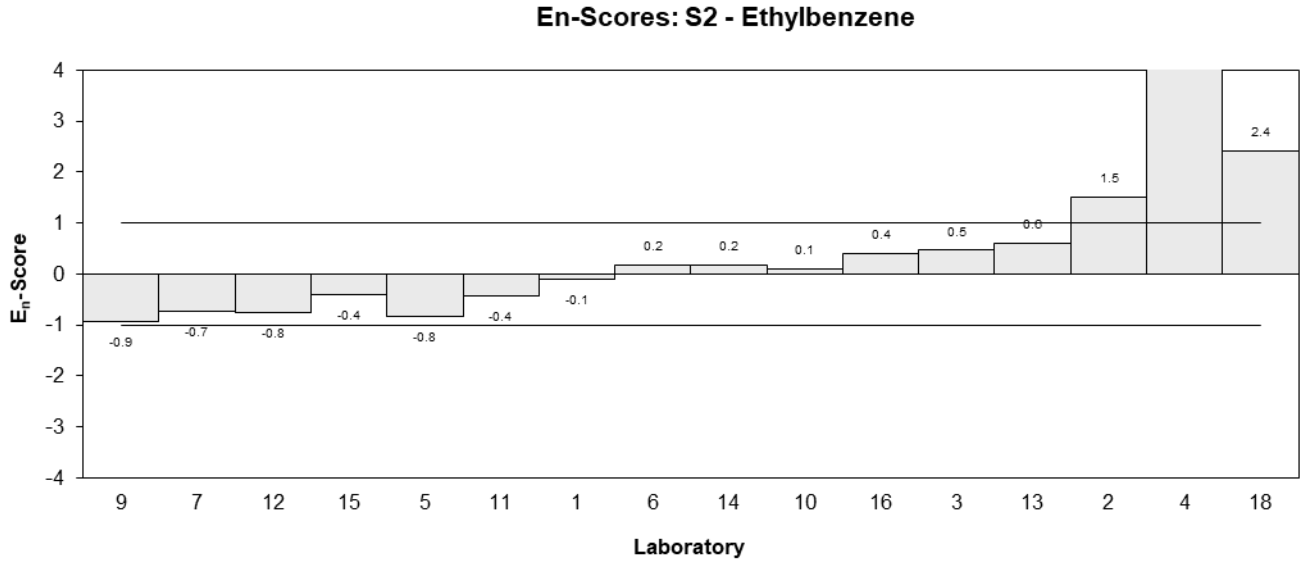

En-Scores: S2 - Ethylbenzene
En-Score
-0.9 -0.7 -0.8 -0.4 -0.8 -0.4 -0.1 0.2 0.2 0.1 0.4 0.5 0.6 1.5 2.4
9 7 12 15 5 11 1 6 14 10 16 3 13 2 4 18
Laboratory

Figure 8

Table 14

Sample Details

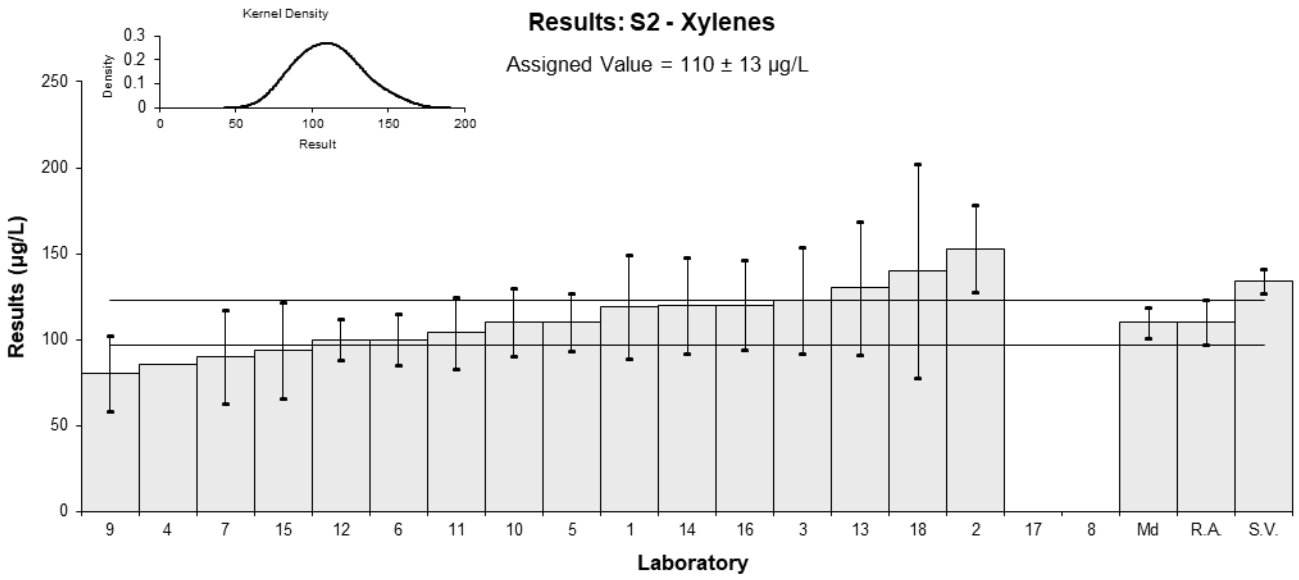
Sample No.	S2
Matrix	Water
Analyte	Xylenes
Units	µg/L

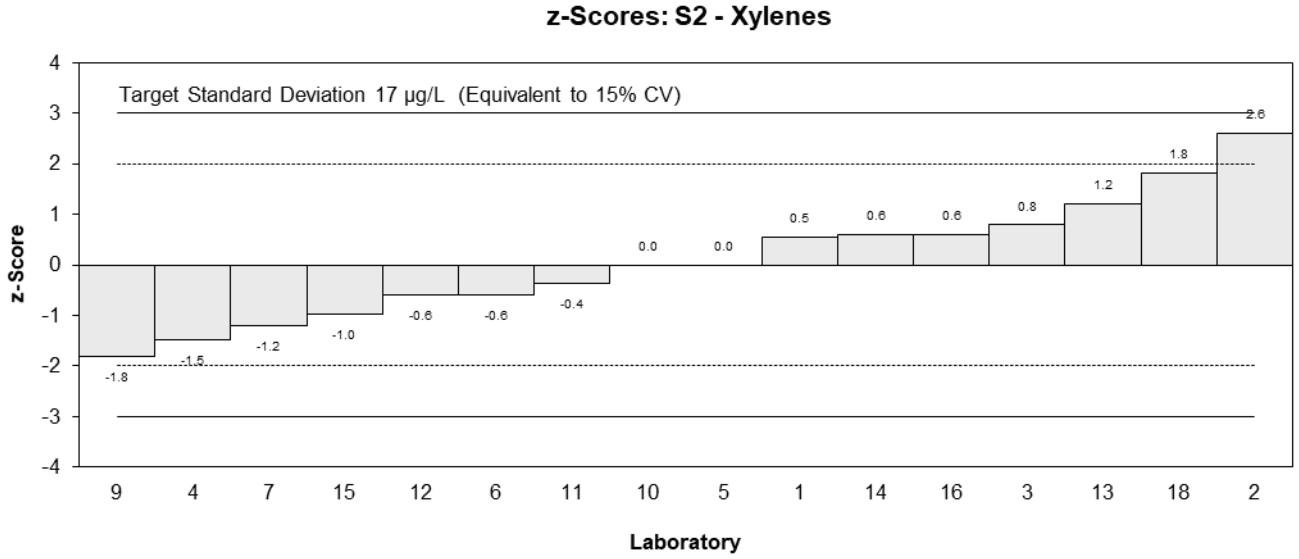
Participant Results

Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	119	30	0.55	0.28
2	153	25.7	2.61	1.49
3	123	30.8	0.79	0.39
4	85.5	NR	-1.48	-1.88
5	110	17	0.00	0.00
6	100	15	-0.61	-0.50
7	90	27	-1.21	-0.67
8	NT	NT		
9	80	22	-1.82	-1.17
10	110	20	0.00	0.00
11	104.1	20.9	-0.36	-0.24
12	100	12	-0.61	-0.57
13	130	39	1.21	0.49
14	120	28	0.61	0.32
15	94	28	-0.97	-0.52
16	120	26	0.61	0.34
17	NT	NT		
18	140	62.4	1.82	0.47

Statistics

Assigned Value	110	13
Spike	134	7
Robust Average	110	13
Median	110	9
Mean	111	
N	16	
Max.	153	
Min.	80	
Robust SD	21	
Robust CV	19%	


Kernel Density
Density
Result
Results: S2 - Xylenes
Assigned Value = 110 ± 13 µg/L
Results (µg/L)
Laboratory
9 4 7 15 12 6 11 10 5 1 14 16 3 13 18 2 17 8 Md R.A. S.V.


z-Scores: S2 - Xylenes
Target Standard Deviation 17 µg/L (Equivalent to 15% CV)
z-Score
Laboratory
9 4 7 15 12 6 11 10 5 1 14 16 3 13 18 2
-1.8 -1.5 -1.2 -1.0 -0.6 -0.6 -0.4 0.0 0.0 0.5 0.6 0.6 0.8 1.2 1.8 2.6

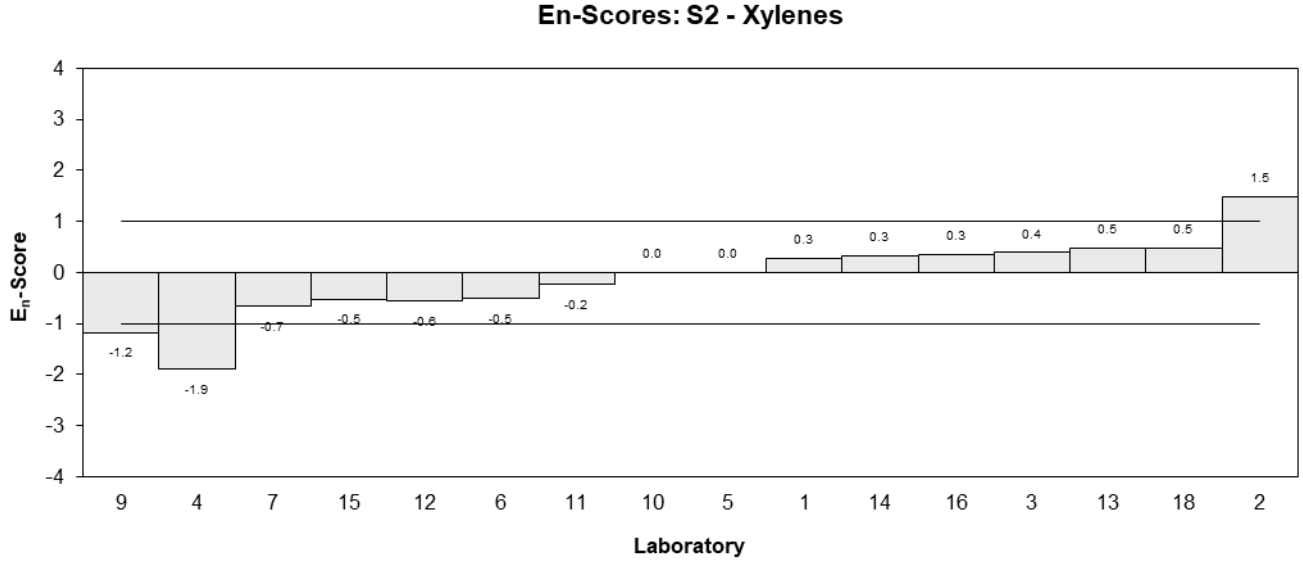

En-Scores: S2 - Xylenes
En-Score
Laboratory
9 4 7 15 12 6 11 10 5 1 14 16 3 13 18 2
-1.2 -1.9 -0.7 -0.5 -0.6 -0.5 -0.2 0.0 0.0 0.3 0.3 0.3 0.4 0.5 0.5 1.5

Figure 9

Table 15

Sample Details

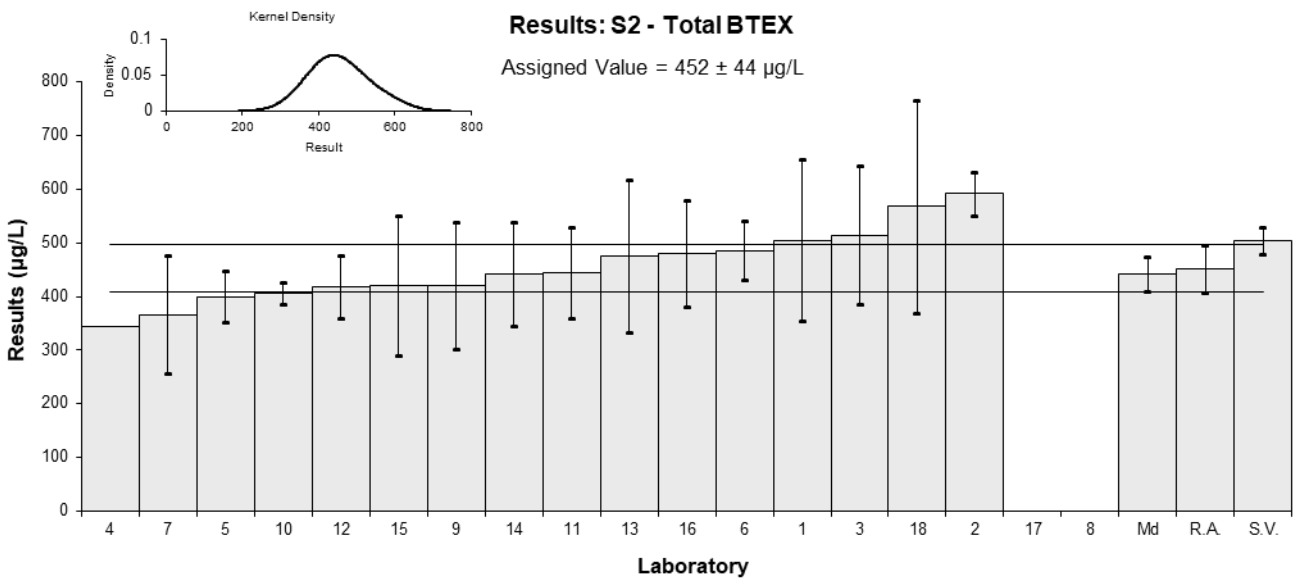
Sample No.	S2
Matrix	Water
Analyte	Total BTEX
Units	µg/L

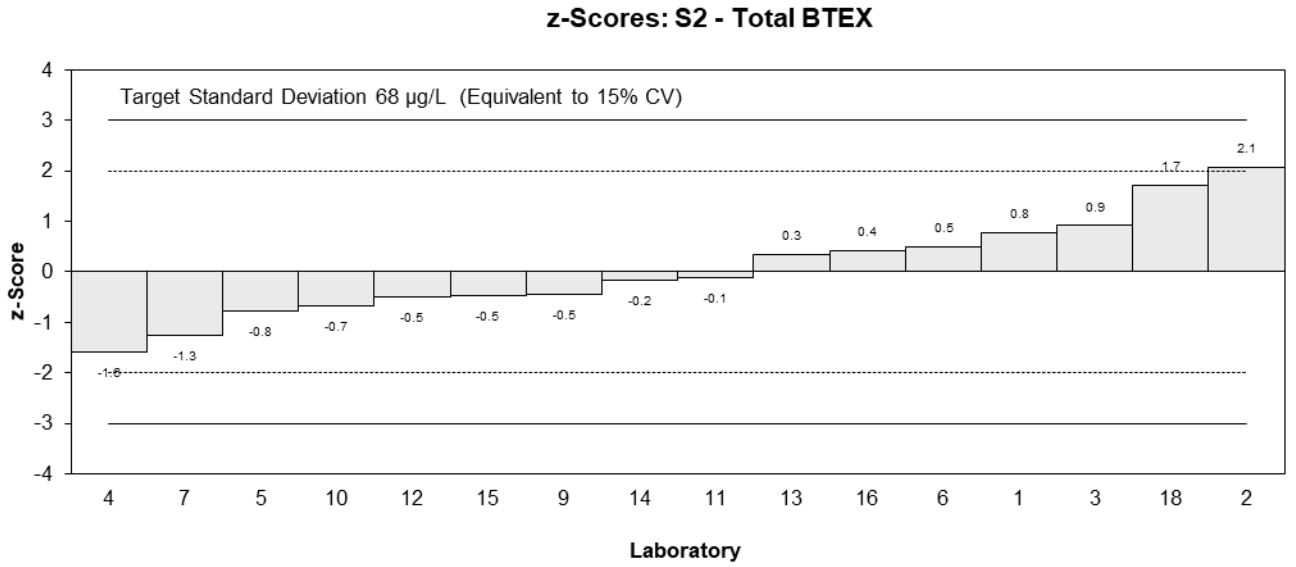
Participant Results

Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	505	151	0.78	0.34
2	592	41	2.06	2.33
3	514	129	0.91	0.45
4	343.5	NR	-1.60	-2.47
5	400	49	-0.77	-0.79
6	486	54	0.50	0.49
7	366	110	-1.27	-0.73
8	NT	NT		
9	421	118	-0.46	-0.25
10	406	20	-0.68	-0.95
11	443.8	84.7	-0.12	-0.09
12	418	58	-0.50	-0.47
13	475	142	0.34	0.15
14	441.1	97	-0.16	-0.10
15	420	130	-0.47	-0.23
16	480	100	0.41	0.26
17	NT	NT		
18	568	198.8	1.71	0.57

Statistics

Assigned Value	452	44
Spike	504	25
Robust Average	452	44
Median	442	32
Mean	455	
N	16	
Max.	592	
Min.	343.5	
Robust SD	71	
Robust CV	16%	


Kernel Density
Density
Result
Results: S2 - Total BTEX
Assigned Value = 452 ± 44 µg/L
Results (µg/L)
Laboratory
4 7 5 10 12 15 9 14 11 13 16 6 1 3 18 2 17 8 Md R.A. S.V.


z-Scores: S2 - Total BTEX
Target Standard Deviation 68 µg/L (Equivalent to 15% CV)
z-Score
Laboratory
4 7 5 10 12 15 9 14 11 13 16 6 1 3 18 2
-1.6 -1.3 -0.8 -0.7 -0.5 -0.5 -0.5 -0.2 -0.1 0.3 0.4 0.5 0.8 0.9 1.7 2.1

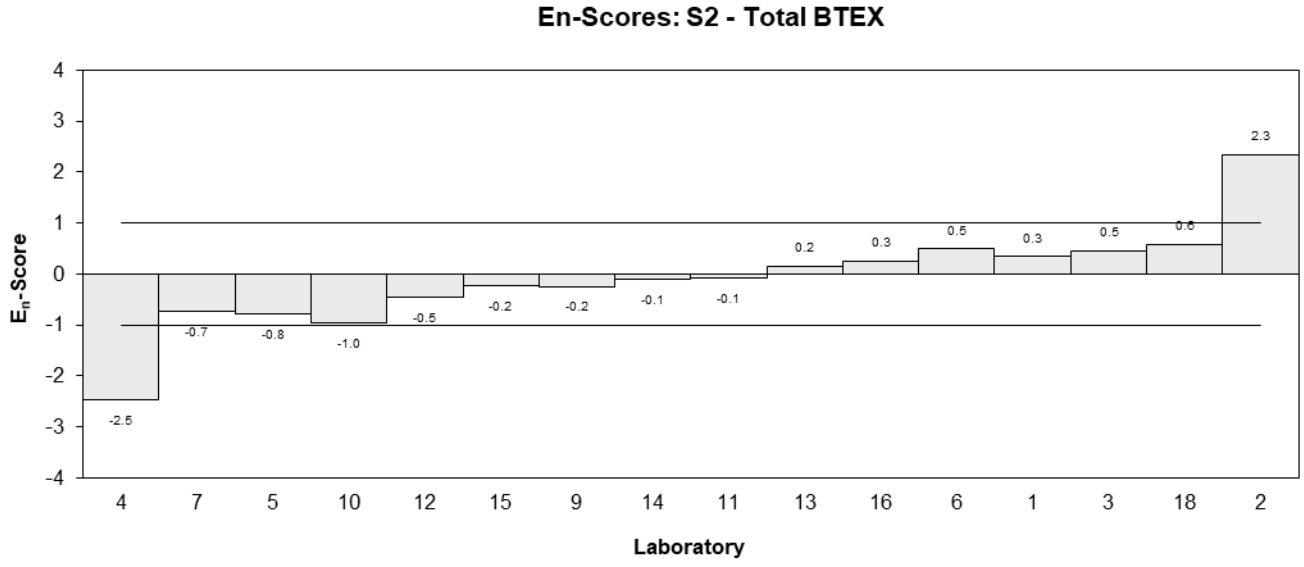

En-Scores: S2 - Total BTEX
En-Score
Laboratory
4 7 5 10 12 15 9 14 11 13 16 6 1 3 18 2
-2.5 -0.7 -0.8 -1.0 -0.5 -0.2 -0.2 -0.1 -0.1 0.2 0.3 0.5 0.3 0.5 0.6 2.3

Figure 10

Table 16

Sample Details

Sample No.	S3
Matrix	Water
Analyte	Anthracene
Units	µg/L

Participant Results

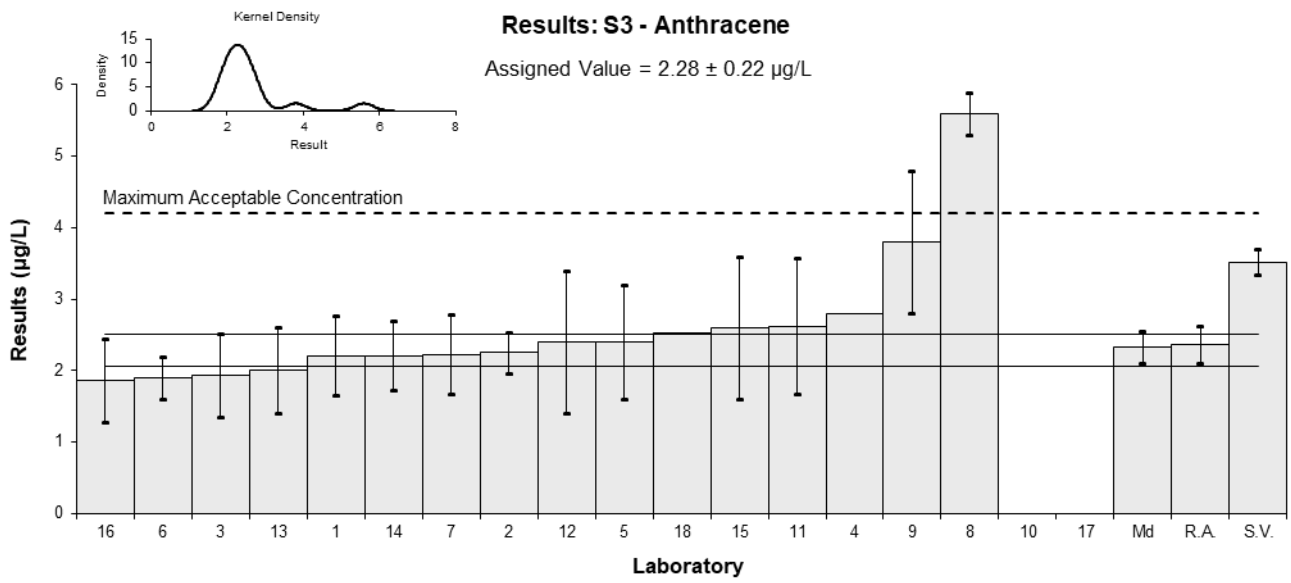
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	2.21	0.55	-0.20	-0.12
2	2.25	0.29	-0.09	-0.08
3	1.93	0.579	-1.02	-0.57
4	2.8	NR	1.52	2.36
5	2.4	0.8	0.35	0.14
6	1.9	0.3	-1.11	-1.02
7	2.23	0.56	-0.15	-0.08
8	5.59	0.3	9.68	8.90
9**	3.8	1	2.00	1.00
10	NR	NR		
11	2.62	0.95	0.99	0.35
12	2.4	1	0.35	0.12
13	2	0.6	-0.82	-0.44
14	2.21	0.48	-0.20	-0.13
15	2.6	1.0	0.94	0.31
16	1.863	0.580	-1.22	-0.67
17	NT	NT		
18	2.53	NR	0.73	1.14

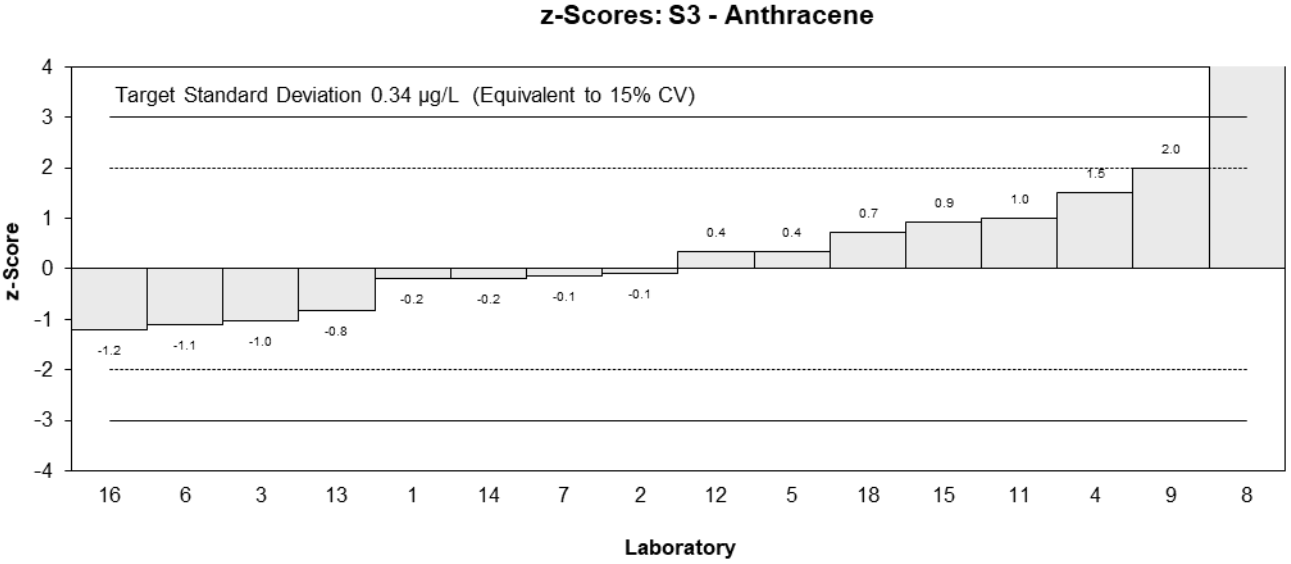
Statistics

Assigned Value*	2.28	0.22
Spike	3.52	0.18
Max. Acceptable Concentration**	4.20	
Robust Average	2.37	0.26
Median	2.33	0.23
Mean	2.58	
N	16	
Max.	5.59	
Min.	1.863	
Robust SD	0.41	
Robust CV	17%	

* Robust average excluding Laboratories 8 and 9.

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


Kernel Density
Density
Result
Results: S3 - Anthracene
Assigned Value = 2.28 ± 0.22 µg/L
Maximum Acceptable Concentration
Results (µg/L)
16 6 3 13 1 14 7 2 12 5 18 15 11 4 9 8 10 17 Md R.A. S.V.
Laboratory


z-Scores: S3 - Anthracene
Target Standard Deviation 0.34 µg/L (Equivalent to 15% CV)
z-Score
-1.2 -1.1 -1.0 -0.8 -0.2 -0.2 -0.1 -0.1 0.4 0.4 0.7 0.9 1.0 1.5 2.0
16 6 3 13 1 14 7 2 12 5 18 15 11 4 9 8
Laboratory

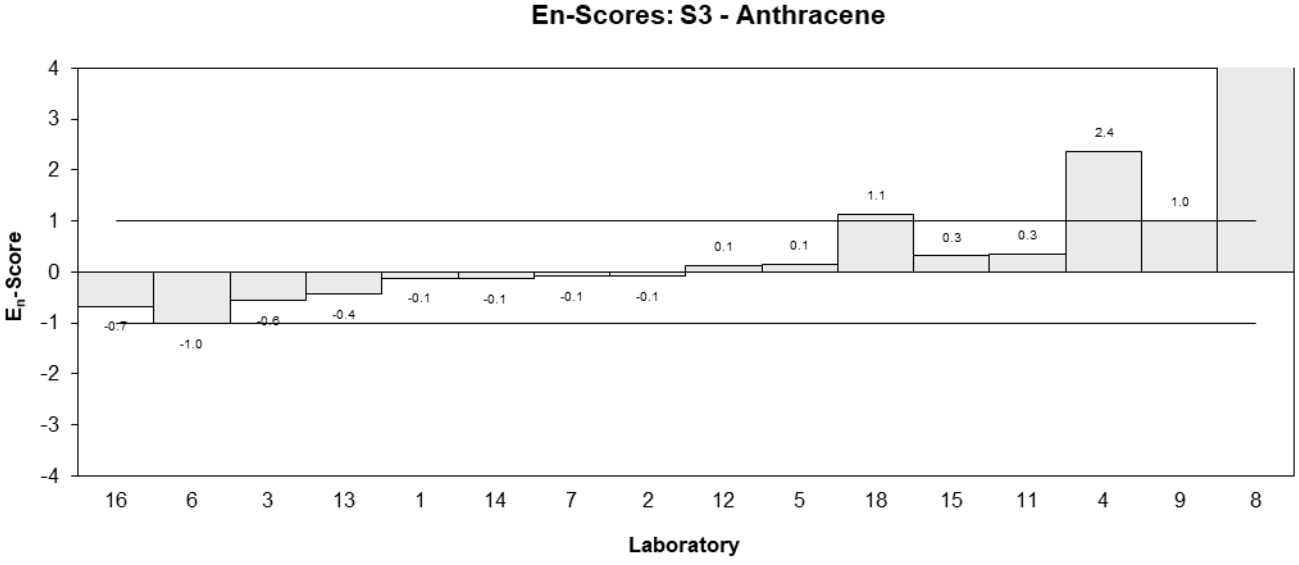

En-Scores: S3 - Anthracene
En-Score
-0.7 -1.0 -0.6 -0.4 -0.1 -0.1 -0.1 -0.1 0.1 0.1 1.1 0.3 0.3 2.4 1.0
16 6 3 13 1 14 7 2 12 5 18 15 11 4 9 8
Laboratory

Figure 11

Table 17

Sample Details

Sample No.	S3
Matrix	Water
Analyte	Benzo(a)pyrene
Units	µg/L

Participant Results

Lab. Code	Result	Uncertainty
1	3.41	0.85
2	3.3	0.22
3	<1	0.3
4	8.7	NR
5	7.3	2.3
6	3.7	1.2
7	9.63	2.4
8	13.92	0.5
9	6.5	2
10	NR	NR
11	5.5	2.7
12	7.1	3
13	3.5	1
14	3.47	0.791
15	7.8	3.1
16	1.561	0.501
17	NT	NT
18	5.23	NR

Statistics

Assigned Value	Not Set	
Spike	8.48	0.42
Robust Average	5.8	1.9
Median	5.5	1.7
Mean	6.0	
N	15	
Max.	13.92	
Min.	1.561	
Robust SD	3.0	
Robust CV	51%	

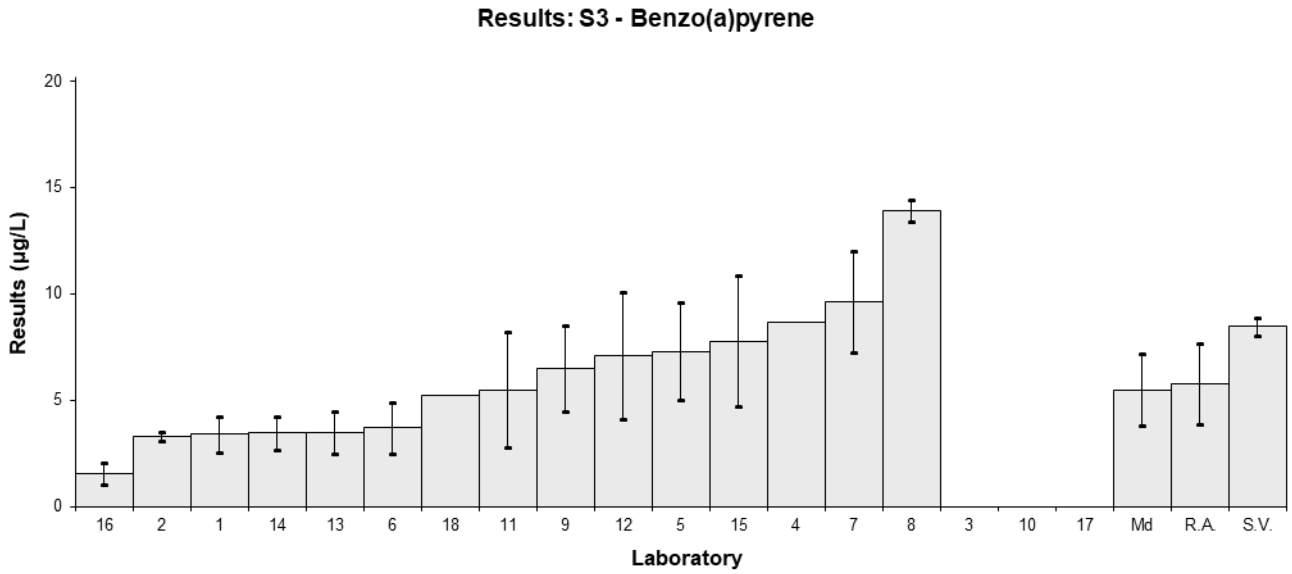

Results: S3 - Benzo(a)pyrene
Results (µg/L)
20
15
10
5
0
16
2
1
14
13
6
18
11
9
12
5
15
4
7
8
3
10
17
Md
R.A.
S.V.
Laboratory

Figure 12

Table 18

Sample Details

Sample No.	S3
Matrix	Water
Analyte	Fluoranthene
Units	µg/L

Participant Results

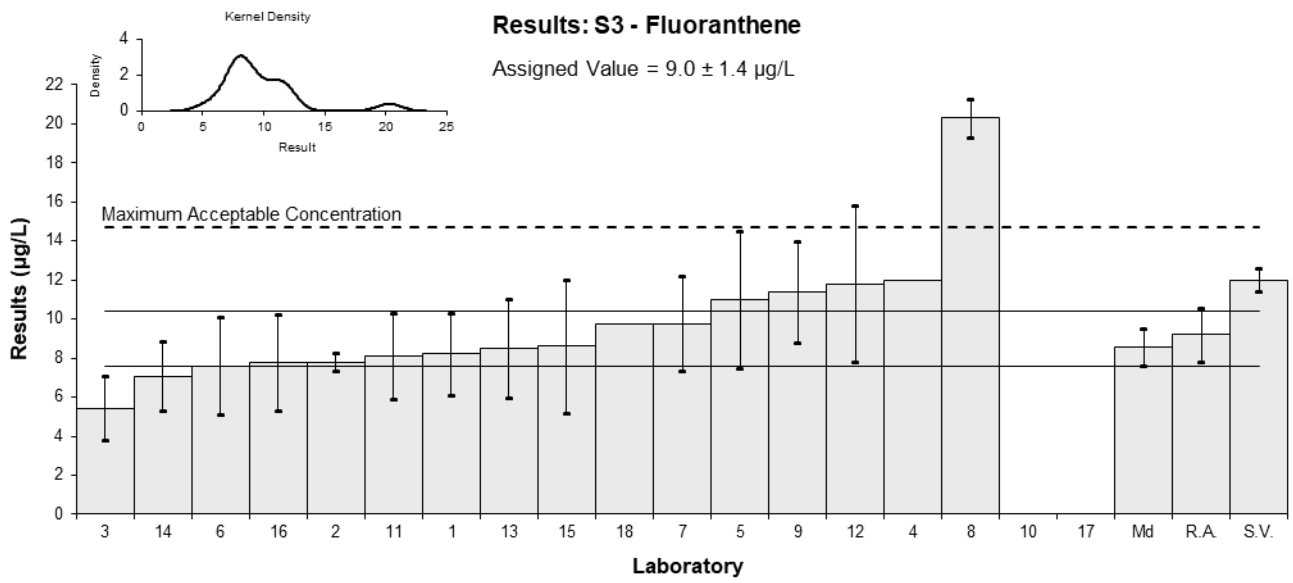
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	8.22	2.1	-0.58	-0.31
2	7.81	0.43	-0.88	-0.81
3	5.44	1.63	-2.64	-1.66
4**	12	NR	2.00	1.00
5	11	3.5	1.48	0.53
6	7.6	2.5	-1.04	-0.49
7	9.77	2.44	0.57	0.27
8	20.30	1	8.37	6.57
9	11.4	2.6	1.78	0.81
10	NR	NR		
11	8.1	2.2	-0.67	-0.35
12**	11.8	4	2.00	0.66
13	8.5	2.5	-0.37	-0.17
14	7.08	1.76	-1.42	-0.85
15	8.6	3.4	-0.30	-0.11
16	7.781	2.486	-0.90	-0.43
17	NT	NT		
18	9.77	NR	0.57	0.55

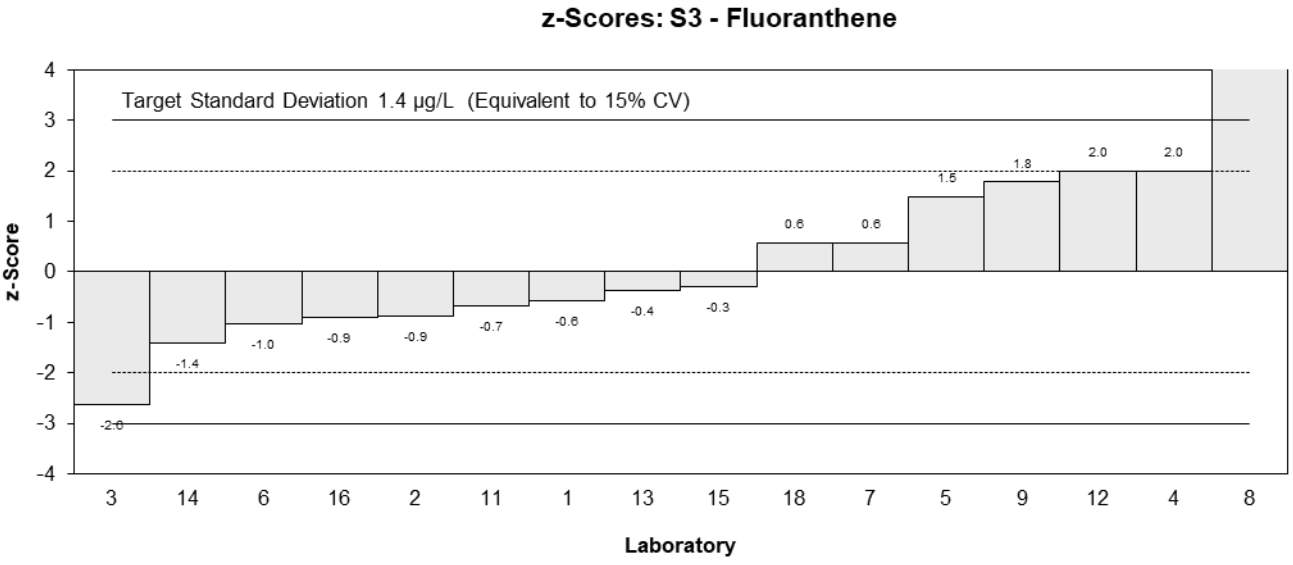
Statistics

Assigned Value*	9.0	1.4
Spike	12.0	0.6
Max. Acceptable Concentration**	14.7	
Robust Average	9.2	1.4
Median	8.55	0.96
Mean	9.70	
N	16	
Max.	20.3	
Min.	5.44	
Robust SD	2.3	
Robust CV	25%	

* Robust average excluding Laboratory 8.

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


Kernel Density
Density
Result
Results: S3 - Fluoranthene
Assigned Value = 9.0 ± 1.4 µg/L
Maximum Acceptable Concentration
Results (µg/L)
3 14 6 16 2 11 1 13 15 18 7 5 9 12 4 8 10 17 Md R.A. S.V.
Laboratory


z-Scores: S3 - Fluoranthene
Target Standard Deviation 1.4 µg/L (Equivalent to 15% CV)
z-Score
-2.6 -1.4 -1.0 -0.9 -0.9 -0.7 -0.6 -0.4 -0.3 0.6 0.6 1.5 1.8 2.0 2.0
3 14 6 16 2 11 1 13 15 18 7 5 9 12 4 8
Laboratory

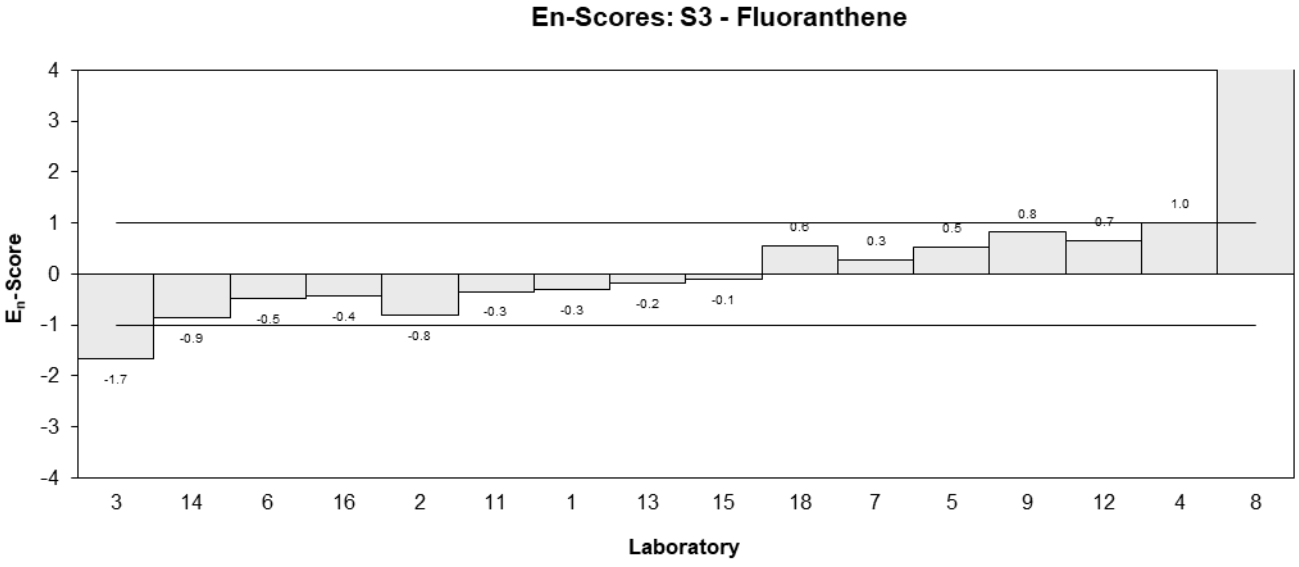

En-Scores: S3 - Fluoranthene
En-Score
-1.7 -0.9 -0.5 -0.4 -0.8 -0.3 -0.3 -0.2 -0.1 0.6 0.3 0.5 0.8 0.7 1.0
3 14 6 16 2 11 1 13 15 18 7 5 9 12 4 8
Laboratory

Figure 13

Table 19

Sample Details

Sample No.	S3
Matrix	Water
Analyte	Fluorene
Units	µg/L

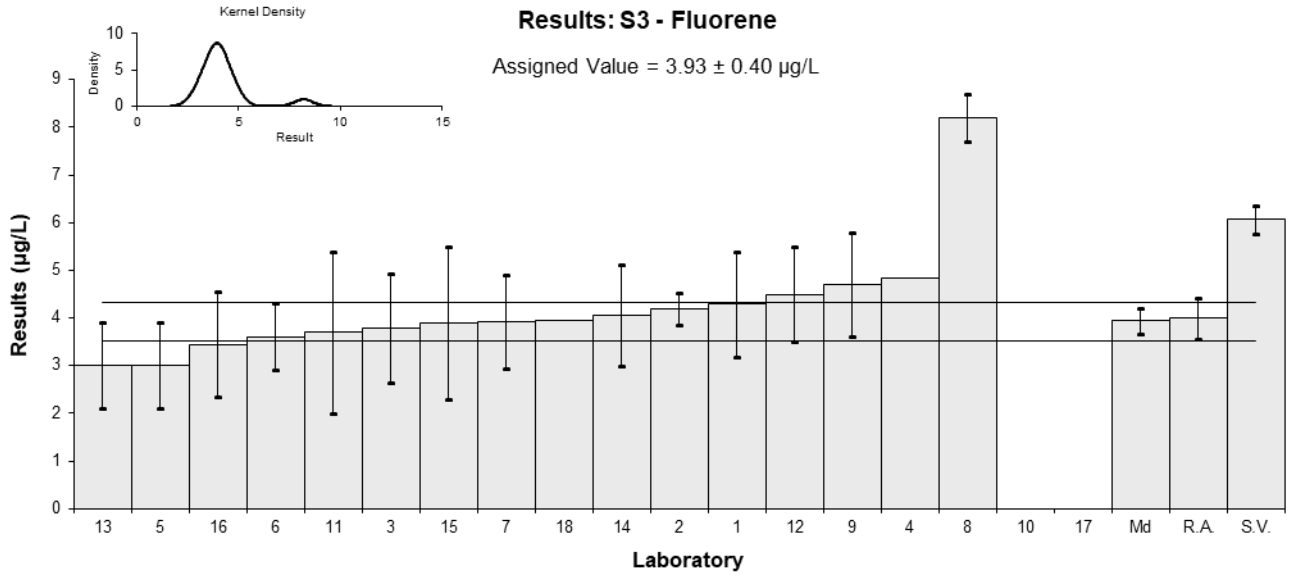
Participant Results

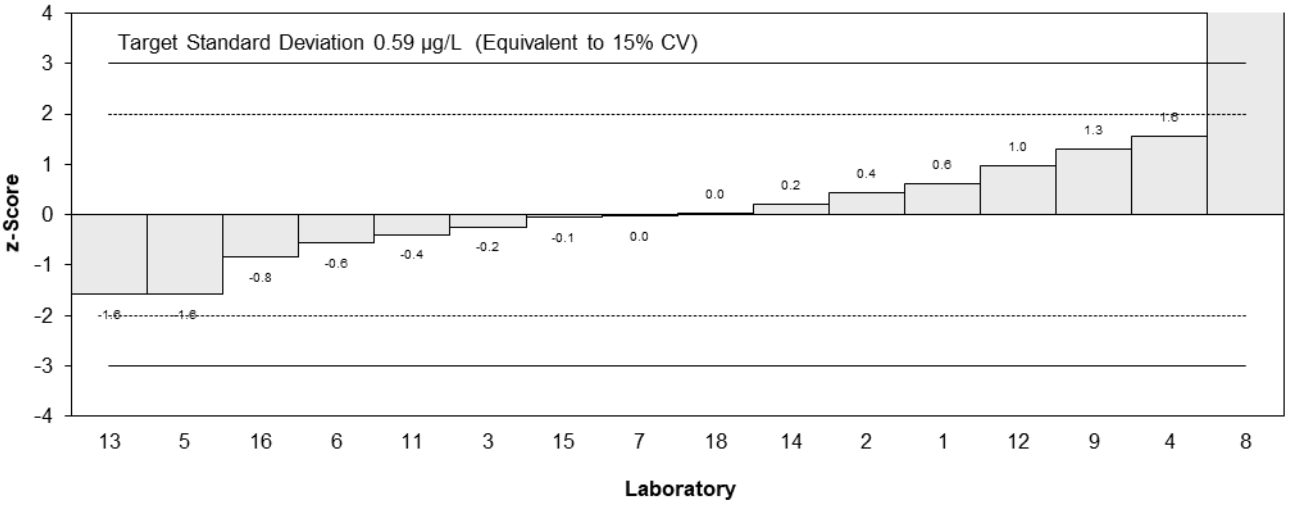
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	4.29	1.1	0.61	0.31
2	4.19	0.34	0.44	0.50
3	3.79	1.14	-0.24	-0.12
4	4.85	NR	1.56	2.30
5	3.0	0.9	-1.58	-0.94
6	3.6	0.7	-0.56	-0.41
7	3.92	0.98	-0.02	-0.01
8	8.21	0.5	7.26	6.68
9	4.7	1.1	1.31	0.66
10	NR	NR		
11	3.7	1.7	-0.39	-0.13
12	4.5	1	0.97	0.53
13	3	0.9	-1.58	-0.94
14	4.05	1.06	0.20	0.11
15	3.9	1.6	-0.05	-0.02
16	3.438	1.104	-0.83	-0.42
17	NT	NT		
18	3.95	NR	0.03	0.05

Statistics

Assigned Value*	3.93	0.40
Spike	6.07	0.30
Robust Average	3.99	0.42
Median	3.94	0.27
Mean	4.19	
N	16	
Max.	8.21	
Min.	3	
Robust SD	0.67	
Robust CV	17%	

* Robust average excluding Laboratory 8.


Kernel Density
Density
Result
Results: S3 - Fluorene
Assigned Value = 3.93 ± 0.40 µg/L
Results (µg/L)
13 5 16 6 11 3 15 7 18 14 2 1 12 9 4 8 10 17 Md R.A. S.V.
Laboratory


z-Scores: S3 - Fluorene
Target Standard Deviation 0.59 µg/L (Equivalent to 15% CV)
z-Score
-1.6 -1.6 -0.8 -0.6 -0.4 -0.2 -0.1 0.0 0.0 0.2 0.4 0.6 1.0 1.3 1.6
13 5 16 6 11 3 15 7 18 14 2 1 12 9 4 8
Laboratory

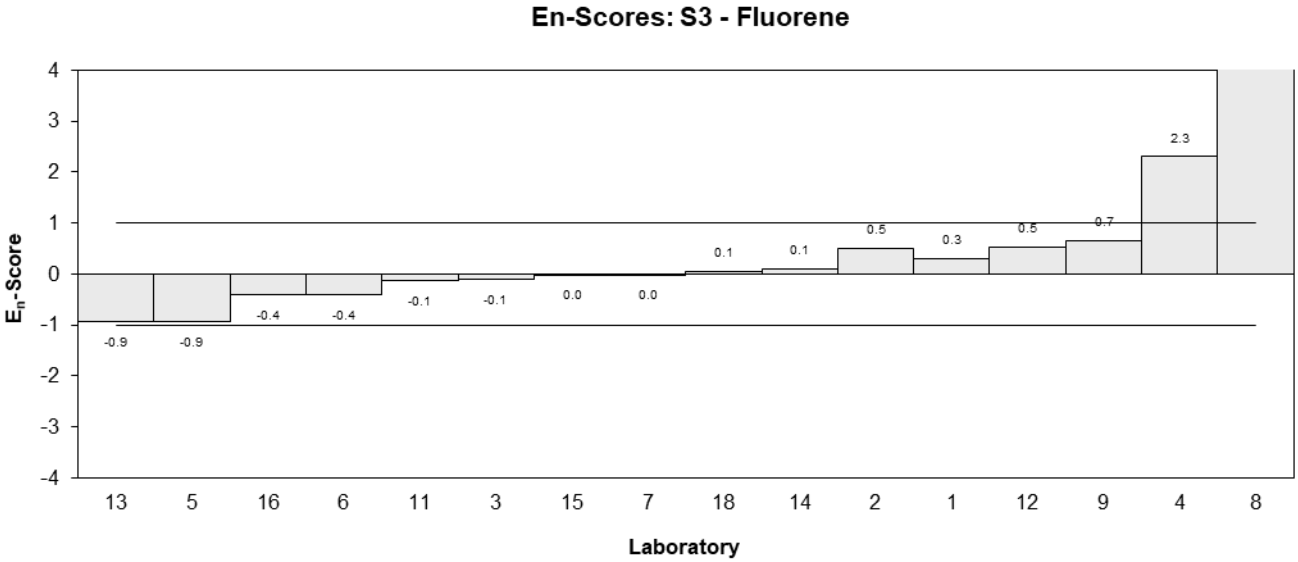

En-Scores: S3 - Fluorene
En-Score
-0.9 -0.9 -0.4 -0.4 -0.1 -0.1 0.0 0.0 0.1 0.1 0.5 0.3 0.5 0.7 2.3
13 5 16 6 11 3 15 7 18 14 2 1 12 9 4 8
Laboratory

Figure 14

Table 20

Sample Details

Sample No.	S3
Matrix	Water
Analyte	Phenanthrene
Units	µg/L

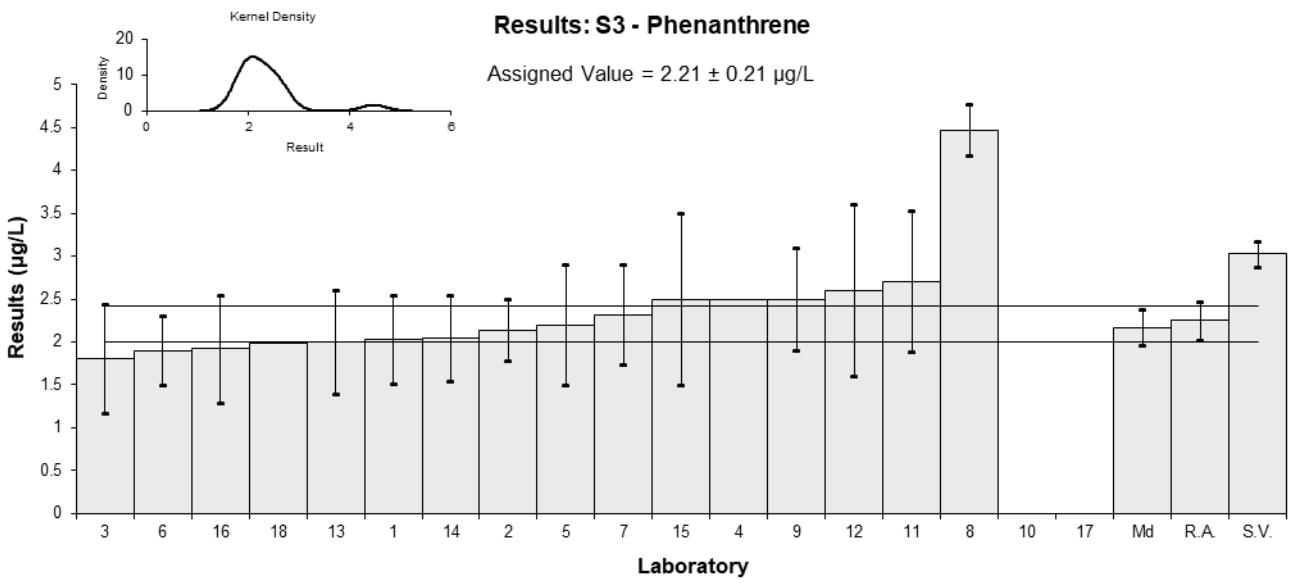
Participant Results

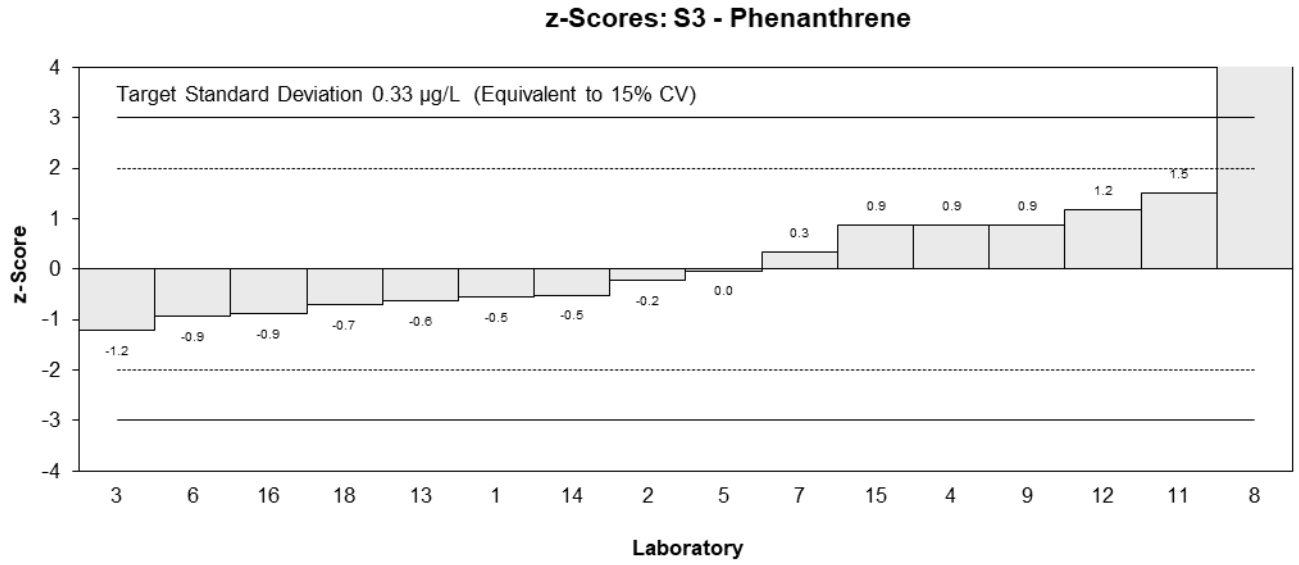
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	2.03	0.51	-0.54	-0.33
2	2.14	0.36	-0.21	-0.17
3	1.81	0.634	-1.21	-0.60
4	2.5	NR	0.87	1.38
5	2.2	0.7	-0.03	-0.01
6	1.9	0.4	-0.94	-0.69
7	2.32	0.58	0.33	0.18
8	4.47	0.3	6.82	6.17
9	2.5	0.6	0.87	0.46
10	NR	NR		
11	2.71	0.82	1.51	0.59
12	2.6	1	1.18	0.38
13	2	0.6	-0.63	-0.33
14	2.04	0.5	-0.51	-0.31
15	2.5	1.0	0.87	0.28
16	1.919	0.627	-0.88	-0.44
17	NT	NT		
18	1.98	NR	-0.69	-1.10

Statistics

Assigned Value*	2.21	0.21
Spike	3.03	0.15
Robust Average	2.25	0.22
Median	2.17	0.21
Mean	2.35	
N	16	
Max.	4.47	
Min.	1.81	
Robust SD	0.36	
Robust CV	16%	

* Robust average excluding Laboratory 8.


Kernel Density
Density
Result
Results: S3 - Phenanthrene
Assigned Value = 2.21 ± 0.21 µg/L
Results (µg/L)
3 6 16 18 13 1 14 2 5 7 15 4 9 12 11 8 10 17 Md R.A. S.V.
Laboratory


z-Scores: S3 - Phenanthrene
Target Standard Deviation 0.33 µg/L (Equivalent to 15% CV)
z-Score
-1.2 -0.9 -0.9 -0.7 -0.6 -0.5 -0.5 -0.2 0.0 0.3 0.9 0.9 0.9 1.2 1.5
3 6 16 18 13 1 14 2 5 7 15 4 9 12 11 8
Laboratory

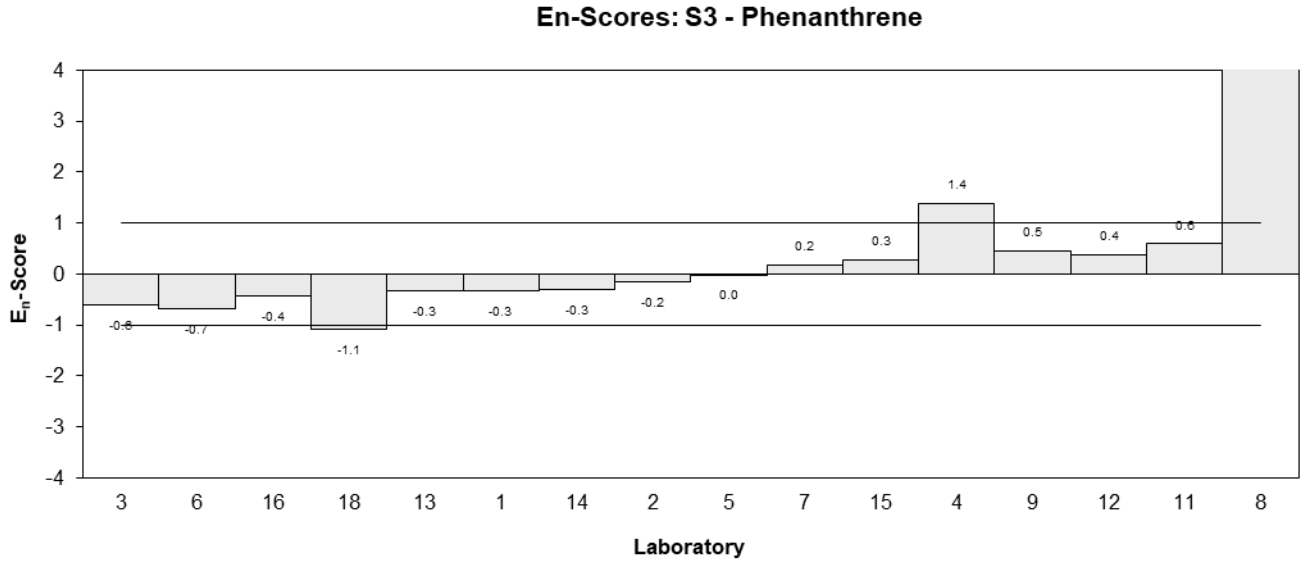

En-Scores: S3 - Phenanthrene
En-Score
-0.6 -0.7 -0.4 -1.1 -0.3 -0.3 -0.3 -0.2 0.0 0.2 0.3 1.4 0.5 0.4 0.6
3 6 16 18 13 1 14 2 5 7 15 4 9 12 11 8
Laboratory

Figure 15

Table 21

Sample Details

Sample No.	S3
Matrix	Water
Analyte	Pyrene
Units	µg/L

Participant Results

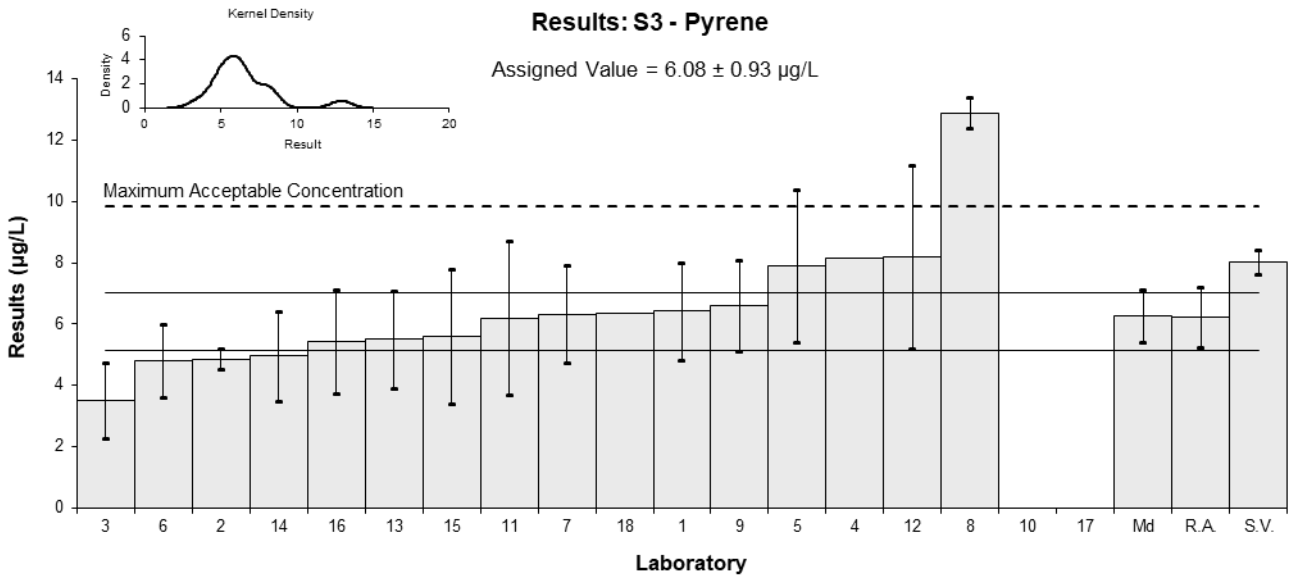
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	6.42	1.6	0.37	0.18
2	4.85	0.34	-1.35	-1.24
3	3.52	1.23	-2.81	-1.66
4**	8.15	NR	2.00	1.00
5	7.9	2.5	2.00	0.68
6	4.8	1.2	-1.40	-0.84
7	6.32	1.58	0.26	0.13
8	12.90	0.5	7.48	6.46
9	6.6	1.5	0.57	0.29
10	NR	NR		
11	6.2	2.5	0.13	0.04
12**	8.2	3	2.00	0.67
13	5.5	1.6	-0.64	-0.31
14	4.96	1.47	-1.23	-0.64
15	5.6	2.2	-0.53	-0.20
16	5.417	1.693	-0.73	-0.34
17	NT	NT		
18	6.36	NR	0.31	0.30

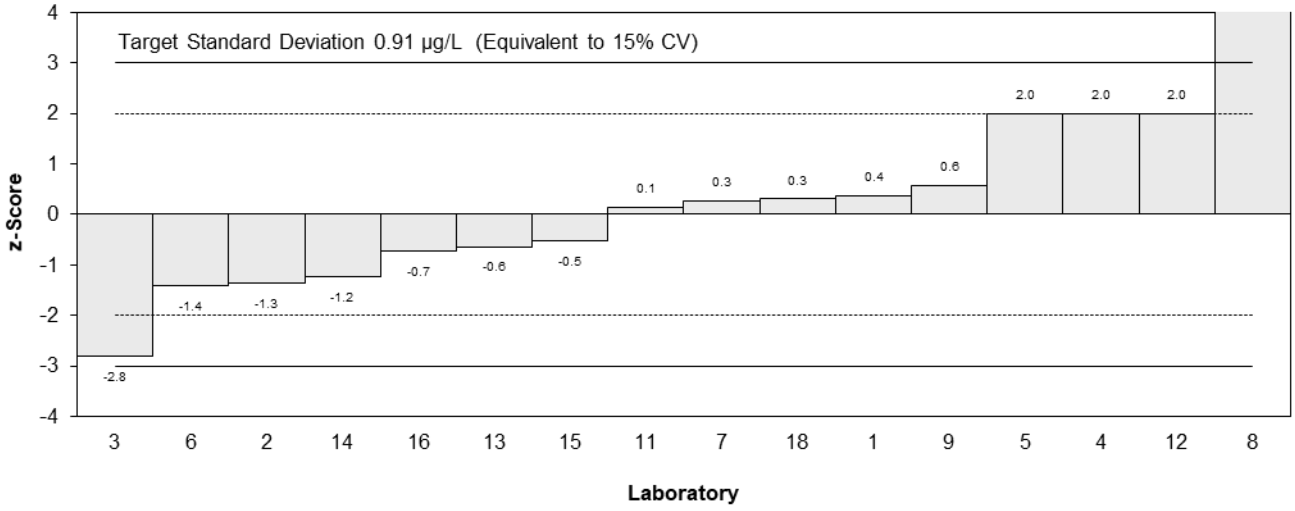
Statistics

Assigned Value*	6.08	0.93
Spike	8.02	0.40
Max. Acceptable Concentration**	9.84	
Robust Average	6.23	0.98
Median	6.26	0.85
Mean	6.48	
N	16	
Max.	12.90	
Min.	3.52	
Robust SD	1.6	
Robust CV	25%	

* Robust average excluding Laboratory 8.

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


Results: S3 - Pyrene
Kernel Density
Density
Result
Assigned Value = 6.08 ± 0.93 µg/L
Maximum Acceptable Concentration
Results (µg/L)
3 6 2 14 16 13 15 11 7 18 1 9 5 4 12 8 10 17 Md R.A. S.V.
Laboratory


z-Scores: S3 - Pyrene
Target Standard Deviation 0.91 µg/L (Equivalent to 15% CV)
z-Score
-2.8 -1.4 -1.3 -1.2 -0.7 -0.6 -0.5 0.1 0.3 0.3 0.4 0.6 2.0 2.0 2.0
3 6 2 14 16 13 15 11 7 18 1 9 5 4 12 8
Laboratory

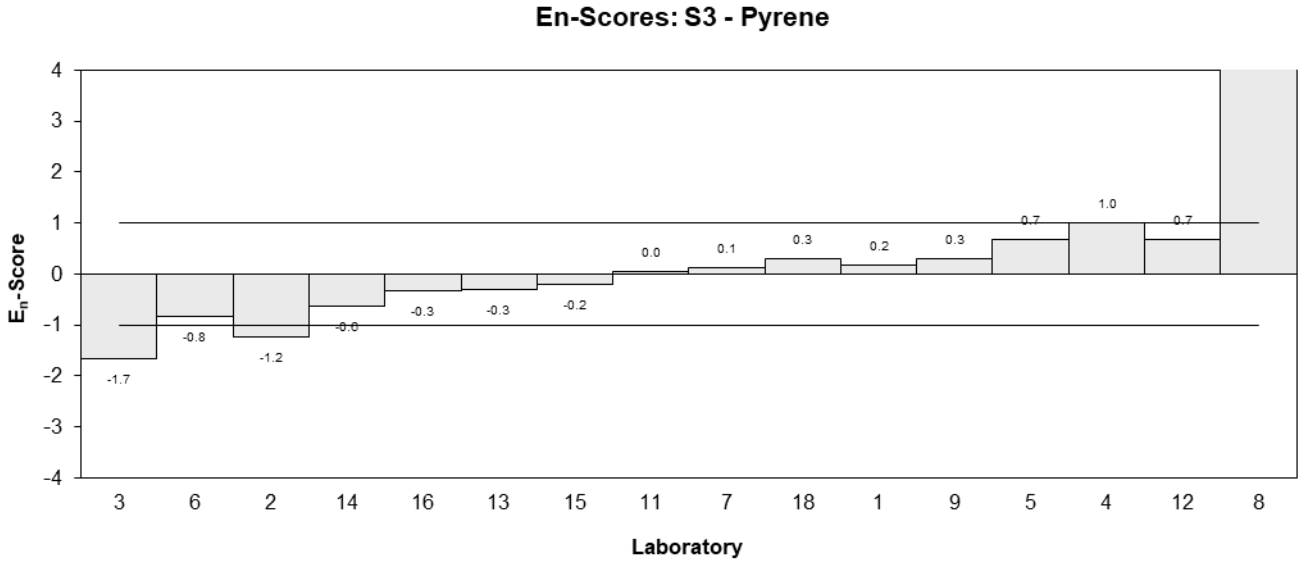

En-Scores: S3 - Pyrene
En-Score
-1.7 -0.8 -1.2 -0.6 -0.3 -0.3 -0.2 0.0 0.1 0.3 0.2 0.3 0.7 1.0 0.7
3 6 2 14 16 13 15 11 7 18 1 9 5 4 12 8
Laboratory

Figure 16

Table 22

Sample Details

Sample No.	S4
Matrix	Water
Analyte	Anthracene
Units	µg/L

Participant Results

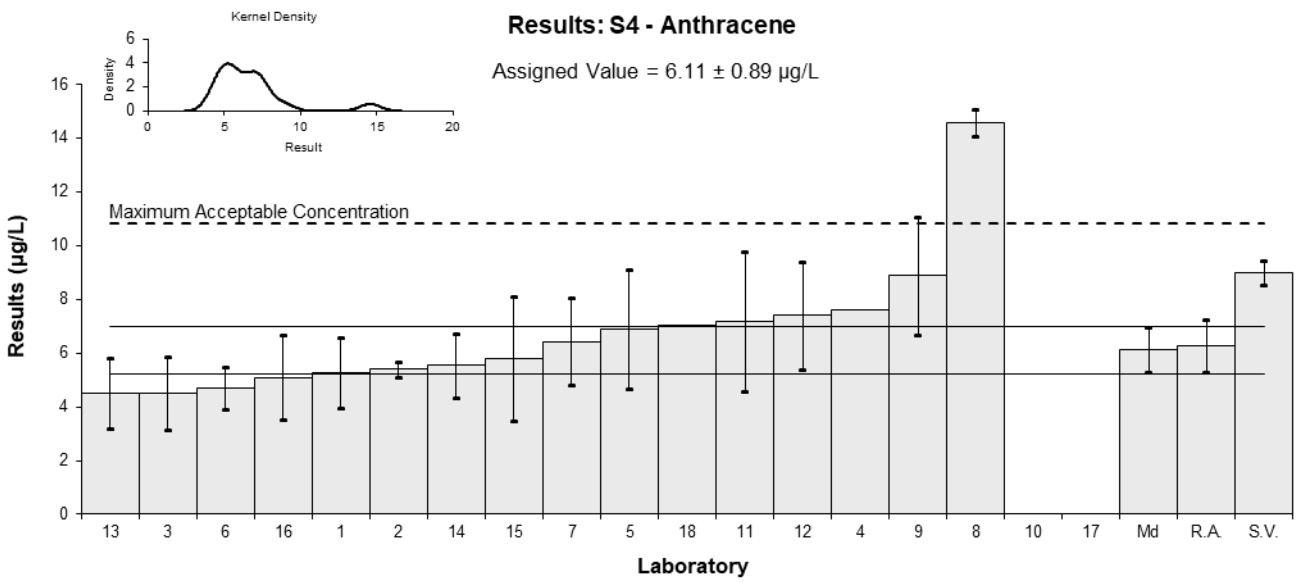
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	5.28	1.3	-0.91	-0.53
2	5.4	0.27	-0.77	-0.76
3	4.52	1.36	-1.73	-0.98
4	7.6	NR	1.63	1.67
5	6.9	2.2	0.86	0.33
6	4.7	0.8	-1.54	-1.18
7	6.44	1.61	0.36	0.18
8	14.57	0.5	9.23	8.29
9**	8.9	2.2	2.00	1.00
10	NR	NR		
11	7.2	2.6	1.19	0.40
12	7.4	2	1.41	0.59
13	4.5	1.3	-1.76	-1.02
14	5.54	1.21	-0.62	-0.38
15	5.8	2.3	-0.34	-0.13
16	5.106	1.591	-1.10	-0.55
17	NT	NT		
18	7.02	NR	0.99	1.02

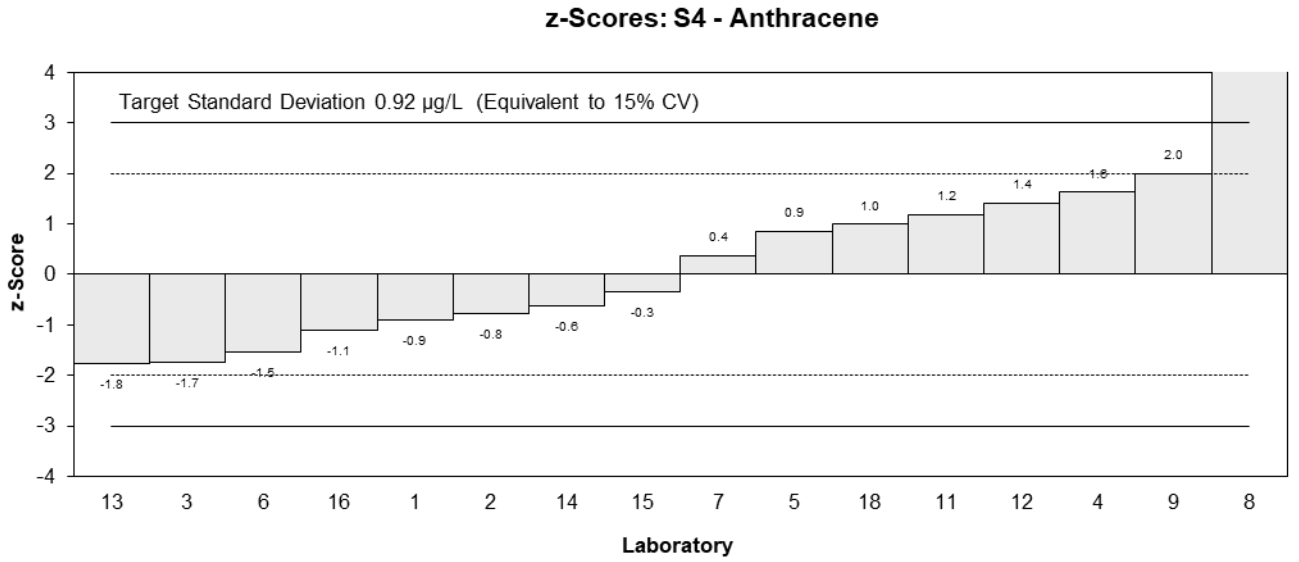
Statistics

Assigned Value*	6.11	0.89
Spike	8.99	0.45
Max. Acceptable Concentration**	10.82	
Robust Average	6.29	0.98
Median	6.12	0.83
Mean	6.68	
N	16	
Max.	14.57	
Min.	4.5	
Robust SD	1.6	
Robust CV	25%	

* Robust average excluding Laboratory 8.

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


Kernel Density
Density
Result
Results: S4 - Anthracene
Assigned Value = 6.11 ± 0.89 µg/L
Maximum Acceptable Concentration
Results (µg/L)
13 3 6 16 1 2 14 15 7 5 18 11 12 4 9 8 10 17 Md R.A. S.V.
Laboratory


z-Scores: S4 - Anthracene
Target Standard Deviation 0.92 µg/L (Equivalent to 15% CV)
z-Score
-1.8 -1.7 -1.5 -1.1 -0.9 -0.8 -0.6 -0.3 0.4 0.9 1.0 1.2 1.4 1.6 2.0
13 3 6 16 1 2 14 15 7 5 18 11 12 4 9 8
Laboratory

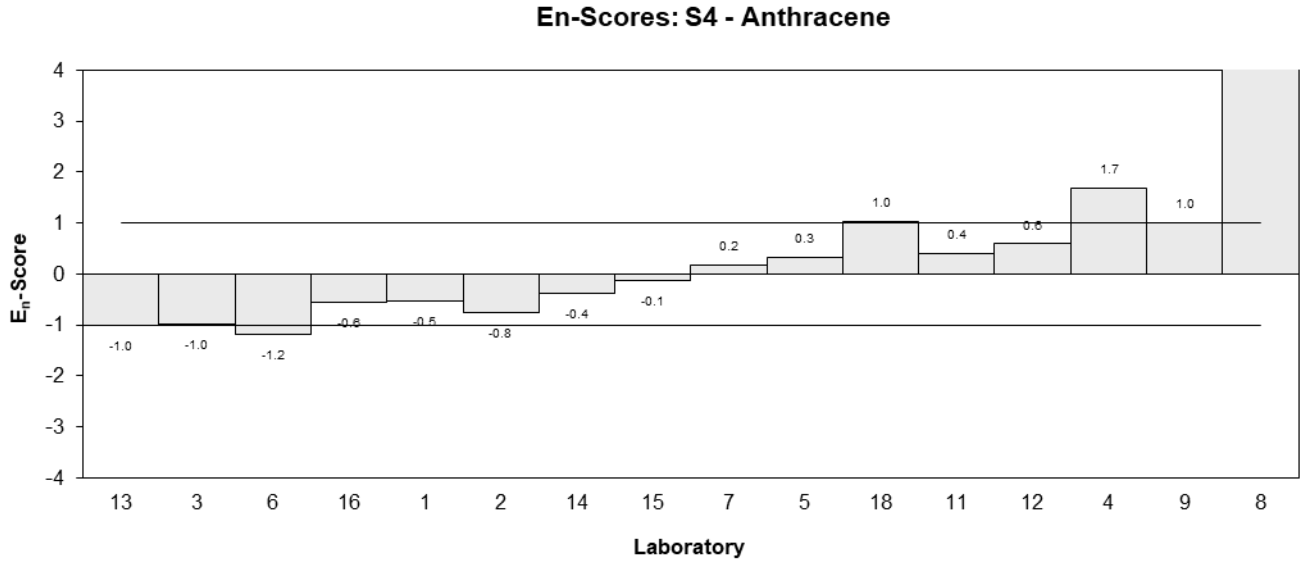

En-Scores: S4 - Anthracene
En-Score
-1.0 -1.0 -1.2 -0.6 -0.5 -0.8 -0.4 -0.1 0.2 0.3 1.0 0.4 0.6 1.7 1.0
13 3 6 16 1 2 14 15 7 5 18 11 12 4 9 8
Laboratory

Figure 17

Table 23

Sample Details

Sample No.	S4
Matrix	Water
Analyte	Benzo(a)pyrene
Units	µg/L

Participant Results

Lab. Code	Result	Uncertainty
1	0.41	0.1
2	<1	0.3
3	<1	0.3
4	0.79	NR
5	0.7	0.2
6	0.47	0.16
7	0.6	0.15
8	1.25	0.1
9	<0.1	NR
10	NR	NR
11	0.52	0.26
12	<1	1
13	0.35	0.1
14	< 1	0.3
15	0.73	0.14
16	< 1	0.3
17	NT	NT
18	0.683	NR

Statistics

Assigned Value	Not Set	
Spike	0.921	0.046
Robust Average	0.62	0.16
Median	0.64	0.14
Mean	0.65	
N	10	
Max.	1.25	
Min.	0.35	
Robust SD	0.21	
Robust CV	33%	

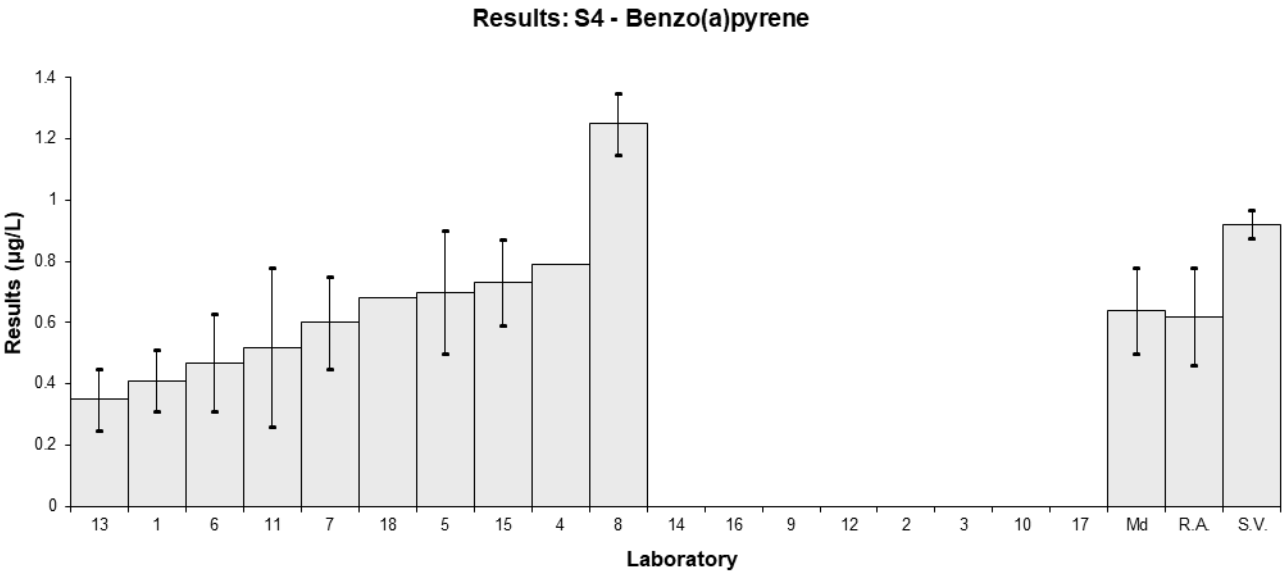

Results: S4 - Benzo(a)pyrene
Results (µg/L)
1.4
1.2
1
0.8
0.6
0.4
0.2
0
13
1
6
11
7
18
5
15
4
8
14
16
9
12
2
3
10
17
Md
R.A.
S.V.
Laboratory

Figure 18

Table 24

Sample Details

Sample No.	S4
Matrix	Water
Analyte	Fluoranthene
Units	µg/L

Participant Results

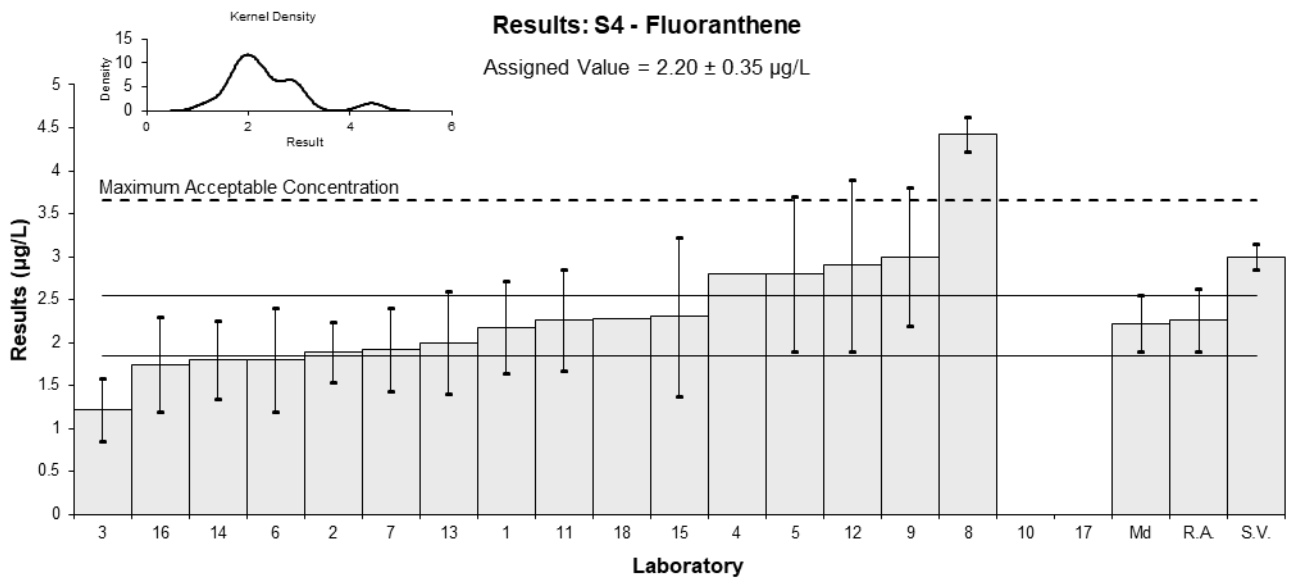
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	2.18	0.54	-0.06	-0.03
2	1.89	0.35	-0.94	-0.63
3	1.22	0.367	-2.97	-1.93
4	2.8	NR	1.82	1.71
5	2.8	0.9	1.82	0.62
6	1.8	0.6	-1.21	-0.58
7	1.92	0.48	-0.85	-0.47
8	4.42	0.2	6.73	5.51
9**	3	0.8	2.00	0.92
10	NR	NR		
11	2.26	0.59	0.18	0.09
12**	2.9	1	2.00	0.66
13	2	0.6	-0.61	-0.29
14	1.8	0.45	-1.21	-0.70
15	2.3	0.92	0.30	0.10
16	1.747	0.558	-1.37	-0.69
17	NT	NT		
18	2.28	NR	0.24	0.23

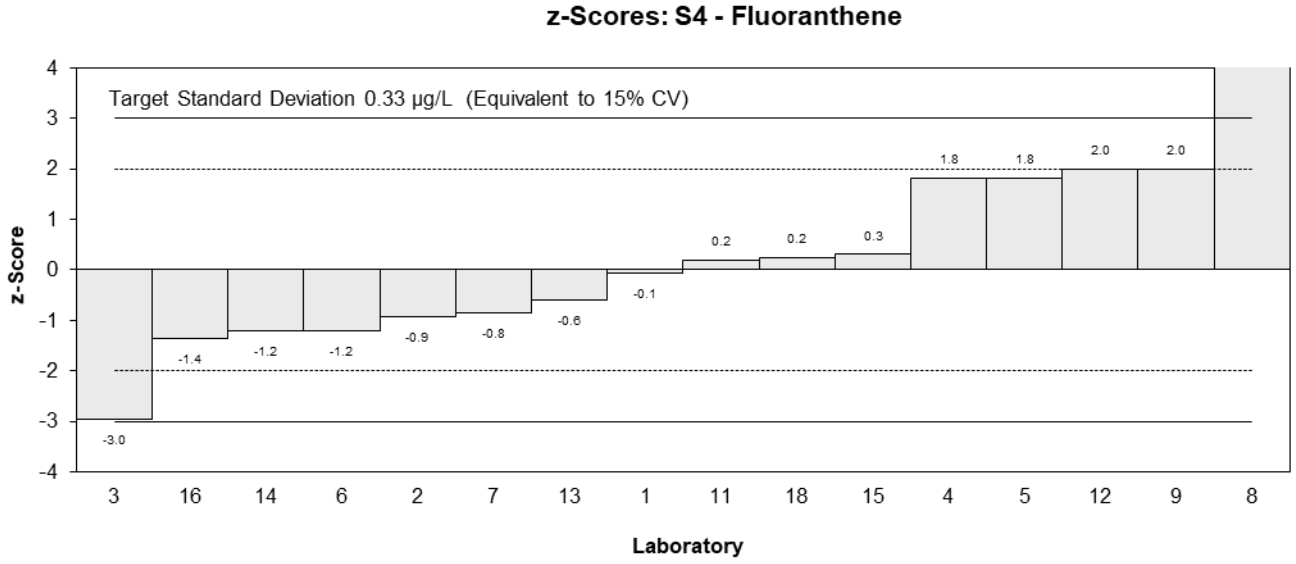
Statistics

Assigned Value*	2.20	0.35
Spike	3.00	0.15
Max. Acceptable Concentration**	3.66	
Robust Average	2.26	0.37
Median	2.22	0.33
Mean	2.33	
N	16	
Max.	4.42	
Min.	1.22	
Robust SD	0.60	
Robust CV	26%	

* Robust average excluding Laboratory 8.

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


Kernel Density
Density
Result
Results: S4 - Fluoranthene
Assigned Value = 2.20 ± 0.35 µg/L
Maximum Acceptable Concentration
Results (µg/L)
3 16 14 6 2 7 13 1 11 18 15 4 5 12 9 8 10 17 Md R.A. S.V.
Laboratory


z-Scores: S4 - Fluoranthene
Target Standard Deviation 0.33 µg/L (Equivalent to 15% CV)
z-Score
-3.0 -1.4 -1.2 -1.2 -0.9 -0.8 -0.6 -0.1 0.2 0.2 0.3 1.8 1.8 2.0 2.0
3 16 14 6 2 7 13 1 11 18 15 4 5 12 9 8
Laboratory

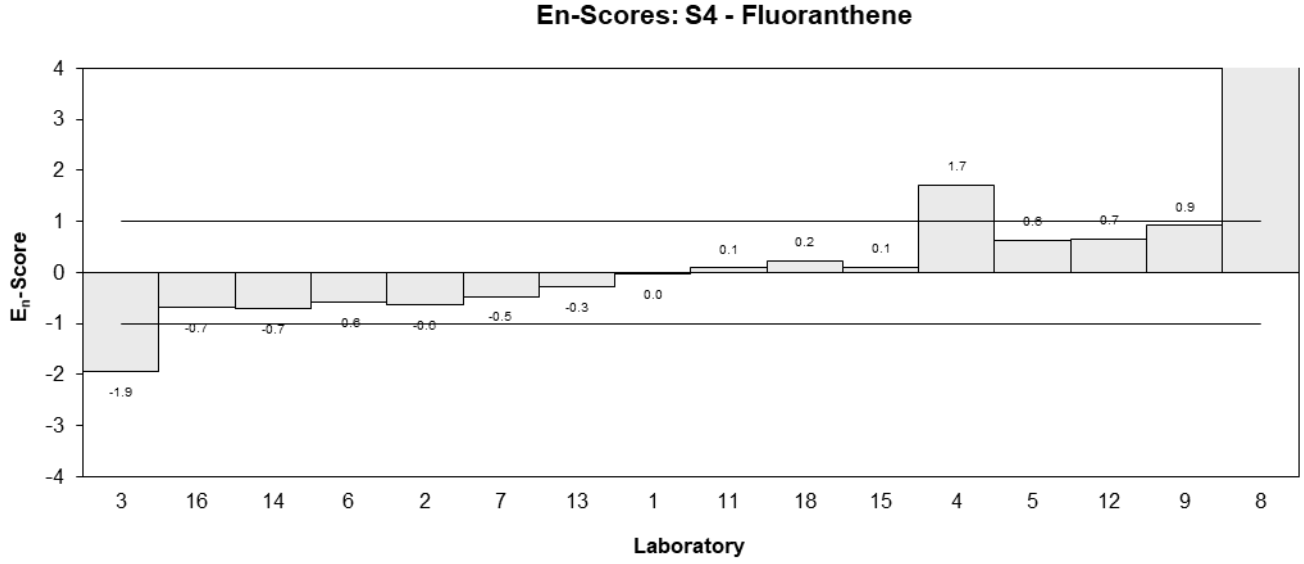

En-Scores: S4 - Fluoranthene
En-Score
-1.9 -0.7 -0.7 -0.6 -0.6 -0.5 -0.3 0.0 0.1 0.2 0.1 1.7 0.6 0.7 0.9
3 16 14 6 2 7 13 1 11 18 15 4 5 12 9 8
Laboratory

Figure 19

Table 25

Sample Details

Sample No.	S4
Matrix	Water
Analyte	Fluorene
Units	µg/L

Participant Results

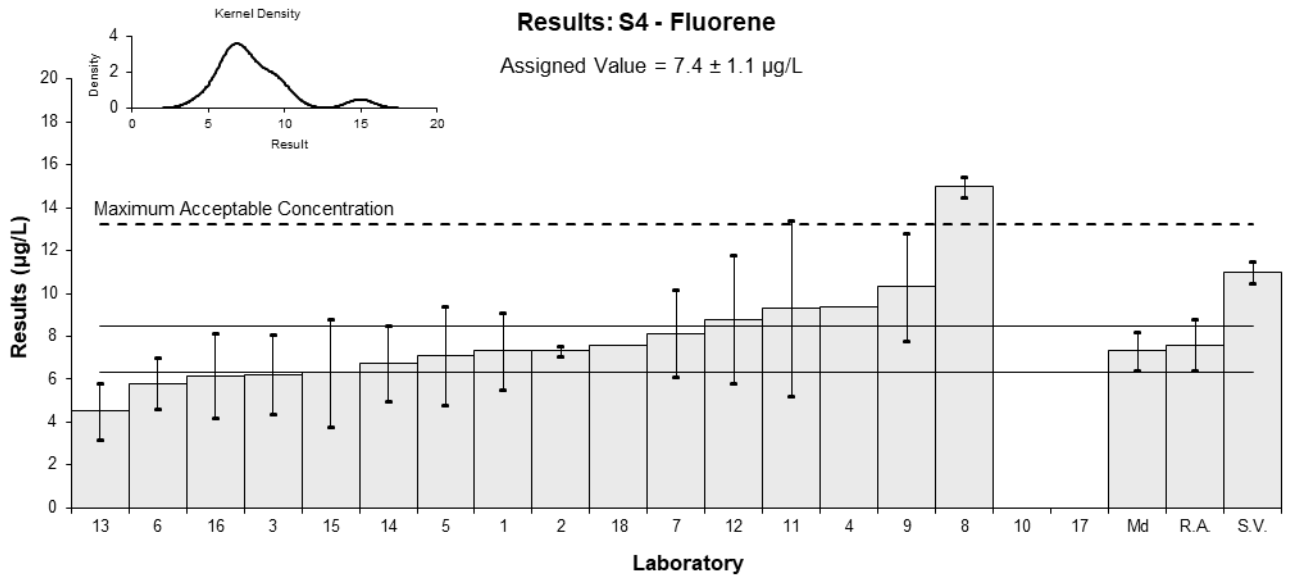
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	7.31	1.8	-0.08	-0.04
2	7.31	0.24	-0.08	-0.08
3	6.23	1.87	-1.05	-0.54
4	9.4	NR	1.80	1.82
5	7.1	2.3	-0.27	-0.12
6	5.8	1.2	-1.44	-0.98
7	8.14	2.03	0.67	0.32
8	14.97	0.5	6.82	6.26
9**	10.3	2.5	2.00	1.00
10	NR	NR		
11	9.3	4.1	1.71	0.45
12	8.8	3	1.26	0.44
13	4.5	1.3	-2.61	-1.70
14	6.74	1.76	-0.59	-0.32
15	6.3	2.5	-0.99	-0.40
16	6.150	1.976	-1.13	-0.55
17	NT	NT		
18	7.59	NR	0.17	0.17

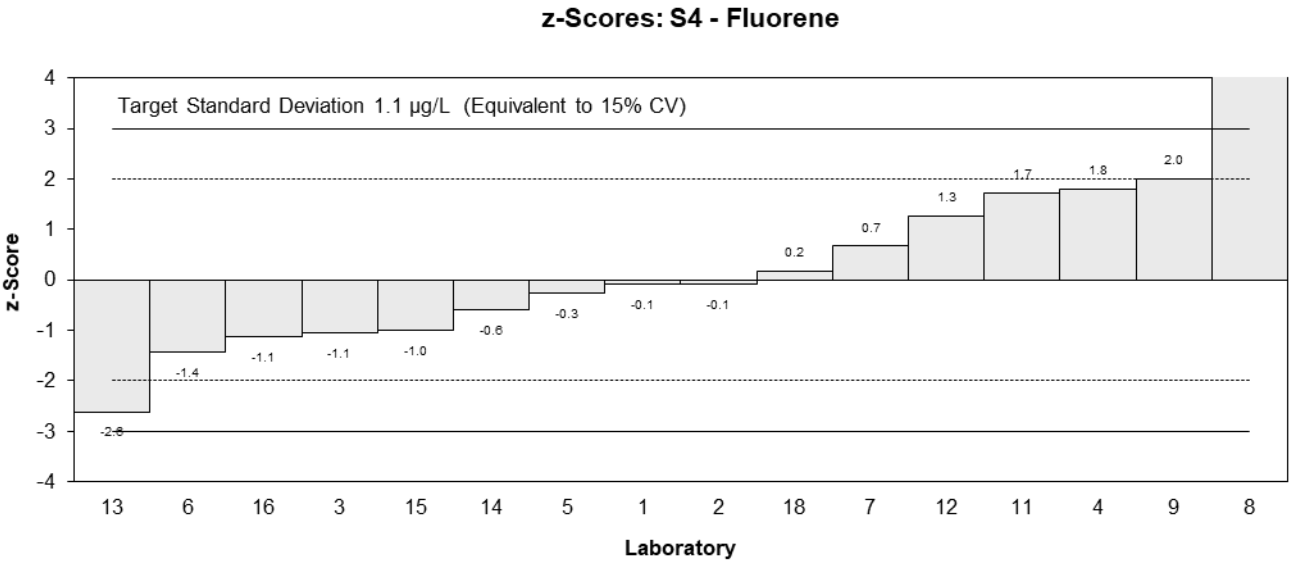
Statistics

Assigned Value*	7.4	1.1
Spike	11.0	0.5
Max. Acceptable Concentration**	13.2	
Robust Average	7.6	1.2
Median	7.31	0.88
Mean	7.87	
N	16	
Max.	14.97	
Min.	4.5	
Robust SD	1.9	
Robust CV	25%	

* Robust average excluding Laboratory 8.

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


Kernel Density
Density
Result
Results: S4 - Fluorene
Assigned Value = 7.4 ± 1.1 µg/L
Maximum Acceptable Concentration
Results (µg/L)
13 6 16 3 15 14 5 1 2 18 7 12 11 4 9 8 10 17 Md R.A. S.V.
Laboratory


z-Scores: S4 - Fluorene
Target Standard Deviation 1.1 µg/L (Equivalent to 15% CV)
z-Score
-2.6 -1.4 -1.1 -1.1 -1.0 -0.6 -0.3 -0.1 -0.1 0.2 0.7 1.3 1.7 1.8 2.0
13 6 16 3 15 14 5 1 2 18 7 12 11 4 9 8
Laboratory

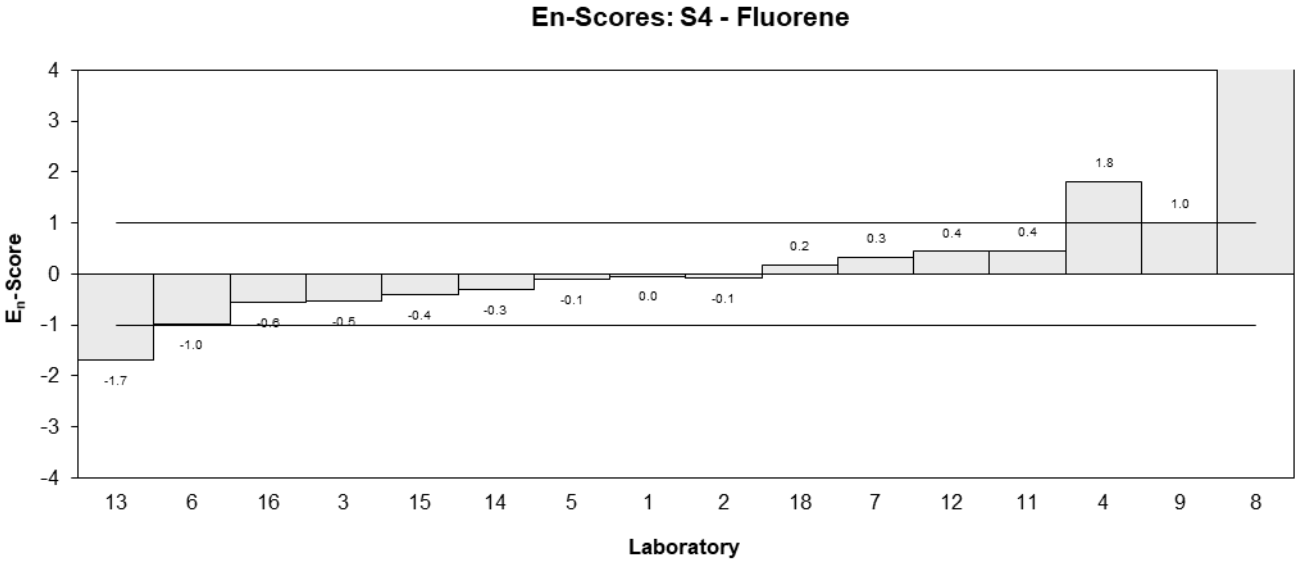

En-Scores: S4 - Fluorene
En-Score
-1.7 -1.0 -0.6 -0.5 -0.4 -0.3 -0.1 0.0 -0.1 0.2 0.3 0.4 0.4 1.8 1.0
13 6 16 3 15 14 5 1 2 18 7 12 11 4 9 8
Laboratory

Figure 20

Table 26

Sample Details

Sample No.	S4
Matrix	Water
Analyte	Phenanthrene
Units	µg/L

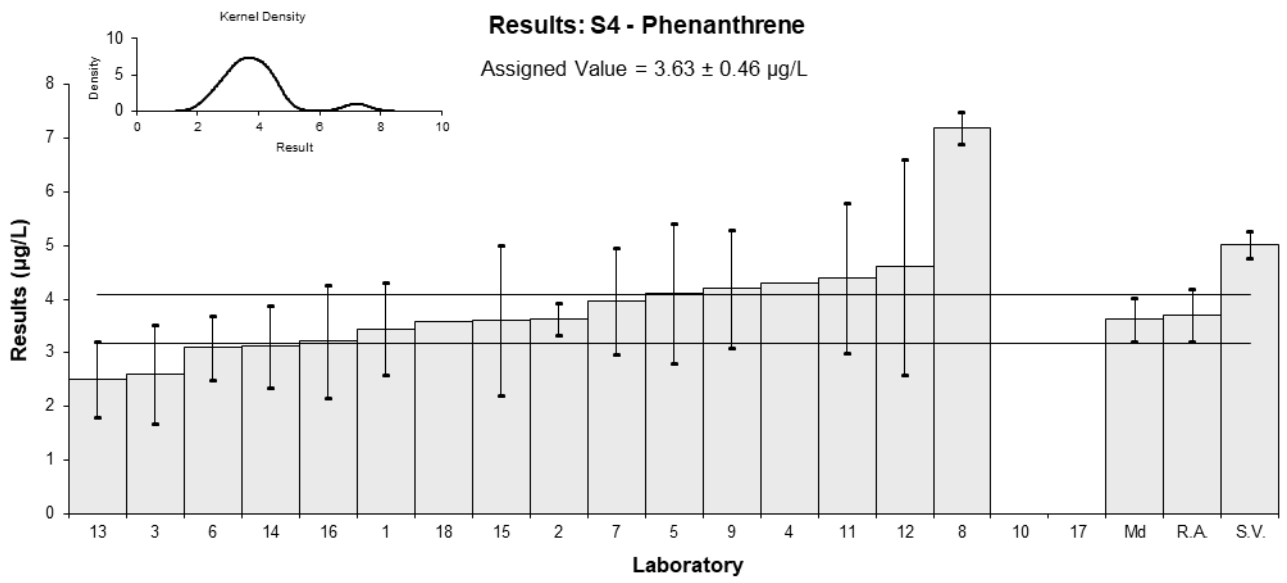
Participant Results

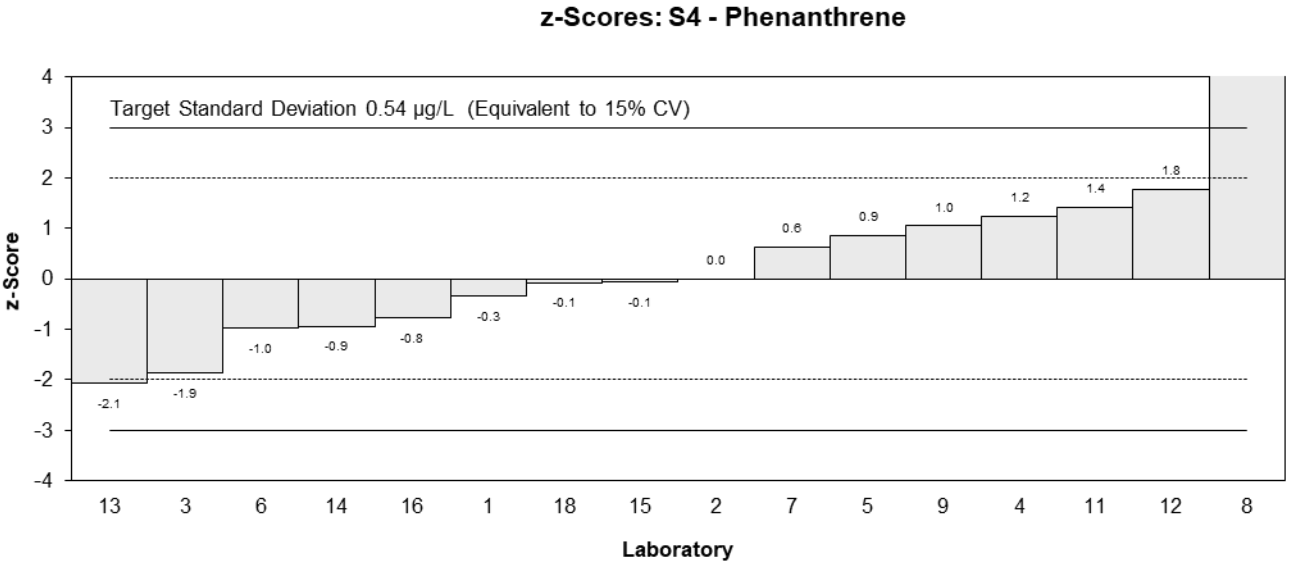
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	3.45	0.86	-0.33	-0.18
2	3.63	0.3	0.00	0.00
3	2.61	0.92	-1.87	-0.99
4	4.3	NR	1.23	1.46
5	4.1	1.3	0.86	0.34
6	3.1	0.6	-0.97	-0.70
7	3.97	0.99	0.62	0.31
8	7.20	0.3	6.56	6.50
9	4.2	1.1	1.05	0.48
10	NR	NR		
11	4.4	1.4	1.41	0.52
12	4.6	2	1.78	0.47
13	2.5	0.7	-2.08	-1.35
14	3.12	0.77	-0.94	-0.57
15	3.6	1.4	-0.06	-0.02
16	3.212	1.049	-0.77	-0.36
17	NT	NT		
18	3.59	NR	-0.07	-0.09

Statistics

Assigned Value*	3.63	0.46
Spike	5.02	0.25
Robust Average	3.71	0.49
Median	3.62	0.40
Mean	3.85	
N	16	
Max.	7.20	
Min.	2.5	
Robust SD	0.78	
Robust CV	21%	

* Robust average excluding Laboratory 8.


Kernel Density
Density
Result
Results: S4 - Phenanthrene
Assigned Value = 3.63 ± 0.46 µg/L
Results (µg/L)
13 3 6 14 16 1 18 15 2 7 5 9 4 11 12 8 10 17 Md R.A. S.V.
Laboratory


z-Scores: S4 - Phenanthrene
Target Standard Deviation 0.54 µg/L (Equivalent to 15% CV)
z-Score
-2.1 -1.9 -1.0 -0.9 -0.8 -0.3 -0.1 -0.1 0.0 0.6 0.9 1.0 1.2 1.4 1.8
13 3 6 14 16 1 18 15 2 7 5 9 4 11 12 8
Laboratory

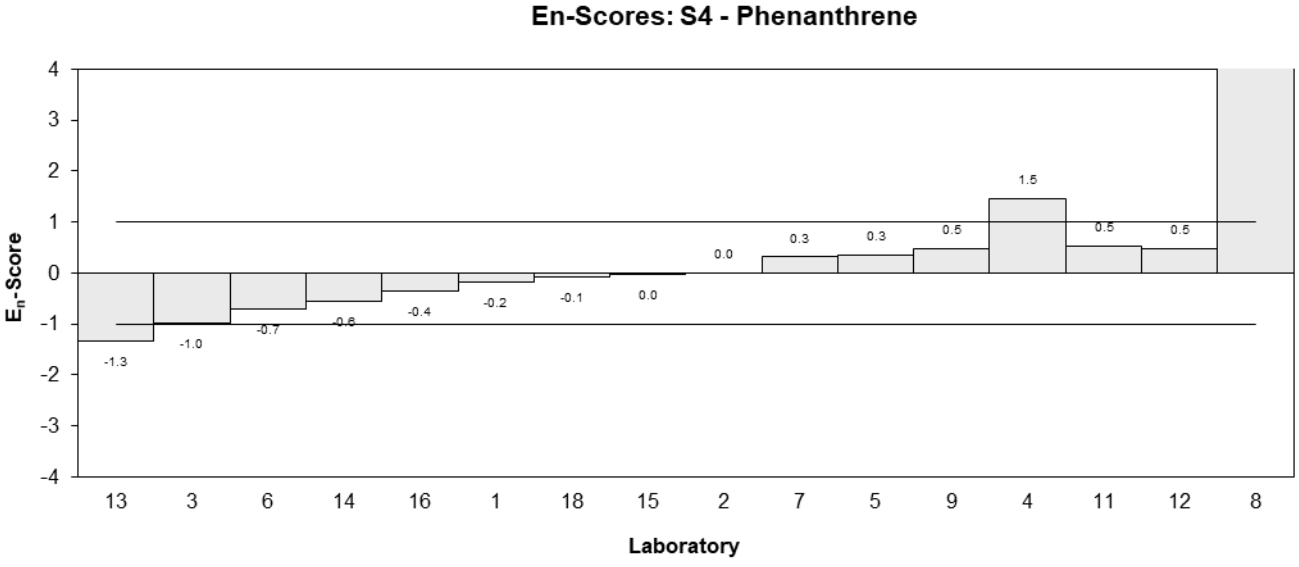

En-Scores: S4 - Phenanthrene
En-Score
-1.3 -1.0 -0.7 -0.6 -0.4 -0.2 -0.1 0.0 0.0 0.3 0.3 0.5 1.5 0.5 0.5
13 3 6 14 16 1 18 15 2 7 5 9 4 11 12 8
Laboratory

Figure 21

Table 27

Sample Details

Sample No.	S4
Matrix	Water
Analyte	Pyrene
Units	µg/L

Participant Results

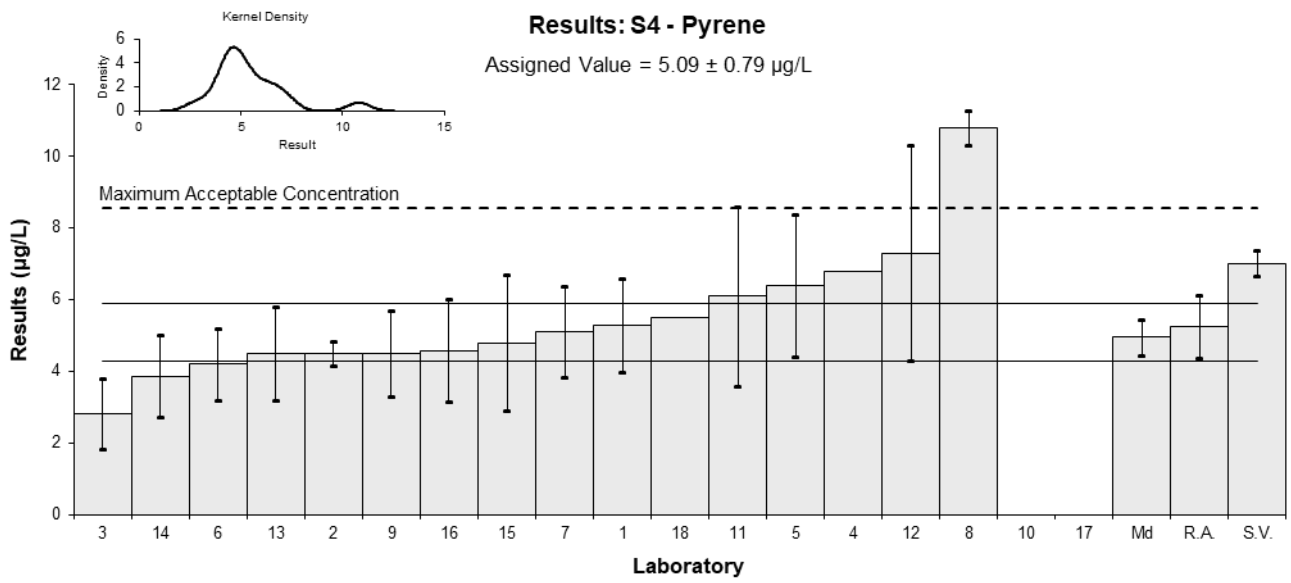
Lab. Code	Result	Uncertainty	z-Score	E_n-Score
1	5.29	1.3	0.26	0.13
2	4.5	0.33	-0.77	-0.69
3	2.81	0.983	-2.99	-1.81
4**	6.8	NR	2.00	1.00
5	6.4	2	1.72	0.61
6	4.2	1.0	-1.17	-0.70
7	5.1	1.28	0.01	0.01
8	10.80	0.5	7.48	6.11
9	4.5	1.2	-0.77	-0.41
10	NR	NR		
11	6.1	2.5	1.32	0.39
12**	7.3	3	2.00	0.71
13	4.5	1.3	-0.77	-0.39
14	3.86	1.15	-1.61	-0.88
15	4.8	1.9	-0.38	-0.14
16	4.573	1.429	-0.68	-0.32
17	NT	NT		
18	5.49	NR	0.52	0.51

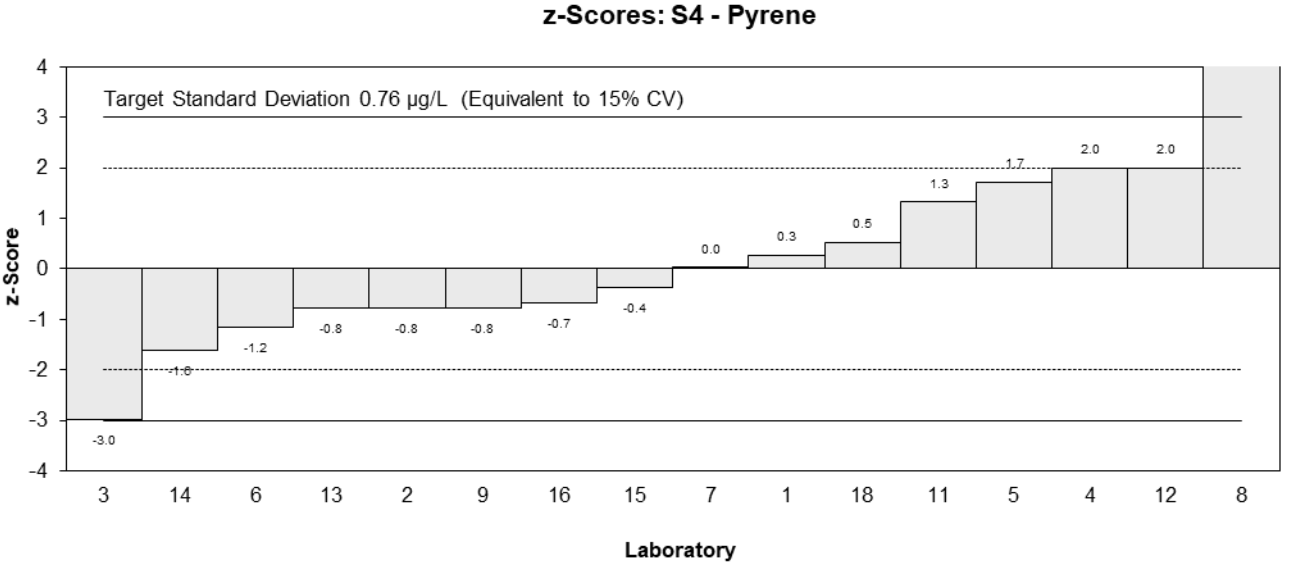
Statistics

Assigned Value*	5.09	0.79
Spike	7.02	0.35
Max. Acceptable Concentration**	8.55	
Robust Average	5.24	0.88
Median	4.95	0.51
Mean	5.44	
N	16	
Max.	10.80	
Min.	2.81	
Robust SD	1.4	
Robust CV	27%	

* Robust average excluding Laboratory 8.

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


Kernel Density
Density
Result
Results: S4 - Pyrene
Assigned Value = 5.09 ± 0.79 µg/L
Maximum Acceptable Concentration
Results (µg/L)
3 14 6 13 2 9 16 15 7 1 18 11 5 4 12 8 10 17 Md R.A. S.V.
Laboratory


z-Scores: S4 - Pyrene
Target Standard Deviation 0.76 µg/L (Equivalent to 15% CV)
z-Score
-3.0 -1.6 -1.2 -0.8 -0.8 -0.8 -0.7 -0.4 0.0 0.3 0.5 1.3 1.7 2.0 2.0
3 14 6 13 2 9 16 15 7 1 18 11 5 4 12 8
Laboratory

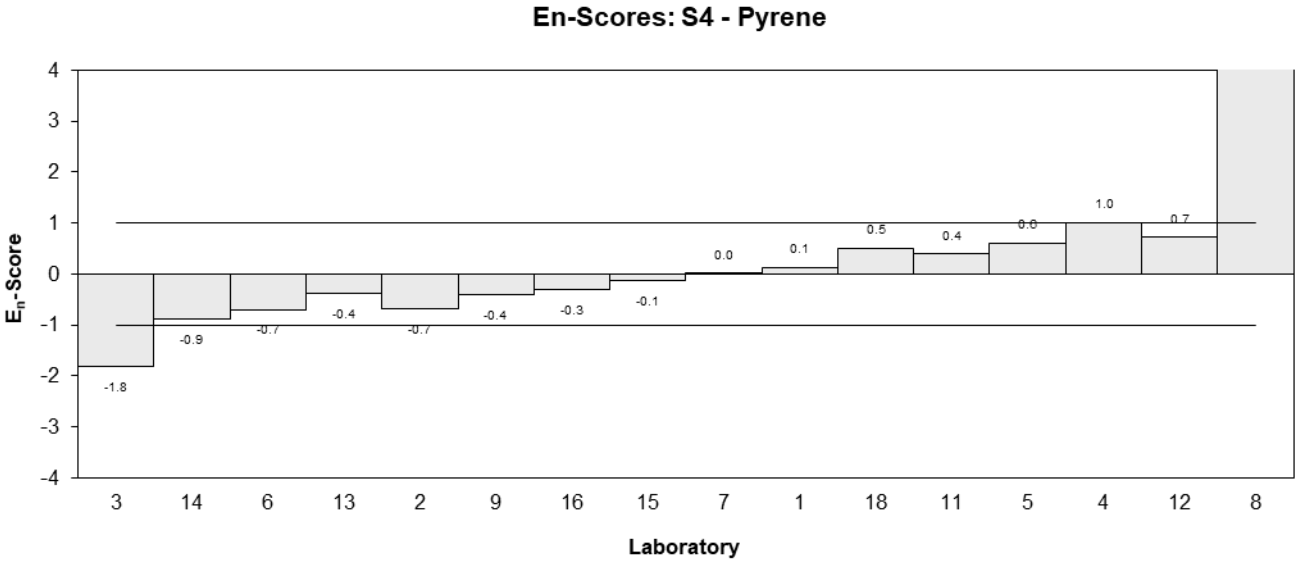

En-Scores: S4 - Pyrene
En-Score
-1.8 -0.9 -0.7 -0.4 -0.7 -0.4 -0.3 -0.1 0.0 0.1 0.5 0.4 0.6 1.0 0.7
3 14 6 13 2 9 16 15 7 1 18 11 5 4 12 8
Laboratory

Figure 22

6 DISCUSSION OF RESULTS

6.1 Assigned Value

The robust average of participants' results was used as the assigned value for all scored analytes. The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528:2015.[7] Results less than 50% and greater than 150% of the robust average were removed before the calculation of the assigned value.[3,4] The calculation of the expanded uncertainty for robust averages is presented in Appendix 3, using Sample S2 benzene as an example.

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

No assigned values were set for Sample S1 >C34-C40 as there were too few numerical results, and for Samples S3 and S4 benzo(a)pyrene as reported numeric results were highly variable. Sample S2 C6-C10 range was also not scored; historically this has been due to its volatile nature and therefore data has been provided for information only, though participants' CV for this has been improving over the past few Hydrocarbons in Water PT studies.

A comparison of the assigned values (or robust average if no assigned value was set) and the spiked values is presented in Table 28. Similar ratios of assigned value to spiked value have been observed in previous NMI Hydrocarbons in Water PT studies, and in this study assigned values were set if there was a reasonable consensus of participants' results.

Table 28 Comparison of Assigned Value (or Robust Average) and Spiked Value

Sample	Analyte	Assigned Value *(Robust Average)* (µg/L)	Spiked Value (µg/L)	Assigned Value *(Robust Average)* / Spiked Value (%)
S1	TRH	1280	1970	65
S2	Benzene	60.5	67.4	90
	Toluene	265	285	93
	Ethylbenzene	18.2	17.9	102
	Xylenes	110	134	82
	Total BTEX	452	504	90
S3	Anthracene	2.28	3.52	65
	Benzo(a)pyrene	*(5.8)*	8.48	*(68)*
	Fluoranthene	9.0	12.0	75
	Fluorene	3.93	6.07	65
	Phenanthrene	2.21	3.03	73
	Pyrene	6.08	8.02	76
S4	Anthracene	6.11	8.99	68
	Benzo(a)pyrene	*(0.62)*	0.921	*(67)*
	Fluoranthene	2.20	3.00	73
	Fluorene	7.4	11.0	67
	Phenanthrene	3.63	5.02	72
	Pyrene	5.09	7.02	73

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report the expanded uncertainty estimates associated with their results and the basis of this uncertainty. It is a requirement of ISO/IEC 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.[9]

Of 327 numeric results submitted for analytes of interest in this study, 296 (91%) were reported with an associated uncertainty. Participants used a wide variety of procedures to estimate their expanded MU (Table 3). Some participants reported using the NATA General Accreditation Guidance Estimating and Reporting MU as their guide; NATA no longer publishes this document.[11]

Laboratory **4** did not report any uncertainties, and Laboratory **18** did not report uncertainties for PAHs. These participants both reported being accredited to ISO/IEC 17025. Laboratory **10** TRH result had no uncertainty as the result was calculated by the study coordinator by summing the individual hydrocarbon ranges reported.

The magnitude of reported uncertainties was within the range of 3.2% to 100% relative. In general, an expanded measurement uncertainty of less than 10% relative is likely to be unrealistically small for the routine measurement of a hydrocarbon pollutant in water, while an expanded uncertainty of over 50% is likely to be too large. Of 296 MUs, 32 were below 10% relative while one was greater than 50% relative. Participants reporting these uncertainties should consider if their MUs are suitable or fit-for-purpose.

Laboratory **10** reported an uncertainty of '20' for all numeric results, even when the different analytes had significantly different concentrations. This participant may have reported their uncertainties as relative instead of absolute values, however as values were not reported with a '%' or equivalent, no modifications were made by the study coordinator.

Uncertainties associated with results returning a satisfactory z-score but an unsatisfactory E_n-score may have been underestimated.

An estimate of uncertainty expressed as a value should not be attached to a non-value result.[10] Laboratories **2**, **3**, **10**, **12**, **14** and **16** attached an uncertainty to some of their non-value results.

In some cases, results were reported with an inappropriate number of significant figures. Including too many significant figures may inaccurately reflect the measurement precision. 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 7.781 ± 2.486 µg/L, it is better to report this as 7.8 ± 2.5 µg/L.[10]

6.3 z-Score

Target SDs equivalent to 15% and 20% PCV were used to calculate z-scores. CVs predicted by the Thompson-Horwitz equation,[8] target SDs (as PCV), and the between-laboratory CVs obtained in this study for scored analytes are presented for comparison in Table 29.

Table 29 Comparison of Thompson-Horwitz CVs, Target SDs, and Between-Laboratory CVs

Sample	Analyte	Assigned Value (µg/L)	Thompson-Horwitz CV (%)	Target SD (as PCV) (%)	Between-Laboratory CV* (%)
S1	>C10-C16	770	17	20	29
	>C16-C34	700	17	20	26
	TRH	1280	15	15	34

Sample	Analyte	Assigned Value (µg/L)	Thompson-Horwitz CV (%)	Target SD (as PCV) (%)	Between-Laboratory CV* (%)
S2	Benzene	60.5	22	15	14
	Toluene	265	20	15	13
	Ethylbenzene	18.2	22	15	17
	Xylenes	110	22	15	19
	Total BTEX	452	18	15	16
S3	Anthracene	2.28	22	15	14
	Fluoranthene	9.0	22	15	23
	Fluorene	3.93	22	15	16
	Phenanthrene	2.21	22	15	15
	Pyrene	6.08	22	15	24
S4	Anthracene	6.11	22	15	23
	Fluoranthene	2.20	22	15	25
	Fluorene	7.4	22	15	22
	Phenanthrene	3.63	22	15	20
	Pyrene	5.09	22	15	24

* Robust between-laboratory CV with outliers removed, if applicable.

To account for possible low bias in the consensus values due to laboratories using inefficient extraction or analytical techniques, a total of 15 z-scores were adjusted across the following analytes: Sample S1 TRH, Sample S3 anthracene, fluoranthene and pyrene, and Sample S4 anthracene, fluoranthene, fluorene and pyrene. When the assigned value was less than 80% of the spiked value, a maximum acceptable concentration was set to two target SDs more than the spiked value, and results lower than the maximum acceptable concentration but with a z-score greater than 2.0 had their z-score adjusted to 2.0. This ensured that laboratories reporting results close to the spiked value were not penalised. z-Scores for results higher than the maximum acceptable concentration were not adjusted, and z-scores less than 2.0 were left unaltered.

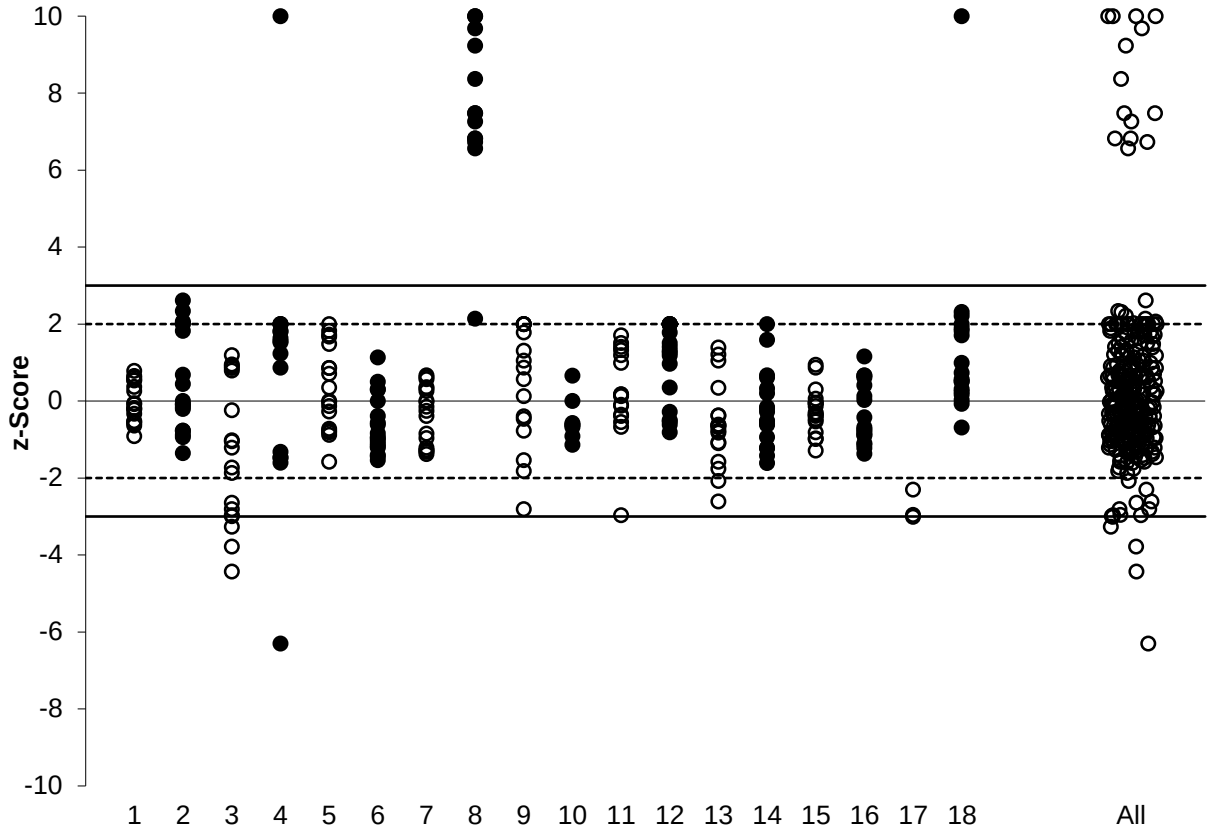
Of 288 results for which z-scores were calculated, 252 (88%) returned a satisfactory score of $|z| \leq 2.0$, indicating a satisfactory performance.

Laboratories **1**, **2**, **3**, **5**, **6**, **7**, **12**, **13**, **14**, **15**, **16** and **18** reported results for all 18 scored analytes. Of these participants, Laboratories **1**, **5**, **6**, **7**, **12**, **14**, **15** and **16** returned satisfactory z-scores for all analytes.

Satisfactory z-scores were achieved for all scored analytes reported by Laboratories **10** (8).

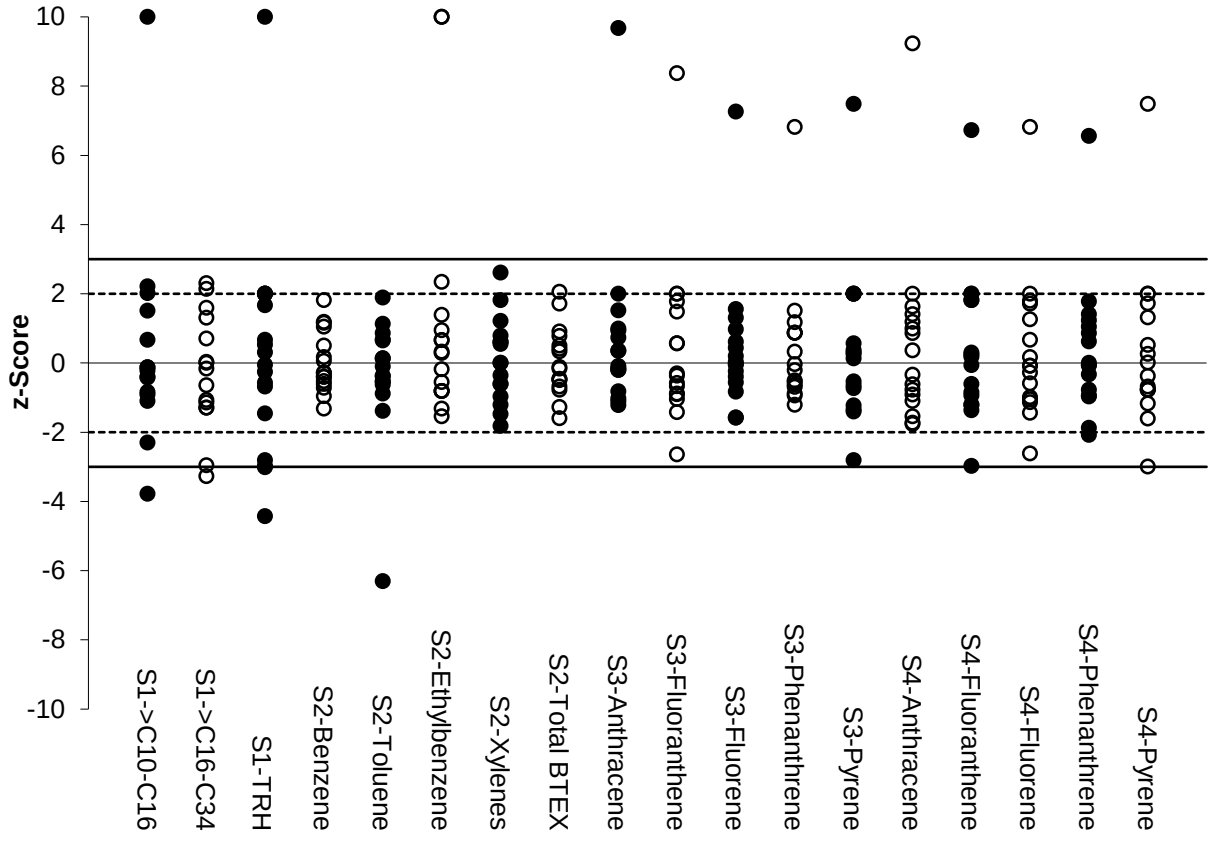
No results reported by Laboratories **8** (13) and **17** (3) returned a satisfactory z-score. All results reported by Laboratory **8** returned z-scores greater than the assigned value (positive bias), and all results reported by Laboratory **17** returned z-scores lower than the assigned value (negative bias). These participants may need to investigate the source of these biases.

The dispersal of participants' z-scores is presented by laboratory in Figure 23 and by analyte in Figure 24.



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

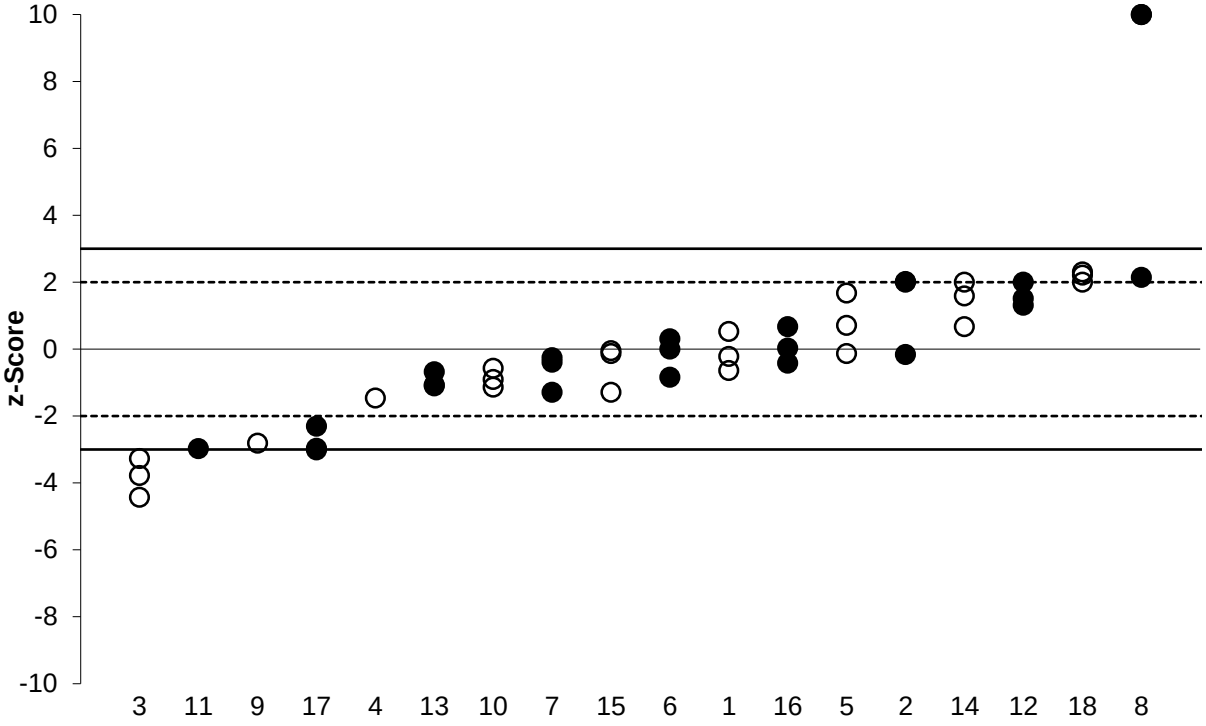
Figure 23 z-Score Dispersal by Laboratory



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

Figure 24 z-Score Dispersal by Sample and Analyte

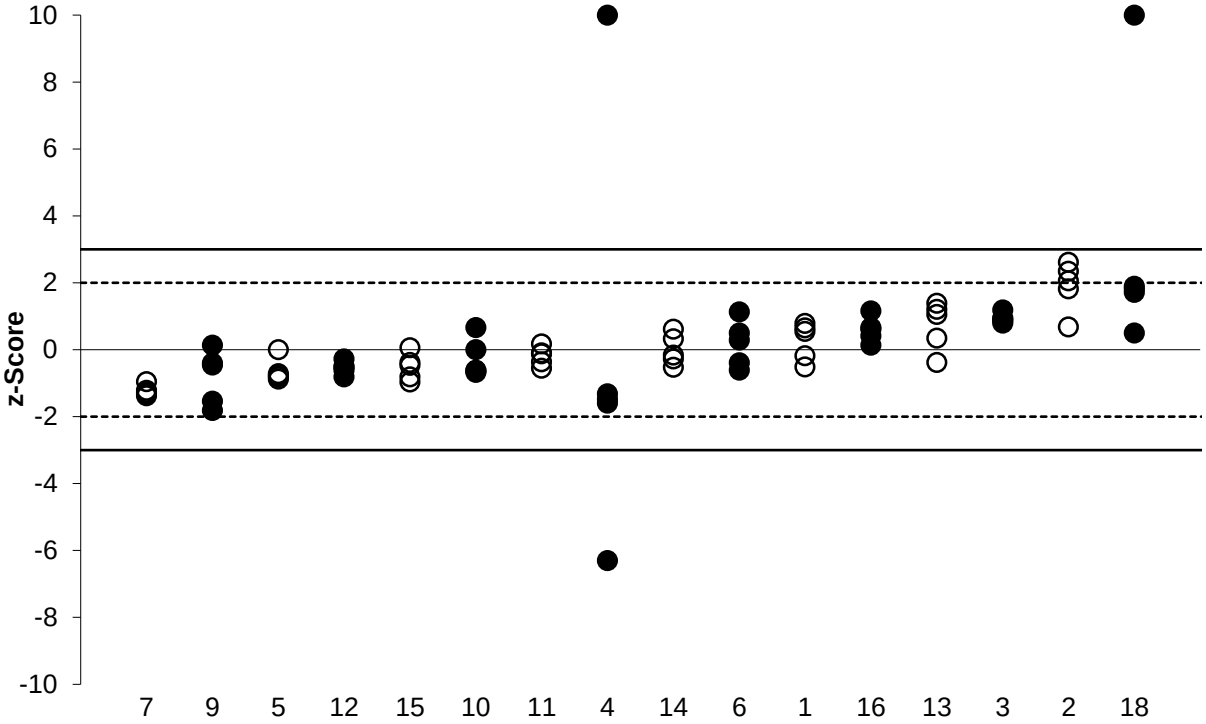
Figure 25 presents participants' z-scores for Sample S1 only. Participants with a trend of z-scores below the zero line likely had an inefficient extraction process for TRH. As the ratio of the assigned value to the spiked value was 65% for TRH, participants reporting results with higher satisfactory z-scores may have more efficient extraction methodologies.



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

Figure 25 Sample S1 (TRH) z-Score Dispersal by Laboratory

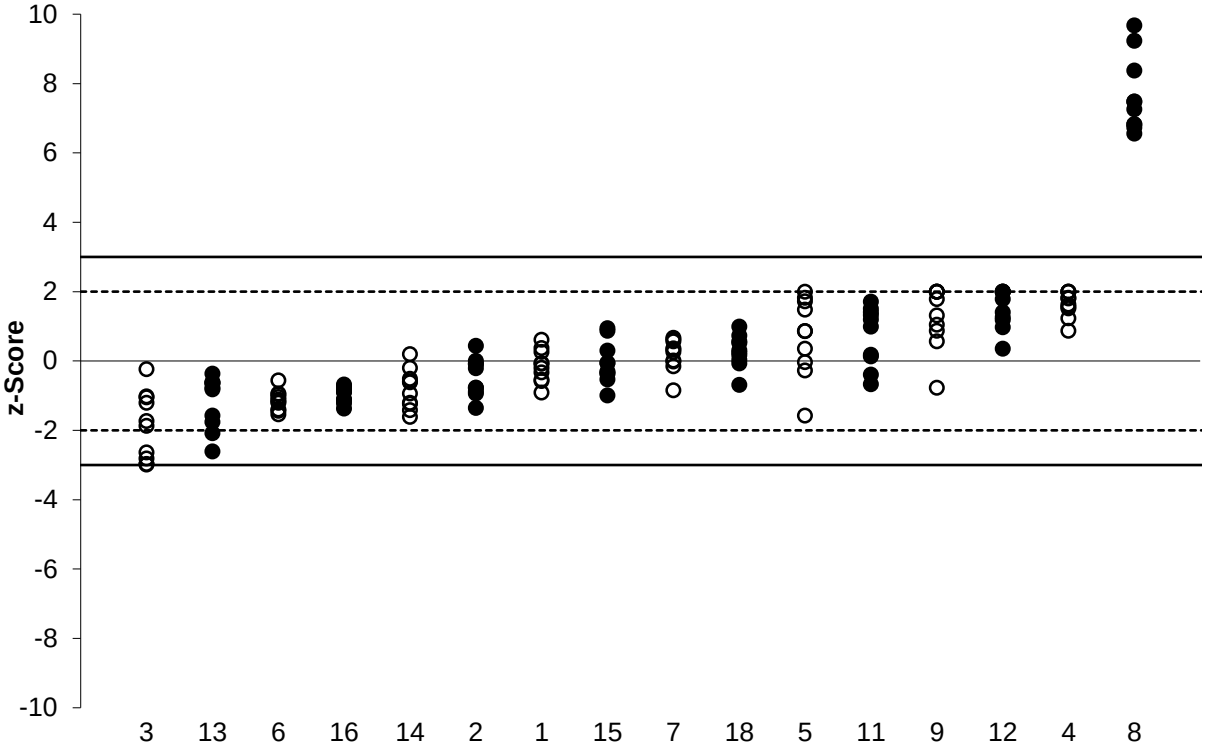
Figure 26 presents participants' z-scores for Sample S2 only. A trend of z-scores on one side of the zero line may indicate laboratory bias for BTEX analytes.



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

Figure 26 Sample S2 (BTEX) z-Score Dispersal by Laboratory

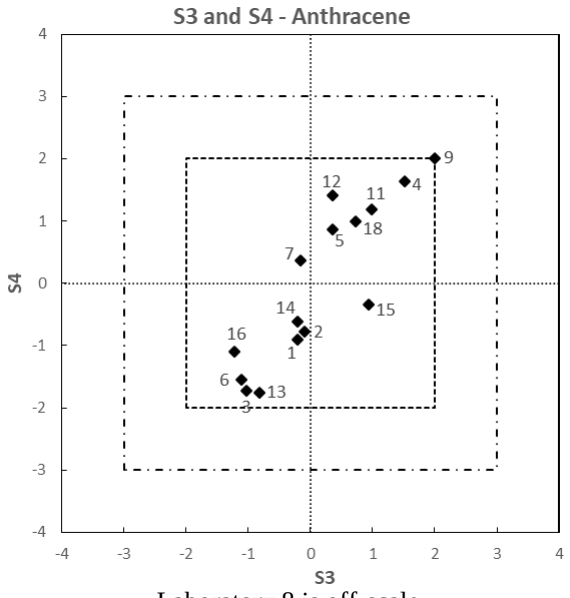
Figure 27 presents participants' z-scores for Samples S3 and S4 only. Participants with a trend of z-scores below the zero line may have an inefficient extraction process for PAHs. As the ratio of the assigned values to the spiked values ranged from 65% to 76%, results with higher satisfactory z-scores may correspond to the more efficient extraction of PAHs.



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

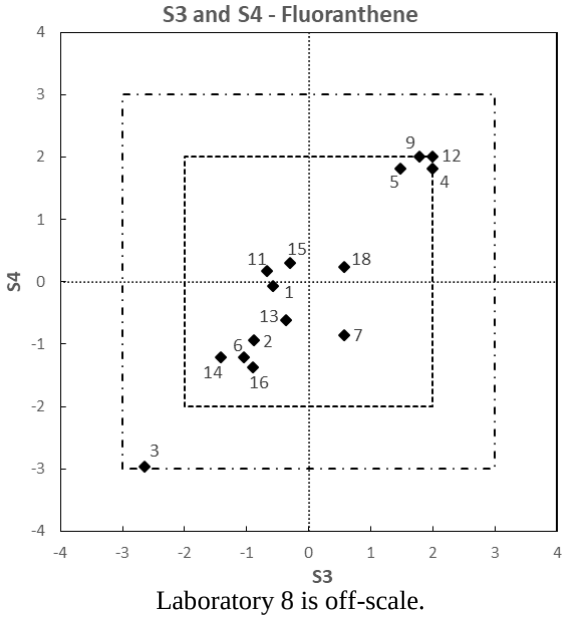
Figure 27 Samples S3 and S4 (PAHs) z-Score Dispersal by Laboratory

Scatter plots of z-scores for anthracene, fluoranthene, fluorene, phenanthrene and pyrene in Samples S3 and S4 are presented in Figures 28 to 32. Scores are predominantly in the upper right and lower left quadrants, indicating that laboratory bias is the major contributor to the variability of results. Points close to the diagonal axis demonstrate excellent repeatability while points close to the zero demonstrate excellent repeatability and accuracy.



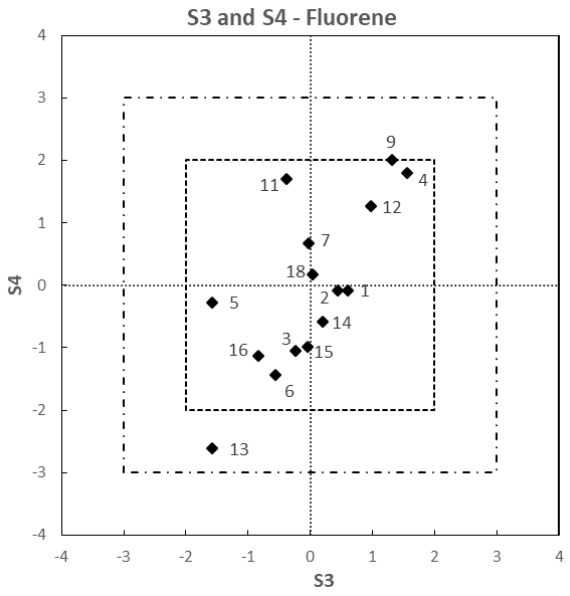
Laboratory 8 is off-scale.

Figure 28 z-Score Scatter Plot – Anthracene



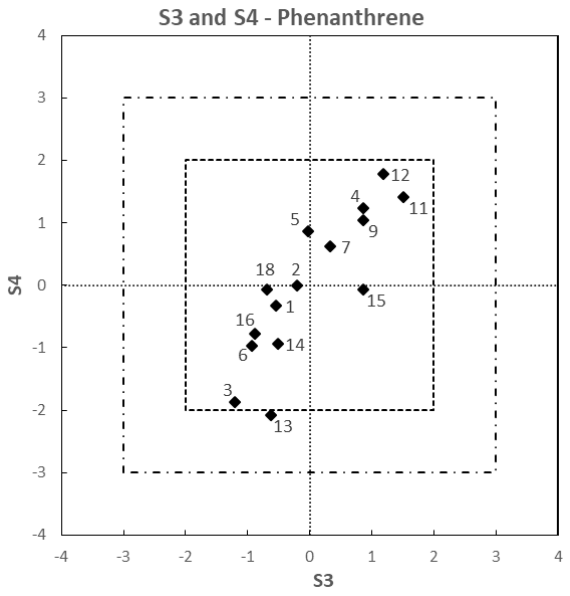
Laboratory 8 is off-scale.

Figure 29 z-Score Scatter Plot – Fluoranthene



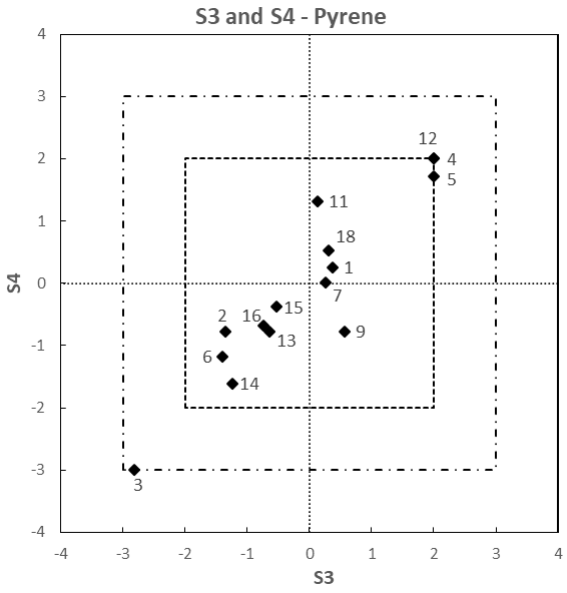
Laboratory 8 is off-scale.

Figure 30 z-Score Scatter Plot – Fluorene



Laboratory 8 is off-scale.

Figure 31 z-Score Scatter Plot – Phenanthrene



Laboratory 8 is off-scale.

Figure 32 z-Score Scatter Plot – Pyrene

6.4 E_n-Score

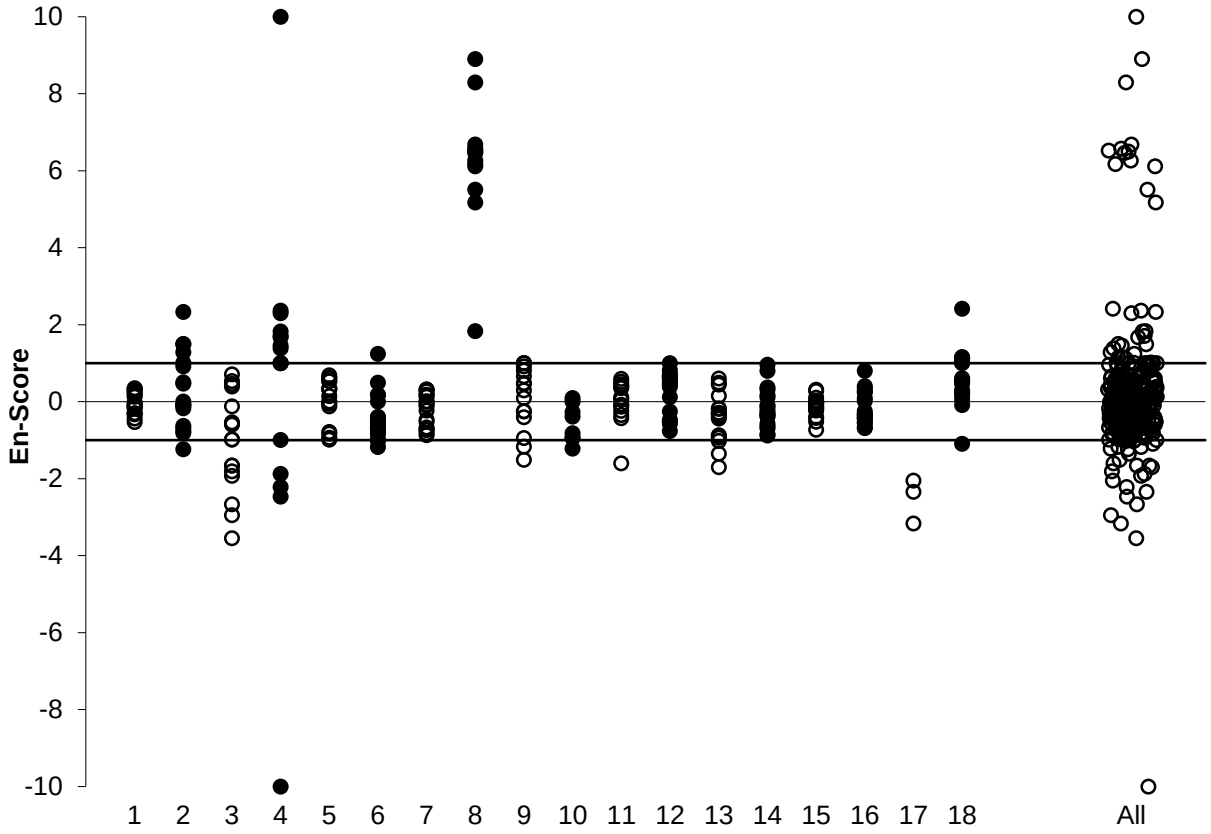
E_n-scores should be interpreted in conjunction with z-scores; an unsatisfactory E_n-score can either be caused by an inappropriate measurement or uncertainty, or both. If a participant did not report an expanded measurement uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the E_n-score. For results whose z-scores were adjusted as discussed in Section 6.3 z-Scores, any E_n-scores greater than 1.0 were set to 1.0.

Of 288 results for which E_n-scores were calculated, 231 (80%) returned a satisfactory score of $|E_n| \leq 1.0$, indicating agreement of the participant's result with the assigned value within their respective uncertainties.

Laboratories **1**, **5**, **7**, **12**, **14**, **15** and **16** returned satisfactory E_n-scores for all 18 scored analytes.

All results reported by Laboratories **8** (13) and **17** (3) returned unsatisfactory E_n-scores.

A summary of E_n-score dispersal by laboratory is presented in Figure 33.



E_n-scores greater than 10 or less than -10 have been plotted at 10 and -10 respectively.

Figure 33 E_n-Score Dispersal by Laboratory

6.5 False Negatives

Table 30 presents false negative results. These are analytes present in the samples which a participant tested for, but did not report a numeric result (for example, participants reporting a 'less than' result (< *x*) when the assigned value was higher than their limit of reporting (LOR), or participants that did not report anything). For analytes where no assigned value was set, results have only been considered to be false negatives where the robust average and spiked value were significantly higher than the participants' LOR, or if no value was reported.

Table 30 False Negatives

Lab. Code	Sample	Analyte	Assigned Value *(Robust Average)* (µg/L)	Spiked Value (µg/L)	Result* (µg/L)
3	S3	Benzo(a)pyrene	*(5.8)*	8.48	<1
9	S4	Benzo(a)pyrene	*(0.62)*	0.921	<0.1
10	S3	Anthracene	2.28	3.52	NR
		Benzo(a)pyrene	*(5.8)*	8.48	NR
		Fluoranthene	9.0	12.0	NR
		Fluorene	3.93	6.07	NR
		Phenanthrene	2.21	3.03	NR
		Pyrene	6.08	8.02	NR

Lab. Code	Sample	Analyte	Assigned Value *(Robust Average)* (µg/L)	Spiked Value (µg/L)	Result* (µg/L)
	S4	Anthracene	6.11	8.99	NR
		Benzo(a)pyrene	*(0.62)*	0.921	NR
		Fluoranthene	2.20	3.00	NR
		Fluorene	7.4	11.0	NR
		Phenanthrene	3.63	5.02	NR
		Pyrene	5.09	7.02	NR

* Results reported as NR may or may not be false negatives, depending on the participants' actual LOR.

6.6 Participants' Analytical Methods

TRH (Sample S1)

Five participants reported taking 500 mL (i.e. the whole sample) for analysis, while the other participants reported sample test portions ranging from 35 – 250 mL. There was no evident correlation between the results obtained and the reported sample volume used (Figure 34).

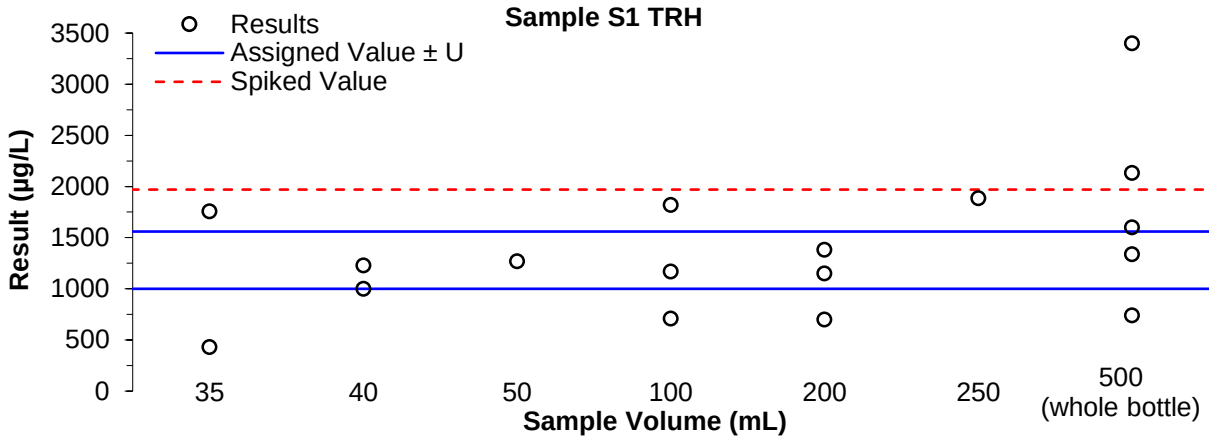


Figure 34 Sample S1 TRH Results vs Sample Volume

All participants reporting methodologies used liquid-liquid extraction (LLE), with dichloromethane (DCM), hexane (HEX), or a mixture of pentane (PENT) and hexane as the solvent. Three participants reported an additional silica clean-up step for the samples. Most participants used GC-FID for analysis, except for one participant who used GC-MS instead. The most common methodology employed was LLE with DCM, and using GC-FID for analysis. A summary of results vs methodology is presented in Figure 35.

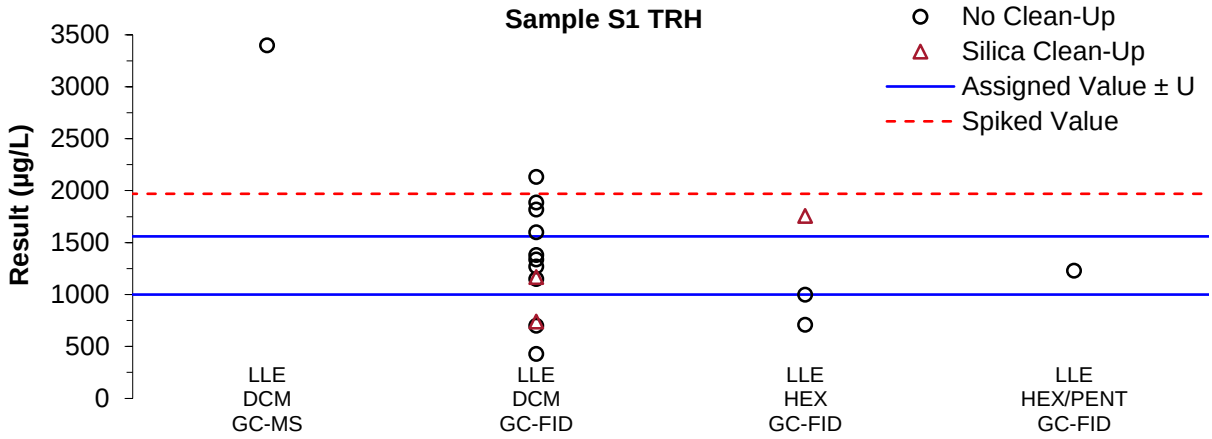


Figure 35 Sample S1 TRH Results vs Methodology

BTEX (Sample S2)

Eight participants reported taking the whole sample (40 mL) for analysis, while the other participants reported sample test portions of either 5 mL or 10 mL (Figure 36).

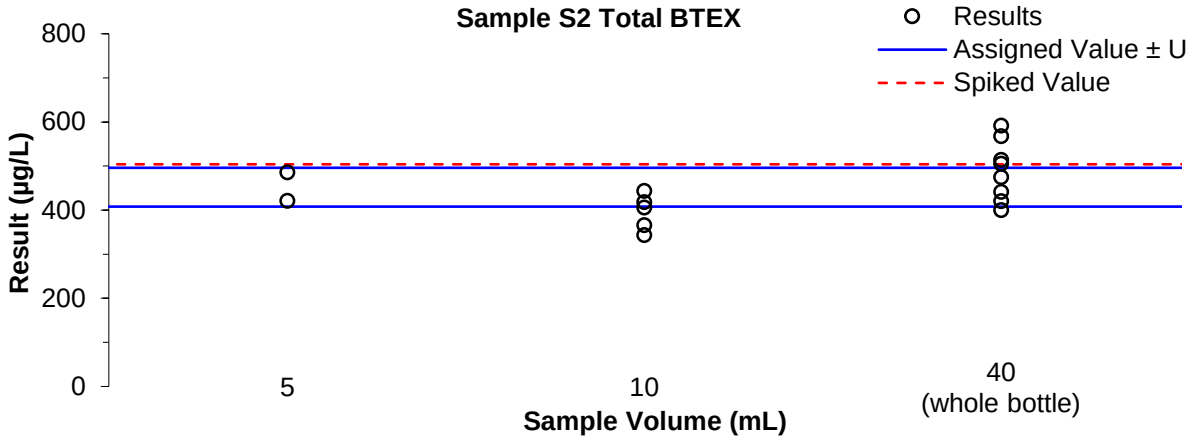


Figure 36 Sample S2 Total BTEX Results vs Sample Volume

For BTEX analysis, participants used either purge-and-trap (P&T) GC-MS(/MS) or headspace (HS) GC-MS(/MS). Two participants reported LLE as part of their preparation. Two participants reported using methanol as an extraction solvent. The most common methodology was P&T GC-MS. A summary of results vs methodology is presented in Figure 37.

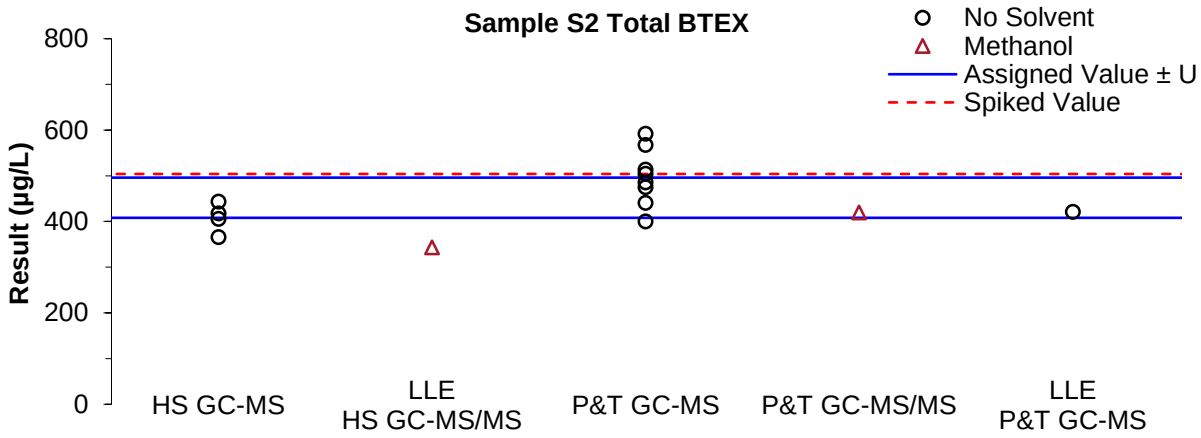


Figure 37 Sample S2 Total BTEX Results vs Methodology

PAHs (Samples S3 and S4)

All results reported by Laboratory **8** for PAHs in Samples S3 and S4 were approximately two times greater than the assigned values. This may have been due to a dilution factor or calculation error, and this participants' results have been excluded from the following discussion.

Five participants reported taking 500 mL (i.e. the whole sample) for analysis, while other participants reported test portions ranging from 35 – 250 mL. z-Scores obtained as compared to the sample volume used is presented in Figure 38. In this study, those participants using 35 mL for analysis were generally biased low. Participants taking a smaller volume for sample analysis should ensure that they are taking a suitable representative amount of sample. Heavier PAHs such as benzo(a)pyrene may be adsorbing to the container, causing higher variability for this analyte in both Samples S3 and S4. Participants may need to shake or sonicate the sample prior to sampling.

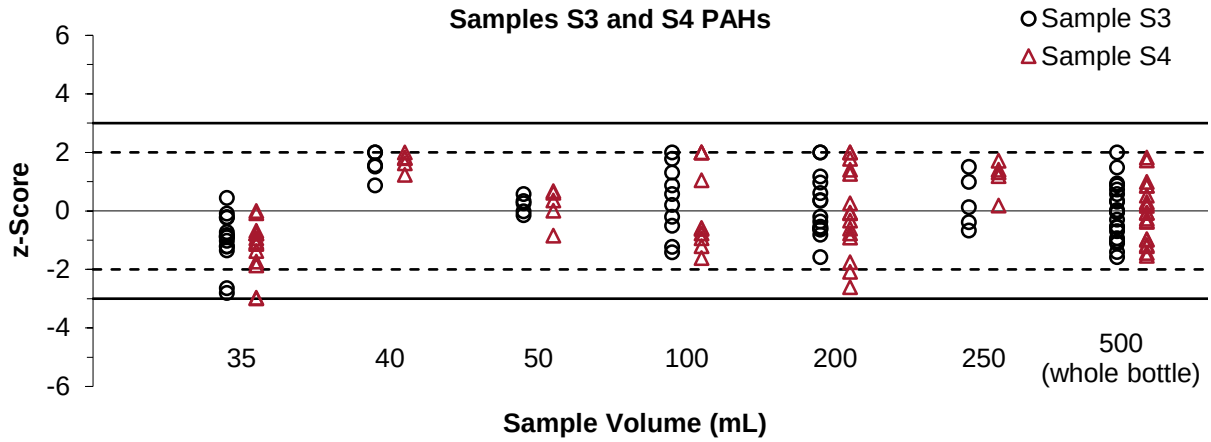


Figure 38 Samples S3 and S4 PAHs z-Scores vs Sample Volume

The majority of participants used liquid-liquid extraction, except for one participant who used solid-phase extraction (SPE). DCM, HEX, and a mixture of DCM and ethyl acetate (EtOAc) were reported as extraction solvents. All participants used GC-MS(/MS) for analysis. The most common methodology employed for PAHs was LLE with DCM, using GC-MS for analysis. A summary of results vs methodology is presented in Figure 39. As the ratio of assigned values to spiked values ranged from 65% to 76%, results with higher satisfactory z-scores (for example in this study, participants who used LLE with HEX, or SPE with DCM/EtOAc) may correspond to the more efficient extraction of PAHs.

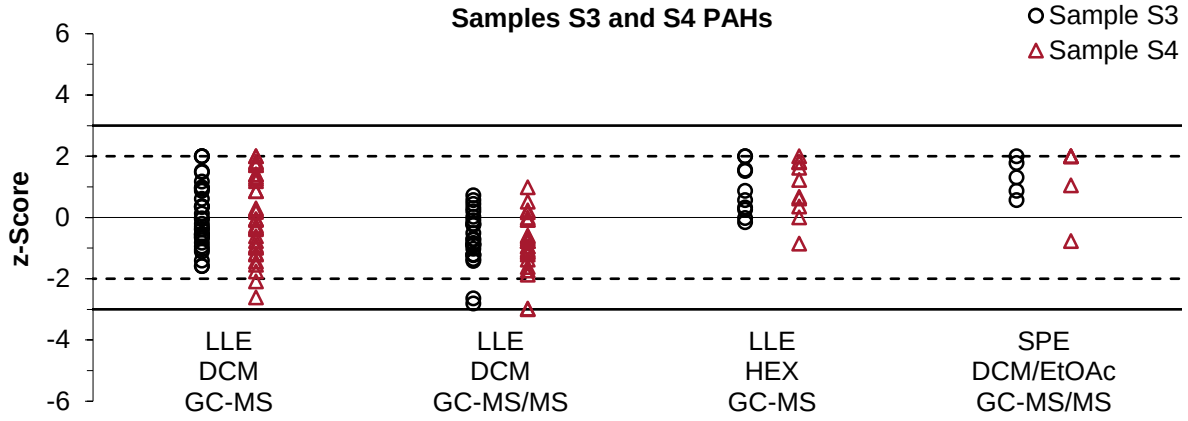


Figure 39 Samples S3 and S4 PAHs z-Scores vs Methodology

6.7 Certified Reference Materials

Participants were requested to report whether certified standards or matrix reference materials had been used as part of the quality assurance for the analysis.

Twelve participants reported using certified standards, one participant reported using matrix reference materials, and one participant reported using both certified standards and matrix reference materials. The following were reported:

- NMI MX015
- AccuStandard
- Agilent
- ChemService
- Sigma-Aldrich
- Restek

These materials may or may not meet the internationally recognised definition of a CRM:

> '***reference material***, *accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceabilities, using valid procedures*'[12]

6.8 Summary of Participants' Performance

Summaries of participants' results and performances for scored analytes in this PT study are presented in Tables 31 and 32, and Figure 40.

Table 31 Summary of Participants' Samples S1 and S2 Results for Scored Analytes (all values are in µg/L)*

Lab. Code	S1 >C10-C16	S1 >C16-C34	S1 TRH	S2 Benzene	S2 Toluene	S2 Ethylbenzene	S2 Xylenes	S2 Total BTEX
Assigned Value	770	700	1280	60.5	265	18.2	110	452
1	736	611	1381	55.8	291	17.7	119	505
2	1081	677	1758	77	292	24.6	153	592
3	188	242	430	71.3	299	20.8	123	514
4	NR	NR	1000	48.5	14.5	195	85.5	343.5
5	750	800	1600	54	230	16	110	400
6	640	700	1340	57	310	19	100	486
7	710	520	1230	51.8	210	14.6	90	366
8	2400	1000	3400	NT	NT	NT	NT	NT
9	NR	NR	740	56.9	270	14	80	421
10	630	540	1170	55	240	20	110	406
11	NR	NR	710	62	261	16.7	104.1	443.8
12	1003	884	1887	58	244	16	100	418
13	600	550	1150	70	250	22	130	475
14	873	922	1820	58	244	19.1	120	441.1
15	750	520	1270	61	250	16	94	420
16	705	704	1409	71	270	20	120	480
17	415.7	285.7	701.4	NT	NT	NT	NT	NT
18	1110	1023	2133	65	340	220	140	568

* Shaded cells are results which returned a questionable or unsatisfactory z-score.

Table 32 Summary of Participants' Samples S3 and S4 Results for Scored Analytes (all values are in µg/L)*

Lab. Code	S3 Anthracene	S3 Fluoranthene	S3 Fluorene	S3 Phenanthrene	S3 Pyrene	S4 Anthracene	S4 Fluoranthene	S4 Fluorene	S4 Phenanthrene	S4 Pyrene
Assigned Value	2.28	9.0	3.93	2.21	6.08	6.11	2.20	7.4	3.63	5.09
1	2.21	8.22	4.29	2.03	6.42	5.28	2.18	7.31	3.45	5.29
2	2.25	7.81	4.19	2.14	4.85	5.4	1.89	7.31	3.63	4.5
3	1.93	5.44	3.79	1.81	3.52	4.52	1.22	6.23	2.61	2.81
4	2.8	12	4.85	2.5	8.15	7.6	2.8	9.4	4.3	6.8
5	2.4	11	3.0	2.2	7.9	6.9	2.8	7.1	4.1	6.4
6	1.9	7.6	3.6	1.9	4.8	4.7	1.8	5.8	3.1	4.2
7	2.23	9.77	3.92	2.32	6.32	6.44	1.92	8.14	3.97	5.1
8	5.59	20.30	8.21	4.47	12.90	14.57	4.42	14.97	7.20	10.80
9	3.8	11.4	4.7	2.5	6.6	8.9	3	10.3	4.2	4.5
10	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
11	2.62	8.1	3.7	2.71	6.2	7.2	2.26	9.3	4.4	6.1
12	2.4	11.8	4.5	2.6	8.2	7.4	2.9	8.8	4.6	7.3
13	2	8.5	3	2	5.5	4.5	2	4.5	2.5	4.5
14	2.21	7.08	4.05	2.04	4.96	5.54	1.8	6.74	3.12	3.86
15	2.6	8.6	3.9	2.5	5.6	5.8	2.3	6.3	3.6	4.8
16	1.863	7.781	3.438	1.919	5.417	5.106	1.747	6.150	3.212	4.573
17	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
18	2.53	9.77	3.95	1.98	6.36	7.02	2.28	7.59	3.59	5.49

* Shaded cells are results which returned a questionable or unsatisfactory z-score.

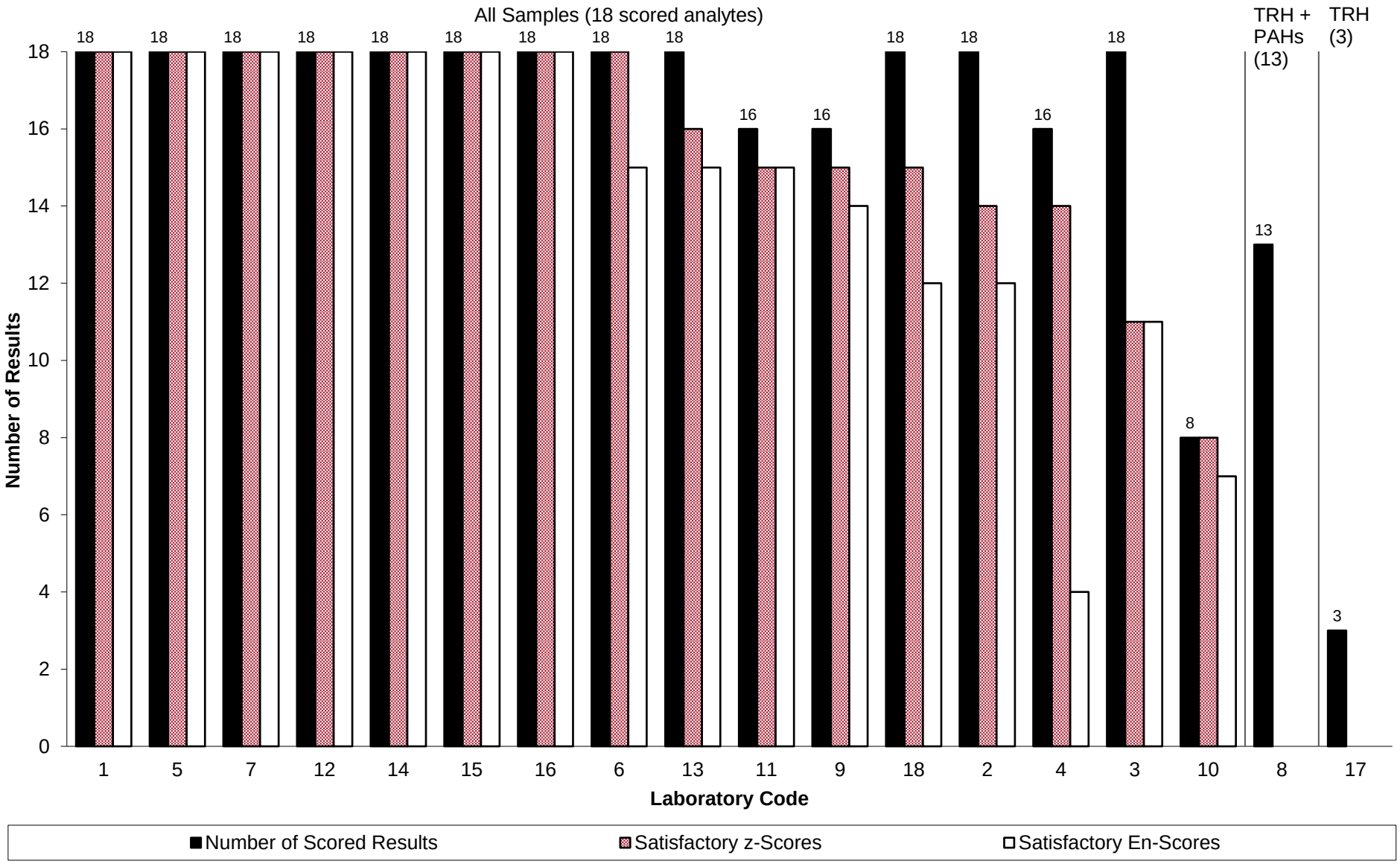


Figure 40 Summary of Participants' Performance

6.9 Comparison with Previous Studies

To enable direct comparison with previous studies, the target SD (as PCV) used to calculate z-scores has been kept constant between PT studies.

TRH

A summary of z-scores and E_n-scores (presented as a percentage of the total number of scores for each study) obtained by participants for TRH in water over the last 10 studies where this was scored (2012–2021) is presented in Figure 41. Over this period, the average proportion of satisfactory scores was 74% for z-scores and 65% for E_n-scores.

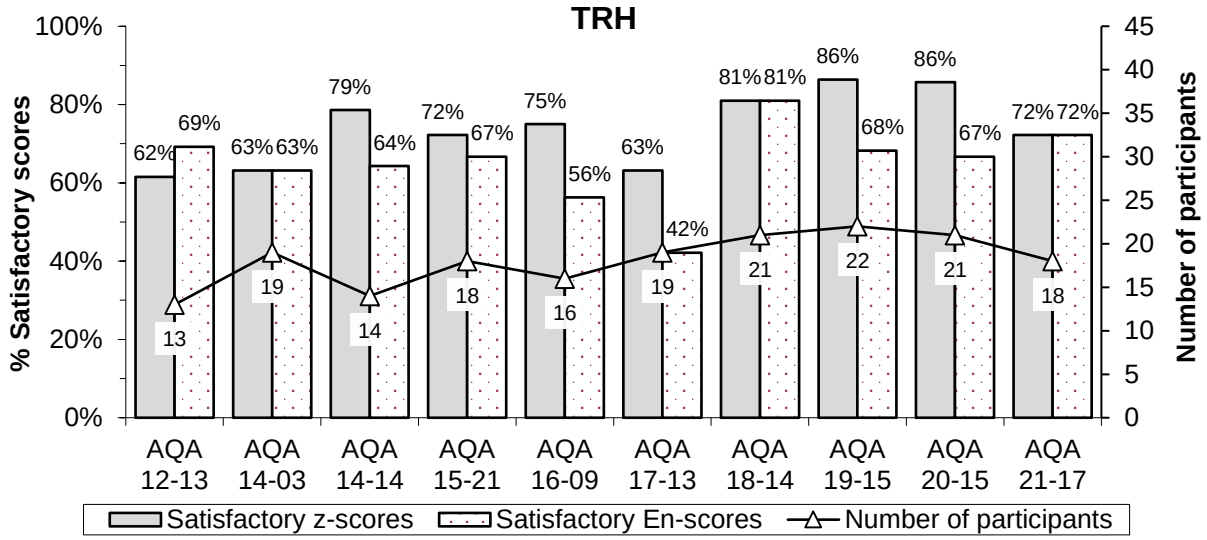


Figure 41 Summary of Satisfactory Scores for TRH (TPH for AQA 12-13) in Water PT Studies

Total BTEX

A summary of z-scores and E_n-scores (presented as a percentage of the total number of scores for each study) obtained by participants for Total BTEX in water over the last 10 studies where this was scored (2014–2021) is presented in Figure 42. Over this period, the proportion of satisfactory scores has remained high, with an average proportion of 92% and 87% for z-scores and E_n-scores respectively.

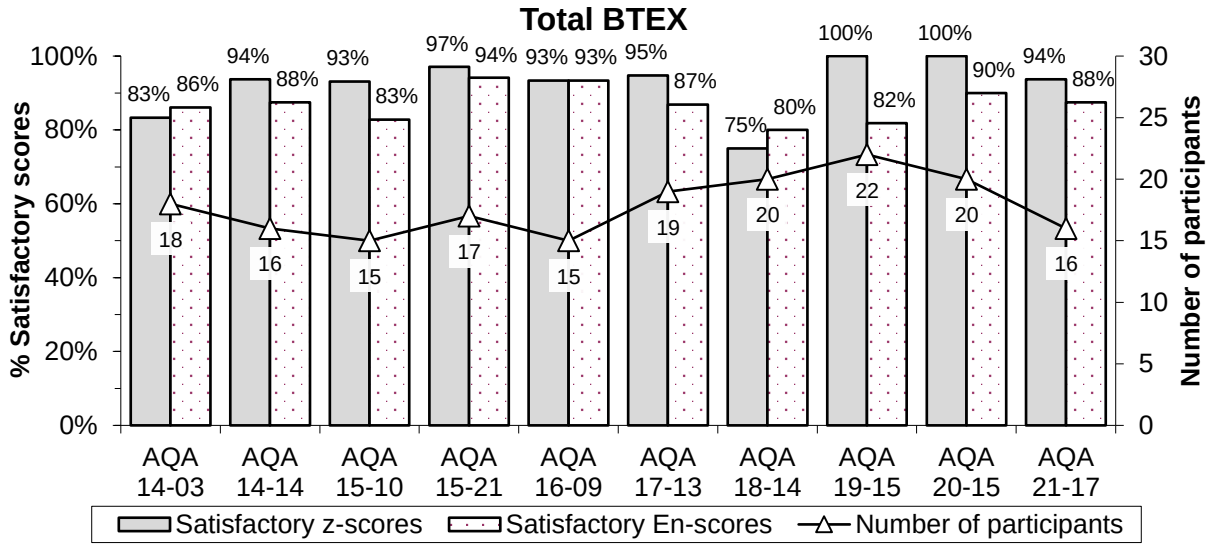


Figure 42 Summary of Satisfactory Scores for Total BTEX in Water PT Studies

PAHs

NMI has included PAHs in water as PT samples since 2015. A summary of z-scores and E_n-scores (presented as a percentage of the total number of scores for each study) obtained by participants for PAHs in water over the last 7 studies (2015–2021) is presented in Figure 43. Over this period, the proportion of satisfactory scores has fairly consistent, with an average proportion of 86% and 80% for z-scores and E_n-scores respectively.

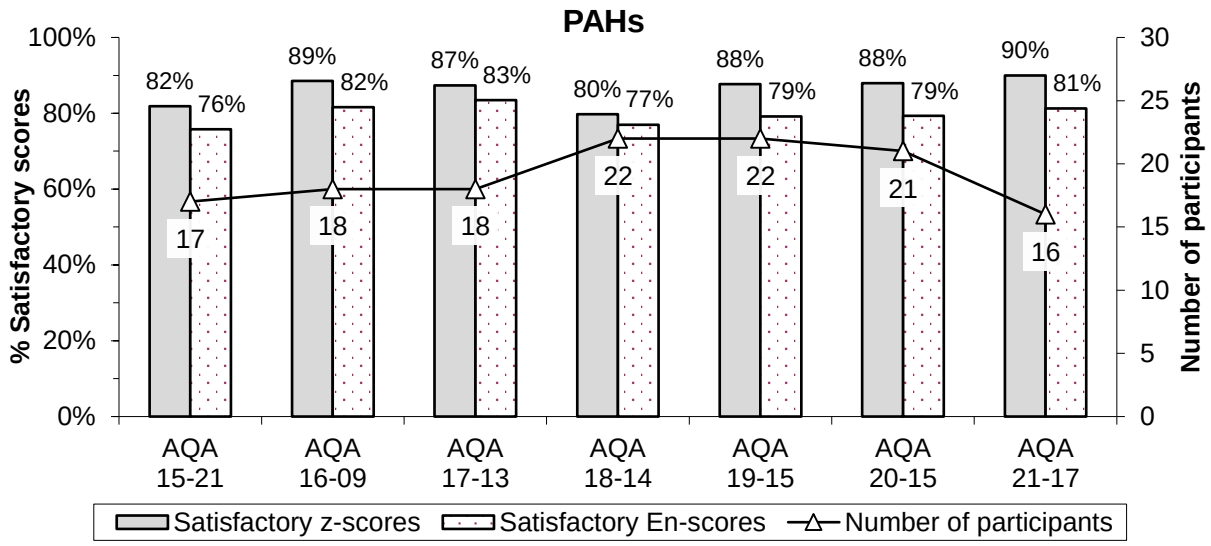


Figure 43 Summary of Satisfactory Scores for PAHs in Water PT Studies

A plot of the assigned value, expressed as a percentage of the spiked value, for PAHs in water since 2015 is presented in Figure 44. In this study, the recoveries for anthracene, fluoranthene and pyrene were lower than the average of previous studies, while for fluorene the recoveries were higher than the average. For phenanthrene, the recoveries were similar to the average.

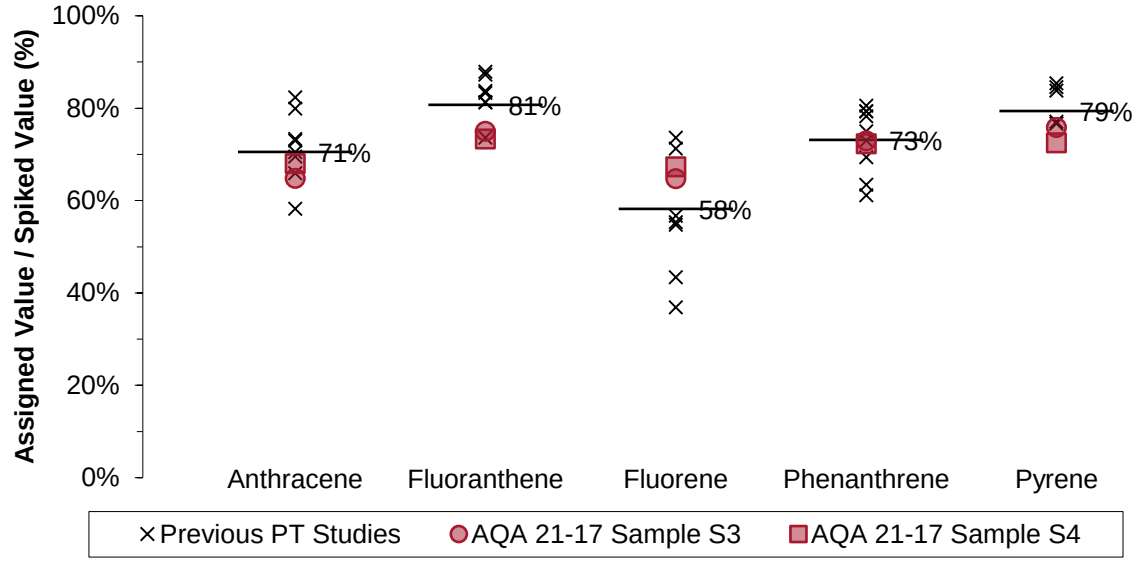


Figure 44 Ratio of Assigned Value to Spiked Value for PAHs in Water PT Studies (the line indicates the average for each PAH)

7 REFERENCES

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APPENDIX 1 – SAMPLE PREPARATION

A1.1 Diesel Fuel and River Water Preparation

Diesel fuel was purchased from a local retail outlet and treated to remove volatiles. Approximately 500 mL of diesel fuel was placed in a heated (80 °C) open container and sparged with nitrogen. Treatment continued until the GC-FID chromatogram indicated that essentially all the hydrocarbons eluting before C_{10} had been removed. This same treated diesel fuel has been used in previous NMI Hydrocarbon PT studies.

Water was sampled from Browns Waterhole in Sydney. The water was filtered under vacuum through an Advantec 150 mm glass fibre filter. After filtration the water was placed in 10 L Schott bottles and autoclaved.

A1.2 Test Sample Preparation

Sample S1

A diesel spiking solution was prepared by weighing a portion of the treated diesel fuel into a 500 mL volumetric flask and making to volume with methanol. Amber glass bottles of approximately 500 mL capacity were rinsed with acetone and dried. The cleaned bottles were placed in an air-conditioned room overnight. Filtered autoclaved water (498.5 ± 0.2 g, or 500 mL at 25 °C) was weighed into the bottles. Methanol/diesel spiking solution (0.98 mL) was added to each bottle using a Hamilton dispenser. The bottles were immediately capped and inverted to mix the solution. Each bottle was then labelled and shrink-wrapped.

Sample S2

Filtered autoclaved water (41.88 ± 0.05 g, or 42 mL at 25 °C) was weighed into Agilent vials. A composite spike solution was prepared by adding aliquots of diesel and unleaded petrol to methanol. Two of the BTEX compounds was fortified with additional laboratory solvent. The composite spiking solution was made up to volume with methanol. Composite spiking solution (1.0 mL) was added to each vial. Each vial was capped after spiking, and then labelled and shrink-wrapped.

Samples S3 and S4

The spiking solutions were prepared by dissolving each standard material in dichloromethane. Diluted spiking solutions were prepared using acetone. The autoclaved water was placed in a stainless steel container. After spiking the water was stirred using a top-driven impeller stirrer for at least 2 hours. The samples were then dispensed into 500 mL amber glass bottles which were labelled and shrink-wrapped.

Between preparation and dispatch all samples were stored in a cool room at 4 °C.

APPENDIX 2 – PARTICIPANTS' TEST METHODS

Participants were requested to provide information about their test methods. Responses are presented in Tables 33 to 35. Some responses may be modified so that the participant cannot be identified.

Table 33 Methodology – Sample S1 TRH

Lab. Code	Sample Volume (mL)	Extraction Details	Extraction Solvent	Clean-Up	Measurement Instrument	Method Reference
1	200	Liquid-Liquid	DCM	None	GC-FID	USEPA 8015B
2	35	Liquid-Liquid	Hexane	Silica Gel	GC-FID	USEPA 3510
3	35	Liquid-Liquid	DCM	None	GC-FID	
4	40	Liquid-Liquid	Hexane	None	GC-FID	USEPA 8015
5	500	Liquid-Liquid	DCM	None	GC-FID	In-house
6	500	Liquid-Liquid	DCM	none	GC-FID	
7	40	Liquid-Liquid	Hexane:Pentane	None	GC-FID	in-house
8	500	Liquid-Liquid	DCM		GC-MS	USEPA 8260
9	500	Liquid-Liquid	DCM	Silica	GC-FID	
10	100	Liquid-Liquid	DCM	Silica	GC-FID	USEPA 8260
11	100	Solvent extraction	Hexane	None	GC-FID	US EPA 8015
12	250	Liquid-Liquid	DCM	None	GC-FID	In house based on USEPA 3550, 3510 & 8000B
13	200	Liquid-Liquid	DCM	None	GC-FID	USEPA 8015B
14	100	Liquid-Liquid	DCM	None	GC-FID	USEPA 8015
15	50	Liquid-Liquid	DCM	None	GC-FID	USEPA3510
16						
17	200	Liquid-Liquid	DCM	None	GC-FID	In house method
18	500	Liquid-Liquid	DCM	None	GC-FID	USEPA 8260

Table 34 Methodology – Sample S2 BTEX

Lab. Code	Sample Volume (mL)	Extraction Details	Extraction Solvent	Clean-Up	Measurement Instrument	Method Reference
1	40	Purge and Trap	None	None	P&T GC-MS	USEPA 8260
2	43	Purge & Trap	N/A	N/A	GC-MS	USEPA 8260
3	40	none	none	none	P&T GC-MS	
4	10	Liquid-Liquid	Methanol	None	Headspace GC-MS/MS	USEPA 5021A
5	40	Direct analysis	n/a	None	P&T GC-MS	In-house
6	5	P+T			GCMS	
7	10			none	Headspace GC-MS	in-house
8	NT					
9	5	Liquid-Liquid			P&T GC-MS	USEPA 8260
10	10		Headspace	Nil	GCMS	USEPA3550C
11	10	Headspace	-	-	Headspace GC-MS	US EPA 8260 & 5021
12	10	Headspace	None	None	Headspace GC-MS	In house based on USEPA 8260B & 8000B
13	40	Purge and Trap	None	None	P&T GC-MS	USEPA 8260
14	40	Purge & Trap	N/A	None	P&T GC-MS	USEPA 8260
15	44	Purge and Trap	Methanol	None	P&T GC-MS/MS	USEPA 8260
16						
17	NT					
18	42	N/A	N/A	None	P&T GC-MS	USEPA 8260

Table 35 Methodology – Samples S3 and S4 PAHs

Lab. Code	Sample Volume (mL)	Extraction Details	Extraction Solvent	Clean-Up	Measurement Instrument	Method Reference
1	200	Liquid-Liquid	DCM	None	GC-MS	USEPA 8015B
2	35	Liquid-Liquid	DCM	N/A	GC-MS/MS	USEPA 8270
3	35	Liquid-Liquid	DCM	none	GC-MS/MS	
4	40	Liquid-Liquid	Hexane	None	GC-MS	USEPA 3500C 8270D
5	500	Liquid-Liquid	DCM	None	GC-MS	In-house
6	500	Liquid-Liquid	DCM	none	GCMS	
7	50	Liquid-Liquid	Hexane	none	GC-MS	in-house
8	500	Liquid-Liquid	DCM		GC-MS	USEPA 8270
9	100	SPE	DCM-EtOAc	None	GC-MS/MS	USEPA 8270
10						
11	250	Liquid-Liquid	DCM	None	GC-MS	US EPA 8270
12	200	Liquid-Liquid	DCM	None	GC-MS	In house based on USEPA 3550, 3510 & 8270C
13	200	Liquid-Liquid	DCM	None	GC-MS	USEPA 8015B
14	100	Liquid-Liquid	DCM	None	GC-MS/MS	USEPA 8270
15	500	Liquid-Liquid	DCM	None	GC-MS	USEPA 8270
16	35	Liquid-Liquid	DCM	None	GC-MS/MS	USEPA8260
17	NT					
18	500	Liquid-Liquid	DCM	None	GC-MS/MS	USEPA 8270

APPENDIX 3 – ROBUST AVERAGE AND ASSOCIATED UNCERTAINTY, z-SCORE AND E_N-SCORE CALCULATIONS

A3.1 Robust Average and Associated Uncertainty

Robust averages were calculated using the procedure described in ISO 13258:2015 Annex C.[7] The associated uncertainties were estimated as according to Equation 4.

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

where:

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

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

p is the number of results

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

A worked example is set out below in Table 36.

Table 36 Uncertainty of the Robust Average for Sample S2 Benzene

Number of results (p)	16
Robust Average	60.5 µg/L
$S_{rob\ av}$	8.6 µg/L
$u_{rob\ av}$	2.7 µg/L
k	2
$U_{rob\ av}$	5.4 µg/L

Therefore, the robust average for benzene in Sample S2 is 60.5 ± 5.4 µg/L.

A3.2 z-Score and E_n-Score Calculation

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

A worked example is set out below in Table 37.

Table 37 z-Score and E_n-Score for Sample S1 >C10-C16 Result Reported by Laboratory 1

Participant Result (µg/L)	Assigned Value (µg/L)	Target Standard Deviation	z-Score	E_n-Score
736 ± 184	770 ± 150	20% as PCV, or: 0.2 × 770 = 154 µg/L	z-Score = $\frac{736-770}{154}$ = -0.22	E_n-score = $\frac{736-770}{\sqrt{184^2+150^2}}$ = -0.14

APPENDIX 4 – ACRONYMS AND ABBREVIATIONS

BTEX	Benzene, Toluene, Ethylbenzene, Xylenes
CITAC	Cooperation on International Traceability in Analytical Chemistry
CRM	Certified Reference Material
CV	Coefficient of Variation
DCM	Dichloromethane
EtOAc	Ethyl Acetate
FID	Flame Ionisation Detector
GAG	General Accreditation Guidance (NATA)
GC	Gas Chromatography
GUM	Guide to the expression of Uncertainty in Measurement
HEX	Hexane
HS	Headspace
IEC	International Electrotechnical Commission
ISO	International Organization for Standardization
LLE	Liquid-Liquid Extraction
LOR	Limit Of Reporting
Max.	Maximum value
Md	Median value
Min.	Minimum value
MS	Mass Spectrometry
MS/MS	Tandem Mass Spectrometry
MU	Measurement Uncertainty
N	Number of numeric results
NATA	National Association of Testing Authorities, Australia
NEPM	National Environmental Protection Measure
NMI	National Measurement Institute, Australia
NR	Not Reported
NT	Not Tested
P&T	Purge and Trap
PAH	Polycyclic Aromatic Hydrocarbon
PCV	Performance Coefficient of Variation
PENT	Pentane
PT	Proficiency Test
R.A.	Robust Average

RM	Reference Material
S.V.	Spiked Value (or formulated concentration of a PT sample)
SD	Standard Deviation
SI	International System of Units
SPE	Solid Phase Extraction
SS	Spiked Samples
TPH	Total Petroleum Hydrocarbons
TRH	Total Recoverable Hydrocarbons
USEPA	United States Environmental Protection Agency

END OF REPORT