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Department of Industry, Science and Resources

National Measurement Institute

Proficiency Test Final Report AQA 26-07 Hydrocarbons in Soil

July 2026

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Jenny Xu

Luminita Antin

Geoff Morschel

Jasmine Duong

Aaron Mamo

Zoë Baker-Moss

Scott Chen

Annmaree Kenny

Gabriela Saveluc

Jihong Liu

Raluca Iavetz

Manager, Chemical Reference Values

105 Delhi Road, North Ryde, NSW 2113, Australia

Phone: +61 2 9449 0234

Email: proficiency@measurement.gov.au

Accredited for compliance with ISO/IEC 17043

Accreditation No. 198; Site No. 14485

EXECUTIVE SUMMARY

This report presents the results of AQA 26-07 Hydrocarbons in Soil.

Twenty-three laboratories enrolled to participate, and all submitted results.

The sample set consisted of 3 soil samples, and participants were asked to report total recoverable hydrocarbons (TRH) in Sample S1; benzene, toluene, ethylbenzene and xylenes (BTEX) and volatile hydrocarbons (C6 to C10) in Sample S2; and polycyclic aromatic hydrocarbons (PAHs) in Sample S3.

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

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:

i. Compare participants' performances and assess their capabilities to identify and measure hydrocarbon pollutants in soil.

Laboratories 2, 6, 9, 12, 13, 20, and 22 reported numeric results for all 19 scored analytes.

Four participants reported false negatives ('less than' results where the assigned value was significantly higher than their limit of reporting, or participants reporting NR; total of 4 results).

Two participants reported additional analytes that were not spiked into the test samples (total of 2 results).

Of 363 z-scores, 316 (87%) returned $|z| \leq 2.0$, indicating an acceptable performance.

Of 358 E_n-scores, 291 (81%) returned $|E_n| < 1.0$, indicating agreement of the participant's result with the assigned value within their respective uncertainties.

Laboratories 2 and 6 returned acceptable z-scores and E_n-scores for all 19 scored analytes.

ii. Evaluate participants' methods for the measurement of hydrocarbon pollutants in soil.

There was no significant trend observed between methodology and results for TRH and PAHs, with good agreement between participants' results despite a range of different methodologies being employed.

For BTEX, those participants who used headspace GC-MS were biased low and may need to review their parameters to identify the source of this low bias.

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

Of 402 numeric results, 396 results (99%) were reported with an associated expanded measurement uncertainty. The magnitude of the reported expanded uncertainties was within the range 0.011% to 100% of the reported result.

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

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

NMIA also has certified reference material MX015 Hydrocarbon-Contaminated Soil available for purchase.

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1 INTRODUCTION

1.1 NMIA Proficiency Testing Program

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

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

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

1.2 Study Aims

The aims of the study were to:

- compare participants' performances and assess their capabilities to identify and measure hydrocarbon pollutants in soil;
- evaluate participants' methods for the measurement of hydrocarbon pollutants in soil;
- develop the practical application of measurement uncertainty, and provide participants with information that will be useful in assessing their uncertainty evaluations; 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 NMIA proficiency tests is described in the NMIA Study Protocol for Proficiency Testing.[2] The statistical methods used are described in the NMIA Chemical Proficiency Testing Statistical Manual.[3] These documents have been prepared with reference to ISO/IEC 17043 and The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories.[1,4]

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

2 STUDY INFORMATION

2.1 Study Timetable

The timetable of the study was:

Invitations sent	2 March 2026
Samples sent	23 March 2026
Results due	28 April 2026
Interim Report	30 April 2026
Preliminary Report	8 May 2026

2.2 Participation and Laboratory Code

Twenty-three laboratories enrolled to participate, and all participants were assigned a confidential laboratory code number for this study. All participants submitted results.

2.3 Selection of Analytes and Test Material Preparation

The hydrocarbons in this study and their spiked levels were typical of those encountered by environmental testing laboratories. Investigation levels for hydrocarbons are set out in the National Environment Protection (Assessment of Site Contamination) Measure (NEPM) Schedule B1 *Guideline on Investigation Levels for Soil and Groundwater*.[5]

Three test samples were provided for analysis:

- Sample S1 was 50 g soil spiked with treated diesel fuel and commercially purchased hydraulic oil. This sample assessed NEPM fractions >C10-C16, >C16-C34, >C34-C40, and TRH.
- Sample S2 was 50 g soil spiked with unleaded petrol, treated diesel fuel, and toluene. This sample assessed volatile hydrocarbons (C6-C10), benzene, toluene, ethylbenzene, xylenes, and Total BTEX.
- Sample S3 was 50 g soil spiked with polycyclic aromatic hydrocarbons (PAHs). The spiked values for analytes in this sample are presented in Table 1.

Table 1 Sample S3 Spiked Values

Sample	Analyte	Spiked Value (mg/kg)
S3	Naphthalene	2.01
S3	Acenaphthylene	0.601
S3	Acenaphthene	0.606
S3	Fluorene	0.251
S3	Phenanthrene	1.31
S3	Anthracene	0.202
S3	Pyrene	0.530
S3	Benz[a]anthracene	1.21
S3	Chrysene	3.03
S3	Benzo[b]fluoranthene	1.60
S3	Benzo[a]pyrene	0.106

Further information on the preparation of the samples is given in Appendix 1.

2.4 Homogeneity and Stability of Test Materials

No homogeneity or stability testing was conducted for this PT study. The samples were prepared, packaged, stored, and dispatched using a process that has been demonstrated to produce homogeneous and stable samples in previous NMIA hydrocarbons in soil PT studies. The storage stability of petroleum hydrocarbons in soil has also been previously established.[6]

Participants' results did not give reason to question the homogeneity or transport stability of the samples (Appendix 2). To further assess possible instability, participants' results were compared to the spiked values where available. Assigned values for Sample S3 PAHs were within the ranges of 57% to 83% of the spiked values, which is similar to ratios observed in previous NMIA hydrocarbons in soil PT studies.

2.5 Sample Storage, Dispatch and Receipt

Prior to dispatch, Samples S1 and S3 were stored in a refrigerator at approximately 4°C, and Sample S2 was stored in a freezer at approximately -20°C. The samples were packaged in insulated polystyrene foam boxes with cooler bricks and dispatched by courier on 23 March 2026.

The following items were also sent to participants:

- a 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.

2.6 Instructions to Participants

Participants were informed that Sample S1 was spiked with semi-volatile petroleum pollutants (>C10-C40), Sample S2 was spiked with volatile petroleum pollutants (BTEX components), and Sample S3 was spiked with selected PAHs. The list of possible PAHs given to participants is presented in Table 2.

Table 2 List of Possible PAHs for Sample S3

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

Participants were instructed as follows:

- If samples cannot be analysed on the day of receipt, please store Samples S1 and S3 refrigerated, and Sample S2 frozen.
- Quantitatively analyse the samples using your routine test method. It is recommended to mix all samples well before subsampling.
- Do not test for volatile hydrocarbons (C6-C10) or BTEX components in Sample S1.
- Participants need not test for all listed analytes.
- Report results on as received basis in units of mg/kg for the following:
    - Sample S1: Semi-volatile hydrocarbons (>C10-C40) and Total Recoverable Hydrocarbons (TRH). Use your laboratory's chosen quantitation ranges and indicate

what these ranges are. Results will be assessed using Australian NEPM fractions >C10-C16, >C16-C34, >C34-C40, and TRH. The spike concentration range is between 1000 – 20000 mg/kg.

- Sample S2: Volatile Hydrocarbons (C6-C10), Benzene, Toluene, Ethylbenzene and Total Xylenes, and Total BTEX. Individual BTEX components spike concentration is between 50 – 5000 mg/kg.
- Sample S3: PAHs. The spike concentration range is between 0.05 – 50 mg/kg.

- This figure will be used in all statistical analysis in the study report.
- Report results as you would report to a client, i.e. corrected for recovery or not according to your standard procedure and applying the limit of reporting of the method used for analysis (no limit of reporting has been set for this study).
- For each analyte in each sample, report the associated expanded uncertainty in units of mg/kg (e.g. 2000 ± 200 mg/kg).
- Report any listed analyte not tested as NT as the result.
- Report the basis of your uncertainty evaluations as requested in the results sheet (e.g. uncertainty budget, repeatability precision, long term result variability).
- Please complete the method details as requested in the results sheet.
- Return the completed results sheet by email (proficiency@measurement.gov.au).
- Please return results by 20 April 2026. Late results may not be included in the study report.

The results due date was later extended to 28 April 2026 for all participants to account for public holidays.

2.7 Interim Report and Preliminary Report

An Interim Report was emailed to all participants on 30 April 2026.

A Preliminary Report was emailed to all participants on 8 May 2026. This report included a summary of the results reported by laboratories, assigned values, performance coefficient of variations, z-scores, and E_n-scores for each analyte in this study. The following has been changed from the Preliminary Report in the present Final Report:

- A result has been flagged as an extreme outlier in Sample S2 benzene. Statistics have been updated but the analyte remains unscored.
- The spiked value and scoring of Sample S3 pyrene (Table 3). A high recovery of the spiked value for Sample S3 pyrene was observed, and an investigation was conducted. The spiking standard solution was a higher concentration than the expected preparation value, potentially due to an error during spiking standard solution preparation. The spiked value has therefore been adjusted accordingly. Participants' results reported for this analyte were in good agreement with each other and with the adjusted spiked value. An assigned value was set for this analyte, and results have now been scored.

Table 3 Sample S3 Pyrene Expected Spiked Value and Adjusted Spiked Value

Sample	Analyte	Expected Spiked Value (mg/kg)	Adjusted Spiked Value (mg/kg)
S3	Pyrene	0.298	0.530

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Methods Reported by Participants

Participants were requested to provide information about their test methods. Responses received are presented in Appendix 4. Some responses may be modified so that the participant cannot be identified.

3.2 Basis of Participants' Measurement Uncertainty Evaluations

Participants were requested to provide information about their basis of measurement uncertainty (MU). Responses received are presented in Table 4. Some responses may be modified so that the participant cannot be identified.

Table 4 Basis of Expanded Uncertainty Evaluation

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
1	Top Down - precision and evaluation of the method and laboratory bias k = 2	Control samples - SS Instrument calibration	Instrument calibration Recoveries of SS Standard purity	Eurachem/CITAC Guide
2	Top Down - precision and evaluation of the method and laboratory bias k = 2	Control samples - SS		Eurachem/CITAC Guide
3	Top Down - precision and evaluation of the method and laboratory bias Coverage factor not reported	Control samples - CRM	CRM Recoveries of SS	Eurachem/CITAC Guide
4	Top Down - precision and evaluation of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis Instrument calibration	CRM Instrument calibration Recoveries of SS	NMIA Uncertainty Course
5	Top Down - precision and evaluation of the method and laboratory bias Coverage factor not reported	Control samples Duplicate analysis Instrument calibration	Instrument calibration	
6	Top Down - precision and evaluation of the method and laboratory bias k = 2	Duplicate analysis	Recoveries of SS	Eurachem/CITAC Guide
7	Top Down - precision and evaluation of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis	Instrument calibration Recoveries of SS	Eurachem/CITAC Guide
8	Top Down - precision and evaluation of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis Instrument calibration	CRM Instrument calibration Recoveries of SS	ISO/GUM
9	Top Down - precision and evaluation of the method	Control samples - SS		Eurachem/CITAC Guide

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
	and laboratory bias k = 2			
10	Top Down - precision and evaluation of the method and laboratory bias k = 2	Control samples - SS Duplicate analysis Instrument calibration	CRM Instrument calibration Recoveries of SS	ISO/GUM
11	TRH: Top Down - precision and evaluation of the method and laboratory bias k = 2 PAHs: Professional judgment k = 2	Control samples - SS Duplicate analysis Instrument calibration	Instrument calibration Recoveries of SS Standard purity	Eurachem/CITAC Guide
12	Top Down - precision and evaluation of the method and laboratory bias Coverage factor not reported	Control samples - SS Duplicate analysis Instrument calibration	CRM Instrument calibration Recoveries of SS	Eurachem/CITAC Guide
13	Top Down - precision and evaluation of the method and laboratory bias Coverage factor not reported	Control samples - CRM Duplicate analysis Instrument calibration	CRM Instrument calibration Recoveries of SS Standard purity	ISO/GUM
14	Standard uncertainty based on historical data k = 2	Duplicate analysis Instrument calibration	CRM Instrument calibration Standard purity	Eurachem/CITAC Guide
15	Bottom Up (ISO/GUM, fish bone/cause and effect diagram) Coverage factor not reported	Control samples - RM / Ex PT Sample Duplicate analysis Instrument calibration		
16	Top Down - reproducibility (standard deviation) from PT studies used directly Coverage factor not reported	Duplicate analysis	Instrument calibration	
17	Top Down - precision and evaluation of the method and laboratory bias k = 2	Control samples - CRM Duplicate analysis Instrument calibration	CRM Instrument calibration	Eurachem/CITAC Guide
18	Top Down - precision and evaluation of the method and laboratory bias Coverage factor not reported	Control samples - CRM Duplicate analysis		Eurachem/CITAC Guide
19	Top Down - precision and evaluation of the method and laboratory bias	Control samples - SS	CRM Recoveries of SS	ISO/GUM

Lab. Code	Approach to Evaluating MU	Information Sources for MU Evaluation*		Guide Document for Evaluating MU
		Precision	Method Bias	
	Coverage factor not reported			
20	Top Down - precision and evaluation of the method and laboratory bias k = 2	Control samples - SS		Eurachem/CITAC Guide
21	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control samples - SS	Recoveries of SS	
22	Standard deviation of replicate analyses multiplied by 2 or 3 k = 2	Standard deviation from PT studies only		ISO/GUM
23	Top Down - precision and evaluation of the method and laboratory bias k = 2	Control samples - RM / Ex PT Sample	CRM	ISO/GUM

*CRM = Certified Reference Material; RM = Reference Material; SS = Spiked Samples; PT = Proficiency Testing

3.3 Participants' Comments

Participants were invited to comment on this study or future studies. Such feedback may be useful in improving future studies. No comments were received for this PT study.

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 5 to 26 with summary statistics and other estimates of analyte mass fraction. 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.

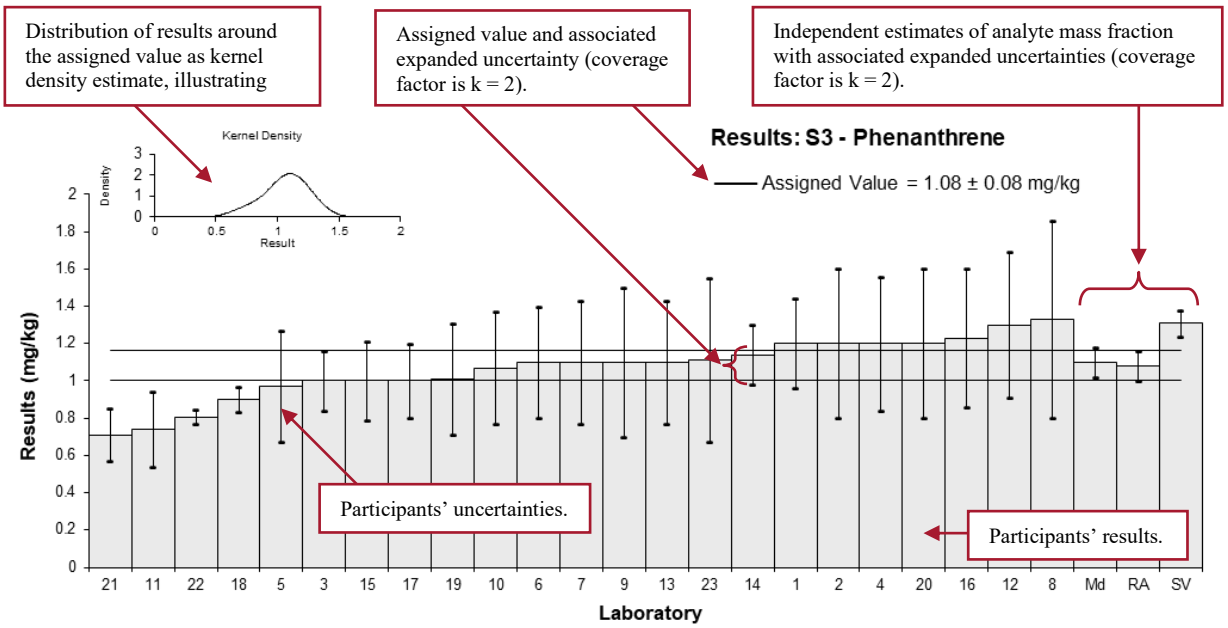


Figure 1 Guide to Presentation of Results

4.2 Outliers and Extreme Outliers

Outliers were results less than 50% and greater than 150% of the robust average and were removed before the calculation of the assigned value.[3,4] Extreme outliers were obvious blunders, such as those with incorrect units, decimal errors, or for a different analyte or sample, and such results were removed for the calculation of all summary statistics.[3]

4.3 Assigned Value

The assigned value is defined as the 'value attributed to a particular property or characteristic of a proficiency testing item'.[1] In this PT study, this property is the mass fraction of the analytes in the samples. Assigned values were the robust averages of participants' results, outliers and extreme outliers removed; the expanded uncertainties were evaluated 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.[7] Robust CV as presented in the Section 5 (Statistics table) is the robust between-laboratory CV including outliers but excluding extreme outliers. Everywhere else in the report the robust between-laboratory CV refers to the robust CV calculated with both outliers and extreme outliers removed.

4.5 Performance Coefficient of Variation

The performance coefficient of variation (PCV) is a set measure of the between-laboratory variation that in the judgement of the study coordinator would be expected from participants, given the levels of analytes present. The PCV is not the CV of participants' results; it is set by the study coordinator and is based on the mass fraction of the analytes and experience from

previous studies, and is supported by mathematical models such as the Thompson-Horwitz equation.[8] By setting a realistic value for the PCV, a participant's performance does not depend on other participants' performance and can be compared from study to study when the PCV remains the same.

4.6 Standard Deviation for Proficiency Assessment

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

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

4.7 z-Score

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

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

where:

z is z-score

χ is a participant's result

X is the assigned value

σ is the SDPA from Equation 1

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

For the absolute value of a z-score: $|z| \leq 2.0$ is acceptable; $2.0 < |z| < 3.0$ is questionable; and $|z| \geq 3.0$ is not acceptable.

4.8 E_n-Score

The E_n-score is complementary to the z-score in assessment of laboratory performance. E_n-score includes expanded 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: $|E_n| < 1.0$ is acceptable; and $|E_n| \geq 1.0$ is not acceptable.

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

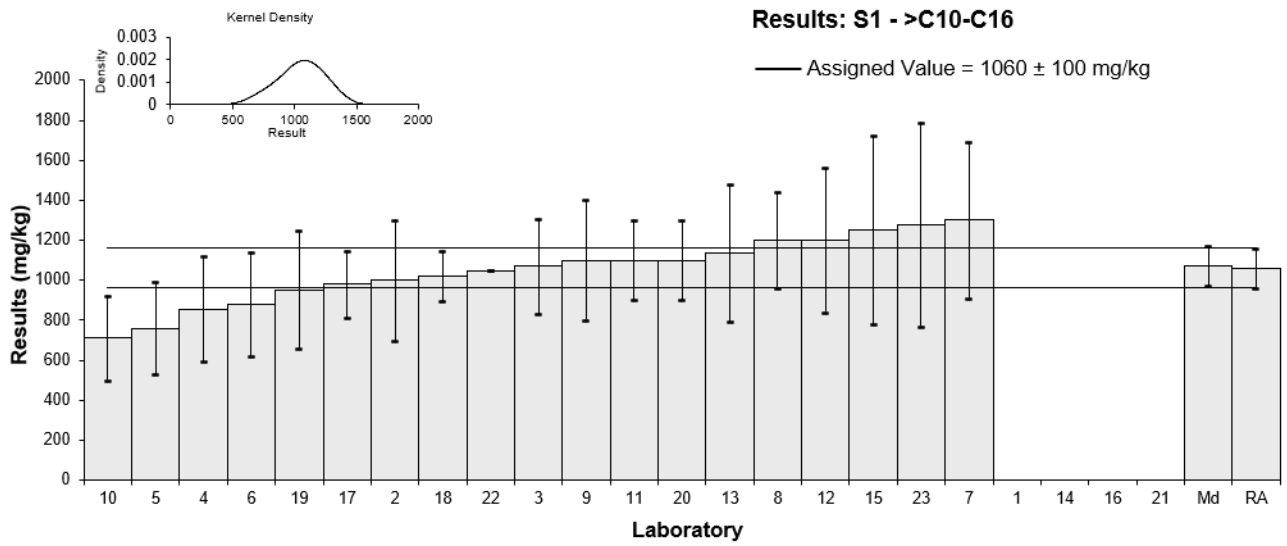
Sample No.	S1
Matrix	Soil
Analyte	>C10-C16
Unit	mg/kg

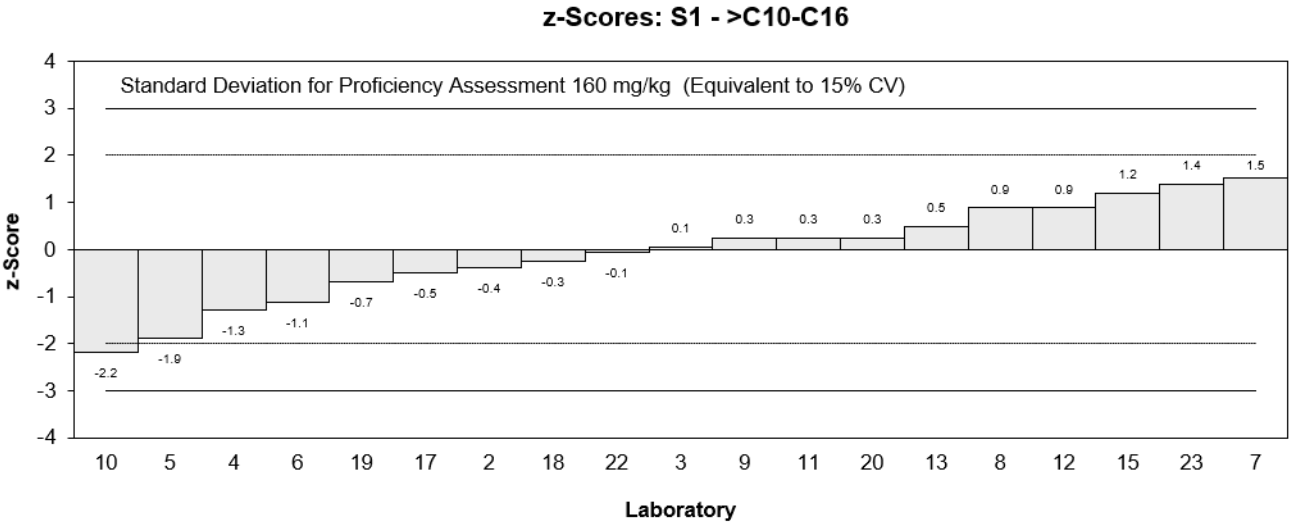
Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	NR	NR		
2	1000	300	-0.38	-0.19
3	1070	239	0.06	0.04
4	857	260	-1.28	-0.73
5	760	230	-1.89	-1.20
6	880	260	-1.13	-0.65
7	1300	390	1.51	0.60
8	1200	240	0.88	0.54
9	1100	300	0.25	0.13
10	711	213	-2.19	-1.48
11	1100	200	0.25	0.18
12	1200	360	0.88	0.37
13	1136	340.8	0.48	0.21
14	NR	NR		
15	1250	470	1.19	0.40
16	NT	NT		
17	980	169	-0.50	-0.41
18	1020	127.5	-0.25	-0.25
19	951	295	-0.69	-0.35
20	1100	200	0.25	0.18
21	NR	NR		
22	1048.9	0.9	-0.07	-0.11
23	1279	511	1.38	0.42

Statistics

Assigned Value	1060	100
Robust Average	1060	100
Median	1070	100
Mean	1050	
N	19	
Max	1300	
Min	711	
Robust SD	180	
Robust CV	17%	


Kernel Density
Density
Result
Results: S1 - >C10-C16
Assigned Value = 1060 ± 100 mg/kg
Results (mg/kg)
Laboratory
10 5 4 6 19 17 2 18 22 3 9 11 20 13 8 12 15 23 7 1 14 16 21 Md RA


z-Scores: S1 - >C10-C16
Standard Deviation for Proficiency Assessment 160 mg/kg (Equivalent to 15% CV)
z-Score
-2.2 -1.9 -1.3 -1.1 -0.7 -0.5 -0.4 -0.3 -0.1 0.1 0.3 0.3 0.3 0.5 0.9 0.9 1.2 1.4 1.5
Laboratory
10 5 4 6 19 17 2 18 22 3 9 11 20 13 8 12 15 23 7

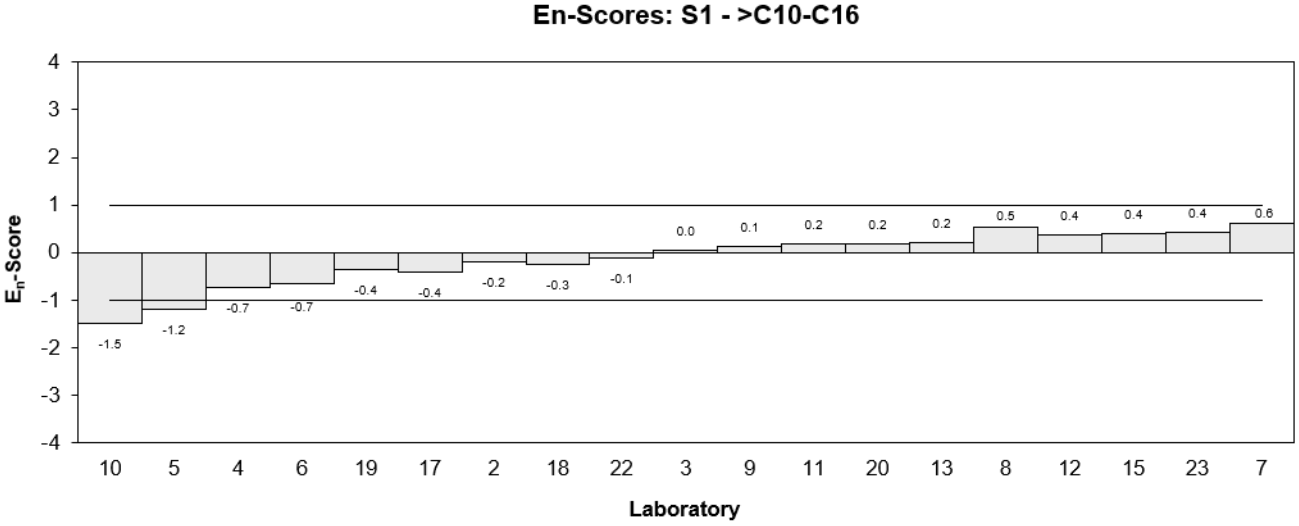

En-Scores: S1 - >C10-C16
En-Score
-1.5 -1.2 -0.7 -0.7 -0.4 -0.4 -0.2 -0.3 -0.1 0.0 0.1 0.2 0.2 0.2 0.5 0.4 0.4 0.4 0.6
Laboratory
10 5 4 6 19 17 2 18 22 3 9 11 20 13 8 12 15 23 7

Figure 2

Table 6

Sample Details

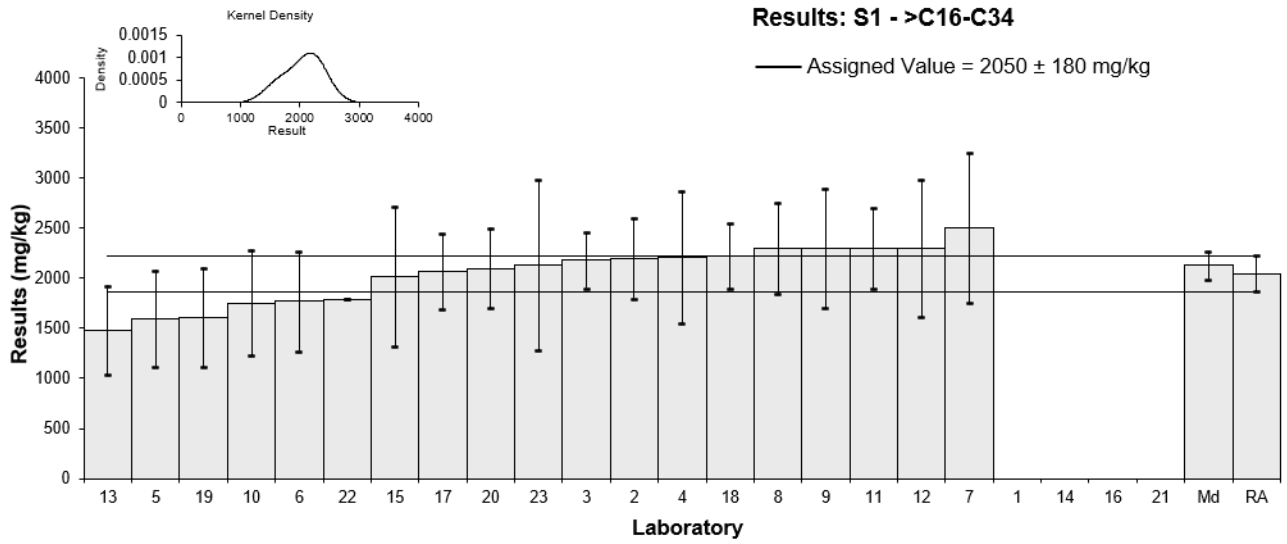
Sample No.	S1
Matrix	Soil
Analyte	>C16-C34
Unit	mg/kg

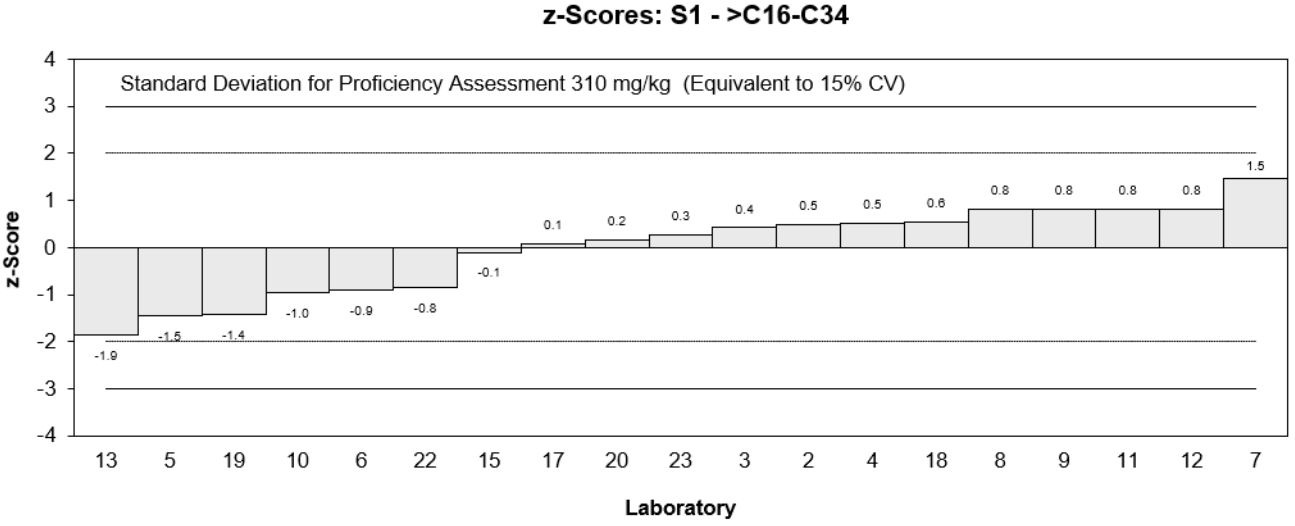
Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	NR	NR		
2	2200	400	0.49	0.34
3	2180	280	0.42	0.39
4	2210	665	0.52	0.23
5	1600	480	-1.46	-0.88
6	1770	500	-0.91	-0.53
7	2500	750	1.46	0.58
8	2300	460	0.81	0.51
9	2300	600	0.81	0.40
10	1755	527	-0.96	-0.53
11	2300	400	0.81	0.57
12	2300	690	0.81	0.35
13	1482	444.6	-1.85	-1.18
14	NR	NR		
15	2020	700	-0.10	-0.04
16	NT	NT		
17	2070	375	0.07	0.05
18	2220	328.1	0.55	0.45
19	1609	499	-1.43	-0.83
20	2100	400	0.16	0.11
21	NR	NR		
22	1790.6	0.2	-0.84	-1.44
23	2131	852	0.26	0.09

Statistics

Assigned Value	2050	180
Robust Average	2050	180
Median	2130	140
Mean	2040	
N	19	
Max	2500	
Min	1482	
Robust SD	320	
Robust CV	16%	


Kernel Density
Density
Result
Results: S1 - >C16-C34
Assigned Value = 2050 ± 180 mg/kg
Results (mg/kg)
Laboratory
13 5 19 10 6 22 15 17 20 23 3 2 4 18 8 9 11 12 7 1 14 16 21 Md RA


z-Scores: S1 - >C16-C34
Standard Deviation for Proficiency Assessment 310 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
13 5 19 10 6 22 15 17 20 23 3 2 4 18 8 9 11 12 7

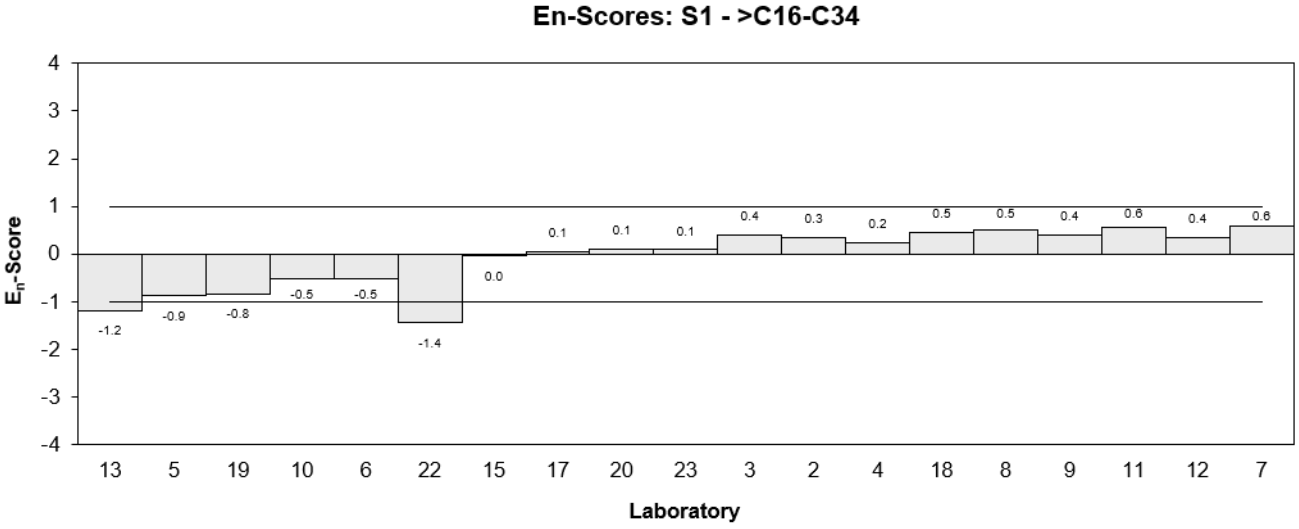

En-Scores: S1 - >C16-C34
En-Score
Laboratory
13 5 19 10 6 22 15 17 20 23 3 2 4 18 8 9 11 12 7

Figure 3

Table 7

Sample Details

Sample No.	S1
Matrix	Soil
Analyte	>C34-C40
Unit	mg/kg

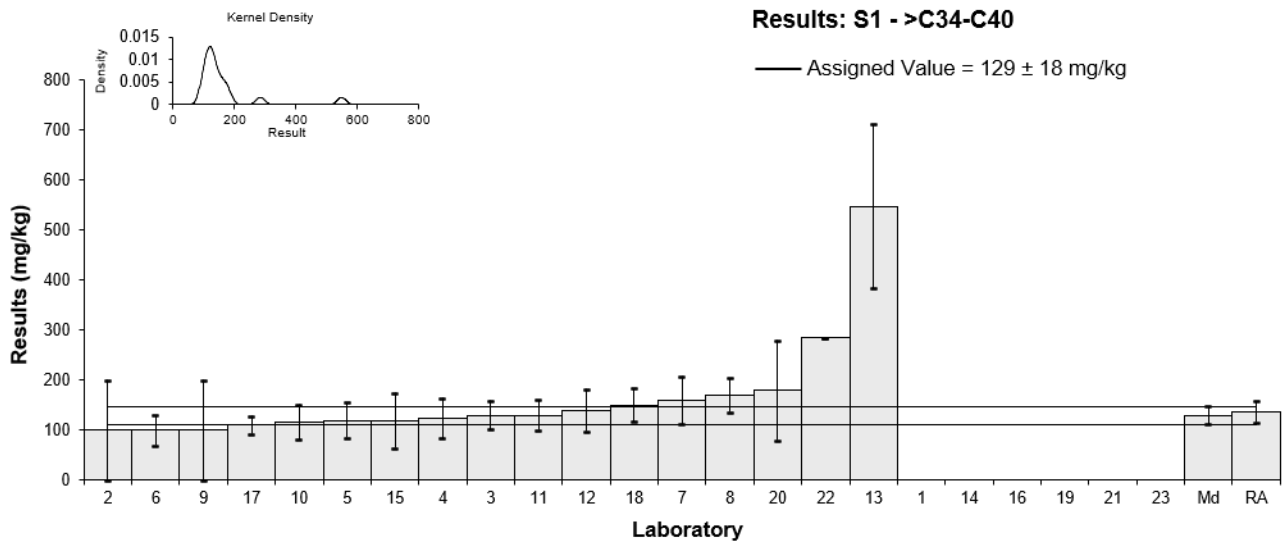
Participant Results

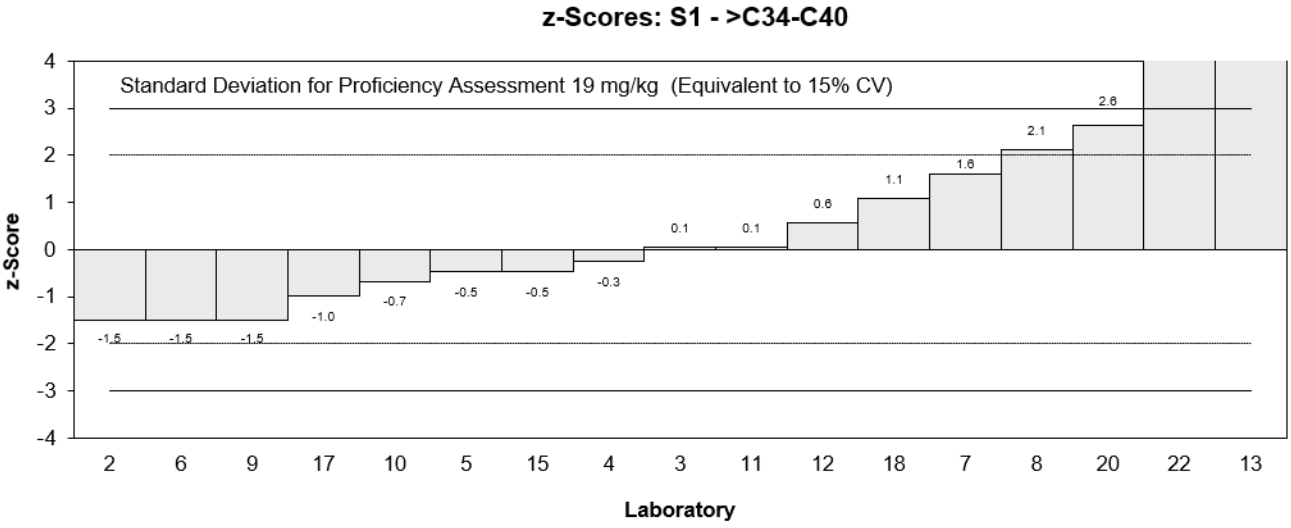
Lab. Code	Result	Uncertainty	z	E_n
1	NR	NR		
2	100	100	-1.50	-0.29
3	130	29	0.05	0.03
4	124	40	-0.26	-0.11
5	120	35	-0.47	-0.23
6	100	30	-1.50	-0.83
7	160	48	1.60	0.60
8	170	34	2.12	1.07
9	100	100	-1.50	-0.29
10	116	35	-0.67	-0.33
11	130	30	0.05	0.03
12	140	42	0.57	0.24
13*	548	164.4	21.65	2.53
14	NR	NR		
15	120	55	-0.47	-0.16
16	NT	NT		
17	110	18	-0.98	-0.75
18	150	33	1.09	0.56
19	< 100	NR		
20	180	100	2.64	0.50
21	NR	NR		
22*	284.3	0.6	8.03	8.62
23	<100	100		

* Outlier, see Section 4.2

Statistics

Assigned Value	129	18
Robust Average	137	21
Median	130	18
Mean	164	
N	17	
Max	548	
Min	100	
Robust SD	35	
Robust CV	26%	


Kernel Density
Density
Result
Results: S1 - >C34-C40
Assigned Value = 129 ± 18 mg/kg
Results (mg/kg)
Laboratory
2 6 9 17 10 5 15 4 3 11 12 18 7 8 20 22 13 1 14 16 19 21 23 Md RA


z-Scores: S1 - >C34-C40
Standard Deviation for Proficiency Assessment 19 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
2 6 9 17 10 5 15 4 3 11 12 18 7 8 20 22 13

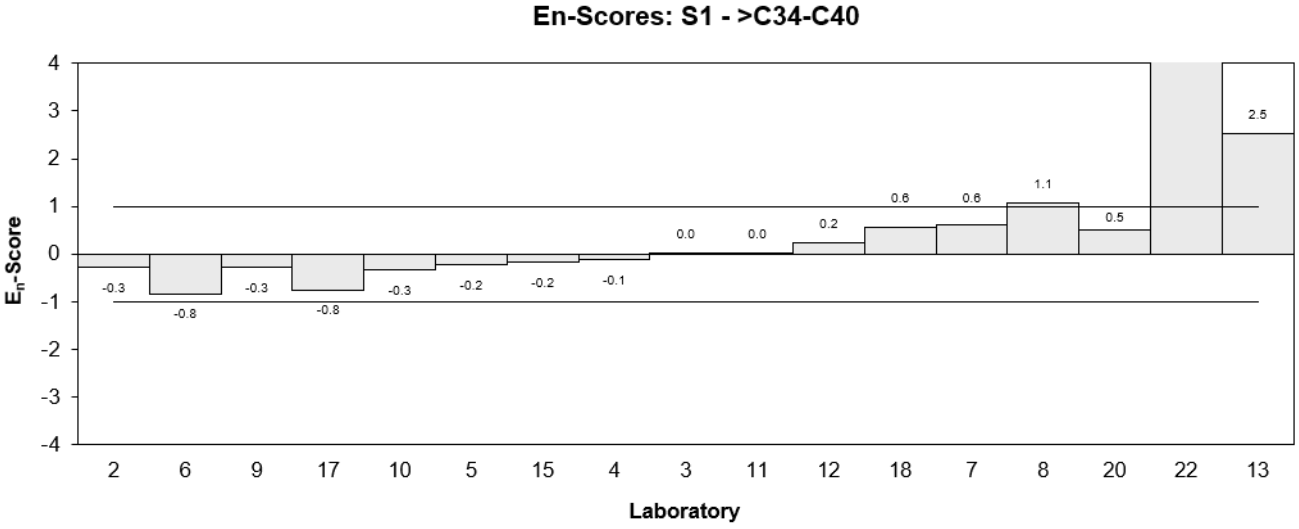

En-Scores: S1 - >C34-C40
En-Score
Laboratory
2 6 9 17 10 5 15 4 3 11 12 18 7 8 20 22 13

Figure 4

Table 8

Sample Details

Sample No.	S1
Matrix	Soil
Analyte	TRH
Unit	mg/kg

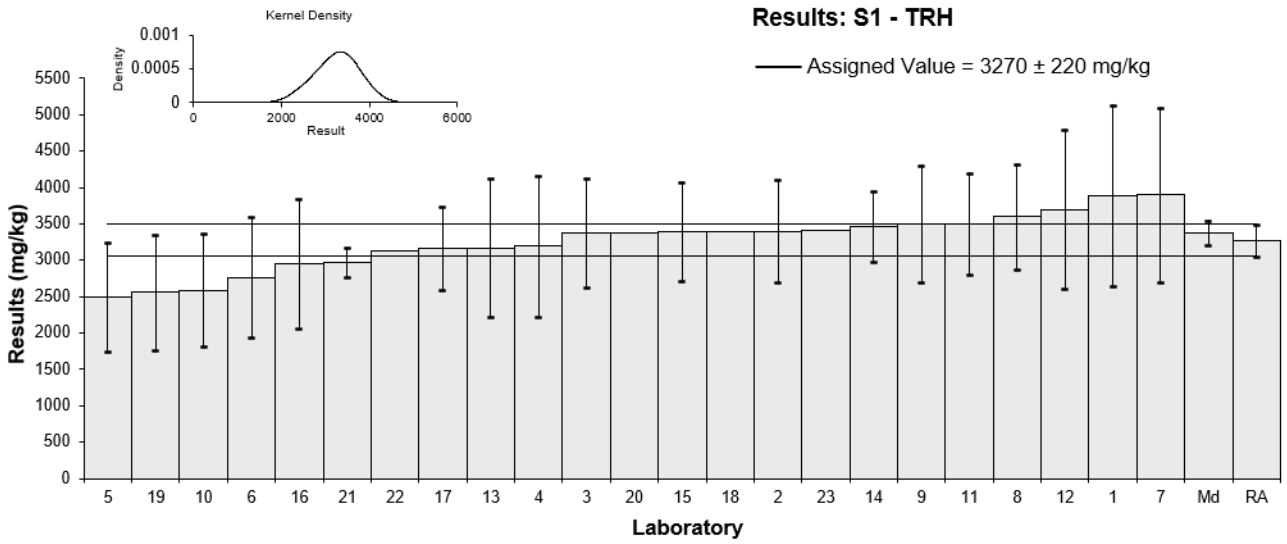
Participant Results

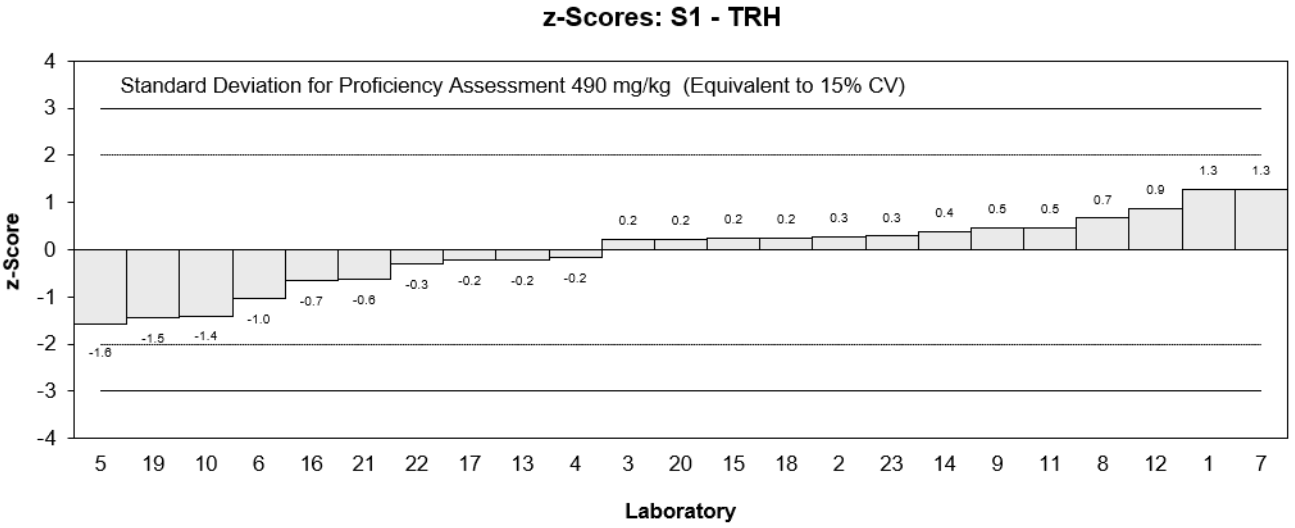
Lab. Code	Result	Uncertainty	z	E_n
1	3891	1245	1.27	0.49
2	3400	700	0.27	0.18
3	3380	747	0.22	0.14
4	3191	965	-0.16	-0.08
5	2500	750	-1.57	-0.99
6	2760	830	-1.04	-0.59
7	3900	1200	1.28	0.52
8	3600	720	0.67	0.44
9	3500	800	0.47	0.28
10	2583	775	-1.40	-0.85
11	3500	700	0.47	0.31
12	3700	1100	0.88	0.38
13	3166	949.8	-0.21	-0.11
14	3460	480	0.39	0.36
15	3390	680	0.24	0.17
16	2950	885	-0.65	-0.35
17	3160	573	-0.22	-0.18
18	3390	NR	0.24	0.55
19	2560	794	-1.45	-0.86
20	3380	NR	0.22	0.50
21	2966.01	207.62	-0.62	-1.00
22	3123.8	NR	-0.30	-0.66
23	3410	NR	0.29	0.64

Statistics

Assigned Value	3270	220
Robust Average	3270	220
Median	3380	170
Mean	3250	
N	23	
Max	3900	
Min	2500	
Robust SD	420	
Robust CV	13%	

Laboratories 20 and 23 did not report a TRH result. The study coordinator calculated TRH values for these laboratories by summing the individual hydrocarbon ranges they reported. Uncertainty evaluations were not performed for these results.


Kernel Density
Density
0.001
0.0005
0
0
2000
4000
6000
Result
Results: S1 - TRH
Assigned Value = 3270 ± 220 mg/kg
Results (mg/kg)
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
5 19 10 6 16 21 22 17 13 4 3 20 15 18 2 23 14 9 11 8 12 1 7 Md RA
Laboratory


z-Scores: S1 - TRH
Standard Deviation for Proficiency Assessment 490 mg/kg (Equivalent to 15% CV)
z-Score
4
3
2
1
0
-1
-2
-3
-4
-1.6 -1.5 -1.4 -1.0 -0.7 -0.6 -0.3 -0.2 -0.2 -0.2 0.2 0.2 0.2 0.2 0.3 0.3 0.4 0.5 0.5 0.7 0.9 1.3 1.3
5 19 10 6 16 21 22 17 13 4 3 20 15 18 2 23 14 9 11 8 12 1 7
Laboratory

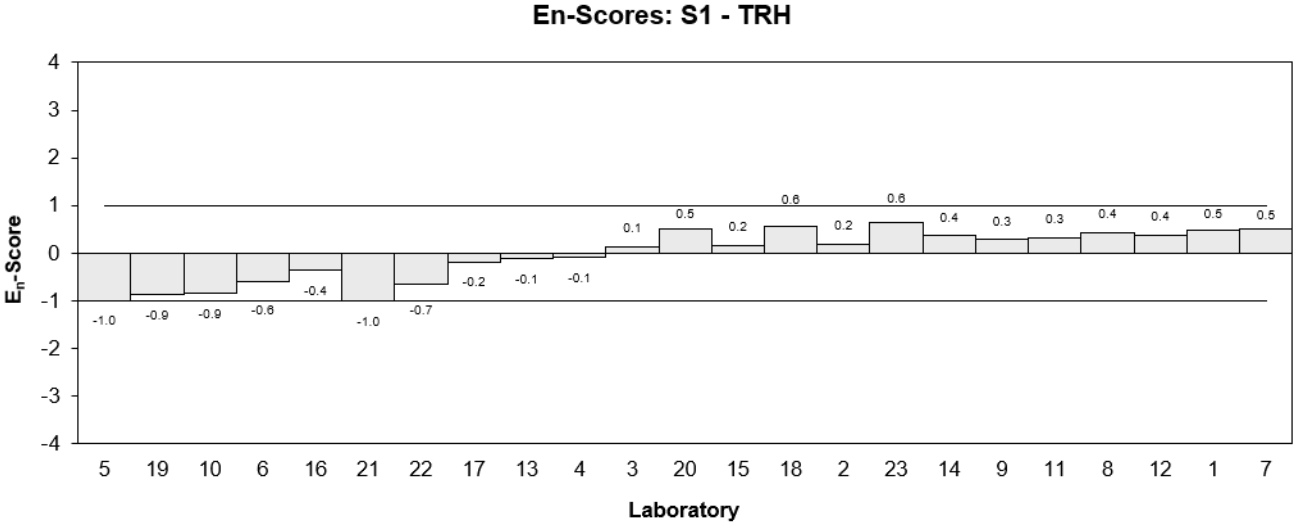

En-Scores: S1 - TRH
En-Score
4
3
2
1
0
-1
-2
-3
-4
-1.0 -0.9 -0.9 -0.6 -0.4 -1.0 -0.7 -0.2 -0.1 -0.1 0.1 0.5 0.2 0.6 0.2 0.6 0.4 0.3 0.3 0.4 0.4 0.5 0.5
5 19 10 6 16 21 22 17 13 4 3 20 15 18 2 23 14 9 11 8 12 1 7
Laboratory

Figure 5

Table 9 Non-NEPM Hydrocarbon Ranges Reported by Participants for Sample S1

Lab. Code	Range	Result (mg/kg)	Uncertainty (mg/kg)
1	C7-C9	31	12
	C10-C14	552	193
	C15-C36	3308	992
14	C7-C9	<20	6.7
	C10-C14	364	80
	C15-C36	3090	470
15	C10-C14	560	210
	C15-C28	2530	770
16	C10-C36	2950	885
21	C7-C9	<100	NR
	C10-C14	526.78	36.87
	C15-C36	2439.23	170.75

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Table 10

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	C6-C10
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty
1**	NT	NT
2	2200	500
3	2570	601
4	2109	635
5	2100	650
6	2700	670
7	1400	420
8	2400	480
9	2500	600
10	2259.54	677.9
11	NT	NT
12	2600	780
13	1367	410.1
14**	NT	NT
15	2270	620
16**	NT	NT
17	2930	705
18	2740	602.8
19	2600	830
20	2100	500
21	NT	NT
22	2090	42.44
23**	12900	5100

** Excluded Result, see Section 6.1

Statistics

Assigned Value	Not Set	
Robust Average	2330	220
Median	2270	210
Mean	2290	
N	17	
Max	2930	
Min	1367	
Robust SD	370	
Robust CV	16%	

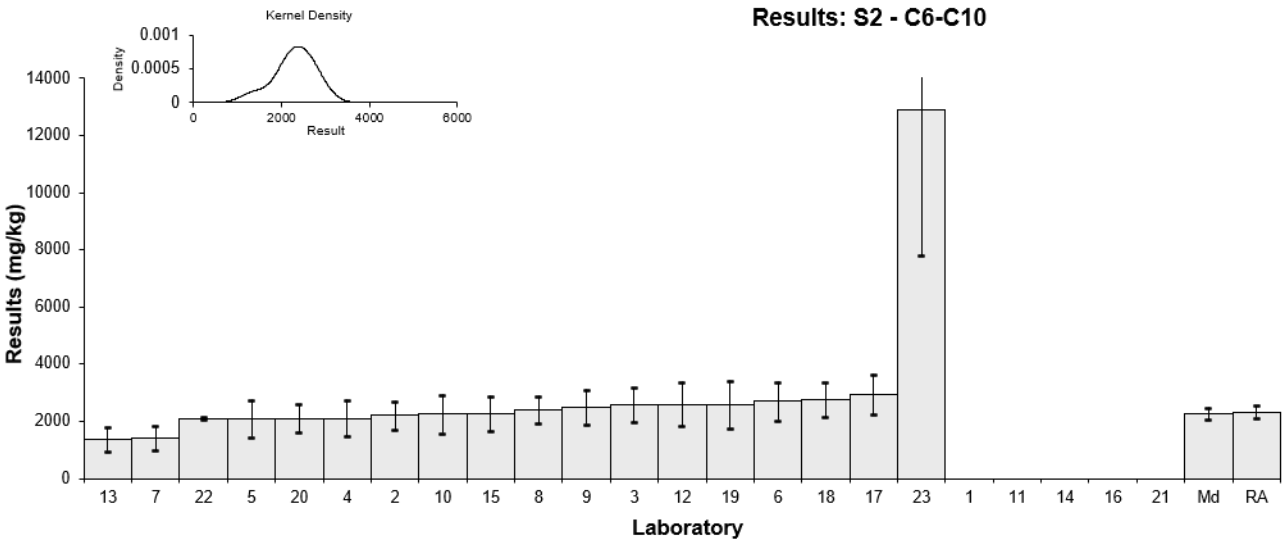

Kernel Density
Density
0.001
0.0005
0
0
2000
4000
6000
Result
Results: S2 - C6-C10
Results (mg/kg)
14000
12000
10000
8000
6000
4000
2000
0
13
7
22
5
20
4
2
10
15
8
9
3
12
19
6
18
17
23
1
11
14
16
21
Md
RA
Laboratory

Figure 6

Table 11

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	Benzene
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty
1**	4.7	1.6
2	19	5
3	19	3.95
4	13	5
5	16	4.8
6	21	5
7	12	3.6
8	15	3
9	17	5
10	18.97	5.7
11	NT	NT
12	10	3
13*	1.6	0.48
14**	7.89	0.73
15	15.8	4.1
16**	1.88	0.564
17	27.2	3.8
18	19.3	4
19	16	5
20	13	8
21	NT	NT
22	27.49	5.5
23**	8.6	3.4

* Extreme Outlier, see Section 4.2; ** Excluded Result, see Section 6.1

Statistics

Assigned Value	Not Set	
Robust Average	17.1	2.8
Median	16.5	2.5
Mean	17.5	
N	16	
Max	27.49	
Min	10	
Robust SD	4.5	
Robust CV	26%	

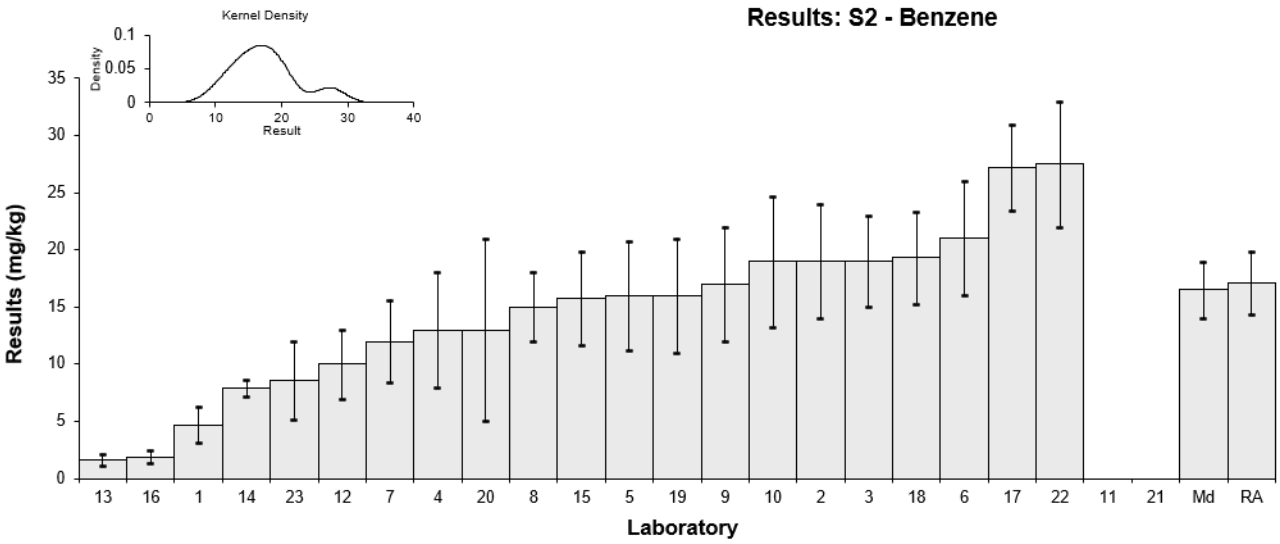

Results: S2 - Benzene
Kernel Density
Density
0.1
0.05
0
0
10
20
30
40
Result
Results (mg/kg)
35
30
25
20
15
10
5
0
13
16
1
14
23
12
7
4
20
8
15
5
19
9
10
2
3
18
6
17
22
11
21
Md
RA
Laboratory

Figure 7

Table 12

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	Toluene
Unit	mg/kg

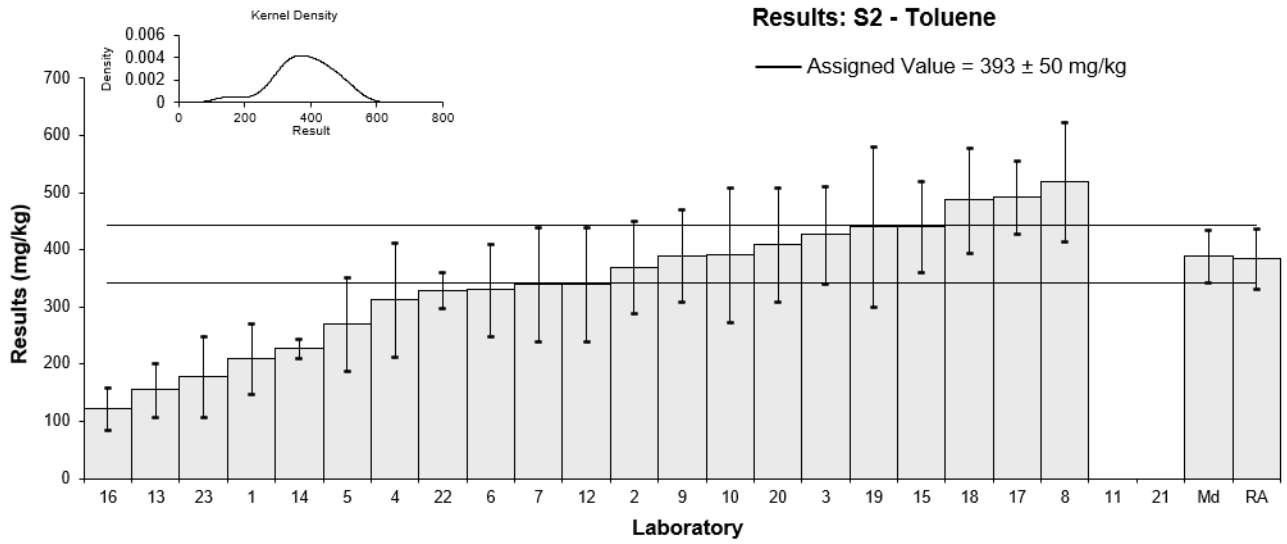
Participant Results

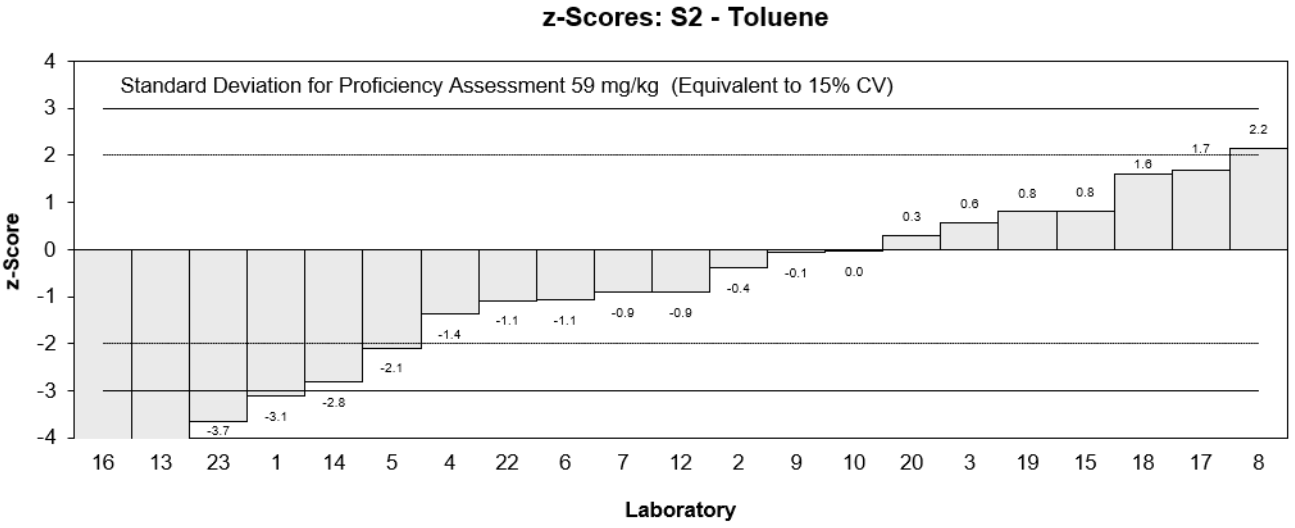
Lab. Code	Result	Uncertainty	z	E_n
1**	210	61	-3.10	-2.32
2	370	80	-0.39	-0.24
3	427	85	0.58	0.34
4	313	100	-1.36	-0.72
5	270	82	-2.09	-1.28
6	330	80	-1.07	-0.67
7	340	100	-0.90	-0.47
8	520	104	2.15	1.10
9	390	80	-0.05	-0.03
10	391.43	117.4	-0.03	-0.01
11	NT	NT		
12	340	100	-0.90	-0.47
13*	155	46.5	-4.04	-3.49
14**	227	17	-2.82	-3.14
15	441	80	0.81	0.51
16**	123	36.9	-4.58	-4.34
17	492	64	1.68	1.22
18	487	91.6	1.59	0.90
19	440	140	0.80	0.32
20	410	100	0.29	0.15
21	NT	NT		
22	329.1	31.6	-1.08	-1.08
23**	178	71	-3.65	-2.48

* Outlier, see Section 4.2; ** Excluded Result, see Section 6.1

Statistics

Assigned Value	393	50
Robust Average	385	52
Median	390	46
Mean	379	
N	17	
Max	520	
Min	155	
Robust SD	86	
Robust CV	22%	


Kernel Density
Density
0.006
0.004
0.002
0
0
200
400
600
800
Result
Results: S2 - Toluene
Assigned Value = 393 ± 50 mg/kg
Results (mg/kg)
700
600
500
400
300
200
100
0
16 13 23 1 14 5 4 22 6 7 12 2 9 10 20 3 19 15 18 17 8 11 21 Md RA
Laboratory


z-Scores: S2 - Toluene
Standard Deviation for Proficiency Assessment 59 mg/kg (Equivalent to 15% CV)
z-Score
4
3
2
1
0
-1
-2
-3
-4
-3.7 -3.1 -2.8 -2.1 -1.4 -1.1 -1.1 -0.9 -0.9 -0.4 -0.1 0.0 0.3 0.6 0.8 0.8 1.6 1.7 2.2
16 13 23 1 14 5 4 22 6 7 12 2 9 10 20 3 19 15 18 17 8
Laboratory

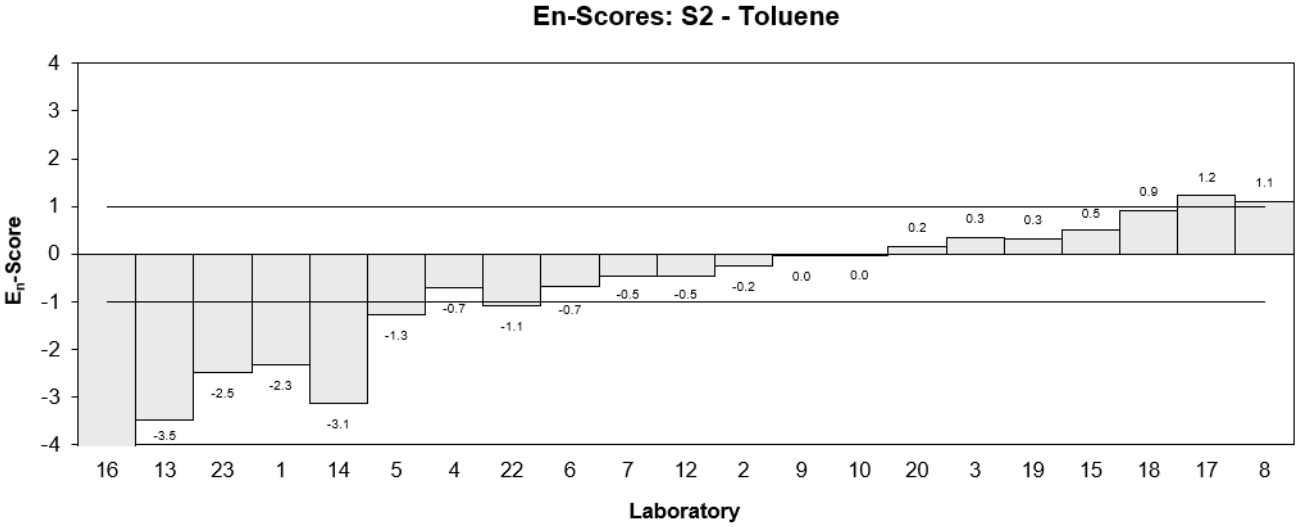

En-Scores: S2 - Toluene
En-Score
4
3
2
1
0
-1
-2
-3
-4
-3.5 -2.5 -2.3 -3.1 -1.3 -0.7 -1.1 -0.7 -0.5 -0.5 -0.2 0.0 0.0 0.2 0.3 0.3 0.5 0.9 1.2 1.1
16 13 23 1 14 5 4 22 6 7 12 2 9 10 20 3 19 15 18 17 8
Laboratory

Figure 8

Table 13

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	Ethylbenzene
Unit	mg/kg

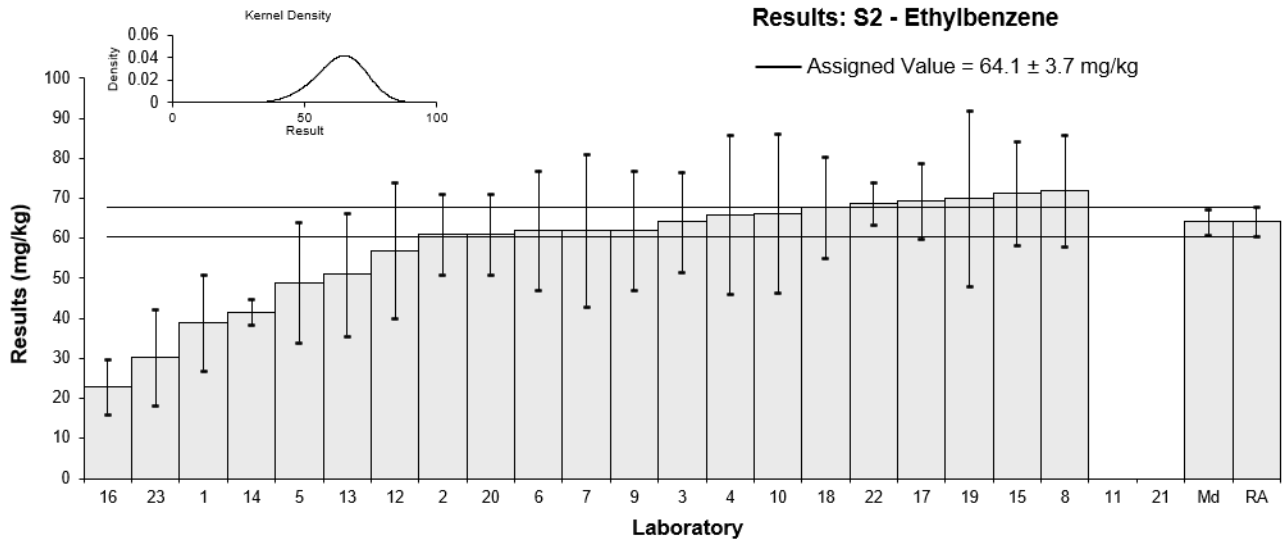
Participant Results

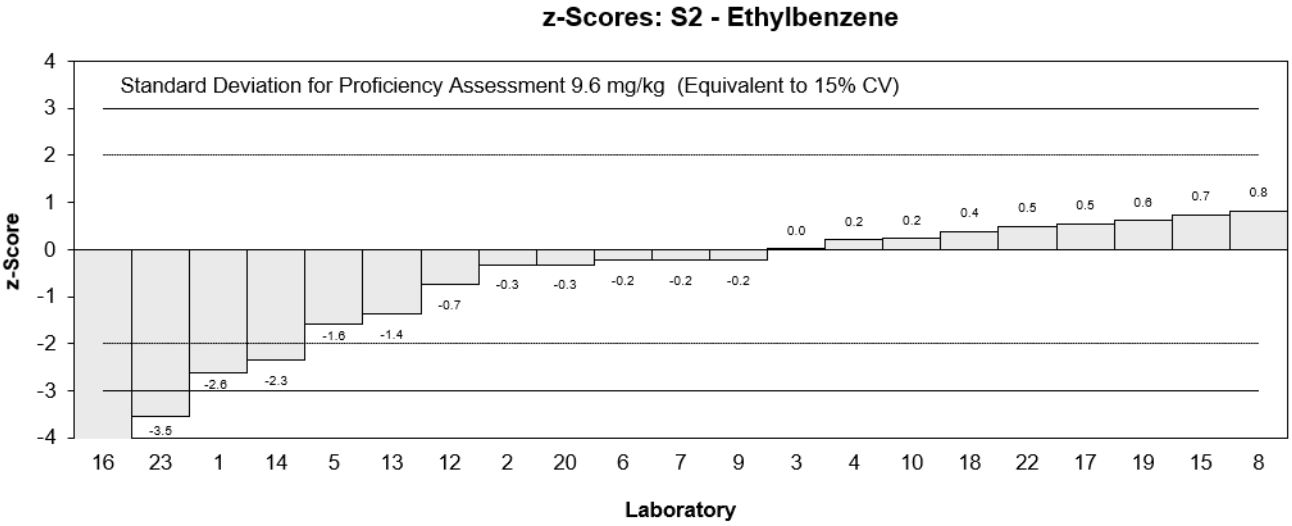
Lab. Code	Result	Uncertainty	z	E_n
1**	39	12	-2.61	-2.00
2	61	10	-0.32	-0.29
3	64.2	12.5	0.01	0.01
4	66	20	0.20	0.09
5	49	15	-1.57	-0.98
6	62	15	-0.22	-0.14
7	62	19	-0.22	-0.11
8	72	14	0.82	0.55
9	62	15	-0.22	-0.14
10	66.30	19.9	0.23	0.11
11	NT	NT		
12	57	17	-0.74	-0.41
13	51	15.3	-1.36	-0.83
14**	41.6	3.2	-2.34	-4.60
15	71.2	13	0.74	0.53
16**	22.9	6.87	-4.28	-5.28
17	69.3	9.5	0.54	0.51
18	67.8	12.7	0.38	0.28
19	70	22	0.61	0.26
20	61	10	-0.32	-0.29
21	NT	NT		
22	68.84	5.3	0.49	0.73
23**	30.2	12	-3.53	-2.70

** Excluded Result, see Section 6.1

Statistics

Assigned Value	64.1	3.7
Robust Average	64.1	3.7
Median	64.2	3.2
Mean	63.6	
N	17	
Max	72	
Min	49	
Robust SD	6.1	
Robust CV	9.6%	


Kernel Density
Density
Result
Results: S2 - Ethylbenzene
Assigned Value = 64.1 ± 3.7 mg/kg
Results (mg/kg)
Laboratory
16 23 1 14 5 13 12 2 20 6 7 9 3 4 10 18 22 17 19 15 8 11 21 Md RA


z-Scores: S2 - Ethylbenzene
Standard Deviation for Proficiency Assessment 9.6 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
16 23 1 14 5 13 12 2 20 6 7 9 3 4 10 18 22 17 19 15 8
-3.5 -2.6 -2.3 -1.6 -1.4 -0.7 -0.3 -0.3 -0.2 -0.2 -0.2 0.0 0.2 0.2 0.4 0.5 0.5 0.6 0.7 0.8

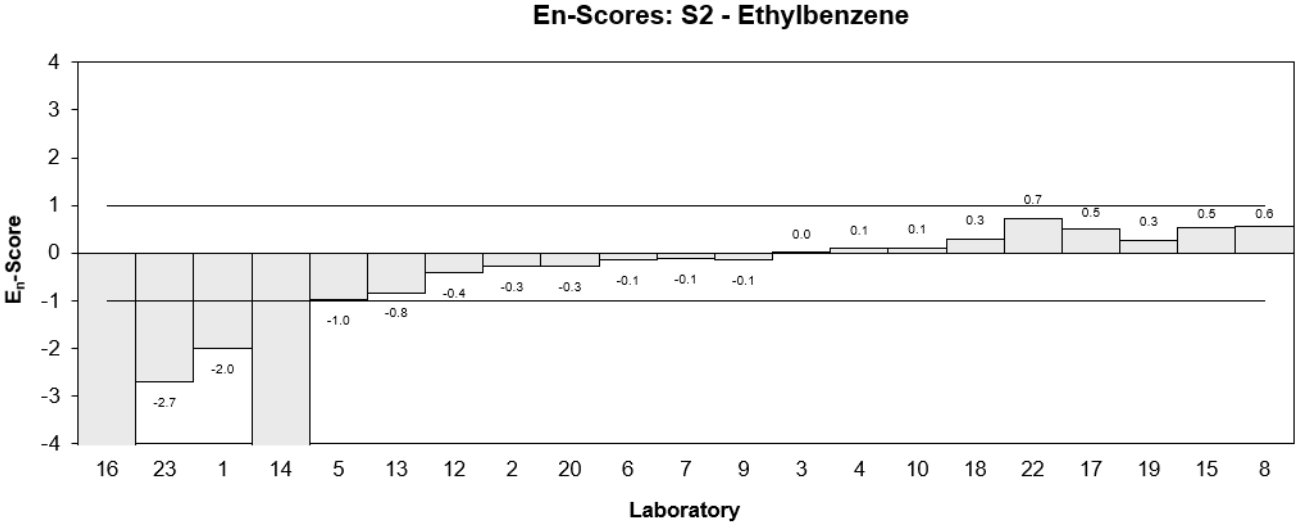

En-Scores: S2 - Ethylbenzene
En-Score
Laboratory
16 23 1 14 5 13 12 2 20 6 7 9 3 4 10 18 22 17 19 15 8
-2.7 -2.0 -1.0 -0.8 -0.4 -0.3 -0.3 -0.1 -0.1 -0.1 0.0 0.1 0.1 0.3 0.7 0.5 0.3 0.5 0.6

Figure 9

Table 14

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	Xylenes
Unit	mg/kg

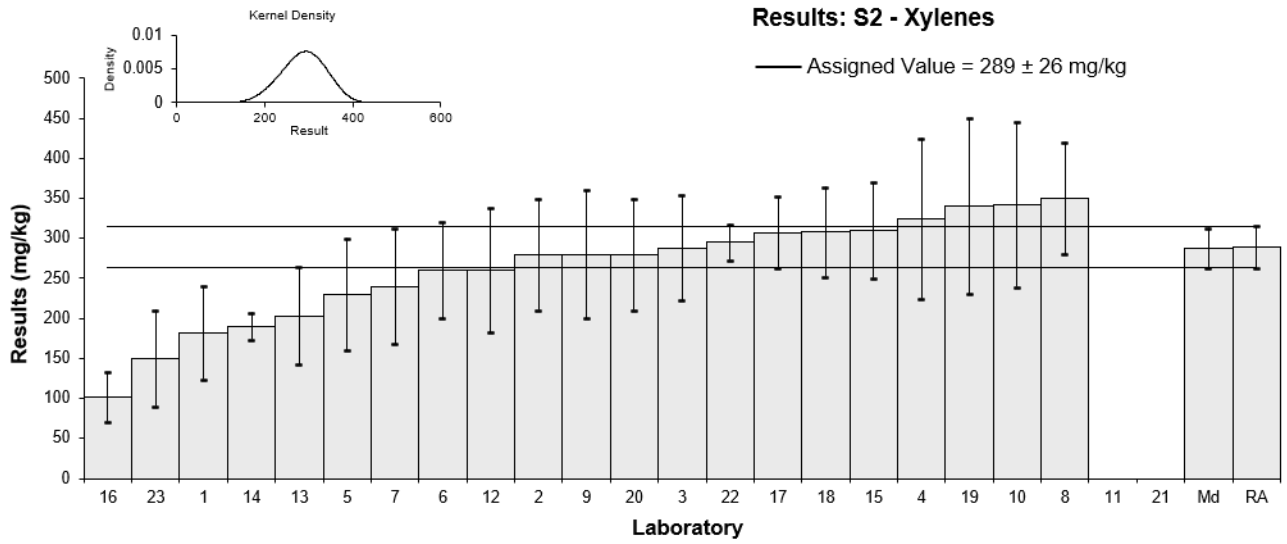
Participant Results

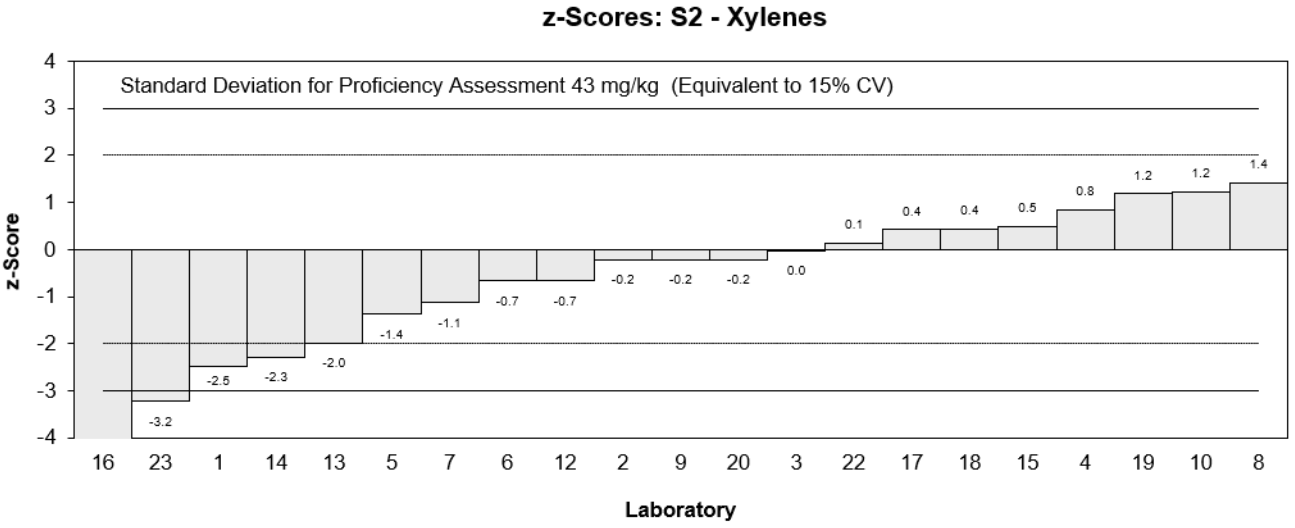
Lab. Code	Result	Uncertainty	z	E_n
1**	182	58	-2.47	-1.68
2	280	70	-0.21	-0.12
3	288	66	-0.02	-0.01
4	325	100	0.83	0.35
5	230	70	-1.36	-0.79
6	260	60	-0.67	-0.44
7	240	72	-1.13	-0.64
8	350	70	1.41	0.82
9	280	80	-0.21	-0.11
10	342.00	102.6	1.22	0.50
11	NT	NT		
12	260	78	-0.67	-0.35
13	203	60.9	-1.98	-1.30
14**	190.3	16.5	-2.28	-3.21
15	310	60	0.48	0.32
16**	101.8	30.54	-4.32	-4.67
17	307	45	0.42	0.35
18	308	56.3	0.44	0.31
19	340	110	1.18	0.45
20	280	70	-0.21	-0.12
21	NT	NT		
22	295.08	22.5	0.14	0.18
23**	150	60	-3.21	-2.13

** Excluded Result, see Section 6.1

Statistics

Assigned Value	289	26
Robust Average	289	26
Median	288	25
Mean	288	
N	17	
Max	350	
Min	203	
Robust SD	44	
Robust CV	15%	


Kernel Density
Density
Result
Results: S2 - Xylenes
Assigned Value = 289 ± 26 mg/kg
Results (mg/kg)
Laboratory
16 23 1 14 13 5 7 6 12 2 9 20 3 22 17 18 15 4 19 10 8 11 21 Md RA


z-Scores: S2 - Xylenes
Standard Deviation for Proficiency Assessment 43 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
16 23 1 14 13 5 7 6 12 2 9 20 3 22 17 18 15 4 19 10 8
-3.2 -2.5 -2.3 -2.0 -1.4 -1.1 -0.7 -0.7 -0.2 -0.2 -0.2 0.0 0.1 0.4 0.4 0.5 0.8 1.2 1.2 1.4

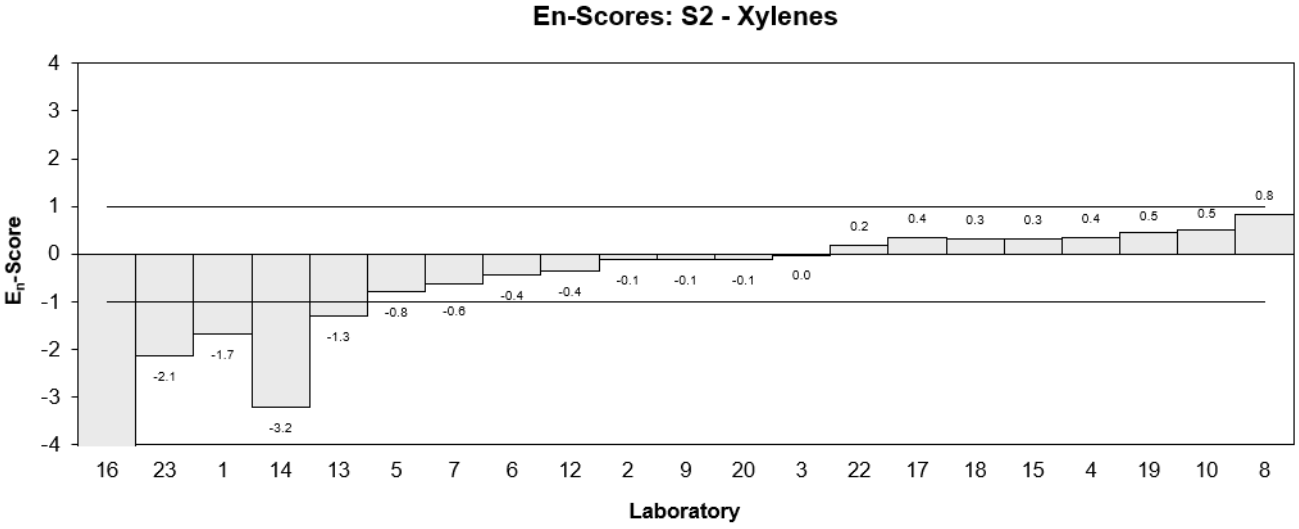

En-Scores: S2 - Xylenes
En-Score
Laboratory
16 23 1 14 13 5 7 6 12 2 9 20 3 22 17 18 15 4 19 10 8
-2.1 -1.7 -3.2 -1.3 -0.8 -0.6 -0.4 -0.4 -0.1 -0.1 -0.1 0.0 0.2 0.4 0.3 0.3 0.4 0.5 0.5 0.8

Figure 10

Table 15

Sample Details

Sample No.	S2
Matrix	Soil
Analyte	Total BTEX
Unit	mg/kg

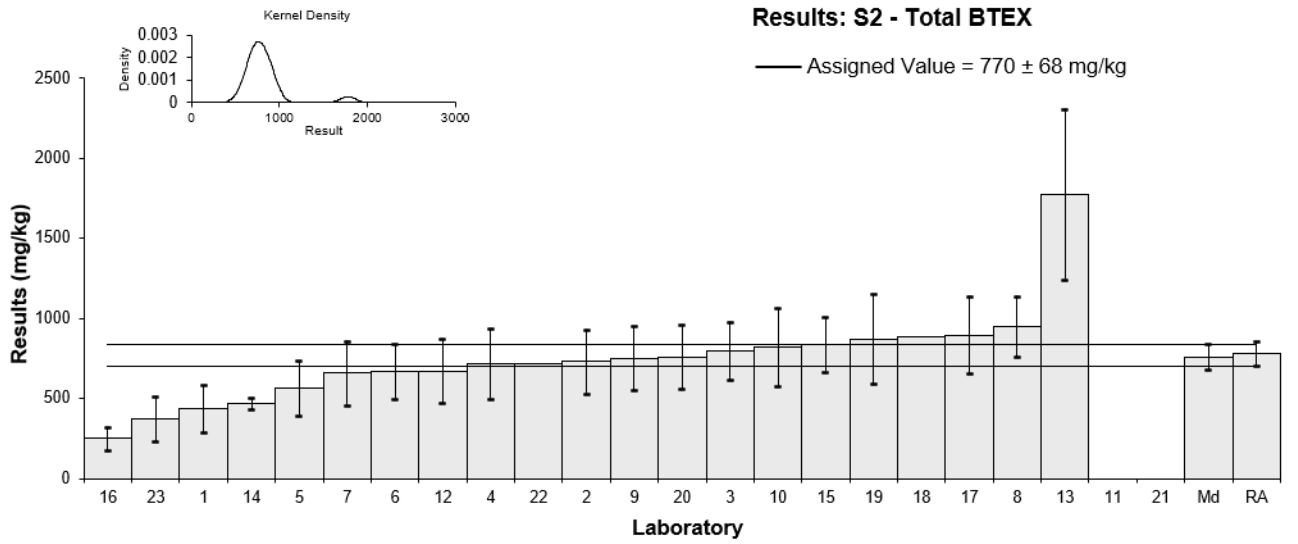
Participant Results

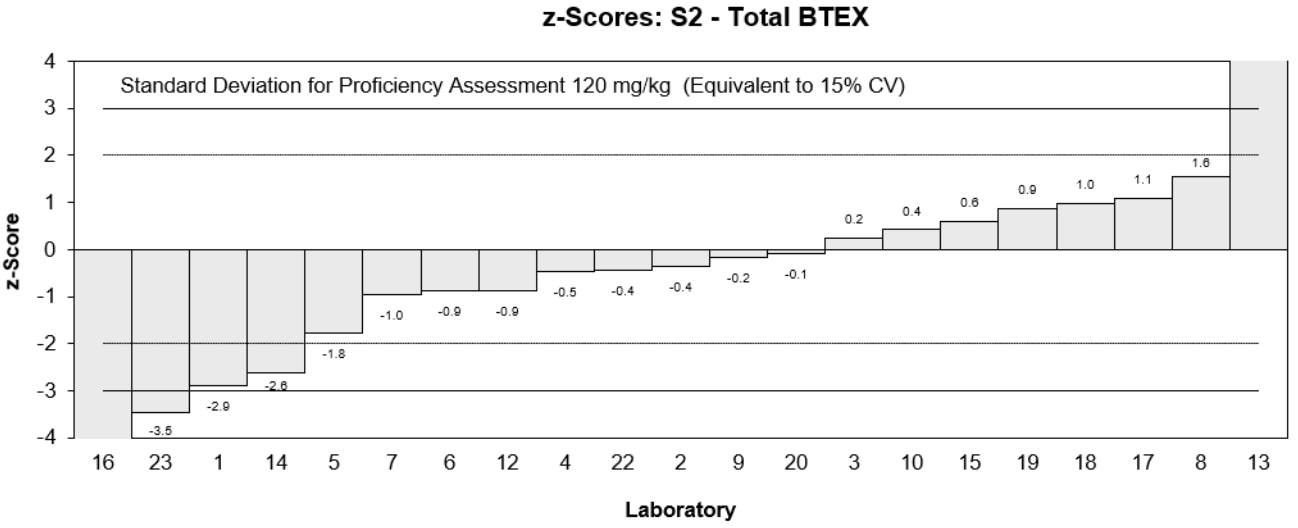
Lab. Code	Result	Uncertainty	z	E_n
1**	436	148	-2.89	-2.05
2	730	200	-0.35	-0.19
3	798	183	0.24	0.14
4	717	220	-0.46	-0.23
5	564	170	-1.78	-1.13
6	670	170	-0.87	-0.55
7	660	200	-0.95	-0.52
8	950	190	1.56	0.89
9	750	200	-0.17	-0.09
10	818.70	245.6	0.42	0.19
11	NT	NT		
12	670	200	-0.87	-0.47
13*	1777.6	533.28	8.72	1.87
14**	467	37	-2.62	-3.91
15	838	170	0.59	0.37
16**	249.58	74.874	-4.51	-5.15
17	896	243	1.09	0.50
18	882	NR	0.97	1.65
19	870	280	0.87	0.35
20	760	200	-0.09	-0.05
21	NT	NT		
22	720.51	NR	-0.43	-0.73
23**	370	140	-3.46	-2.57

* Outlier, see Section 4.2; ** Excluded Result, see Section 6.1

Statistics

Assigned Value	770	68
Robust Average	782	73
Median	760	81
Mean	830	
N	17	
Max	1777.6	
Min	564	
Robust SD	120	
Robust CV	15%	


Kernel Density
Density
Result
Results: S2 - Total BTEX
Assigned Value = 770 ± 68 mg/kg
Results (mg/kg)
Laboratory
16 23 1 14 5 7 6 12 4 22 2 9 20 3 10 15 19 18 17 8 13 11 21 Md RA


z-Scores: S2 - Total BTEX
Standard Deviation for Proficiency Assessment 120 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
16 23 1 14 5 7 6 12 4 22 2 9 20 3 10 15 19 18 17 8 13

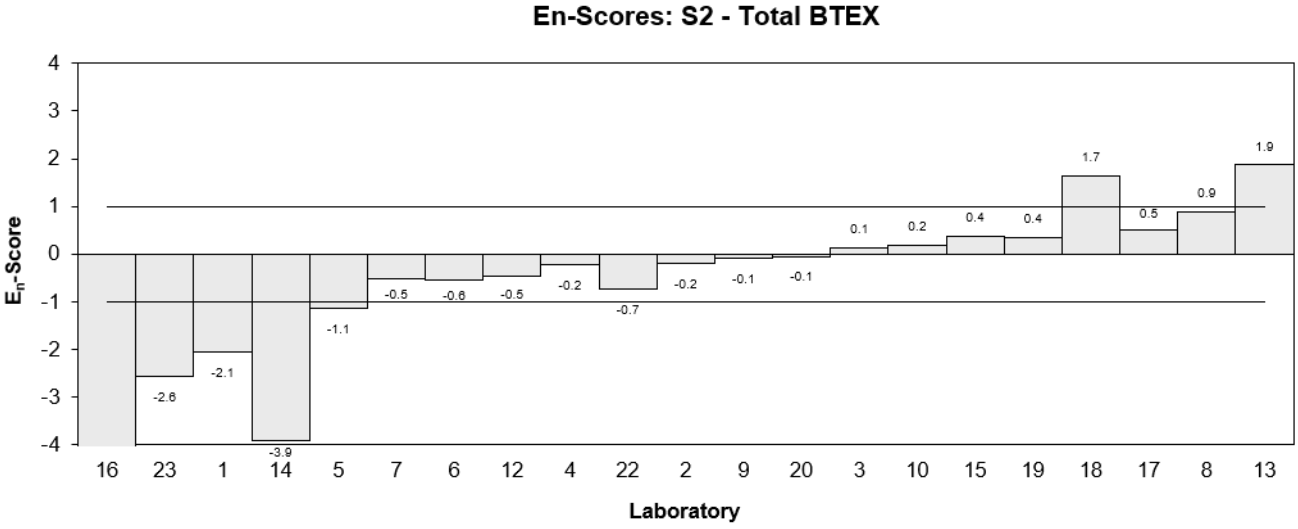

En-Scores: S2 - Total BTEX
En-Score
Laboratory
16 23 1 14 5 7 6 12 4 22 2 9 20 3 10 15 19 18 17 8 13

Figure 11

Table 16

Sample Details

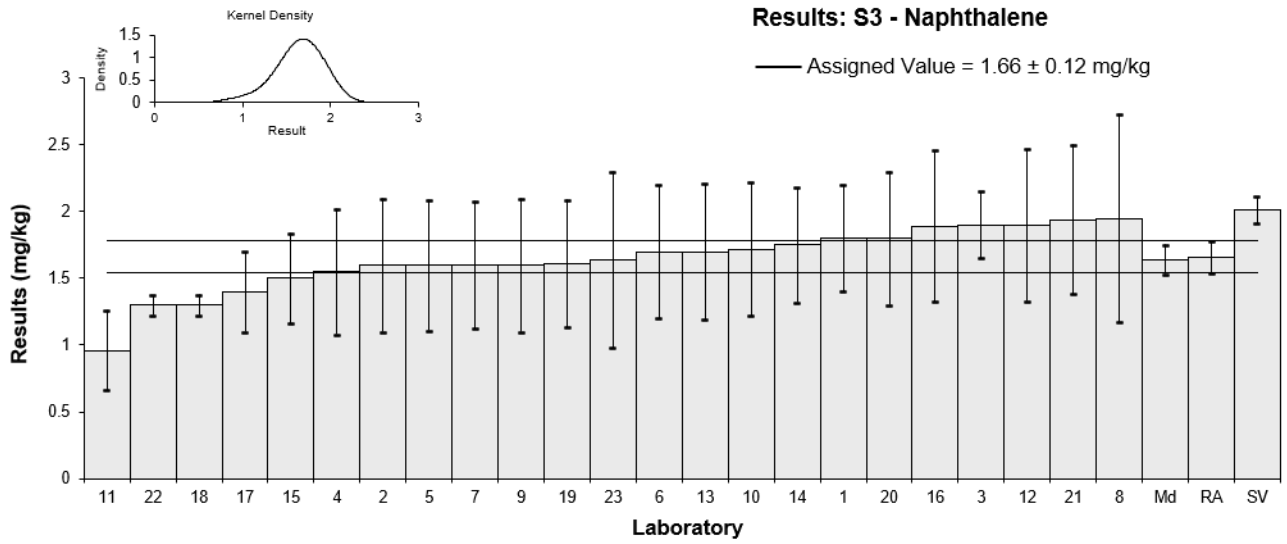
Sample No.	S3
Matrix	Soil
Analyte	Naphthalene
Unit	mg/kg

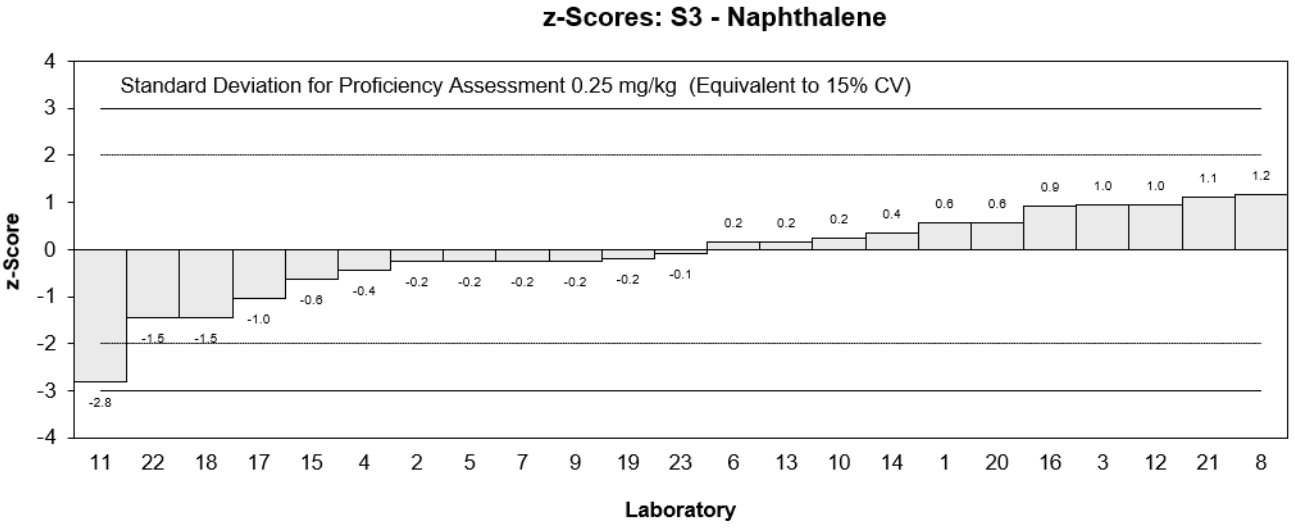
Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	1.8	0.4	0.56	0.34
2	1.6	0.5	-0.24	-0.12
3	1.9	0.25	0.96	0.87
4	1.55	0.47	-0.44	-0.23
5	1.6	0.49	-0.24	-0.12
6	1.7	0.5	0.16	0.08
7	1.6	0.48	-0.24	-0.12
8	1.95	0.78	1.16	0.37
9	1.6	0.5	-0.24	-0.12
10	1.72	0.5	0.24	0.12
11	0.96	0.3	-2.81	-2.17
12	1.9	0.57	0.96	0.41
13	1.7	0.51	0.16	0.08
14	1.75	0.43	0.36	0.20
15	1.5	0.34	-0.64	-0.44
16	1.89	0.567	0.92	0.40
17	1.4	0.3	-1.04	-0.80
18	1.3	0.078	-1.45	-2.52
19	1.61	0.48	-0.20	-0.10
20	1.8	0.5	0.56	0.27
21	1.94	0.56	1.12	0.49
22	1.298	0.075	-1.45	-2.56
23	1.64	0.66	-0.08	-0.03

Statistics

Assigned Value	1.66	0.12
Spike Value	2.01	0.10
Robust Average	1.66	0.12
Median	1.64	0.11
Mean	1.64	
N	23	
Max	1.95	
Min	0.96	
Robust SD	0.22	
Robust CV	13%	


Kernel Density
Density
Result
Results: S3 - Naphthalene
Assigned Value = 1.66 ± 0.12 mg/kg
Results (mg/kg)
11 22 18 17 15 4 2 5 7 9 19 23 6 13 10 14 1 20 16 3 12 21 8 Md RA SV
Laboratory


z-Scores: S3 - Naphthalene
Standard Deviation for Proficiency Assessment 0.25 mg/kg (Equivalent to 15% CV)
z-Score
-2.8 -1.5 -1.5 -1.0 -0.6 -0.4 -0.2 -0.2 -0.2 -0.2 -0.2 -0.1 0.2 0.2 0.2 0.4 0.6 0.6 0.9 1.0 1.0 1.1 1.2
11 22 18 17 15 4 2 5 7 9 19 23 6 13 10 14 1 20 16 3 12 21 8
Laboratory

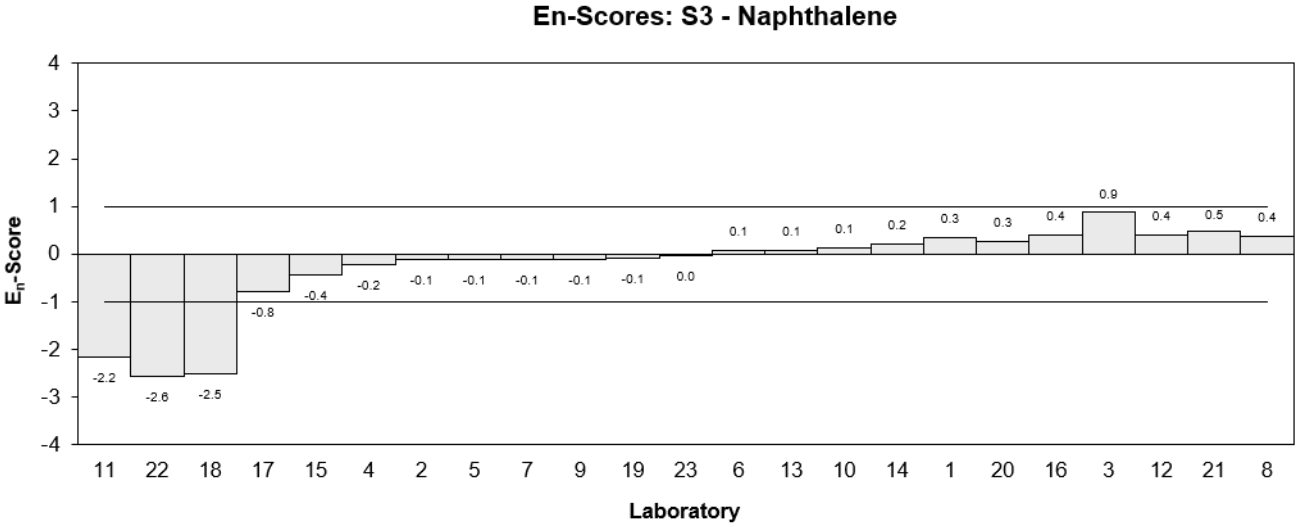

En-Scores: S3 - Naphthalene
En-Score
-2.2 -2.6 -2.5 -0.8 -0.4 -0.2 -0.1 -0.1 -0.1 -0.1 -0.1 0.0 0.1 0.1 0.1 0.2 0.3 0.3 0.4 0.9 0.4 0.5 0.4
11 22 18 17 15 4 2 5 7 9 19 23 6 13 10 14 1 20 16 3 12 21 8
Laboratory

Figure 12

Table 17

Sample Details

Sample No.	S3
Matrix	Soil
Analyte	Acenaphthylene
Unit	mg/kg

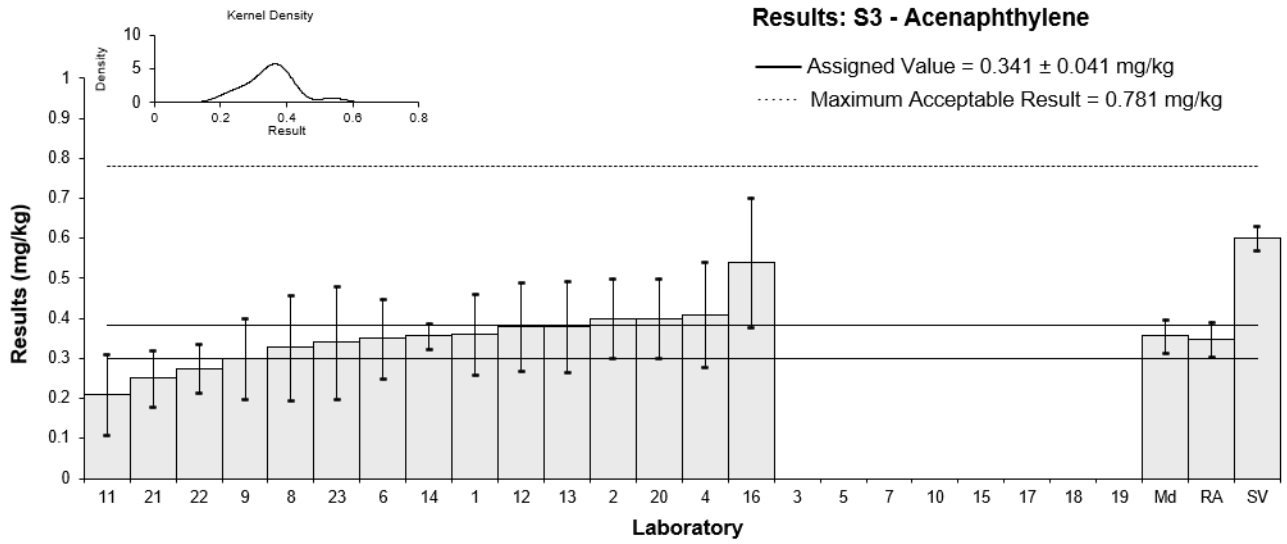
Participant Results

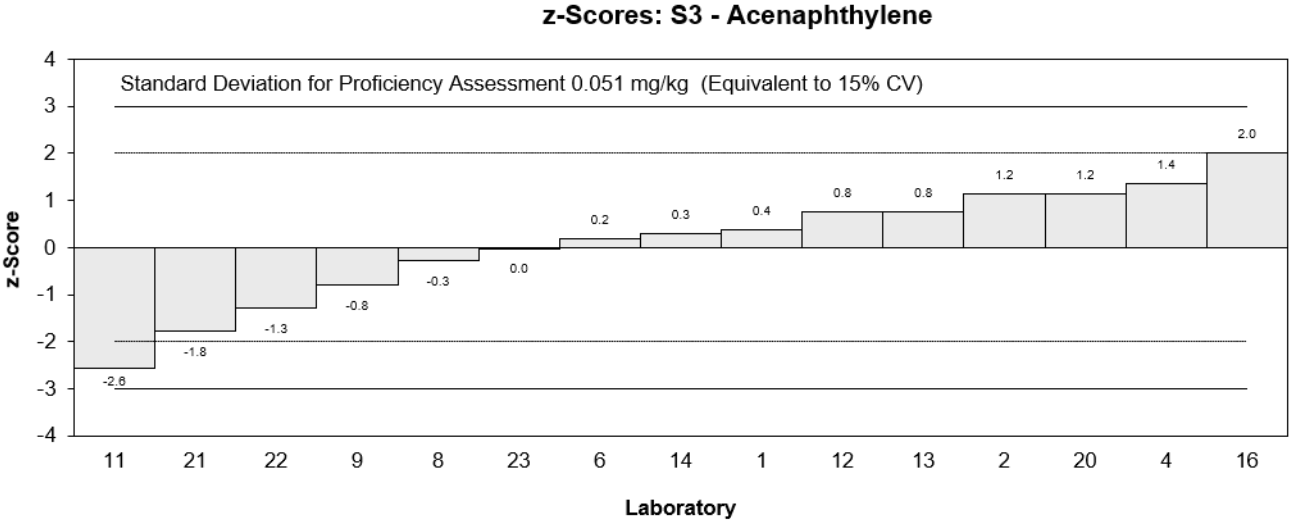
Lab. Code	Result	Uncertainty	z	E_n
1	0.36	0.1	0.37	0.18
2	0.4	0.1	1.15	0.55
3	<0.5	NR		
4	0.41	0.13	1.35	0.51
5	<0.5	NR		
6	0.35	0.1	0.18	0.08
7	<0.5	NR		
8	0.327	0.13	-0.27	-0.10
9	0.3	0.1	-0.80	-0.38
10	< 0.5	NR		
11	0.21	0.1	-2.56	-1.21
12	0.38	0.11	0.76	0.33
13	0.38	0.114	0.76	0.32
14	0.356	0.033	0.29	0.29
15	<0.5	0.15		
16*	0.540	0.162	2.00▼	
17	<0.5	NR		
18	<0.5	NR		
19	< 0.5	NR		
20	0.4	0.1	1.15	0.55
21	0.25	0.07	-1.78	-1.12
22	0.275	0.061	-1.29	-0.90
23	0.34	0.14	-0.02	-0.01

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

Statistics

Assigned Value	0.341	0.041
Spike Value	0.601	0.030
Robust Average	0.348	0.044
Max Acceptable Result	0.781	
Median	0.356	0.042
Mean	0.352	
N	15	
Max	0.540	
Min	0.21	
Robust SD	0.068	
Robust CV	19%	


Kernel Density
Density
Result
Results: S3 - Acenaphthylene
Assigned Value = 0.341 ± 0.041 mg/kg
Maximum Acceptable Result = 0.781 mg/kg
Results (mg/kg)
Laboratory
11 21 22 9 8 23 6 14 1 12 13 2 20 4 16 3 5 7 10 15 17 18 19 Md RA SV


z-Scores: S3 - Acenaphthylene
Standard Deviation for Proficiency Assessment 0.051 mg/kg (Equivalent to 15% CV)
z-Score
-2.6 -1.8 -1.3 -0.8 -0.3 0.0 0.2 0.3 0.4 0.8 0.8 1.2 1.2 1.4 2.0
Laboratory
11 21 22 9 8 23 6 14 1 12 13 2 20 4 16

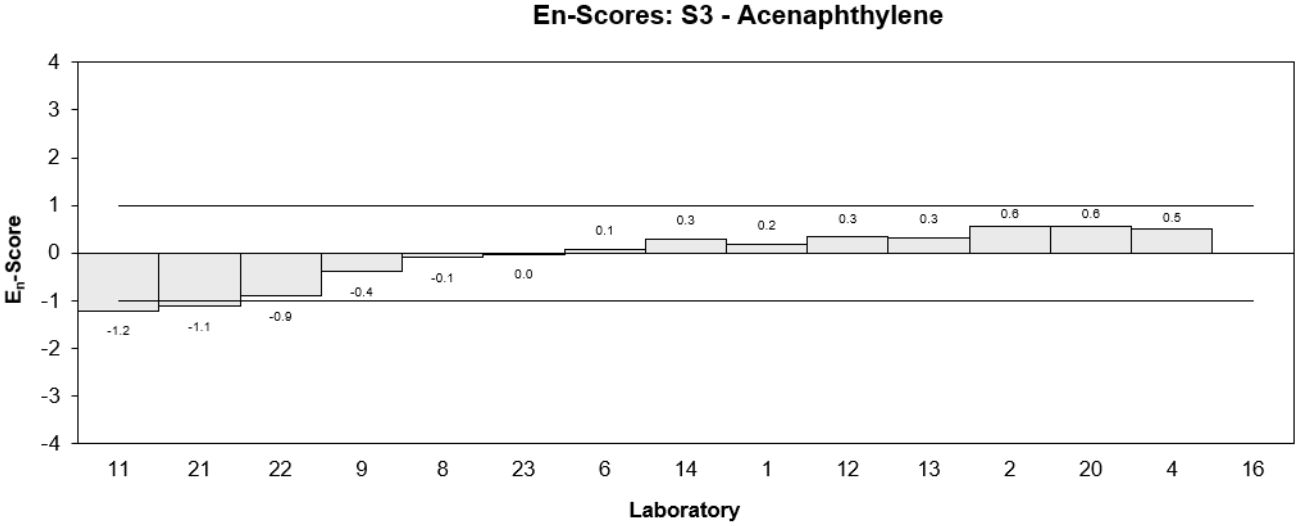

En-Scores: S3 - Acenaphthylene
En-Score
-1.2 -1.1 -0.9 -0.4 -0.1 0.0 0.1 0.3 0.2 0.3 0.3 0.6 0.6 0.5
Laboratory
11 21 22 9 8 23 6 14 1 12 13 2 20 4 16

Figure 13

Table 18

Sample Details

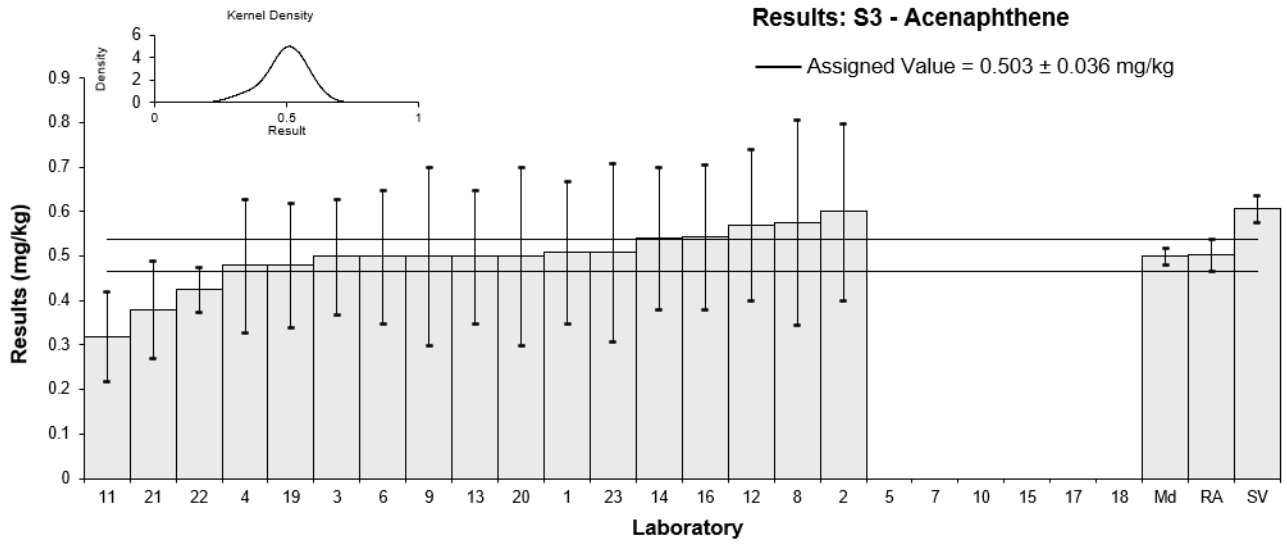
Sample No.	S3
Matrix	Soil
Analyte	Acenaphthene
Unit	mg/kg

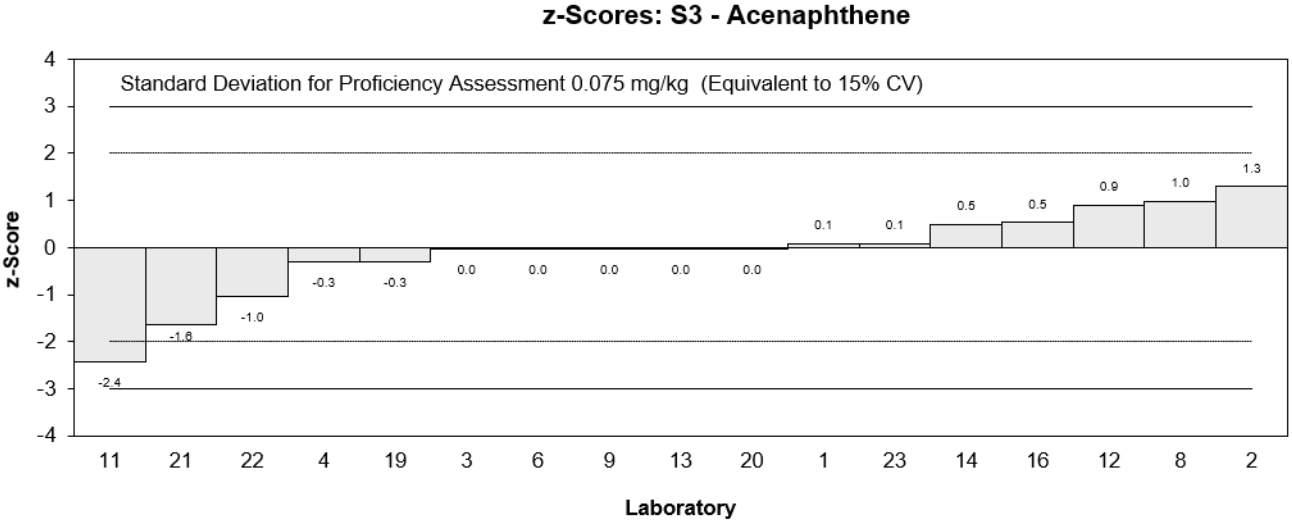
Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	0.51	0.16	0.09	0.04
2	0.6	0.2	1.29	0.48
3	0.5	0.13	-0.04	-0.02
4	0.48	0.15	-0.30	-0.15
5	<0.5	NR		
6	0.50	0.15	-0.04	-0.02
7	<0.5	NR		
8	0.576	0.23	0.97	0.31
9	0.5	0.2	-0.04	-0.01
10	< 0.5	NR		
11	0.32	0.1	-2.43	-1.72
12	0.57	0.17	0.89	0.39
13	0.5	0.15	-0.04	-0.02
14	0.54	0.16	0.49	0.23
15	<0.5	0.12		
16	0.543	0.1629	0.53	0.24
17	<0.5	NR		
18	<0.5	NR		
19	0.48	0.14	-0.30	-0.16
20	0.5	0.2	-0.04	-0.01
21	0.38	0.11	-1.63	-1.06
22	0.425	0.05	-1.03	-1.27
23	0.51	0.2	0.09	0.03

Statistics

Assigned Value	0.503	0.036
Spike Value	0.606	0.030
Robust Average	0.503	0.036
Median	0.500	0.018
Mean	0.496	
N	17	
Max	0.6	
Min	0.32	
Robust SD	0.059	
Robust CV	12%	


Kernel Density
Density
Result
Results: S3 - Acenaphthene
Assigned Value = 0.503 ± 0.036 mg/kg
Results (mg/kg)
Laboratory
11 21 22 4 19 3 6 9 13 20 1 23 14 16 12 8 2 5 7 10 15 17 18 Md RA SV


z-Scores: S3 - Acenaphthene
Standard Deviation for Proficiency Assessment 0.075 mg/kg (Equivalent to 15% CV)
z-Score
-2.4 -1.6 -1.0 -0.3 -0.3 0.0 0.0 0.0 0.0 0.0 0.1 0.1 0.5 0.5 0.9 1.0 1.3
Laboratory
11 21 22 4 19 3 6 9 13 20 1 23 14 16 12 8 2

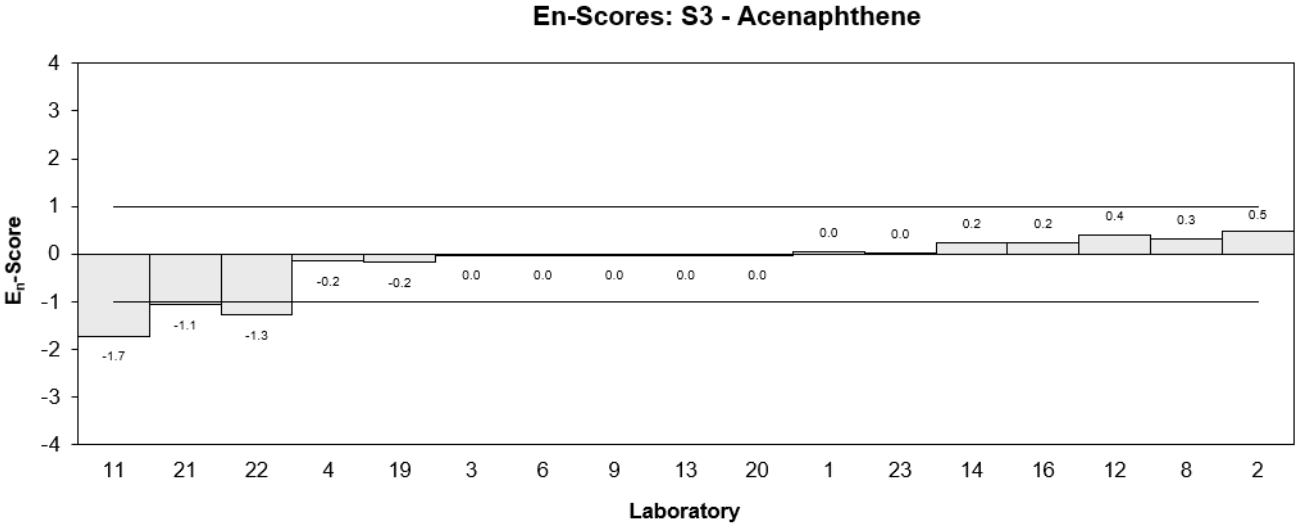

En-Scores: S3 - Acenaphthene
En-Score
-1.7 -1.1 -1.3 -0.2 -0.2 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.2 0.2 0.4 0.3 0.5
Laboratory
11 21 22 4 19 3 6 9 13 20 1 23 14 16 12 8 2

Figure 14

Table 19

Sample Details

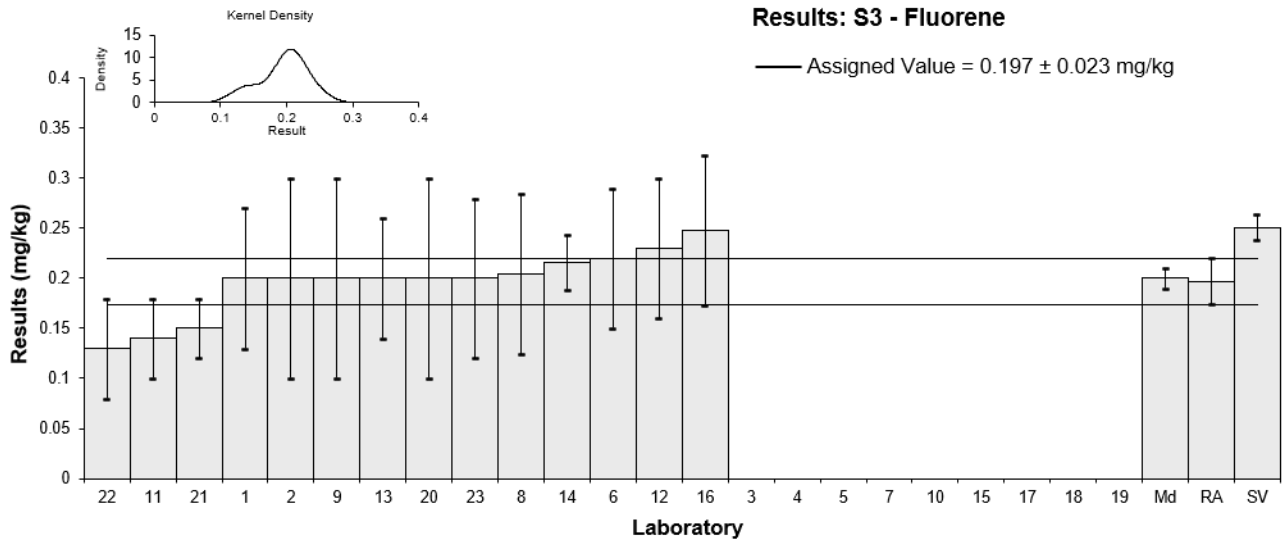
Sample No.	S3
Matrix	Soil
Analyte	Fluorene
Unit	mg/kg

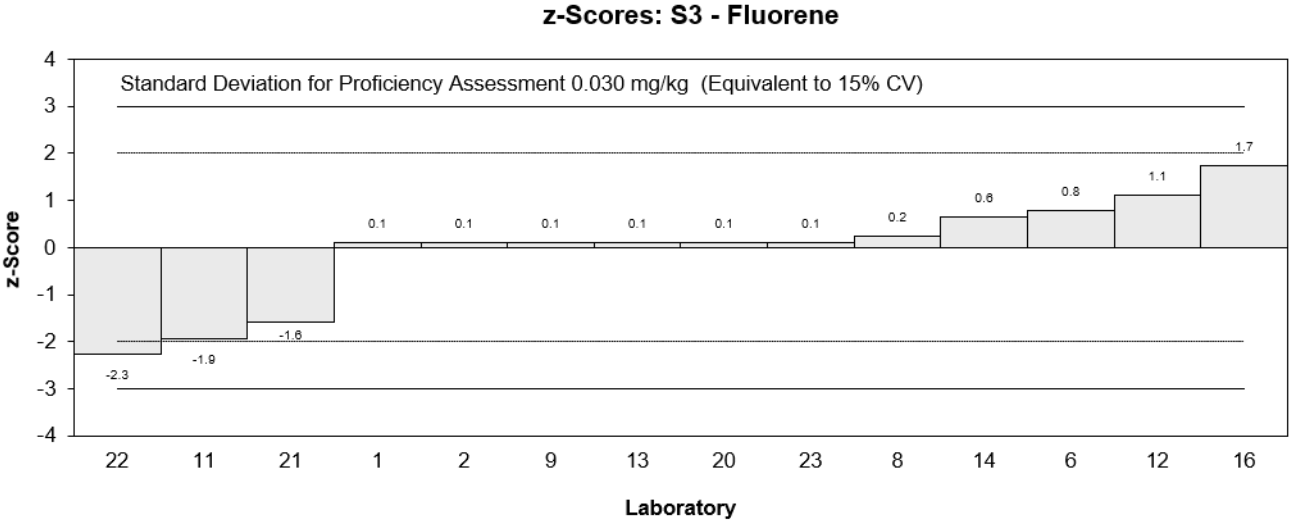
Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	0.2	0.07	0.10	0.04
2	0.2	0.1	0.10	0.03
3	<0.5	NR		
4	< 0.5	NR		
5	<0.5	NR		
6	0.22	0.07	0.78	0.31
7	<0.5	NR		
8	0.204	0.08	0.24	0.08
9	0.2	0.1	0.10	0.03
10	< 0.5	NR		
11	0.14	0.04	-1.93	-1.24
12	0.23	0.07	1.12	0.45
13	0.2	0.06	0.10	0.05
14	0.216	0.027	0.64	0.54
15	<0.5	0.12		
16	0.248	0.0744	1.73	0.65
17	<0.5	NR		
18	<0.5	NR		
19	< 0.5	NR		
20	0.2	0.1	0.10	0.03
21	0.15	0.03	-1.59	-1.24
22	0.13	0.05	-2.27	-1.22
23	0.2	0.08	0.10	0.04

Statistics

Assigned Value	0.197	0.023
Spike Value	0.251	0.013
Robust Average	0.197	0.023
Median	0.200	0.010
Mean	0.196	
N	14	
Max	0.248	
Min	0.13	
Robust SD	0.035	
Robust CV	18%	


Kernel Density
Density
Result
Results: S3 - Fluorene
Assigned Value = 0.197 ± 0.023 mg/kg
Results (mg/kg)
Laboratory
22 11 21 1 2 9 13 20 23 8 14 6 12 16 3 4 5 7 10 15 17 18 19 Md RA SV


z-Scores: S3 - Fluorene
Standard Deviation for Proficiency Assessment 0.030 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
22 11 21 1 2 9 13 20 23 8 14 6 12 16
-2.3
-1.9
-1.6
0.1
0.2
0.6
0.8
1.1
1.7

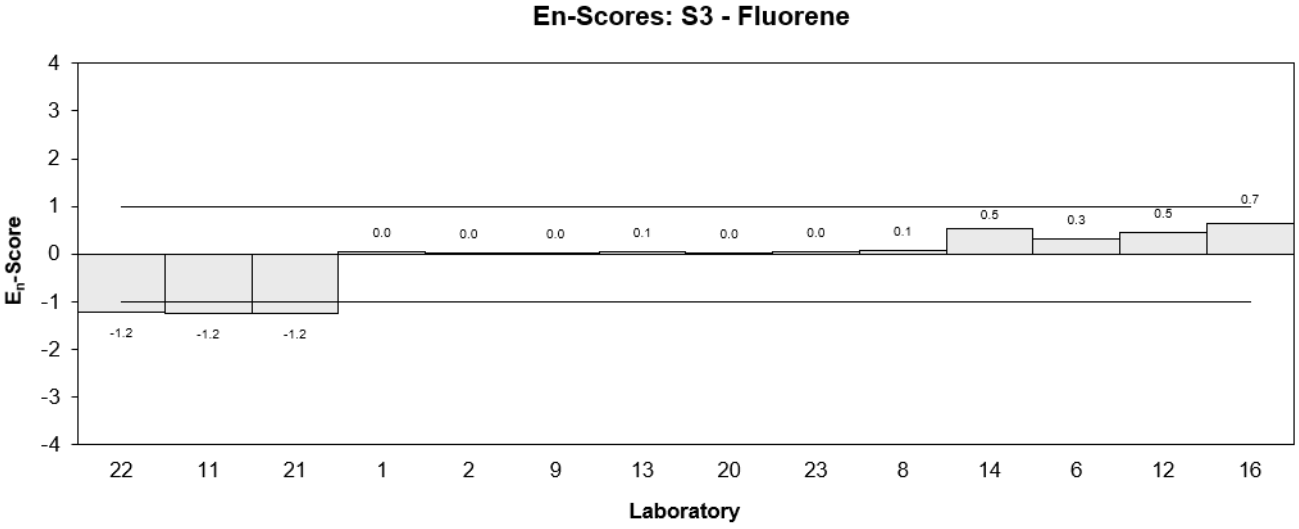

En-Scores: S3 - Fluorene
En-Score
Laboratory
22 11 21 1 2 9 13 20 23 8 14 6 12 16
-1.2
0.0
0.1
0.5
0.3
0.7

Figure 15

Table 20

Sample Details

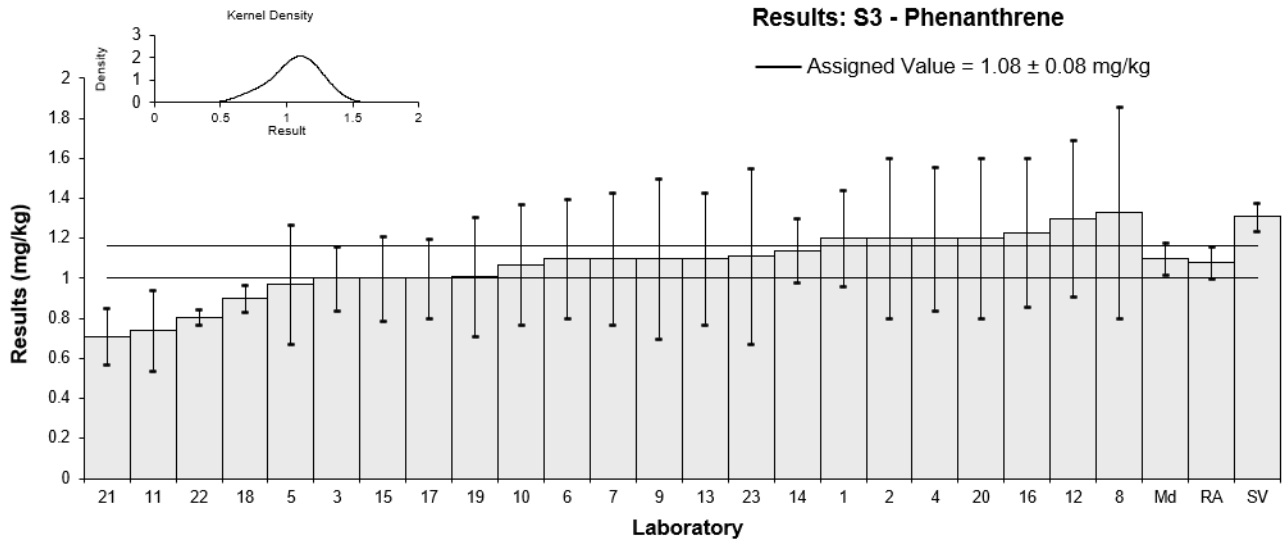
Sample No.	S3
Matrix	Soil
Analyte	Phenanthrene
Unit	mg/kg

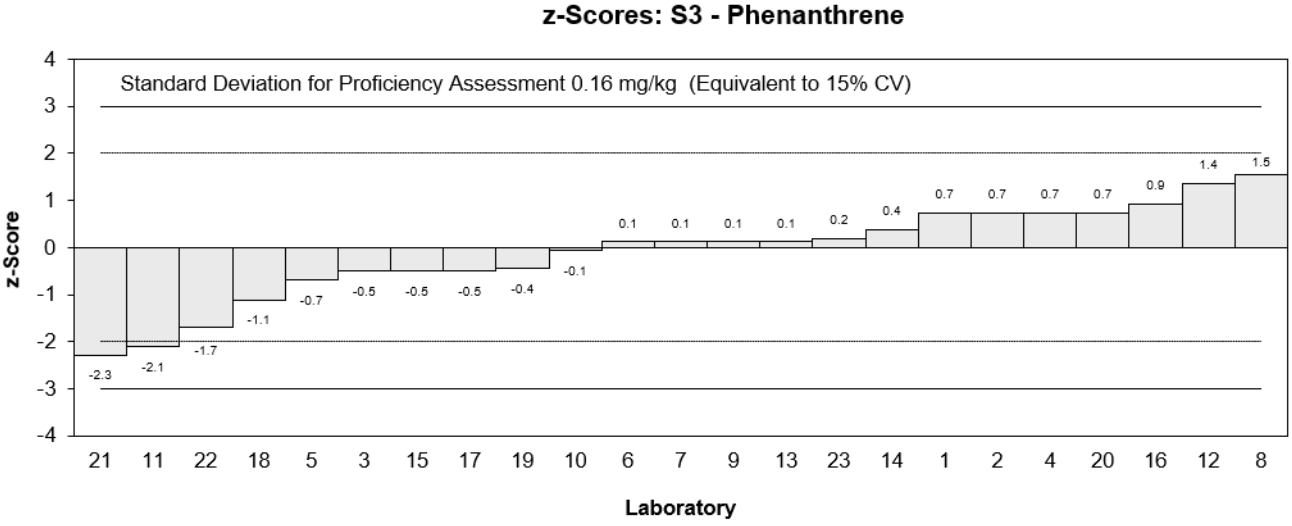
Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	1.2	0.24	0.74	0.47
2	1.2	0.4	0.74	0.29
3	1.0	0.16	-0.49	-0.45
4	1.2	0.36	0.74	0.33
5	0.97	0.3	-0.68	-0.35
6	1.1	0.3	0.12	0.06
7	1.1	0.33	0.12	0.06
8	1.33	0.53	1.54	0.47
9	1.1	0.4	0.12	0.05
10	1.07	0.3	-0.06	-0.03
11	0.74	0.2	-2.10	-1.58
12	1.3	0.39	1.36	0.55
13	1.1	0.33	0.12	0.06
14	1.14	0.16	0.37	0.34
15	1.0	0.21	-0.49	-0.36
16	1.23	0.369	0.93	0.40
17	1.0	0.2	-0.49	-0.37
18	0.9	0.069	-1.11	-1.70
19	1.01	0.30	-0.43	-0.23
20	1.2	0.4	0.74	0.29
21	0.71	0.14	-2.28	-2.29
22	0.806	0.037	-1.69	-3.11
23	1.11	0.44	0.19	0.07

Statistics

Assigned Value	1.08	0.08
Spike Value	1.31	0.07
Robust Average	1.08	0.08
Median	1.10	0.08
Mean	1.07	
N	23	
Max	1.33	
Min	0.71	
Robust SD	0.16	
Robust CV	15%	


Kernel Density
Density
Result
Results: S3 - Phenanthrene
Assigned Value = 1.08 ± 0.08 mg/kg
Results (mg/kg)
21 11 22 18 5 3 15 17 19 10 6 7 9 13 23 14 1 2 4 20 16 12 8 Md RA SV
Laboratory


z-Scores: S3 - Phenanthrene
Standard Deviation for Proficiency Assessment 0.16 mg/kg (Equivalent to 15% CV)
z-Score
-2.3 -2.1 -1.7 -1.1 -0.7 -0.5 -0.5 -0.5 -0.4 -0.1 0.1 0.1 0.1 0.1 0.2 0.4 0.7 0.7 0.7 0.7 0.9 1.4 1.5
21 11 22 18 5 3 15 17 19 10 6 7 9 13 23 14 1 2 4 20 16 12 8
Laboratory

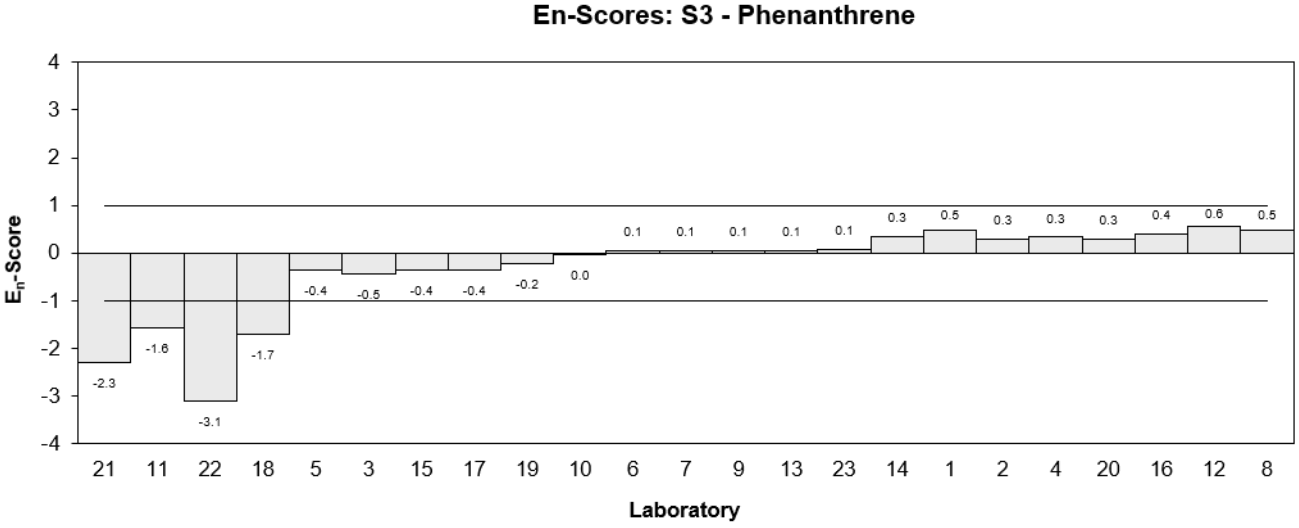

En-Scores: S3 - Phenanthrene
En-Score
-2.3 -1.6 -3.1 -1.7 -0.4 -0.5 -0.4 -0.4 -0.2 0.0 0.1 0.1 0.1 0.1 0.1 0.3 0.5 0.3 0.3 0.3 0.4 0.6 0.5
21 11 22 18 5 3 15 17 19 10 6 7 9 13 23 14 1 2 4 20 16 12 8
Laboratory

Figure 16

Table 21

Sample Details

Sample No.	S3
Matrix	Soil
Analyte	Anthracene
Unit	mg/kg

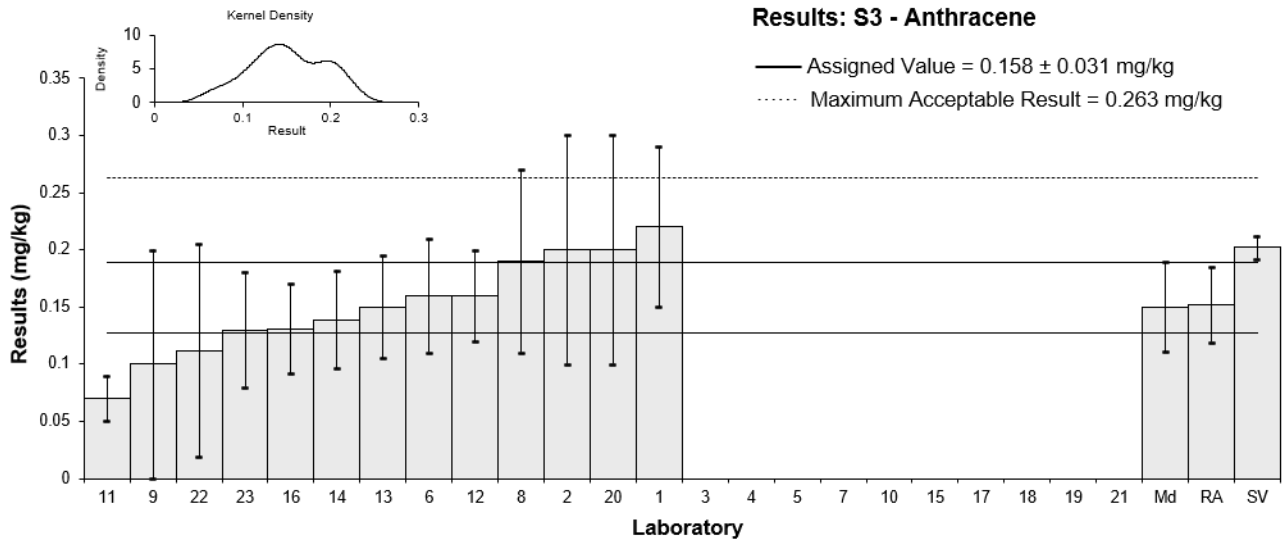
Participant Results

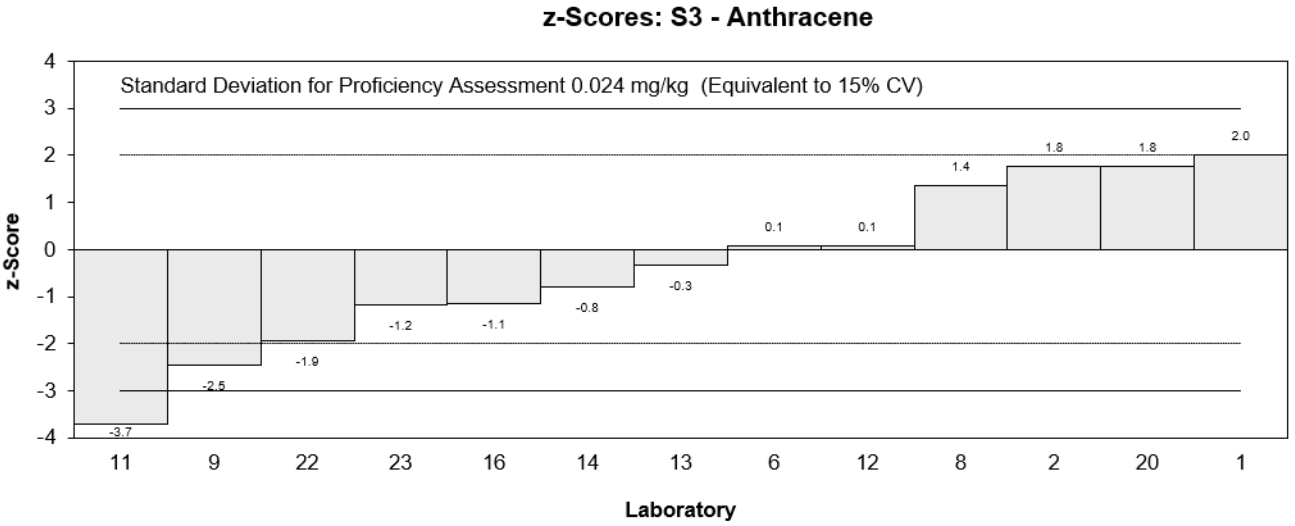
Lab. Code	Result	Uncertainty	z	E_n
1	0.22	0.07	2.00▼	
2	0.2	0.1	1.77	0.40
3	<0.5	NR		
4	< 0.5	NR		
5	<0.5	NR		
6	0.16	0.05	0.08	0.03
7	<0.5	NR		
8	0.19	0.08	1.35	0.37
9	0.1	0.1	-2.45	-0.55
10	< 0.5	NR		
11*	0.07	0.02	-3.71	-2.39
12	0.16	0.04	0.08	0.04
13	0.15	0.045	-0.34	-0.15
14	0.139	0.043	-0.80	-0.36
15	<0.5	0.13		
16	0.131	0.0393	-1.14	-0.54
17	<0.5	NR		
18	<0.5	NR		
19	< 0.5	NR		
20	0.2	0.1	1.77	0.40
21	<0.05	NR		
22	0.112	0.093	-1.94	-0.47
23	0.13	0.05	-1.18	-0.48

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

Statistics

Assigned Value	0.158	0.031
Spike Value	0.202	0.010
Robust Average	0.152	0.033
Max Acceptable Result	0.263	
Median	0.150	0.039
Mean	0.151	
N	13	
Max	0.22	
Min	0.07	
Robust SD	0.048	
Robust CV	32%	


Results: S3 - Anthracene
Assigned Value = 0.158 ± 0.031 mg/kg
Maximum Acceptable Result = 0.263 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
11 9 22 23 16 14 13 6 12 8 2 20 1 3 4 5 7 10 15 17 18 19 21 Md RA SV


z-Scores: S3 - Anthracene
Standard Deviation for Proficiency Assessment 0.024 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
11 9 22 23 16 14 13 6 12 8 2 20 1
-3.7 -2.5 -1.9 -1.2 -1.1 -0.8 -0.3 0.1 0.1 1.4 1.8 1.8 2.0

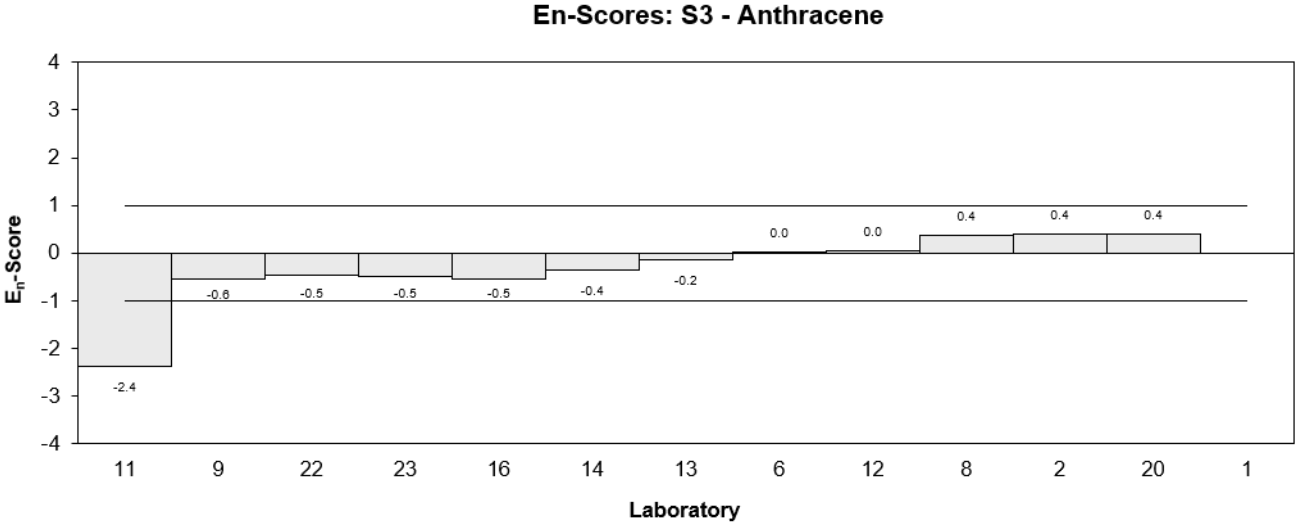

En-Scores: S3 - Anthracene
En-Score
Laboratory
11 9 22 23 16 14 13 6 12 8 2 20 1
-2.4 -0.6 -0.5 -0.5 -0.5 -0.4 -0.2 0.0 0.0 0.4 0.4 0.4

Figure 17

Table 22

Sample Details

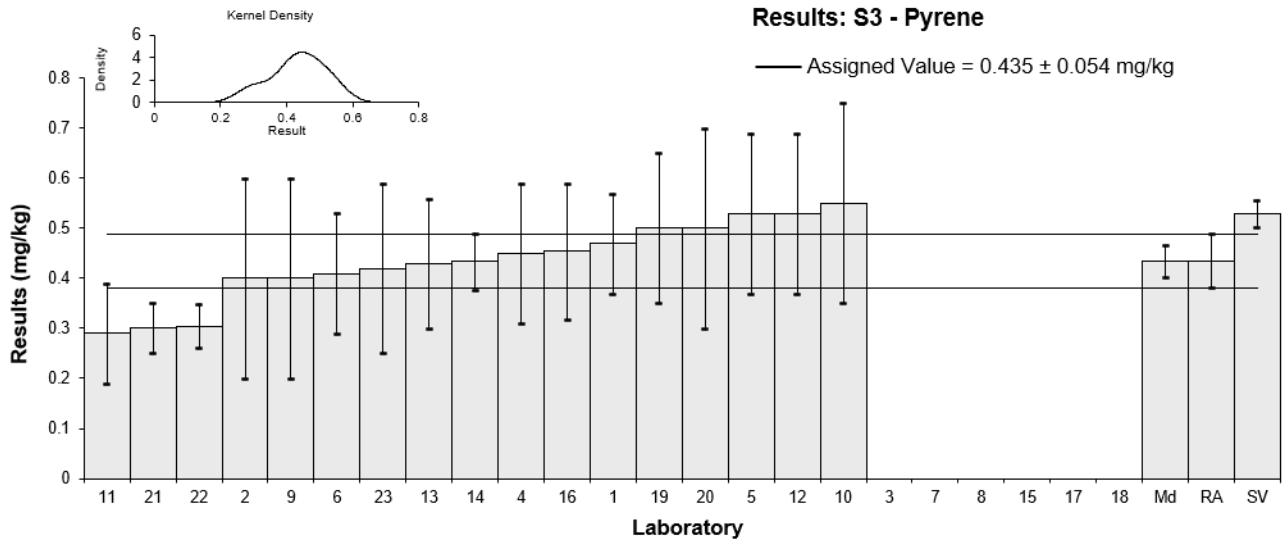
Sample No.	S3
Matrix	Soil
Analyte	Pyrene
Unit	mg/kg

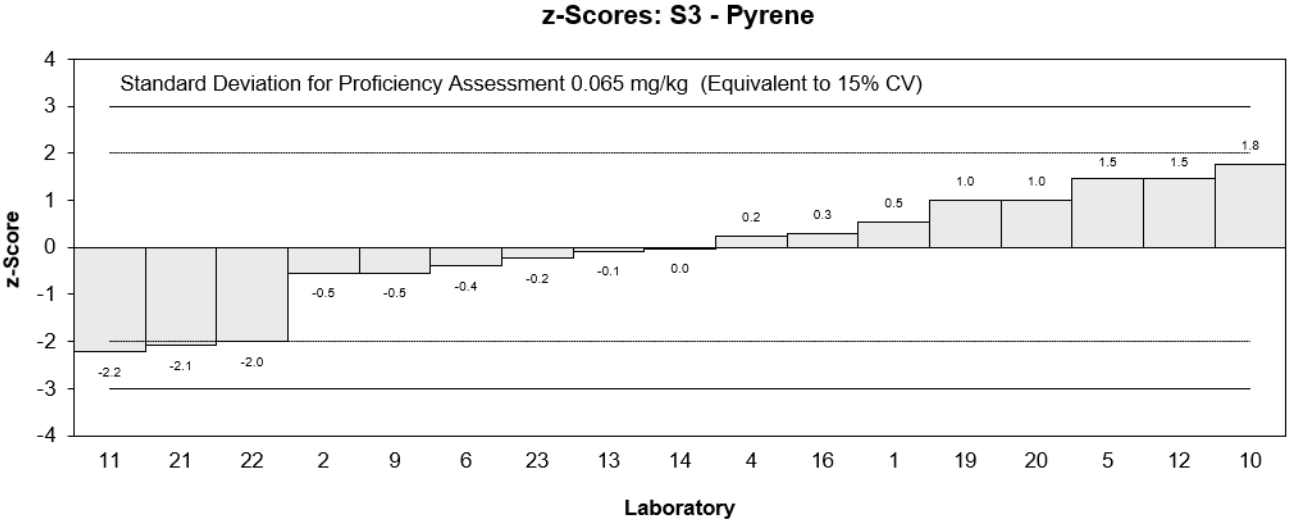
Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	0.47	0.1	0.54	0.31
2	0.4	0.2	-0.54	-0.17
3	<0.5	NR		
4	0.45	0.14	0.23	0.10
5	0.53	0.16	1.46	0.56
6	0.41	0.12	-0.38	-0.19
7	<0.5	NR		
8	<0.5	NR		
9	0.4	0.2	-0.54	-0.17
10	0.55	0.2	1.76	0.56
11	0.29	0.1	-2.22	-1.28
12	0.53	0.16	1.46	0.56
13	0.43	0.129	-0.08	-0.04
14	0.434	0.056	-0.02	-0.01
15	<0.5	0.17		
16	0.454	0.1362	0.29	0.13
17	<0.5	NR		
18	<0.5	NR		
19	0.50	0.15	1.00	0.41
20	0.5	0.2	1.00	0.31
21	0.3	0.05	-2.07	-1.83
22	0.305	0.044	-1.99	-1.87
23	0.42	0.17	-0.23	-0.08

Statistics

Assigned Value	0.435	0.054
Spike Value	0.530	0.027
Robust Average	0.435	0.054
Median	0.434	0.032
Mean	0.434	
N	17	
Max	0.55	
Min	0.29	
Robust SD	0.088	
Robust CV	20%	


Results: S3 - Pyrene
Assigned Value = 0.435 ± 0.054 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
11 21 22 2 9 6 23 13 14 4 16 1 19 20 5 12 10 3 7 8 15 17 18 Md RA SV


z-Scores: S3 - Pyrene
Standard Deviation for Proficiency Assessment 0.065 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
11 21 22 2 9 6 23 13 14 4 16 1 19 20 5 12 10
-2.2 -2.1 -2.0 -0.5 -0.5 -0.4 -0.2 -0.1 0.0 0.2 0.3 0.5 1.0 1.0 1.5 1.5 1.8

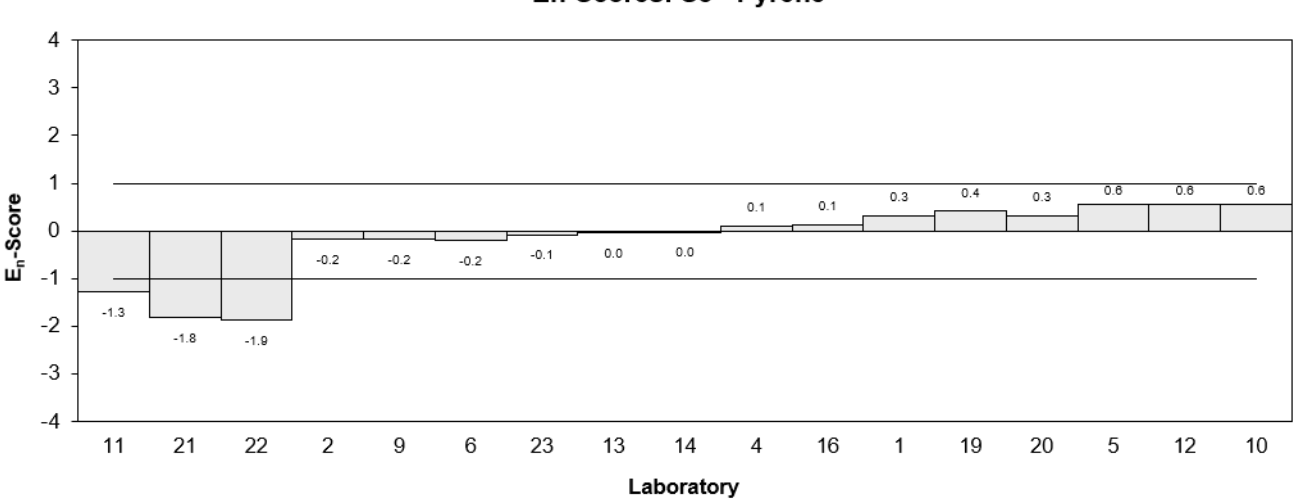

En-Scores: S3 - Pyrene
En-Score
Laboratory
11 21 22 2 9 6 23 13 14 4 16 1 19 20 5 12 10
-1.3 -1.8 -1.9 -0.2 -0.2 -0.2 -0.1 0.0 0.0 0.1 0.1 0.3 0.4 0.3 0.6 0.6 0.6

Figure 18

Table 23

Sample Details

Sample No.	S3
Matrix	Soil
Analyte	Benz[a]anthracene
Unit	mg/kg

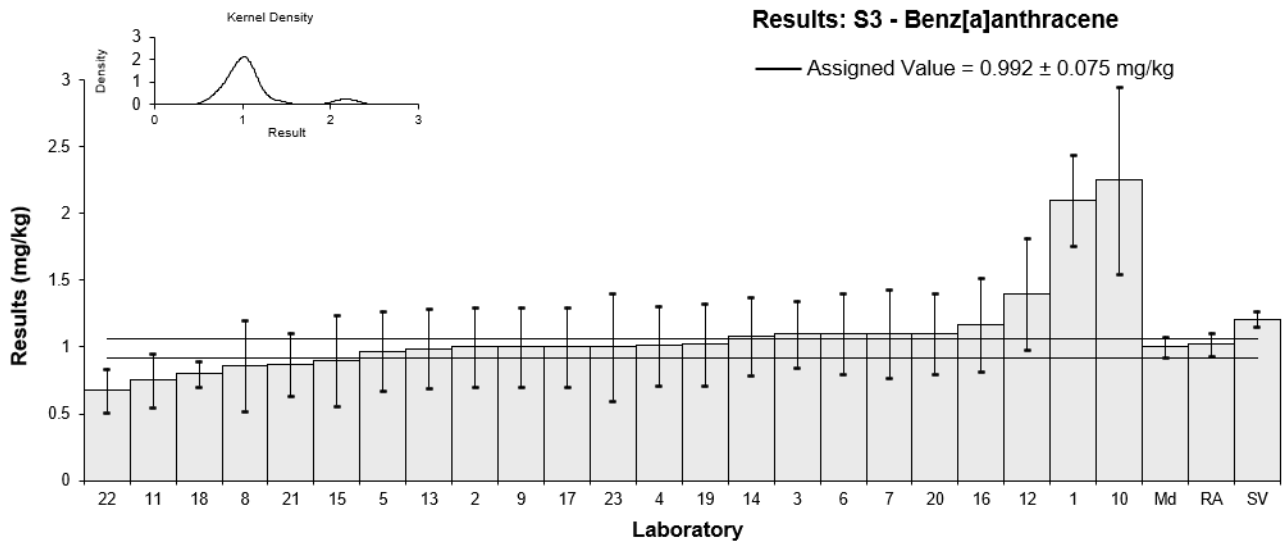
Participant Results

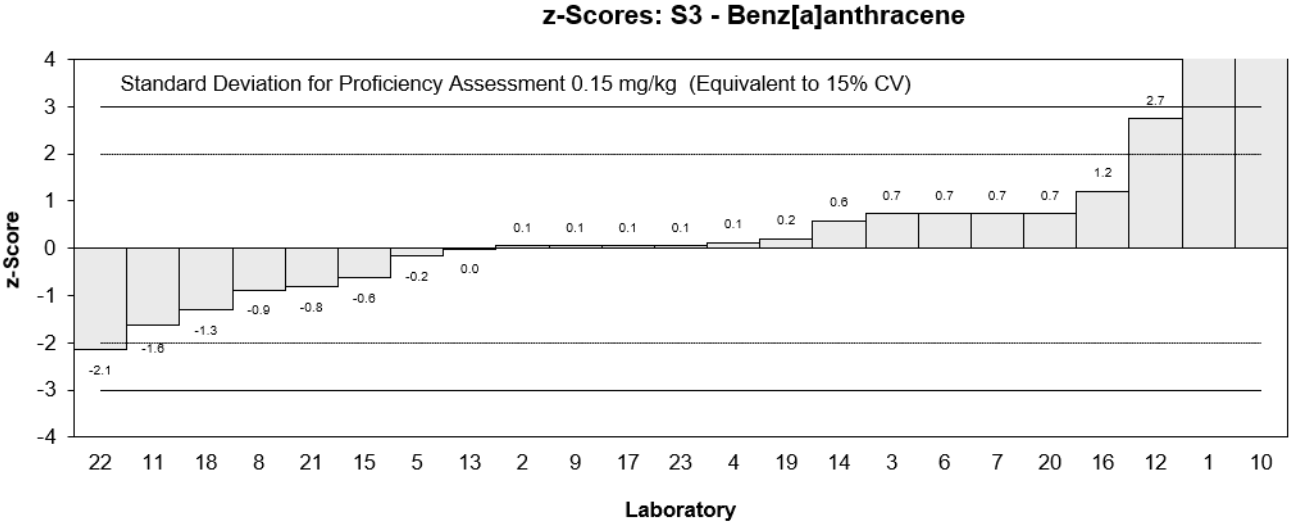
Lab. Code	Result	Uncertainty	z	E_n
1*	2.1	0.34	7.45	3.18
2	1.0	0.3	0.05	0.03
3	1.1	0.25	0.73	0.41
4	1.01	0.3	0.12	0.06
5	0.97	0.3	-0.15	-0.07
6	1.1	0.3	0.73	0.35
7	1.1	0.33	0.73	0.32
8	0.861	0.34	-0.88	-0.38
9	1	0.3	0.05	0.03
10*	2.25	0.7	8.45	1.79
11	0.75	0.2	-1.63	-1.13
12	1.4	0.42	2.74	0.96
13	0.99	0.297	-0.01	-0.01
14	1.08	0.29	0.59	0.29
15	0.9	0.34	-0.62	-0.26
16	1.17	0.351	1.20	0.50
17	1.0	0.3	0.05	0.03
18	0.8	0.097	-1.29	-1.57
19	1.02	0.31	0.19	0.09
20	1.1	0.3	0.73	0.35
21	0.87	0.24	-0.82	-0.49
22	0.674	0.163	-2.14	-1.77
23	1	0.4	0.05	0.02

* Outlier, see Section 4.2

Statistics

Assigned Value	0.992	0.075
Spike Value	1.21	0.06
Robust Average	1.02	0.09
Median	1.00	0.08
Mean	1.10	
N	23	
Max	2.25	
Min	0.674	
Robust SD	0.17	
Robust CV	16%	


Kernel Density
Density
Result
Results: S3 - Benz[a]anthracene
Assigned Value = 0.992 ± 0.075 mg/kg
Results (mg/kg)
Laboratory
22 11 18 8 21 15 5 13 2 9 17 23 4 19 14 3 6 7 20 16 12 1 10 Md RA SV


z-Scores: S3 - Benz[a]anthracene
Standard Deviation for Proficiency Assessment 0.15 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
22 11 18 8 21 15 5 13 2 9 17 23 4 19 14 3 6 7 20 16 12 1 10

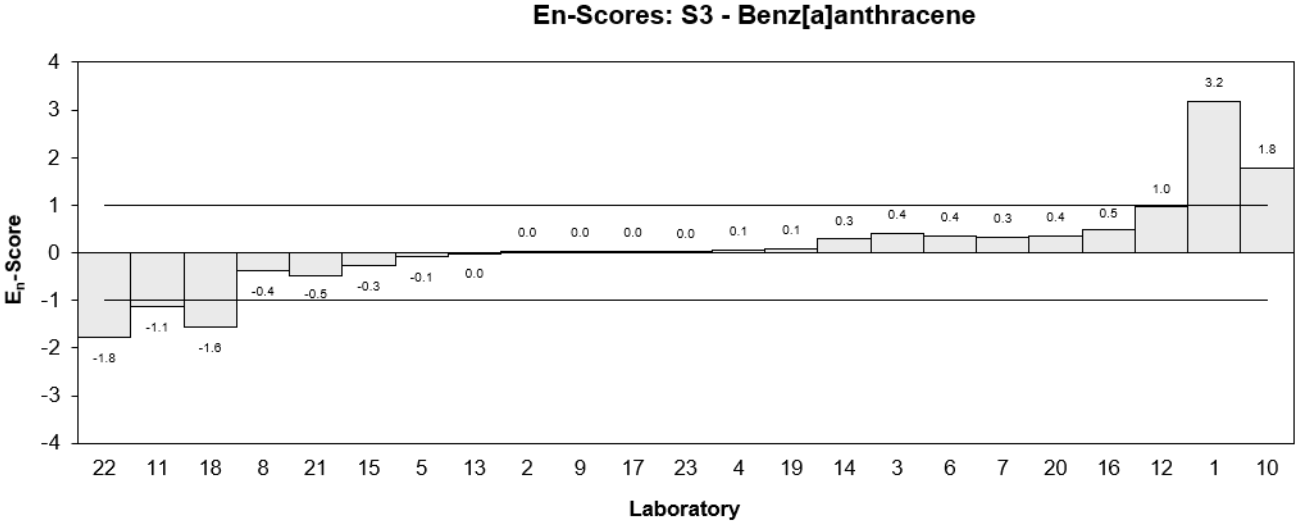

En-Scores: S3 - Benz[a]anthracene
En-Score
Laboratory
22 11 18 8 21 15 5 13 2 9 17 23 4 19 14 3 6 7 20 16 12 1 10

Figure 19

Table 24

Sample Details

Sample No.	S3
Matrix	Soil
Analyte	Chrysene
Unit	mg/kg

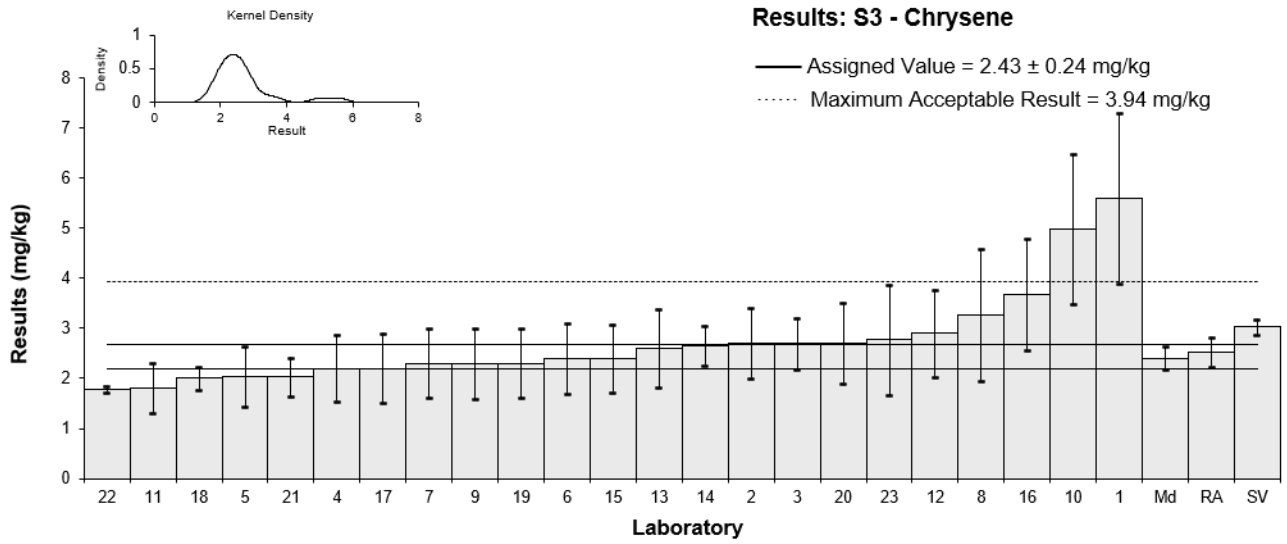
Participant Results

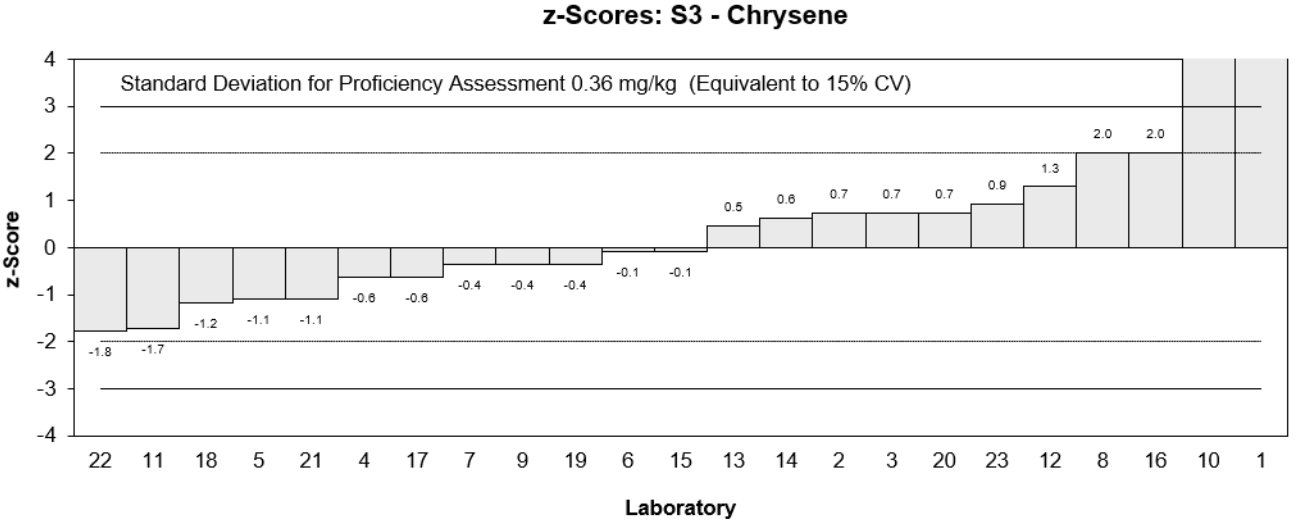
Lab. Code	Result	Uncertainty	z	E_n
1*	5.6	1.7	8.70	1.85
2	2.7	0.7	0.74	0.36
3	2.7	0.51	0.74	0.48
4	2.2	0.66	-0.63	-0.33
5	2.03	0.6	-1.10	-0.62
6	2.4	0.7	-0.08	-0.04
7	2.3	0.69	-0.36	-0.18
8	3.27	1.31	2.00▼	
9	2.3	0.7	-0.36	-0.18
10*	4.98	1.5	7.00	1.68
11	1.8	0.5	-1.73	-1.14
12	2.9	0.87	1.29	0.52
13	2.6	0.78	0.47	0.21
14	2.66	0.4	0.63	0.49
15	2.4	0.68	-0.08	-0.04
16	3.68	1.104	2.00▼	
17	2.2	0.7	-0.63	-0.31
18	2	0.223	-1.18	-1.31
19	2.30	0.69	-0.36	-0.18
20	2.7	0.8	0.74	0.32
21	2.03	0.39	-1.10	-0.87
22	1.781	0.055	-1.78	-2.64
23	2.77	1.11	0.93	0.30

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

Statistics

Assigned Value	2.43	0.24
Spike Value	3.03	0.15
Robust Average	2.53	0.29
Max Acceptable Result	3.94	
Median	2.40	0.23
Mean	2.71	
N	23	
Max	5.6	
Min	1.781	
Robust SD	0.55	
Robust CV	22%	


Kernel Density
Density
Result
Results: S3 - Chrysene
Assigned Value = 2.43 ± 0.24 mg/kg
Maximum Acceptable Result = 3.94 mg/kg
Results (mg/kg)
22 11 18 5 21 4 17 7 9 19 6 15 13 14 2 3 20 23 12 8 16 10 1 Md RA SV
Laboratory


z-Scores: S3 - Chrysene
Standard Deviation for Proficiency Assessment 0.36 mg/kg (Equivalent to 15% CV)
z-Score
-1.8 -1.7 -1.2 -1.1 -1.1 -0.6 -0.6 -0.4 -0.4 -0.4 -0.1 -0.1 0.5 0.6 0.7 0.7 0.7 0.9 1.3 2.0 2.0
22 11 18 5 21 4 17 7 9 19 6 15 13 14 2 3 20 23 12 8 16 10 1
Laboratory

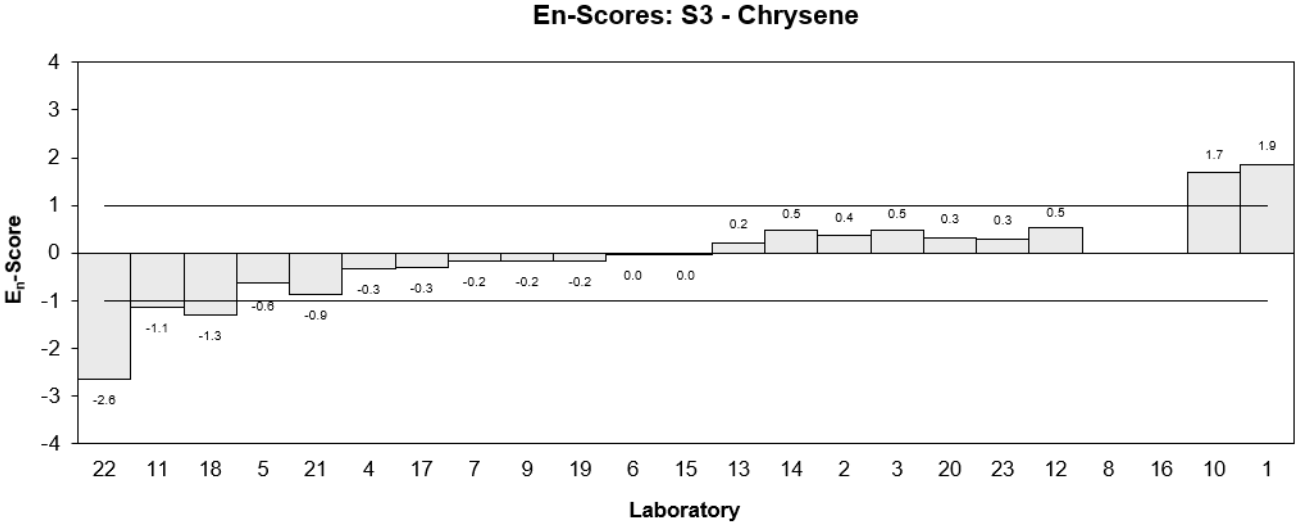

En-Scores: S3 - Chrysene
En-Score
-2.6 -1.1 -1.3 -0.6 -0.9 -0.3 -0.3 -0.2 -0.2 -0.2 0.0 0.0 0.2 0.5 0.4 0.5 0.3 0.3 0.5 1.7 1.9
22 11 18 5 21 4 17 7 9 19 6 15 13 14 2 3 20 23 12 8 16 10 1
Laboratory

Figure 20

Table 25

Sample Details

Sample No.	S3
Matrix	Soil
Analyte	Benzo[b]fluoranthene
Unit	mg/kg

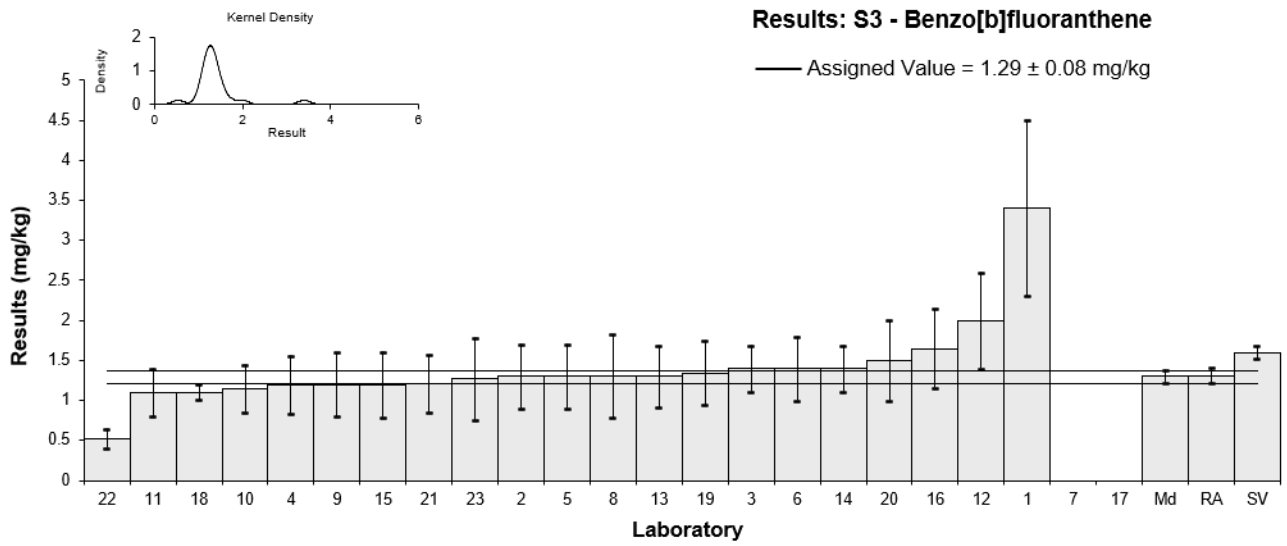
Participant Results

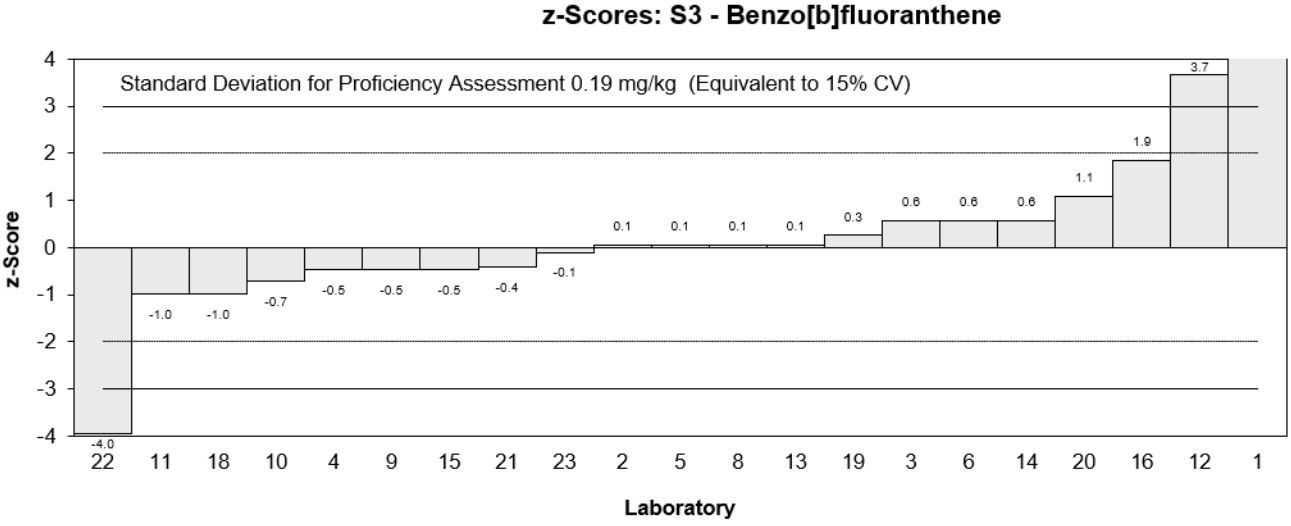
Lab. Code	Result	Uncertainty	z	E_n
1*	3.4	1.1	10.90	1.91
2	1.3	0.4	0.05	0.02
3	1.4	0.29	0.57	0.37
4	1.2	0.36	-0.47	-0.24
5	1.3	0.4	0.05	0.02
6	1.4	0.4	0.57	0.27
7	<0.5	NR		
8	1.3	0.52	0.05	0.02
9	1.2	0.4	-0.47	-0.22
10	1.15	0.3	-0.72	-0.45
11	1.1	0.3	-0.98	-0.61
12*	2	0.6	3.67	1.17
13	1.3	0.39	0.05	0.03
14	1.4	0.29	0.57	0.37
15	1.2	0.41	-0.47	-0.22
16	1.65	0.495	1.86	0.72
17	NT	NT		
18	1.1	0.098	-0.98	-1.50
19	1.34	0.40	0.26	0.12
20	1.5	0.5	1.09	0.41
21	1.21	0.36	-0.41	-0.22
22*	0.526	0.121	-3.95	-5.27
23	1.27	0.51	-0.10	-0.04

* Outlier, see Section 4.2

Statistics

Assigned Value	1.29	0.08
Spike Value	1.60	0.08
Robust Average	1.31	0.10
Median	1.30	0.08
Mean	1.39	
N	21	
Max	3.4	
Min	0.526	
Robust SD	0.18	
Robust CV	14%	


Kernel Density
Density
Result
Results: S3 - Benzo[b]fluoranthene
Assigned Value = 1.29 ± 0.08 mg/kg
Results (mg/kg)
22 11 18 10 4 9 15 21 23 2 5 8 13 19 3 6 14 20 16 12 1 7 17 Md RA SV
Laboratory


z-Scores: S3 - Benzo[b]fluoranthene
Standard Deviation for Proficiency Assessment 0.19 mg/kg (Equivalent to 15% CV)
z-Score
-4.0 -1.0 -1.0 -0.7 -0.5 -0.5 -0.5 -0.4 -0.1 0.1 0.1 0.1 0.1 0.3 0.6 0.6 0.6 1.1 1.9 3.7
22 11 18 10 4 9 15 21 23 2 5 8 13 19 3 6 14 20 16 12 1
Laboratory

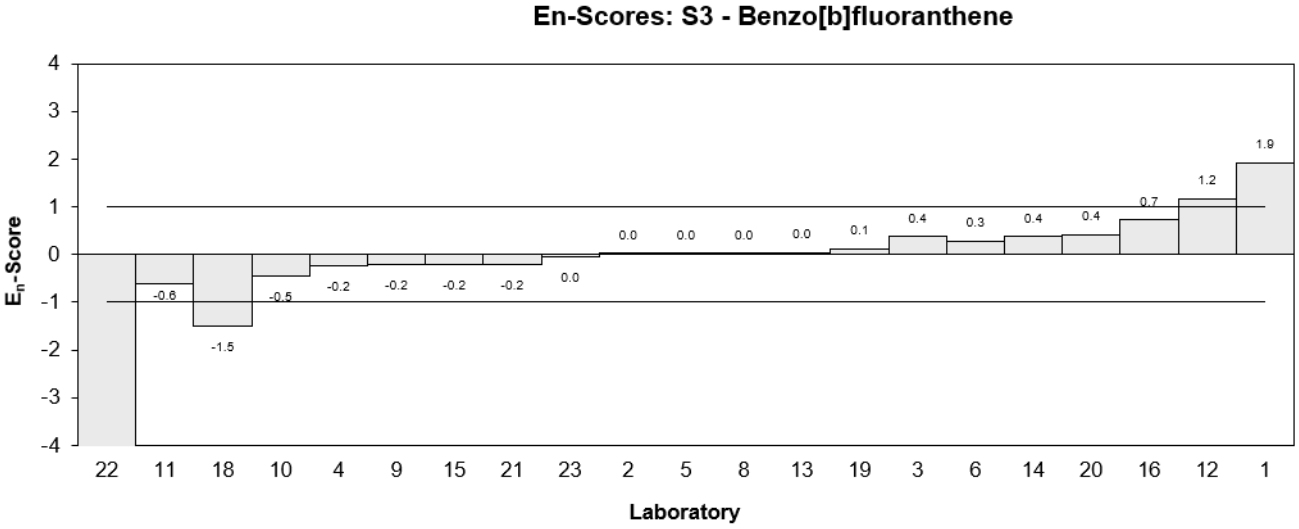

En-Scores: S3 - Benzo[b]fluoranthene
En-Score
-0.6 -1.5 -0.5 -0.2 -0.2 -0.2 -0.2 0.0 0.0 0.0 0.0 0.0 0.1 0.4 0.3 0.4 0.4 0.7 1.2 1.9
22 11 18 10 4 9 15 21 23 2 5 8 13 19 3 6 14 20 16 12 1
Laboratory

Figure 21

Table 26

Sample Details

Sample No.	S3
Matrix	Soil
Analyte	Benzo[a]pyrene
Unit	mg/kg

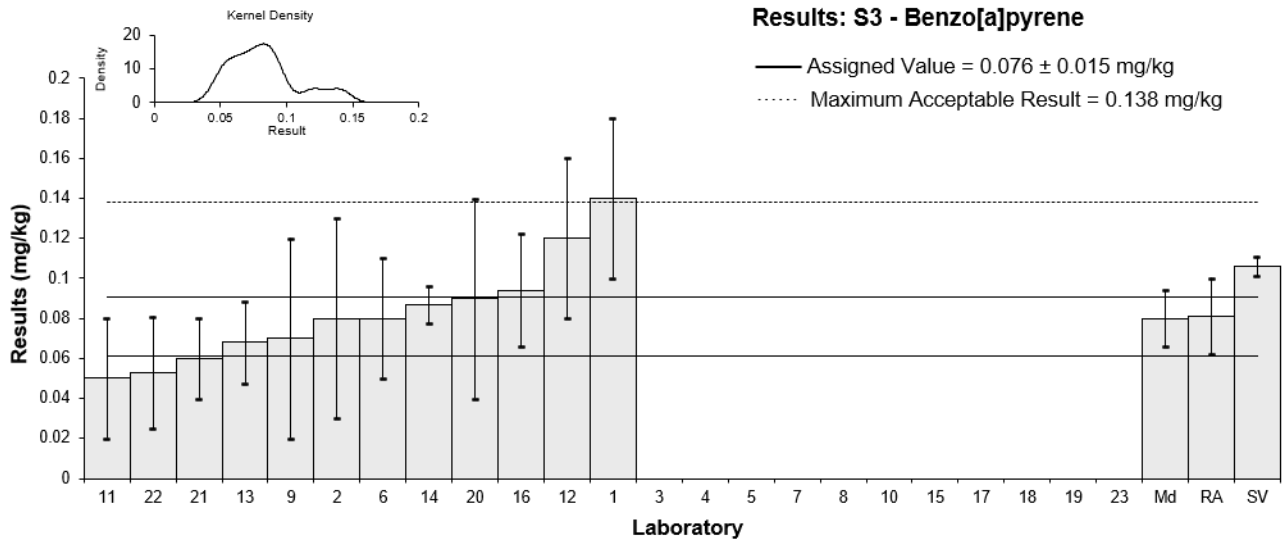
Participant Results

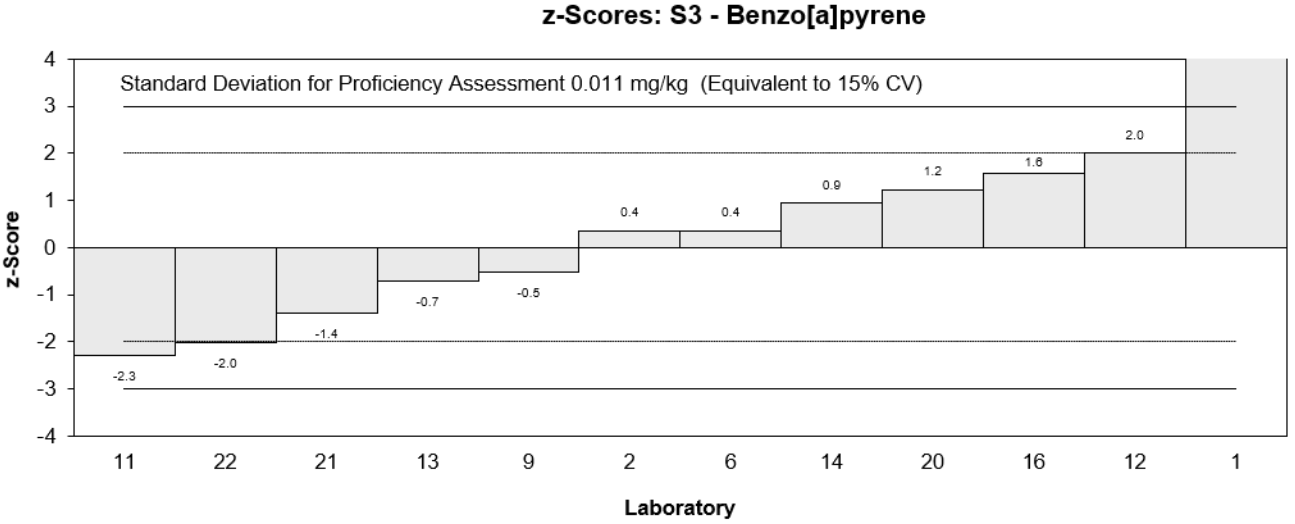
Lab. Code	Result	Uncertainty	z	E_n
1*	0.14	0.04	5.61	1.50
2	0.08	0.05	0.35	0.08
3	<0.5	NR		
4	< 0.5	NR		
5	<0.5	NR		
6	0.08	0.03	0.35	0.12
7	<0.5	NR		
8	<0.1	NR		
9	0.07	0.05	-0.53	-0.11
10	< 0.5	NR		
11	0.05	0.03	-2.28	-0.78
12	0.12	0.04	2.00▼	
13	0.068	0.0204	-0.70	-0.32
14	0.0867	0.0094	0.94	0.60
15	<0.5	0.21		
16	0.094	0.0282	1.58	0.56
17	<0.5	NR		
18	<0.5	NR		
19	< 0.5	NR		
20	0.09	0.05	1.23	0.27
21	0.06	0.02	-1.40	-0.64
22	0.053	0.028	-2.02	-0.72
23	<0.1	0.1		

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

Statistics

Assigned Value	0.076	0.015
Spike Value	0.106	0.005
Robust Average	0.081	0.019
Max Acceptable Result	0.138	
Median	0.080	0.014
Mean	0.083	
N	12	
Max	0.14	
Min	0.05	
Robust SD	0.026	
Robust CV	32%	


Kernel Density
Density
Result
Results: S3 - Benzo[a]pyrene
Assigned Value = 0.076 ± 0.015 mg/kg
Maximum Acceptable Result = 0.138 mg/kg
Results (mg/kg)
Laboratory
11 22 21 13 9 2 6 14 20 16 12 1 3 4 5 7 8 10 15 17 18 19 23 Md RA SV


z-Scores: S3 - Benzo[a]pyrene
Standard Deviation for Proficiency Assessment 0.011 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
11 22 21 13 9 2 6 14 20 16 12 1
-2.3 -2.0 -1.4 -0.7 -0.5 0.4 0.4 0.9 1.2 1.6 2.0

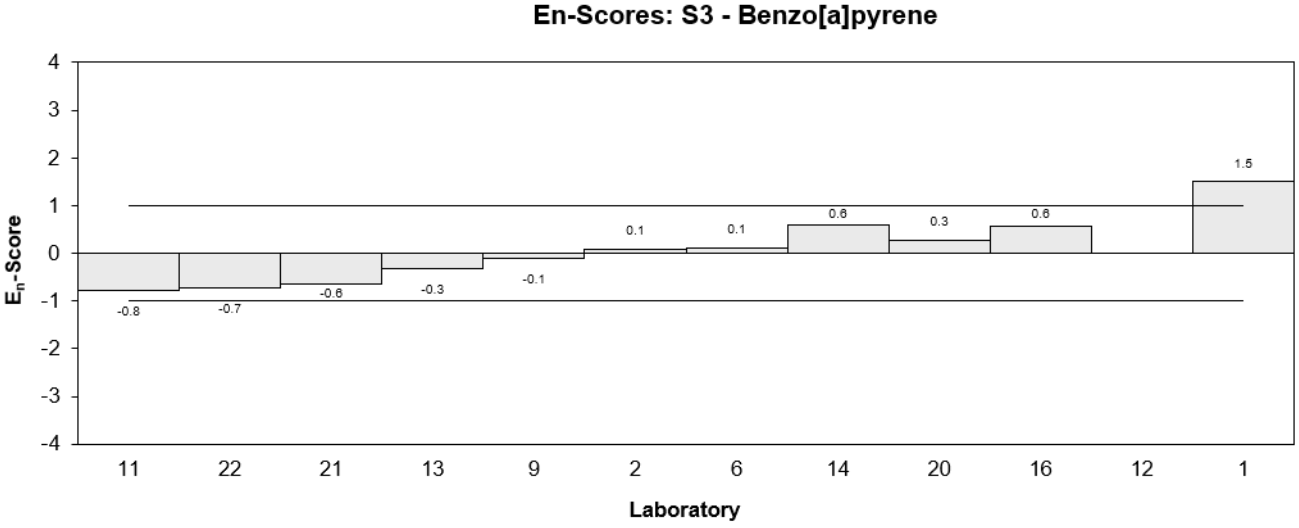

En-Scores: S3 - Benzo[a]pyrene
En-Score
Laboratory
11 22 21 13 9 2 6 14 20 16 12 1
-0.8 -0.7 -0.6 -0.3 -0.1 0.1 0.1 0.6 0.3 0.6 1.5

Figure 22

6 DISCUSSION OF RESULTS

6.1 Assigned Value

Assigned values for all scored analytes were the robust averages of participants' results. Results less than 50% or greater than 150% of the robust average were excluded from the calculation of each assigned value.[3,4] The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO 13528.[7] The calculation of the expanded uncertainty for robust averages is presented in Appendix 3, using Sample S3 acenaphthene as an example.

No assigned value was set for Sample S2 benzene, as numeric results reported for this analyte were highly variable; this may be due to the volatility of this compound. Sample S2 C6-C10 was also not scored because of its volatile nature and results have been provided for information only, though participants' results in this study were in reasonable consensus with each other.

The results reported by Laboratories 1, 14, and 16 in Sample S2 were consistently lower than the consensus value by approximately the same factor respectively. The results reported by Laboratory 23 in Sample S2 were consistently lower than the consensus value, except for C6-C10 which was significantly higher than the consensus value. To avoid unfair scoring, these results were considered invalid and were excluded from the robust average calculations as they would bias the assigned value; they were also excluded from the calculation of all summary statistics.

Samples S1 and S2 were spiked with commercially purchased products, and spiked values are not available. For Sample S3, a comparison of the assigned values and the spiked values is presented in Table 27. Assigned values were within the range of 57% to 83% of the spiked value, which is similar to ratios observed in previous NMIA hydrocarbons in soil PT studies. Assigned values were set if there was a reasonable consensus of participants' results.

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

Table 27 Ratio of Assigned Value to Spiked Value for Sample S3

Sample	Analyte	Assigned Value / Spiked Value (%)
S3	Naphthalene	83
S3	Acenaphthylene	57
S3	Acenaphthene	83
S3	Fluorene	78
S3	Phenanthrene	82
S3	Anthracene	78
S3	Pyrene	82
S3	Benz[a]anthracene	82
S3	Chrysene	80
S3	Benzo[b]fluoranthene	81
S3	Benzo[a]pyrene	72

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an evaluation of the expanded uncertainty associated with their results. It is a requirement of ISO/IEC 17025 that laboratories have procedures to evaluate the uncertainty of chemical measurements and to report this uncertainty in specific circumstances, including when the client's instruction so requires.[9]

Of 402 numeric results, 396 results (99%) were reported with an associated expanded MU. Participants used a wide variety of procedures to evaluate their uncertainty (Table 4).

Laboratories 18 and 22 did not report uncertainties for Sample S1 TRH and Sample S2 Total BTEX results (they did provide uncertainties for individual components). Both participants reported being accredited to ISO/IEC 17025.

Sample S1 TRH results from Laboratories 20 and 23 had no uncertainties, as the results were calculated by the study coordinator by summing the individual hydrocarbon range results reported, and no evaluations of the uncertainty were made.

The magnitude of the reported expanded uncertainties was within the range 0.011% to 100% of the reported result. In general, an expanded uncertainty of less than 15% relative may be unrealistically small for the routine measurement of a hydrocarbon pollutant in soil, while an expanded uncertainty of over 50% may be too large to be fit-for-purpose. In this study, 348 (88%) relative uncertainties were between 15% and 50%, 37 were less than 15%, and 11 were greater than 50%.

The following participants may need to review the procedure they use to evaluate and report uncertainties, or review if an error was made when reporting their uncertainties:

- Laboratory 2 reported relative uncertainties ranging from 16% to 63% for all analytes, except for Sample S1 >C34-C40 where they reported an uncertainty equal to the reported measurement (100% relative uncertainty).
- Laboratory 9 reported relative uncertainties ranging from 21% to 71% for all analytes, except for Sample S1 >C34-C40 and Sample S3 anthracene where they reported uncertainties equal to the reported measurements (100% relative uncertainty).
- Laboratory 22 reported a wide range of relative uncertainties. For Sample S1, relative uncertainties were all unrealistically small, ranging from 0.011% to 0.21%. For Sample S2, the relative uncertainties ranged from 2.0% to 20%. For Sample S3, the relative uncertainties ranged from 3.1% to 83%.

Participants were also requested to report the coverage factor associated with their uncertainties (Table 4). All participants reporting their coverage factor reported k = 2.

Uncertainties associated with results returning an acceptable *z*-score but a not acceptable E_n-score may have been underestimated.

Laboratories 15 and 23 attached evaluations of the expanded MU for results reported as less than their limit of reporting (LOR). An evaluation of uncertainty expressed as a value cannot be attached to a result expressed as a range.[10]

In some cases, the results were reported with an inappropriate number of significant figures. Including too many significant figures may inaccurately reflect the precision of measurements. The recommended format is to write 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 0.674 ± 0.163 mg/kg, report this as 0.67 ± 0.16 mg/kg.[10]

6.3 z-Score

The *z*-score compares the participant's deviation from the assigned value based on the standard deviation for proficiency assessment (SDPA). The SDPA defines acceptable performance in a PT study. SDPAs equivalent to 15% performance coefficient of variation (PCV) were used to calculate *z*-scores in this study. Unlike the standard deviation based on between-laboratory CV, setting the SDPA as a realistic value enables *z*-scores to be used as fixed reference value points for assessment of individual laboratory performance, independent of the group's performance.

The CVs predicted by the Thompson-Horwitz equation,[8] between-laboratory CVs, and SDPAs (as PCV) in this study are presented for comparison in Table 28.

Table 28 Comparison of Between-Laboratory CV, Thompson-Horwitz CV, and SDPA

Sample	Analyte	Numeric Results Reported*	Assigned Value (mg/kg)	Between-Laboratory CV* (%)	Thompson-Horwitz CV (%)	SDPA (as PCV, %)
S1	>C10-C16	19	1060	17	5.6	15
S1	>C16-C34	19	2050	16	5.1	15
S1	>C34-C40	15	129	21	7.7	15
S1	TRH	23	3270	13	4.7	15
S2	C6-C10	17	Not Set	16**	5**	Not Set
S2	Benzene	16	Not Set	26**	10**	Not Set
S2	Toluene	16	393	20	6.5	15
S2	Ethylbenzene	17	64.1	9.6	8.6	15
S2	Xylenes	17	289	15	6.8	15
S2	Total BTEX	16	770	14	5.9	15
S3	Naphthalene	23	1.66	13	15	15
S3	Acenaphthylene	14	0.341	18	19	15
S3	Acenaphthene	17	0.503	12	18	15
S3	Fluorene	14	0.197	18	20	15
S3	Phenanthrene	23	1.08	15	16	15
S3	Anthracene	12	0.158	27	21	15
S3	Pyrene	17	0.435	20	18	15
S3	Benz[a]anthracene	21	0.992	14	16	15
S3	Chrysene	21	2.43	18	14	15
S3	Benzo[b]fluoranthene	18	1.29	11	15	15
S3	Benzo[a]pyrene	11	0.076	26	22	15

*Values exclude outliers if applicable. Shaded cells represent between-laboratory CVs higher than both the SDPA and the Thompson-Horwitz CV for scored analytes. **Calculated using robust average (assigned value not set).

To account for possible low bias in the consensus values due to participants using inefficient analytical or extraction techniques, a total of five *z*-scores were adjusted across the following: Sample S3 acenaphthylene, anthracene, chrysene, and benzo[a]pyrene. A maximum acceptable result was set as the spiked value plus two SDPAs of the spiked value. Results

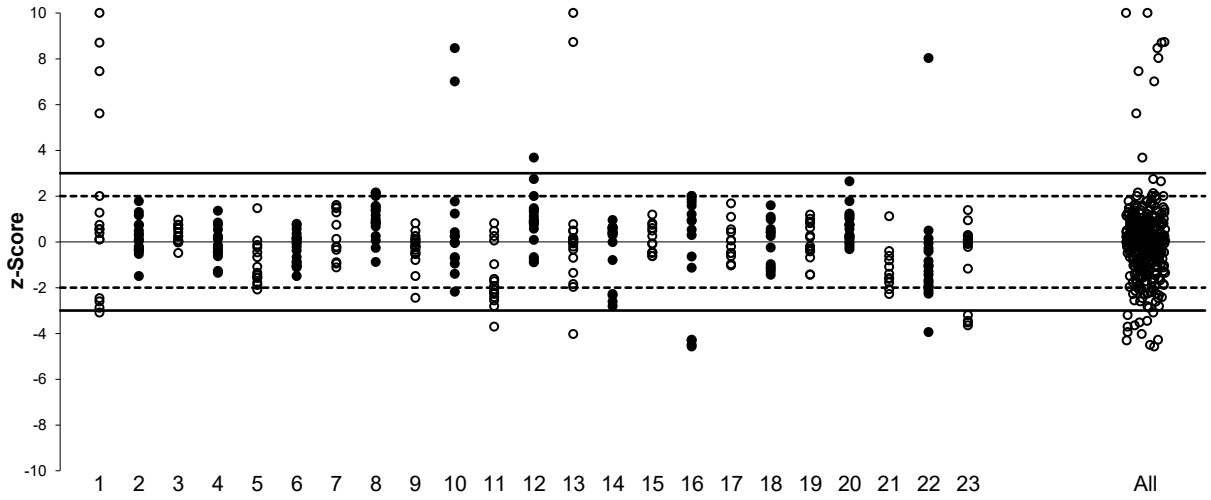
lower than the maximum acceptable result but with a *z*-score greater than 2.0 had their *z*-score adjusted to 2.0. This ensured that participants reporting results close to the spiked value were not penalised. *z*-Scores for results higher than the maximum acceptable result and *z*-scores less than 2.0 were left unaltered.

Of 363 results for which *z*-scores were calculated, 316 (87%) returned a score of $|z| \leq 2.0$, indicating an acceptable performance.

Laboratories 2, 6, 9, 12, 13, 20, and 22 reported numeric results for all 19 scored analytes. Of these participants, Laboratories 2 and 6 returned acceptable *z*-scores for all scored analytes.

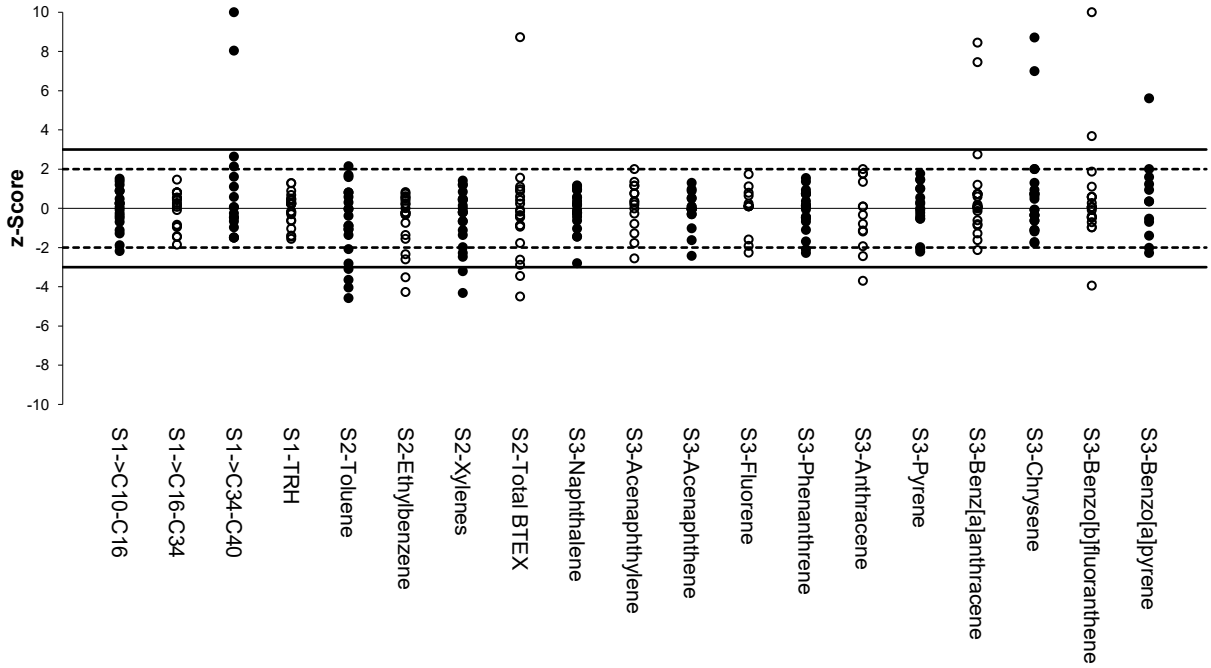
Seven other participants received acceptable *z*-scores for all scored analytes that they reported results for: Laboratories 4 (16), 3 (14), 19 (14), 15 (13), 18 (13), 7 (12), and 17 (12).

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



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

Figure 23 *z*-Score Dispersal by Laboratory

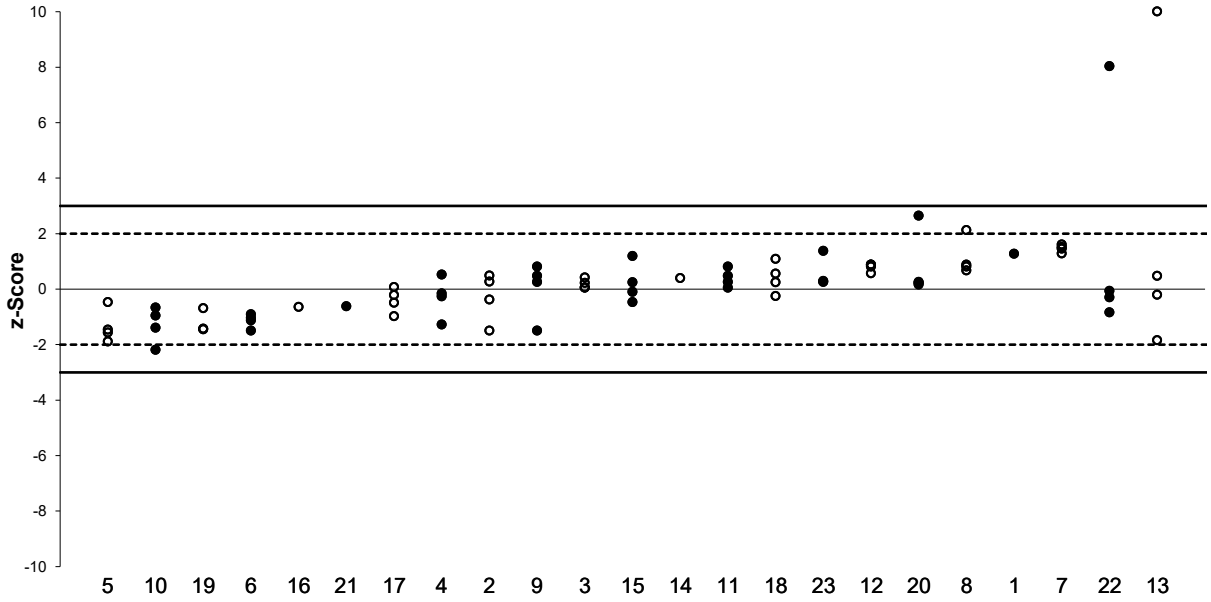


z-Scores greater than 10.0 have been plotted at 10.0.

Figure 24 *z*-Score Dispersal by Analyte

Participants' *z*-scores for Sample S1 TRH, Sample S2 BTEX, and Sample S3 PAHs are presented separately in Figures 25 to 27 respectively.

A trend of questionable or not acceptable *z*-scores on one side of the zero line may indicate laboratory bias. In particular, laboratories whose results consistently returned questionable or not acceptable *z*-scores below the zero line may have an inefficient extraction process for that type of analyte. Conversely, participants reporting results with higher acceptable *z*-scores may have more efficient methodologies.



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

Figure 25 Sample S1 TRH *z*-Score Dispersal by Laboratory

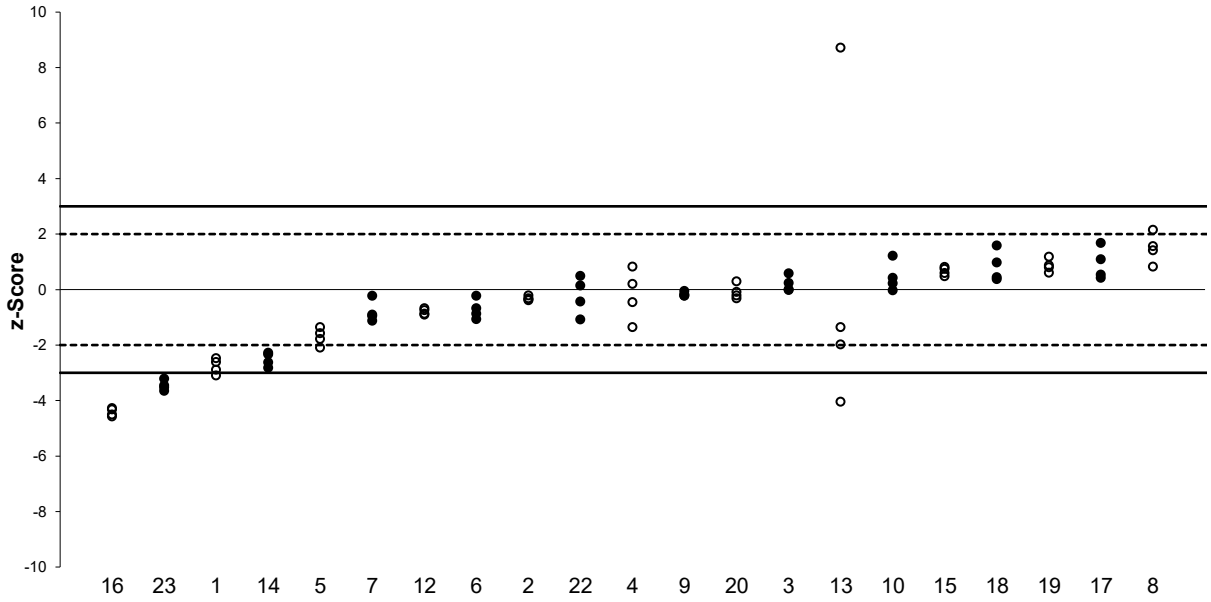
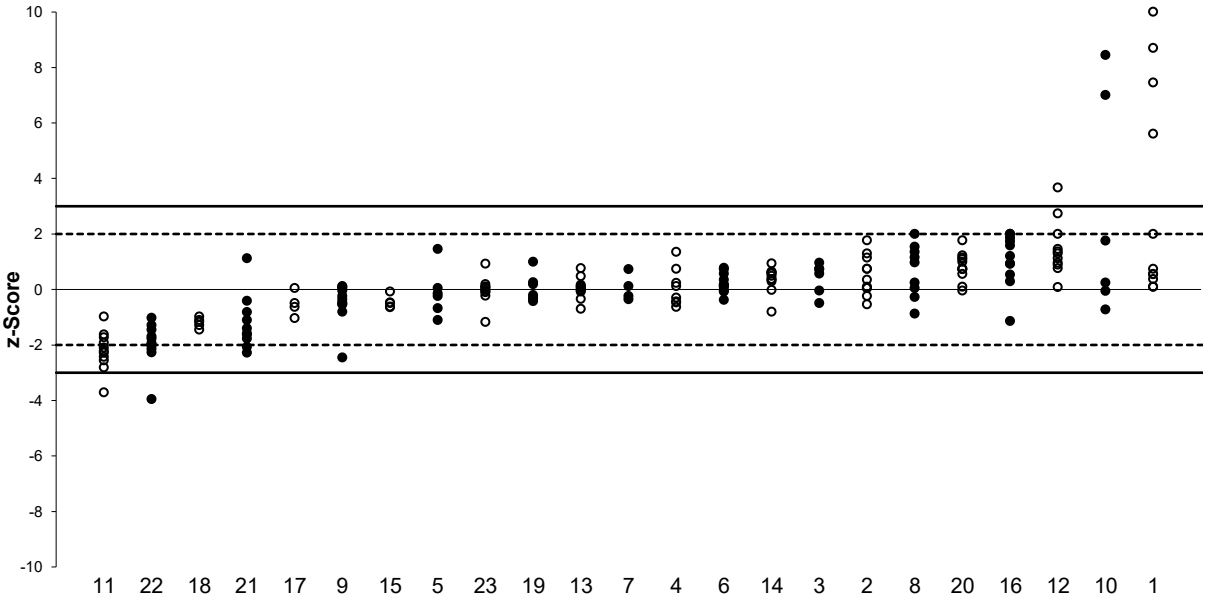


Figure 26 Sample S2 BTEX *z*-Score Dispersal by Laboratory



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

Figure 27 Sample S3 PAHs *z*-Score Dispersal by Laboratory

6.4 E_n-Score

The E_n-score indicates how closely a result agrees with the assigned value taking into consideration the respective uncertainties. E_n-scores can be interpreted in conjunction with *z*-scores, as a not acceptable E_n-score can either be caused by issues with measurement, an inappropriate evaluation of measurement uncertainty, or both.

Where a laboratory did not report an expanded uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the E_n-score. For results for which *z*-scores were adjusted as discussed in Section 6.3, no E_n-score has been reported.

Of 358 results for which E_n-scores were calculated, 291 (81%) returned an acceptable score of $|E_n| < 1.0$, indicating agreement of the participant's result with the assigned value within their respective uncertainties.

Laboratories 2, 6, 9, and 20 returned acceptable E_n-scores for all 19 scored analytes.

Five other participants received acceptable E_n-scores for all scored analytes they reported results for: Laboratories 4 (16), 3 (14), 19 (14), 15 (13), and 7 (12).

The dispersal of participants' E_n-scores is presented graphically by laboratory in Figure 28.

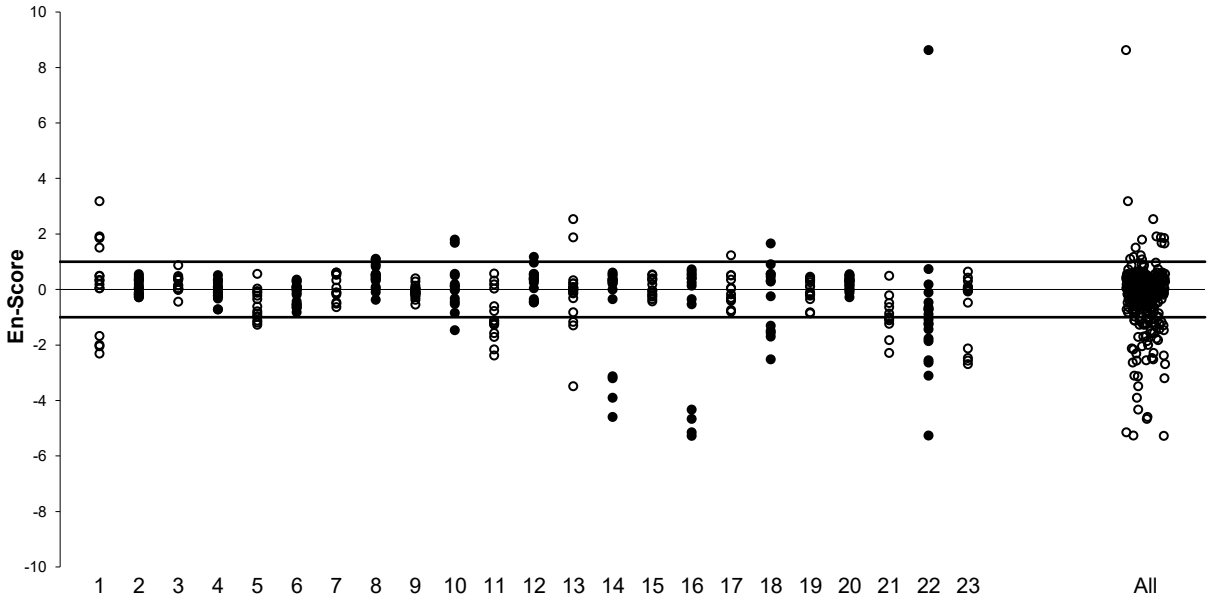


Figure 28 E_n-Score Dispersal by Laboratory

6.5 False Negatives

False negative results are presented in Table 29. These are analytes present in the sample 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 significantly higher than their limit of reporting (LOR) (i.e. the assigned value minus the expanded uncertainty was greater than the LOR), or laboratories that did not report anything.

Table 29 False Negatives

Lab. Code	Sample	Analyte	Assigned Value (mg/kg)	Spiked Value (mg/kg)	Result (mg/kg)
7	S3	Benzo[b]fluoranthene	1.29	1.6	<0.5
19	S1	>C34-C40	129	Not Available	< 100
21	S3	Anthracene	0.158	0.202	<0.05
23	S1	>C34-C40	129	Not Available	<100

6.6 Reporting of Additional Analytes

Additional analytes reported by participants are presented in Table 30. These were analytes not spiked into the sample by the study coordinator.

Sample S3 was spiked with benzo[b]fluoranthene. Laboratories 17 and 22 reported the presence of benzo[k]fluoranthene. These two analytes typically closely elute or coelute depending on chromatographic conditions.[11] Participants should take care when interpreting their analytical data.

Laboratory 17 reported that they did not test for benzo[b]fluoranthene. However, they reported for benzo[k]fluoranthene at a concentration similar to the assigned value of benzo[b]fluoranthene.

Laboratory 22 reported a result for benzo[b]fluoranthene that was biased low, and also reported for benzo[k]fluoranthene. Combining their benzo[b]fluoranthene and benzo[k]fluoranthene results gives a value close to the benzo[b]fluoranthene assigned value.

Table 30 Non-Spiked Analytes Reported by Participants

Lab. Code	Sample	Analyte	Result (mg/kg)	Uncertainty (mg/kg)
17	S3	Benzo[k]fluoranthene	1.3	0.3
22	S3	Benzo[k]fluoranthene	0.589	0.12

6.7 Participants' Analytical Methods

A variety of analytical methods were used by participants in this study (Appendix 4).

TRH

Sample S1 was a 50 g soil sample. Participants used a sample size between 2.5 g and 10 g for TRH analysis, with most participants using 10 g. A plot of z-scores against sample mass used for analysis is presented in Figure 29.

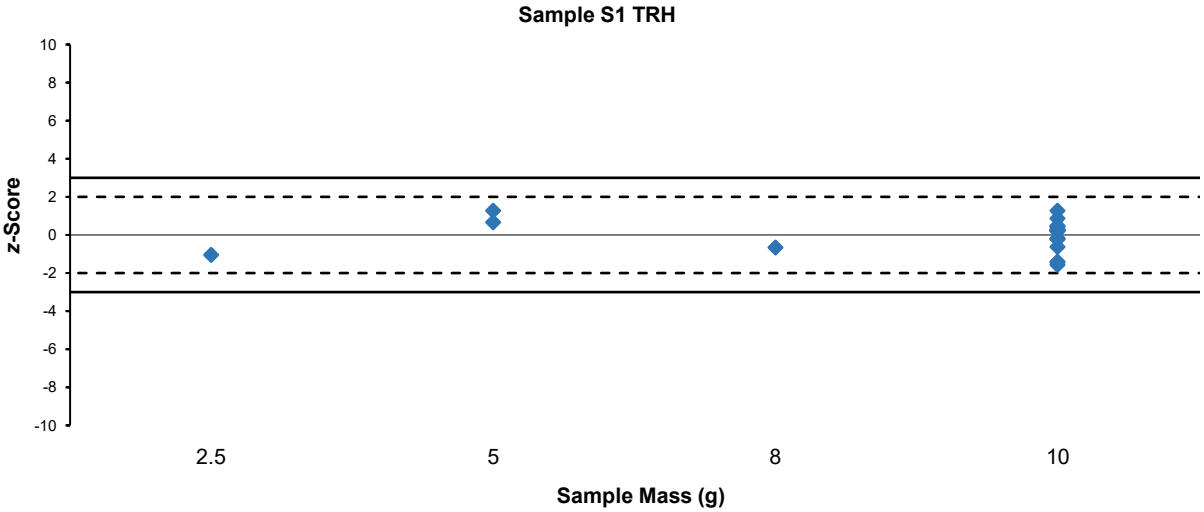


Figure 29 Sample S1 TRH z-Scores vs Sample Mass Used for Analysis

Participants employed a range of extraction techniques, including solid-liquid extraction (SLE) with mechanical agitation (MA), sonication (S), and accelerated solvent extraction (ASE). Extraction solvents used included acetone (ACE), dichloromethane (DCM), hexane (HEX), and/or methanol (MeOH). Some participants also applied a silica clean-up procedure prior to analysis. Participants used gas chromatography (GC) coupled to either flame ionisation detection (FID) or tandem mass spectrometry (MS/MS) for analysis.

A plot of z-scores versus methodology for Sample S1 TRH is presented in Figure 30. The most common methodology used to analyse TRH in this study was SLE (MA) with DCM/ACE as the extraction solvent, no clean-up step, and using GC-FID for analysis. TRH analysis did not challenge participants, with excellent agreement between reported results across the range of methodologies employed.

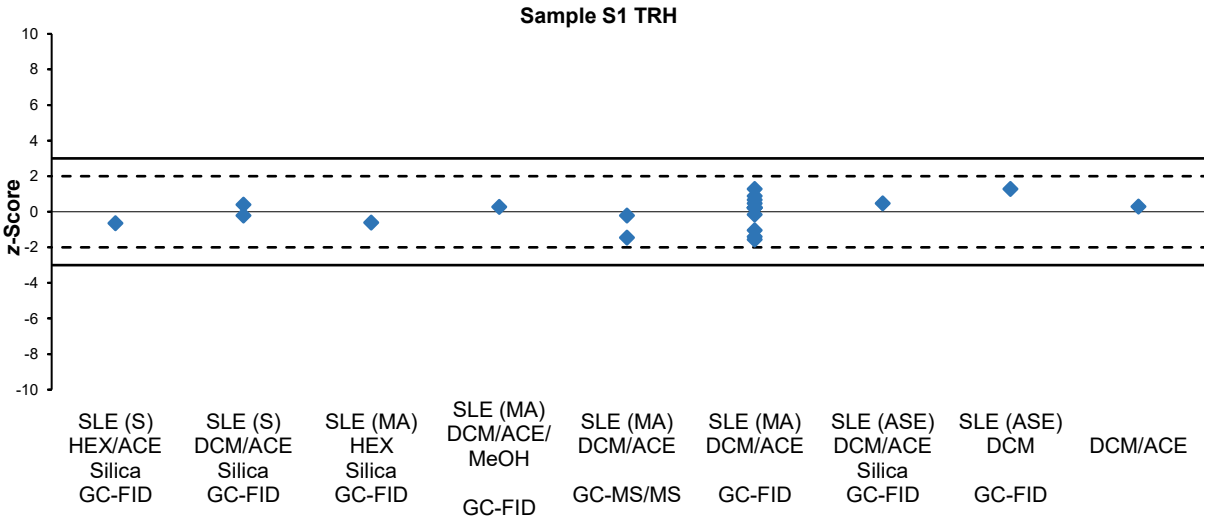


Figure 30 Sample S1 TRH z-Scores vs Methodology

Laboratories 13 and 22 were both biased significantly high for >C34-C40 only, while still returning acceptable results for TRH. Both participants reported results that were acceptable, however biased low, for >C16-C34. These participants may need to review whether their retention time cut-offs for band separation are suitable.

BTEX

Sample S2 was a 50 g soil sample. Participants used a sample size between 2.5 g and 14 g for BTEX analysis, with most participants using 5 g. A plot of z-scores against sample mass used for analysis is presented in Figure 31. Participants using larger sample masses should verify that their methodology achieves adequate extraction efficiency.

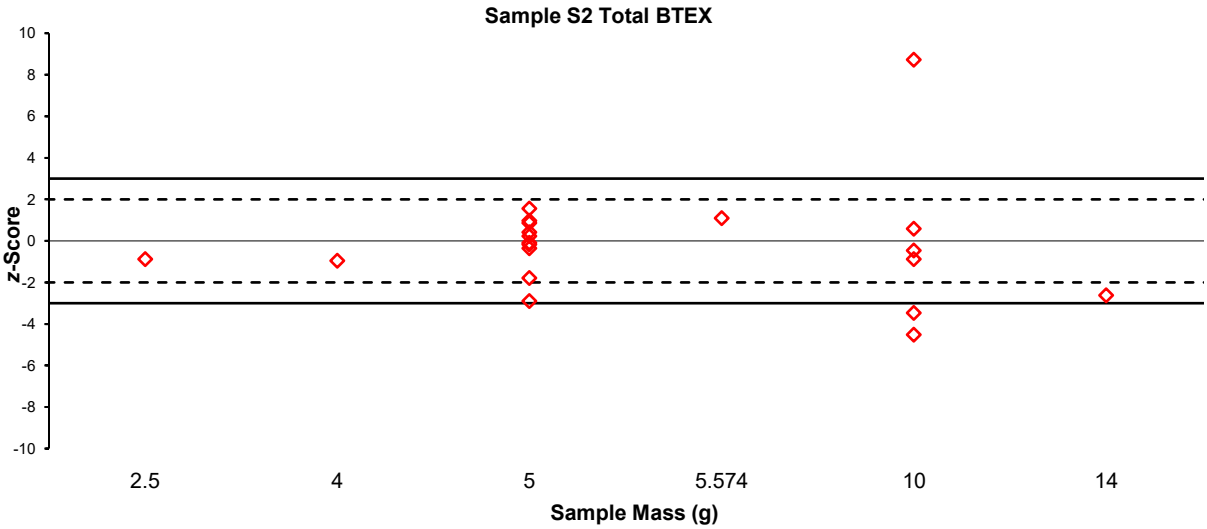


Figure 31 Sample S2 Total BTEX z-Scores vs Sample Mass Used for Analysis

Most participants used purge and trap (P&T) GC-mass spectrometry (MS).

Two participants used headspace (HS) GC-MS instead. These participants were biased low in this study (Figure 32) and may need to review their parameters (for example: temperature, incubation time, or agitation technique) to identify the source of this low bias or implement procedures to correct for the bias.

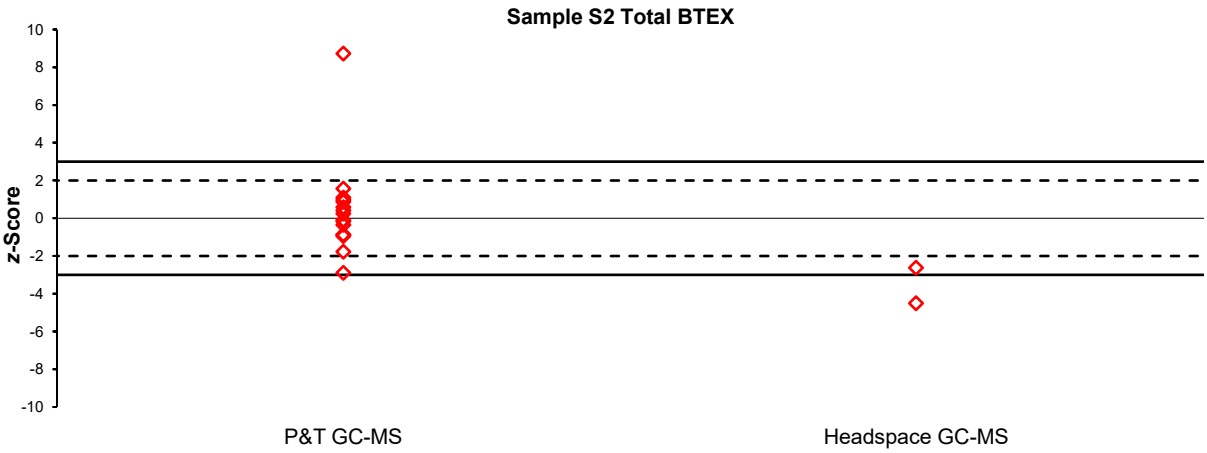


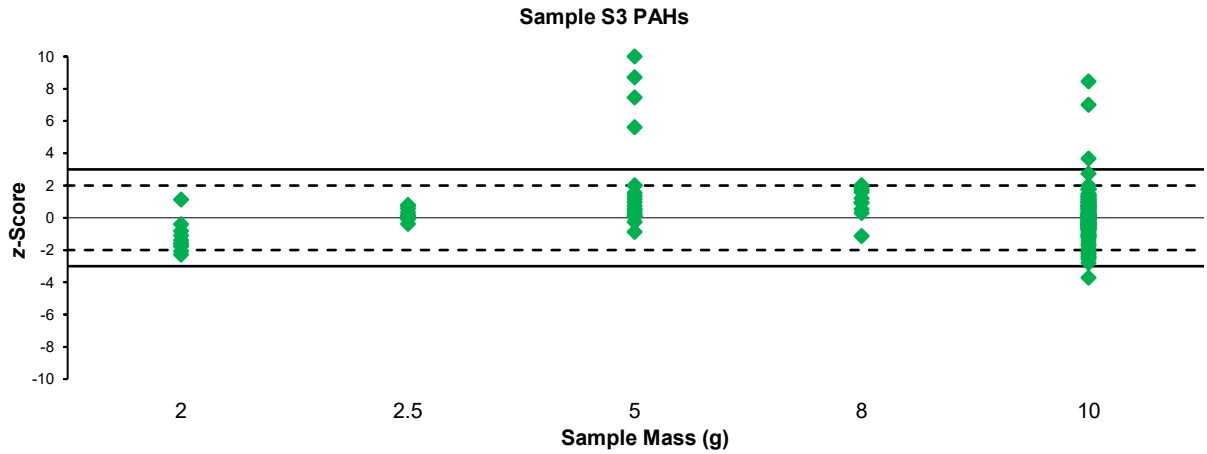
Figure 32 Sample S2 Total BTEX z-Scores vs Instrument

The results reported by Laboratories 1, 14, and 16 in Sample S2 were consistently lower than the consensus value by approximately the same factor respectively. The results reported by Laboratory 23 in Sample S2 were consistently lower than the consensus value, except for C6-C10 which was significantly higher than the consensus value. These participants may have applied an incorrect dilution factor.

The results from Laboratory 13 were extremely varied (Figure 26). The sum of their individual BTEX components did not add up to their total BTEX value. Instead, their total BTEX result is equal to the sum of all BTEX component results and their C6-C10 result. This participant may need to review if they have made a reporting error with their results.

PAHs

Sample S3 was a 50 g soil sample. Participants used a sample size between 2 g and 10 g for PAHs analysis, with the majority of participants using 10 g. A plot of *z*-scores against sample mass used for analysis is presented in Figure 33.



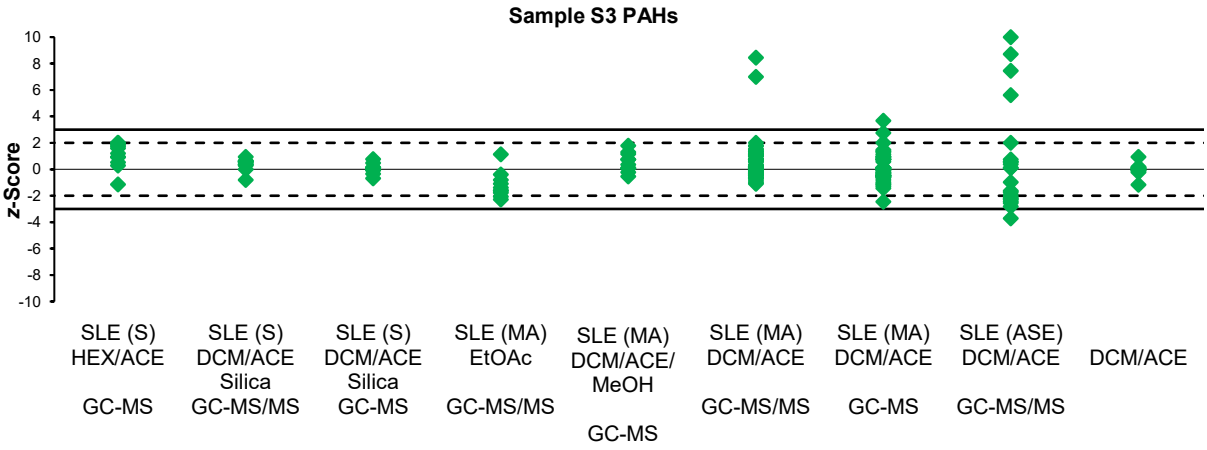
z-Scores greater than 10.0 have been plotted at 10.0.

Figure 33 Sample S3 PAHs *z*-Scores vs Sample Mass Used for Analysis

Participants employed a range of extraction techniques, including SLE (MA), SLE (S), or SLE (ASE). Extraction solvents used included ACE, DCM, HEX, MeOH, and/or ethyl acetate (EtOAc). Some participants also applied a silica clean-up procedure prior to analysis. All participants used GC-MS(/MS) for analysis.

A plot of z-scores against methodology for Sample S3 PAHs is presented in Figure 34. The most common methodology used to analyse PAHs in this study was SLE (MA) with

DCM/ACE as the extraction solvent, no clean-up step, and using GC-MS/MS for analysis. In general, the different methodologies produced compatible results.



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

Figure 34 Sample S3 PAHs *z*-Scores vs Methodology

Laboratories 1 and 10 reported results significantly higher than the assigned value for Sample S3 benz[a]anthracene and chrysene, with z-scores greater than 5.0. These two analytes typically elute at similar times when using gas chromatography.[12] If chromatographic conditions were not optimised and the two peaks were not completely resolved, this may have contributed to the overestimation of both analytes.

As discussed in Section 6.6, Laboratories 17 and 22 reported for benzo[k]fluoranthene, which was not spiked into the sample. This analyte usually closely elutes or coelutes with benzo[b]fluoranthene depending on chromatographic conditions.[11] Incorrect reporting of benzo[k]fluoranthene may have contributed to their underestimation or non-reporting of benzo[b]fluoranthene.

6.8 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 their analysis.

Twelve participants reported using certified standards, 2 participants reported using matrix reference materials, and 3 participants reported using both. The following were reported by participants:

- NMIA MX015
- Accustandard
- ERA, e.g. 727
- o2si
- Sigma Aldrich, e.g. CRM47993, CRM47505, CRM355, CRM143,
- ISO 17034 traceable standards
- PM Separations

These materials may or may not meet the internationally recognised definition of a Certified Reference Material:

> '***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*'[13]

6.9 Summary of Participants' Results and Performances

Summaries of participants' results and performances in this PT study are presented in Tables 31 and 32, and Figure 35.

Table 31 Summary of Participants' Results for Samples S1 and S2*

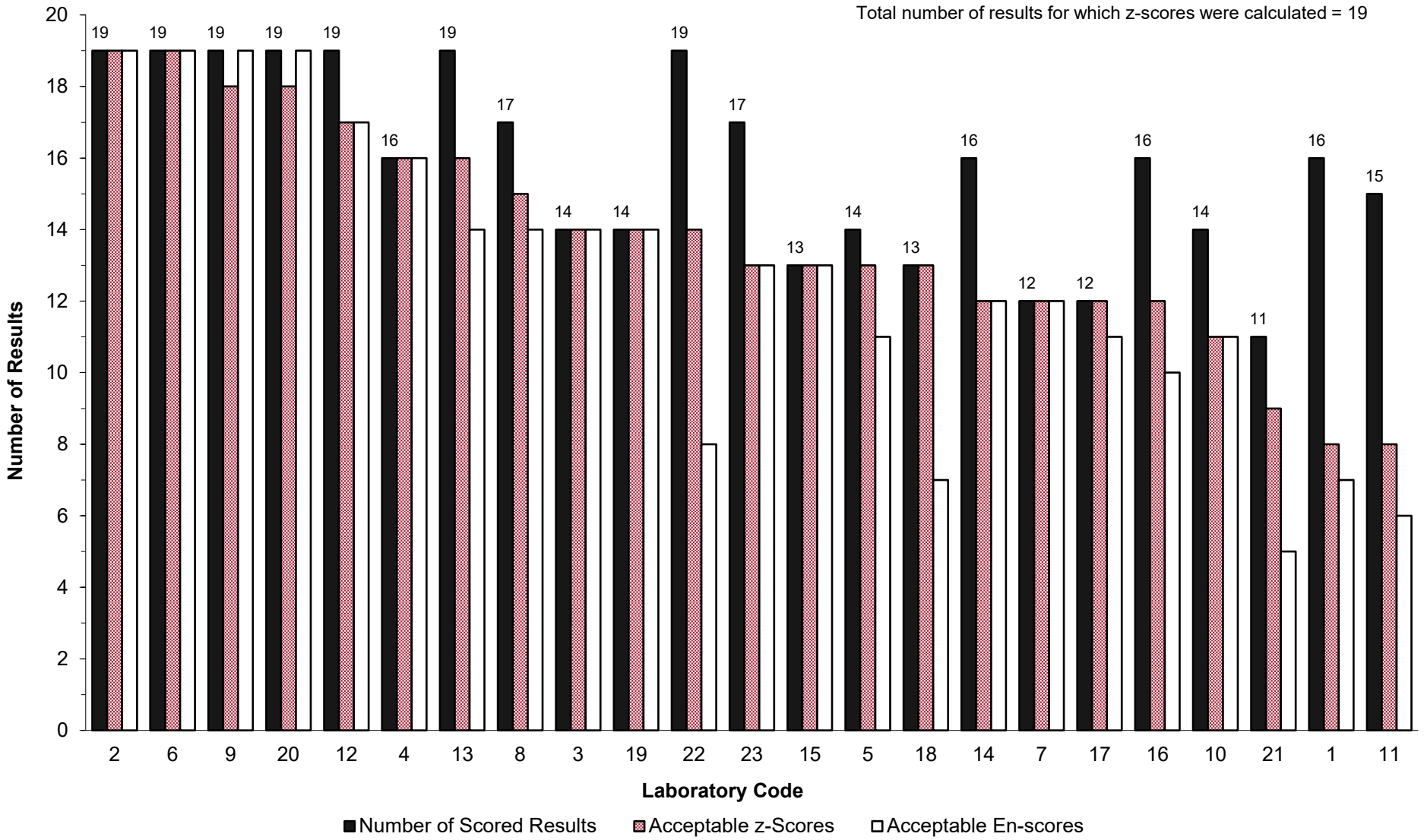
Lab. Code	Sample S1				Sample S2					
	>C10-C16	>C16-C34	>C34-C40	TRH	C6-C10	Benzene	Toluene	Ethylbenzene	Xylenes	Total BTEX
AV	1060	2050	129	3270	Not Set	Not Set	393	64.1	289	770
1	NR	NR	NR	3891	NT	4.7	210	39	182	436
2	1000	2200	100	3400	2200	19	370	61	280	730
3	1070	2180	130	3380	2570	19	427	64.2	288	798
4	857	2210	124	3191	2109	13	313	66	325	717
5	760	1600	120	2500	2100	16	270	49	230	564
6	880	1770	100	2760	2700	21	330	62	260	670
7	1300	2500	160	3900	1400	12	340	62	240	660
8	1200	2300	170	3600	2400	15	520	72	350	950
9	1100	2300	100	3500	2500	17	390	62	280	750
10	711	1755	116	2583	2259.54	18.97	391.43	66.30	342.00	818.70
11	1100	2300	130	3500	NT	NT	NT	NT	NT	NT
12	1200	2300	140	3700	2600	10	340	57	260	670
13	1136	1482	548	3166	1367	1.6	155	51	203	1777.6
14	NR	NR	NR	3460	NT	7.89	227	41.6	190.3	467
15	1250	2020	120	3390	2270	15.8	441	71.2	310	838
16	NT	NT	NT	2950	NT	1.88	123	22.9	101.8	249.58
17	980	2070	110	3160	2930	27.2	492	69.3	307	896
18	1020	2220	150	3390	2740	19.3	487	67.8	308	882
19	951	1609	< 100	2560	2600	16	440	70	340	870
20	1100	2100	180	3380	2100	13	410	61	280	760
21	NR	NR	NR	2966.01	NT	NT	NT	NT	NT	NT
22	1048.9	1790.6	284.3	3123.8	2090	27.49	329.1	68.84	295.08	720.51
23	1279	2131	<100	3410	12900	8.6	178	30.2	150	370

*All values are in mg/kg. Shaded cells are results which returned a questionable or not acceptable *z*-score. AV = Assigned Value; NT = Not Tested; NR = Not Reported.

Table 32 Summary of Participants' Results for Sample S3*

Lab. Code	Sample S3										
	Naphthalene	Acenaphthylene	Acenaphthene	Fluorene	Phenanthrene	Anthracene	Pyrene	Benz[a]anthracene	Chrysene	Benzo[b]fluoranthene	Benzo[a]pyrene
AV	1.66	0.341	0.503	0.197	1.08	0.158	0.435	0.992	2.43	1.29	0.076
1	1.8	0.36	0.51	0.2	1.2	0.22	0.47	2.1	5.6	3.4	0.14
2	1.6	0.4	0.6	0.2	1.2	0.2	0.4	1.0	2.7	1.3	0.08
3	1.9	<0.5	0.5	<0.5	1.0	<0.5	<0.5	1.1	2.7	1.4	<0.5
4	1.55	0.41	0.48	< 0.5	1.2	< 0.5	0.45	1.01	2.2	1.2	< 0.5
5	1.6	<0.5	<0.5	<0.5	0.97	<0.5	0.53	0.97	2.03	1.3	<0.5
6	1.7	0.35	0.50	0.22	1.1	0.16	0.41	1.1	2.4	1.4	0.08
7	1.6	<0.5	<0.5	<0.5	1.1	<0.5	<0.5	1.1	2.3	<0.5	<0.5
8	1.95	0.327	0.576	0.204	1.33	0.19	<0.5	0.861	3.27	1.3	<0.1
9	1.6	0.3	0.5	0.2	1.1	0.1	0.4	1	2.3	1.2	0.07
10	1.72	< 0.5	< 0.5	< 0.5	1.07	< 0.5	0.55	2.25	4.98	1.15	< 0.5
11	0.96	0.21	0.32	0.14	0.74	0.07	0.29	0.75	1.8	1.1	0.05
12	1.9	0.38	0.57	0.23	1.3	0.16	0.53	1.4	2.9	2	0.12
13	1.7	0.38	0.5	0.2	1.1	0.15	0.43	0.99	2.6	1.3	0.068
14	1.75	0.356	0.54	0.216	1.14	0.139	0.434	1.08	2.66	1.4	0.0867
15	1.5	<0.5	<0.5	<0.5	1.0	<0.5	<0.5	0.9	2.4	1.2	<0.5
16	1.89	0.540	0.543	0.248	1.23	0.131	0.454	1.17	3.68	1.65	0.094
17	1.4	<0.5	<0.5	<0.5	1.0	<0.5	<0.5	1.0	2.2	NT	<0.5
18	1.3	<0.5	<0.5	<0.5	0.9	<0.5	<0.5	0.8	2	1.1	<0.5
19	1.61	< 0.5	0.48	< 0.5	1.01	< 0.5	0.50	1.02	2.30	1.34	< 0.5
20	1.8	0.4	0.5	0.2	1.2	0.2	0.5	1.1	2.7	1.5	0.09
21	1.94	0.25	0.38	0.15	0.71	<0.05	0.3	0.87	2.03	1.21	0.06
22	1.298	0.275	0.425	0.13	0.806	0.112	0.305	0.674	1.781	0.526	0.053
23	1.64	0.34	0.51	0.2	1.11	0.13	0.42	1	2.77	1.27	<0.1

*All values are in mg/kg. Shaded cells are results which returned a questionable or not acceptable *z*-score. AV = Assigned Value; NT = Not Tested.



Laboratories 11 and 21 only analysed Samples S1 and S3 (total number of results for which *z*-scores were calculated = 15).

Figure 35 Summary of Participants' Performance

6.10 Comparison with Previous Hydrocarbons in Soil PT Studies

To enable direct comparison with results from previous NMIA hydrocarbons in soil PT studies, the SDPA used to calculate *z*-scores has been kept constant at 15% PCV.

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

TRH

A summary of the acceptable performance (presented as a percentage of the total number of scores) obtained by participants for TRH in soil over the last 10 studies (2017 – 2026) is presented in Figure 36. Over this period, the average proportion of acceptable *z*-scores was 93%, and the average proportion of acceptable E_n-scores was 78%. This study had the highest performing group over this period, with excellent proportions of acceptable results for both *z*-scores and E_n-scores.

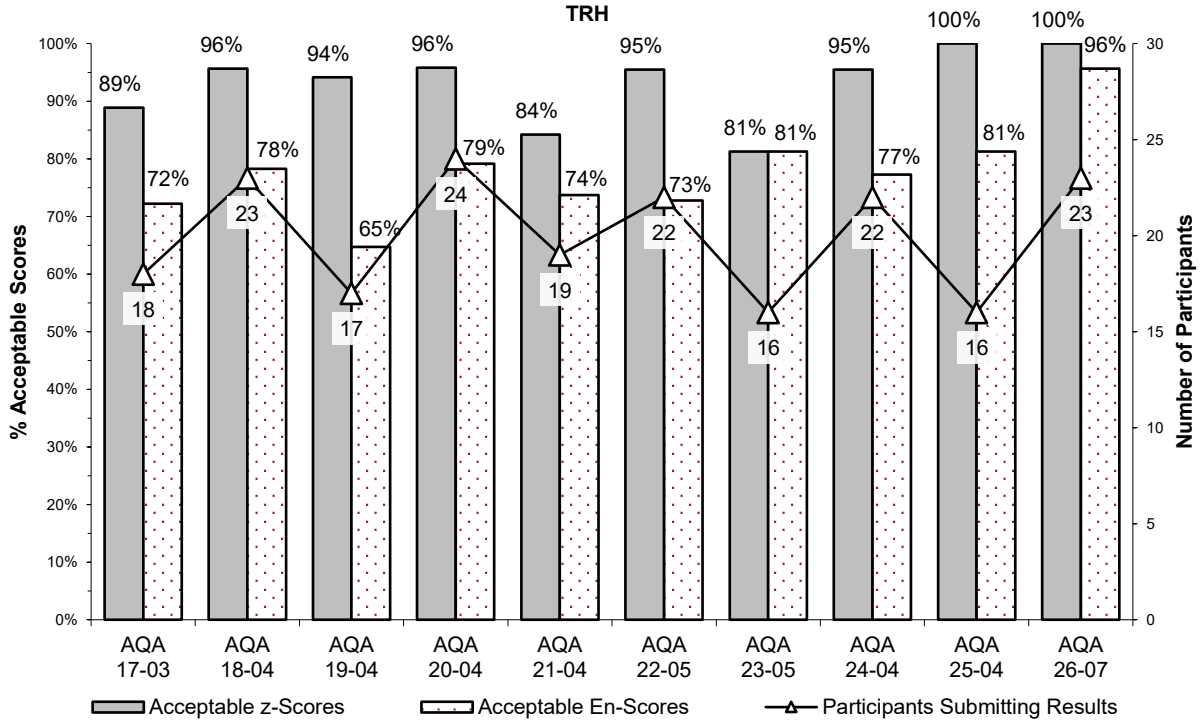


Figure 36 Participants' Performance for TRH in NMIA Hydrocarbons in Soil PT Studies

Total BTEX

A summary of the acceptable performance (presented as a percentage of the total number of scores) obtained by participants for Total BTEX in soil over the last 10 studies (2017 – 2026) is presented in Figure 37. Over this period, the average proportion of acceptable *z*-scores was 82%, and the average proportion of acceptable E_n-scores was 77%. While this study shows an improvement as compared to the previous 2 studies, the results are still generally lower than those achieved in earlier studies.

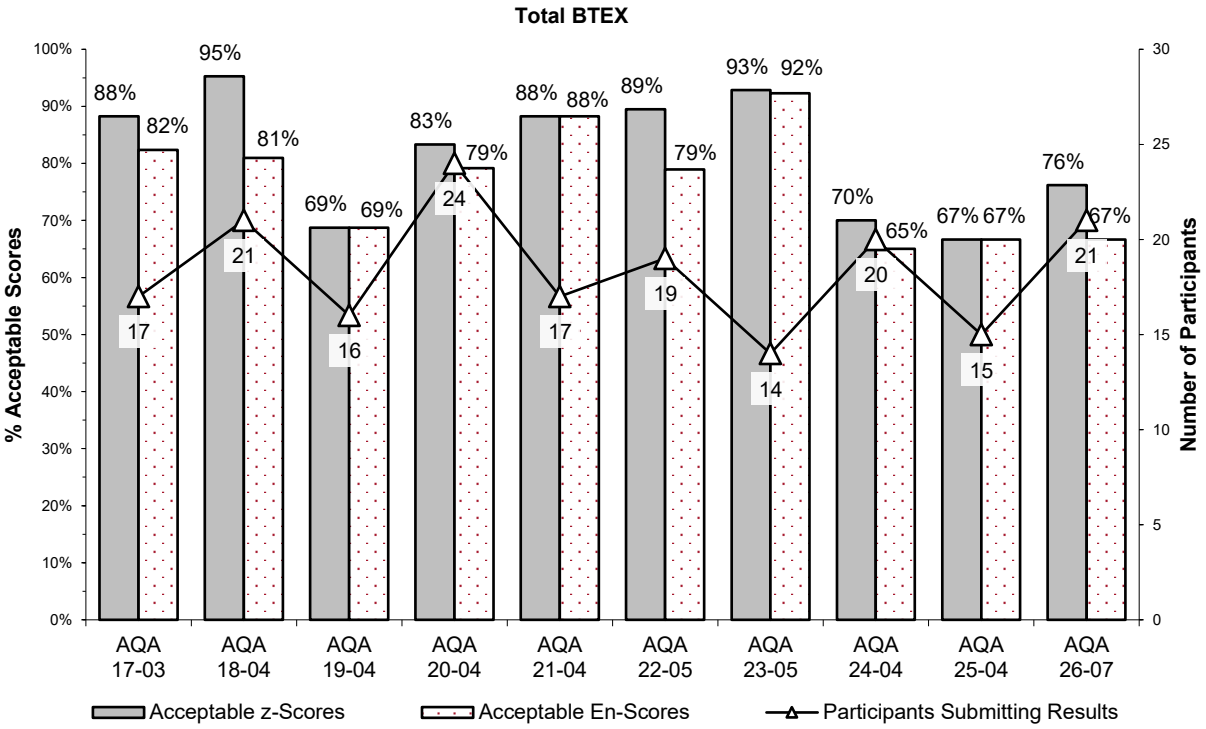


Figure 37 Participants' Performance for Total BTEX in NMIA Hydrocarbons in Soil PT Studies

PAHs

A summary of the acceptable performance (presented as a percentage of the total number of scores) obtained by participants for PAHs in soil over the last 10 studies (2017 – 2026) is presented in Figure 38. Over this period, the average proportion of acceptable *z*-scores was 91%, and the average proportion of acceptable E_n-scores was 86%. Participants' performance remains similar to previous studies.

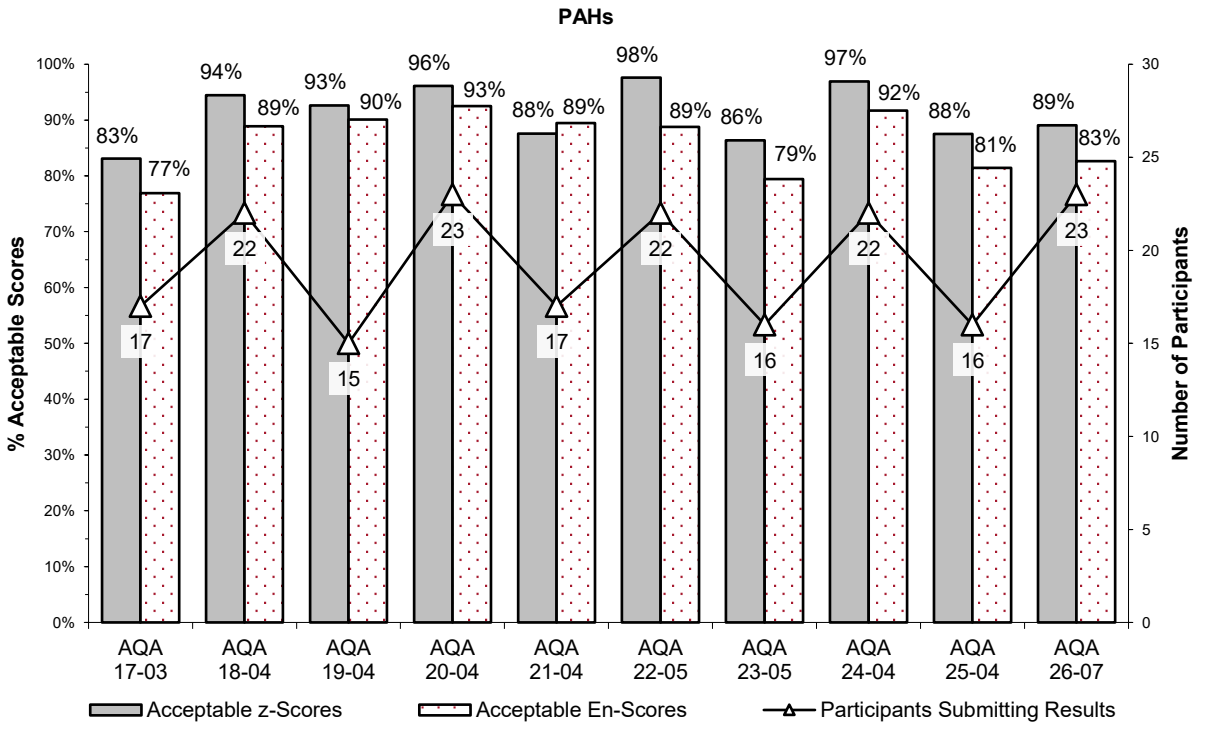


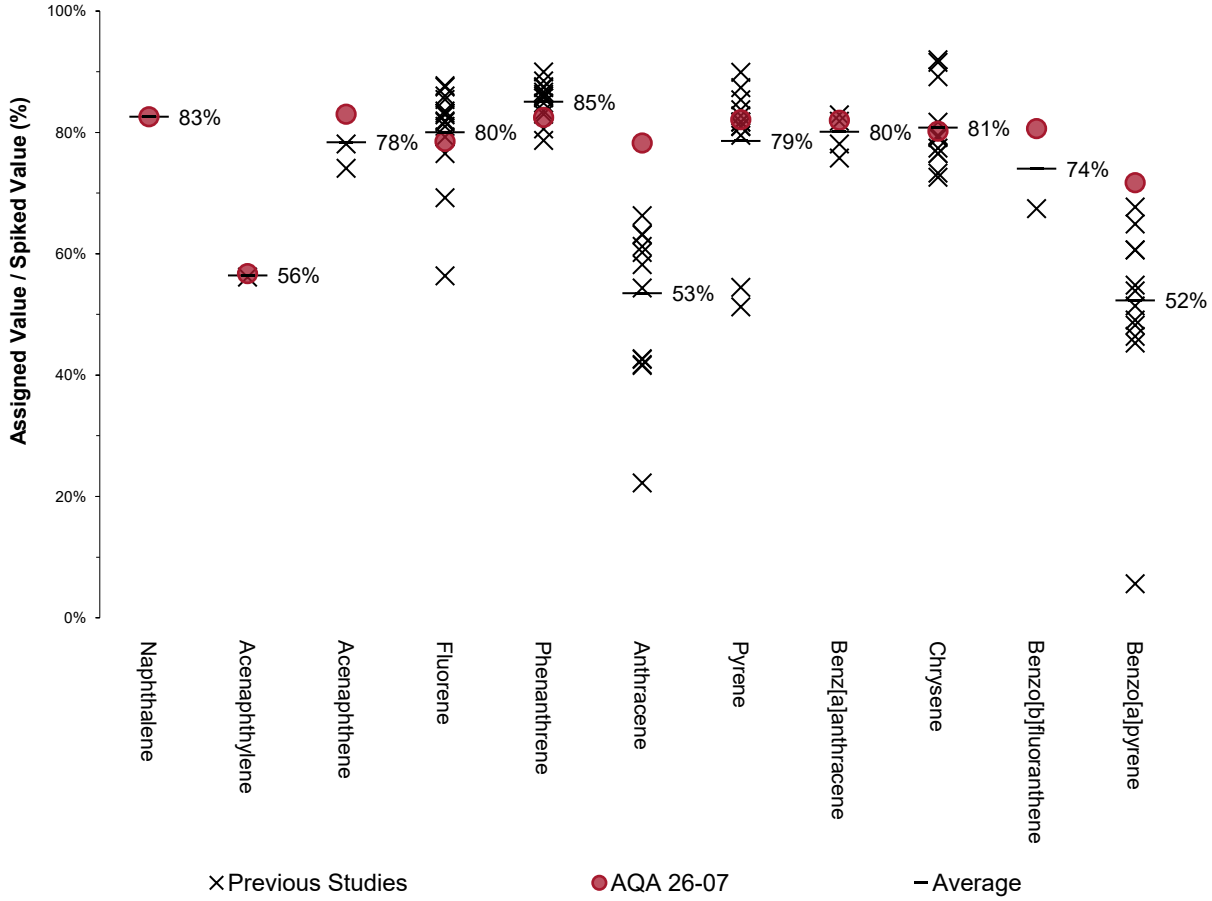
Figure 38 Participants' Performance for PAHs in NMIA Hydrocarbons in Soil PT Studies

A plot of the assigned value expressed as a percentage of the spiked value for PAHs in soil over the last 10 studies (2017 – 2026) is presented in Figure 39. Results from samples with other matrices have not been included as it has been previously seen that the nature of the matrix can substantially affect the recovery of some analytes.[14]

Throughout NMIA hydrocarbons in soil PT studies, acenaphthylene, anthracene and benzo[a]pyrene have consistently had lower results reported by participants as compared to the spiked values, averaging 56%, 53%, and 52% respectively for the robust average to spiked value. A high assigned value to spiked value ratio was achieved by participants in this study for anthracene (78%) and benzo[a]pyrene (72%) as compared to previous studies.

This was the first study to include naphthalene as a spiked analyte, and the assigned value to spiked value ratio for this analyte was 83%.

For most analytes, participants performed similarly or better as compared to previous studies.



For previous studies where no assigned value was set, the robust average has been used in calculations instead.

Figure 39 Recoveries of PAHs in Soil for NMIA Hydrocarbons in Soil PT Studies

Measurement Uncertainty

As discussed in Section 6.2, it is a requirement of ISO/IEC 17025 that laboratories report their uncertainties.[9] Figure 40 presents a summary of the relative uncertainties as reported by participants over the last 10 studies (2017 – 2026). On average per study, 92% of participants reported that they were accredited to ISO/IEC 17025. The vast majority of numeric results were reported with uncertainties, and most relative uncertainties were between 15% and 50%. However, around 14% of relative uncertainties were outside this range, and may have been unrealistically small or too large to be fit-for-purpose.

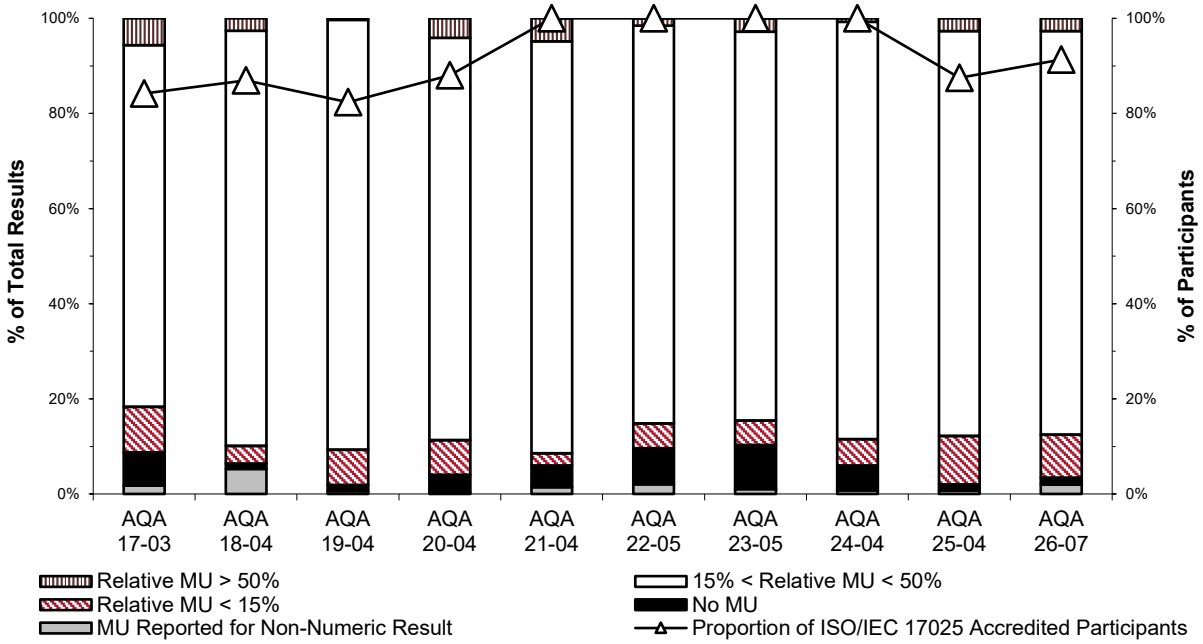


Figure 40 Summary of Participants' Relative Uncertainties for NMIA Hydrocarbons in Soil PT Studies

7 REFERENCES

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

[1] ISO/IEC 17043, *Conformity assessment – General requirements for the competence of proficiency testing providers*.

[2] Commonwealth of Australia, Department of Industry, Science and Resources, NMIA, 2026, *Study Protocol for Proficiency Testing*, viewed July 2026, <https://www.industry.gov.au/sites/default/files/2020-10/cpt_study_protocol.pdf>.

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[8] Thompson, M., 2000, 'Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing', *Analyst*, vol. 125, pp. 385-386.

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[11] US EPA, 2018, *Method 8270E: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, Revision 6, viewed July 2026, <https://www.epa.gov/sites/default/files/2020-10/documents/method_8270e_update_vi_06-2018_0.pdf>

[12] US EPA, 1984, *Method 610: Polynuclear Aromatic Hydrocarbons*, viewed July 2026, <https://www.epa.gov/sites/default/files/2015-10/documents/method_610_1984.pdf>

[13] JCGM 200:2012, *International vocabulary of metrology – Basic and general concepts and associated terms (VIM),* 3rd edition.

[14] NMIA, 2018, *Proficiency Test Report AQA 18-04 Hydrocarbons in Soil*.

APPENDIX 1 SAMPLE PREPARATION

A1.1 Diesel Fuel Preparation

Diesel fuel was purchased from a local retail outlet and treated to remove volatiles. The diesel was analysed to confirm that essentially all the hydrocarbons eluting before C_{10} had been removed. This same treated diesel fuel was used in previous NMIA hydrocarbon PT studies.

A1.2 Test Sample Preparation

For Samples S1, S2, and S3, soil from a Victorian farm which was dried and then sieved was used as the starting matrix.

Sample S1: Soil was spiked with sparged diesel and hydraulic fluid, before being divided into equal portions of 50 g each. The samples were stored at 4°C until dispatch.

Sample S2: Soil was spiked with unleaded petrol, sparged diesel, and toluene, before being scooped into jars and topped up to minimise vapour space. Samples were wrapped with gel ice and then stored at -20°C until dispatch.

Sample S3: Soil was spiked with PAHs, before being divided into equal portions of 50 g each. The samples were stored at 4°C until dispatch.

APPENDIX 2 ASSESSMENT OF HOMOGENEITY AND STABILITY

A2.1 Homogeneity

No homogeneity testing was completed for this study as the samples were prepared using a process previously demonstrated to produce sufficiently homogeneous samples. The results of this study also gave no reason to question the samples' homogeneity.

A2.2 Stability

No stability testing was completed for this study as the samples were prepared, stored, and dispatched using a process previously demonstrated to produce sufficiently stable samples for similar analytes and matrices over a similar time frame. After preparation and before dispatch, Samples S1 and S3 were stored in a refrigerator at 4°C, and Sample S2 was stored in a freezer at -20°C. For dispatch, samples were packaged into insulated polystyrene foam boxes with cooler bricks.

The results of this study also gave no reason to question the samples' transportation stability. Comparisons of results to days spent in transit for scored analytes are presented in Figures 41 to 53 (solid blue lines correspond to the assigned value ± U for each analyte; results have not been included here if they were excluded from all statistical calculations in Section 5).

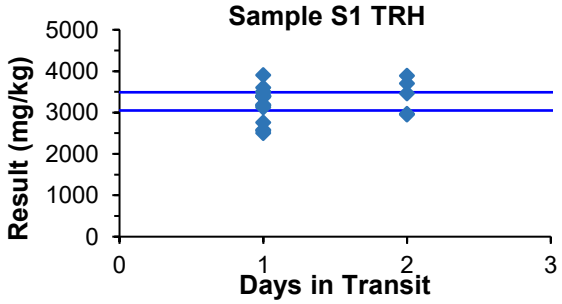

Figure 41 S1 TRH Results vs Transit Days

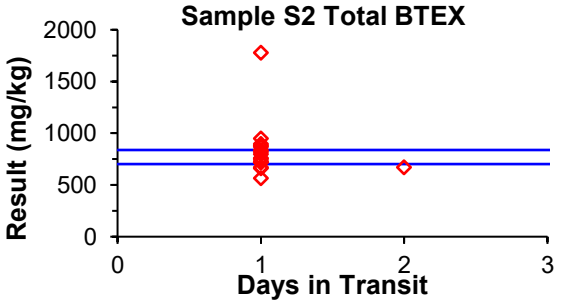

Figure 42 S2 Total BTEX Results vs Transit Days

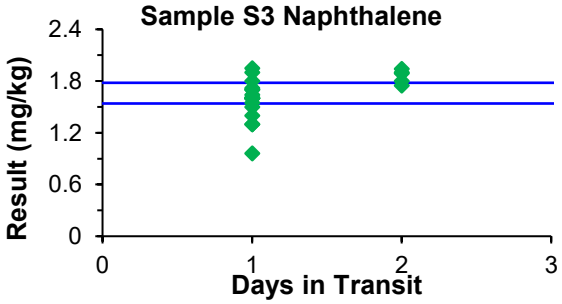

Figure 43 S3 Naphthalene Results vs Transit Days

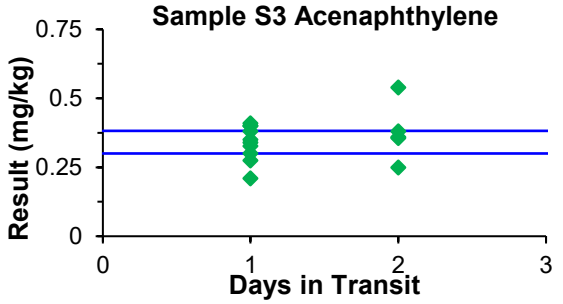

Figure 44 S3 Acenaphthylene Results vs Transit Days

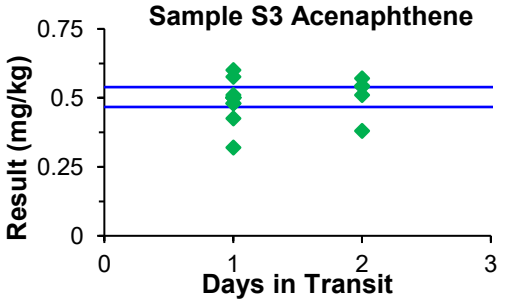

Figure 45 S3 Acenaphthene Results vs Transit Days

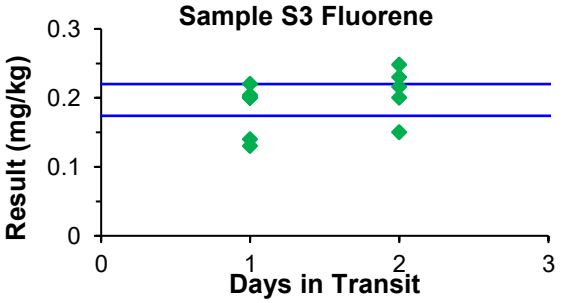

Figure 46 S3 Fluorene Results vs Transit Days

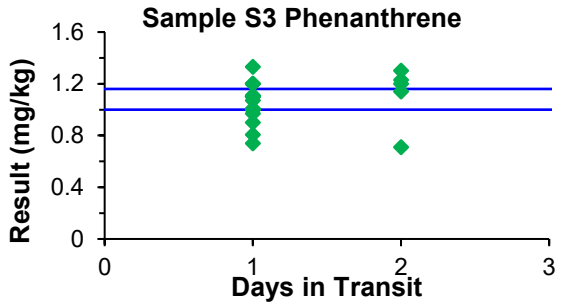

Figure 47 S3 Phenanthrene Results vs Transit Days

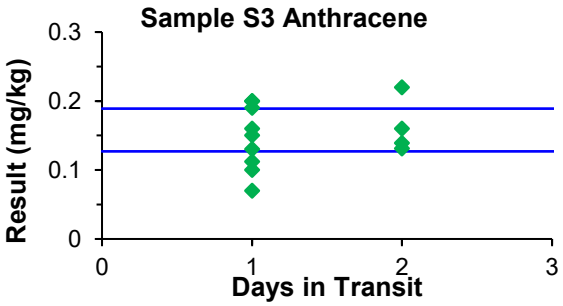

Figure 48 S3 Anthracene Results vs Transit Days

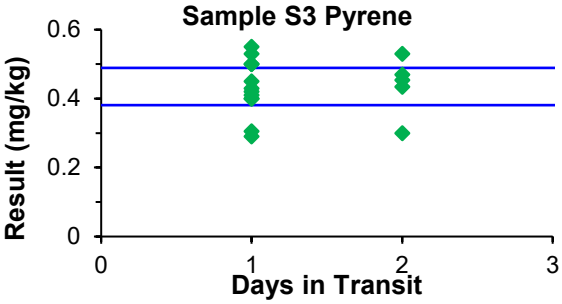

Figure 49 S3 Pyrene Results vs Transit Days

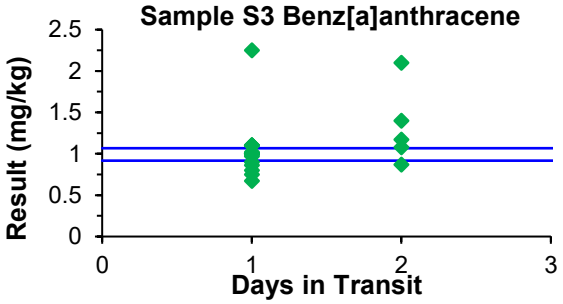

Figure 50 S3 Benz[a]anthracene Results vs Transit Days

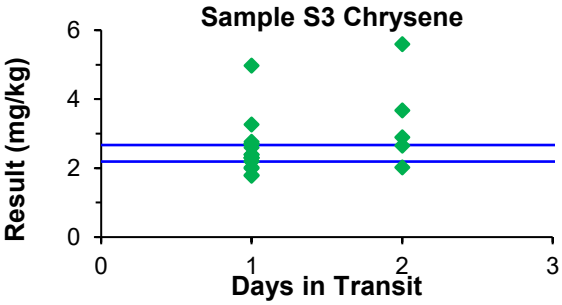

Figure 51 S3 Chrysene Results vs Transit Days

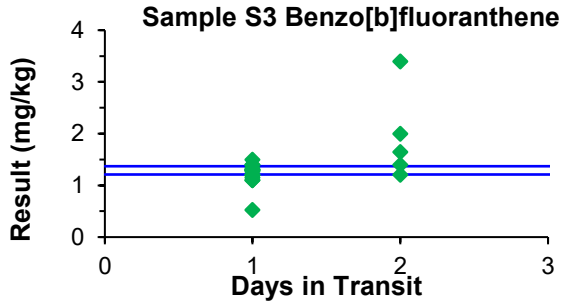

Figure 52 S3 Benzo[b]fluoranthene Results vs Transit Days

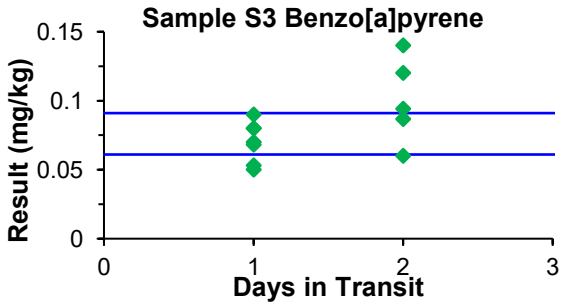

Figure 53 S3 Benzo[a]pyrene Results vs Transit Days

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

A3.1 Robust Average and Associated Uncertainty

Robust averages were calculated using the procedure described in ISO 13528.[7] The associated uncertainties were evaluated as according to Equation 4.

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

where:

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

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

p is the number of results

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

A worked example is set out below in Table 33.

Table 33 Uncertainty of the Robust Average for Sample S3 Acenaphthene

No. Results (p)	17
Robust Average	0.503 mg/kg
$S_{rob\ av}$	0.059 mg/kg
$u_{rob\ av}$	0.018 mg/kg
k	2
$U_{rob\ av}$	0.036 mg/kg

Therefore, the robust average for Sample S3 acenaphthene is 0.503 ± 0.036 mg/kg.

A3.2 z-Score and E_n-Score Calculations

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

A worked example is set out below in Table 34.

Table 34 z-Score and E_n-Score Calculation for Sample S3 Acenaphthene Result Reported by Laboratory 1

Participant Result (mg/kg)	Assigned Value (mg/kg)	Standard Deviation for Proficiency Assessment	z-Score	E_n-Score
0.51 ± 0.16	0.503 ± 0.036	15% as PCV, or: 0.15 × 0.503 = 0.07545 mg/kg	$z = \frac{0.51 - 0.503}{0.07545} = 0.09$	$E_n = \frac{0.51 - 0.503}{\sqrt{0.16^2 + 0.036^2}} = 0.04$

APPENDIX 4 TEST METHODS REPORTED BY PARTICIPANTS

Table 35 Measurement Methods and Instrumental Techniques for Sample S1 TRH

Lab. Code	Sample Mass (g)	Extraction Technique	Extraction Solvent	Extraction Time (min)	Extraction Volume and Number of Steps (if staggered extraction)	Extract Concentration Technique	Extract Concentration Time (min)	Extract Concentration Temperature (°C)	Clean-Up	Measurement Technique	Internal Standard Used
1	5	Solid-Liquid (ASE)	DCM	5	2	Evaporation under nitrogen	5	40	None	GC-FID	None
2	10	Solid-Liquid (Mechanical Agitation)	DCM:Acetone: Methanol	120	20mL, 1 step	N/A	N/A	N/A	N/A	GC-FID	N/A
3	10	Solid-Liquid (Mechanical Agitation)	1:1 DCM/Acetone	60	20mL	n/a	n/a	n/a	n/a	GC-FID	n/a
4	10	Solid-Liquid (Mechanical Agitation)	DCM/ACETONE	60	20 mL					GC-FID	
5	10	Solid-Liquid (Mechanical Agitation)	DCM:Acetone	60min	20ml,1 step					GC-FID	
6	2.5	Solid-Liquid (Mechanical Agitation)	1:1 Dichloromethane: Acetone	30	5 mL, single extraction	N/A	N/A	N/A	No clean-up	GC-FID	No ISTD
7	10	Solid-Liquid (Mechanical Agitation)	DCM:Acetone 1:1	60		None				GC-FID	None
8	5	Solid-Liquid (Mechanical Agitation)	50:50 DCM:Acetone	60	25ml	N/A	N/A	N/A	N/A	GC-FID	None
9	10	Solid-Liquid (Mechanical Agitation)	DCM:ACETONE	120	20mL, 1 step	NA	NA	NA	N/A	GC-FID	NA
10	10	Solid-Liquid (Mechanical Agitation)	Acetone:DCM (1:1 v/v)	60min	20ml				None	GC-FID	
11	10	Solid-Liquid (ASE)	DCM:Acetone	50	100mL	Evaporation under nitrogen	20	35	Silica	GC-FID	NA

Lab. Code	Sample Mass (g)	Extraction Technique	Extraction Solvent	Extraction Time (min)	Extraction Volume and Number of Steps (if staggered extraction)	Extract Concentration Technique	Extract Concentration Time (min)	Extract Concentration Temperature (°C)	Clean-Up	Measurement Technique	Internal Standard Used
12	10	Solid-Liquid (Mechanical Agitation)	DCM/Acetone	60	20 mL	N/A	N/A	N/A	N/A	GC-FID	N/A
13	10	Solid-Liquid (Sonication)	DCM/Acetone	15		NA	NA	NA	Silica	GC-FID	
14	10	Solid-Liquid (Sonication)	DCM:Acetone (1:1)	30	20ml	-	-	-	Silica	GC-FID	-
15	10	Solid-Liquid (Mechanical Agitation)	50:50 DCM:Acetone	60	20mL, 1 Step	NA	NA	NA	None	GC-FID	None
16	8	Solid-Liquid (Sonication)	Hex:Ace	50	25mL, Step 1	NA	NA	NA	Silica	GC-FID	NA
17	10	Solid-Liquid (Mechanical Agitation)	DCM:Acetone (1:1)	120	30	N/A	N/A	N/A	N/A	GC-MS/MS	N/A
18	10	Solid-Liquid (Mechanical Agitation)	DCM and ACETONE	1 hour	30 mL					GC-FID	N/A
19	10	Solid-Liquid (Mechanical Agitation)	Acetone:DCM (1:1 v/v)	60min	20ml				None	GC-MS/MS	
20	10	Solid-Liquid (Mechanical Agitation)	DCM/Acetone	120	20mL, 1 step	None	N/A	N/A	N/A	GC-FID	N/A
21	10	Solid-Liquid (Mechanical Agitation)	Hexane	60	20mL	None	None	None	Silica	GC-FID	None
23	10		Acetone/DCM						NA		

Table 36 Methodology Additional Information for Sample S1 TRH

Lab. Code	Methodology Additional Information
4	USEPA 8015
10	USEPA 8015
11	USEPA 8260
14	Method Reference: US EPA 8015

Table 37 Measurement Methods and Instrumental Techniques for Sample S2 BTEX

Lab. Code	Sample Mass (g)	Blank Subtracted?	Dilution? If yes, please write the dilution factor.	Measurement Technique	Internal Standard Used
1	5	no	100	P&T GC-MS	d6-benzene,d8-toluene,d10-ethylbenzene
2	5	No	10	P&T GC-MS	8260 Internal Standard Mix
3	5g	C6-C10 blank subtracted	no	P&T GC-MS	1,2-Dichlorobenzene-D4, 1,4-Difluorobenzene, Chlorobenzene-d5
4	10	No	100-fold		Fluorobenzene
5	5	No	800 and 8000	P&T GC-MS	Fluorobenzene
6	2.5	No		P&T GC-MS	1,4-Difluorobenzene, Chlorobenzene-d5
7	4	Yes		P&T GC-MS	Pentafluorobenzene, Difluorobenzene, d4-1,4-dichlorobenzne, d5-Chlorobenzene
8	5	Yes	1:25	P&T GC-MS	Surrogate
9	5	No	20	P&T GC-MS	various isotope labelled
10	5	No	100	P&T GC-MS	Fluorobenzene
12	10	No	Yes, for toluene. 100x dilution.	P&T GC-MS	Fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4
13	10	No		P&T GC-MS	
14	14	Yes	Yes, 10x	Headspace GC-MS	Toluene-d8
15	10	No	25	P&T GC-MS	Various
16	10	Yes	x10 & x100	Headspace GC-MS	yes, Fluorobenzene-(IS) & Chlorobenzene-d5-(IS)
17	5.574	No	x10 & x20 (for Toluene)	P&T GC-MS	1,4-difluorobenzene, chlorobenzene-d5 & naphthalene-d8
18	5	No		P&T GC-MS	
19	5	No	100	P&T GC-MS	Fluorobenzene

Lab. Code	Sample Mass (g)	Blank Subtracted?	Dilution? If yes, please write the dilution factor.	Measurement Technique	Internal Standard Used
20	5	No	Yes, x100 and x10	P&T GC-MS	8260 Internal Standards
23	10	No			

Table 38 Methodology Additional Information for Sample S2 BTEX

Lab. Code	Methodology Additional Information
4	USEPA 8260
6	2.5 g sample extracted into 5mL methanol, 250uL extract diluted to 40mL in H2O
7	Only hydrocarbons blank subtracted, not BTEX
10	USEPA 8260
14	Method Reference: US EPA 8260 & 5021

Table 39 Extraction Methodology for Sample S3 PAHs

Lab. Code	Sample Mass (g)	Extraction Technique	Extraction Solvent	Extraction Time (min)	Extraction Volume and Number of Steps (if staggered extraction)	Extract Concentration Technique	Extract Concentration Time (min)	Extract Concentration Temperature (°C)	Clean-Up
1	5	Solid-Liquid (ASE)	DCM:acetone 1:1	15	2	Evaporation under nitrogen	5	40	None
2	10	Solid-Liquid (Mechanical Agitation)	DCM:Acetone:Methanol	120	20mL, 1 step	N/A	N/A	N/A	N/A
3	10g	Solid-Liquid (Mechanical Agitation)	1:1 DCM/Acetone	60	20mL	n/a	n/a	n/a	n/a
4	10g	Solid-Liquid (Mechanical Agitation)	DCM/ACETONE	60	20 mL				
5	10	Solid-Liquid (Mechanical Agitation)	DCM:Acetone	60min	20ml,1 step				

Lab. Code	Sample Mass (g)	Extraction Technique	Extraction Solvent	Extraction Time (min)	Extraction Volume and Number of Steps (if staggered extraction)	Extract Concentration Technique	Extract Concentration Time (min)	Extract Concentration Temperature (°C)	Clean-Up
6	2.5	Solid-Liquid (Mechanical Agitation)	1:1 Dichloromethane:Acetone	30	5 mL, Single extraction	NA	NA	NA	No clean-up
7	10	Solid-Liquid (Mechanical Agitation)	DCM:Acetone 1:1	60		None			None
8	5	Solid-Liquid (Mechanical Agitation)	50:50 DCM:Acetone	60	25ml	N/A	N/A	N/A	N/A
9	10	Solid-Liquid (Mechanical Agitation)	DCM:ACETONE	120	20, 1 step	NA	NA	NA	NA
10	10	Solid-Liquid (Mechanical Agitation)	Acetone:DCM (1:1 v/v)	60min	20ml				None
11	10	Solid-Liquid (ASE)	DCM:Acetone	50	100mL	Evaporation under nitrogen	20	35	NA
12	10	Solid-Liquid (Mechanical Agitation)	DCM/Acetone	60	20 mL	N/A	N/A	N/A	N/A
13	10	Solid-Liquid (Sonication)	DCM/Acetone	15	NA	NA	NA	NA	Silica
14	10	Solid-Liquid (Sonication)	DCM:Acetone (1:1)	30	20ml	-	-	-	Silica
15	10	Solid-Liquid (Mechanical Agitation)	50:50 DCM:Acetone	60	20mL, 1 Step	NA	NA	NA	None
16	8	Solid-Liquid (Sonication)	Hex:Ace	60	25mL, Step 1	NA	NA	NA	NA
17	10	Solid-Liquid (Mechanical Agitation)	DCM:Acetone (1:1)	120	30	N/A	N/A	N/A	N/A

Lab. Code	Sample Mass (g)	Extraction Technique	Extraction Solvent	Extraction Time (min)	Extraction Volume and Number of Steps (if staggered extraction)	Extract Concentration Technique	Extract Concentration Time (min)	Extract Concentration Temperature (°C)	Clean-Up
18	10	Solid-Liquid (Mechanical Agitation)	DCM and ACETONE	1 hour					
19	10	Solid-Liquid (Mechanical Agitation)	Acetone:DCM (1:1 v/v)	60min	20ml				None
20	10	Solid-Liquid (Mechanical Agitation)	DCM/Acetone	120	20mL, 1 step	None	N/A	N/A	N/A
21	2	Solid-Liquid (Mechanical Agitation)	Ethyl Acetate	30	15mL in 2 steps (10mL + 5mL)	Rotovap		38	None
23	10		Acetone/DCM						

Table 40 Measurement Methods and Instrumental Techniques for Sample S3 PAHs

Lab. Code	Measurement Technique	Type of Internal Standard Used	Internal Standard Used
1	GC-MS/MS	PAHs	acenapthene-d10,chrysene d12,napthalene-d8,phenanthrene-d10perylene-d12
2	GC-MS	PAHs	8270 Internal Standard Mix
3	GC-MS	PAHs	EPA 8270 Semivolatile Internal Standard Mix
4	GC-MS/MS	PAHs	Naphthalene-d8, Acenaphthene-d10, Chrysene-d12 and Perylene-d12
5	GC-MS/MS		Naphthalene-D8,Acenaphthene-D10,Chrysene-D12,Perylene-D12
6	GC-MS/MS	PAHs	Naphthalene-d8, Phenanthrene-d10, Chrysene-d12, Perylene-d12
7	GC-MS	PAHs	Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
8	GC-MS/MS	PAHs	Accustandard
9	GC-MS	PAHs	Deuterated PAHs
10	GC-MS/MS	Deuterated PAHs internal standards	Naphthalene-d8, Acenaphthene-d10, Chrysene-d12 and Perylene-d12
11	GC-MS/MS		Acenaphthene-d10, Chrysene-d12
12	GC-MS	N/A	d8-Naphthalene, d10-Acenaphthalene, d10-Phenanthrene, d12-Chrysene, d12-Perylene

Lab. Code	Measurement Technique	Type of Internal Standard Used	Internal Standard Used
13	GC-MS		
14	GC-MS/MS	Deuterated PAHs	Mixture of purchased internal standards
15	GC-MS	PAHs	Various
16	GC-MS	PAHs	Naphthalene-d8-(IS), Acenaphthene-d10-(IS), Phenanthrene-d10-(IS) & Chrysene-D12-(IS)
17	GC-MS/MS	deuterated internal standards (ISTDs)	1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12
18	GC-MS		Semi volatile Internal standard
19	GC-MS/MS	Deuterated PAHs internal standards	Naphthalene-d8
20	GC-MS/MS	PAHs	8270 Internal Standards
21	GC-MS/MS	PAHs	Phenanthrene-d10
23			

Table 41 Methodology Additional Information for Sample S3 PAHs

Lab. Code	Methodology Additional Information
4	USEPA 8270
10	USEPA 8270
11	USEPA 610
13	Laboratory method is unable to report Benzo[b]fluoranthene and Benzo[k]fluoranthene individually. Reported result for Benzo[b]fluoranthene is reported as Benzo[b,j,k]fluoranthene
14	Method Reference: US EPA 8270
21	Benzo[b]fluoranthene is reported as total of Benzo[b+k]fluoranthene.

APPENDIX 5 ACRONYMS AND ABBREVIATIONS

ACE	Acetone
AV	Assigned Value
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 Detection
GC	Gas Chromatography
GUM	Guide to the expression of Uncertainty in Measurement
HEX	Hexane
HS	Headspace (GC)
IEC	International Electrotechnical Commission
ISO	International Standards Organization
k	Coverage Factor
LOR	Limit Of Reporting
Max	Maximum
Md	Median
MeOH	Methanol
Min	Minimum
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
NMIA	National Measurement Institute Australia
NR	Not Reported
NT	Not Tested
P&T	Purge and Trap (GC)
PAH	Polycyclic Aromatic Hydrocarbon
PCV	Performance Coefficient of Variation
PT	Proficiency Testing

RA	Robust Average
RM	Reference Material
SD	Standard Deviation
SDPA	Standard Deviation for Proficiency Assessment
SI	International System of Units
SLE	Solid-Liquid Extraction
SLE (ASE)	Solid-Liquid Extraction (Accelerated Solvent Extraction)
SLE (MA)	Solid-Liquid Extraction (Mechanical Agitation)
SLE (S)	Solid-Liquid Extraction (Sonication)
SS	Spiked Samples
SV	Spiked Value, or formulated concentration of a PT sample
TRH	Total Recoverable Hydrocarbons
U	Expanded Uncertainty
US EPA	United States Environmental Protection Agency

END OF REPORT