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National Measurement Institute

Proficiency Test Report AQA 19-12 Metals, Anions and Nutrients in Soil

November 2019

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

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

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Accredited for compliance with ISO/IEC 17043

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1 SUMMARY

This report presents the results of the proficiency test AQA 19-12 Metals, Anions and Nutrients in Soil. The study focused on the measurement of acid extractable elements: Al, As, Ba, Be, Ca, Cd, Co, Cr, Cs, Cu, Fe, Ga, Hg, K, La, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Rb, S, Sb, Se, Sn, Sr, Th, U, V and Zn. Measurement of pH, electrical conductivity (EC), water soluble chloride (Cl^-), fluoride (F^-), sulphate (SO_4^{2-}), orthophosphate-P (PO_4^{3-}-P) and total Kjeldahl nitrogen and 2M KCl extractable ammonium nitrogen (NH_4^+-N) and 2M KCl extractable nitrate nitrogen (NO_3^--N) were also included in the program.

The sample set consisted of three dried soil samples.

Twenty five laboratories registered to participate and twenty four submitted results.

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

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

i. *compare the performance of participant laboratories and assess their accuracy;*

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

Of 721 results, 639 (89%) returned a satisfactory score of $|z| \leq 2$.

Of 721 E_n-scores, 538 (75%) were satisfactory with $| E_n | \leq 1$.

ii. *evaluate the laboratories 'methods used in determination of inorganic analytes in soil;*

The request was for acid extractable elements. An aggressive digestion regime that involves a digestion temperature of 170°C can facilitate metal extraction from the fraction which is naturally present in the soil matrix. Alternatively, weak digestion regimes which involve an extraction time of only 30 minutes extract just a fraction of the soil contaminants. When an assessment is made of the anthropogenic impact of metal content in a soil sample, aggressive or weak digestion regimes can produce misleading results.

iii. *evaluate within laboratory reproducibility;*

Sample S3 of the present study was distributed as Sample S3 of AQA 19-02. Although the assigned values set for the two samples were not significantly different, in some cases, the reported results varied greatly.

iv. *compare the performance of participant laboratories with their past performance;*

On average participants' performance remained fairly consistent.

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

Of 748 numerical results, 615 (82%) were reported with an expanded measurement uncertainty. An example of estimating measurement uncertainty using the proficiency testing data only is given in Appendix 3.

2 INTRODUCTION

2.1 NMI Proficiency Testing Program

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

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

- inorganic analytes in soil, water, food and pharmaceuticals;
- pesticide residues in fruit and vegetables, soil and water;
- petroleum hydrocarbons in soil and water;
- PFAS in water, soil, biota and food;
- allergens in food;
- controlled drug assay; and
- folic acid in flour.

AQA 19-12 is the 25th NMI proficiency study of inorganic analytes in soil.

2.2 Study Aims

The aims of the study were to:

- compare the performance of participant laboratories and assess their accuracy;
- evaluate the laboratories' methods used in determination of inorganic analytes in soil;
- evaluate within-laboratory reproducibility;
- compare the performance of participant laboratories with their past performance; and
- develop the practical application of traceability and measurement uncertainty.

2.3 Study Conduct

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

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

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

3 STUDY INFORMATION

3.1 Selection of Matrices and Inorganic Analytes

The 53 tests were selected from those for which an investigation level is published in the Guideline on the Investigation Levels for Soil and Groundwater, promulgated by the National Environmental Protection Council (NEPC)[5] and from analytes commonly measured in soil.

3.2 Participation

Twenty-five laboratories participated and twenty-four submitted results.

The timetable of the study was:

Invitation issued:	07 August 2019
Samples dispatched:	26 August 2019
Results due:	23 September 2019
Interim report issued:	24 September 2019

3.3 Test Material Specification

Three samples were provided for analysis:

- Sample S1 was 30 g of dried low level contaminated soil;
- Sample S2 was 25 g of dried sediment; and
- Sample S3 was 75 g of dried agricultural soil, previously distributed as Sample S3 of proficiency testing study AQA 19-02.[6]

3.4 Laboratory Code

All participant laboratories were assigned a confidential code number.

3.5 Sample Preparation, Analysis and Homogeneity Testing

Test samples from previous studies have been demonstrated to be sufficiently homogeneous for evaluation of participants' performance.[6, 7] Therefore, only a partial homogeneity test was conducted for all elements in S1 and S2 as the same preparation procedure was followed as in previous studies.[1] The results from the partial homogeneity testing for these samples are reported in the present study as the homogeneity value.

A full homogeneity test was conducted for Sample S3, with the exception of 2M KCl extractable ammonium nitrogen (NH_4^+-N) and 2M KCl extractable nitrate nitrogen (NO_3^--N). Sample S3 was demonstrated to be sufficiently homogeneous for the evaluation of participants' performance.

The preparation, analysis and homogeneity testing of the study samples are described in Appendix 1.

3.6 Stability of Analytes

No stability study was carried out in the present study. Stability studies conducted for the previous proficiency tests of inorganic analytes in soil found no significant changes in any of the analytes' concentration.[6, 7]

3.7 Sample Storage, Dispatch and Receipt

The samples were dispatched by courier on 26 August 2019.

A description of the test samples and instructions for participants, and a form for participants to confirm the receipt of the test samples, were sent with the samples.

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

3.8 Instructions to Participants

Participants were instructed as follows:

- Quantitatively analyse the samples using your normal test method.
- For S3 determination of the 2M KCl extractable ammonium nitrogren (NH_4^+-N) and nitrate nitrogen (NO_3^--N), participants are asked to follow the procedure described below:

  "Prepare a 1:10 w/v soil/2M KCl extracting solution. For example, weigh 5 g of soil into a suitable bottle or jar and add 50 mL of 2M KCl extracting solution. Mechanically shake (end-over-end preferred), at room temperature for 1 h. Allow around 20-30 min for soil to settle and clarify and then take a known aliquot for the measurement technique employed. Further dilution of the aliquot may be required."

Measure the analytes using a colorimetric method; and to report results of 1:10 soil/2M KCl extracting solution on as received basis in units of mg/kg for: 2M KCl extractable ammonium-N (NH_4^+-N) and 2M KCl extractable nitrate-N (NO_3^--N).

- Report results for all the other tests in S1, S2 and S3 on a as received basis in units of mg/kg.

SAMPLE S1 (soil)		SAMPLE S2 (sediment)		SAMPLE S3 (agricultural soil)	
Test Acid Extractable	Approximate Conc. Range mg/kg	Test Acid Extractable	Approximate Conc. Range mg/kg	Test	Approximate Conc. Range mg/kg
As	0.5-20	Al	2500-50000	Ca (acid extractable)	500-20000
Ba	25-1000	As	2.5-100	Fe (acid extractable)	500-20000
Be	<5	Cd	5-200	K (acid extractable)	50-2000
Ca	25-1000	Co	5-200	Mg (acid extractable)	50-2000
Co	1-40	Cr	5-200	Na (acid extractable)	25-1000
Cr	1-40	Cu	5-200	P (acid extractable)	50-1000
Cs	<5	Hg	2.5-100	S (acid extractable)	50-1000
Cu	1-40	Mn	250-1000	Sr (acid extractable)	10-200
Ga	1-40	Mo	5-200	Water Soluble Chloride (Cl^-)- 1:5 soil/water extract	10-200
La	1-40	Ni	5-200	Water Soluble Fluoride (F^-)- 1:5 soil/water extract	0.5-10
Li	0.5-20	Pb	5-200	Water Soluble Sulphate (SO_4^{2-}) - 1:5 soil/water extract	25-300
Mn	25-500	Sb	2.5-100	Water Soluble Orthophosphate-P (PO_4^{3-} -P) - 1:5 soil/water extract	0.5-10
Ni	1-40	Se	2.5-100	pH of 1:5 soil/water suspension	>3
P	5-250	Th	2.5-100	EC of 1:5 soil/water extract (µS/cm)	>250
Pb	1-40	U	<5	2M KCl Extractable (Nitrate-N)	NA
Rb	1-40	V	5-200	2M KCl Extractable (Ammonium-N)	NA
Se	<5	Zn	5-200	Total Kjeldahl nitrogen (TKN)	NA
Sn	<5				
Zn	1-40				

NA = Not Available

- Report results as you would report to a client.
- For each analyte in each sample, report the expanded measurement uncertainty associated with your analytical result (eg 5.21 ± 0.51 mg/kg). Report the basis of your uncertainty estimates (i.e. control charts, proficiency testing).
- Please send us all the requested details regarding the test method.
- Return the completed results sheet by 23 September 2019.

3.9 Interim Report

An interim report was emailed to participants on 24 September 2019.

4 PARTICIPANT LABORATORY INFORMATION

4.1 Test Method Summaries

Summaries of test methods are transcribed in Tables 1 to 8.

Table 1 Methodology for Acid Extractable Elements

Lab. Code	Method Reference	Sample Mass (g)	Digestion Temp. (ºC)	Digestion Time (min)	Vol. HNO_3 (mL)	Vol. HNO_3 (1:1) (mL)	Vol. HCl (mL)	Vol. HCl (1:1) (mL)	Vol. H_2O_2 (mL)	Other (mL)
1	EPA (Environmental Protection Agency) 1994 Method 200.8	2	109	60	800		400			1200
2	USEPA 200.8	1	95	120	2.5		2.5			10
3[a]	Soil Chemmical Methods - Australasia (Rayment &Lyons) method 17B1	3	95	120	22.5		7.5			
4	US EPA 3050B	0.5	95	120	7.5		5		1.5	
5	In House S6 - referencing APHA 3125	0.4	120	60	2.5		7.5			
6	USEPA methods 3050B, 3051A, 6020B	2	95	60	4		12			4 (H_2O)
7	AS 4479.2-1997, AS4479.4-1999	0.5	95	120	1		3			
8	EPA3050B, 6020B	2	90-95	60	4		12			
9	USEPA METHOD 3010, USEPA METHOD 6020	2	98	60	4		12			4 (H_2O)
10	USEPA Method 3050	0.5	85	240	5		5			
11	USEPA3050	1	98	150	5		5			
12	US EPA 200.2 adaption for Hot Block	0.5	85-95	30	3		3		2	
13		0.5	105	120	2		1			
14	USEPA method 200.2 Revision 2.8	1	95	60		2		10	2	
15	EPA Method 3050B Acid Digestion of Sediments, Sludge and Soils	0.5	85	240	5		5			
16	USEPA method 6020 – ICP-MS	1	95	120	7.5		2.5			
17		1	105	120	3		3			
19	USEPA 3051A (Modification)	1	170	15		8		2		
20	In House	5.4	105	90	5		5		5	
21	US-EPA Method 200.2	1	95	50	2		2			10 (H_2O)
23	USEPA-6010C (Except Mercury by USEPA-7471B)	1	95		5		5		3	
24	USEPA200.7	1	95	30		5		2		
25	USEPA 200.8	1.2	95	30	2.5		2.5			

[a]Additional information in Table 8

Table 2 Methodology for Total Kjeldahl Nitrogen

Lab. Code	Method Reference	Digestion	Distillation	Measurement Method	Instrument
2	USEPA Method 351.2, Rayment & Lyons Method 7A2	Yes	No	Colorimetric - salicylate method	DA
3	In-house method based on APHA 23rd edition 4500-Norg B	Yes	Yes	Colorimetric - salicylate method	DA
5	In house S4a	No	No	TKN = TN-NOx (Dumas)	LECO
6	APHA Method 4500-Norg D	Yes		Colorimetric - salicylate method	FIA
11				TKN = TN-NOx (Dumas)	LECO
14	APHA, 4500-P J. & 4500-N C.	Yes			DA
16	APHA 4500-Norg-D and Thermo Scientific Method D08727 and NEMI METHOD ID: 9171	Yes		Colorimetric - salicylate method	FIA
17		Yes	Yes	Titrimetric method	Manual Analysis
19	APHA Method 4500-N Org B and Method 4500-NH3 F	Yes	Yes	Colorimetric - phenate method	DA

Table 3 Methodology for 2M KCl Extractable Ammonium-N and Nitrate-N

Lab. Code	Method Reference		Sample Mass (g)	Extraction Solution 2M KCl Volume (mL)	Shake time (hours)	Measurement Method		Measurement Instrument	
	NH_4^+-N	NO_3^--N				NH_4^+-N	NO_3^--N	NH_4^+-N	NO_3^--N
2[a]	R&L Method 7C2	R&L Method 7C2	1	10	1	Colorimetric – Phenate method	Colorimetric-Sulfanilamide-NEDD Cd reduction	DA	SFA
3	Soil Chemical Methods, Rayment & Lyons method 7C2	Soil Chemical Methods, Rayment & Lyons method 7B1	5	50	1	Colorimetric – Salicylate method	Colorimetric – vanadium III method	DA	DA
5	In House S37	In House S37	2	20	1	Colorimetric – Salicylate method	Colorimetric – vanadium III method	FIA	FIA
6	APHA 4500 NH3 G	APHA 4500 NO3 I	5	50	1	Colorimetric – Phenate method	Colorimetric-Sulfanilamide-NEDD Cd reduction	FIA	FIA
8	APHA 4500-NH3 F		5	50	1	Colorimetric – Phenate method		DA	
11	R&L	R&L	2	20	1	Colorimetric – Salicylate method	Colorimetric-Sulfanilamide-NEDD Cd reduction	DA	DA
14			5	50	1	Colorimetric – Phenate method		FIA	

Table 3 Methodology for 2M KCl Extractable Ammonium-N and Nitrate-N (continued)

Lab. Code	Method Reference		Sample Mass (g)	Extraction Solution 2M KCl Volume (mL)	Shake time (hours)	Measurement Method		Measurement Instrument	
	NH_4^+-N	NO_3^-N				NH_4^+-N	NO_3^--N	NH_4^+-N	NO_3^-N
17			5	50	1	SFA	Colorimetric-Sulfanilamide-NEDD Cd reduction	SFA	FIA
18	KCl	H2O						DA	DA
19[a]	7C2a	7B1a	3	30	1	Colorimetric – Salicylate method	Colorimetric-Sulfanilamide-NEDD hydrazine reduction	SFA	SFA

[a]Additional information in Table 8

Table 4 Methodology for Water Soluble Cl^-

Lab. Code	Method Reference	Sample Mass (g)	Water Volume (mL)	Shake Time (hours)	Measurement Method	Measurement Instrument
2[a]	R&L Method 5A2	20	100	1	Ferricyanide Colorimetric Method	DA
3	Soil Chemical Methods, Rayment & Lyons	40.08	200	1	Potentiometric Method	Titration
4	APHA 4110 B				Ion Chromatographic Method	IC
5		2	10	1	Ion Chromatographic Method	IC
6	APHA 4500 Cl E	10	50	0.5	Ferricyanide Colorimetric Method	Gallery
8	APHA 4110C	8	40	1	Ion Chromatographic Method	IC
11		10	50	1	Ferricyanide Colorimetric Method	DA
14		15	75	1	Ion Chromatographic Method	IC
15		10	50	1	Ferricyanide Colorimetric Method	DA
16	APHA 4500-Cl E	10	50	1	Mercuric Thiocyanate	DA
17		10	50	1	Ion Chromatographic Method	IC
18					1:5 Water	DA
19	APHA 4110 B	10	50	1	Ion Chromatographic Method	IC

[a]Additional information in Table 8

Table 5 Methodology for Water Soluble F^-

Lab. Code	Method Reference	Sample Mass (g)	Water Volume (mL)	Shake Time (hours)	Measurement Method	Measurement Instrument
2	APHA 4500-F	20	100	1	Ion Selective Electrode Method	Fluoride Selective Electrode
3	Soil Chemical Methods, Rayment & Lyons	40.08	200	1	Ion Selective Electrode Method	Fluoride Selective Electrode
5		2	10	1	Ion Chromatographic Method	IC
6	APHA 4500 F C	10	50	0.5	Ion Selective Electrode Method	Fluoride Selective Electrode
14		15	75	1	Ion Chromatographic Method	IC
16	APHA 4500-F A&C	10	50	1	Ion Selective Electrode Method	Fluoride Selective Electrode
17		10	50	1	Ion Chromatographic Method	IC
19	APHA 4500-F C	10	50	1	Ion Selective Electrode Method	Fluoride Selective Electrode

Table 6 Methodology for Water Soluble SO_4^{2-}

Lab. Code	Method Reference	Sample Mass (g)	Water Volume (mL)	Shake Time (hours)	Measurement Method	Measurement Instrument
2	APHA 3120	20	100	1	ICP Method	ICP-OES
3	Soil Chemical Methods, Rayment & Lyons	40.08	200	1	ICP Method	ICP-OES
4	APHA 4110B				Ion Chromatographic Method	IC
5		2	10	1	ICP Method	ICP-OES
6	APHA 4500 SO4	10	50	0.5	Turbidimetric Method	Gallery
8	APHA 4110C	8	40	1	Ion Chromatographic Method	IC
11		10	50	1	ICP Method	ICP-OES
14		15	75	1	Ion Chromatographic Method	IC
16	APHA 4500-SO4-E	10	50	1	Turbidimetric Method	DA
17		10	50	1	Ion Chromatographic Method	IC
19	APHA 4110B	10	50	1	Ion Chromatographic Method	IC

Table 7 Methodology for Water Soluble Orthophosphate-P

Lab. Code	Method Reference	Sample Mass (g)	Water Volume (mL)	Shake Time (hours)	Measurement Method	Measurement Instrument
2	APHA 4500-P	20	100	1	Ascorbic Acid Colorimetric Method	DA
3		40.08	200	1	Ascorbic Acid Colorimetric Method	DA
5		2	10	1	Vanadomolybdophosphoric Colorimetric Method	FIA
6	APHA 4500 P E	10	50	0.5	Ascorbic Acid Colorimetric Method	Gallery
8	APHA 4500-P E	8	40	1	Ascorbic Acid Colorimetric Method	DA
11		10	50	1	Other	ICP-OES
14		15	75	1	Other	DA
16	APHA 4500-P B	10	50	1	Ascorbic Acid Colorimetric Method	DA
17		10	50	1	Ascorbic Acid Colorimetric Method	DA
19*	APHA 4500-P G	10	50	1	Ascorbic Acid Colorimetric Method	FIA

[a]Additional information in Table 8

4.2 Instruments Used for Measurements

The instruments and settings used by participants for acid extractable elements are presented in Appendix 4.

4.3 Additional Information

Participants had the option to report additional information for each sample analysed. These are transcribed in Table 8.

Table 8 Additional information

Lab. Code	Additional Information
2	Extractable N: R&L = G.E. Rayment and D.J.Lyons,Soil Chemical Methods - Australasia, CSIRO Publishing, 2011 Water Soluble Anions: R&L = G.E. Rayment and D.J.Lyons,Soil Chemical Methods - Australasia, CSIRO Publishing, 2011
3	Acid Extractable elements: "Arsenic (following extraction) was analysed by hydried generation on AAS Mercury is analysed by in-house method based on USEPA method 245.7 using FIMS"
4	S2: Estimated result for Sb is 5 mg/Kg
19	S3: Insufficient sample to complete fluoride analysis. Extractable N: Method reference - Soil Chemical Methods Australasia, G. E. Rayment & David J Lyons

4.4 Basis of Participants' Measurement Uncertainty Estimates

Participants were requested to provide information about the basis of their uncertainty estimates (Tables 9 and 10).

Table 9 Basis of Uncertainty Estimate

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation		Guide Document for Estimating MU
		Precision[a]	Method Bias[a]	
1	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples – RM Duplicate Analysis	Recoveries of SS	
2	Professional Judgement			
3	Top Down - precision and estimates of the method and laboratory bias	Control Samples – SS Duplicate Analysis Instrument Calibration	Recoveries of SS	NATA 2018, Measurement Uncertainty of Chemical Test Results
4	Top Down - precision and estimates of the method and laboratory bias	Control Samples – CRM Duplicate Analysis	CRM Recoveries of SS Instrument Calibration	NATA 2018, Measurement Uncertainty of Chemical Test Results
5	Top Down - precision and estimates of the method and laboratory bias	Control Samples – RM Duplicate Analysis	Instrument Calibration	Nordtest Report TR537
6	Top Down - precision and estimates of the method and laboratory bias	Control Samples – CRM	CRM Recoveries of SS Instrument Calibration	NATA 2018, Measurement Uncertainty of Chemical Test Results
7	Calculated from Standard deviation and concentration of long term in house QC samples	Control Samples – RM Duplicate Analysis	CRM Laboratory bias from PT studies Recoveries of SS	NATA 2018, Measurement Uncertainty of Chemical Test Results
8*	See Table 12	Control Samples Duplicate Analysis Instrument Calibration	CRM	See Table 12
9	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples - CRM		NATA 2018, Measurement Uncertainty of Chemical Test Results
10	Top Down - precision and estimates of the method and laboratory bias	Control Samples – CRM Duplicate Analysis	CRM Laboratory bias from PT studies Recoveries of SS Instrument Calibration Standard Purity Variation in sample moisture content	NATA 2018, Measurement Uncertainty of Chemical Test Results
11	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples - RM Duplicate Analysis	CRM Laboratory bias from PT studies	NATA 2018, Measurement Uncertainty of Chemical Test Results
12	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples – CRM Duplicate Analysis Instrument Calibration	CRM Recoveries of SS Instrument Calibration	NATA 2018, Measurement Uncertainty of Chemical Test Results
13			CRM	
14*	Top Down - precision and estimates of the method and laboratory bias	Control Samples – RM Duplicate Analysis Instrument Calibration	CRM Laboratory bias from PT studies Instrument Calibration	NATA 2018, Measurement Uncertainty of Chemical Test Results

15	Top Down - precision and estimates of the method and laboratory bias	Control Samples – SS Duplicate Analysis	Laboratory bias from PT studies	NATA 2018, Measurement Uncertainty of Chemical Test Results
16	Top Down - precision and estimates of the method and laboratory bias	Control Samples – SS Duplicate Analysis	CRM Recoveries of SS	Eurachem/CITAC Guide
17	Top Down - precision and estimates of the method and laboratory bias	Control Samples Duplicate Analysis	CRM Recoveries of SS	Nordtest Report TR537
19	Top Down - precision and estimates of the method and laboratory bias	Control Samples – CRM Duplicate Analysis	CRM	NMI Uncertainty Course
20	Top Down - precision and estimates of the method and laboratory bias	Control Samples – RM	CRM	NATA 2018, Measurement Uncertainty of Chemical Test Results
21	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples – CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration	Eurachem/CITAC Guide
23	Top Down - precision and estimates of the method and laboratory bias	Control Samples – CRM Duplicate Analysis	CRM Recoveries of SS Instrument Calibration	NMI Uncertainty Course
24	Bottom Up (ISO/GUM, fish bone/ cause and effect diagram)	Control Samples – CRM Duplicate Analysis	Laboratory bias from PT studies	NATA 2018, Measurement Uncertainty of Chemical Test Results

[a] RM = Reference Material, CRM = Certified Reference Material, SS =Spiked samples; * Additional Information in Table 10

Table 10 Additional Information for Basis of Uncertainty Estimate

Lab. Code	Additional Information
8	Estimation of MU from within-laboratory data on bias and precision has been calculated by using the procedures outlined in ASTM E2554-13 Standard Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques
14	MU Calculation Pack based on QC Data

4.5 Participant Comments on this PT Study or Suggestions for Future Studies

The study co-ordinator welcomes comments or suggestions from participants about this study or possible future studies. Such feedback may be useful in improving future studies. Participants' comments are reproduced in Table 11.

Table 11 Participants' Comments

Lab Code	Participants' Comments	Study Co-ordinator's Response
13	Sample number 3 has some root pieces that makes this sample look not very well homogenised	The samples used in NMI PT studies are prepared from "natural" materials. These are the best approximations of routine test materials, with the analyte being in a "native" state. A full homogeneity test was conducted for this sample. Sample S3 was demonstrated to be sufficiently homogeneous for evaluation of participants' performance.

5 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

5.1 Results Summary

Participant results are listed in Tables 12 to 64 with results' summary statistics: robust average, median, maximum, minimum, robust standard deviation (SD_{rob}) and robust coefficient of variation (CV_{rob}). Bar charts of results and performance scores are presented in Figures 2 to 54.

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

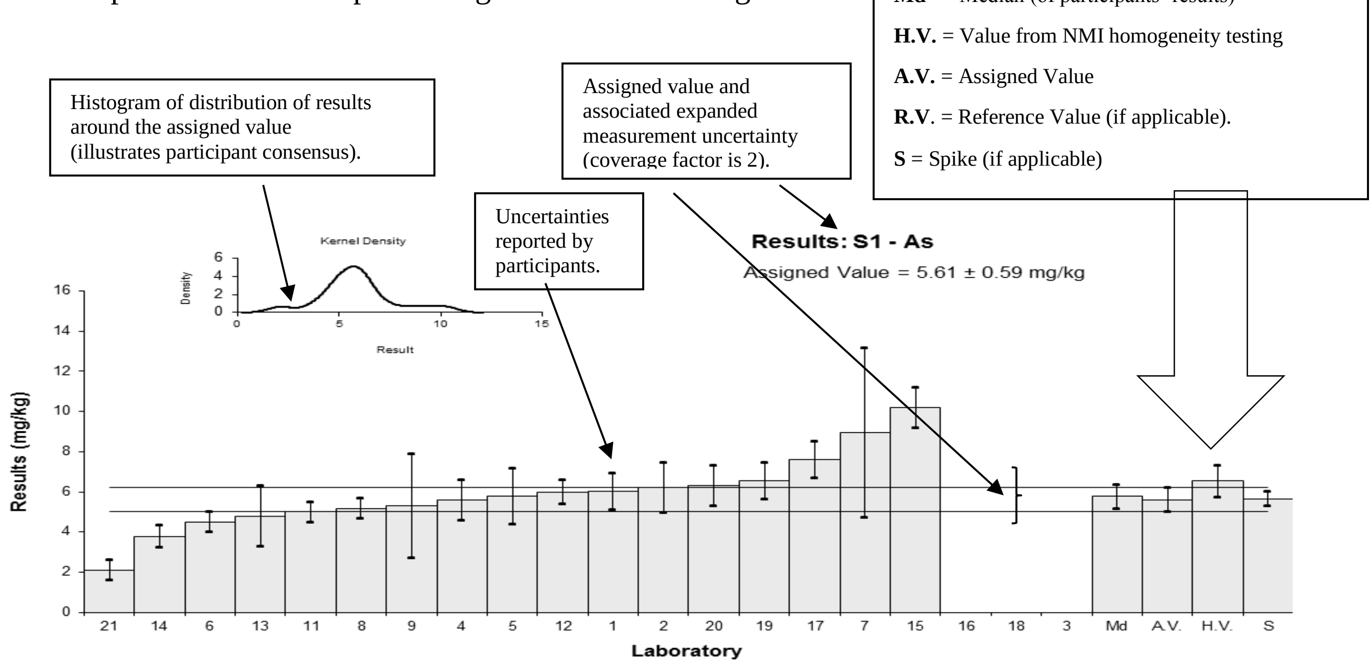


Figure 1 Guide to Presentation of Results

5.2 Assigned Value

An example of an assigned value calculation using data from the present study is given in Appendix 2. The assigned value is defined as: 'the value attributed to a particular property of a proficiency test item.'[1] In this study the property is the mass fraction of analyte. Assigned values were the robust average of participants' results; the expanded uncertainties were estimated from the associated robust standard deviations.

5.3 Robust Average

The robust averages and associated expanded measurement uncertainties were calculated using the procedure described in 'Statistical methods for use in proficiency testing by interlaboratory comparisons, ISO13528:2015(E)'.[8]

5.4 Robust Between-Laboratory Coefficient of Variation

The robust between-laboratory coefficient of variation (CV) is a measure of the variability of participants' results and was calculated using the procedure described in ISO13528:2015(E).[8]

5.5 Target Standard Deviation

The target standard deviation (σ) is the product of the assigned value (*X*) and the performance coefficient of variation (PCV) as presented in Equation 1

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

This value is used for calculation of participant z-score and provides scaling for laboratory deviation from the assigned value. It is important to note that the PCV is a fixed value and is not the standard deviation of participants' results. The fixed value set for PCV is based on the existing regulation, the acceptance criteria indicated by the methods, the matrix, the concentration level of analyte and on experience from previous studies. It is backed up by mathematical models such as Thompson Horwitz equation.[9] By setting a fixed and realistic value for the PCV, the participants' performance does not depends on other participants' performance and can be compared from study to study and against achievable performance.

5.6 z-Score

An example of z-score calculation using data from the present study is given in Appendix 2.

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

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

where:

z is z-score
χ is participants' result
X is the study assigned value
σ is the target standard deviation

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

- |z| ≤ 2 is satisfactory;
- 2 < |z| < 3 is questionable;
- |z| ≥3 is unsatisfactory.

5.7 E_n-Score

An example of E_n-score calculation using data from the present study is given in Appendix 2.

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

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

where:

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

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

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

5.8 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025:2018[10] must establish and demonstrate the traceability and measurement uncertainty associated with their test results. Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.[11]

6 TABLES AND FIGURES

Table 12

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	As
Units	mg/kg

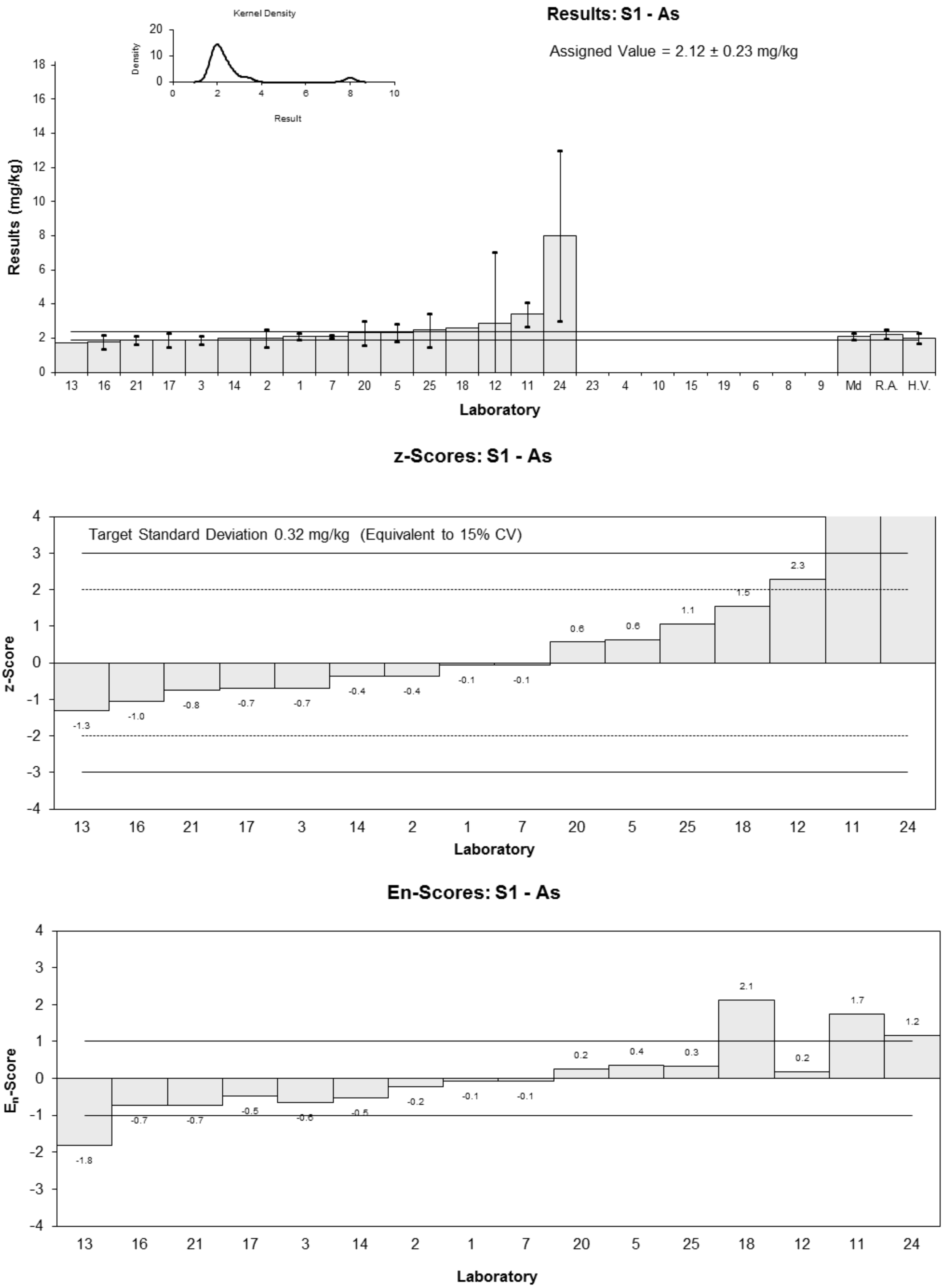
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	2.10	0.21	-0.06	-0.06
2	2.0	0.5	-0.38	-0.22
3	1.90	0.251	-0.69	-0.65
4	<3	NR		
5	2.32	0.5	0.63	0.36
6	NT	NT		
7	2.1	0.1	-0.06	-0.08
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	3.4	0.7	4.03	1.74
12	2.85	4.2	2.30	0.17
13	1.7	NR	-1.32	-1.83
14	2.0	NR	-0.38	-0.52
15	NT	NT		
16	1.787	0.4	-1.05	-0.72
17	1.9	0.4	-0.69	-0.48
18	2.61	NR	1.54	2.13
19	NT	NT		
20	2.3	0.69	0.57	0.25
21	1.88	0.23	-0.75	-0.74
23	<3	NR		
24	8	5	18.49	1.17
25	2.46	1.00	1.07	0.33

Statistics

Assigned Value*	2.12	0.23
Spike	Not Spiked	
Homogeneity Value	2.02	0.30
Robust Average	2.24	0.29
Median	2.10	0.17
Mean	2.58	
N	16	
Max.	8	
Min.	1.7	
Robust SD	0.34	
Robust CV	15%	

*Robust Average excluding Laboratories 11 and 24.



Results: S1 - As
Assigned Value = 2.12 ± 0.23 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
13 16 21 17 3 14 2 1 7 20 5 25 18 12 11 24 23 4 10 15 19 6 8 9 Md R.A. H.V.
z-Scores: S1 - As
Target Standard Deviation 0.32 mg/kg (Equivalent to 15% CV)
z-Score
-1.3 -1.0 -0.8 -0.7 -0.7 -0.4 -0.4 -0.1 -0.1 0.6 0.6 1.1 1.5 2.3
13 16 21 17 3 14 2 1 7 20 5 25 18 12 11 24
Laboratory
En-Scores: S1 - As
En-Score
-1.8 -0.7 -0.7 -0.5 -0.6 -0.5 -0.2 -0.1 -0.1 0.2 0.4 0.3 2.1 0.2 1.7 1.2
13 16 21 17 3 14 2 1 7 20 5 25 18 12 11 24
Laboratory

Figure 2

Table 13

Sample Details

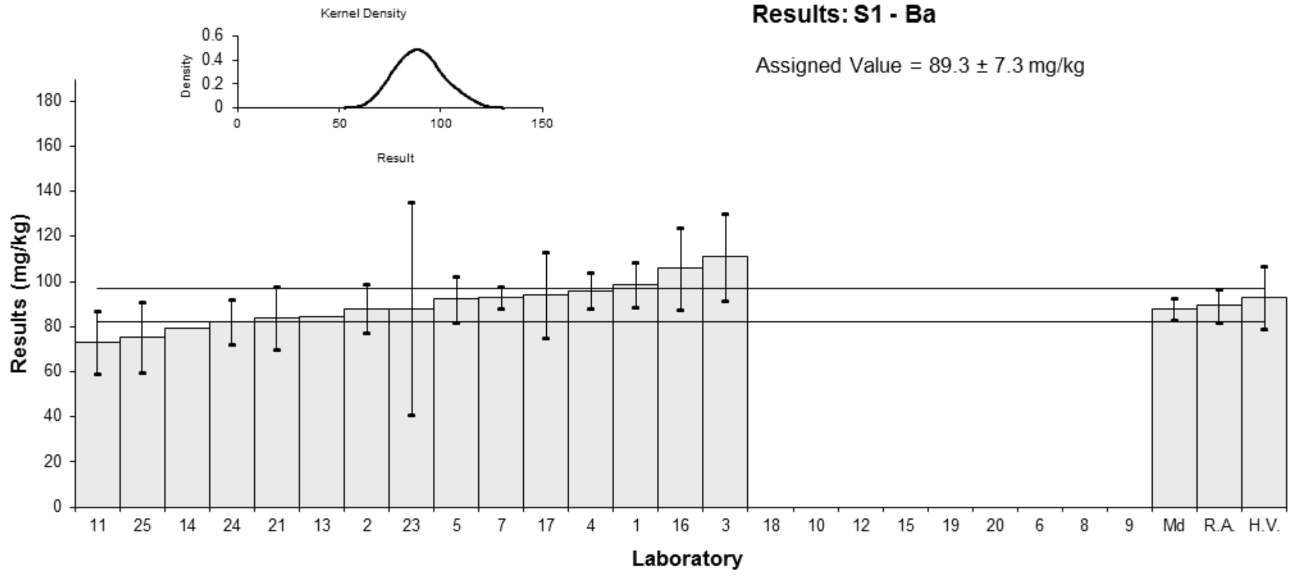
Sample No.	S1
Matrix.	Soil
Analyte.	Ba
Units	mg/kg

Participant Results

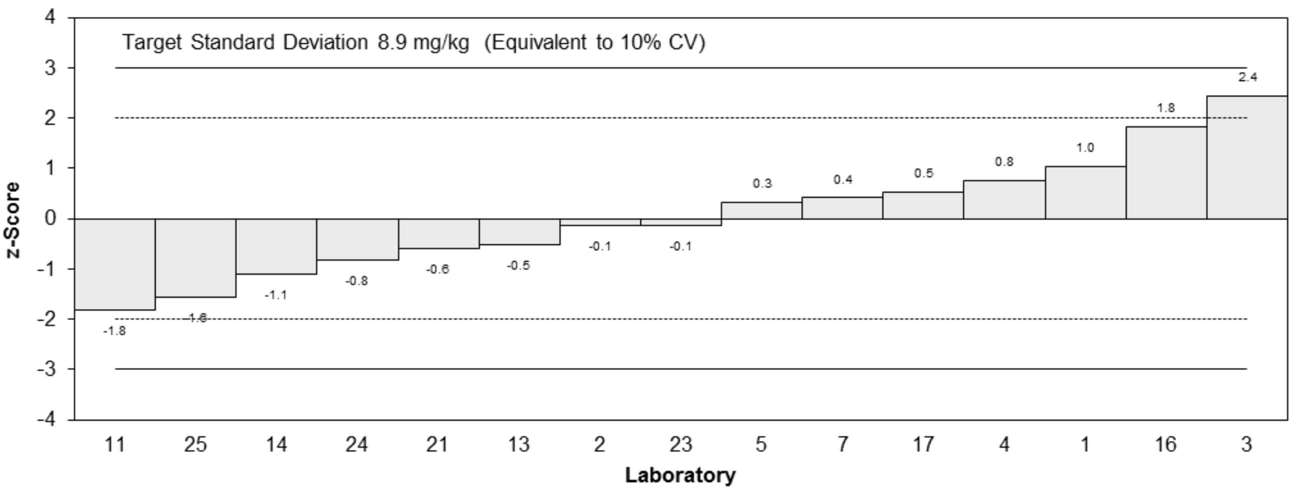
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	98.50	9.85	1.03	0.75
2	88	11	-0.15	-0.10
3	110.99	19.31	2.43	1.05
4	96	8	0.75	0.62
5	92.1	10	0.31	0.23
6	NT	NT		
7	93	5.0	0.41	0.42
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	73	14	-1.83	-1.03
12	NT	NT		
13	84.6	NR	-0.53	-0.64
14	79.5	NR	-1.10	-1.34
15	NT	NT		
16	105.7	18	1.84	0.84
17	94	19	0.53	0.23
18	NR	NR		
19	NT	NT		
20	NT	NT		
21	84	14	-0.59	-0.34
23	88	47.3	-0.15	-0.03
24	82	10	-0.82	-0.59
25	75.4	15.6	-1.56	-0.81

Statistics

Assigned Value	89.3	7.3
Spike	Not Spiked	
Homogeneity Value	93	14
Robust Average	89.3	7.3
Median	88.0	4.9
Mean	89.7	
N	15	
Max.	110.99	
Min.	73	
Robust SD	11	
Robust CV	12%	



z-Scores: S1 - Ba



En-Scores: S1 - Ba

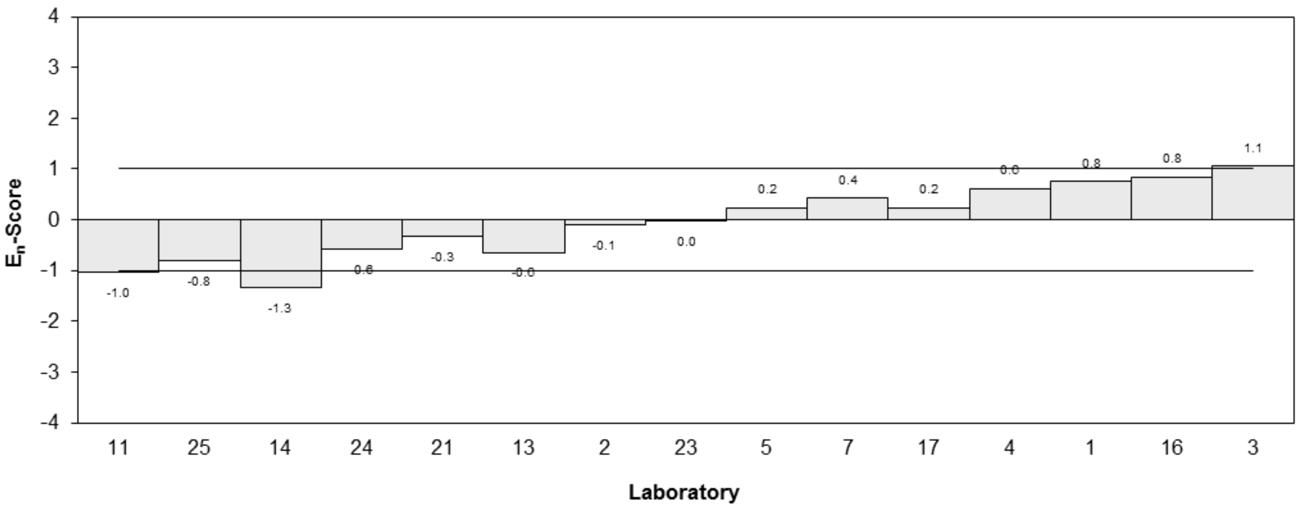


Figure 3

Table 14

Sample Details

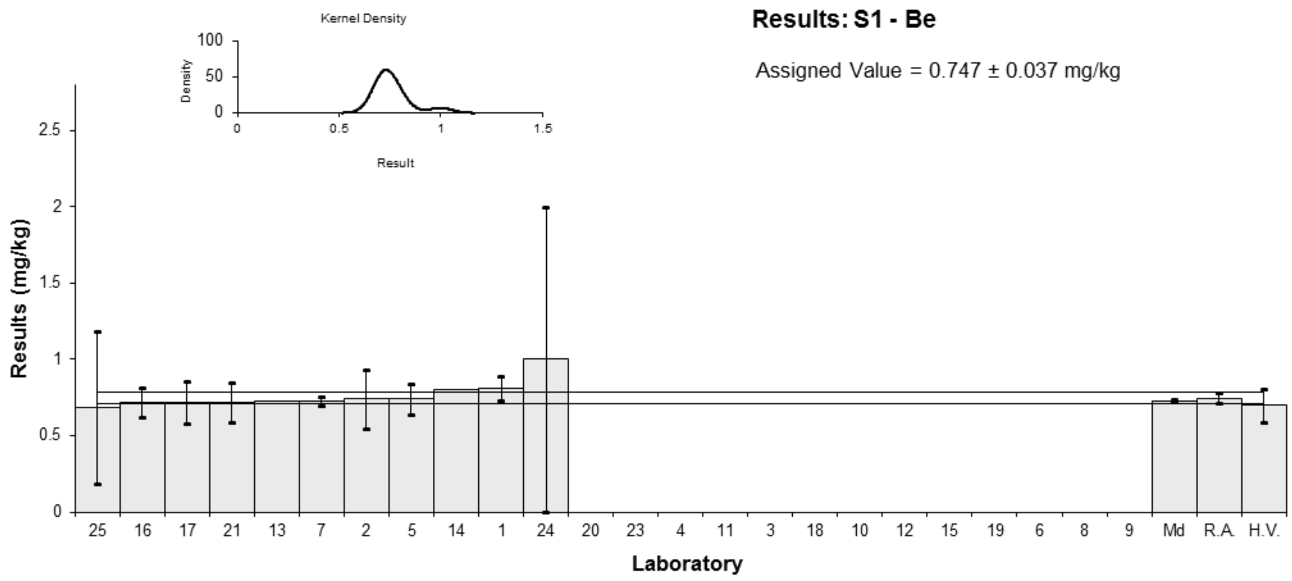
Sample No.	S1
Matrix.	Soil
Analyte.	Be
Units	mg/kg

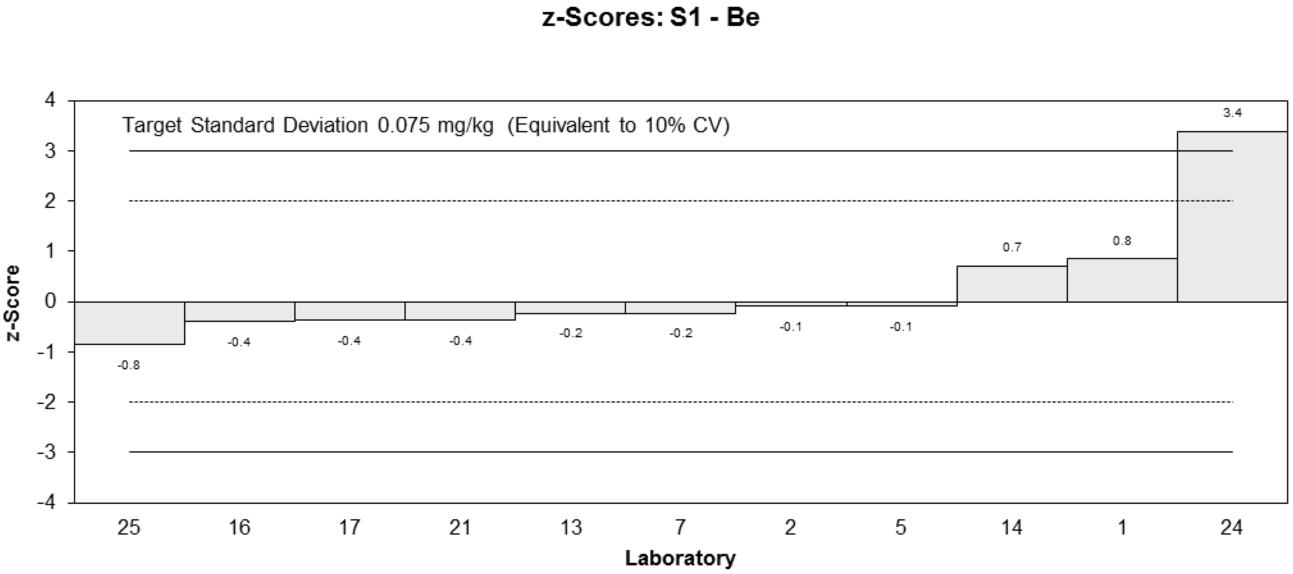
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.81	0.08	0.84	0.71
2	0.74	0.19	-0.09	-0.04
3	<5	NR		
4	<1	NR		
5	0.74	0.1	-0.09	-0.07
6	NT	NT		
7	0.73	0.03	-0.23	-0.36
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	<5	NR		
12	NT	NT		
13	0.73	NR	-0.23	-0.46
14	0.8	NR	0.71	1.43
15	NT	NT		
16	0.7182	0.1	-0.39	-0.27
17	0.72	0.14	-0.36	-0.19
18	NR	NR		
19	NT	NT		
20	<1	NR		
21	0.72	0.13	-0.36	-0.20
23	<1	NR		
24	1	1	3.39	0.25
25	0.684	0.50	-0.84	-0.13

Statistics

Assigned Value	0.747	0.037
Spike	Not Spiked	
Homogeneity Value	0.70	0.11
Robust Average	0.747	0.037
Median	0.730	0.010
Mean	0.763	
N	11	
Max.	1	
Min.	0.684	
Robust SD	0.050	
Robust CV	6.7%	


Results: S1 - Be
Assigned Value = 0.747 ± 0.037 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
25
16
17
21
13
7
2
5
14
1
24
20
23
4
11
3
18
10
12
15
19
6
8
9
Md
R.A.
H.V.


z-Scores: S1 - Be
Target Standard Deviation 0.075 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-0.8
-0.4
-0.4
-0.4
-0.2
-0.2
-0.1
-0.1
0.7
0.8
3.4

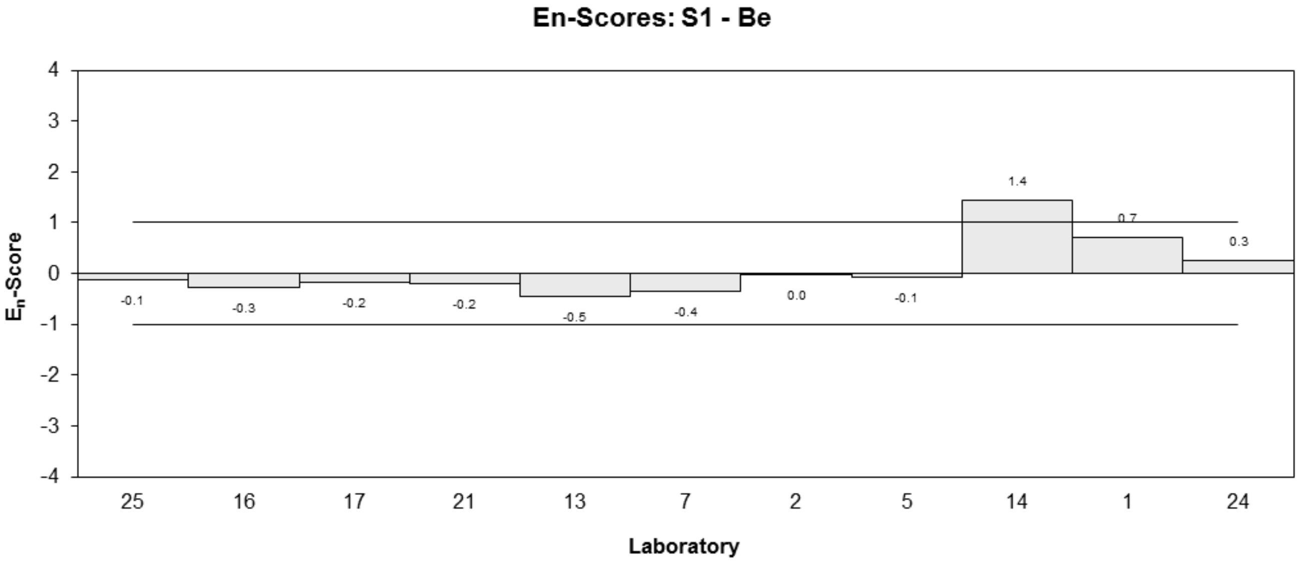

En-Scores: S1 - Be
E_n-Score
Laboratory
-0.1
-0.3
-0.2
-0.2
-0.5
-0.4
0.0
-0.1
1.4
0.7
0.3

Figure 4

Table 15

Sample Details

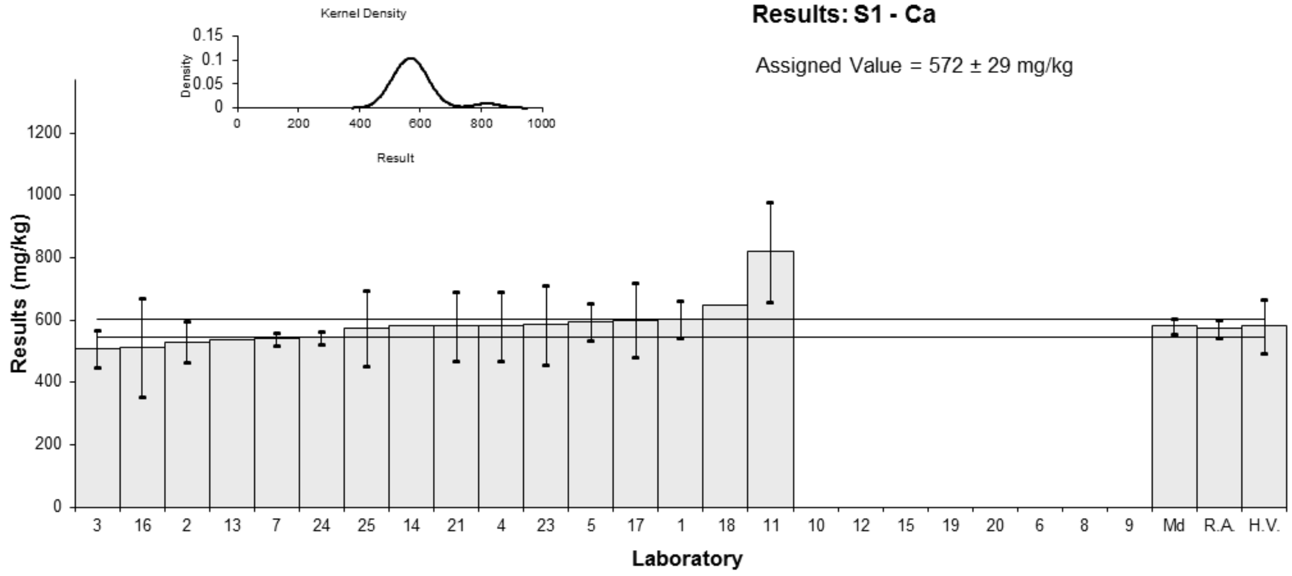
Sample No.	S1
Matrix.	Soil
Analyte.	Ca
Units	mg/kg

Participant Results

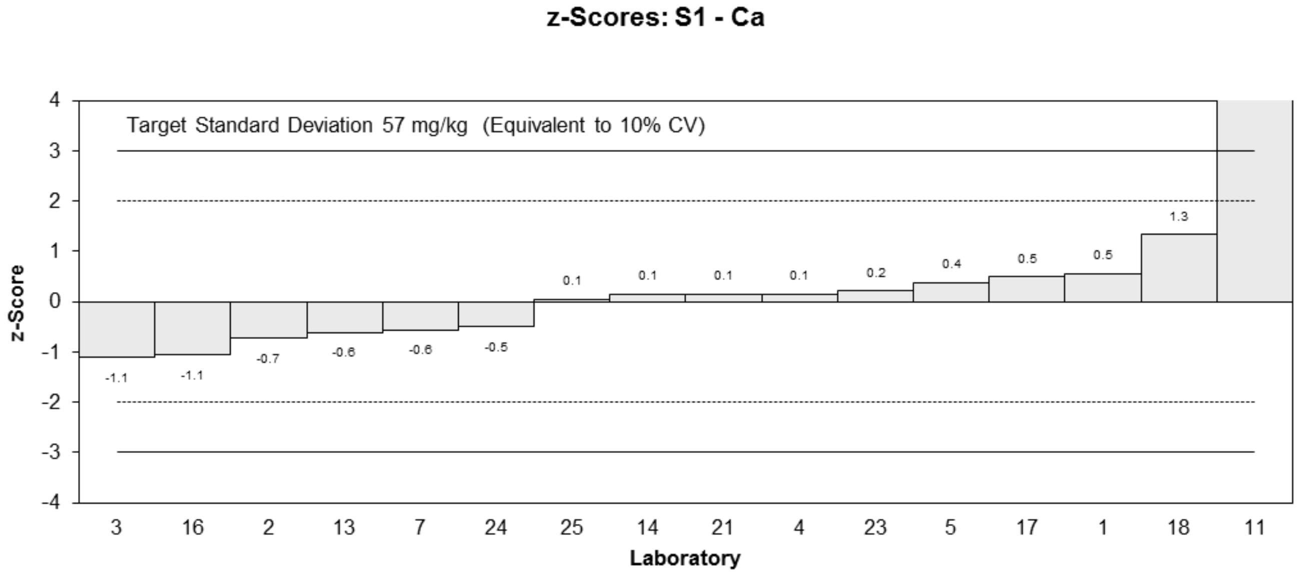
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	603.00	60.30	0.54	0.46
2	530	67	-0.73	-0.58
3	508	58.4	-1.12	-0.98
4	580	110	0.14	0.07
5	593	60	0.37	0.32
6	NT	NT		
7	540	20	-0.56	-0.91
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	819	160	4.32	1.52
12	NT	NT		
13	537	NR	-0.61	-1.21
14	580	NR	0.14	0.28
15	NT	NT		
16	511.8	160	-1.05	-0.37
17	600	120	0.49	0.23
18	648	NR	1.33	2.62
19	NT	NT		
20	NT	NT		
21	580	110	0.14	0.07
23	584	126	0.21	0.09
24	543	20	-0.51	-0.82
25	575	121	0.05	0.02

Statistics

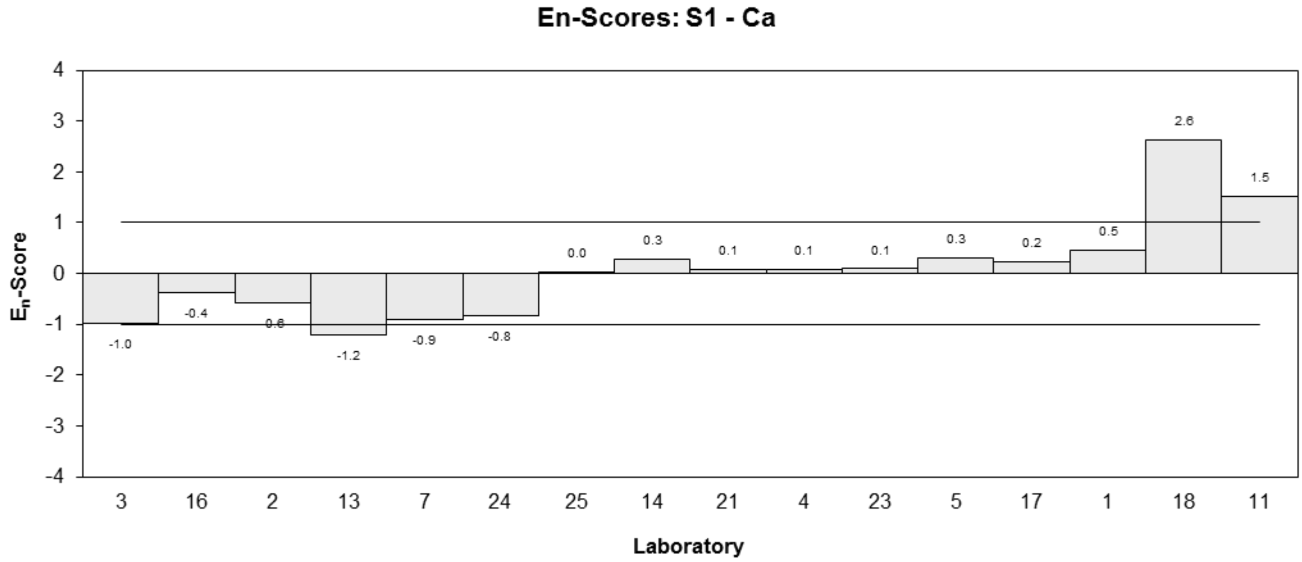
Assigned Value	572	29
Spike	Not Spiked	
Homogeneity Value	580	87
Robust Average	572	29
Median	580	24
Mean	583	
N	16	
Max.	819	
Min.	508	
Robust SD	46	
Robust CV	8%	



Results: S1 - Ca
Assigned Value = 572 ± 29 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
3
16
2
13
7
24
25
14
21
4
23
5
17
1
18
11
10
12
15
19
20
6
8
9
Md
R.A.
H.V.



z-Scores: S1 - Ca
Target Standard Deviation 57 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-1.1
-1.1
-0.7
-0.6
-0.6
-0.5
0.1
0.1
0.1
0.1
0.2
0.4
0.5
0.5
1.3
3
16
2
13
7
24
25
14
21
4
23
5
17
1
18
11



En-Scores: S1 - Ca
En-Score
Laboratory
-1.0
-0.4
-0.6
-1.2
-0.9
-0.8
0.0
0.3
0.1
0.1
0.1
0.3
0.2
0.5
2.6
1.5
3
16
2
13
7
24
25
14
21
4
23
5
17
1
18
11

Figure 5

Table 16

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	Co
Units	mg/kg

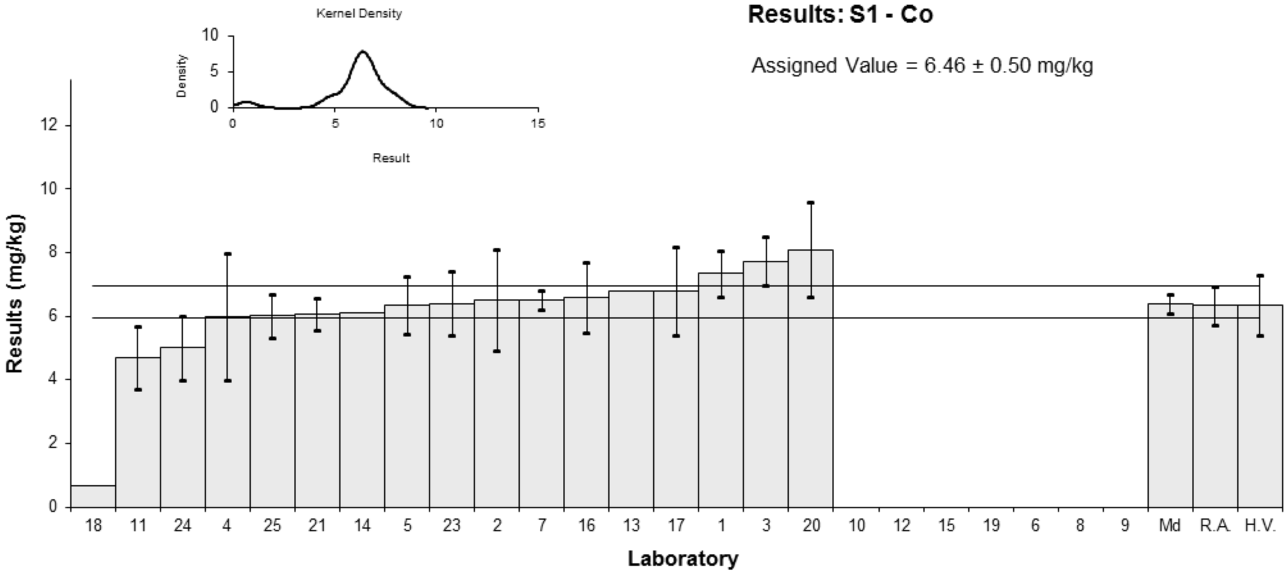
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	7.34	0.73	1.36	0.99
2	6.5	1.6	0.06	0.02
3	7.73	0.773	1.97	1.38
4	6	2	-0.71	-0.22
5	6.35	0.9	-0.17	-0.11
6	NT	NT		
7	6.5	0.3	0.06	0.07
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	4.7	1	-2.72	-1.57
12	NT	NT		
13	6.8	NR	0.53	0.68
14	6.1	NR	-0.56	-0.72
15	NT	NT		
16	6.598	1.1	0.21	0.11
17	6.8	1.4	0.53	0.23
18	0.67	NR	-8.96	-11.58
19	NT	NT		
20	8.1	1.5	2.54	1.04
21	6.07	0.49	-0.60	-0.56
23	6.4	1	-0.09	-0.05
24	5	1	-2.26	-1.31
25	6.02	0.69	-0.68	-0.52

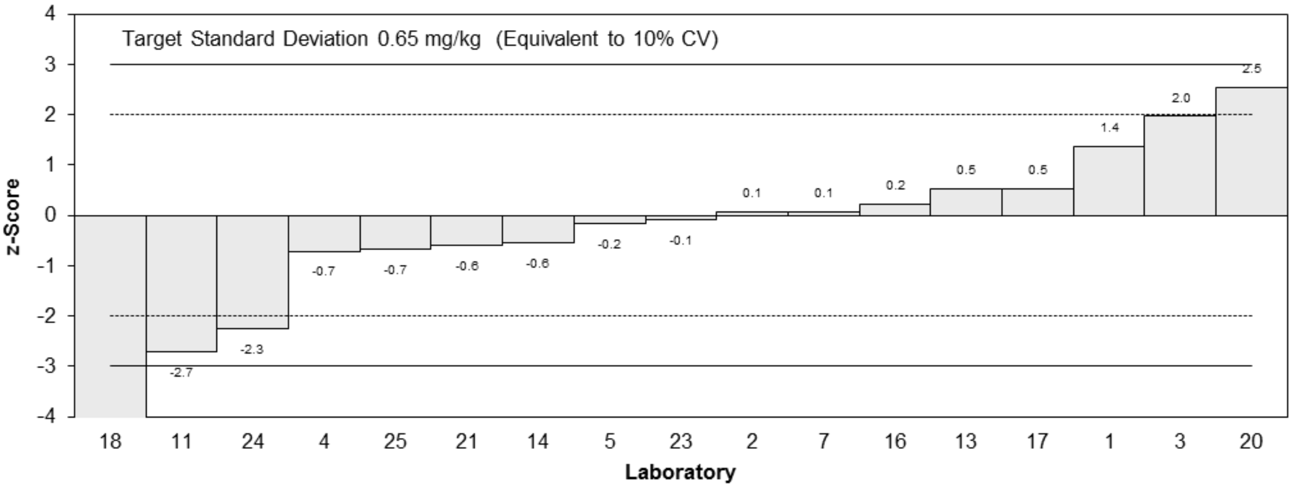
Statistics

Assigned Value*	6.46	0.50
Spike	Not Spiked	
Homogeneity Value	6.35	0.95
Robust Average	6.34	0.60
Median	6.40	0.30
Mean	6.10	
N	17	
Max.	8.1	
Min.	0.67	
Robust SD	0.80	
Robust CV	13%	

*Robust Average excluding Laboratory 18.



z-Scores: S1 - Co



En-Scores: S1 - Co

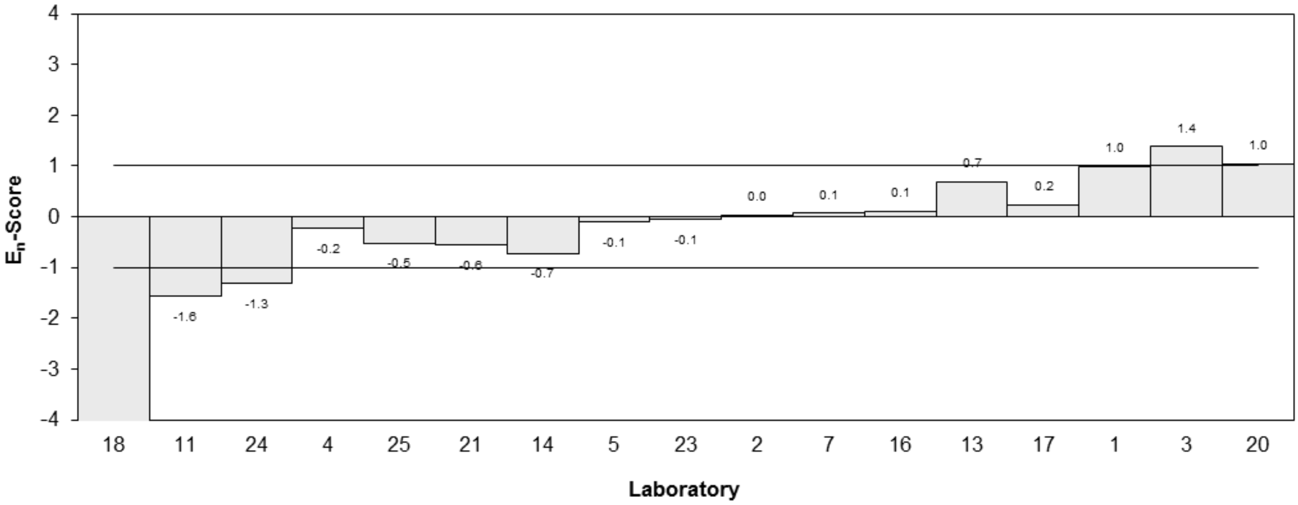


Figure 6

Table 17

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	Cr
Units	mg/kg

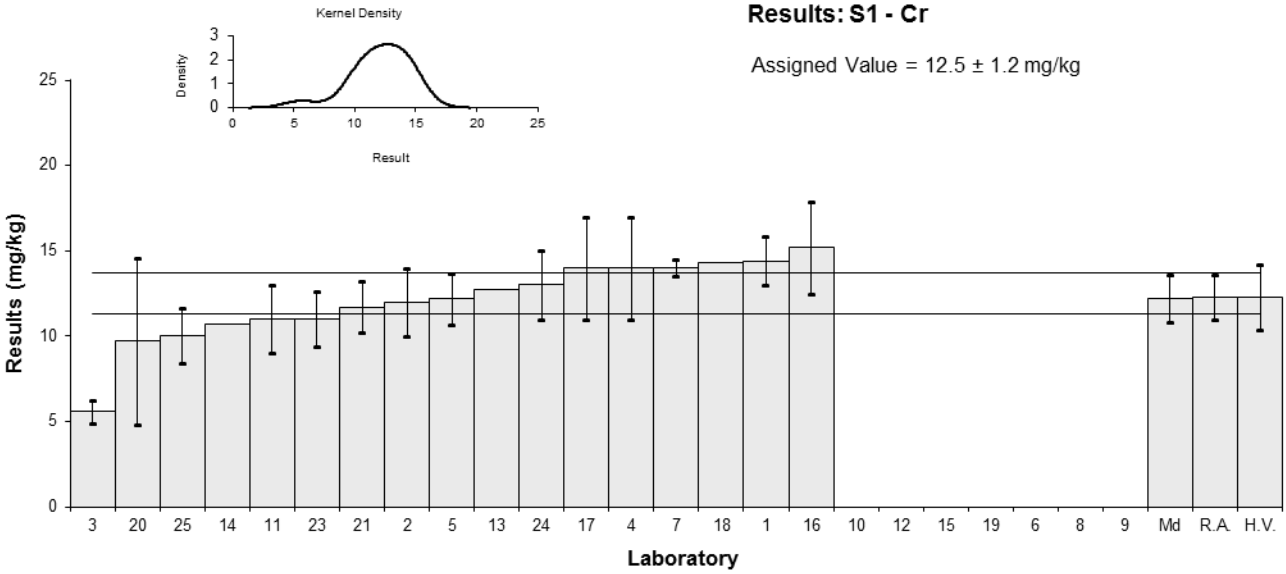
Participant Results

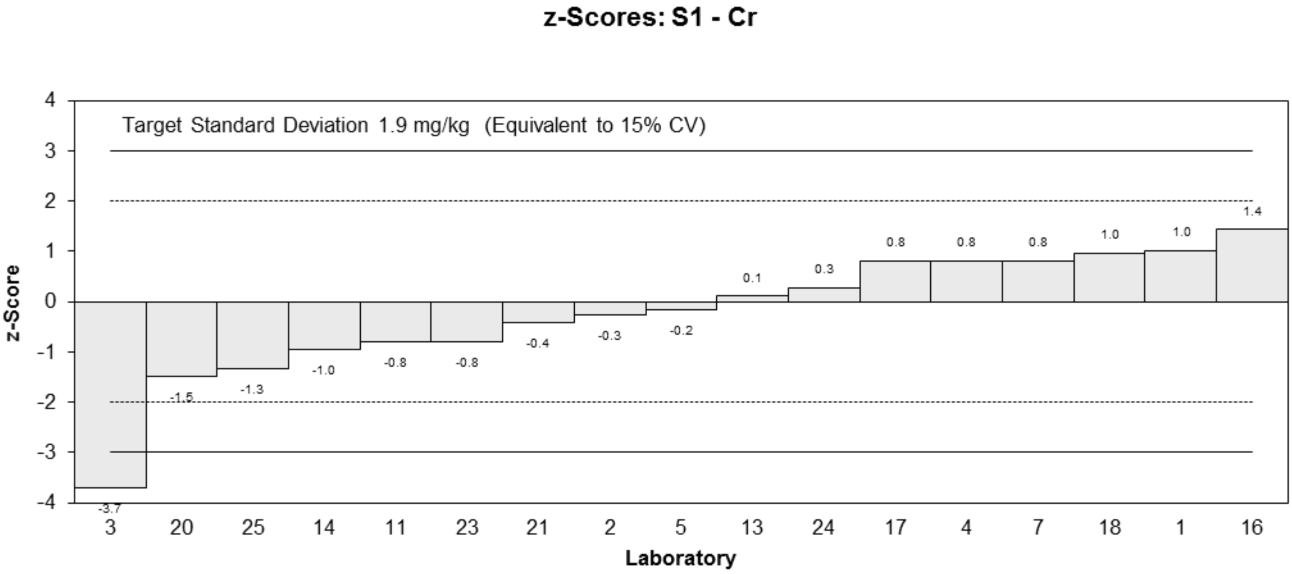
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	14.40	1.44	1.01	1.01
2	12	2	-0.27	-0.21
3	5.57	0.668	-3.70	-5.05
4	14	3	0.80	0.46
5	12.2	1.5	-0.16	-0.16
6	NT	NT		
7	14	0.5	0.80	1.15
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	11	2	-0.80	-0.64
12	NT	NT		
13	12.7	NR	0.11	0.17
14	10.7	NR	-0.96	-1.50
15	NT	NT		
16	15.18	2.7	1.43	0.91
17	14	3	0.80	0.46
18	14.3	NR	0.96	1.50
19	NT	NT		
20	9.7	4.9	-1.49	-0.56
21	11.7	1.5	-0.43	-0.42
23	11	1.6	-0.80	-0.75
24	13	2	0.27	0.21
25	10	1.62	-1.33	-1.24

Statistics

Assigned Value*	12.5	1.2
Spike	Not Spiked	
Homogeneity Value	12.3	1.9
Robust Average	12.3	1.3
Median	12.2	1.4
Mean	12.1	
N	17	
Max.	15.18	
Min.	5.57	
Robust SD	1.9	
Robust CV	15%	

*Robust Average excluding Laboratory 3.


Results: S1 - Cr
Assigned Value = 12.5 ± 1.2 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
3
20
25
14
11
23
21
2
5
13
24
17
4
7
18
1
16
10
12
15
19
6
8
9
Md
R.A.
H.V.


z-Scores: S1 - Cr
Target Standard Deviation 1.9 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory

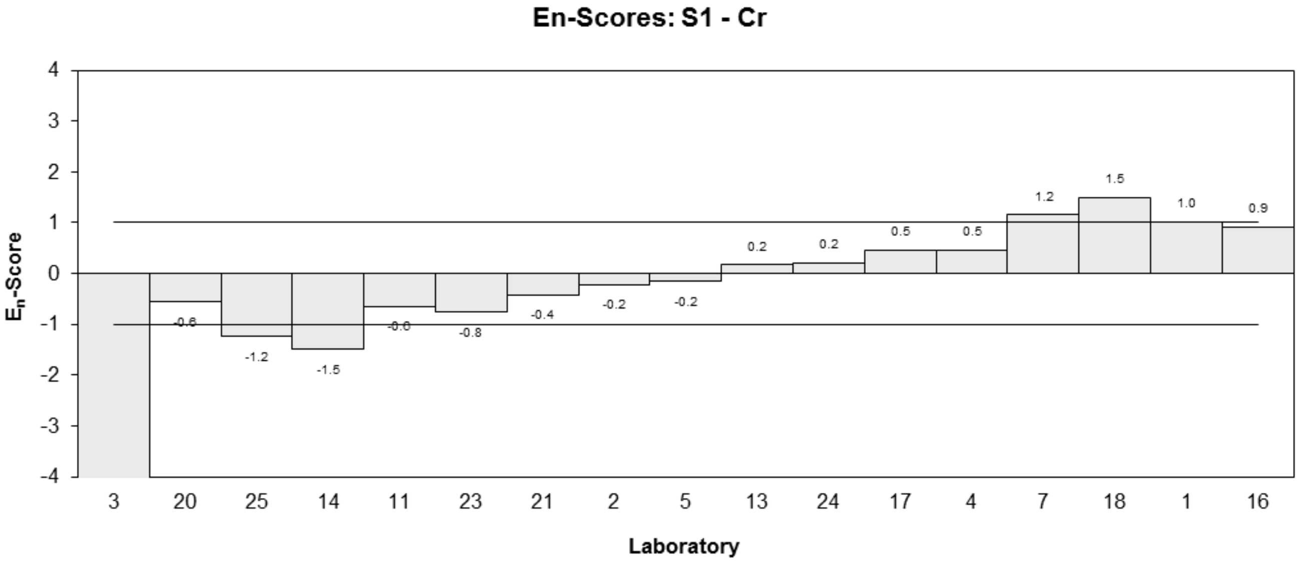

En-Scores: S1 - Cr
En-Score
Laboratory

Figure 7

Table 18

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	Cs
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	0.64	0.06
2	NT	NT
3	NT	NT
4	NT	NT
5	NT	NT
6	NT	NT
7	NT	NT
8	NT	NT
9	NT	NT
10	NT	NT
11	NT	NT
12	NT	NT
13	NT	NT
14	0.4	NR
15	NT	NT
16	NR	NR
17	0.62	0.12
18	NR	NR
19	NT	NT
20	NT	NT
21	0.556	0.069
23	NT	NT
24	NR	NR
25	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.70	0.10
Robust Average	0.55	0.15
Median	0.59	0.10
Mean	0.55	
N	4	
Max.	0.64	
Min.	0.4	
Robust SD	0.12	
Robust CV	22%	

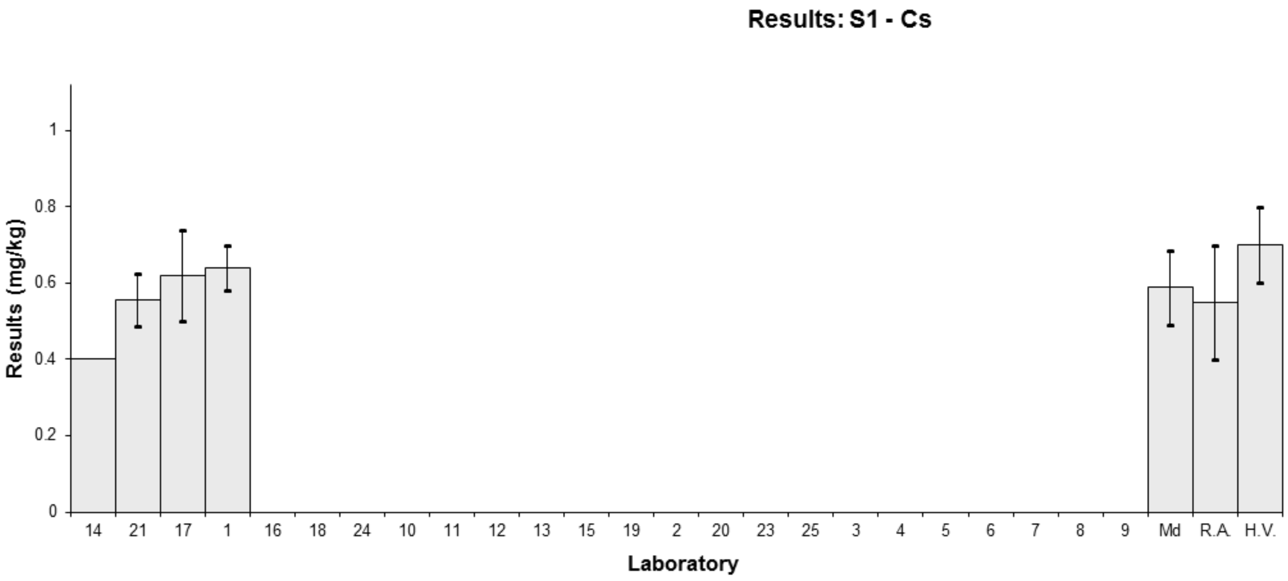

Results: S1 - Cs
Results (mg/kg)
1
0.8
0.6
0.4
0.2
0
14
21
17
1
16
18
24
10
11
12
13
15
19
2
20
23
25
3
4
5
6
7
8
9
Md
R.A.
H.V.
Laboratory

Figure 8

Table 19

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	Cu
Units	mg/kg

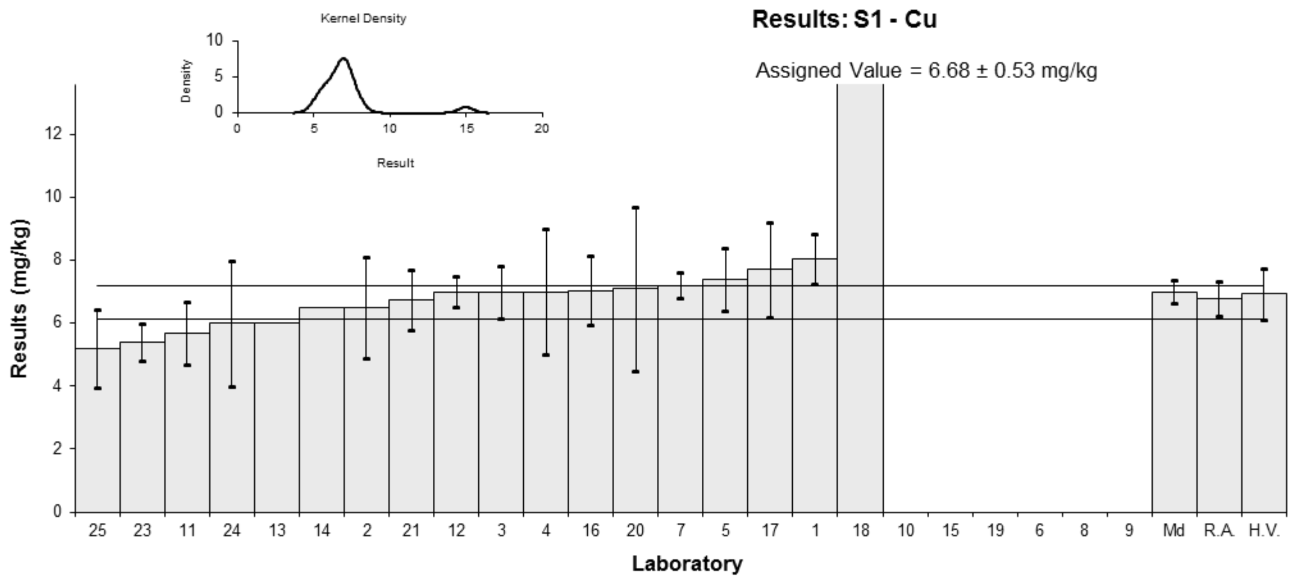
Participant Results

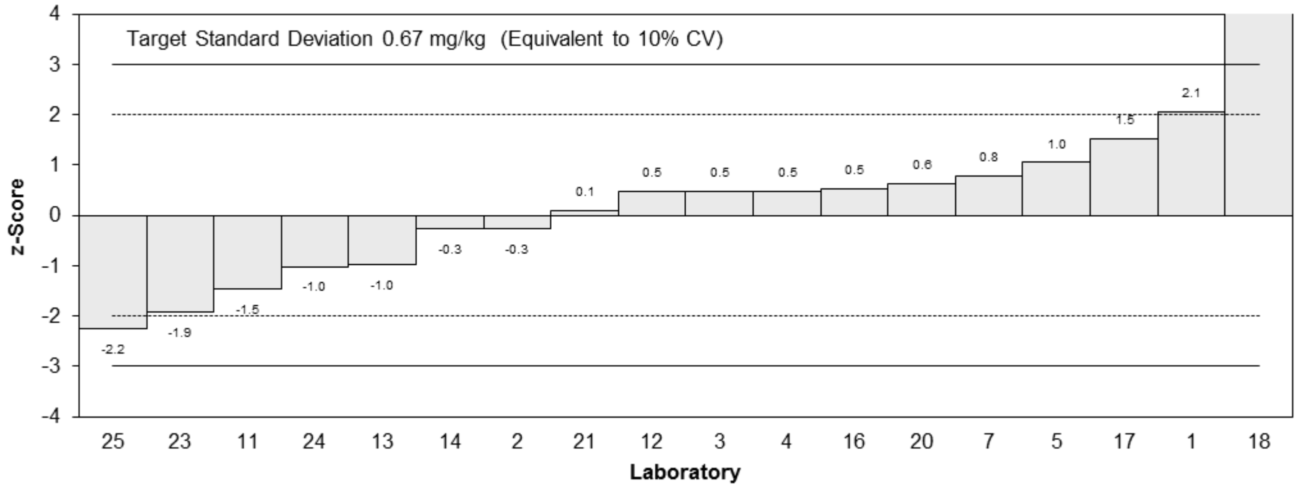
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	8.05	0.80	2.05	1.43
2	6.5	1.6	-0.27	-0.11
3	7.00	0.840	0.48	0.32
4	7	2	0.48	0.15
5	7.38	1.0	1.05	0.62
6	NT	NT		
7	7.2	0.4	0.78	0.78
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	5.7	1	-1.47	-0.87
12	7	0.5	0.48	0.44
13	6.02	NR	-0.99	-1.25
14	6.5	NR	-0.27	-0.34
15	NT	NT		
16	7.029	1.1	0.52	0.29
17	7.7	1.5	1.53	0.64
18	15.0	NR	12.46	15.70
19	NT	NT		
20	7.1	2.6	0.63	0.16
21	6.74	0.96	0.09	0.05
23	5.4	0.6	-1.92	-1.60
24	6	2	-1.02	-0.33
25	5.18	1.24	-2.25	-1.11

Statistics

Assigned Value*	6.68	0.53
Spike	Not Spiked	
Homogeneity Value	6.93	0.83
Robust Average	6.77	0.55
Median	7.00	0.37
Mean	7.14	
N	18	
Max.	15	
Min.	5.18	
Robust SD	0.87	
Robust CV	13%	

Robust Average excluding Laboratory 18.


Results: S1 - Cu
Assigned Value = 6.68 ± 0.53 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
25 23 11 24 13 14 2 21 12 3 4 16 20 7 5 17 1 18 10 15 19 6 8 9 Md R.A. H.V.
Laboratory


z-Scores: S1 - Cu
Target Standard Deviation 0.67 mg/kg (Equivalent to 10% CV)
z-Score
-2.2 -1.9 -1.5 -1.0 -1.0 -0.3 -0.3 0.1 0.5 0.5 0.5 0.5 0.6 0.8 1.0 1.5 2.1
25 23 11 24 13 14 2 21 12 3 4 16 20 7 5 17 1 18
Laboratory

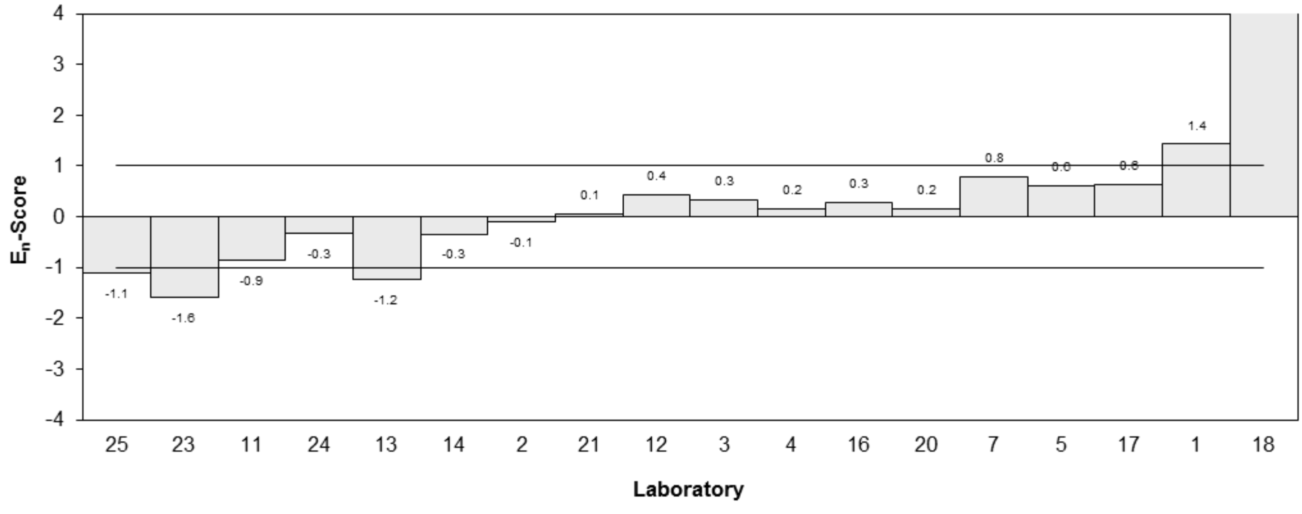

En-Scores: S1 - Cu
E_n-Score
-1.1 -1.6 -0.9 -0.3 -1.2 -0.3 -0.1 0.1 0.4 0.3 0.2 0.3 0.2 0.8 0.6 0.6 1.4
25 23 11 24 13 14 2 21 12 3 4 16 20 7 5 17 1 18
Laboratory

Figure 9

Table 20

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	Ga
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	5	2
3	NT	NT
4	NT	NT
5	NT	NT
6	NT	NT
7	NT	NT
8	NT	NT
9	NT	NT
10	NT	NT
11	NT	NT
12	NT	NT
13	NT	NT
14	2.3	NR
15	NT	NT
16	NR	NR
17	2.9	0.6
18	NR	NR
19	NT	NT
20	NT	NT
21	NT	NT
23	NT	NT
24	NR	NR
25	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	2.9	0.4
Robust Average	3.4	2.3
Median	2.9	2.2
Mean	3.4	
N	3	
Max.	5	
Min.	2.3	
Robust SD	1.6	
Robust CV	47%	

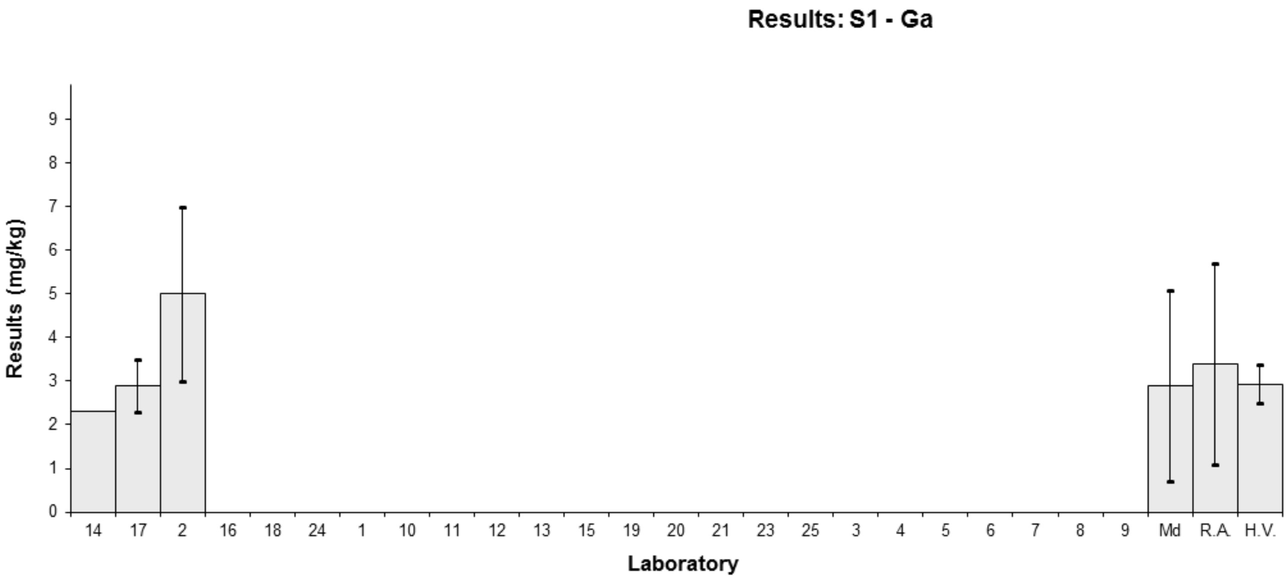

Results: S1 - Ga
Results (mg/kg)
0
1
2
3
4
5
6
7
8
9
14
17
2
16
18
24
1
10
11
12
13
15
19
20
21
23
25
3
4
5
6
7
8
9
Md
R.A.
H.V.
Laboratory

Figure 10

Table 21

Sample Details

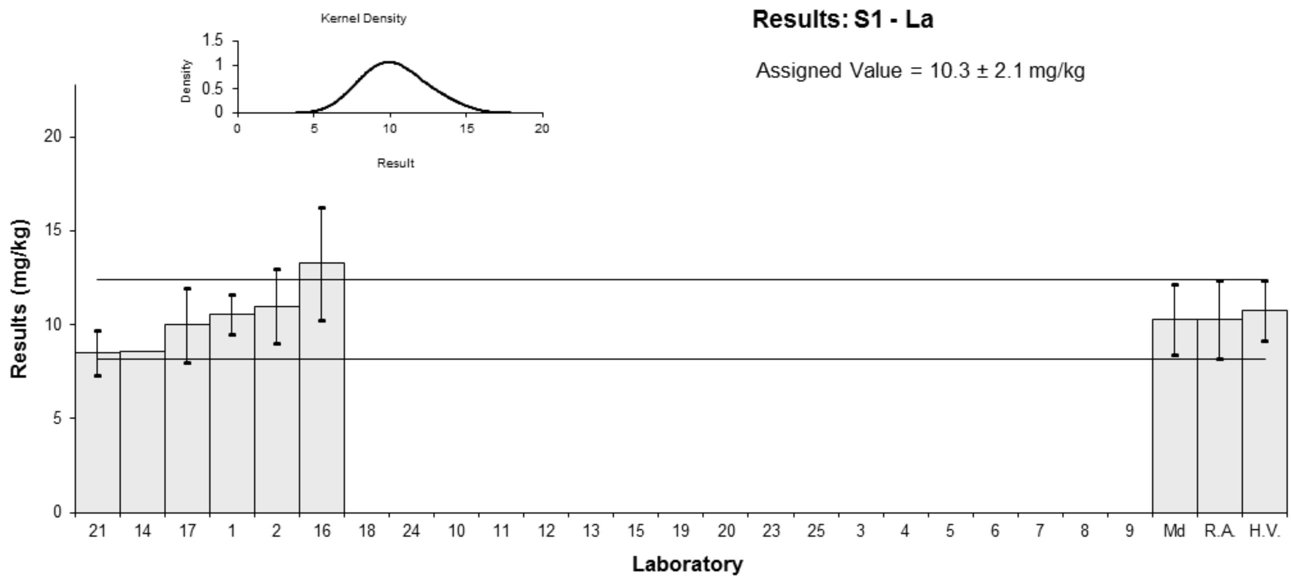
Sample No.	S1
Matrix.	Soil
Analyte.	La
Units	mg/kg

Participant Results

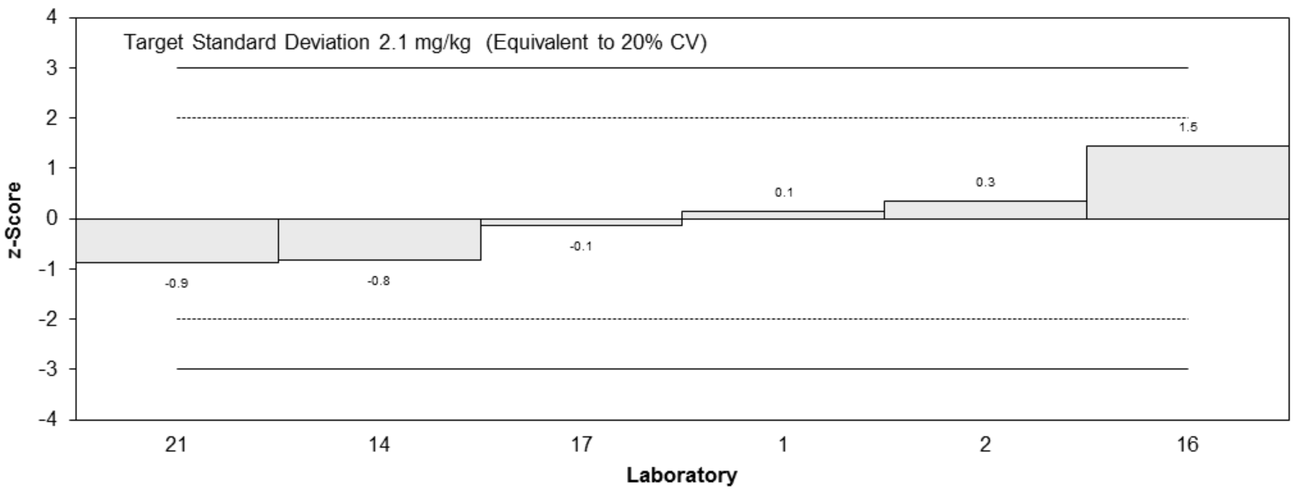
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	10.60	1.06	0.15	0.13
2	11	2	0.34	0.24
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	NT	NT		
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	NT	NT		
12	NT	NT		
13	NT	NT		
14	8.6	NR	-0.83	-0.81
15	NT	NT		
16	13.29	3	1.45	0.82
17	10	2	-0.15	-0.10
18	NR	NR		
19	NT	NT		
20	NT	NT		
21	8.5	1.2	-0.87	-0.74
23	NT	NT		
24	NR	NR		
25	NT	NT		

Statistics

Assigned Value	10.3	2.1
Spike	Not Spiked	
Homogeneity Value	10.8	1.6
Robust Average	10.3	2.1
Median	10.3	1.9
Mean	10.3	
N	6	
Max.	13.29	
Min.	8.5	
Robust SD	2.0	
Robust CV	19%	



z-Scores: S1 - La



En-Scores: S1 - La

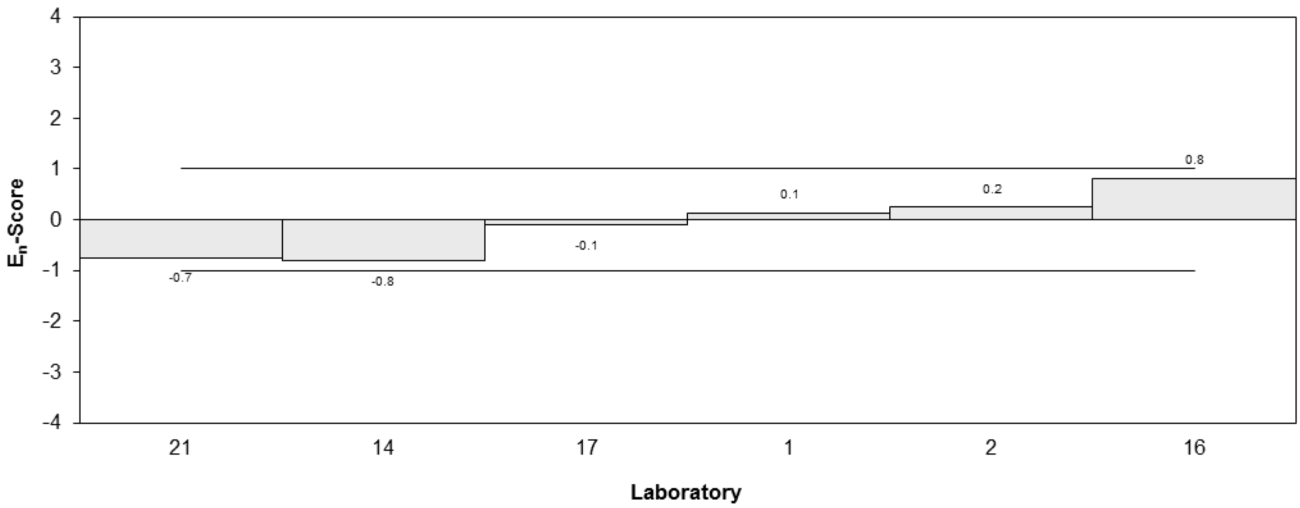


Figure 11

Table 22

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	Li
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	2.80	0.28	0.93	0.78
2	1.9	0.5	-0.97	-0.66
3	22.6	5.42	42.88	3.72
4	2	2	-0.76	-0.17
5	2.36	0.5	0.00	0.00
6	NT	NT		
7	2.8	0.2	0.93	0.83
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	2	0.4	-0.76	-0.57
12	NT	NT		
13	<5	NR		
14	1.8	NR	-1.19	-1.14
15	NT	NT		
16	NR	NR		
17	3.2	0.6	1.78	1.08
18	NR	NR		
19	NT	NT		
20	NT	NT		
21	1.76	0.34	-1.27	-1.01
23	NT	NT		
24	3	1	1.36	0.57
25	0.786	0.500	-3.33	-2.25

Statistics

Assigned Value*	2.36	0.49
Spike	Not Spiked	
Homogeneity Value	2.60	0.39
Robust Average	2.36	0.58
Median	2.18	0.49
Mean	3.92	
N	12	
Max.	22.6	
Min.	0.786	
Robust SD	0.61	
Robust CV	26%	

*Robust Average excluding Laboratories 3 and 25.

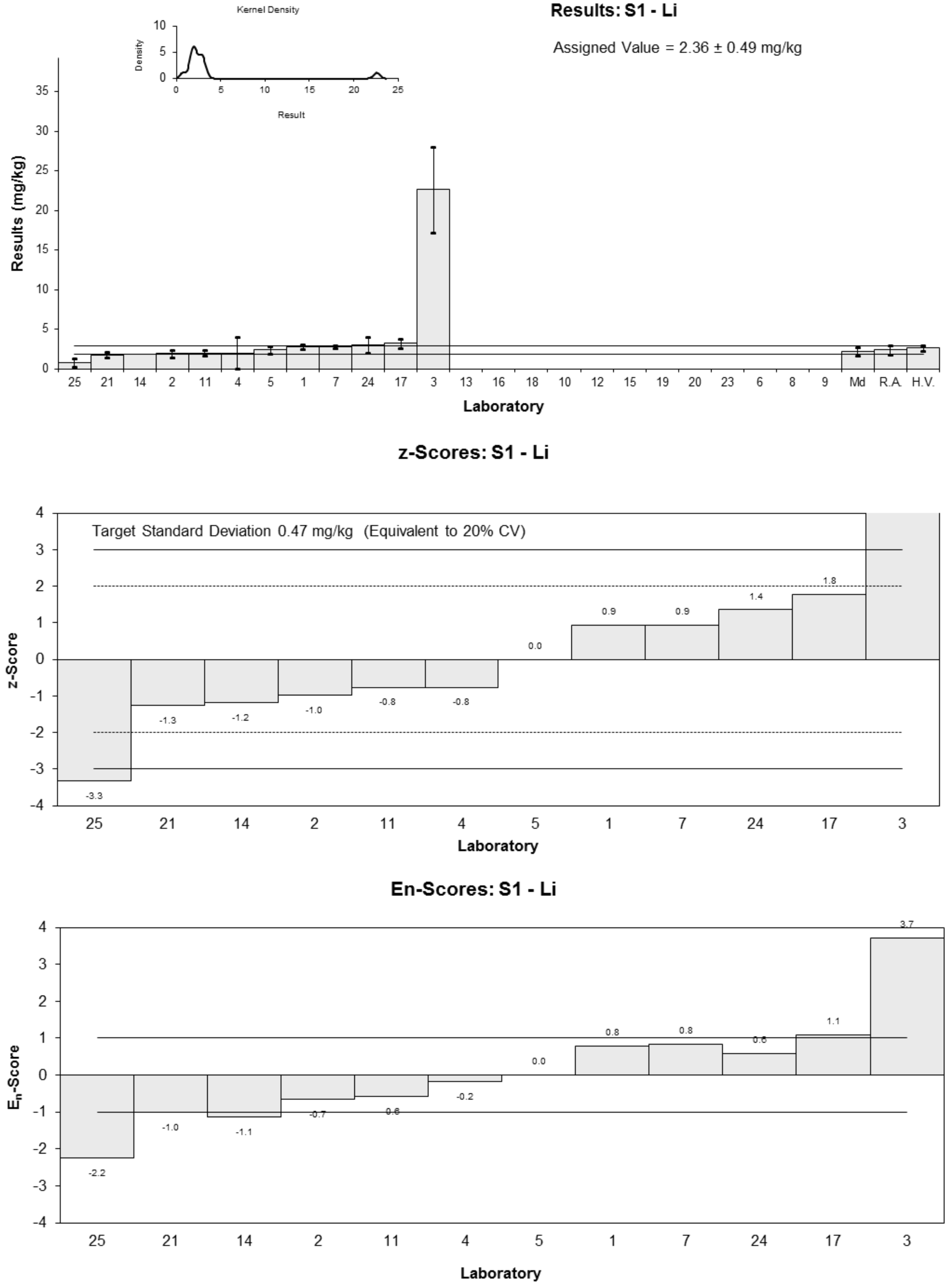

Results: S1 - Li
Assigned Value = 2.36 ± 0.49 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
25 21 14 2 11 4 5 1 7 24 17 3 13 16 18 10 12 15 19 20 23 6 8 9 Md R.A. H.V.
Laboratory
z-Scores: S1 - Li
Target Standard Deviation 0.47 mg/kg (Equivalent to 20% CV)
z-Score
-3.3 -1.3 -1.2 -1.0 -0.8 -0.8 0.0 0.9 0.9 1.4 1.8
25 21 14 2 11 4 5 1 7 24 17 3
Laboratory
En-Scores: S1 - Li
En-Score
-2.2 -1.0 -1.1 -0.7 -0.6 -0.2 0.0 0.8 0.8 0.6 1.1 3.7
25 21 14 2 11 4 5 1 7 24 17 3
Laboratory

Figure 12

Table 23

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	Mn
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	398.00	39.80	0.56	0.51
2	370	47	-0.19	-0.15
3	396	39.6	0.50	0.47
4	390	30	0.34	0.41
5	362	40	-0.40	-0.36
6	NT	NT		
7	380	20	0.08	0.13
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	329	60	-1.27	-0.79
12	NT	NT		
13	381	NR	0.11	0.40
14	380	NR	0.08	0.30
15	NT	NT		
16	365.9	68	-0.29	-0.16
17	380	80	0.08	0.04
18	418	NR	1.09	4.10
19	NT	NT		
20	363	54	-0.37	-0.25
21	388	39	0.29	0.27
23	373	54.6	-0.11	-0.07
24	370	20	-0.19	-0.31
25	357	46.8	-0.53	-0.42

Statistics

Assigned Value	377	10
Spike	Not Spiked	
Homogeneity Value	362	54
Robust Average	377	10
Median	380	8
Mean	377	
N	17	
Max.	418	
Min.	329	
Robust SD	17	
Robust CV	4.5%	

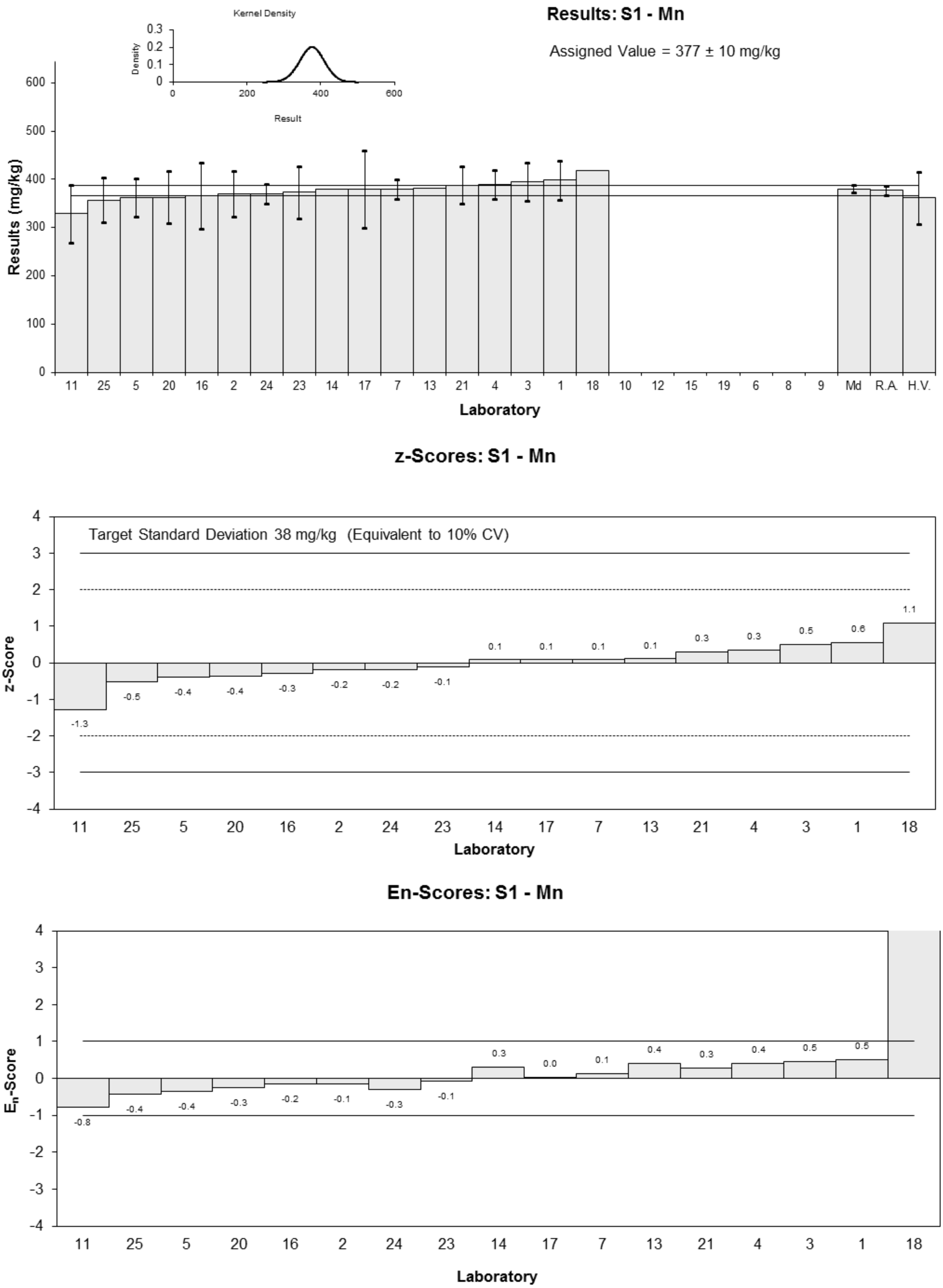

Results: S1 - Mn
Assigned Value = 377 ± 10 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
11 25 5 20 16 2 24 23 14 17 7 13 21 4 3 1 18 10 12 15 19 6 8 9 Md R.A. H.V.
z-Scores: S1 - Mn
Target Standard Deviation 38 mg/kg (Equivalent to 10% CV)
z-Score
-1.3 -0.5 -0.4 -0.4 -0.3 -0.2 -0.2 -0.1 0.1 0.1 0.1 0.1 0.3 0.3 0.5 0.6 1.1
11 25 5 20 16 2 24 23 14 17 7 13 21 4 3 1 18
Laboratory
En-Scores: S1 - Mn
En-Score
-0.8 -0.4 -0.4 -0.3 -0.2 -0.1 -0.3 -0.1 0.3 0.0 0.1 0.4 0.3 0.4 0.5 0.5
11 25 5 20 16 2 24 23 14 17 7 13 21 4 3 1 18
Laboratory

Figure 13

Table 24

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	Ni
Units	mg/kg

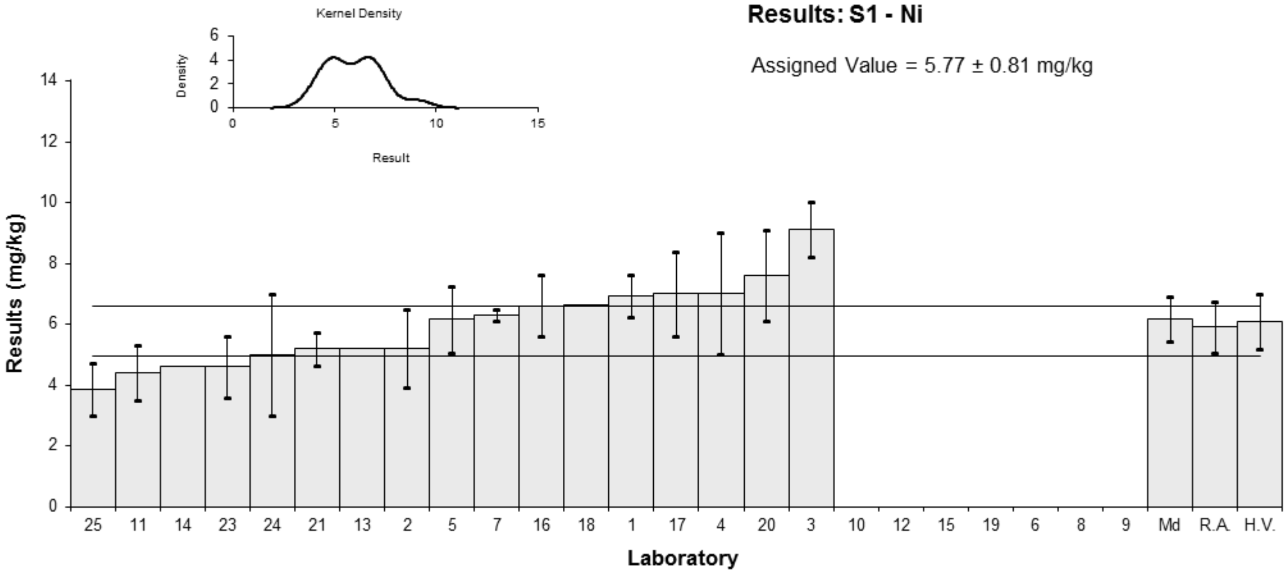
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	6.93	0.69	1.34	1.09
2	5.2	1.3	-0.66	-0.37
3	9.12	0.912	3.87	2.75
4	7	2	1.42	0.57
5	6.16	1.1	0.45	0.29
6	NT	NT		
7	6.3	0.2	0.61	0.64
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	4.4	0.9	-1.58	-1.13
12	NT	NT		
13	5.2	NR	-0.66	-0.70
14	4.6	NR	-1.35	-1.44
15	NT	NT		
16	6.614	1	0.98	0.66
17	7.0	1.4	1.42	0.76
18	6.63	NR	0.99	1.06
19	NT	NT		
20	7.6	1.5	2.11	1.07
21	5.19	0.54	-0.67	-0.60
23	4.6	1	-1.35	-0.91
24	5	2	-0.89	-0.36
25	3.86	0.88	-2.21	-1.60

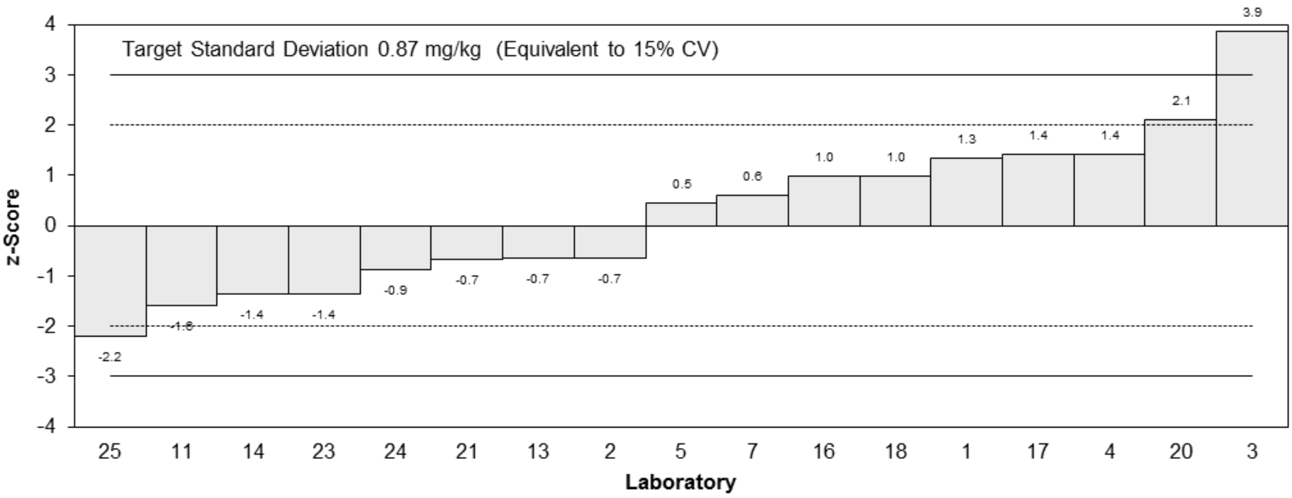
Statistics

Assigned Value*	5.77	0.81
Spike	Not Spiked	
Homogeneity Value	6.08	0.91
Robust Average	5.90	0.84
Median	6.16	0.73
Mean	5.96	
N	17	
Max.	9.12	
Min.	3.86	
Robust SD	1.30	
Robust CV	22%	

*Robust Average excluding Laboratory 3.



z-Scores: S1 - Ni



En-Scores: S1 - Ni

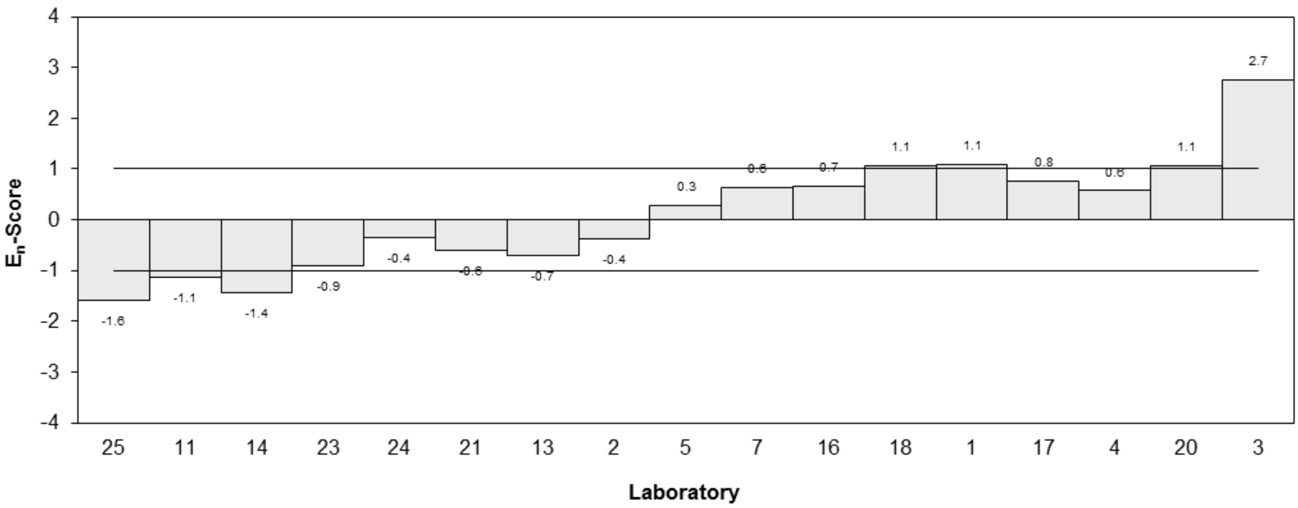


Figure 14

Table 25

Sample Details

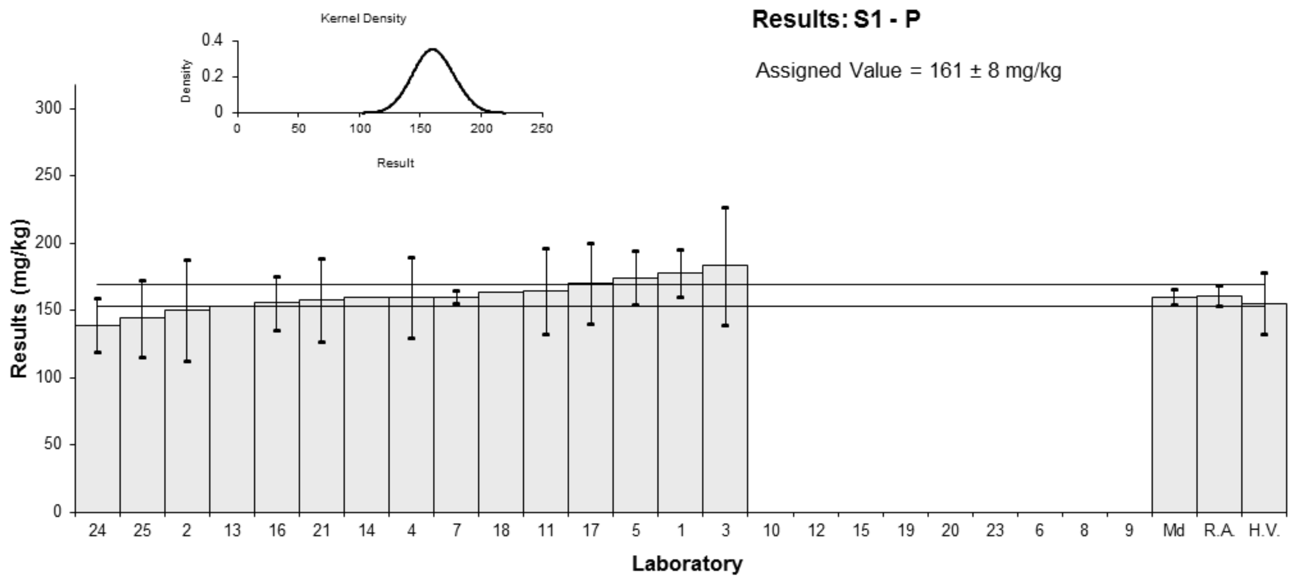
Sample No.	S1
Matrix.	Soil
Analyte.	P
Units	mg/kg

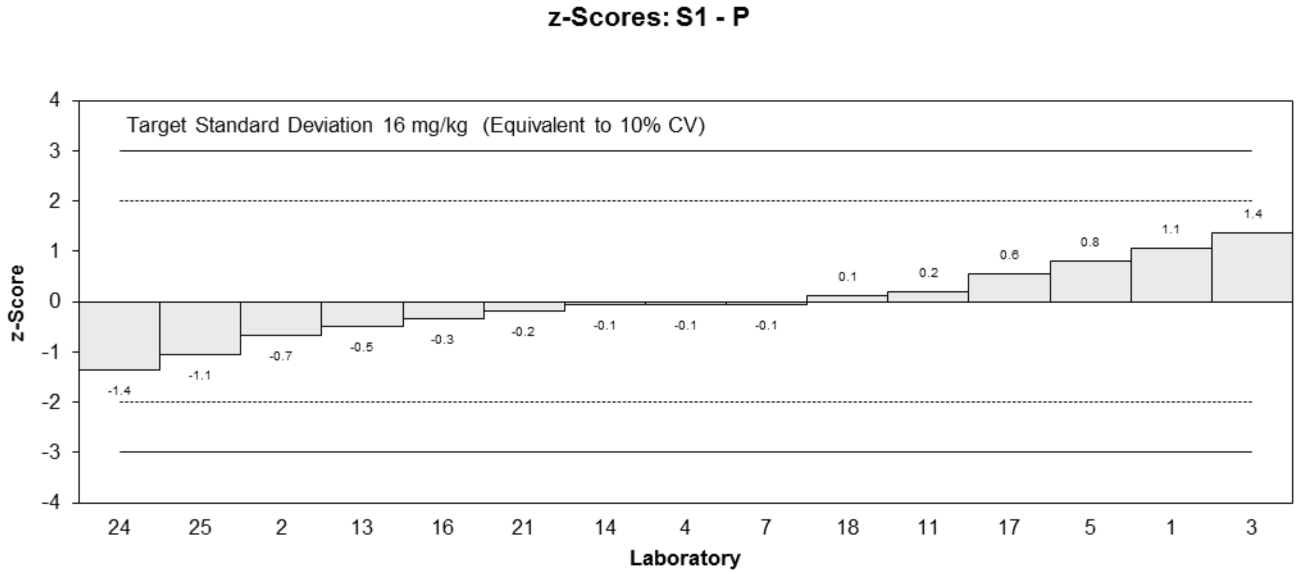
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	178.00	17.80	1.06	0.87
2	150	38	-0.68	-0.28
3	183	43.9	1.37	0.49
4	160	30	-0.06	-0.03
5	174	20	0.81	0.60
6	NT	NT		
7	160	5.0	-0.06	-0.11
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	164	32	0.19	0.09
12	NT	NT		
13	153	NR	-0.50	-1.00
14	160	NR	-0.06	-0.12
15	NT	NT		
16	155.5	20	-0.34	-0.26
17	170	30	0.56	0.29
18	163	NR	0.12	0.25
19	NT	NT		
20	NT	NT		
21	158	31	-0.19	-0.09
23	NT	NT		
24	139	20	-1.37	-1.02
25	144	28.8	-1.06	-0.57

Statistics

Assigned Value	161	8
Spike	Not Spiked	
Homogeneity Value	155	23
Robust Average	161	8
Median	160	6
Mean	161	
N	15	
Max.	183	
Min.	139	
Robust SD	10	
Robust CV	6.2%	


Results: S1 - P
Assigned Value = 161 ± 8 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
24 25 2 13 16 21 14 4 7 18 11 17 5 1 3 10 12 15 19 20 23 6 8 9 Md R.A. H.V.


z-Scores: S1 - P
Target Standard Deviation 16 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
24 25 2 13 16 21 14 4 7 18 11 17 5 1 3

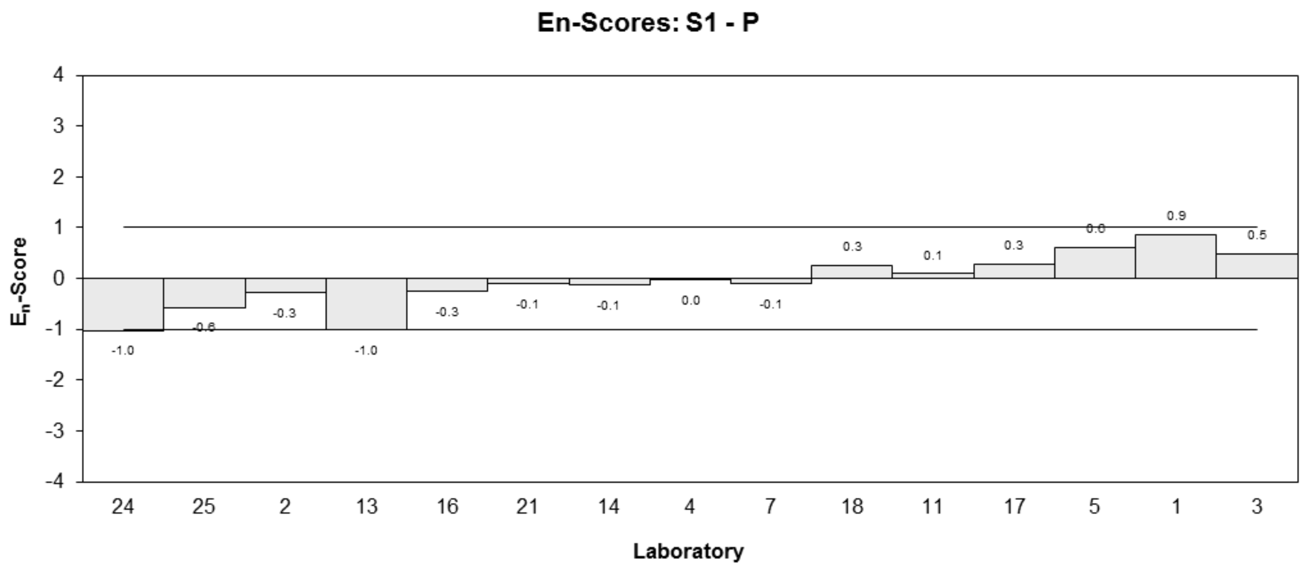

En-Scores: S1 - P
En-Score
Laboratory
24 25 2 13 16 21 14 4 7 18 11 17 5 1 3

Figure 15

Table 26

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	Pb
Units	mg/kg

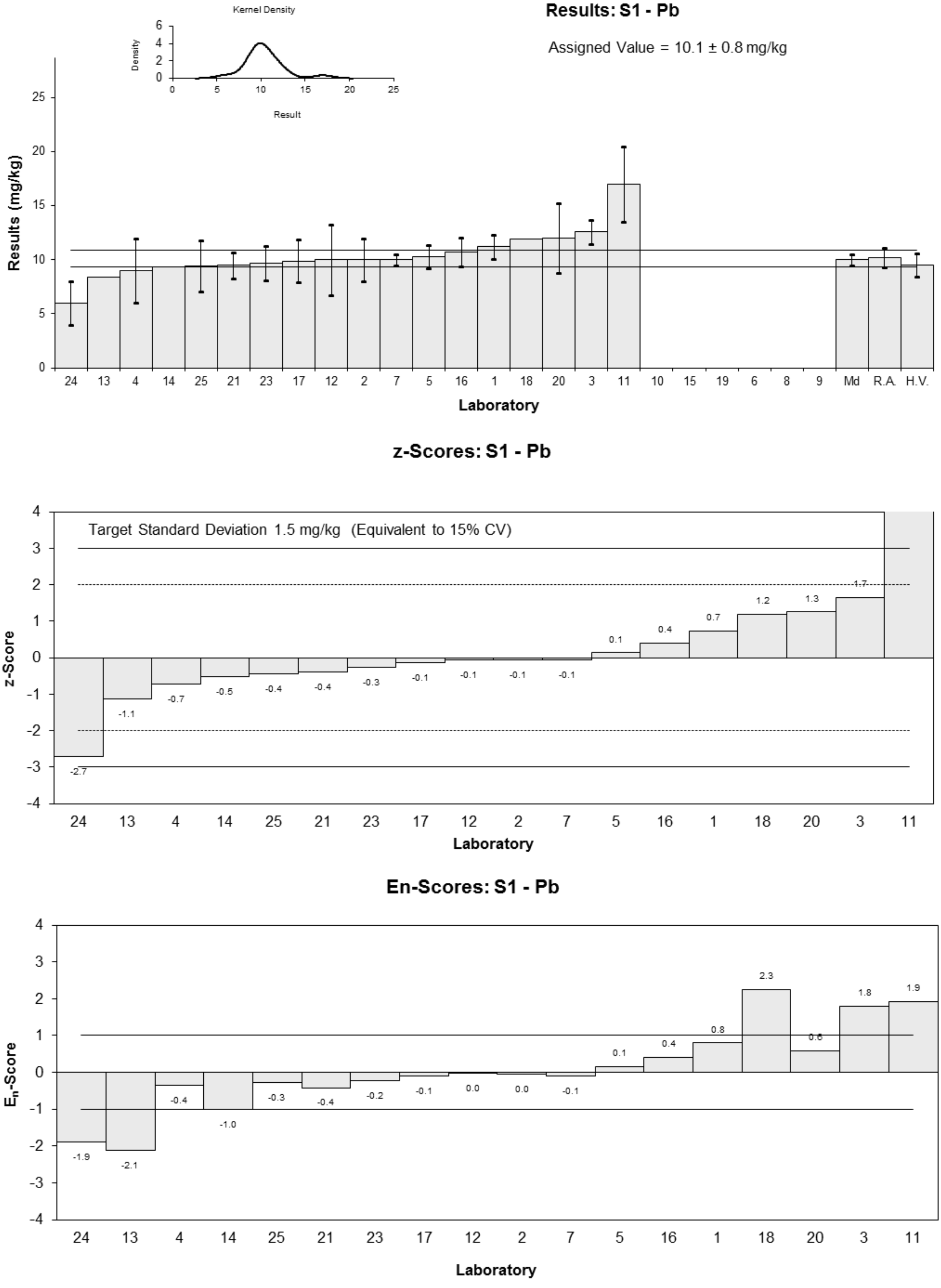
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	11.20	1.12	0.73	0.80
2	10	2	-0.07	-0.05
3	12.6	1.13	1.65	1.81
4	9	3	-0.73	-0.35
5	10.3	1.1	0.13	0.15
6	NT	NT		
7	10	0.5	-0.07	-0.11
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	17	3.5	4.55	1.92
12	10	3.3	-0.07	-0.03
13	8.4	NR	-1.12	-2.12
14	9.3	NR	-0.53	-1.00
15	NT	NT		
16	10.72	1.3	0.41	0.41
17	9.9	2.0	-0.13	-0.09
18	11.9	NR	1.19	2.25
19	NT	NT		
20	12	3.2	1.25	0.58
21	9.5	1.2	-0.40	-0.42
23	9.7	1.6	-0.26	-0.22
24	6	2	-2.71	-1.90
25	9.43	2.36	-0.44	-0.27

Statistics

Assigned Value*	10.1	0.8
Spike	Not Spiked	
Homogeneity Value	9.5	1.1
Robust Average	10.2	0.9
Median	10.0	0.5
Mean	10.4	
N	18	
Max.	17	
Min.	6	
Robust SD	1.0	
Robust CV	9.8%	

*Robust Average excluding Laboratory 11.



Kernel Density
Density
Result
Results: S1 - Pb
Assigned Value = 10.1 ± 0.8 mg/kg
Results (mg/kg)
24 13 4 14 25 21 23 17 12 2 7 5 16 1 18 20 3 11 10 15 19 6 8 9 Md R.A. H.V.
Laboratory
z-Scores: S1 - Pb
Target Standard Deviation 1.5 mg/kg (Equivalent to 15% CV)
z-Score
-2.7 -1.1 -0.7 -0.5 -0.4 -0.4 -0.3 -0.1 -0.1 -0.1 -0.1 0.1 0.4 0.7 1.2 1.3 1.7
24 13 4 14 25 21 23 17 12 2 7 5 16 1 18 20 3 11
Laboratory
En-Scores: S1 - Pb
En-Score
-1.9 -2.1 -0.4 -1.0 -0.3 -0.4 -0.2 -0.1 0.0 0.0 -0.1 0.1 0.4 0.8 2.3 0.6 1.8 1.9
24 13 4 14 25 21 23 17 12 2 7 5 16 1 18 20 3 11
Laboratory

Figure 16

Table 27

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	Rb
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	7.17	0.72
2	NT	NT
3	NT	NT
4	NT	NT
5	NT	NT
6	NT	NT
7	NT	NT
8	NT	NT
9	NT	NT
10	NT	NT
11	NT	NT
12	NT	NT
13	NT	NT
14	4.9	NR
15	NT	NT
16	NR	NR
17	NT	NT
18	NR	NR
19	NT	NT
20	NT	NT
21	5.6	1.2
23	NT	NT
24	NR	NR
25	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	7.9	1.2
Robust Average	5.9	1.9
Median	5.6	2.6
Mean	5.9	
N	3	
Max.	7.17	
Min.	4.9	
Robust SD	1.3	
Robust CV	22%	

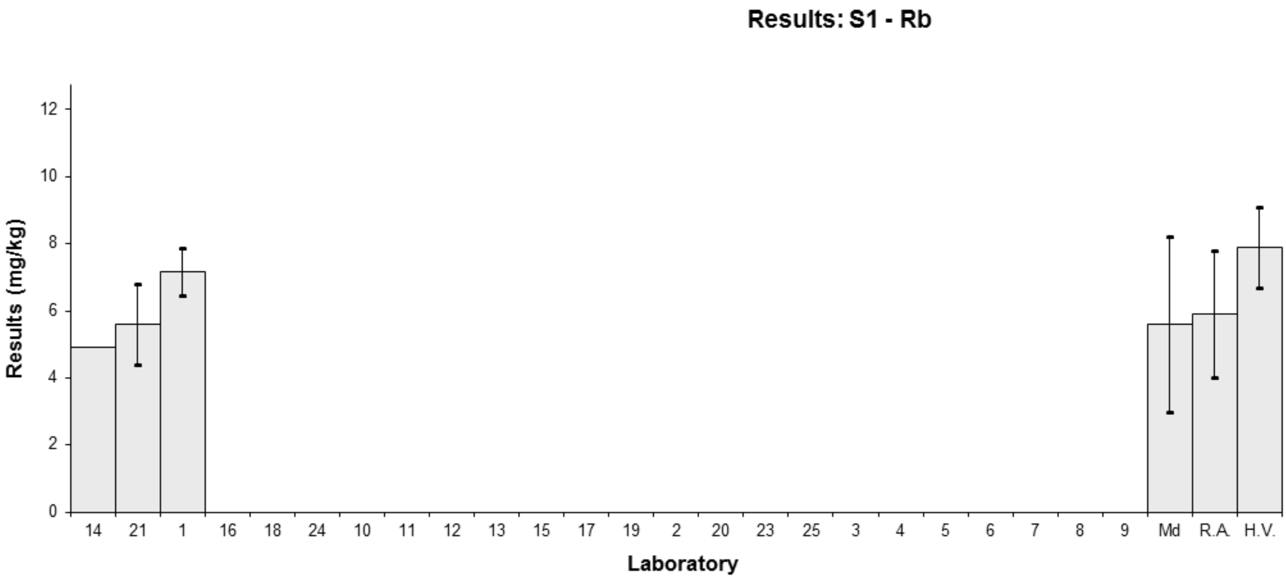

Results: S1 - Rb
Results (mg/kg)
0
2
4
6
8
10
12
14
21
1
16
18
24
10
11
12
13
15
17
19
2
20
23
25
3
4
5
6
7
8
9
Md
R.A.
H.V.
Laboratory

Figure 17

Table 28

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	Se
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	NR	NR
2	<1	1
3	<5	NR
4	<4	NR
5	0.16	0.05
6	NT	NT
7	0.15	0.02
8	NT	NT
9	NT	NT
10	NT	NT
11	<5	NR
12	NT	NT
13	<1	NR
14	<1	NR
15	NT	NT
16	0.1734	0.04
17	<0.5	NR
18	0.87	NR
19	NT	NT
20	<1	NR
21	<2	1.4
23	<3	NR
24	1	1
25	<3	3.00

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.26	0.05
Robust Average	0.47	0.54
Median	0.17	0.04
Mean	0.47	
N	5	
Max.	1	
Min.	0.15	
Robust SD	0.48	
Robust CV	102%	

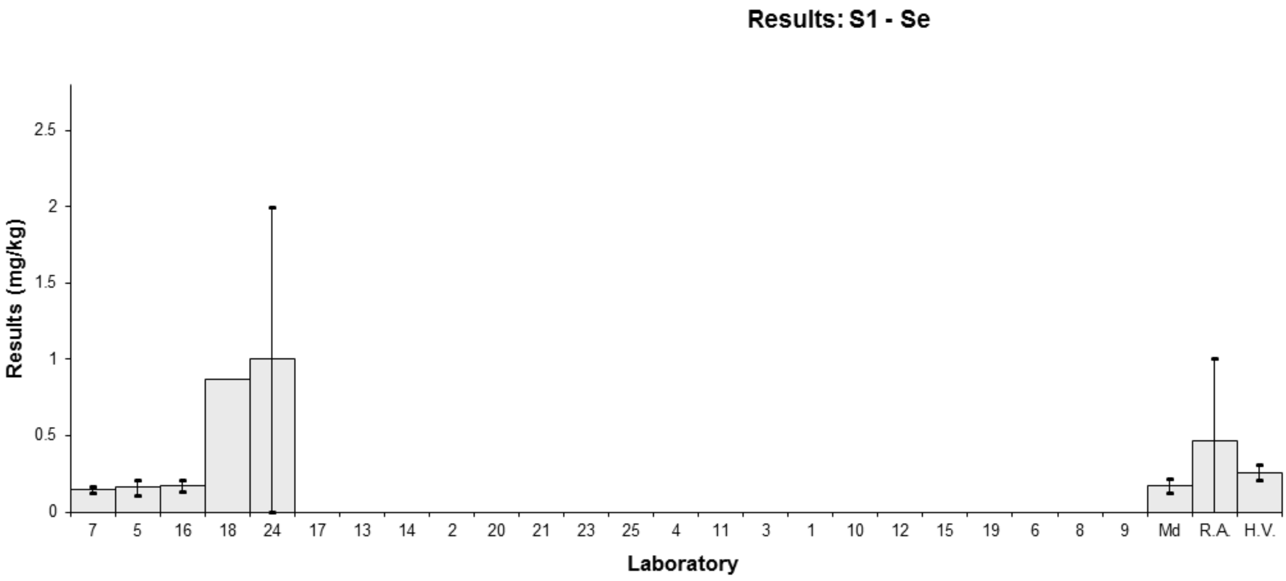

Results: S1 - Se
Results (mg/kg)
2.5
2
1.5
1
0.5
0
7
5
16
18
24
17
13
14
2
20
21
23
25
4
11
3
1
10
12
15
19
6
8
9
Md
R.A.
H.V.
Laboratory

Figure 18

Table 29

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	Sn
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.92	0.09	-0.31	-0.27
2	1.0	0.5	0.10	0.04
3	<5	NR		
4	<2	NR		
5	1.18	0.3	1.02	0.55
6	NT	NT		
7	1.2	0.1	1.12	0.98
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	1.3	0.3	1.63	0.89
12	NT	NT		
13	<5	NR		
14	0.8	NR	-0.92	-0.90
15	NT	NT		
16	0.9852	0.1	0.03	0.02
17	0.67	0.14	-1.58	-1.27
18	NR	NR		
19	NT	NT		
20	<10	NR		
21	0.77	0.13	-1.07	-0.88
23	<2	NR		
24	NR	NR		
25	<3	3.00		

Statistics

Assigned Value	0.98	0.20
Spike	Not Spiked	
Homogeneity Value	0.86	0.13
Robust Average	0.98	0.20
Median	0.99	0.22
Mean	0.98	
N	9	
Max.	1.3	
Min.	0.67	
Robust SD	0.24	
Robust CV	25%	

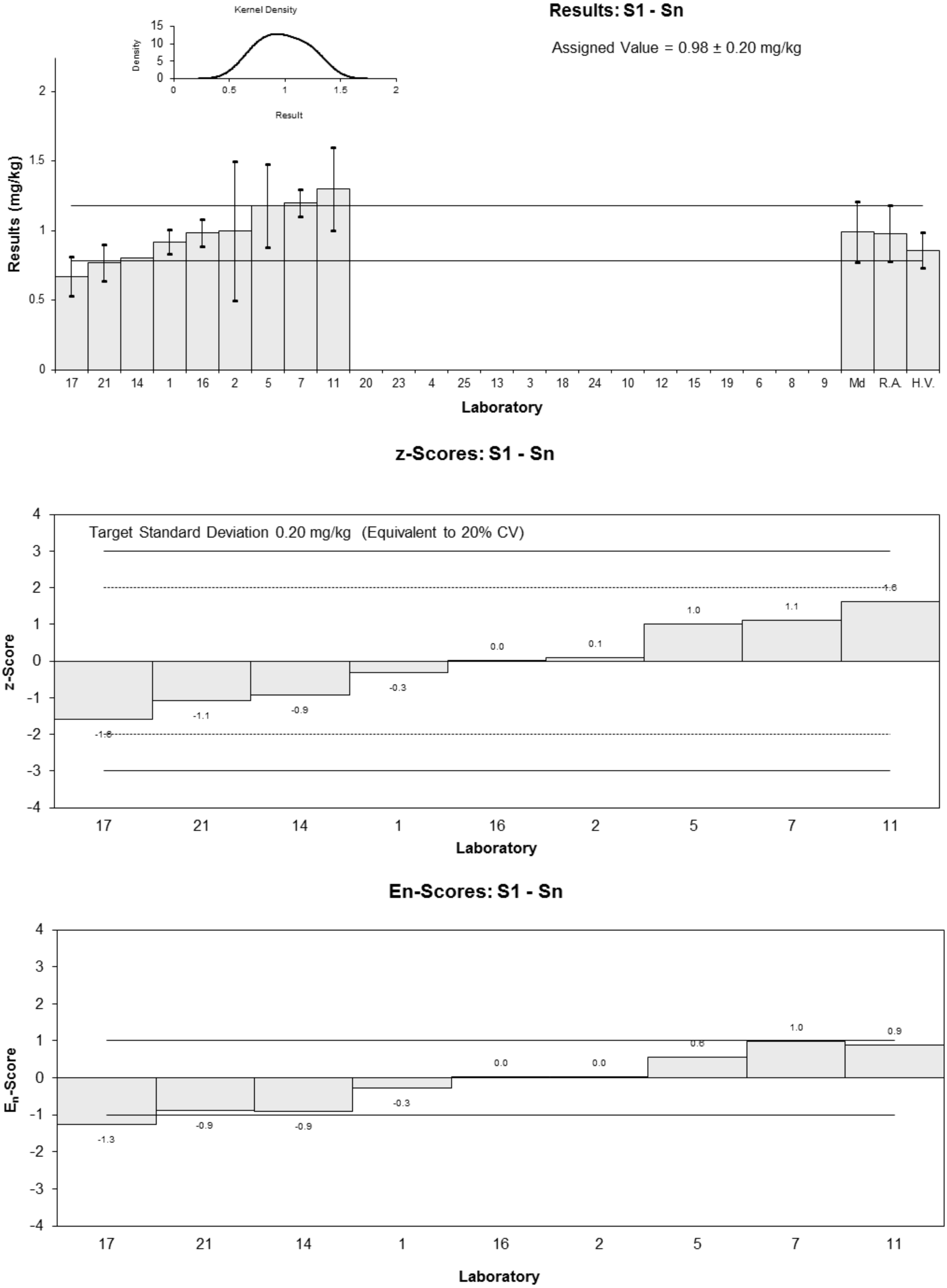

Results: S1 - Sn
Assigned Value = 0.98 ± 0.20 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
17 21 14 1 16 2 5 7 11 20 23 4 25 13 3 18 24 10 12 15 19 6 8 9 Md R.A. H.V.
z-Scores: S1 - Sn
Target Standard Deviation 0.20 mg/kg (Equivalent to 20% CV)
z-Score
-1.6 -1.1 -0.9 -0.3 0.0 0.1 1.0 1.1 1.6
17 21 14 1 16 2 5 7 11
Laboratory
En-Scores: S1 - Sn
En-Score
-1.3 -0.9 -0.9 -0.3 0.0 0.0 0.6 1.0 0.9
17 21 14 1 16 2 5 7 11
Laboratory

Figure 19

Table 30

Sample Details

Sample No.	S1
Matrix.	Soil
Analyte.	Zn
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	16.90	1.69	1.05	1.08
2	14	2	-0.27	-0.25
3	22.7	2.72	3.70	2.69
4	16	4	0.64	0.33
5	14.8	2.0	0.09	0.08
6	NT	NT		
7	15	0.5	0.18	0.29
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	12	2	-1.19	-1.09
12	13	0.7	-0.73	-1.08
13	14.5	NR	-0.05	-0.08
14	10.9	NR	-1.69	-2.85
15	NT	NT		
16	16.09	3.6	0.68	0.39
17	17	3	1.10	0.73
18	17.9	NR	1.51	2.54
19	NT	NT		
20	16	3.7	0.64	0.36
21	13.3	2.2	-0.59	-0.51
23	15	2.8	0.18	0.13
24	14	5	-0.27	-0.12
25	11.7	1.85	-1.32	-1.28

Statistics

Assigned Value*	14.6	1.3
Spike	Not Spiked	
Homogeneity Value	15.7	2.4
Robust Average	14.8	1.4
Median	14.9	1.0
Mean	15.0	
N	18	
Max.	22.7	
Min.	10.9	
Robust SD	2.2	
Robust CV	15%	

*Robust Average excluding Laboratory 3.

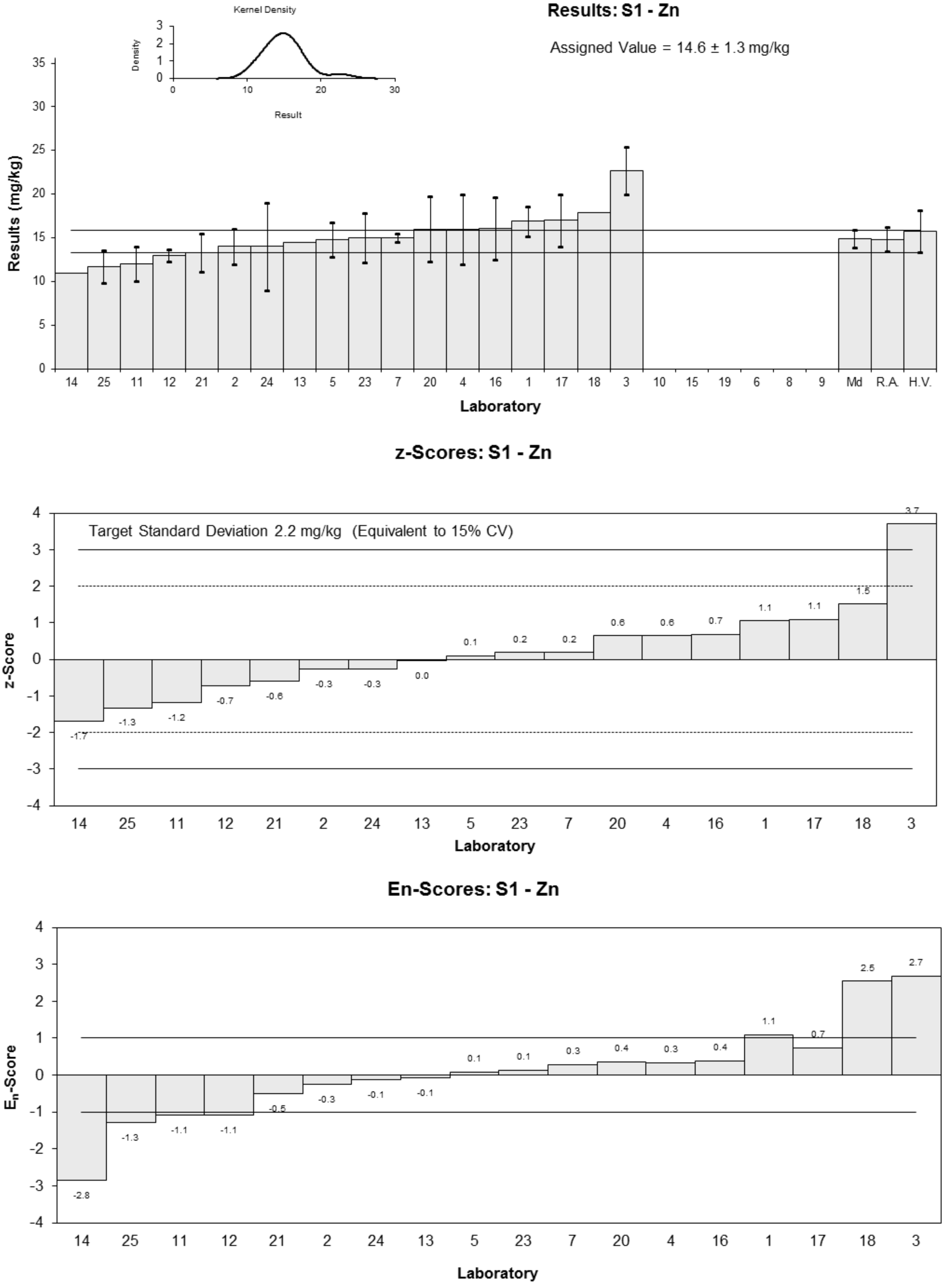

Results: S1 - Zn
Assigned Value = 14.6 ± 1.3 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
14 25 11 12 21 2 24 13 5 23 7 20 4 16 1 17 18 3 10 15 19 6 8 9 Md R.A. H.V.
Laboratory
z-Scores: S1 - Zn
Target Standard Deviation 2.2 mg/kg (Equivalent to 15% CV)
z-Score
-1.7 -1.3 -1.2 -0.7 -0.6 -0.3 -0.3 0.0 0.1 0.2 0.2 0.6 0.6 0.7 1.1 1.1 1.5 3.7
14 25 11 12 21 2 24 13 5 23 7 20 4 16 1 17 18 3
Laboratory
En-Scores: S1 - Zn
E_n-Score
-2.8 -1.3 -1.1 -1.1 -0.5 -0.3 -0.1 -0.1 0.1 0.1 0.3 0.4 0.3 0.4 1.1 0.7 2.5 2.7
14 25 11 12 21 2 24 13 5 23 7 20 4 16 1 17 18 3
Laboratory

Figure 20

Table 31

Sample Details

Sample No.	S2
Matrix.	Sediment
Analyte.	Al
Units	mg/kg

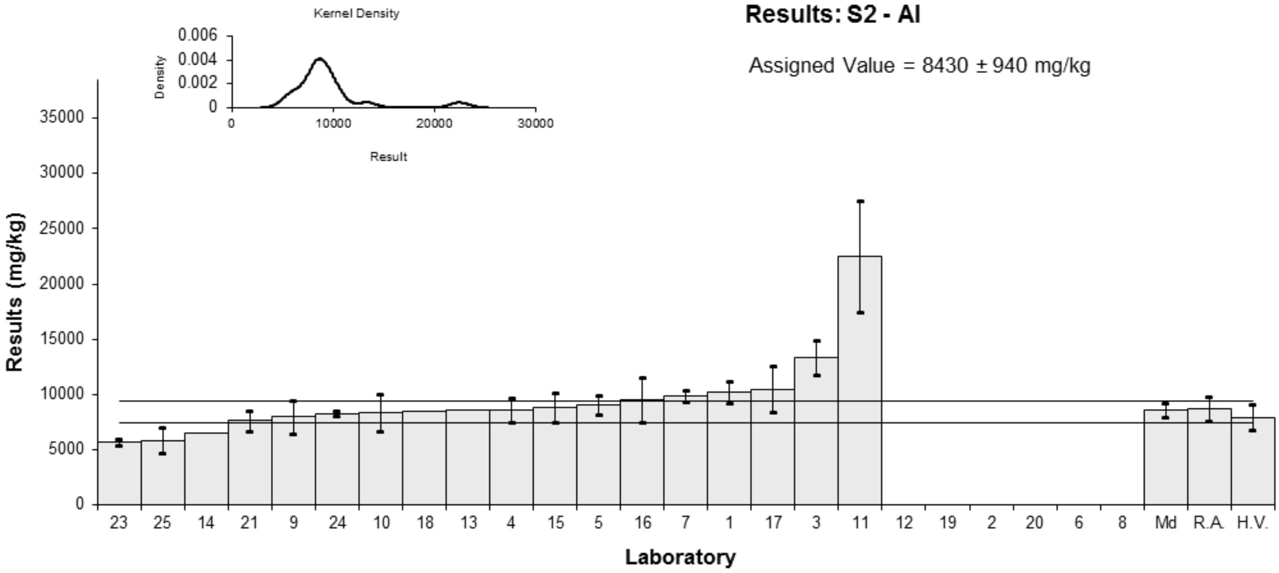
Participant Results

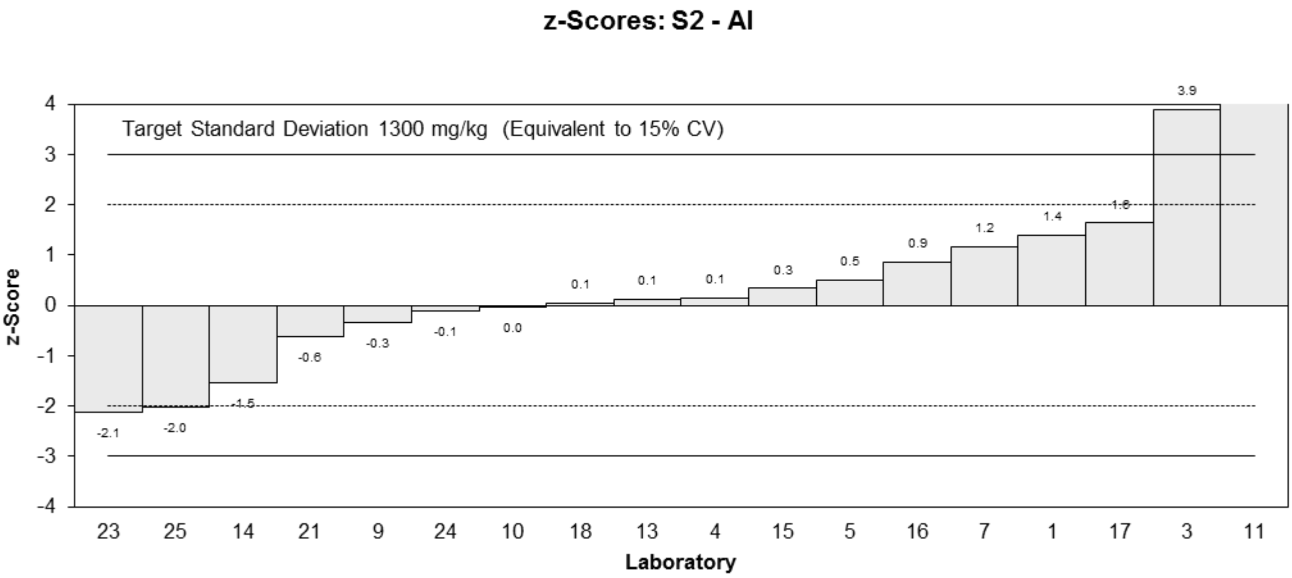
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	10200.00	1020.00	1.40	1.28
2	NT	NT		
3	13362	1603	3.90	2.65
4	8600	1100	0.13	0.12
5	9075	900	0.51	0.50
6	NT	NT		
7	9900	500	1.16	1.38
8	NT	NT		
9	8000	1520	-0.34	-0.24
10	8382.92	1676.58	-0.04	-0.02
11	22500	5000	11.13	2.77
12	NT	NT		
13	8575	NR	0.11	0.15
14	6475	NR	-1.55	-2.08
15	8857	1328	0.34	0.26
16	9507	2000	0.85	0.49
17	10500	2100	1.64	0.90
18	8495	NR	0.05	0.07
19	NT	NT		
20	NT	NT		
21	7640	920	-0.62	-0.60
23	5740	286	-2.13	-2.74
24	8293	200	-0.11	-0.14
25	5870	1109	-2.02	-1.76

Statistics

Assigned Value*	8430	940
Spike	Not Spiked	
Homogeneity Value	7950	1200
Robust Average	8700	1100
Median	8590	690
Mean	9443	
N	18	
Max.	22500	
Min.	5740	
Robust SD	1500	
Robust CV	17%	

*Robust Average excluding Laboratories 3 and 11.


Kernel Density
Results: S2 - Al
Assigned Value = 8430 ± 940 mg/kg
Results (mg/kg)
Laboratory
23 25 14 21 9 24 10 18 13 4 15 5 16 7 1 17 3 11 12 19 2 20 6 8 Md R.A. H.V.


z-Scores: S2 - Al
Target Standard Deviation 1300 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
23 25 14 21 9 24 10 18 13 4 15 5 16 7 1 17 3 11

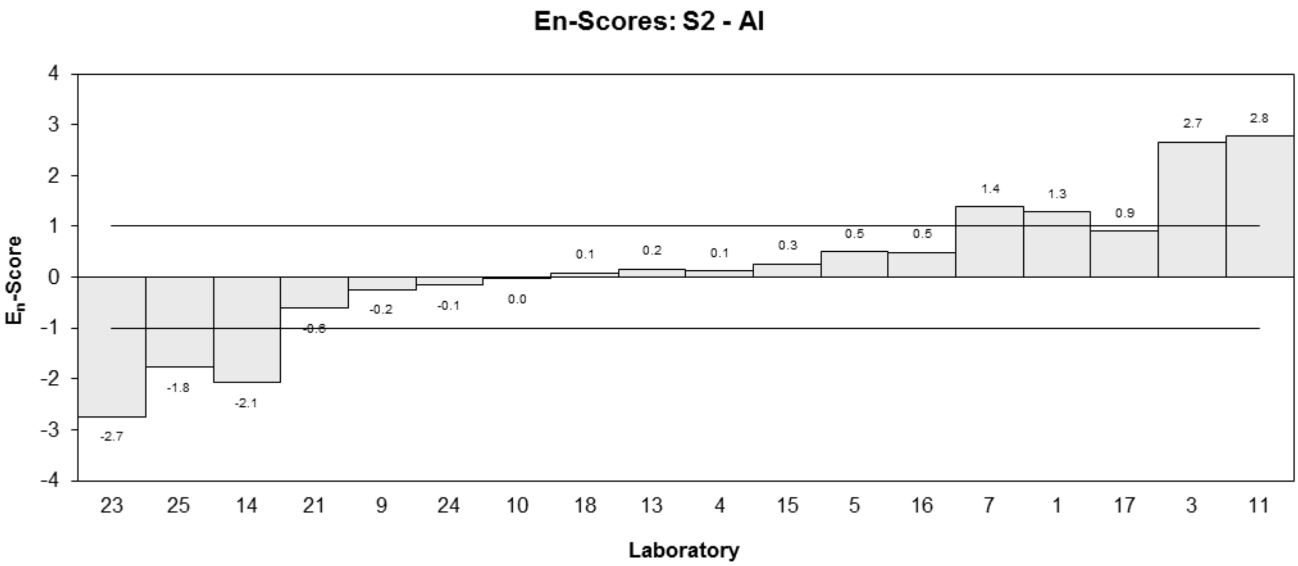

En-Scores: S2 - Al
Laboratory
23 25 14 21 9 24 10 18 13 4 15 5 16 7 1 17 3 11

Figure 21

Table 32

Sample Details

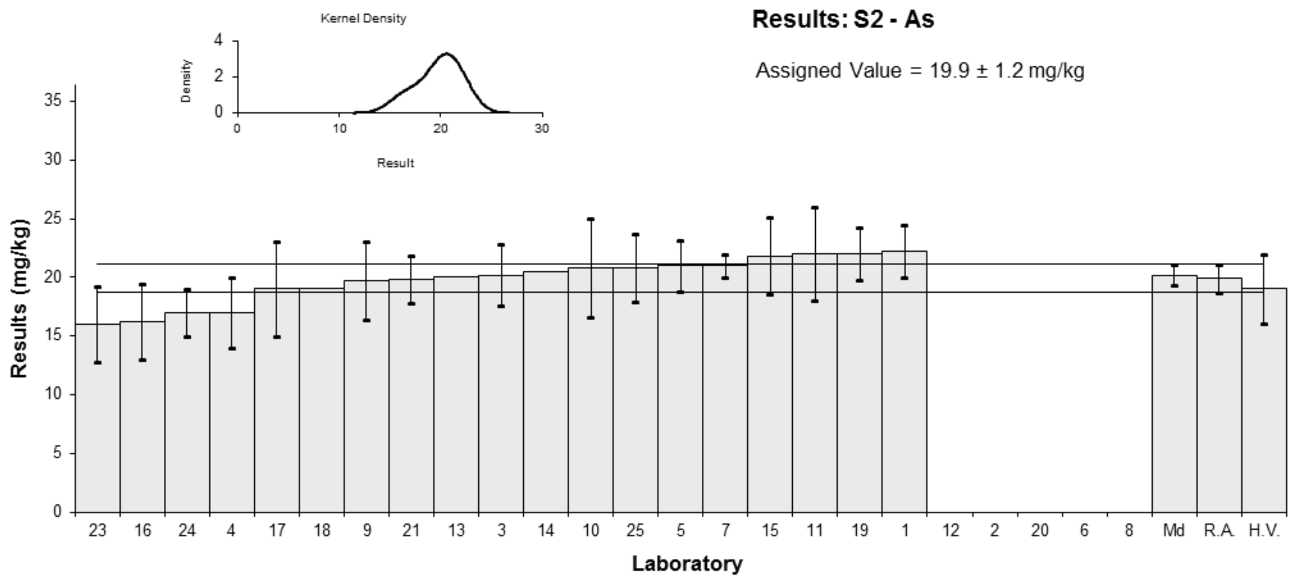
Sample No.	S2
Matrix.	Sediment
Analyte.	As
Units	mg/kg

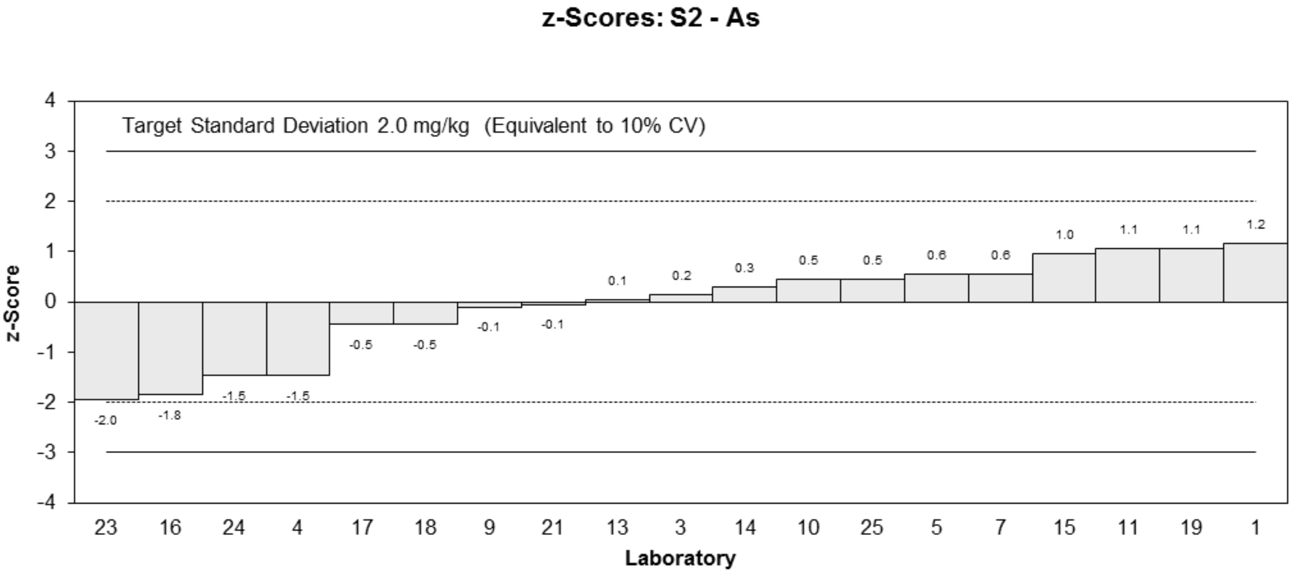
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	22.20	2.22	1.16	0.91
2	NT	NT		
3	20.2	2.67	0.15	0.1
4	17	3	-1.46	-0.9
5	21.0	2.2	0.55	0.44
6	NT	NT		
7	21	1.0	0.55	0.7
8	NT	NT		
9	19.7	3.34	-0.1	-0.06
10	20.79	4.16	0.45	0.21
11	22	4	1.06	0.5
12	NT	NT		
13	20	NR	0.05	0.08
14	20.5	NR	0.3	0.5
15	21.8	3.27	0.95	0.55
16	16.23	3.2	-1.84	-1.07
17	19	4	-0.45	-0.22
18	19.0	NR	-0.45	-0.75
19	22	2.2	1.06	0.84
20	NT	NT		
21	19.8	2.0	-0.05	-0.04
23	16	3.2	-1.96	-1.14
24	17	2	-1.46	-1.24
25	20.8	2.87	0.45	0.29

Statistics

Assigned Value	19.9	1.2
Spike	Not Spiked	
Homogeneity Value	19.0	2.9
Robust Average	19.9	1.2
Median	20.2	0.9
Mean	19.8	
N	19	
Max.	22.2	
Min.	16	
Robust SD	2.1	
Robust CV	11%	


Results: S2 - As
Assigned Value = 19.9 ± 1.2 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
23 16 24 4 17 18 9 21 13 3 14 10 25 5 7 15 11 19 1 12 2 20 6 8 Md R.A. H.V.


z-Scores: S2 - As
Target Standard Deviation 2.0 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
23 16 24 4 17 18 9 21 13 3 14 10 25 5 7 15 11 19 1
-2.0 -1.8 -1.5 -1.5 -0.5 -0.5 -0.1 -0.1 0.1 0.2 0.3 0.5 0.5 0.6 0.6 1.0 1.1 1.1 1.2

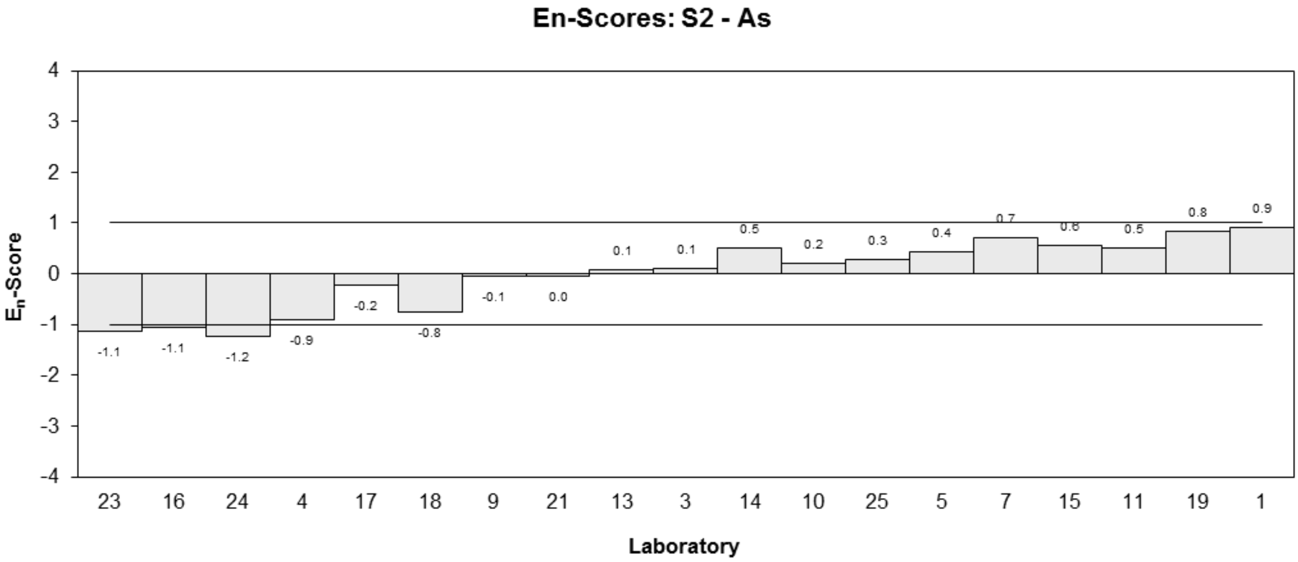

En-Scores: S2 - As
E_n-Score
Laboratory
23 16 24 4 17 18 9 21 13 3 14 10 25 5 7 15 11 19 1
-1.1 -1.1 -1.2 -0.9 -0.2 -0.8 -0.1 0.0 0.1 0.1 0.5 0.2 0.3 0.4 0.7 0.6 0.5 0.8 0.9

Figure 22

Table 33

Sample Details

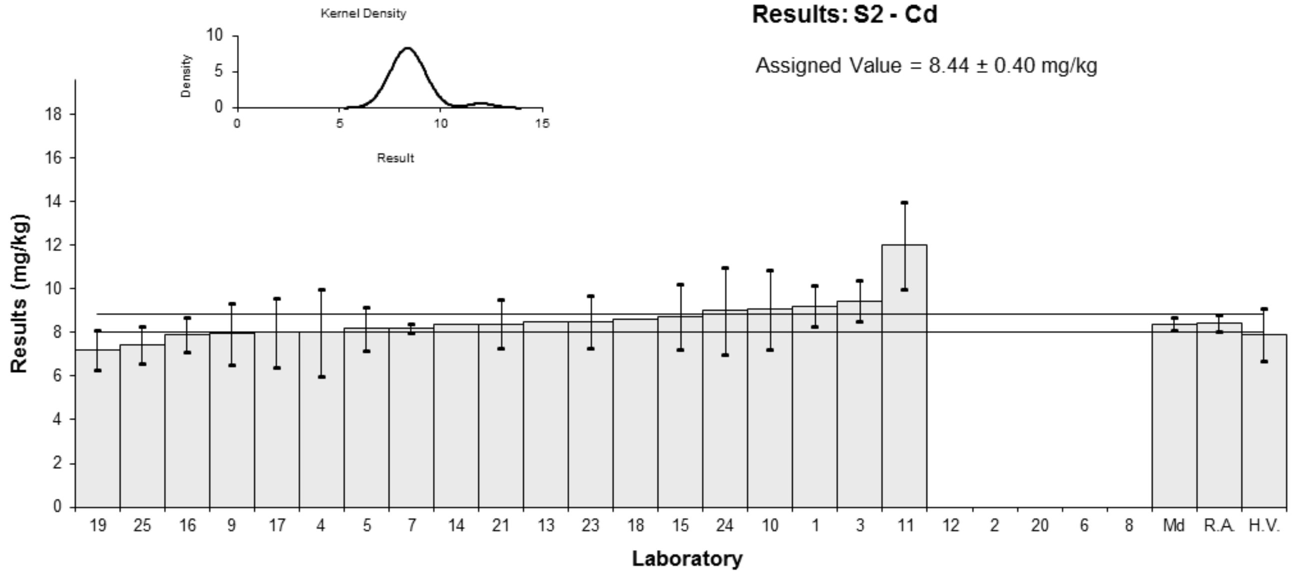
Sample No.	S2
Matrix.	Sediment
Analyte.	Cd
Units	mg/kg

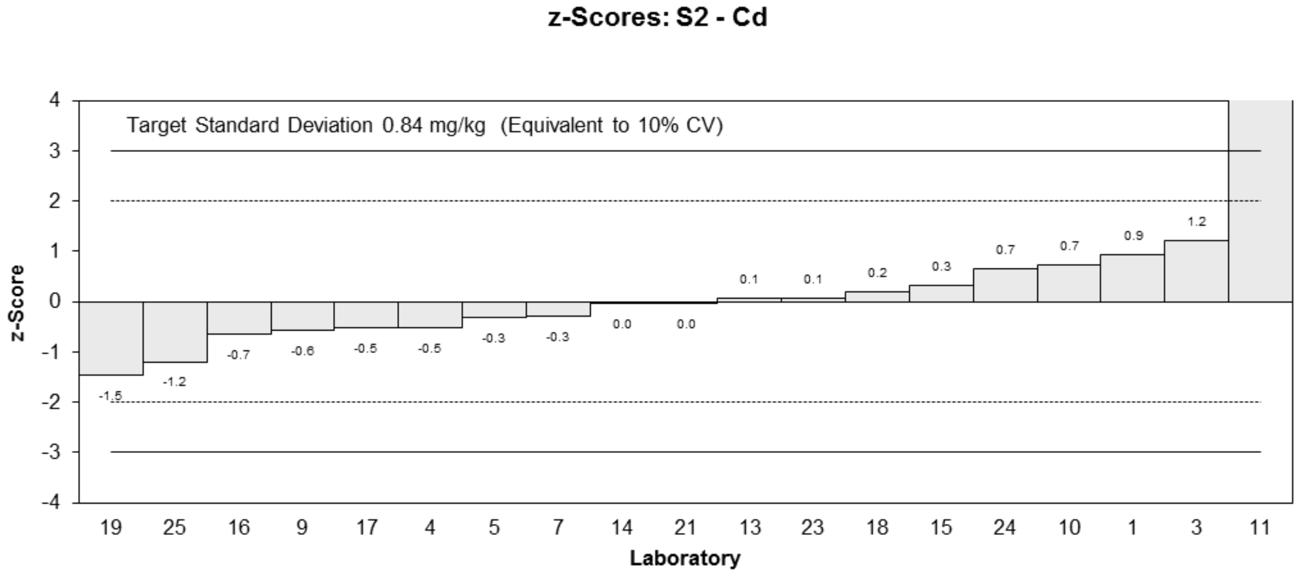
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	9.23	0.92	0.94	0.79
2	NT	NT		
3	9.46	0.946	1.21	0.99
4	8	2	-0.52	-0.22
5	8.18	1.0	-0.31	-0.24
6	NT	NT		
7	8.2	0.2	-0.28	-0.54
8	NT	NT		
9	7.95	1.42	-0.58	-0.33
10	9.06	1.81	0.73	0.33
11	12	2	4.22	1.75
12	NT	NT		
13	8.5	NR	0.07	0.15
14	8.4	NR	-0.05	-0.10
15	8.71	1.5	0.32	0.17
16	7.890	0.8	-0.65	-0.61
17	8.0	1.6	-0.52	-0.27
18	8.60	NR	0.19	0.40
19	7.2	0.9	-1.47	-1.26
20	NT	NT		
21	8.4	1.1	-0.05	-0.03
23	8.5	1.2	0.07	0.05
24	9	2	0.66	0.27
25	7.43	0.869	-1.20	-1.06

Statistics

Assigned Value	8.44	0.40
Spike	Not Spiked	
Homogeneity Value	7.9	1.2
Robust Average	8.44	0.40
Median	8.40	0.29
Mean	8.56	
N	19	
Max.	12	
Min.	7.2	
Robust SD	0.69	
Robust CV	8.2%	


Results: S2 - Cd
Assigned Value = 8.44 ± 0.40 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
19 25 16 9 17 4 5 7 14 21 13 23 18 15 24 10 1 3 11 12 2 20 6 8 Md R.A. H.V.


z-Scores: S2 - Cd
Target Standard Deviation 0.84 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
19 25 16 9 17 4 5 7 14 21 13 23 18 15 24 10 1 3 11
-1.5 -1.2 -0.7 -0.6 -0.5 -0.5 -0.3 -0.3 0.0 0.0 0.1 0.1 0.2 0.3 0.7 0.7 0.9 1.2

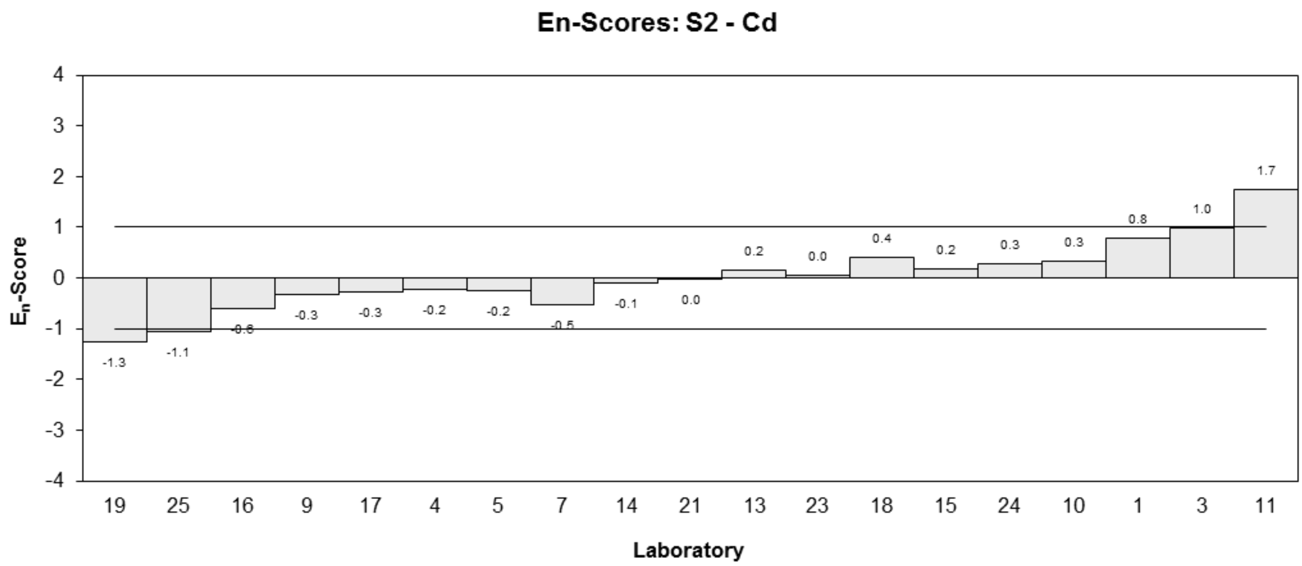

En-Scores: S2 - Cd
En-Score
Laboratory
19 25 16 9 17 4 5 7 14 21 13 23 18 15 24 10 1 3 11
-1.3 -1.1 -0.6 -0.3 -0.3 -0.2 -0.2 -0.5 -0.1 0.0 0.2 0.0 0.4 0.2 0.3 0.3 0.8 1.0 1.7

Figure 23

Table 34

Sample Details

Sample No.	S2
Matrix.	Sediment
Analyte.	Co
Units	mg/kg

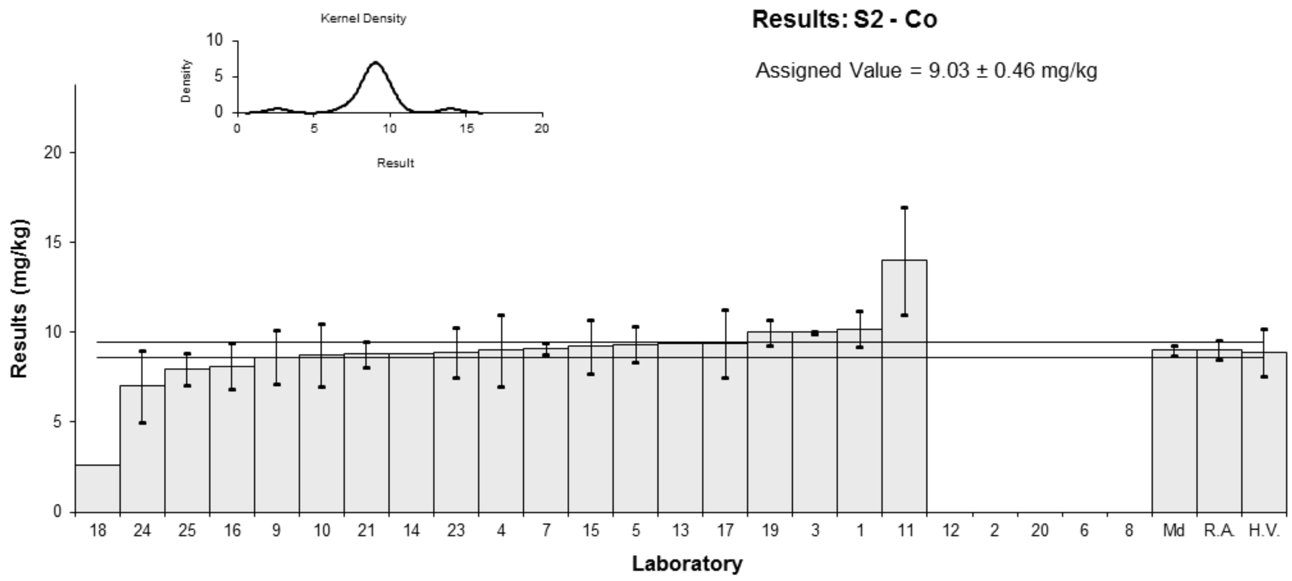
Participant Results

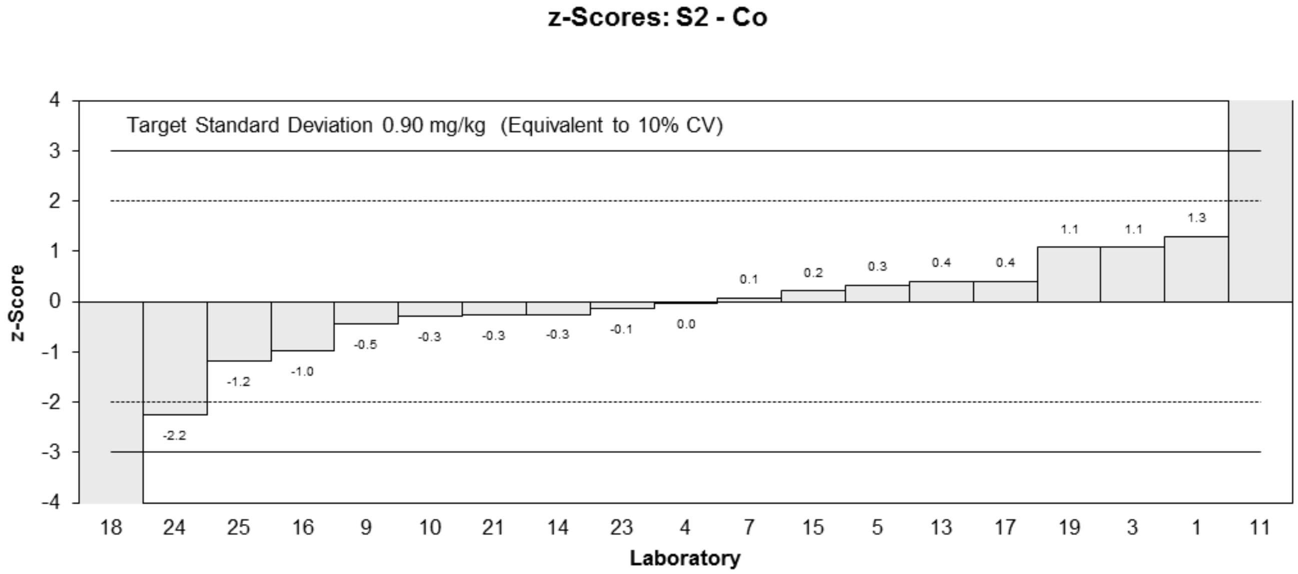
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	10.20	1.02	1.30	1.05
2	NT	NT		
3	10.0	0.100	1.07	2.06
4	9	2	-0.03	-0.01
5	9.33	1.0	0.33	0.27
6	NT	NT		
7	9.1	0.3	0.08	0.13
8	NT	NT		
9	8.62	1.50	-0.45	-0.26
10	8.76	1.75	-0.30	-0.15
11	14	3	5.50	1.64
12	NT	NT		
13	9.4	NR	0.41	0.80
14	8.8	NR	-0.25	-0.50
15	9.22	1.5	0.21	0.12
16	8.139	1.3	-0.99	-0.65
17	9.4	1.9	0.41	0.19
18	2.63	NR	-7.09	-13.91
19	10	0.7	1.07	1.16
20	NT	NT		
21	8.79	0.71	-0.27	-0.28
23	8.9	1.4	-0.14	-0.09
24	7	2	-2.25	-0.99
25	7.97	0.917	-1.17	-1.03

Statistics

Assigned Value*	9.03	0.46
Spike	Not Spiked	
Homogeneity Value	8.9	1.3
Robust Average	9.02	0.52
Median	9.00	0.29
Mean	8.91	
N	19	
Max.	14	
Min.	2.63	
Robust SD	0.76	
Robust CV	8.4%	

*Robust Average excluding Laboratories 11 and 18.


Kernel Density
Density
Result
Results: S2 - Co
Assigned Value = 9.03 ± 0.46 mg/kg
Results (mg/kg)
Laboratory
18 24 25 16 9 10 21 14 23 4 7 15 5 13 17 19 3 1 11 12 2 20 6 8 Md R.A. H.V.


z-Scores: S2 - Co
Target Standard Deviation 0.90 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-2.2 -1.2 -1.0 -0.5 -0.3 -0.3 -0.3 -0.1 0.0 0.1 0.2 0.3 0.4 0.4 1.1 1.1 1.3
18 24 25 16 9 10 21 14 23 4 7 15 5 13 17 19 3 1 11

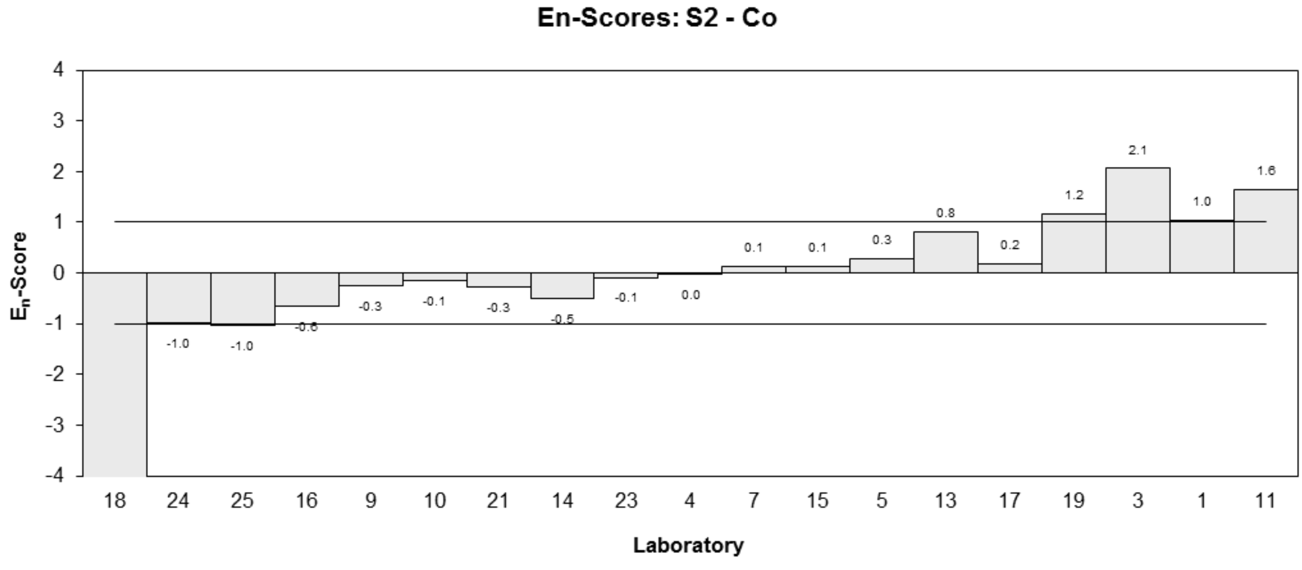

En-Scores: S2 - Co
En-Score
Laboratory
-1.0 -1.0 -0.6 -0.3 -0.1 -0.3 -0.5 -0.1 0.0 0.1 0.1 0.3 0.8 0.2 1.2 2.1 1.0 1.6
18 24 25 16 9 10 21 14 23 4 7 15 5 13 17 19 3 1 11

Figure 24

Table 35

Sample Details

Sample No.	S2
Matrix.	Sediment
Analyte.	Cr
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	23.10	2.31	0.96	0.99
2	NT	NT		
3	10.86	1.30	-3.08	-4.21
4	23	4	0.92	0.64
5	21.4	2.5	0.40	0.39
6	NT	NT		
7	23	1.0	0.92	1.36
8	NT	NT		
9	21.0	3.68	0.26	0.20
10	21.99	4.40	0.59	0.38
11	37	7	5.54	2.32
12	NT	NT		
13	20.6	NR	0.13	0.22
14	16.7	NR	-1.16	-1.94
15	23.0	3.5	0.92	0.71
16	19.72	3.5	-0.16	-0.12
17	23	5	0.92	0.53
18	19.9	NR	-0.10	-0.17
19	32	2.5	3.89	3.83
20	NT	NT		
21	18.5	2.3	-0.56	-0.58
23	18	2.6	-0.73	-0.70
24	19	2	-0.40	-0.45
25	15.6	2.53	-1.52	-1.48

Statistics

Assigned Value*	20.2	1.8
Spike	Not Spiked	
Homogeneity Value	22.0	3.3
Robust Average	20.8	2.0
Median	21.0	1.4
Mean	21.4	
N	19	
Max.	37	
Min.	10.86	
Robust SD	2.9	
Robust CV	14%	

*Robust Average excluding Laboratories 11 and 19.

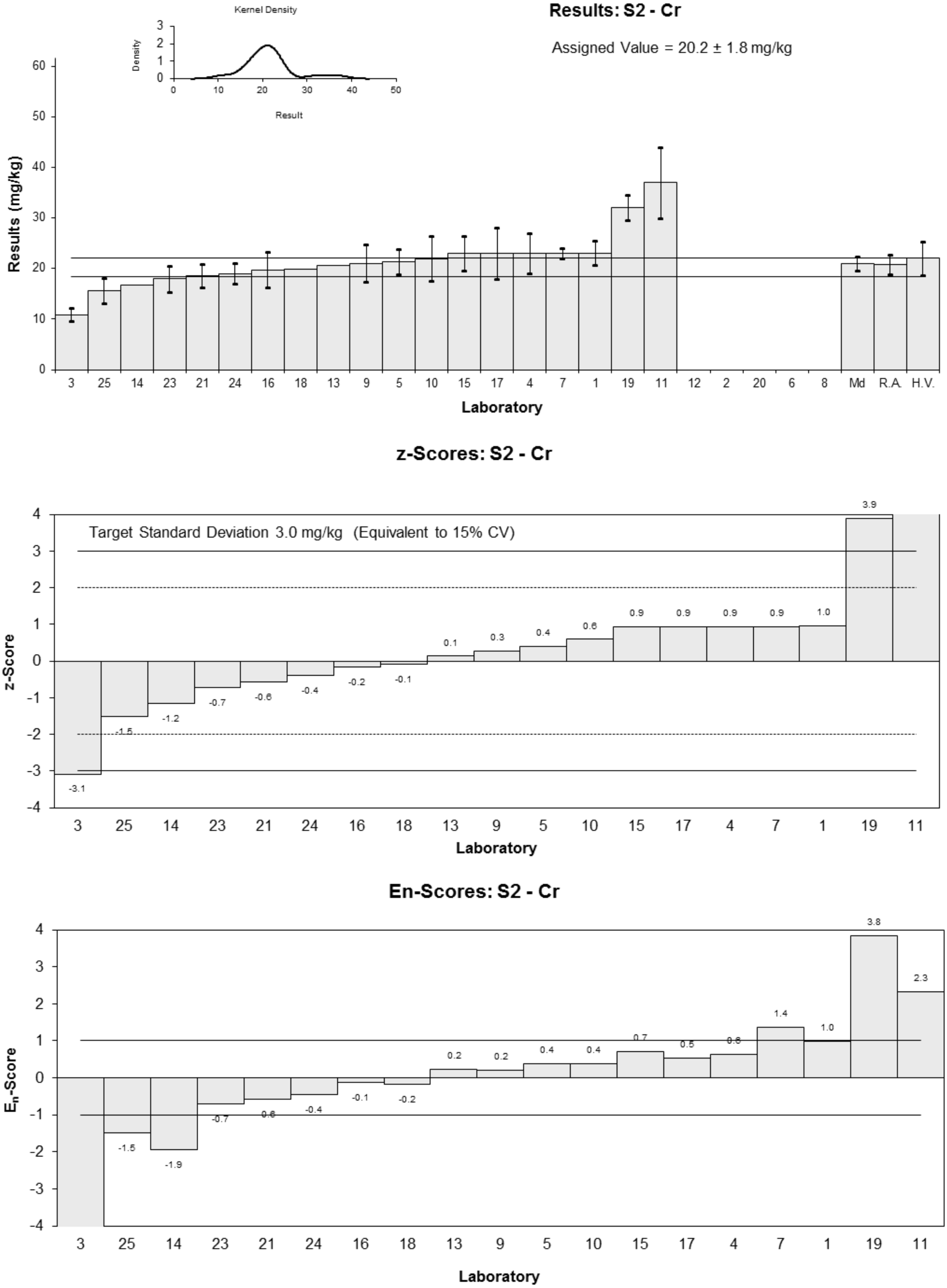

Results: S2 - Cr
Assigned Value = 20.2 ± 1.8 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
3 25 14 23 21 24 16 18 13 9 5 10 15 17 4 7 1 19 11 12 2 20 6 8 Md R.A. H.V.
Laboratory
z-Scores: S2 - Cr
Target Standard Deviation 3.0 mg/kg (Equivalent to 15% CV)
z-Score
-3.1 -1.5 -1.2 -0.7 -0.6 -0.4 -0.2 -0.1 0.1 0.3 0.4 0.6 0.9 0.9 0.9 0.9 1.0 3.9
3 25 14 23 21 24 16 18 13 9 5 10 15 17 4 7 1 19 11
Laboratory
En-Scores: S2 - Cr
En-Score
-1.5 -1.9 -0.7 -0.6 -0.4 -0.1 -0.2 0.2 0.2 0.4 0.4 0.7 0.5 0.6 1.4 1.0 3.8 2.3
3 25 14 23 21 24 16 18 13 9 5 10 15 17 4 7 1 19 11
Laboratory

Figure 25

Table 36

Sample Details

Sample No.	S2
Matrix.	Sediment
Analyte.	Cu
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	27.80	2.78	1.35	1.08
2	NT	NT		
3	23.4	2.81	-0.45	-0.36
4	26	4	0.61	0.36
5	25.4	3.0	0.37	0.28
6	NT	NT		
7	26	1.0	0.61	0.91
8	NT	NT		
9	23.0	4.27	-0.61	-0.34
10	24.15	4.83	-0.14	-0.07
11	42	8	7.14	2.16
12	NT	NT		
13	22.6	NR	-0.78	-1.46
14	23.9	NR	-0.24	-0.46
15	24.4	3.7	-0.04	-0.03
16	20.55	3.2	-1.61	-1.14
17	25	5	0.20	0.10
18	32.8	NR	3.39	6.38
19	25	1.3	0.20	0.27
20	NT	NT		
21	26.9	3.8	0.98	0.60
23	22	2.8	-1.02	-0.81
24	25	2	0.20	0.21
25	21.9	5.23	-1.06	-0.48

Statistics

Assigned Value*	24.5	1.3
Spike	Not Spiked	
Homogeneity Value	25.3	3.8
Robust Average	24.8	1.4
Median	25.0	1.1
Mean	25.7	
N	19	
Max.	42	
Min.	20.55	
Robust SD	2.2	
Robust CV	8.9%	

*Robust Average excluding Laboratory 11.

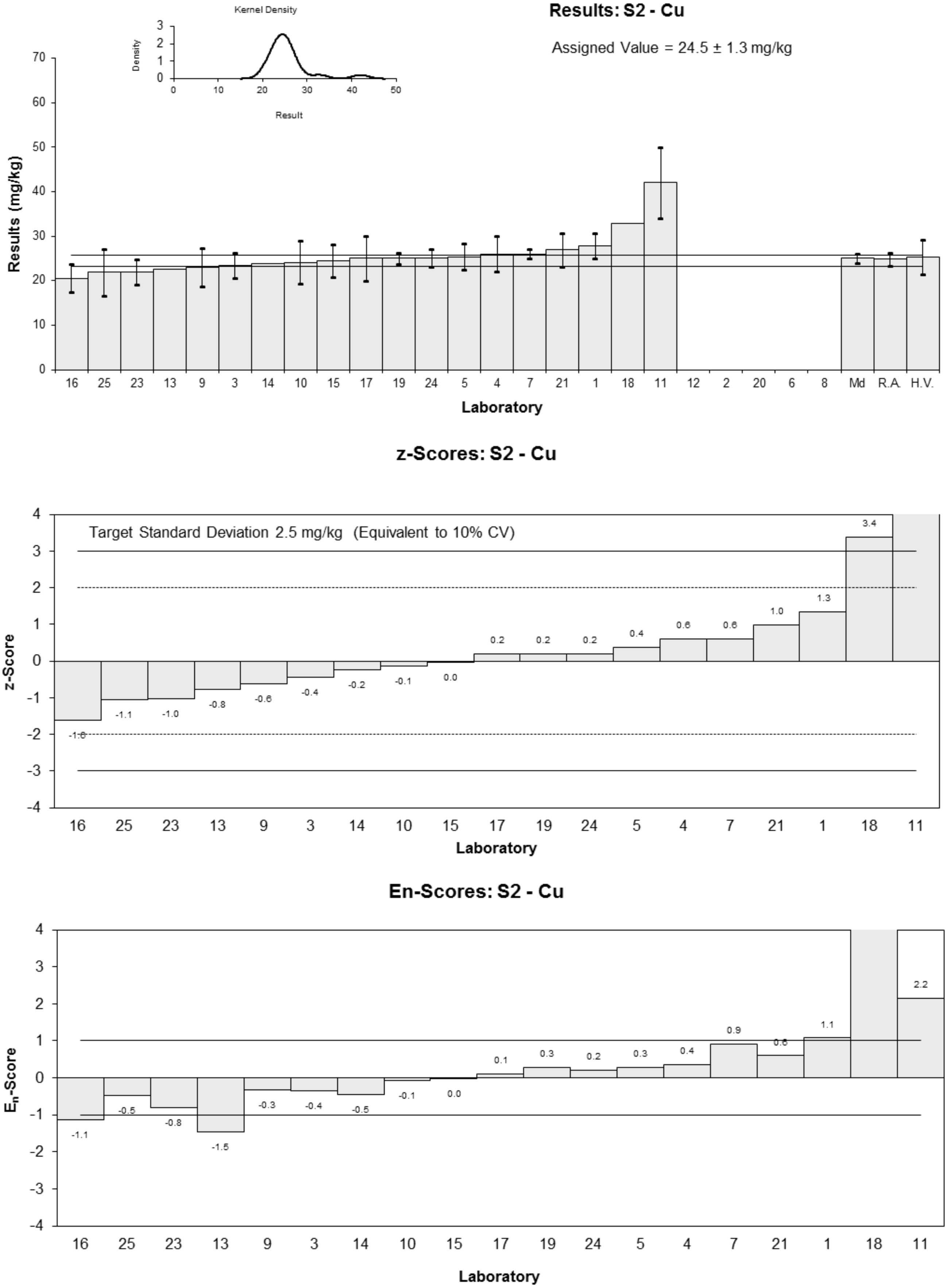

Results: S2 - Cu
Assigned Value = 24.5 ± 1.3 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
16 25 23 13 9 3 14 10 15 17 19 24 5 4 7 21 1 18 11 12 2 20 6 8 Md R.A. H.V.
z-Scores: S2 - Cu
Target Standard Deviation 2.5 mg/kg (Equivalent to 10% CV)
z-Score
-1.6 -1.1 -1.0 -0.8 -0.6 -0.4 -0.2 -0.1 0.0 0.2 0.2 0.2 0.4 0.6 0.6 1.0 1.3 3.4
16 25 23 13 9 3 14 10 15 17 19 24 5 4 7 21 1 18 11
Laboratory
En-Scores: S2 - Cu
E_n-Score
-1.1 -0.5 -0.8 -1.5 -0.3 -0.4 -0.5 -0.1 0.0 0.1 0.3 0.2 0.3 0.4 0.9 0.6 1.1 2.2
16 25 23 13 9 3 14 10 15 17 19 24 5 4 7 21 1 18 11
Laboratory

Figure 26

Table 37

Sample Details

Sample No.	S2
Matrix.	Sediment
Analyte.	Hg
Units	mg/kg

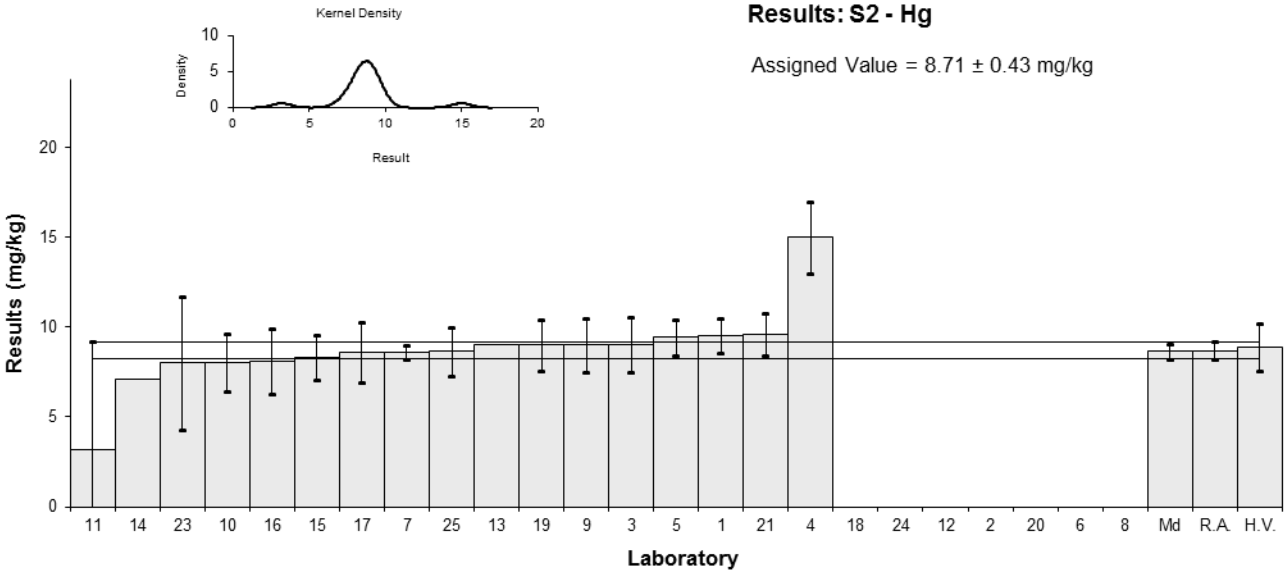
Participant Results

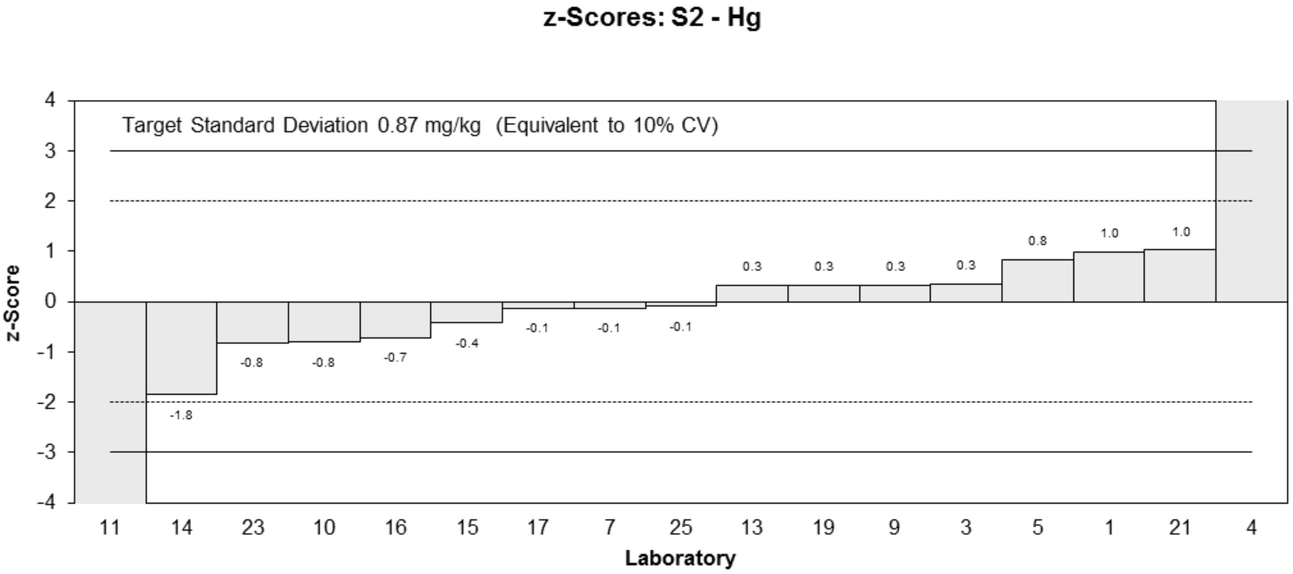
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	9.56	0.96	0.98	0.81
2	NT	NT		
3	9.01	1.53	0.34	0.19
4	15	2	7.22	3.07
5	9.43	1.0	0.83	0.66
6	NT	NT		
7	8.6	0.4	-0.13	-0.19
8	NT	NT		
9	9.00	1.52	0.33	0.18
10	8.02	1.60	-0.79	-0.42
11	3.2	6	-6.33	-0.92
12	NT	NT		
13	9	NR	0.33	0.67
14	7.1	NR	-1.85	-3.74
15	8.34	1.25	-0.42	-0.28
16	8.090	1.8	-0.71	-0.34
17	8.6	1.7	-0.13	-0.06
18	NR	NR		
19	9.0	1.4	0.33	0.20
20	NT	NT		
21	9.6	1.2	1.02	0.70
23	8	3.7	-0.82	-0.19
24	NR	NR		
25	8.64	1.38	-0.08	-0.05

Statistics

Assigned Value*	8.71	0.43
Spike	Not Spiked	
Homogeneity Value	8.9	1.3
Robust Average	8.69	0.51
Median	8.64	0.42
Mean	8.72	
N	17	
Max.	15	
Min.	3.2	
Robust SD	0.67	
Robust CV	7.7%	

*Robust Average excluding Laboratories 4 and 11.


Results: S2 - Hg
Assigned Value = 8.71 ± 0.43 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
11 14 23 10 16 15 17 7 25 13 19 9 3 5 1 21 4 18 24 12 2 20 6 8 Md R.A. H.V.


z-Scores: S2 - Hg
Target Standard Deviation 0.87 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
11 14 23 10 16 15 17 7 25 13 19 9 3 5 1 21 4
-1.8 -0.8 -0.8 -0.7 -0.4 -0.1 -0.1 -0.1 0.3 0.3 0.3 0.3 0.8 1.0 1.0

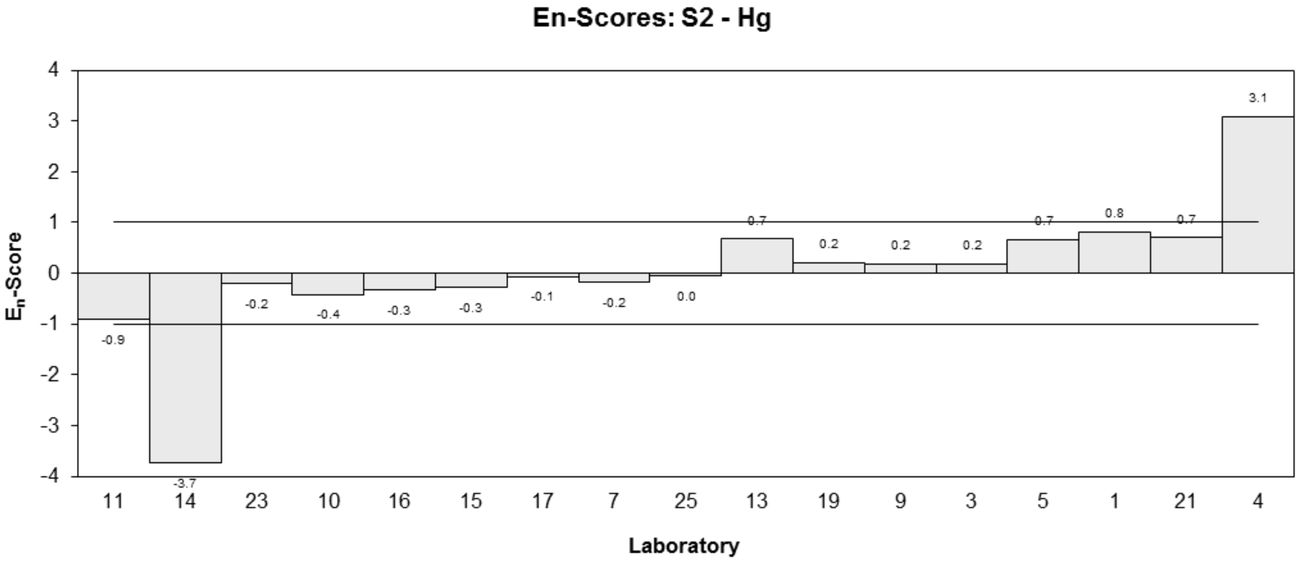

En-Scores: S2 - Hg
En-Score
Laboratory
11 14 23 10 16 15 17 7 25 13 19 9 3 5 1 21 4
-0.9 -3.7 -0.2 -0.4 -0.3 -0.3 -0.1 -0.2 0.0 0.7 0.2 0.2 0.2 0.7 0.8 0.7 3.1

Figure 27

Table 38

Sample Details

Sample No.	S2
Matrix.	Sediment
Analyte.	Mn
Units	mg/kg

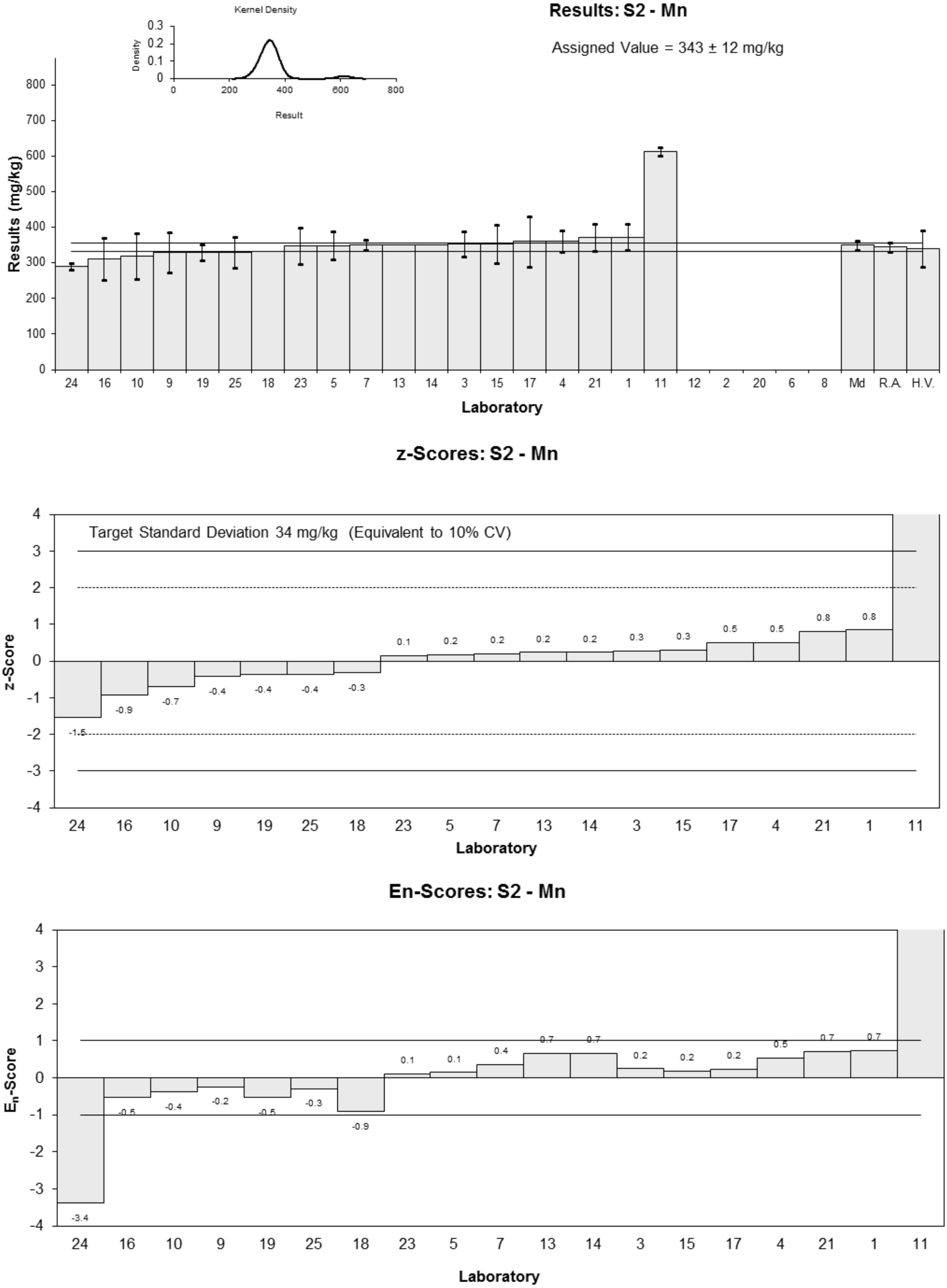
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	372.00	37.20	0.85	0.74
2	NT	NT		
3	352	35.2	0.26	0.24
4	360	30	0.50	0.53
5	349	40	0.17	0.14
6	NT	NT		
7	350	15	0.20	0.36
8	NT	NT		
9	329	57.0	-0.41	-0.24
10	318.97	63.79	-0.70	-0.37
11	613	12	7.87	15.91
12	NT	NT		
13	351	NR	0.23	0.67
14	351	NR	0.23	0.67
15	353	53.0	0.29	0.18
16	311.1	58	-0.93	-0.54
17	360	70	0.50	0.24
18	332	NR	-0.32	-0.92
19	330	22	-0.38	-0.52
20	NT	NT		
21	371	38	0.82	0.70
23	348	51	0.15	0.10
24	290	10	-1.55	-3.39
25	330	43.2	-0.38	-0.29

Statistics

Assigned Value*	343	12
Spike	Not Spiked	
Homogeneity Value	340	50
Robust Average	345	13
Median	350	13
Mean	356	
N	19	
Max.	613	
Min.	290	
Robust SD	21	
Robust CV	6.1%	

*Robust Average excluding Laboratory 11.



Results: S2 - Mn
Assigned Value = 343 ± 12 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
24 16 10 9 19 25 18 23 5 7 13 14 3 15 17 4 21 1 11 12 2 20 6 8 Md R.A. H.V.
z-Scores: S2 - Mn
Target Standard Deviation 34 mg/kg (Equivalent to 10% CV)
z-Score
-1.5 -0.9 -0.7 -0.4 -0.4 -0.4 -0.3 0.1 0.2 0.2 0.2 0.2 0.3 0.3 0.5 0.5 0.8 0.8
24 16 10 9 19 25 18 23 5 7 13 14 3 15 17 4 21 1 11
Laboratory
En-Scores: S2 - Mn
E_n-Score
-3.4 -0.5 -0.4 -0.2 -0.5 -0.3 -0.9 0.1 0.1 0.4 0.7 0.7 0.2 0.2 0.2 0.5 0.7 0.7
24 16 10 9 19 25 18 23 5 7 13 14 3 15 17 4 21 1 11
Laboratory

Figure 28

Table 39

Sample Details

Sample No.	S2
Matrix.	Sediment
Analyte.	Mo
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	9.82	0.98	1.39	1.04
2	NT	NT		
3	71.3	12.4	72.71	5.05
4	11	2	2.76	1.14
5	8.85	0.9	0.27	0.21
6	NT	NT		
7	9.2	0.4	0.67	0.80
8	NT	NT		
9	9.31	1.55	0.80	0.41
10	9.33	1.87	0.82	0.36
11	16	3	8.56	2.41
12	NT	NT		
13	8.4	NR	-0.26	-0.36
14	8.5	NR	-0.14	-0.20
15	9.31	1.4	0.80	0.45
16	7.421	1	-1.39	-1.02
17	7.2	1.4	-1.65	-0.93
18	8.55	NR	-0.08	-0.11
19	8.1	0.8	-0.60	-0.52
20	NT	NT		
21	9.0	1.3	0.44	0.26
23	7.7	2.2	-1.07	-0.40
24	6	1	-3.04	-2.24
25	8.6	1.72	-0.02	-0.01

Statistics

Assigned Value*	8.62	0.61
Spike	Not Spiked	
Homogeneity Value	9.4	1.4
Robust Average	8.89	0.77
Median	8.85	0.34
Mean	12.29	
N	19	
Max.	71.3	
Min.	6	
Robust SD	1.00	
Robust CV	11%	

*Robust Average excluding Laboratories 3 and 11.

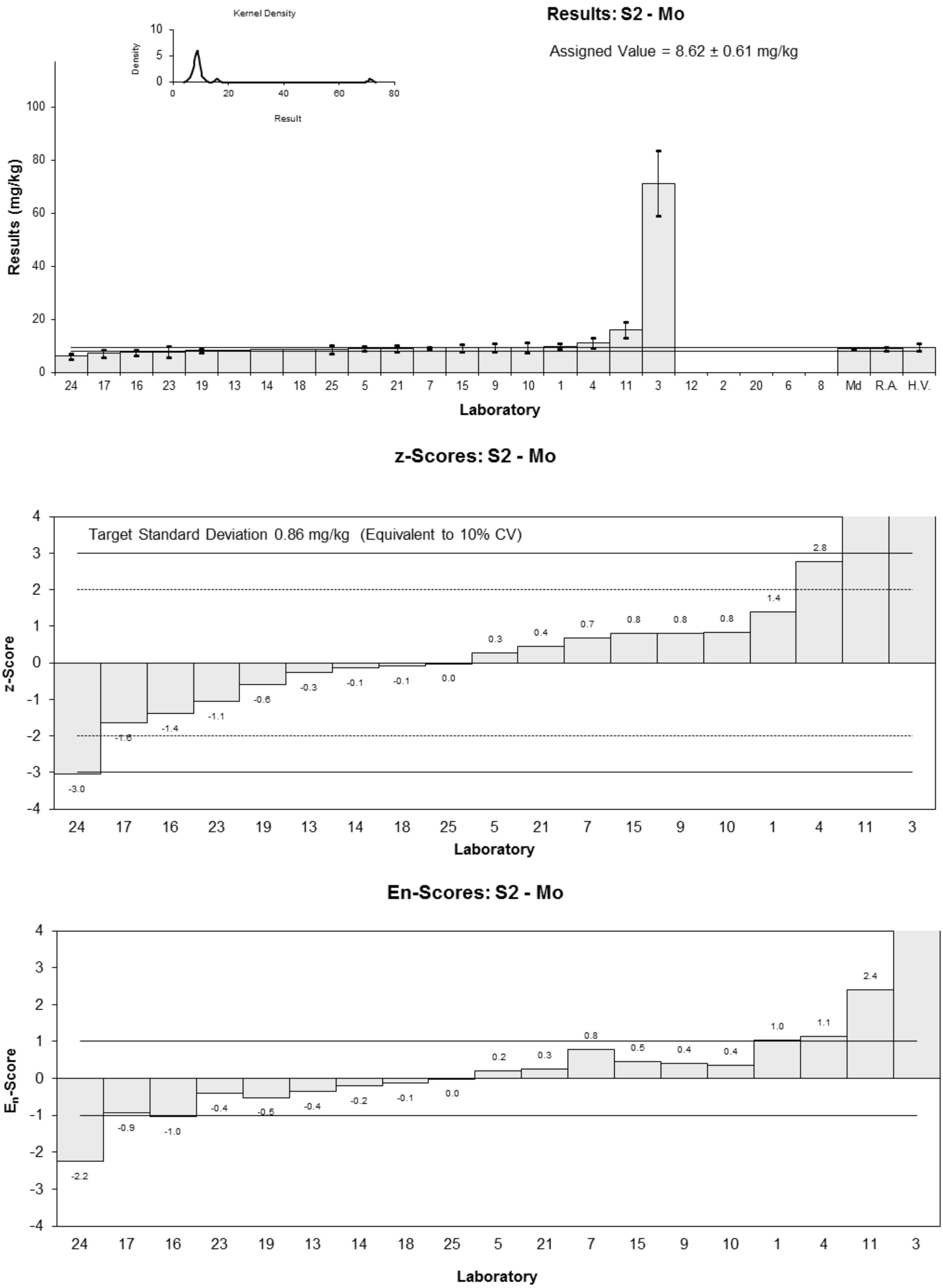

Results: S2 - Mo
Assigned Value = 8.62 ± 0.61 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
24 17 16 23 19 13 14 18 25 5 21 7 15 9 10 1 4 11 3 12 2 20 6 8 Md R.A. H.V.
z-Scores: S2 - Mo
Target Standard Deviation 0.86 mg/kg (Equivalent to 10% CV)
z-Score
-3.0 -1.6 -1.4 -1.1 -0.6 -0.3 -0.1 -0.1 0.0 0.3 0.4 0.7 0.8 0.8 0.8 1.4 2.8
Laboratory
24 17 16 23 19 13 14 18 25 5 21 7 15 9 10 1 4 11 3
En-Scores: S2 - Mo
En-Score
-2.2 -0.9 -1.0 -0.4 -0.5 -0.4 -0.2 -0.1 0.0 0.2 0.3 0.8 0.5 0.4 0.4 1.0 1.1 2.4
Laboratory
24 17 16 23 19 13 14 18 25 5 21 7 15 9 10 1 4 11 3

Figure 29

Table 40

Sample Details

Sample No.	S2
Matrix.	Sediment
Analyte.	Ni
Units	mg/kg

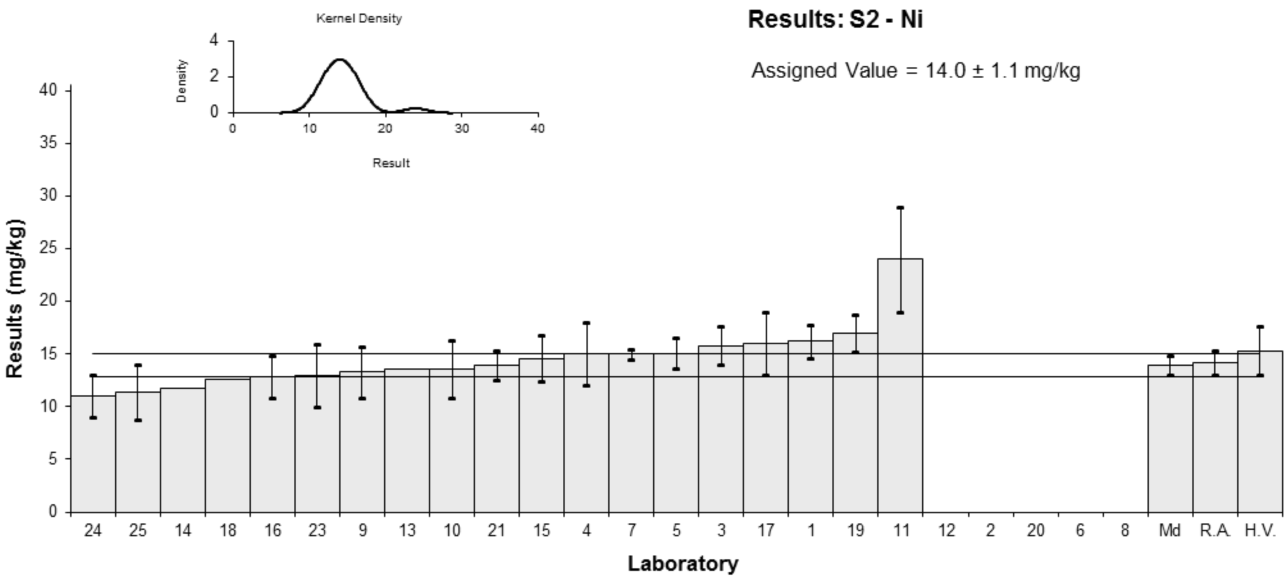
Participant Results

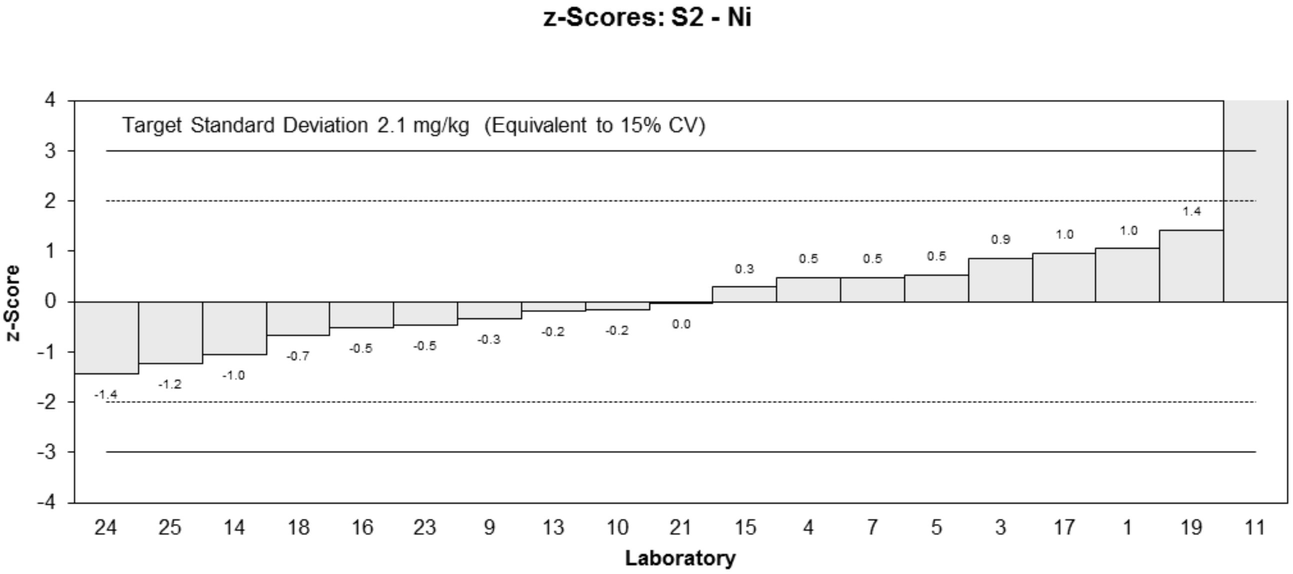
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	16.20	1.62	1.05	1.12
2	NT	NT		
3	15.8	1.82	0.86	0.85
4	15	3	0.48	0.31
5	15.1	1.5	0.52	0.59
6	NT	NT		
7	15	0.5	0.48	0.83
8	NT	NT		
9	13.3	2.44	-0.33	-0.26
10	13.63	2.73	-0.18	-0.13
11	24	5	4.76	1.95
12	NT	NT		
13	13.6	NR	-0.19	-0.36
14	11.8	NR	-1.05	-2.00
15	14.6	2.2	0.29	0.24
16	12.89	2.0	-0.53	-0.49
17	16	3	0.95	0.63
18	12.6	NR	-0.67	-1.27
19	17	1.8	1.43	1.42
20	NT	NT		
21	13.9	1.4	-0.05	-0.06
23	13	3	-0.48	-0.31
24	11	2	-1.43	-1.31
25	11.4	2.59	-1.24	-0.92

Statistics

Assigned Value*	14.0	1.1
Spike	Not Spiked	
Homogeneity Value	15.3	2.3
Robust Average	14.2	1.2
Median	13.9	0.9
Mean	14.5	
N	19	
Max.	24	
Min.	11	
Robust SD	1.9	
Robust CV	13%	

*Robust Average exlcuding Laboratory 11.


Results: S2 - Ni
Assigned Value = 14.0 ± 1.1 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
24 25 14 18 16 23 9 13 10 21 15 4 7 5 3 17 1 19 11 12 2 20 6 8 Md R.A. H.V.


z-Scores: S2 - Ni
Target Standard Deviation 2.1 mg/kg (Equivalent to 15% CV)
z-Score
-1.4 -1.2 -1.0 -0.7 -0.5 -0.5 -0.3 -0.2 -0.2 0.0 0.3 0.5 0.5 0.5 0.9 1.0 1.0 1.4
24 25 14 18 16 23 9 13 10 21 15 4 7 5 3 17 1 19 11
Laboratory

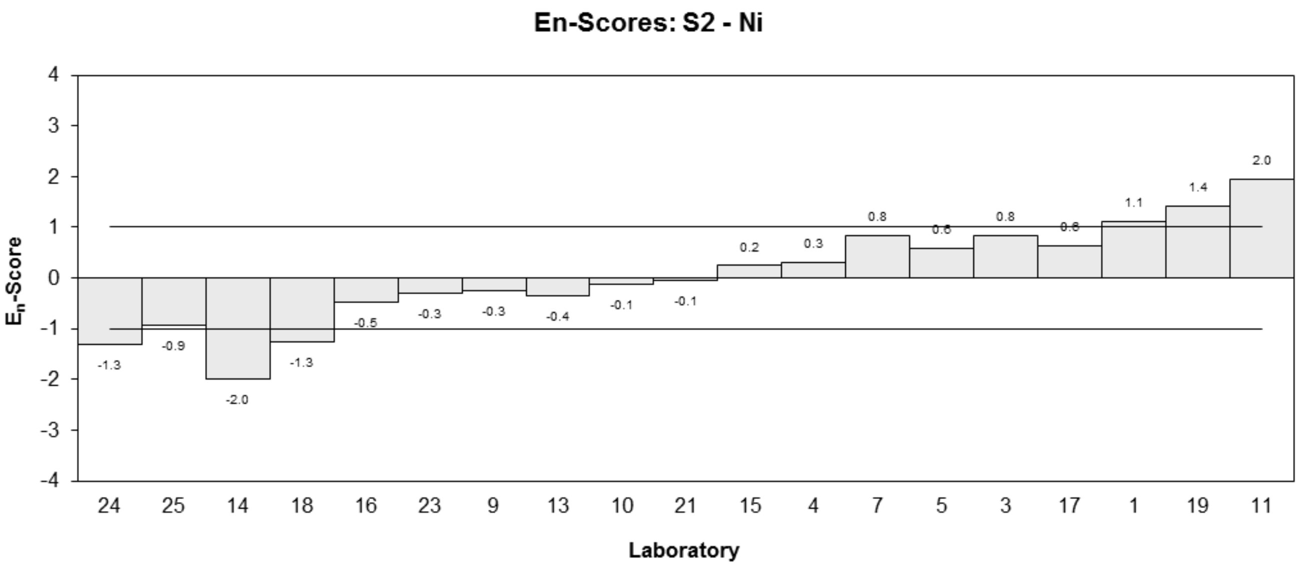

En-Scores: S2 - Ni
E_n-Score
-1.3 -0.9 -2.0 -1.3 -0.5 -0.3 -0.3 -0.4 -0.1 -0.1 0.2 0.3 0.8 0.6 0.8 0.6 1.1 1.4 2.0
24 25 14 18 16 23 9 13 10 21 15 4 7 5 3 17 1 19 11
Laboratory

Figure 30

Table 41

Sample Details

Sample No.	S2
Matrix.	Sediment
Analyte.	Pb
Units	mg/kg

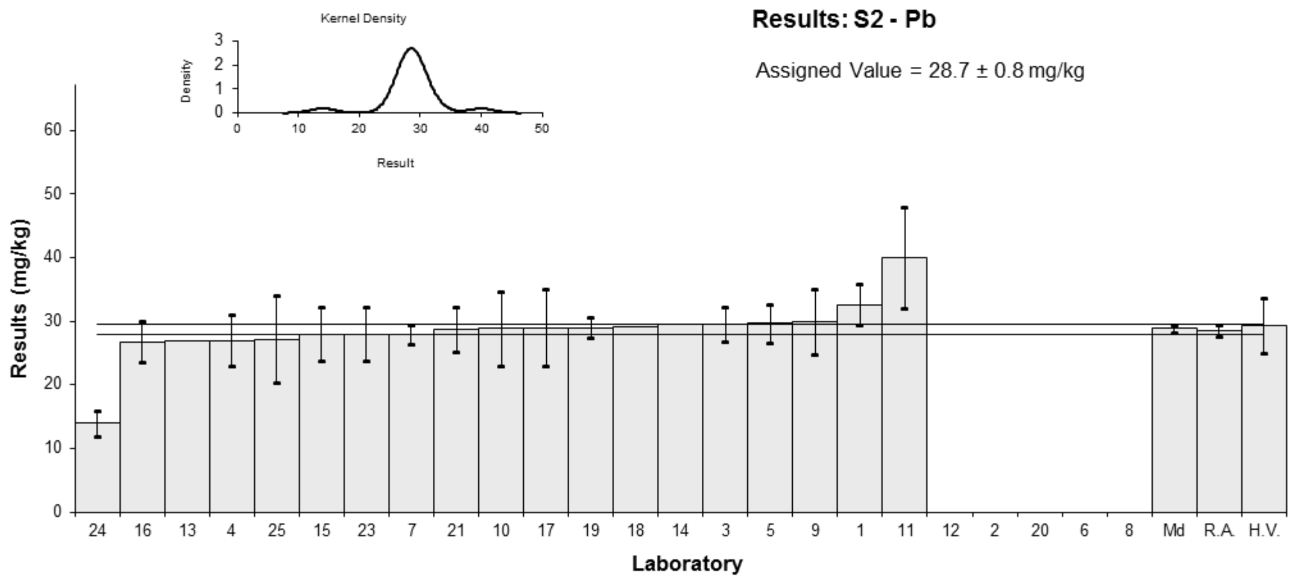
Participant Results

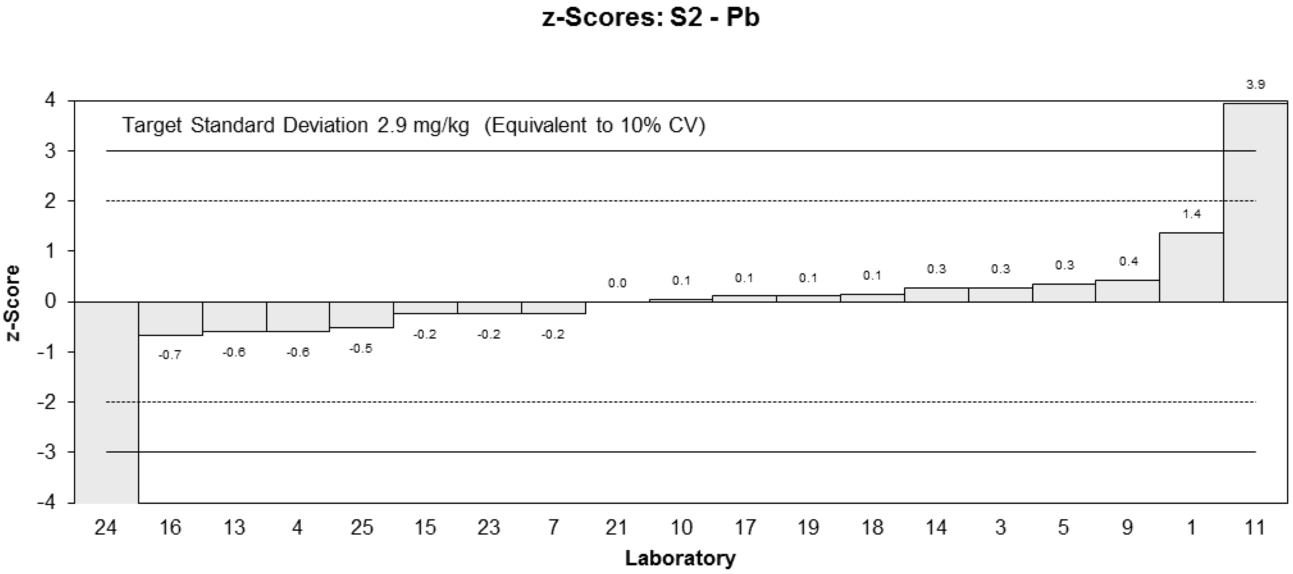
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	32.60	3.26	1.36	1.16
2	NT	NT		
3	29.5	2.66	0.28	0.29
4	27	4	-0.59	-0.42
5	29.7	3.0	0.35	0.32
6	NT	NT		
7	28	1.5	-0.24	-0.41
8	NT	NT		
9	29.9	5.20	0.42	0.23
10	28.85	5.77	0.05	0.03
11	40	8	3.94	1.41
12	NT	NT		
13	27	NR	-0.59	-2.12
14	29.5	NR	0.28	1.00
15	28.0	4.2	-0.24	-0.16
16	26.80	3.2	-0.66	-0.58
17	29	6	0.10	0.05
18	29.1	NR	0.14	0.50
19	29	1.6	0.10	0.17
20	NT	NT		
21	28.7	3.5	0.00	0.00
23	28	4.3	-0.24	-0.16
24	14	2	-5.12	-6.82
25	27.2	6.80	-0.52	-0.22

Statistics

Assigned Value*	28.7	0.8
Spike	Not Spiked	
Homogeneity Value	29.3	4.4
Robust Average	28.6	0.9
Median	28.9	0.6
Mean	28.5	
N	19	
Max.	40	
Min.	14	
Robust SD	1.0	
Robust CV	3.5%	

*Robust Average excluding Laboratory 24.


Results: S2 - Pb
Assigned Value = 28.7 ± 0.8 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
24 16 13 4 25 15 23 7 21 10 17 19 18 14 3 5 9 1 11 12 2 20 6 8 Md R.A. H.V.


z-Scores: S2 - Pb
Target Standard Deviation 2.9 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
24 16 13 4 25 15 23 7 21 10 17 19 18 14 3 5 9 1 11
-0.7 -0.6 -0.6 -0.5 -0.2 -0.2 -0.2 0.0 0.1 0.1 0.1 0.1 0.3 0.3 0.3 0.4 1.4 3.9

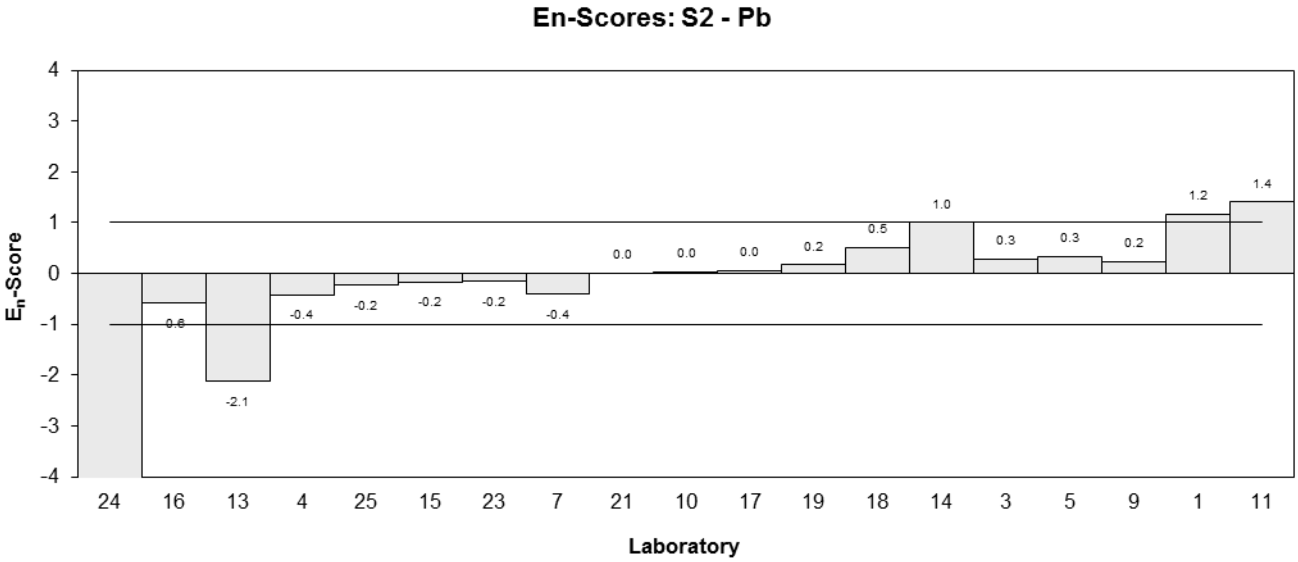

En-Scores: S2 - Pb
E_n-Score
Laboratory
24 16 13 4 25 15 23 7 21 10 17 19 18 14 3 5 9 1 11
-0.6 -2.1 -0.4 -0.2 -0.2 -0.2 -0.4 0.0 0.0 0.0 0.2 0.5 1.0 0.3 0.3 0.2 1.2 1.4

Figure 31

Table 42

Sample Details

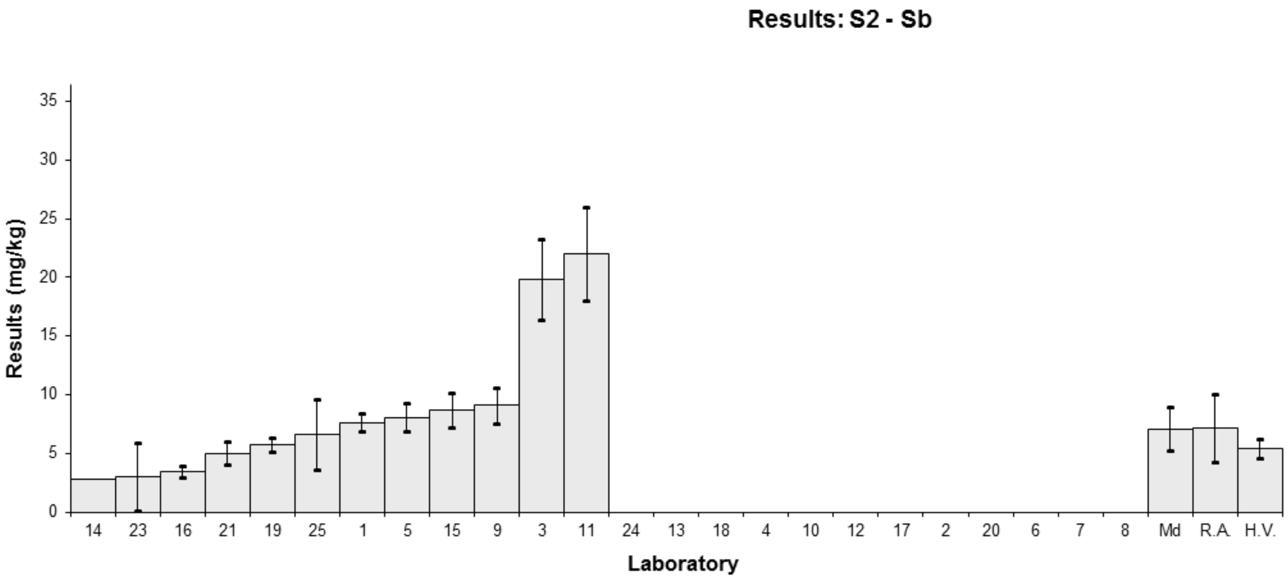
Sample No.	S2
Matrix.	Sediment
Analyte.	Sb
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	7.60	0.76
2	NT	NT
3	19.8	3.44
4	NR	NR
5	8.04	1.2
6	NT	NT
7	NT	NT
8	NT	NT
9	9.07	1.55
10	NT	NT
11	22	4
12	NT	NT
13	<10	NR
14	2.8	NR
15	8.71	1.5
16	3.431	0.5
17	NT	NT
18	NR	NR
19	5.7	0.6
20	NT	NT
21	5.0	1.0
23	3	2.9
24	<1	1
25	6.64	3.00

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	5.4	0.8
Robust Average	7.2	2.9
Median	7.1	1.9
Mean	8.5	
N	12	
Max.	22	
Min.	2.8	
Robust SD	1.7	
Robust CV	24%	



Results: S2 - Sb
Results (mg/kg)
0
5
10
15
20
25
30
35
14 23 16 21 19 25 1 5 15 9 3 11 24 13 18 4 10 12 17 2 20 6 7 8 Md R.A. H.V.
Laboratory

Figure 32

Table 43

Sample Details

Sample No.	S2
Matrix.	Sediment
Analyte.	Se
Units	mg/kg

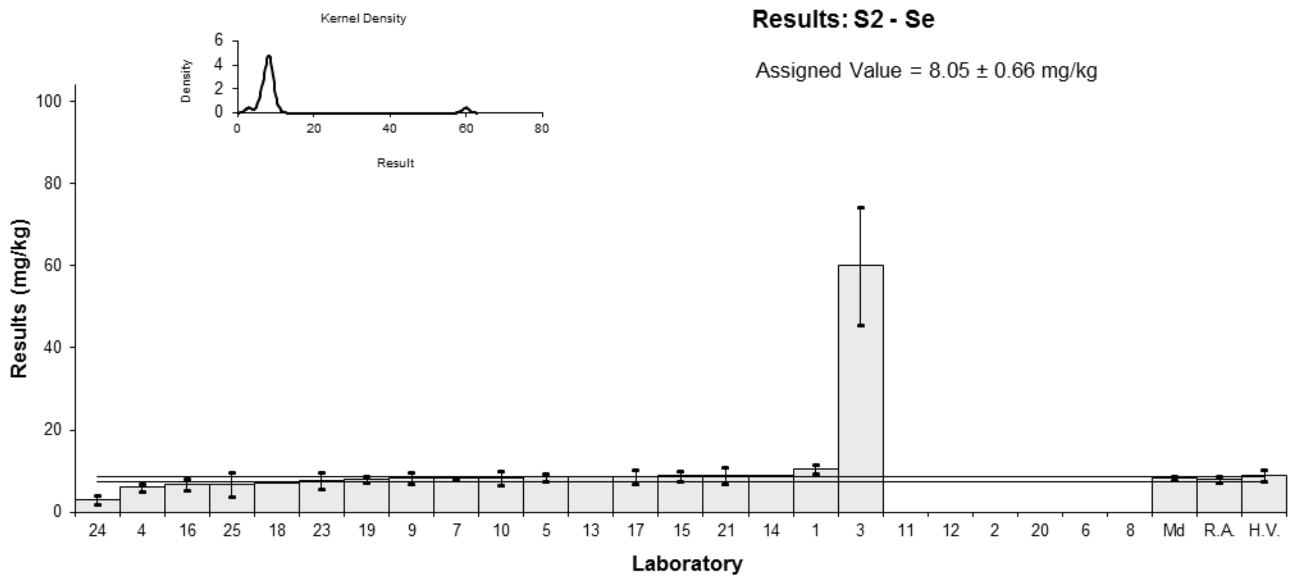
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	10.40	1.04	1.95	1.91
2	NT	NT		
3	59.99	14.39	43.01	3.61
4	6	1	-1.70	-1.71
5	8.48	1.0	0.36	0.36
6	NT	NT		
7	8.2	0.4	0.12	0.19
8	NT	NT		
9	8.15	1.42	0.08	0.06
10	8.40	1.68	0.29	0.19
11	<2	NR		
12	NT	NT		
13	8.5	NR	0.37	0.68
14	9	NR	0.79	1.44
15	8.77	1.3	0.60	0.49
16	6.624	1.4	-1.18	-0.92
17	8.5	1.7	0.37	0.25
18	6.95	NR	-0.91	-1.67
19	8.0	0.7	-0.04	-0.05
20	NT	NT		
21	8.8	2.0	0.62	0.36
23	7.6	2.1	-0.37	-0.20
24	3	1	-4.18	-4.21
25	6.70	3.00	-1.12	-0.44

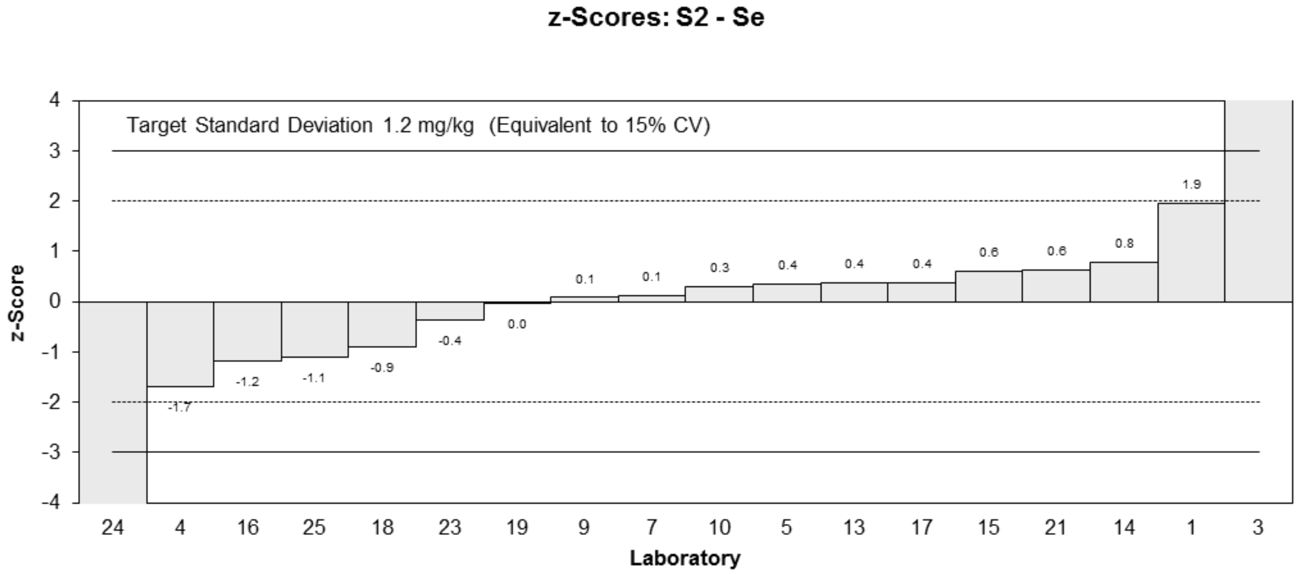
Statistics

Assigned Value*	8.05	0.66
Spike	Not Spiked	
Homogeneity Value	9.0	1.4
Robust Average	8.05	0.81
Median	8.30	0.44
Mean	10.67	
N	18	
Max.	59.99	
Min.	3	
Robust SD	1.1	
Robust CV	14%	

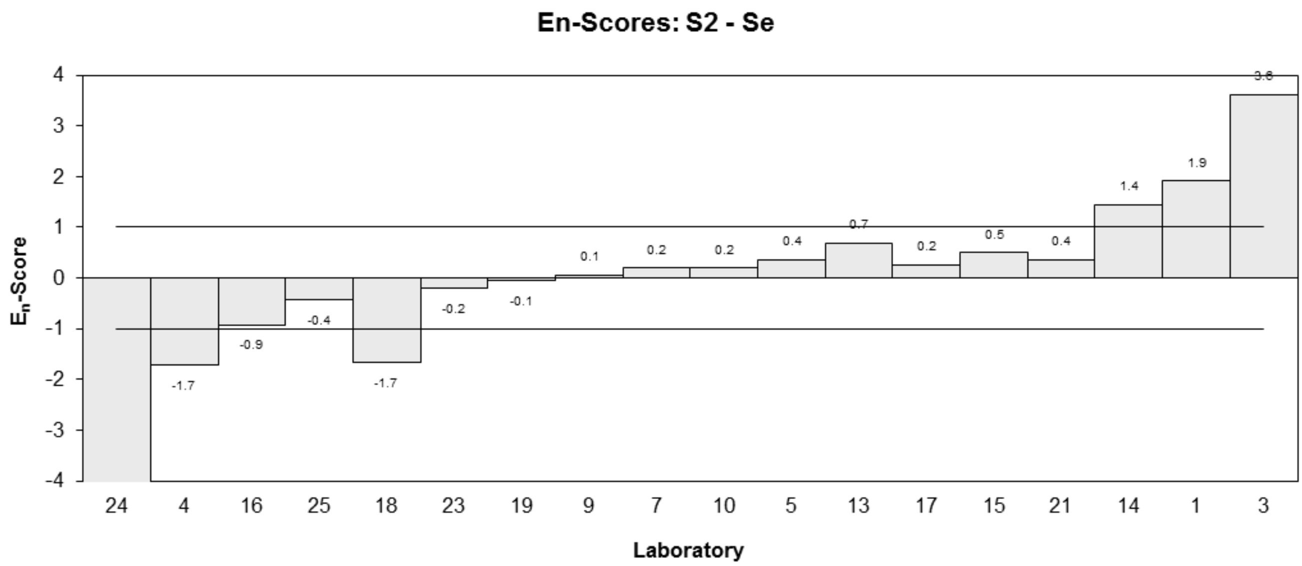
*Robust Average excluding Laboratories 3 and 24.



Results: S2 - Se
Assigned Value = 8.05 ± 0.66 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
24 4 16 25 18 23 19 9 7 10 5 13 17 15 21 14 1 3 11 12 2 20 6 8 Md R.A. H.V.



z-Scores: S2 - Se
Target Standard Deviation 1.2 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
-1.7 -1.2 -1.1 -0.9 -0.4 0.0 0.1 0.1 0.3 0.4 0.4 0.4 0.6 0.6 0.8 1.9
24 4 16 25 18 23 19 9 7 10 5 13 17 15 21 14 1 3



En-Scores: S2 - Se
E_n-Score
Laboratory
-1.7 -0.9 -0.4 -1.7 -0.2 -0.1 0.1 0.2 0.2 0.4 0.7 0.2 0.5 0.4 1.4 1.9 3.6
24 4 16 25 18 23 19 9 7 10 5 13 17 15 21 14 1 3

Figure 33

Table 44

Sample Details

Sample No.	S2
Matrix.	Sediment
Analyte.	Th
Units	mg/kg

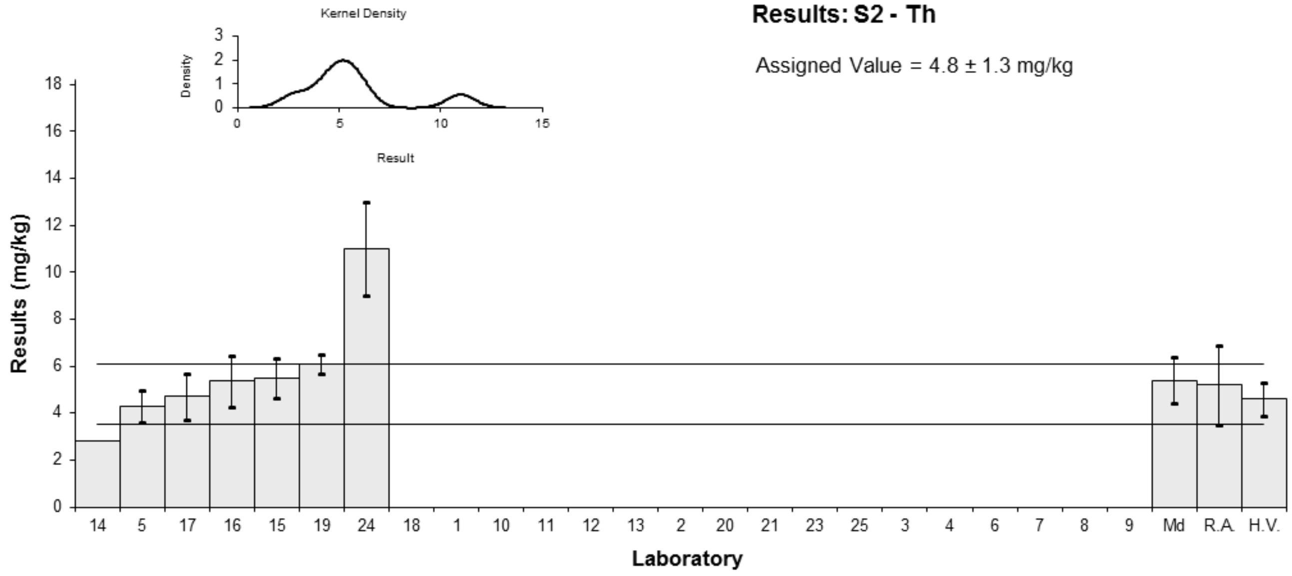
Participant Results

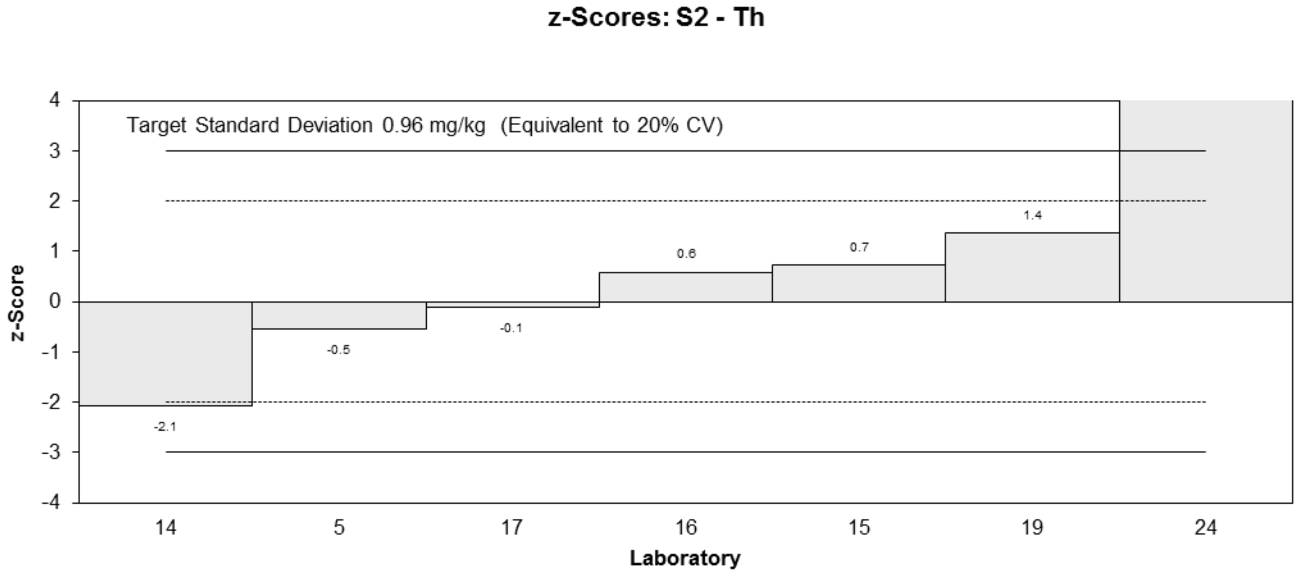
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	NT	NT		
3	NT	NT		
4	NT	NT		
5	4.28	0.7	-0.54	-0.35
6	NT	NT		
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	NT	NT		
12	NT	NT		
13	NT	NT		
14	2.8	NR	-2.08	-1.54
15	5.49	0.83	0.72	0.45
16	5.359	1.1	0.58	0.33
17	4.7	1.0	-0.10	-0.06
18	NR	NR		
19	6.1	0.4	1.35	0.96
20	NT	NT		
21	NT	NT		
23	NT	NT		
24	11	2	6.46	2.60
25	NT	NT		

Statistics

Assigned Value*	4.8	1.3
Spike	Not Spiked	
Homogeneity Value	4.6	0.7
Robust Average	5.2	1.7
Median	5.4	1.0
Mean	5.7	
N	7	
Max.	11	
Min.	2.8	
Robust SD	1.3	
Robust CV	25%	

*Robust Average excluding Laboratory 24.


Results: S2 - Th
Assigned Value = 4.8 ± 1.3 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
14 5 17 16 15 19 24 18 1 10 11 12 13 2 20 21 23 25 3 4 6 7 8 9 Md R.A. H.V.
Laboratory


z-Scores: S2 - Th
Target Standard Deviation 0.96 mg/kg (Equivalent to 20% CV)
z-Score
-2.1
-0.5
-0.1
0.6
0.7
1.4
14 5 17 16 15 19 24
Laboratory

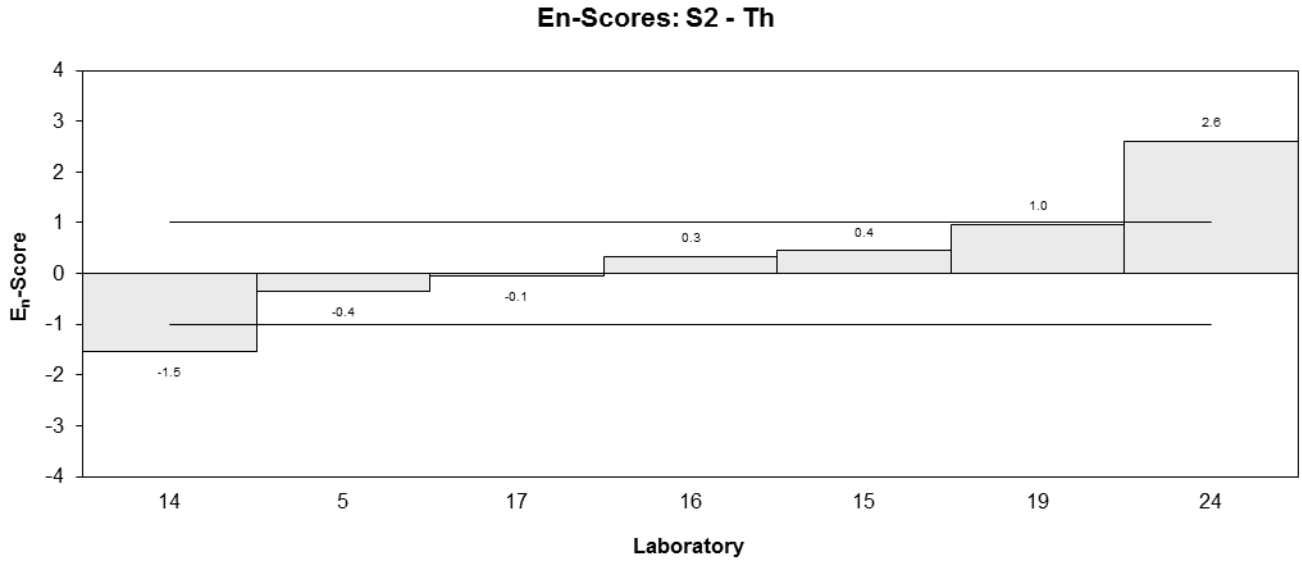

En-Scores: S2 - Th
E_n-Score
-1.5
-0.4
-0.1
0.3
0.4
1.0
2.6
14 5 17 16 15 19 24
Laboratory

Figure 34

Table 45

Sample Details

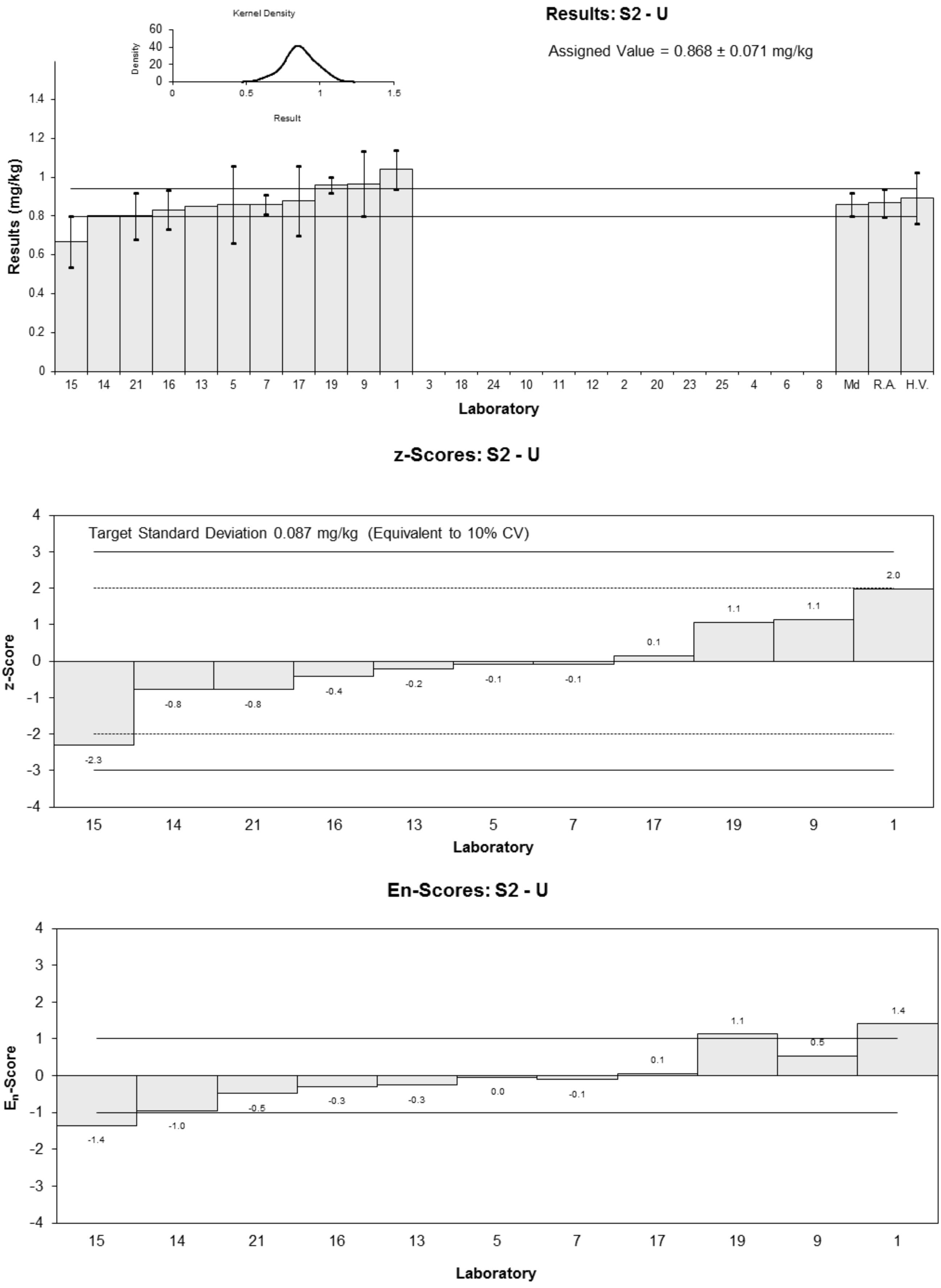
Sample No.	S2
Matrix.	Sediment
Analyte.	U
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1.04	0.10	1.98	1.40
2	NT	NT		
3	<5	NR		
4	NT	NT		
5	0.86	0.2	-0.09	-0.04
6	NT	NT		
7	0.86	0.05	-0.09	-0.09
8	NT	NT		
9	0.967	0.169	1.14	0.54
10	NT	NT		
11	NT	NT		
12	NT	NT		
13	0.85	NR	-0.21	-0.25
14	0.8	NR	-0.78	-0.96
15	0.668	0.13	-2.30	-1.35
16	0.8324	0.1	-0.41	-0.29
17	0.88	0.18	0.14	0.06
18	NR	NR		
19	0.96	0.04	1.06	1.13
20	NT	NT		
21	0.80	0.12	-0.78	-0.49
23	NT	NT		
24	NR	NR		
25	NT	NT		

Statistics

Assigned Value	0.868	0.071
Spike	Not Spiked	
Homogeneity Value	0.89	0.13
Robust Average	0.868	0.071
Median	0.860	0.060
Mean	0.865	
N	11	
Max.	1.04	
Min.	0.668	
Robust SD	0.095	
Robust CV	11%	



Results: S2 - U
Assigned Value = 0.868 ± 0.071 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
15 14 21 16 13 5 7 17 19 9 1 3 18 24 10 11 12 2 20 23 25 4 6 8 Md R.A. H.V.
z-Scores: S2 - U
Target Standard Deviation 0.087 mg/kg (Equivalent to 10% CV)
z-Score
-2.3 -0.8 -0.8 -0.4 -0.2 -0.1 -0.1 0.1 1.1 1.1 2.0
Laboratory
15 14 21 16 13 5 7 17 19 9 1
En-Scores: S2 - U
E_n-Score
-1.4 -1.0 -0.5 -0.3 -0.3 0.0 -0.1 0.1 1.1 0.5 1.4
Laboratory
15 14 21 16 13 5 7 17 19 9 1

Figure 35

Table 46

Sample Details

Sample No.	S2
Matrix.	Sediment
Analyte.	V
Units	mg/kg

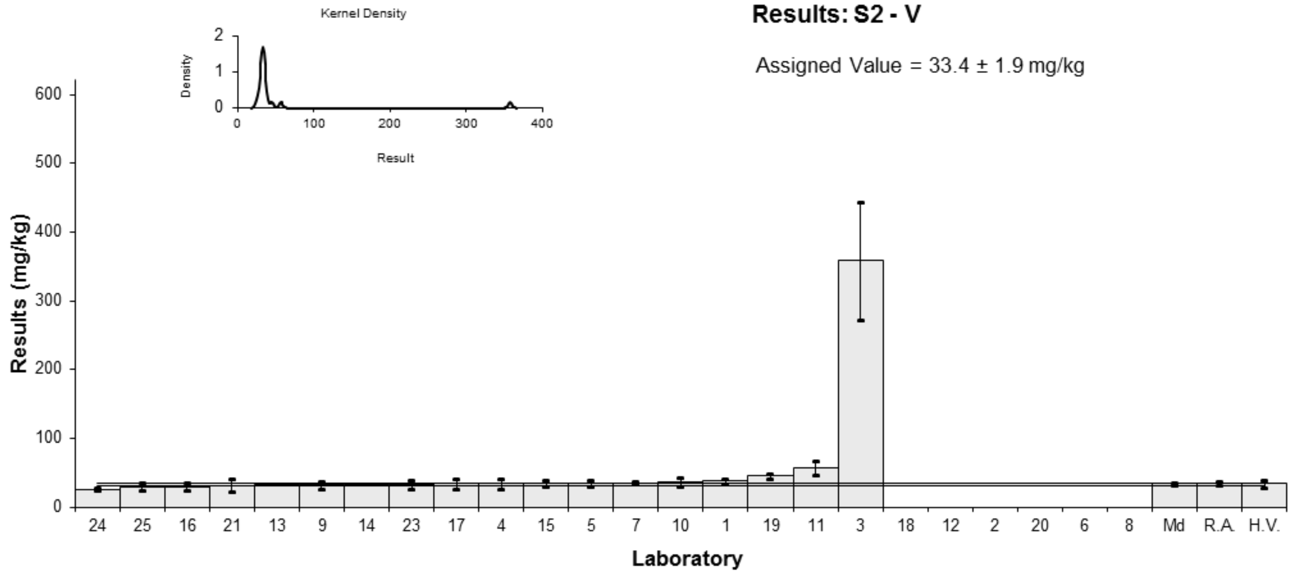
Participant Results

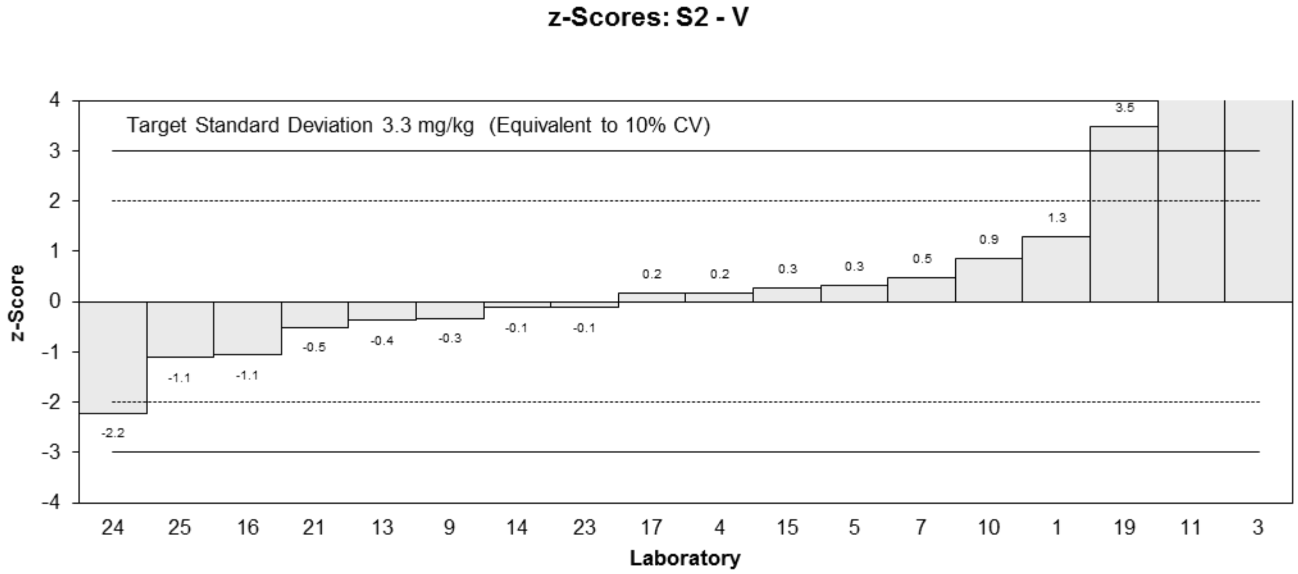
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	37.70	3.77	1.29	1.02
2	NT	NT		
3	358	85.9	97.19	3.78
4	34	8	0.18	0.07
5	34.5	4.0	0.33	0.25
6	NT	NT		
7	35	1.5	0.48	0.66
8	NT	NT		
9	32.3	5.66	-0.33	-0.18
10	36.26	7.25	0.86	0.38
11	57	10	7.07	2.32
12	NT	NT		
13	32.2	NR	-0.36	-0.63
14	33	NR	-0.12	-0.21
15	34.3	5.15	0.27	0.16
16	29.85	5.3	-1.06	-0.63
17	34	7	0.18	0.08
18	NR	NR		
19	45	4	3.47	2.62
20	NT	NT		
21	31.7	8.6	-0.51	-0.19
23	33	6.2	-0.12	-0.06
24	26	2	-2.22	-2.68
25	29.7	5.94	-1.11	-0.59

Statistics

Assigned Value*	33.4	1.9
Spike	Not Spiked	
Homogeneity Value	34.0	5.1
Robust Average	34.3	2.5
Median	34.0	1.5
Mean	53.0	
N	18	
Max.	358	
Min.	26	
Robust SD	3.0	
Robust CV	8.7%	

*Robust Average excluding Laboratories 3 and 11.


Results: S2 - V
Assigned Value = 33.4 ± 1.9 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
24 25 16 21 13 9 14 23 17 4 15 5 7 10 1 19 11 3 18 12 2 20 6 8 Md R.A. H.V.


z-Scores: S2 - V
Target Standard Deviation 3.3 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
24 25 16 21 13 9 14 23 17 4 15 5 7 10 1 19 11 3

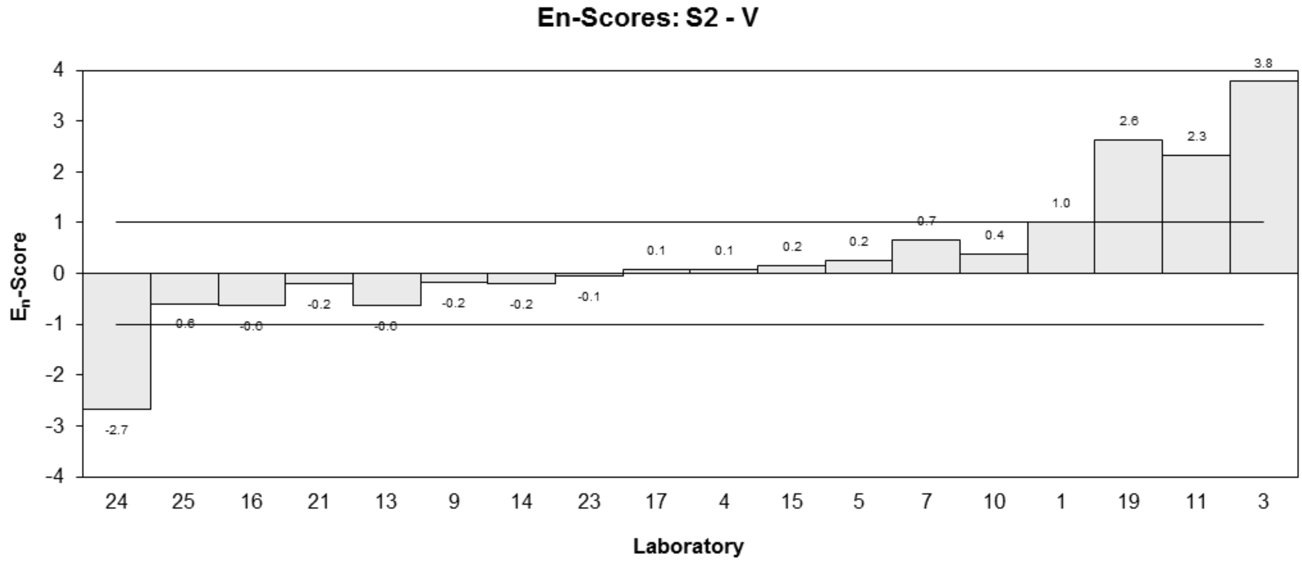

En-Scores: S2 - V
E_n-Score
Laboratory
24 25 16 21 13 9 14 23 17 4 15 5 7 10 1 19 11 3

Figure 36

Table 47

Sample Details

Sample No.	S2
Matrix.	Sediment
Analyte.	Zn
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	81.00	8.10	1.05	0.90
2	NT	NT		
3	75.4	9.05	0.29	0.22
4	74	8	0.10	0.08
5	76.5	8.0	0.44	0.38
6	NT	NT		
7	73	3.0	-0.04	-0.07
8	NT	NT		
9	71.0	12.5	-0.31	-0.18
10	71.03	14.21	-0.31	-0.16
11	115	20	5.69	2.06
12	NT	NT		
13	74.7	NR	0.19	0.50
14	72.2	NR	-0.15	-0.39
15	80.2	12.0	0.94	0.56
16	63.38	14	-1.35	-0.69
17	76	15	0.37	0.18
18	70.6	NR	-0.37	-0.96
19	74	5	0.10	0.12
20	NT	NT		
21	77	13	0.50	0.28
23	73	13.3	-0.04	-0.02
24	62	2	-1.54	-3.28
25	67.2	9.14	-0.83	-0.64

Statistics

Assigned Value*	73.3	2.8
Spike	Not Spiked	
Homogeneity Value	66	10
Robust Average	73.7	3.0
Median	74.0	2.1
Mean	75.1	
N	19	
Max.	115	
Min.	62	
Robust SD	4.7	
Robust CV	6.4%	

*Robust Average excluding Laboratory 11.

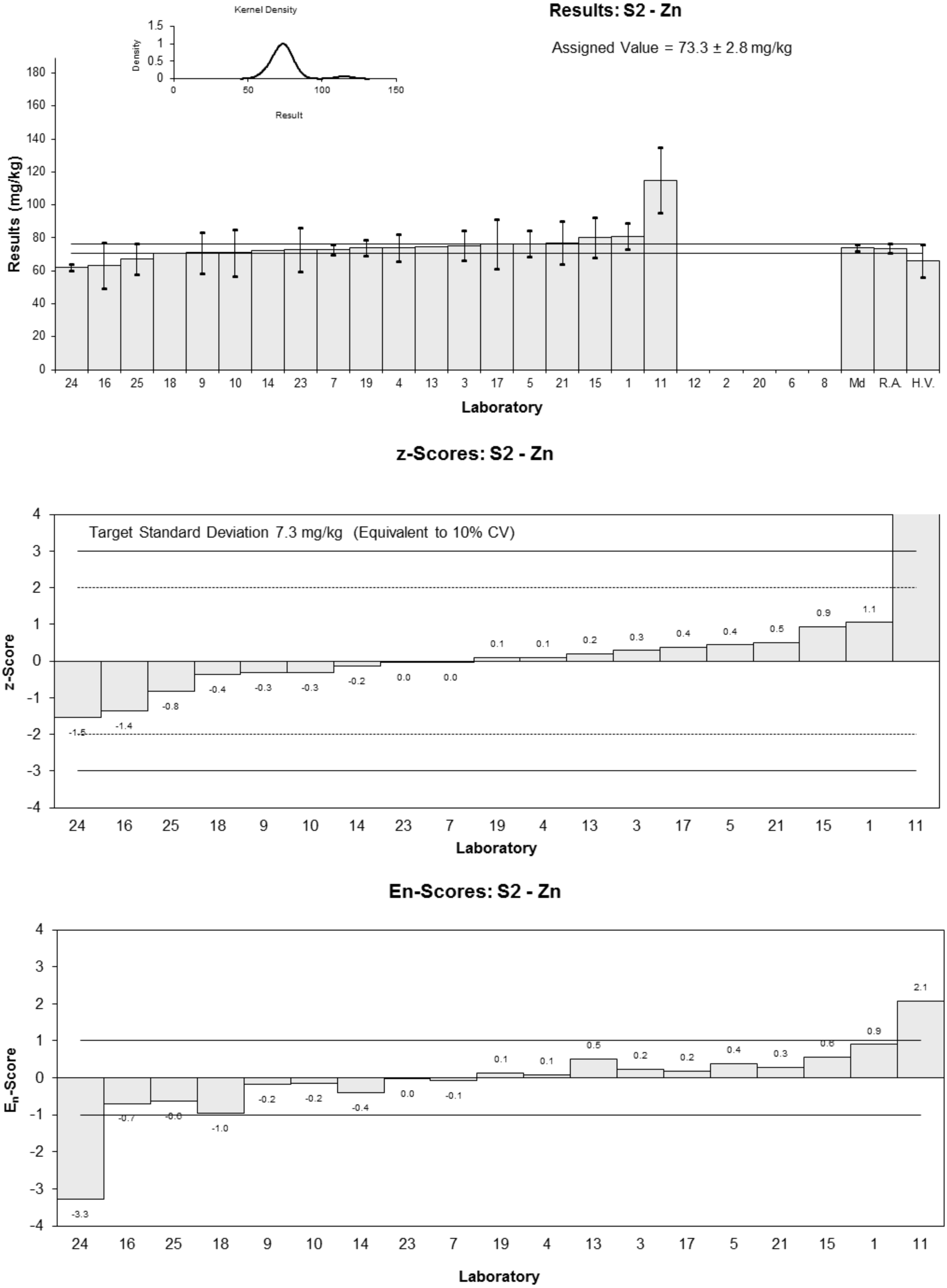

Results: S2 - Zn
Assigned Value = 73.3 ± 2.8 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
24 16 25 18 9 10 14 23 7 19 4 13 3 17 5 21 15 1 11 12 2 20 6 8 Md R.A. H.V.
z-Scores: S2 - Zn
Target Standard Deviation 7.3 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
24 16 25 18 9 10 14 23 7 19 4 13 3 17 5 21 15 1 11
En-Scores: S2 - Zn
E_n-Score
Laboratory
24 16 25 18 9 10 14 23 7 19 4 13 3 17 5 21 15 1 11

Figure 37

Table 48

Sample Details

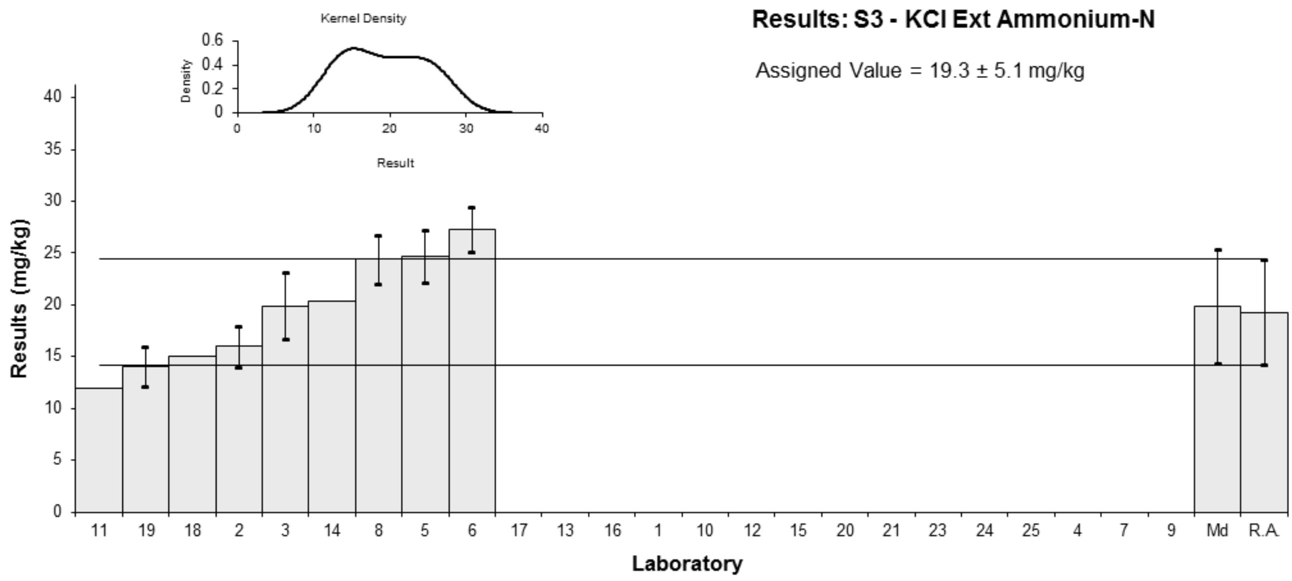
Sample No.	S3
Matrix.	Soil
Analyte.	KCl Ext Ammonium-N
Units	mg/kg

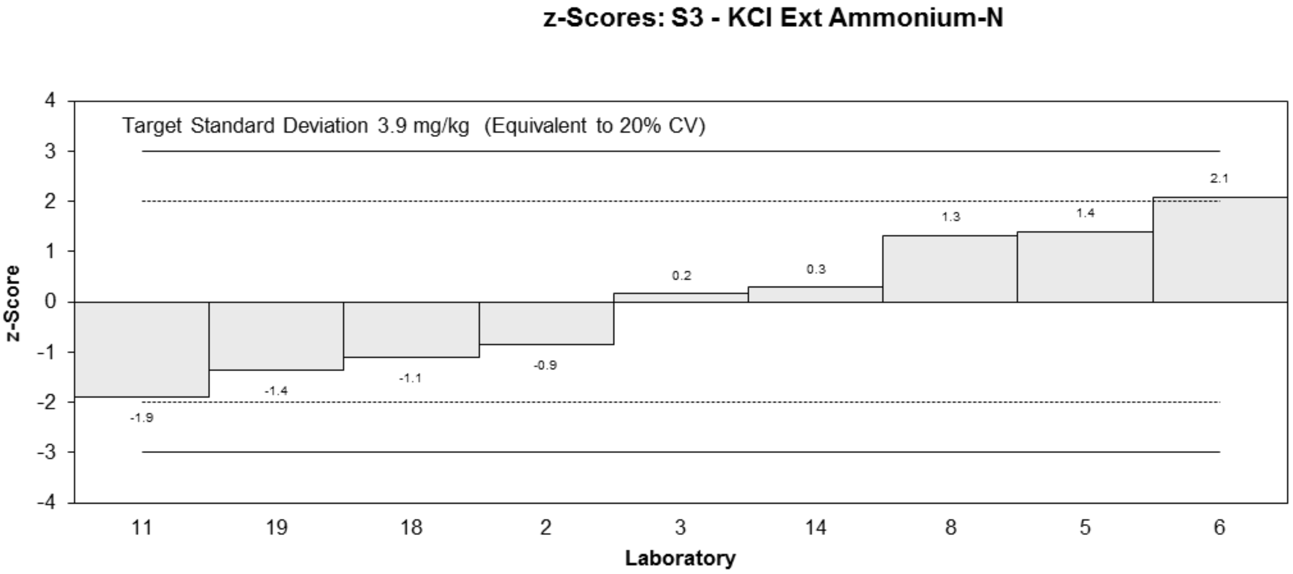
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	16	2	-0.85	-0.60
3	19.9	3.22	0.16	0.10
4	NT	NT		
5	24.7	2.5	1.40	0.95
6	27.3	2.16	2.07	1.44
7	NT	NT		
8	24.4	2.35	1.32	0.91
9	NT	NT		
10	NT	NT		
11	12	NR	-1.89	-1.43
12	NT	NT		
13	NR	NR		
14	20.4	NR	0.28	0.22
15	NT	NT		
16	NR	NR		
17	<30	6		
18	15.0	NR	-1.11	-0.84
19	14	1.9	-1.37	-0.97
20	NT	NT		
21	NT	NT		
23	NT	NT		
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value	19.3	5.1
Spike	Not Spiked	
Robust Average	19.3	5.1
Median	19.9	5.5
Mean	19.3	
N	9	
Max.	27.3	
Min.	12	
Robust SD	6.1	
Robust CV	32%	


Results: S3 - KCl Ext Ammonium-N
Assigned Value = 19.3 ± 5.1 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
11 19 18 2 3 14 8 5 6 17 13 16 1 10 12 15 20 21 23 24 25 4 7 9 Md R.A.


z-Scores: S3 - KCl Ext Ammonium-N
Target Standard Deviation 3.9 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
11 19 18 2 3 14 8 5 6
-1.9 -1.4 -1.1 -0.9 0.2 0.3 1.3 1.4 2.1

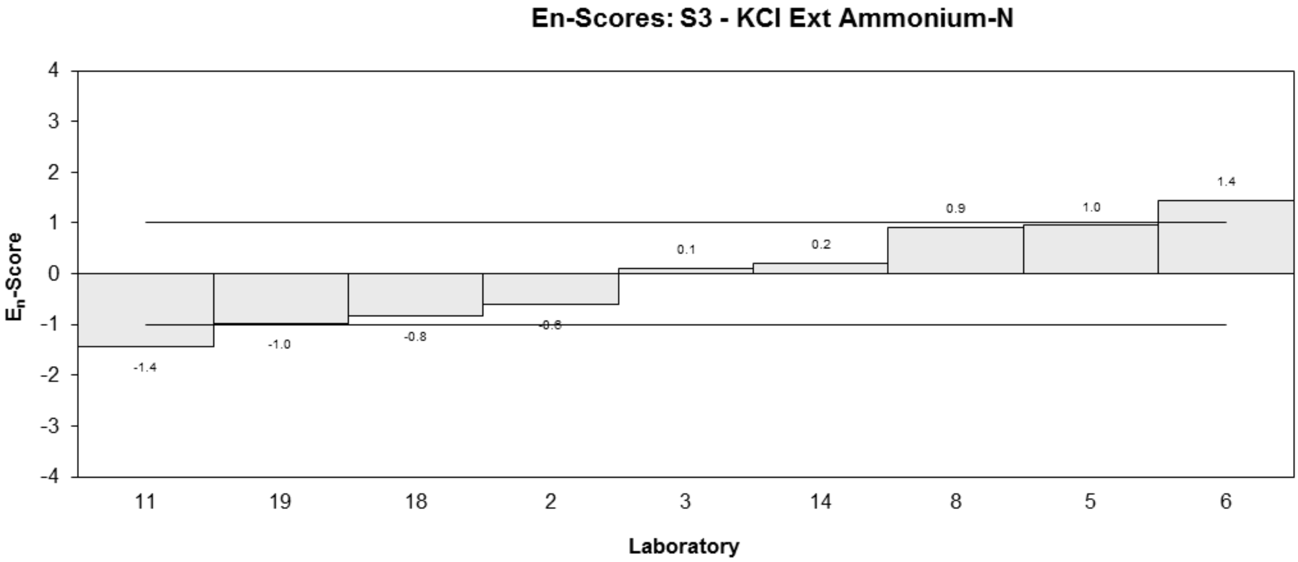

En-Scores: S3 - KCl Ext Ammonium-N
En-Score
Laboratory
11 19 18 2 3 14 8 5 6
-1.4 -1.0 -0.8 -0.6 0.1 0.2 0.9 1.0 1.4

Figure 38

Table 49

Sample Details

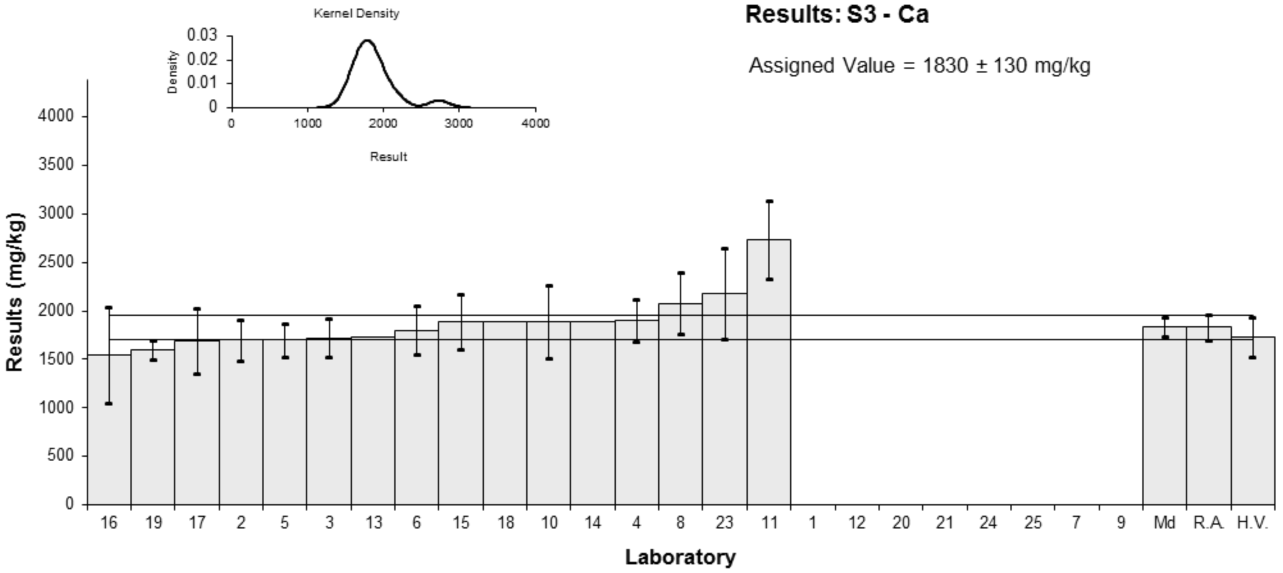
Sample No.	S3
Matrix.	Soil
Analyte.	Ca
Units	mg/kg

Participant Results

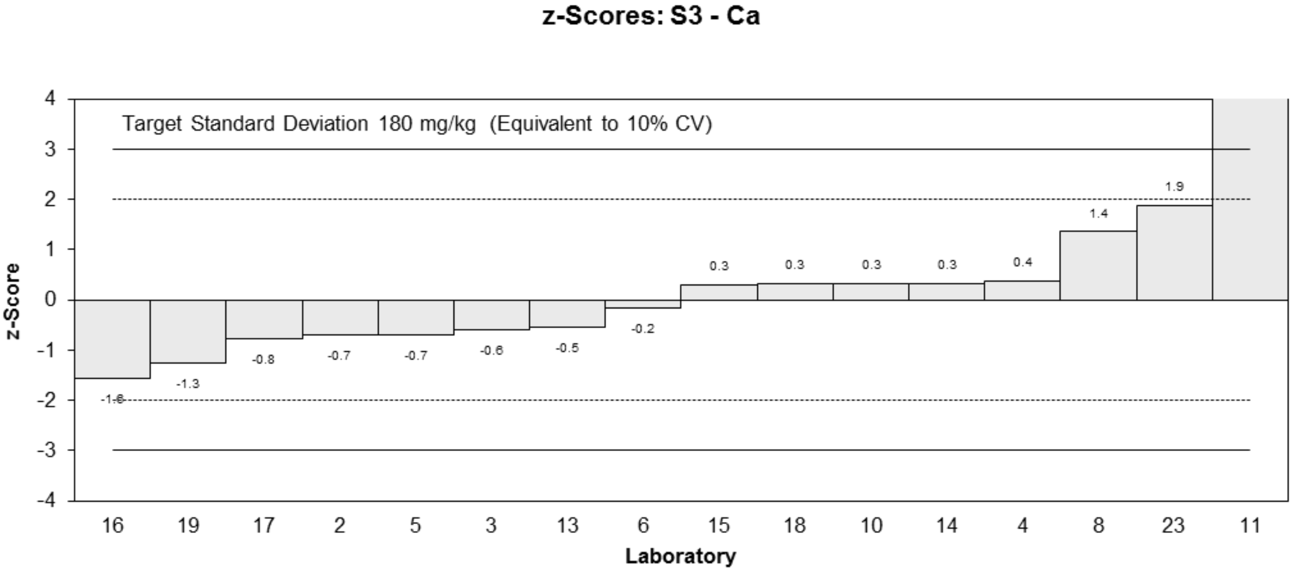
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	1700	210	-0.71	-0.53
3	1720	197.8	-0.60	-0.46
4	1900	220	0.38	0.27
5	1700	170	-0.71	-0.61
6	1800	253	-0.16	-0.11
7	NT	NT		
8	2078	317	1.36	0.72
9	NT	NT		
10	1889.63	377.93	0.33	0.15
11	2730	400	4.92	2.14
12	NT	NT		
13	1731	NR	-0.54	-0.76
14	1890	NR	0.33	0.46
15	1883	282	0.29	0.17
16	1544	490	-1.56	-0.56
17	1690	340	-0.77	-0.38
18	1888	NR	0.32	0.45
19	1600	96	-1.26	-1.42
20	NT	NT		
21	NT	NT		
23	2175	470	1.89	0.71
24	NT	NT		
25	NT	NT		

Statistics

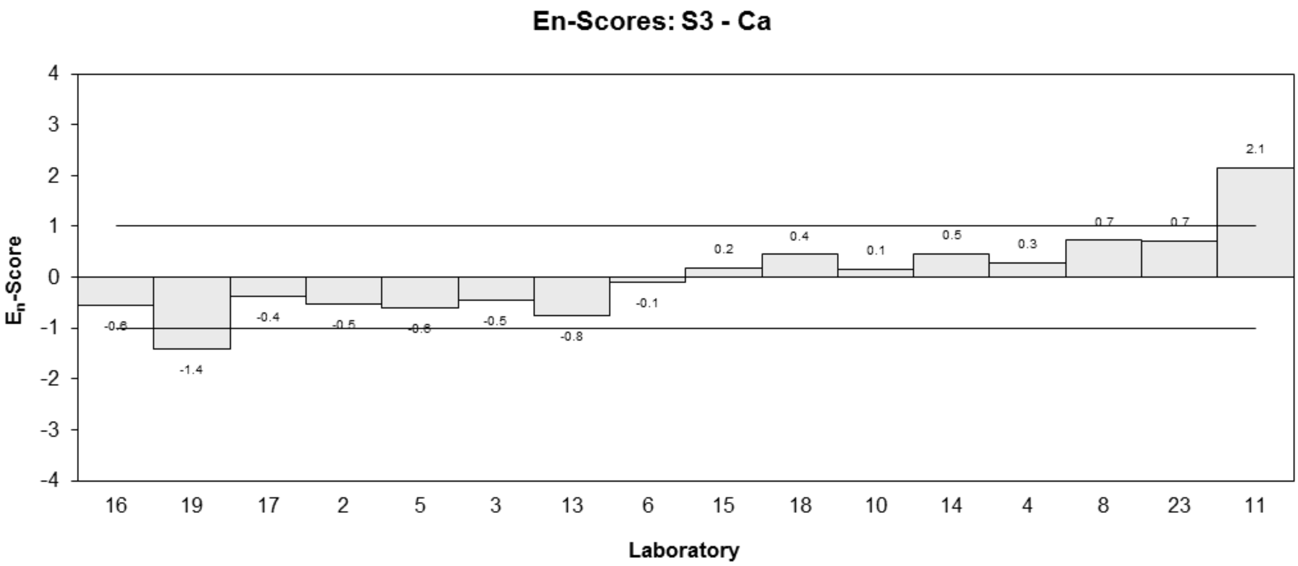
Assigned Value	1830	130
Spike	Not Spiked	
Homogeneity Value	1730	210
Robust Average	1830	130
Median	1840	100
Mean	1870	
N	16	
Max.	2730	
Min.	1544	
Robust SD	200	
Robust CV	11%	



Results: S3 - Ca
Assigned Value = 1830 ± 130 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
16 19 17 2 5 3 13 6 15 18 10 14 4 8 23 11 1 12 20 21 24 25 7 9 Md R.A. H.V.



z-Scores: S3 - Ca
Target Standard Deviation 180 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-1.6 -1.3 -0.8 -0.7 -0.7 -0.6 -0.5 -0.2 0.3 0.3 0.3 0.3 0.4 1.4 1.9
16 19 17 2 5 3 13 6 15 18 10 14 4 8 23 11



En-Scores: S3 - Ca
En-Score
Laboratory
-0.6 -1.4 -0.4 -0.5 -0.6 -0.5 -0.8 -0.1 0.2 0.4 0.1 0.5 0.3 0.7 0.7 2.1
16 19 17 2 5 3 13 6 15 18 10 14 4 8 23 11

Figure 39

Table 50

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	Chloride
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	40	10	0.57	0.34
3	49.0	4.16	1.82	1.63
4	30	3	-0.82	-0.78
5	55	5	2.66	2.24
6	33.4	6.9	-0.35	-0.26
7	NT	NT		
8	25.3	3.30	-1.48	-1.39
9	NT	NT		
10	NT	NT		
11	34	NR	-0.26	-0.28
12	NT	NT		
13	75.17	NR	5.47	5.69
14	30.2	NR	-0.79	-0.83
15	106	15.9	9.76	4.04
16	41	6.8	0.71	0.53
17	25	4	-1.52	-1.37
18	41.3	NR	0.75	0.78
19	31	1.3	-0.68	-0.70
20	NT	NT		
21	NT	NT		
23	NT	NT		
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value*	35.9	6.9
Spike	Not Spiked	
Homogeneity Value	28.1	3.4
Robust Average	39.7	9.3
Median	37.0	5.9
Mean	44.0	
N	14	
Max.	106	
Min.	25	
Robust SD	9.5	
Robust CV	24%	

*Robust Average excluding Laboratories 13 and 15.

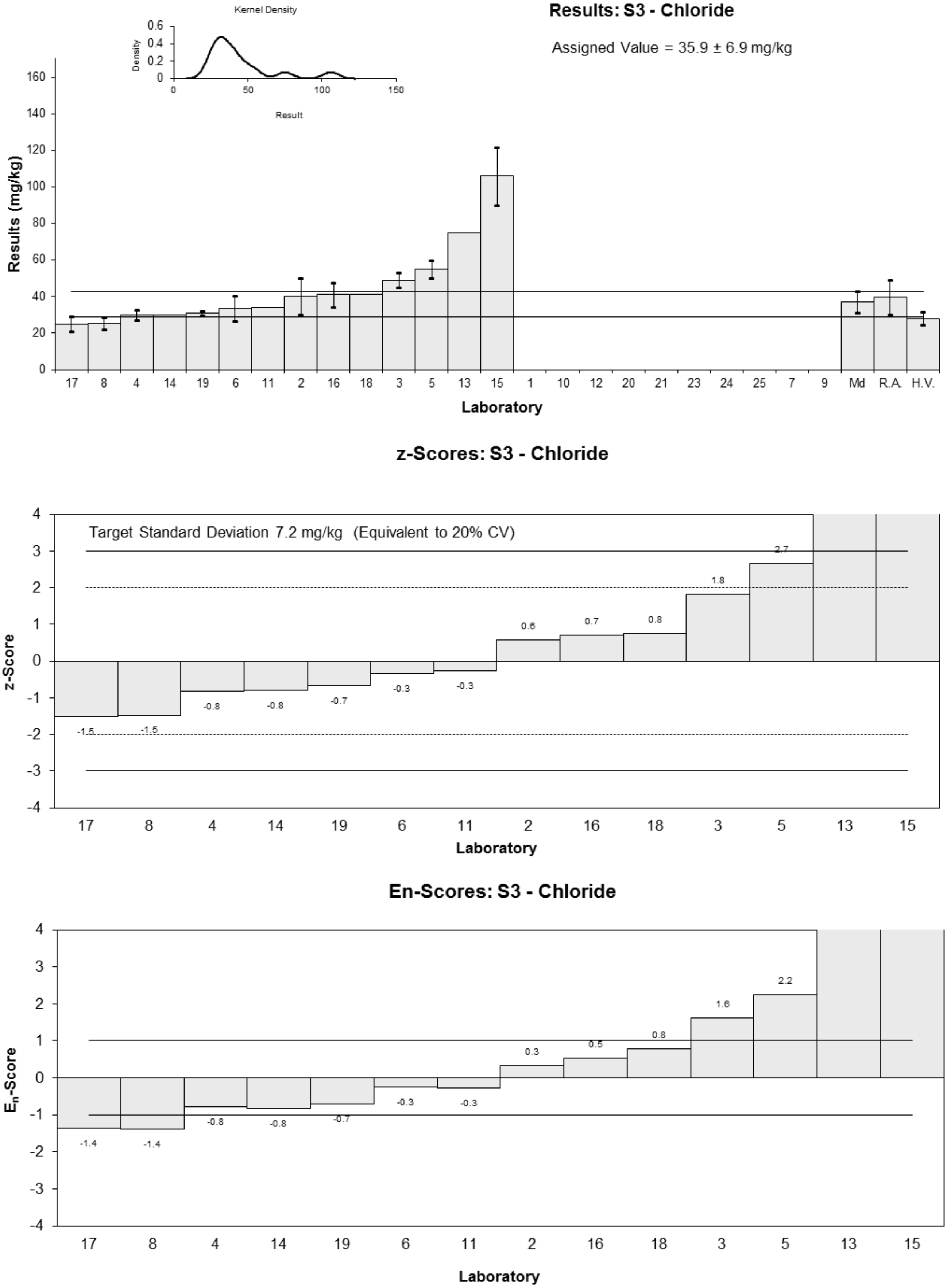

Results: S3 - Chloride
Assigned Value = 35.9 ± 6.9 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
17 8 4 14 19 6 11 2 16 18 3 5 13 15 1 10 12 20 21 23 24 25 7 9 Md R.A. H.V.
z-Scores: S3 - Chloride
Target Standard Deviation 7.2 mg/kg (Equivalent to 20% CV)
z-Score
-1.5 -1.5 -0.8 -0.8 -0.7 -0.3 -0.3 0.6 0.7 0.8 1.8 2.7
17 8 4 14 19 6 11 2 16 18 3 5 13 15
Laboratory
En-Scores: S3 - Chloride
E_n-Score
-1.4 -1.4 -0.8 -0.8 -0.7 -0.3 -0.3 0.3 0.5 0.8 1.6 2.2
17 8 4 14 19 6 11 2 16 18 3 5 13 15
Laboratory

Figure 40

Table 51

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	EC
Units	µS/cm

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	280	35	-0.28	-0.21
3	279	23.44	-0.31	-0.34
4	300	30	0.42	0.37
5	259	26	-1.01	-1.00
6	311	62	0.80	0.36
7	NT	NT		
8	296	23.6	0.28	0.30
9	312	87	0.83	0.27
10	288	29	0.00	0.00
11	280	NR	-0.28	-0.62
12	NT	NT		
13	240	NR	-1.67	-3.69
14	297.7	NR	0.34	0.75
15	296	44	0.28	0.17
16	380	19	3.19	4.00
17	270	40	-0.62	-0.43
18	286	NR	-0.07	-0.15
19	280	14	-0.28	-0.42
20	NT	NT		
21	NT	NT		
23	496	38	7.22	5.18
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value*	288	13
Spike	Not Spiked	
Homogeneity Value	292	35
Robust Average	291	14
Median	288	7
Mean	303	
N	17	
Max.	496	
Min.	240	
Robust SD	20	
Robust CV	6.9%	

*Robust Average excluding Laboratory 23.

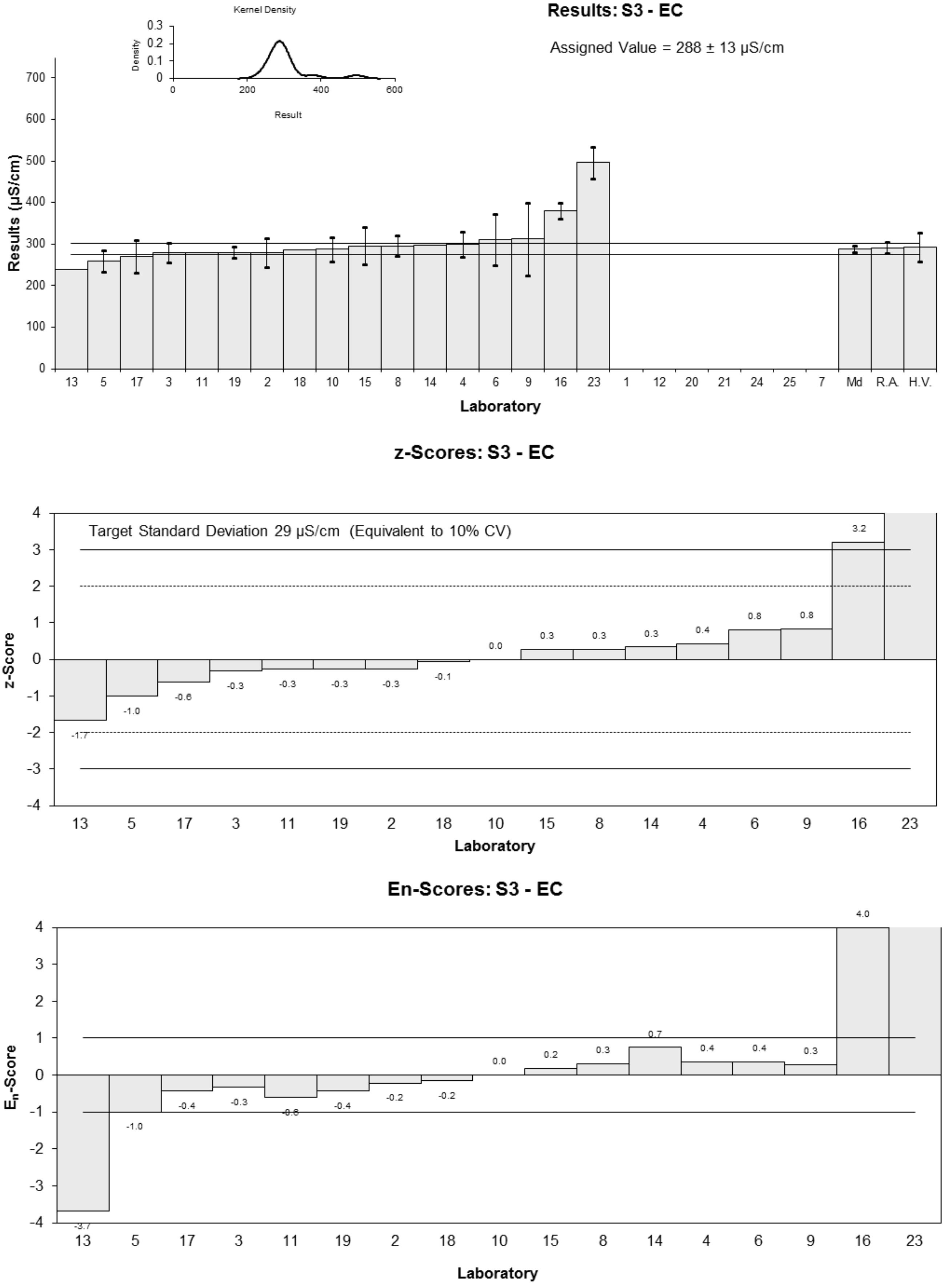

Results: S3 - EC
Assigned Value = 288 ± 13 µS/cm
Kernel Density
Density
Result
Results (µS/cm)
Laboratory
13 5 17 3 11 19 2 18 10 15 8 14 4 6 9 16 23 1 12 20 21 24 25 7 Md R.A. H.V.
z-Scores: S3 - EC
Target Standard Deviation 29 µS/cm (Equivalent to 10% CV)
z-Score
-1.7 -1.0 -0.6 -0.3 -0.3 -0.3 -0.3 -0.1 0.0 0.3 0.3 0.3 0.4 0.8 0.8 3.2
13 5 17 3 11 19 2 18 10 15 8 14 4 6 9 16 23
Laboratory
En-Scores: S3 - EC
E_n-Score
-3.7 -1.0 -0.4 -0.3 -0.6 -0.4 -0.2 -0.2 0.0 0.2 0.3 0.7 0.4 0.4 0.3 4.0
13 5 17 3 11 19 2 18 10 15 8 14 4 6 9 16 23
Laboratory

Figure 41

Table 52

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	Fe
Units	mg/kg

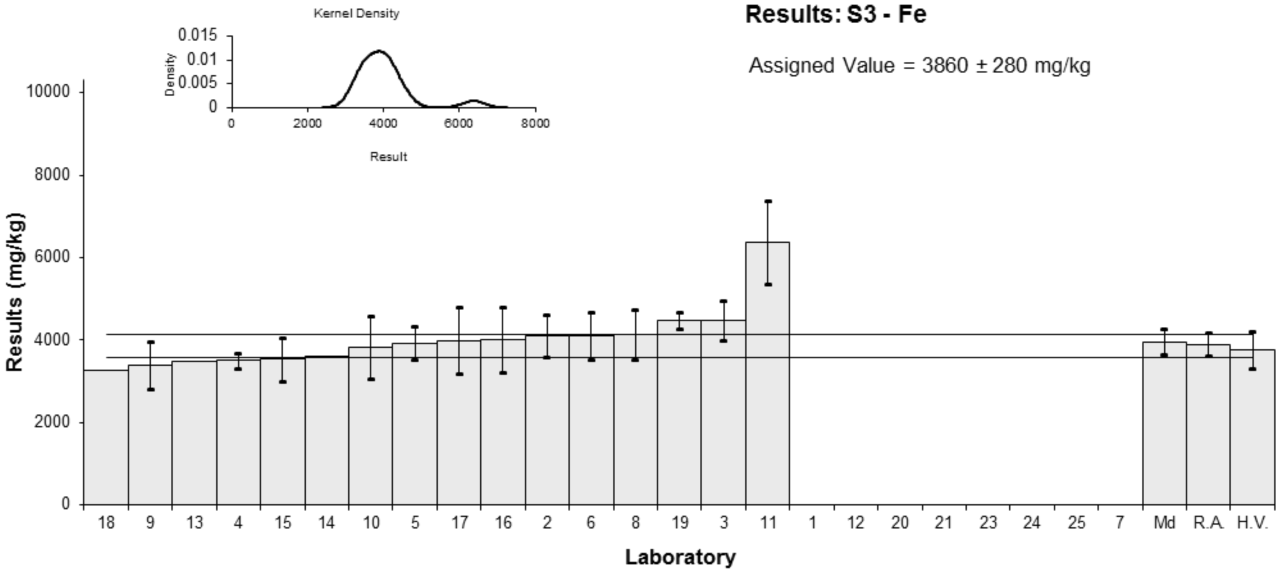
Participant Results

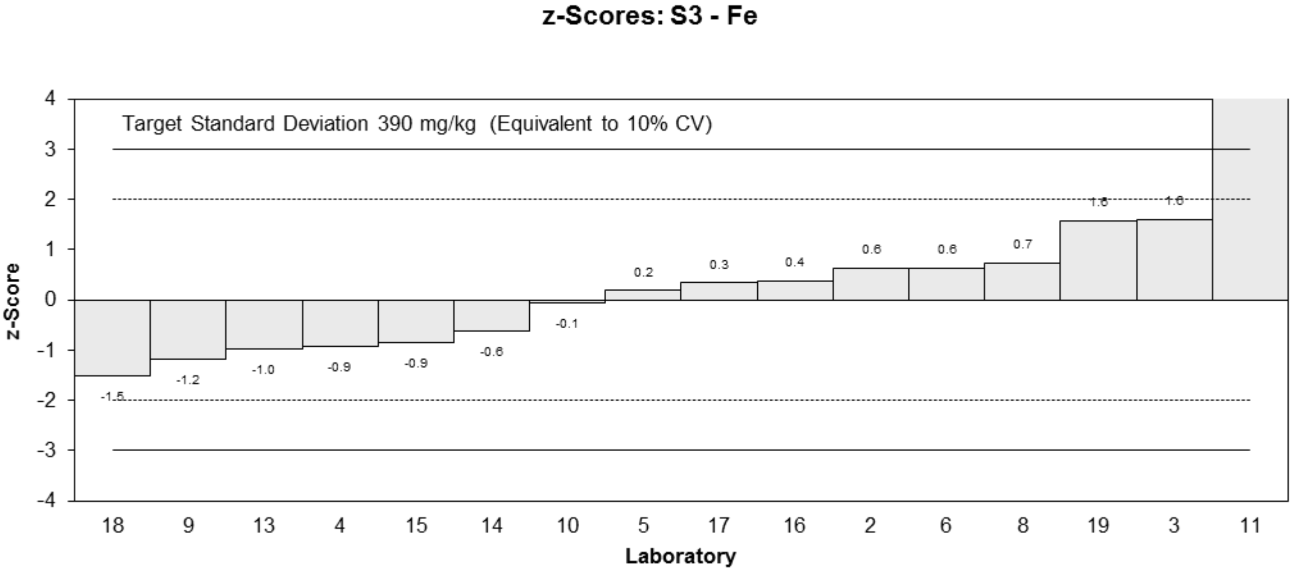
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	4100	510	0.62	0.41
3	4480	492.8	1.61	1.09
4	3500	190	-0.93	-1.06
5	3930	400	0.18	0.14
6	4100	566	0.62	0.38
7	NT	NT		
8	4142	601	0.73	0.43
9	3400	577	-1.19	-0.72
10	3833.13	766.63	-0.07	-0.03
11	6370	1000	6.50	2.42
12	NT	NT		
13	3484	NR	-0.97	-1.34
14	3620	NR	-0.62	-0.86
15	3530	529	-0.85	-0.55
16	4008	780	0.38	0.18
17	3990	800	0.34	0.15
18	3277	NR	-1.51	-2.08
19	4470	210	1.58	1.74
20	NT	NT		
21	NT	NT		
23	NT	NT		
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value*	3860	280
Spike	Not Spiked	
Homogeneity Value	3750	450
Robust Average	3900	290
Median	3960	300
Mean	4015	
N	16	
Max.	6370	
Min.	3277	
Robust SD	430	
Robust CV	11%	

*Robust Average excluding Laboratory 11.


Results: S3 - Fe
Assigned Value = 3860 ± 280 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
18 9 13 4 15 14 10 5 17 16 2 6 8 19 3 11 1 12 20 21 23 24 25 7 Md R.A. H.V.


z-Scores: S3 - Fe
Target Standard Deviation 390 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
18 9 13 4 15 14 10 5 17 16 2 6 8 19 3 11

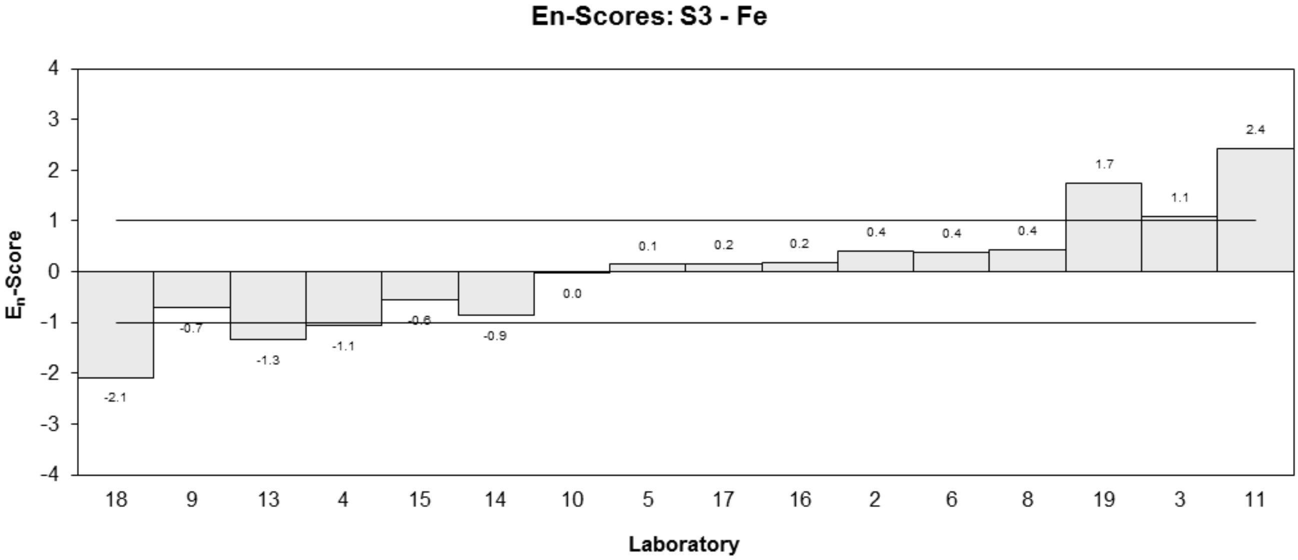

En-Scores: S3 - Fe
En-Score
Laboratory
18 9 13 4 15 14 10 5 17 16 2 6 8 19 3 11

Figure 42

Table 53

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	Fluoride
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	1.6	0.5	0.48	0.25
3	1.22	0.104	-0.82	-0.86
4	NT	NT		
5	1.4	0.2	-0.21	-0.18
6	0.66	0.11	-2.74	-2.83
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	NT	NT		
12	NT	NT		
13	NR	NR		
14	2.82	NR	4.66	5.23
15	NT	NT		
16	<100	NR		
17	1.6	0.3	0.48	0.35
18	NR	NR		
19	NT	NT		
20	NT	NT		
21	NT	NT		
23	NT	NT		
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value*	1.46	0.26
Spike	Not Spiked	
Homogeneity Value	1.67	0.20
Robust Average	1.51	0.73
Median	1.50	0.30
Mean	1.55	
N	6	
Max.	2.82	
Min.	0.66	
Robust SD	0.21	
Robust CV	14%	

*Robust Average excluding Laboratories 6 and 14.

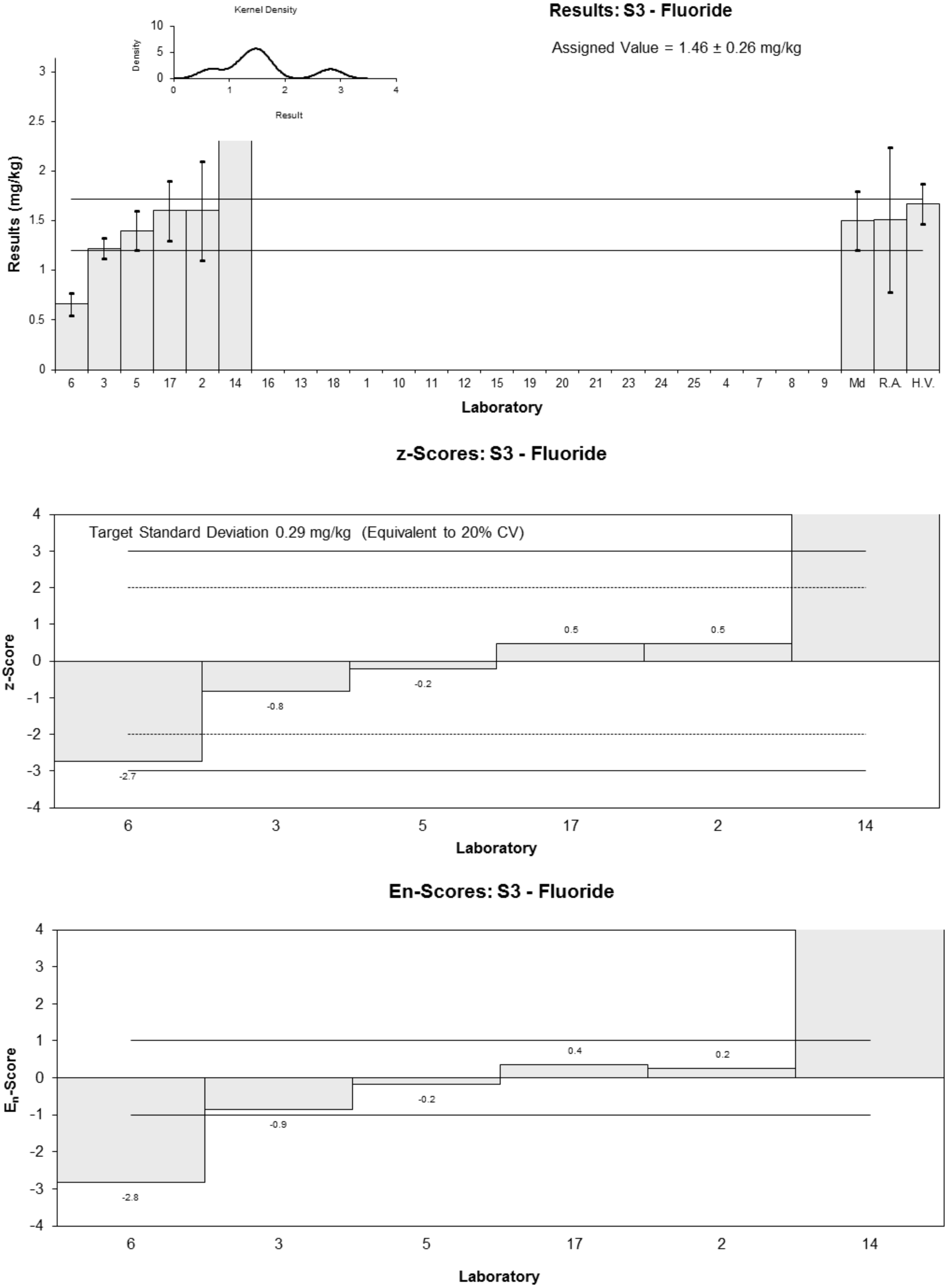

Results: S3 - Fluoride
Assigned Value = 1.46 ± 0.26 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
6 3 5 17 2 14 16 13 18 1 10 11 12 15 19 20 21 23 24 25 4 7 8 9 Md R.A. H.V.
z-Scores: S3 - Fluoride
Target Standard Deviation 0.29 mg/kg (Equivalent to 20% CV)
z-Score
-2.7
-0.8
-0.2
0.5
0.5
Laboratory
En-Scores: S3 - Fluoride
En-Score
-2.8
-0.9
-0.2
0.4
0.2
Laboratory

Figure 43

Table 54

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	K
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	480	60	-0.76	-0.57
3	731	35.82	2.32	1.95
4	500	150	-0.52	-0.24
5	517	50	-0.31	-0.24
6	560	91.8	0.22	0.14
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	417.50	83.50	-1.53	-1.01
11	770	100	2.80	1.69
12	NT	NT		
13	432	NR	-1.35	-1.22
14	570	NR	0.34	0.31
15	470	70.5	-0.89	-0.63
16	615.1	63	0.90	0.67
17	530	110	-0.15	-0.08
18	448	NR	-1.16	-1.04
19	760	100	2.68	1.62
20	NT	NT		
21	NT	NT		
23	360	79.3	-2.24	-1.52
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value	542	90
Spike	Not Spiked	
Homogeneity Value	522	63
Robust Average	542	90
Median	517	57
Mean	544	
N	15	
Max.	770	
Min.	360	
Robust SD	140	
Robust CV	26%	

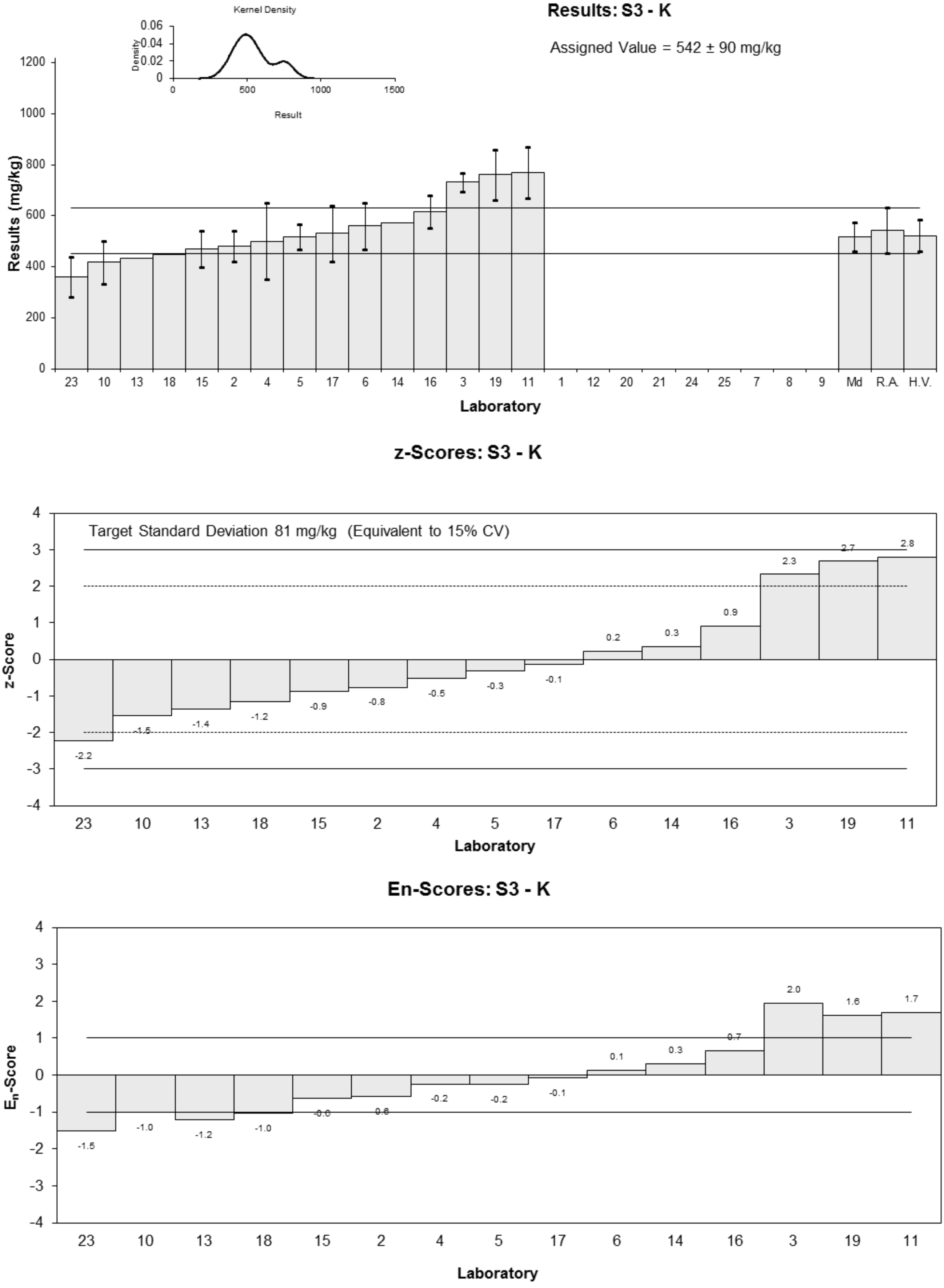

Results: S3 - K
Assigned Value = 542 ± 90 mg/kg
Kernel Density
Density
0.06
0.04
0.02
0
0
500
1000
1500
Result
Results (mg/kg)
1200
1000
800
600
400
200
0
23 10 13 18 15 2 4 5 17 6 14 16 3 19 11 1 12 20 21 24 25 7 8 9 Md R.A. H.V.
Laboratory
z-Scores: S3 - K
Target Standard Deviation 81 mg/kg (Equivalent to 15% CV)
z-Score
4
3
2
1
0
-1
-2
-3
-4
-2.2 -1.5 -1.4 -1.2 -0.9 -0.8 -0.5 -0.3 -0.1 0.2 0.3 0.9 2.3 2.7 2.8
23 10 13 18 15 2 4 5 17 6 14 16 3 19 11
Laboratory
En-Scores: S3 - K
En-Score
4
3
2
1
0
-1
-2
-3
-4
-1.5 -1.0 -1.2 -1.0 -0.6 -0.6 -0.2 -0.2 -0.1 0.1 0.3 0.7 2.0 1.6 1.7
23 10 13 18 15 2 4 5 17 6 14 16 3 19 11
Laboratory

Figure 44

Table 55

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	Mg
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	430	54	-1.49	-1.09
3	580	33.1	1.49	1.38
4	470	150	-0.69	-0.22
5	514	50	0.18	0.14
6	490	52.9	-0.30	-0.22
7	NT	NT		
8	612	92	2.12	1.05
9	NT	NT		
10	478.08	95.62	-0.53	-0.26
11	777	100	5.39	2.50
12	NT	NT		
13	409	NR	-1.90	-2.23
14	500	NR	-0.10	-0.12
15	452	67.8	-1.05	-0.66
16	498.6	160	-0.13	-0.04
17	500	100	-0.10	-0.05
18	592	NR	1.72	2.02
19	575	40	1.39	1.19
20	NT	NT		
21	NT	NT		
23	477	79.1	-0.55	-0.31
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value*	505	43
Spike	Not Spiked	
Homogeneity Value	493	59
Robust Average	513	46
Median	499	30
Mean	522	
N	16	
Max.	777	
Min.	409	
Robust SD	67	
Robust CV	13%	

*Robust Average excluding Laboratory 11.

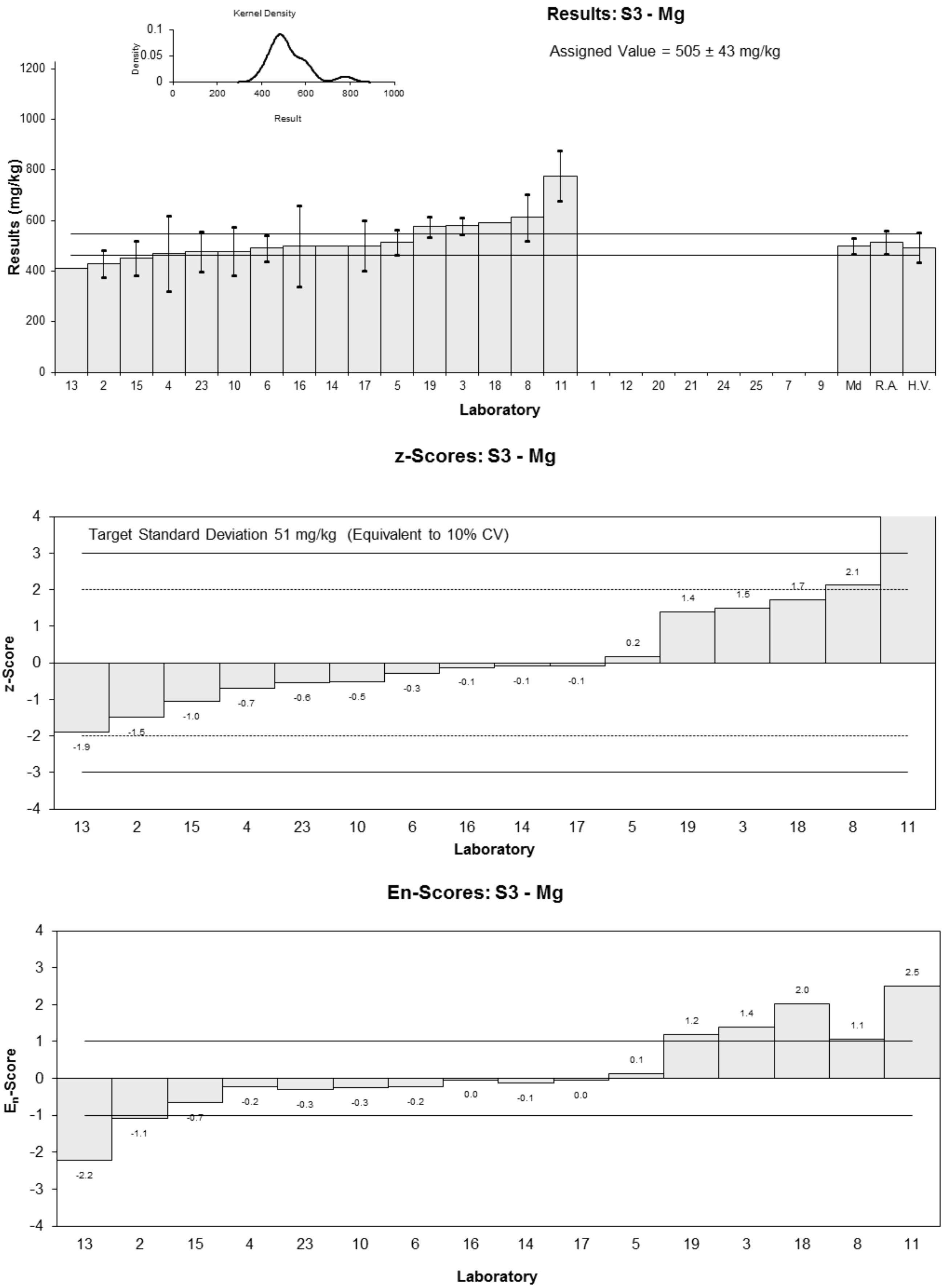

Results: S3 - Mg
Assigned Value = 505 ± 43 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
13 2 15 4 23 10 6 16 14 17 5 19 3 18 8 11 1 12 20 21 24 25 7 9 Md R.A. H.V.
z-Scores: S3 - Mg
Target Standard Deviation 51 mg/kg (Equivalent to 10% CV)
z-Score
-1.9 -1.5 -1.0 -0.7 -0.6 -0.5 -0.3 -0.1 -0.1 -0.1 0.2 1.4 1.5 1.7 2.1
13 2 15 4 23 10 6 16 14 17 5 19 3 18 8 11
Laboratory
En-Scores: S3 - Mg
E_n-Score
-2.2 -1.1 -0.7 -0.2 -0.3 -0.3 -0.2 0.0 -0.1 0.0 0.1 1.2 1.4 2.0 1.1 2.5
13 2 15 4 23 10 6 16 14 17 5 19 3 18 8 11
Laboratory

Figure 45

Table 56

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	Na
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	75	10	0.29	0.29
3	94.0	6.30	1.63	1.97
4	69	16	-0.13	-0.10
5	66	10	-0.35	-0.35
6	70	8.26	-0.06	-0.07
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	82.11	16.42	0.79	0.58
11	161	30	6.35	2.85
12	NT	NT		
13	59.5	NR	-0.80	-1.15
14	70	NR	-0.06	-0.09
15	90.7	13.6	1.40	1.18
16	48.66	5.9	-1.57	-1.93
17	67	14	-0.28	-0.23
18	124	NR	3.74	5.36
19	76	12	0.36	0.33
20	NT	NT		
21	NT	NT		
23	55	8.7	-1.12	-1.21
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value*	70.9	9.9
Spike	Not Spiked	
Homogeneity Value	59.7	7.2
Robust Average	75	12
Median	70.0	8.6
Mean	80.5	
N	15	
Max.	161	
Min.	48.66	
Robust SD	14.0	
Robust CV	19%	

*Robust Average excluding Laboratories 11 and 18.

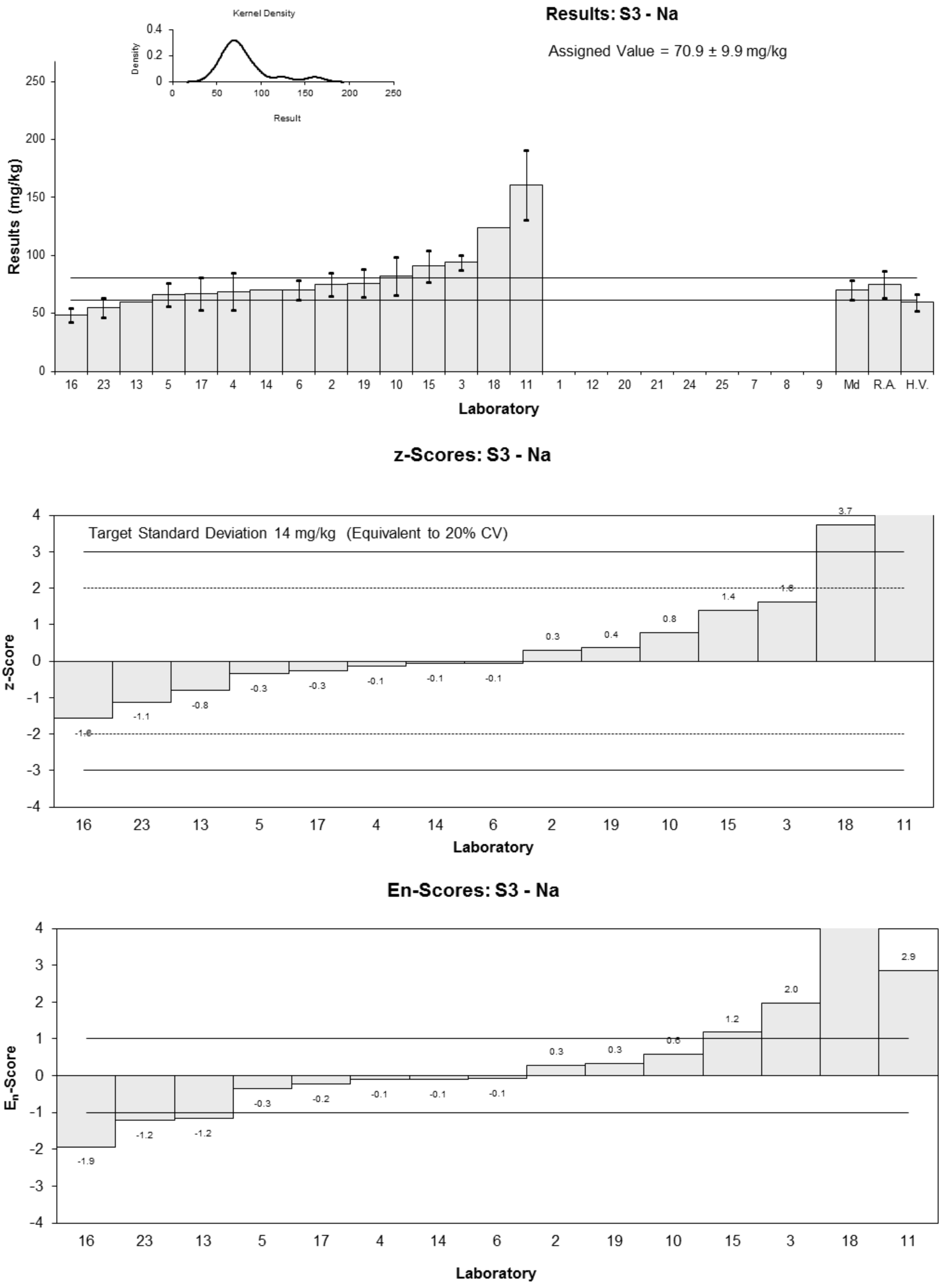

Results: S3 - Na
Assigned Value = 70.9 ± 9.9 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
16 23 13 5 17 4 14 6 2 19 10 15 3 18 11 1 12 20 21 24 25 7 8 9 Md R.A. H.V.
z-Scores: S3 - Na
Target Standard Deviation 14 mg/kg (Equivalent to 20% CV)
z-Score
-1.6 -1.1 -0.8 -0.3 -0.3 -0.1 -0.1 -0.1 0.3 0.4 0.8 1.4 1.6 3.7
16 23 13 5 17 4 14 6 2 19 10 15 3 18 11
Laboratory
En-Scores: S3 - Na
E_n-Score
-1.9 -1.2 -1.2 -0.3 -0.2 -0.1 -0.1 -0.1 0.3 0.3 0.6 1.2 2.0 2.9
16 23 13 5 17 4 14 6 2 19 10 15 3 18 11
Laboratory

Figure 46

Table 57

Sample Details

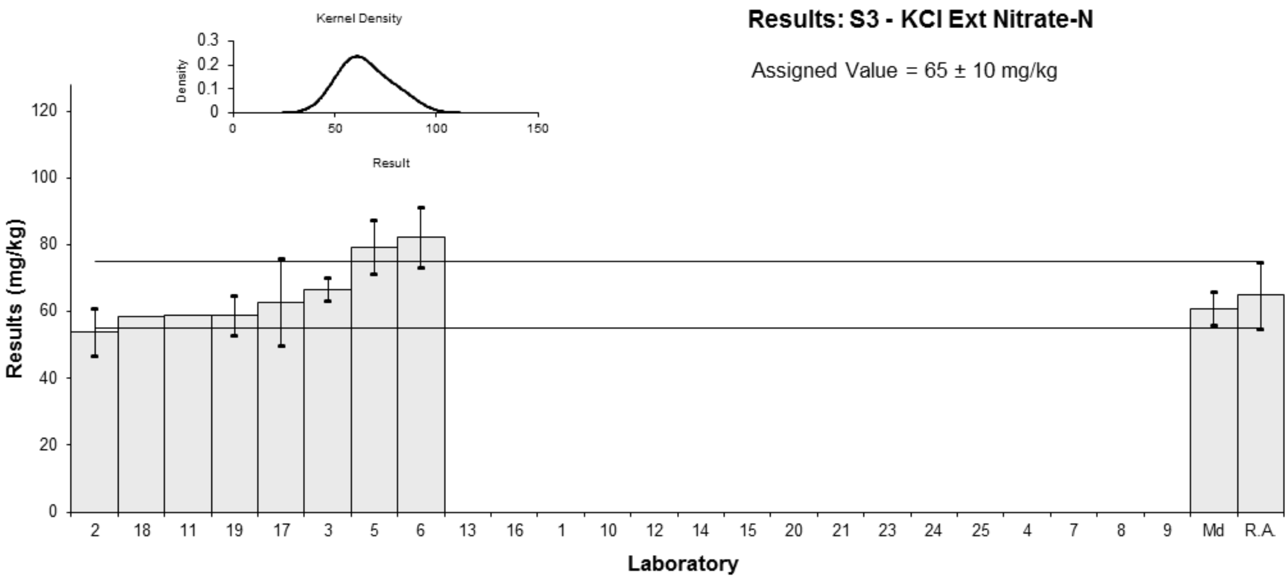
Sample No.	S3
Matrix.	Soil
Analyte.	KCl Ext Nitrate-N
Units	mg/kg

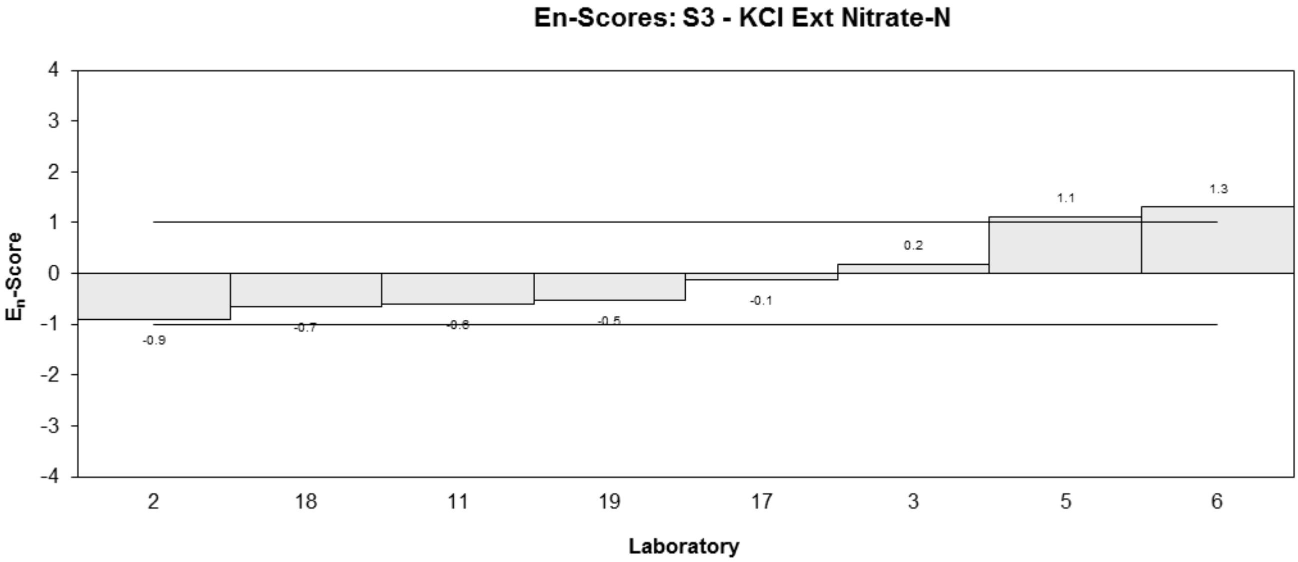
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	54	7	-0.85	-0.90
3	66.8	3.41	0.14	0.17
4	NT	NT		
5	79.4	8.0	1.11	1.12
6	82.5	8.97	1.35	1.30
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	59	NR	-0.46	-0.60
12	NT	NT		
13	NR	NR		
14	NT	NT		
15	NT	NT		
16	NR	NR		
17	63	13	-0.15	-0.12
18	58.5	NR	-0.50	-0.65
19	59	5.8	-0.46	-0.52
20	NT	NT		
21	NT	NT		
23	NT	NT		
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value	65	10
Spike	Not Spiked	
Robust Average	65	10
Median	61	5
Mean	65	
N	8	
Max.	82.5	
Min.	54	
Robust SD	12	
Robust CV	19%	


Results: S3 - KCl Ext Nitrate-N
Assigned Value = 65 ± 10 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
2 18 11 19 17 3 5 6 13 16 1 10 12 14 15 20 21 23 24 25 4 7 8 9 Md R.A.


En-Scores: S3 - KCl Ext Nitrate-N
En-Score
Laboratory
-0.9 -0.7 -0.6 -0.5 -0.1 0.2 1.1 1.3
2 18 11 19 17 3 5 6

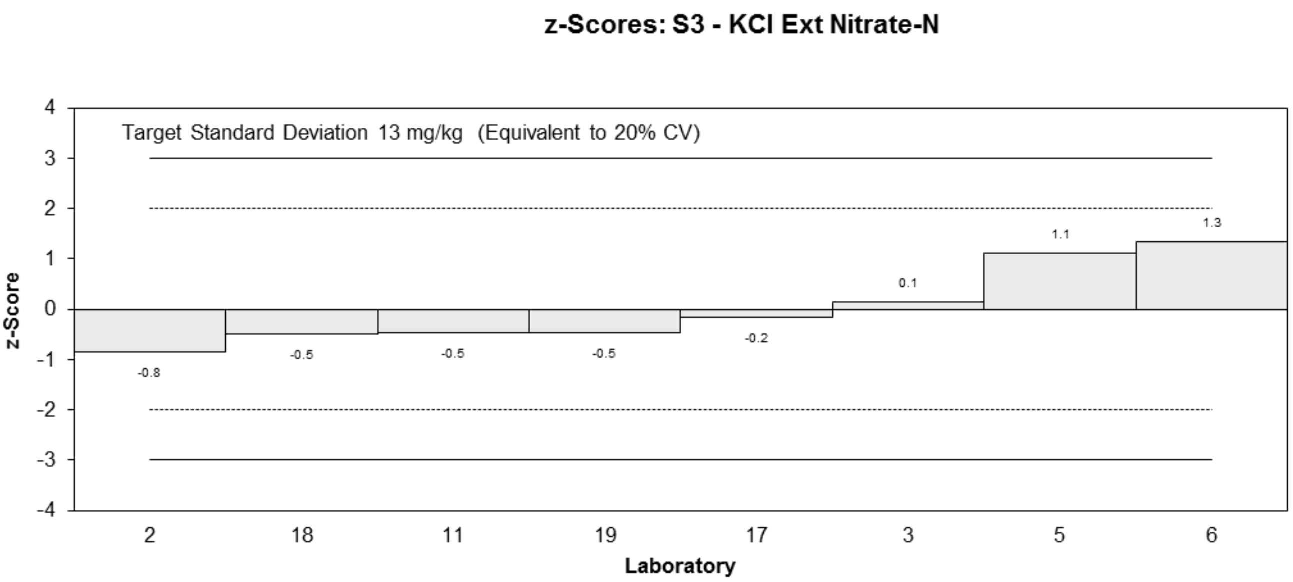

z-Scores: S3 - KCl Ext Nitrate-N
Target Standard Deviation 13 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
-0.8 -0.5 -0.5 -0.5 -0.2 0.1 1.1 1.3
2 18 11 19 17 3 5 6

Figure 47

Table 58

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	Orthophosphate-P
Units	mg/kg

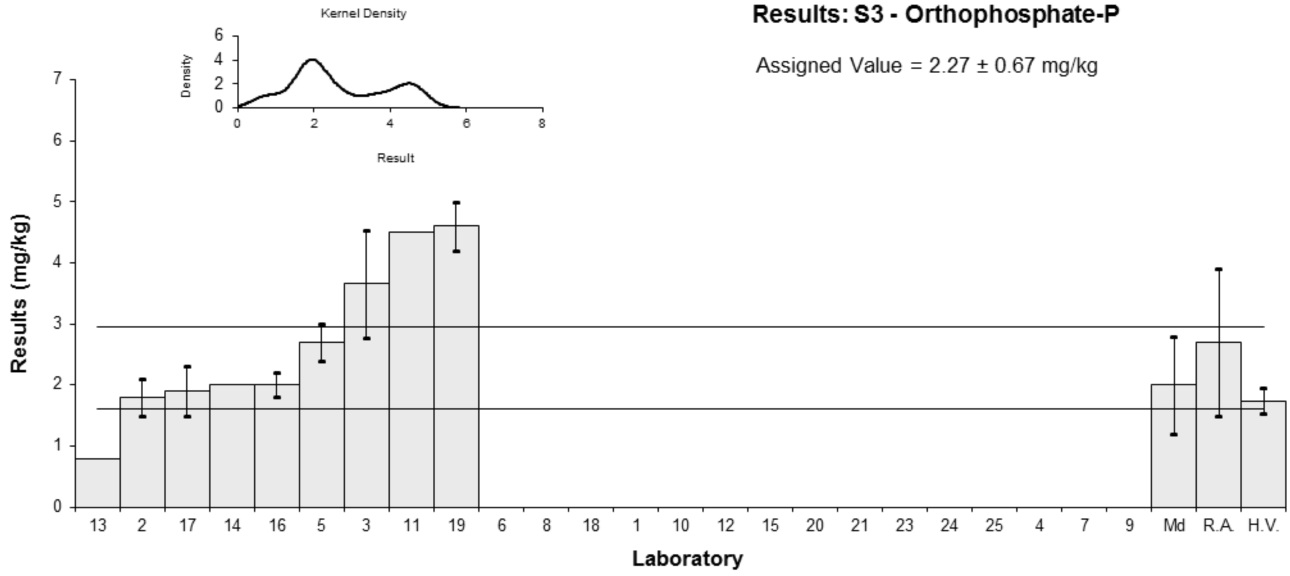
Participant Results

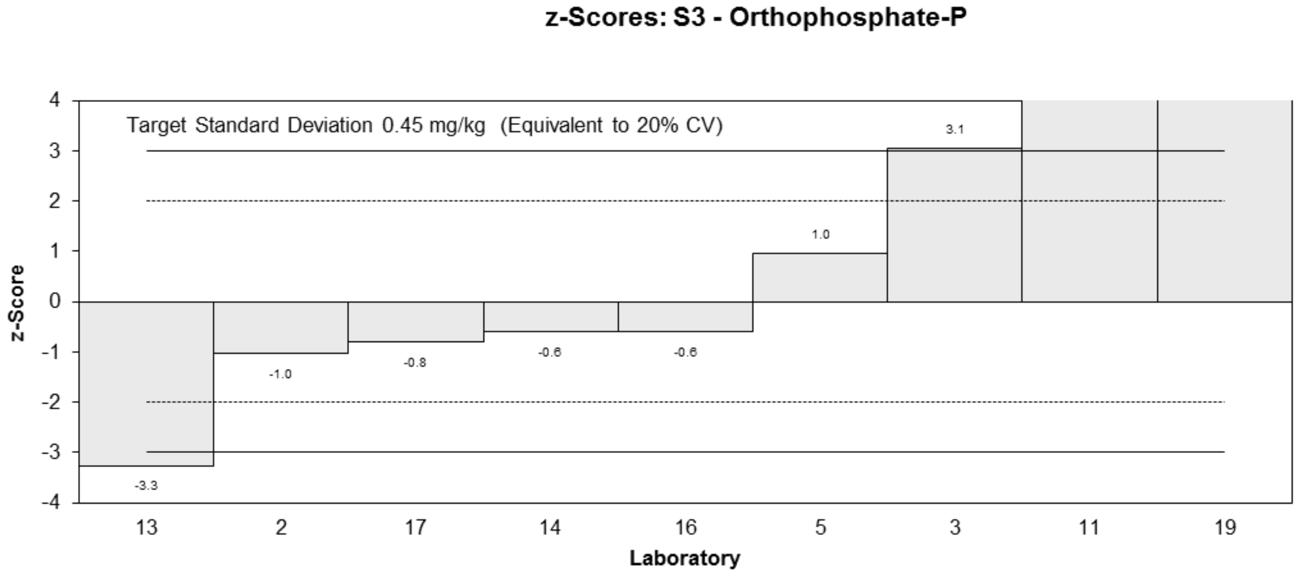
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	1.8	0.3	-1.04	-0.64
3	3.66	0.878	3.06	1.26
4	NT	NT		
5	2.7	0.3	0.95	0.59
6	< 10	1.63		
7	NT	NT		
8	<10	1.3		
9	NT	NT		
10	NT	NT		
11	4.5	NR	4.91	3.33
12	NT	NT		
13	0.788	NR	-3.26	-2.21
14	2.0	NR	-0.59	-0.4
15	NT	NT		
16	2	0.2	-0.59	-0.39
17	1.9	0.4	-0.81	-0.47
18	NR	NR		
19	4.6	0.4	5.13	2.99
20	NT	NT		
21	NT	NT		
23	NT	NT		
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value*	2.27	0.67
Spike	Not Spiked	
Homogeneity Value	1.74	0.21
Robust Average	2.7	1.2
Median	2.00	0.80
Mean	2.66	
N	9	
Max.	4.6	
Min.	0.788	
Robust SD	0.65	
Robust CV	24%	

*Robust Average excluding Laboratories 11, 13 and 19.


Results: S3 - Orthophosphate-P
Assigned Value = 2.27 ± 0.67 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
13
2
17
14
16
5
3
11
19
6
8
18
1
10
12
15
20
21
23
24
25
4
7
9
Md
R.A.
H.V.


z-Scores: S3 - Orthophosphate-P
Target Standard Deviation 0.45 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
-3.3
-1.0
-0.8
-0.6
-0.6
1.0
3.1
13
2
17
14
16
5
3
11
19

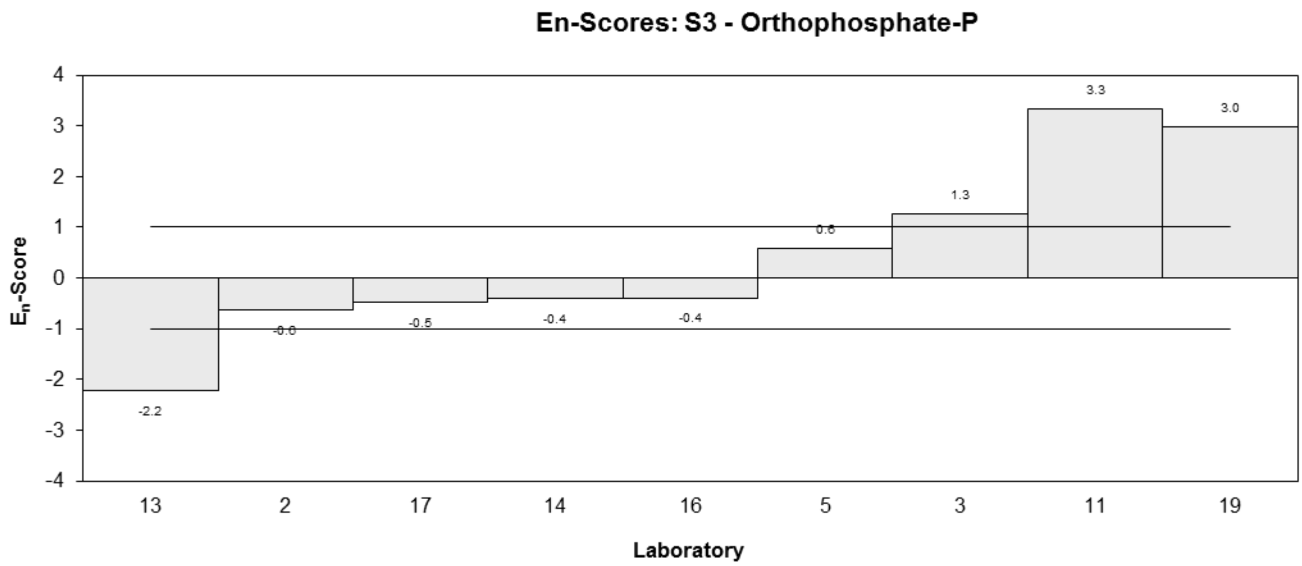

En-Scores: S3 - Orthophosphate-P
E_n-Score
Laboratory
-2.2
-0.5
-0.4
-0.4
1.3
3.3
3.0
13
2
17
14
16
5
3
11
19

Figure 48

Table 59

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	P
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	260	32	-0.58	-0.47
3	278	66.7	0.07	0.03
4	290	50	0.51	0.27
5	294	30	0.65	0.56
6	270	34.8	-0.22	-0.16
7	NT	NT		
8	303	61	0.98	0.44
9	NT	NT		
10	294.04	58.81	0.65	0.30
11	281	50	0.18	0.10
12	NT	NT		
13	267	NR	-0.33	-0.82
14	280	NR	0.14	0.36
15	271	40.7	-0.18	-0.12
16	247.4	32	-1.04	-0.85
17	260	50	-0.58	-0.31
18	289	NR	0.47	1.18
19	260	34	-0.58	-0.45
20	NT	NT		
21	NT	NT		
23	NT	NT		
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value	276	11
Spike	Not Spiked	
Homogeneity Value	264	32
Robust Average	276	11
Median	278	10
Mean	276	
N	15	
Max.	303	
Min.	247.4	
Robust SD	18	
Robust CV	6.5%	

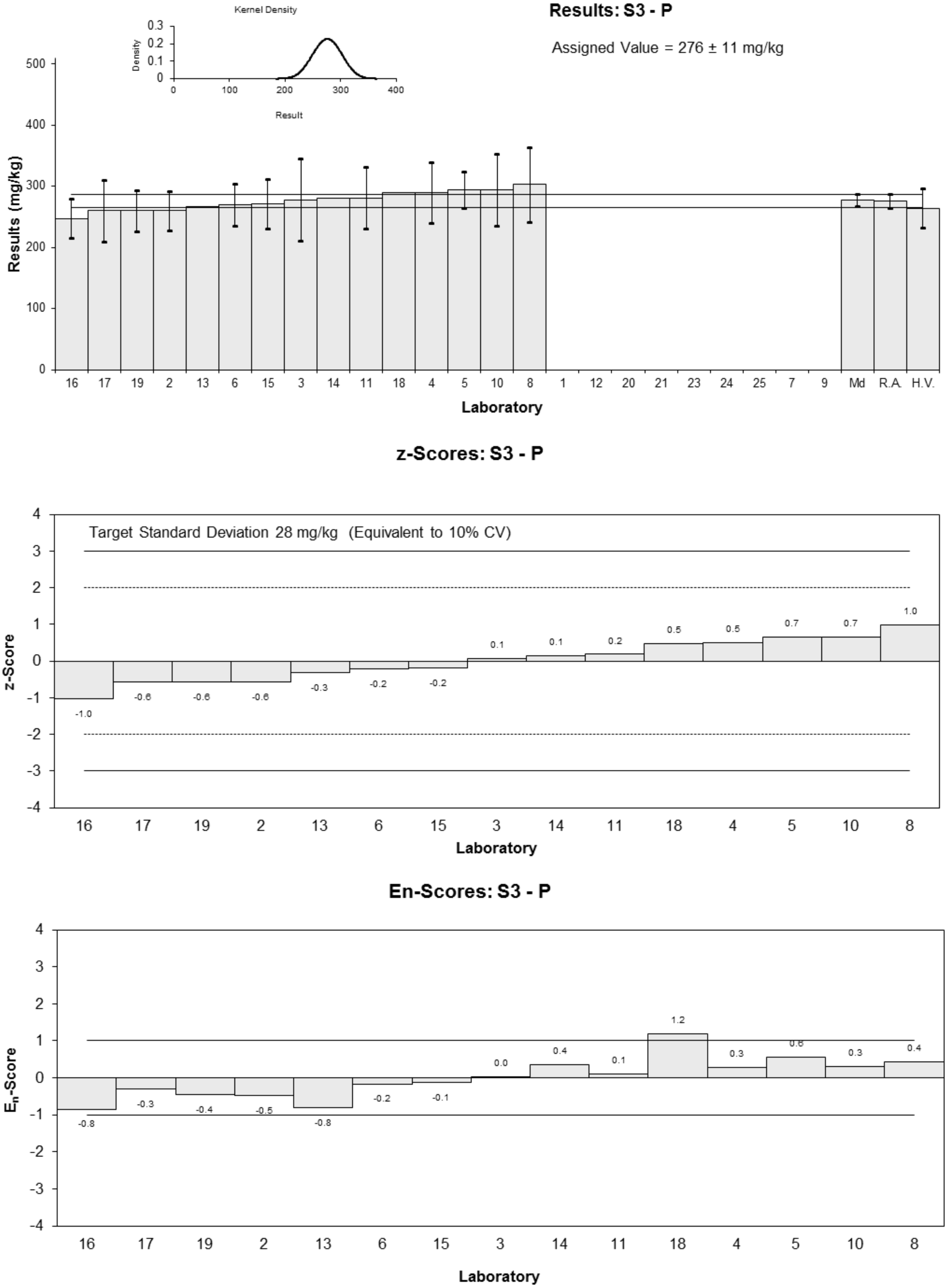

Results: S3 - P
Assigned Value = 276 ± 11 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
16 17 19 2 13 6 15 3 14 11 18 4 5 10 8 1 12 20 21 23 24 25 7 9 Md R.A. H.V.
z-Scores: S3 - P
Target Standard Deviation 28 mg/kg (Equivalent to 10% CV)
z-Score
-1.0 -0.6 -0.6 -0.6 -0.3 -0.2 -0.2 0.1 0.1 0.2 0.5 0.5 0.7 0.7 1.0
16 17 19 2 13 6 15 3 14 11 18 4 5 10 8
Laboratory
En-Scores: S3 - P
En-Score
-0.8 -0.3 -0.4 -0.5 -0.8 -0.2 -0.1 0.0 0.4 0.1 1.2 0.3 0.6 0.3 0.4
16 17 19 2 13 6 15 3 14 11 18 4 5 10 8
Laboratory

Figure 49

Table 60

Sample Details

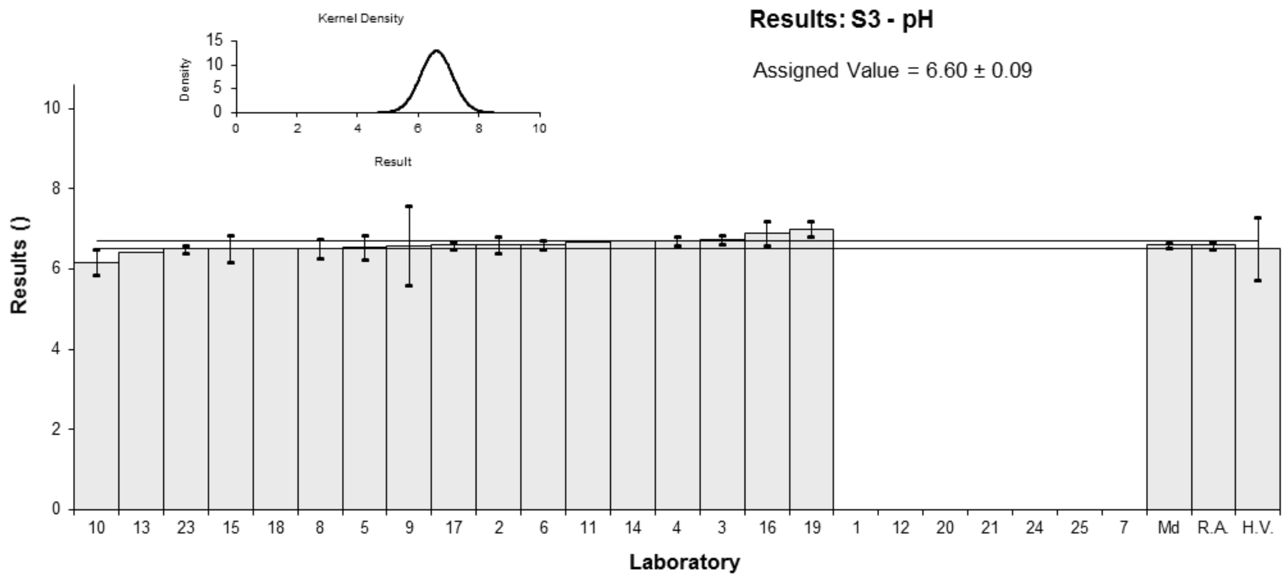
Sample No.	S3
Matrix.	Soil
Analyte.	pH

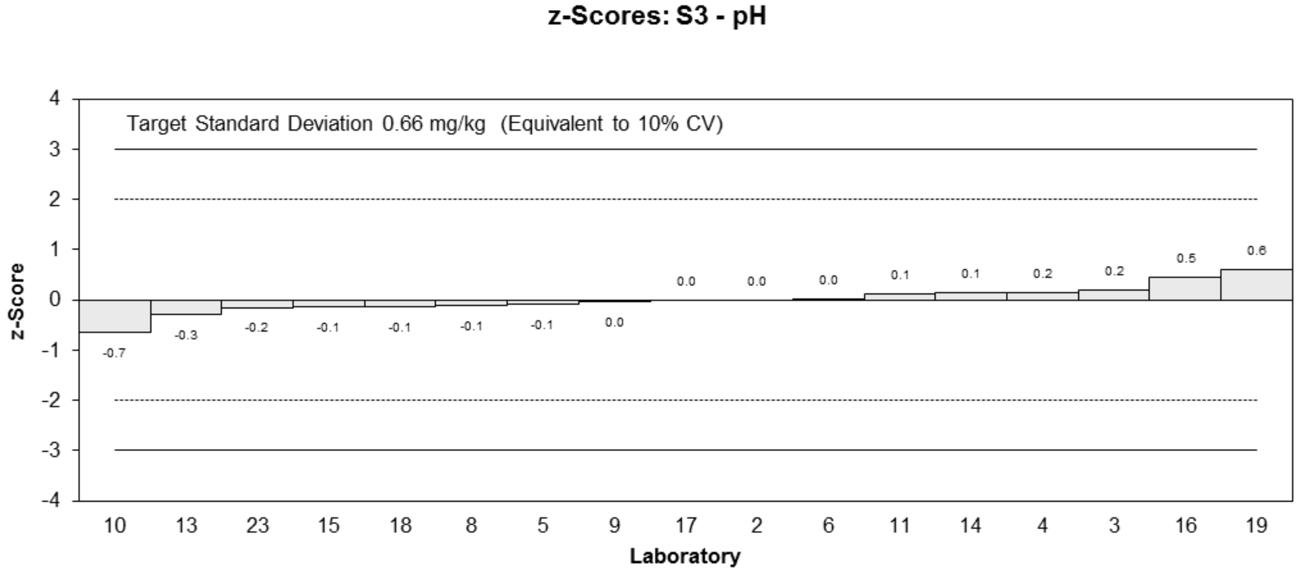
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	6.6	0.2	0.00	0.00
3	6.73	0.108	0.20	0.92
4	6.7	0.1	0.15	0.74
5	6.54	0.3	-0.09	-0.19
6	6.61	0.1	0.02	0.07
7	NT	NT		
8	6.52	0.24	-0.12	-0.31
9	6.58	0.99	-0.03	-0.02
10	6.17	0.31	-0.65	-1.33
11	6.68	NR	0.12	0.89
12	NT	NT		
13	6.4	NR	-0.30	-2.22
14	6.69	NR	0.14	1.00
15	6.51	0.33	-0.14	-0.26
16	6.9	0.3	0.45	0.96
17	6.6	0.09	0.00	0.00
18	6.51	NR	-0.14	-1.00
19	7.0	0.19	0.61	1.90
20	NT	NT		
21	NT	NT		
23	6.5	0.1	-0.15	-0.74
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value	6.60	0.09
Spike	Not Spiked	
Homogeneity Value	6.50	0.78
Robust Average	6.60	0.09
Median	6.60	0.07
Mean	6.60	
N	17	
Max.	7	
Min.	6.17	
Robust SD	0.10	
Robust CV	1.5%	


Results: S3 - pH
Assigned Value = 6.60 ± 0.09
Kernel Density
Density
Result
Results ()
Laboratory
10
13
23
15
18
8
5
9
17
2
6
11
14
4
3
16
19
1
12
20
21
24
25
7
Md
R.A.
H.V.


z-Scores: S3 - pH
Target Standard Deviation 0.66 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory

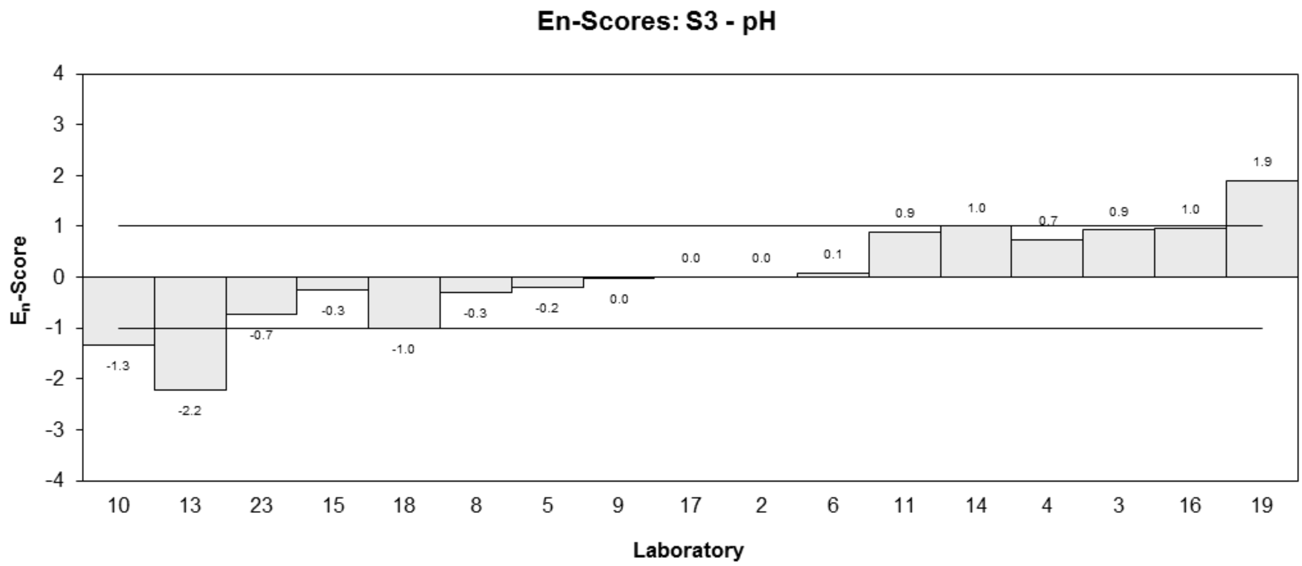

En-Scores: S3 - pH
E_n-Score
Laboratory

Figure 50

Table 61

Sample Details

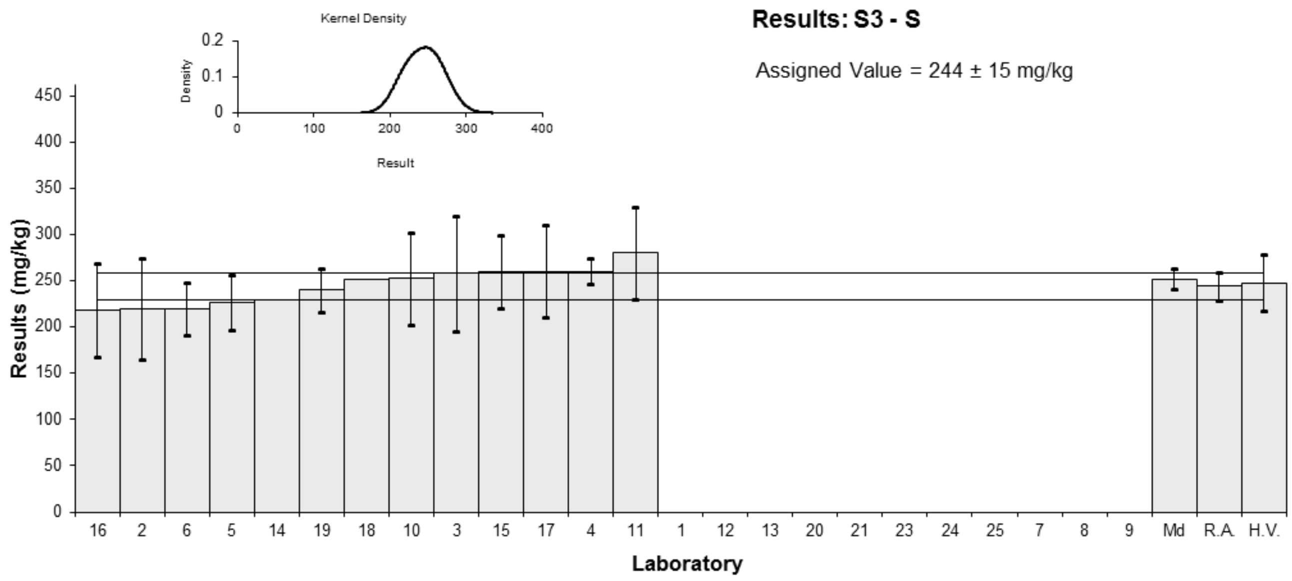
Sample No.	S3
Matrix.	Soil
Analyte.	S
Units	mg/kg

Participant Results

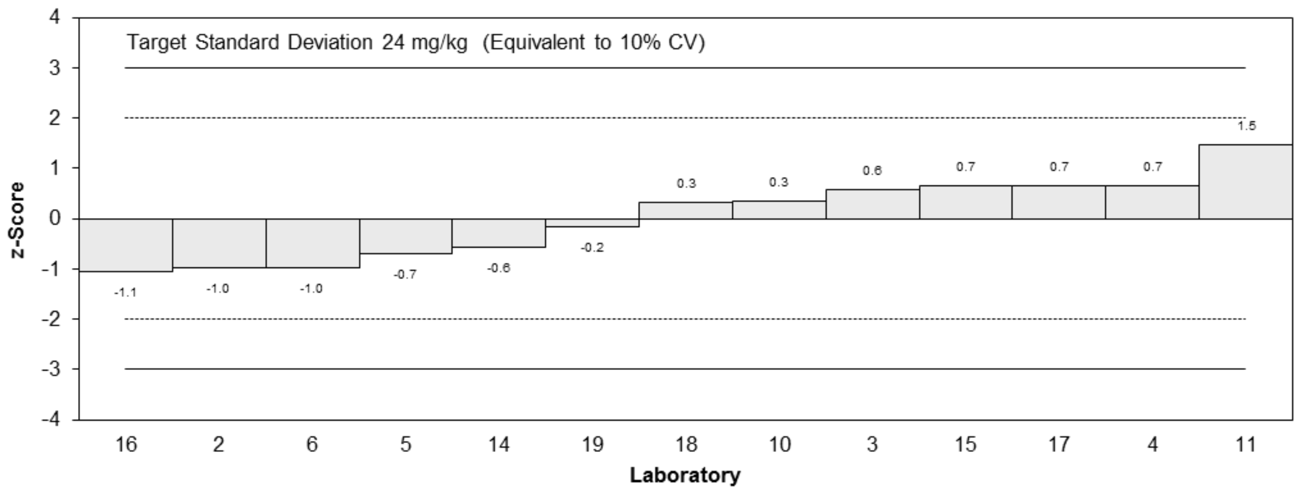
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	220	55	-0.98	-0.42
3	258	61.9	0.57	0.22
4	260	14	0.66	0.78
5	227	30	-0.70	-0.51
6	220	28.4	-0.98	-0.75
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	252.29	50.46	0.34	0.16
11	280	50	1.48	0.69
12	NT	NT		
13	NT	NT		
14	230	NR	-0.57	-0.93
15	260	39	0.66	0.38
16	218.3	51	-1.05	-0.48
17	260	50	0.66	0.31
18	252	NR	0.33	0.53
19	240	24	-0.16	-0.14
20	NT	NT		
21	NT	NT		
23	NT	NT		
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value	244	15
Spike	Not Spiked	
Homogeneity Value	248	30
Robust Average	244	15
Median	252	11
Mean	244	
N	13	
Max.	280	
Min.	218.3	
Robust SD	22	
Robust CV	9%	



z-Scores: S3 - S



En-Scores: S3 - S

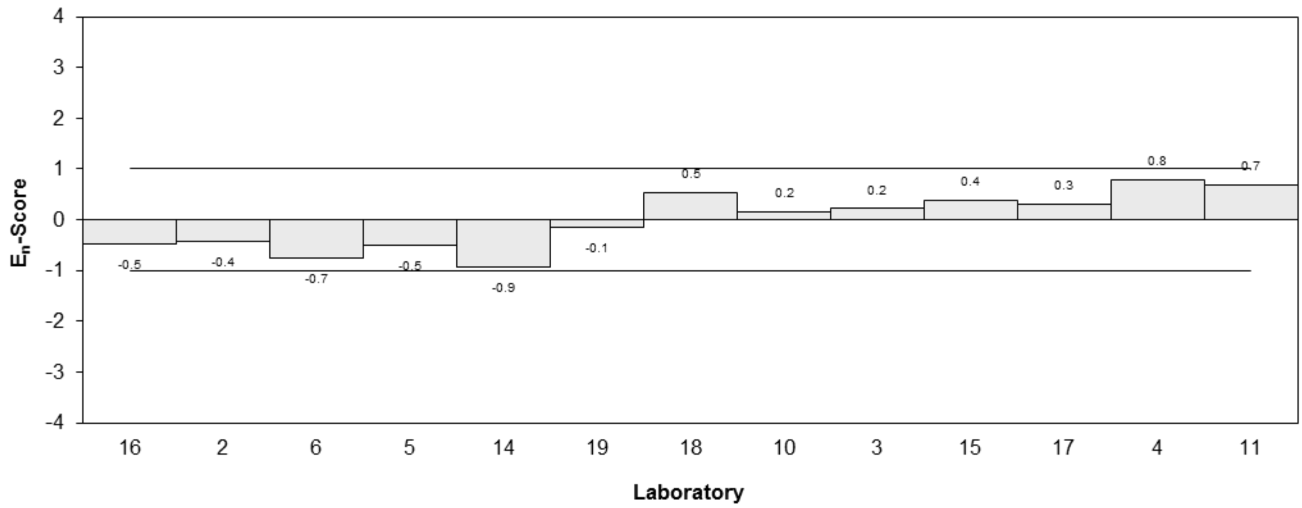


Figure 51

Table 62

Sample Details

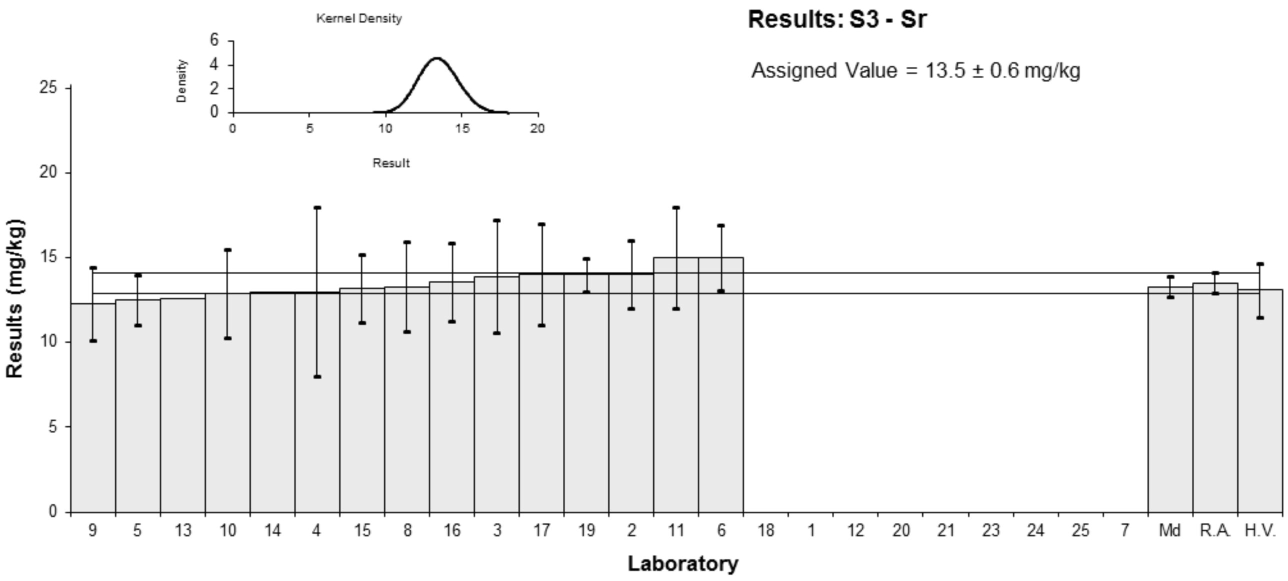
Sample No.	S3
Matrix.	Soil
Analyte.	Sr
Units	mg/kg

Participant Results

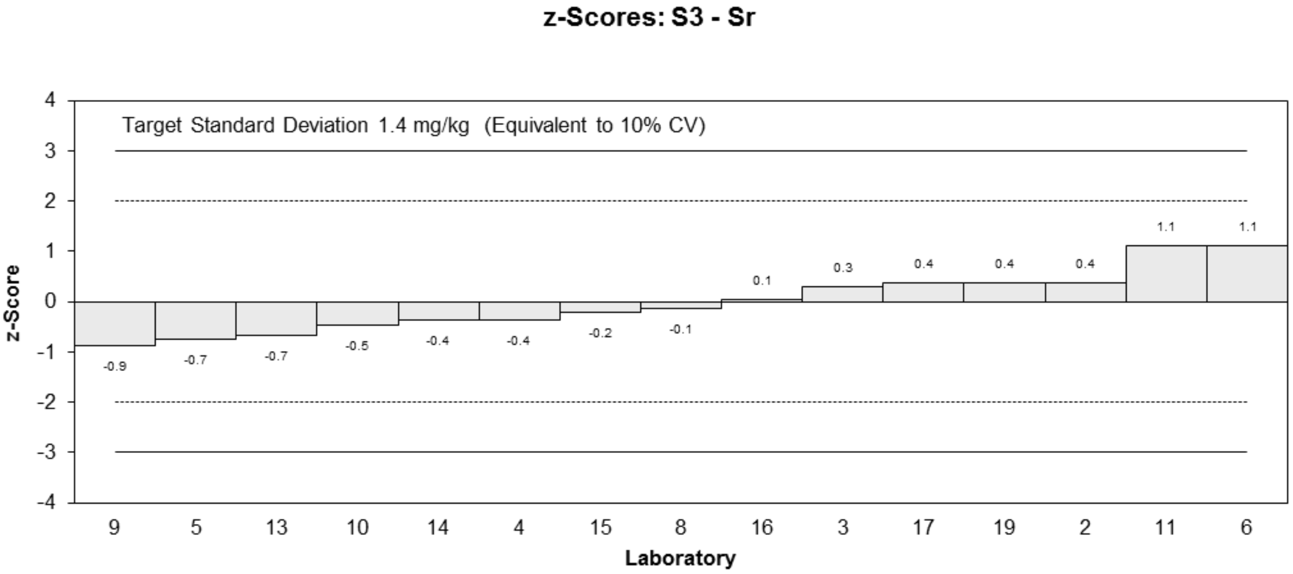
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	14	2	0.37	0.24
3	13.9	3.34	0.30	0.12
4	13	5	-0.37	-0.10
5	12.5	1.5	-0.74	-0.62
6	15	1.92	1.11	0.75
7	NT	NT		
8	13.3	2.66	-0.15	-0.07
9	12.3	2.15	-0.89	-0.54
10	12.88	2.58	-0.46	-0.23
11	15	3	1.11	0.49
12	NT	NT		
13	12.6	NR	-0.67	-1.50
14	13.0	NR	-0.37	-0.83
15	13.2	2.0	-0.22	-0.14
16	13.57	2.3	0.05	0.03
17	14	3	0.37	0.16
18	NR	NR		
19	14	1.0	0.37	0.43
20	NT	NT		
21	NT	NT		
23	NT	NT		
24	NT	NT		
25	NT	NT		

Statistics

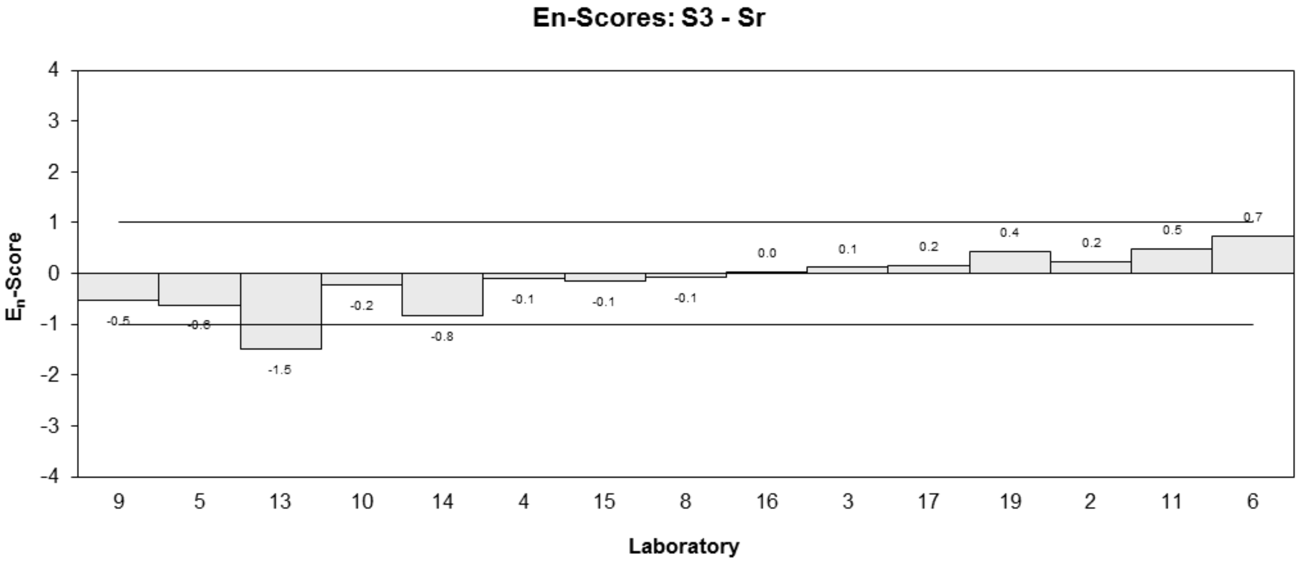
Assigned Value	13.5	0.6
Spike	Not Spiked	
Homogeneity Value	13.1	1.6
Robust Average	13.5	0.6
Median	13.3	0.6
Mean	13.5	
N	15	
Max.	15	
Min.	12.3	
Robust SD	0.9	
Robust CV	6.7%	



Results: S3 - Sr
Assigned Value = 13.5 ± 0.6 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
9 5 13 10 14 4 15 8 16 3 17 19 2 11 6 18 1 12 20 21 23 24 25 7 Md R.A. H.V.
Laboratory



z-Scores: S3 - Sr
Target Standard Deviation 1.4 mg/kg (Equivalent to 10% CV)
z-Score
-0.9 -0.7 -0.7 -0.5 -0.4 -0.4 -0.2 -0.1 0.1 0.3 0.4 0.4 0.4 1.1 1.1
9 5 13 10 14 4 15 8 16 3 17 19 2 11 6
Laboratory



En-Scores: S3 - Sr
En-Score
-0.5 -0.6 -1.5 -0.2 -0.8 -0.1 -0.1 -0.1 0.0 0.1 0.2 0.4 0.2 0.5 0.7
9 5 13 10 14 4 15 8 16 3 17 19 2 11 6
Laboratory

Figure 52

Table 63

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	Sulphate
Units	mg/kg

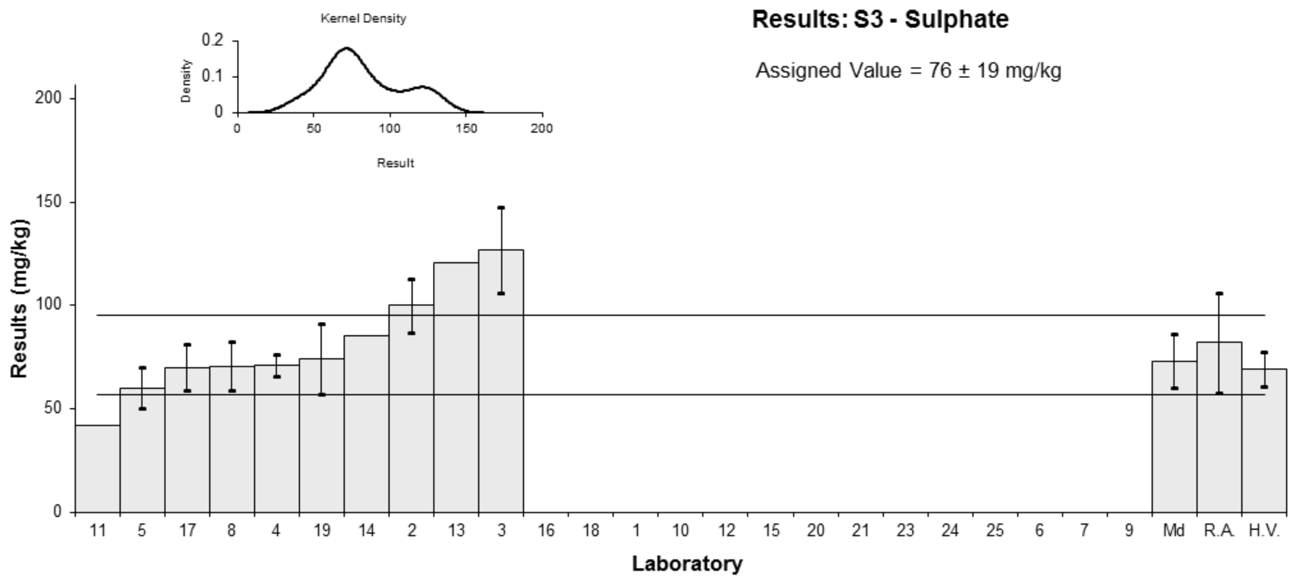
Participant Results

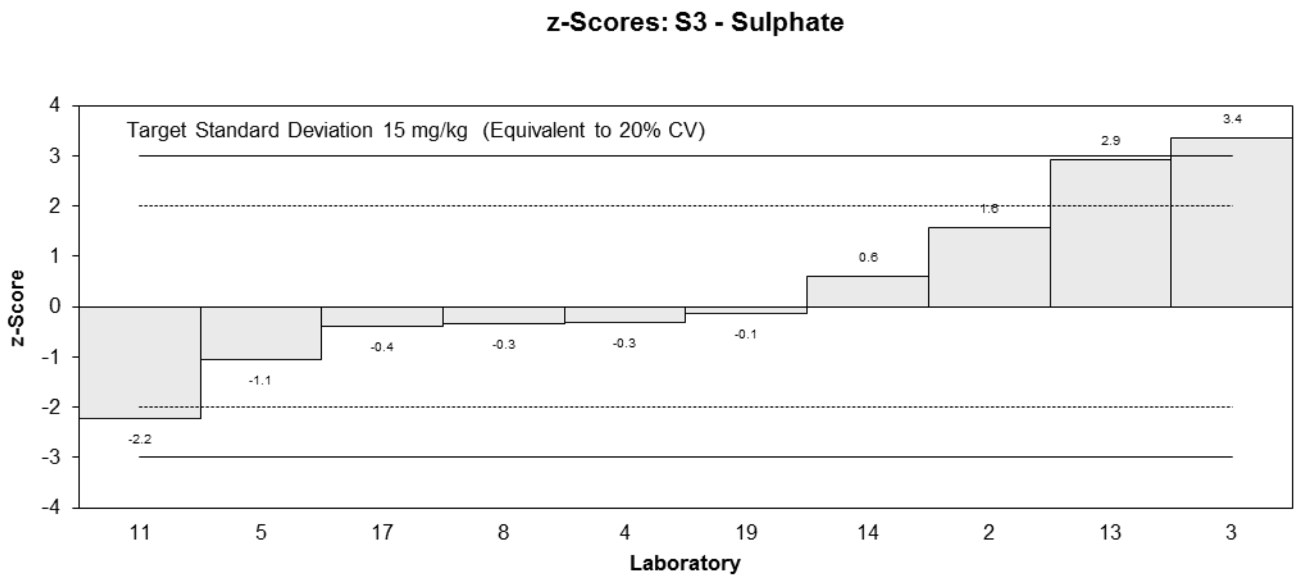
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	100	13	1.58	1.04
3	127	20.70	3.36	1.82
4	71	5	-0.33	-0.25
5	60	10	-1.05	-0.75
6	NT	NT		
7	NT	NT		
8	70.7	12.0	-0.35	-0.24
9	NT	NT		
10	NT	NT		
11	42	NR	-2.24	-1.79
12	NT	NT		
13	120.5	NR	2.93	2.34
14	85.1	NR	0.60	0.48
15	NT	NT		
16	<100	NR		
17	70	11	-0.39	-0.27
18	NR	NR		
19	74	17	-0.13	-0.08
20	NT	NT		
21	NT	NT		
23	NT	NT		
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value*	76	19
Spike	Not Spiked	
Homogeneity Value	69	8
Robust Average	82	24
Median	73	13
Mean	82	
N	10	
Max.	127	
Min.	42	
Robust SD	23	
Robust CV	28%	

*Robust Average excluding Laboratory 3.


Results: S3 - Sulphate
Assigned Value = 76 ± 19 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
11
5
17
8
4
19
14
2
13
3
16
18
1
10
12
15
20
21
23
24
25
6
7
9
Md
R.A.
H.V.


z-Scores: S3 - Sulphate
Target Standard Deviation 15 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
-2.2
-1.1
-0.4
-0.3
-0.3
-0.1
0.6
1.6
2.9
3.4
11
5
17
8
4
19
14
2
13
3

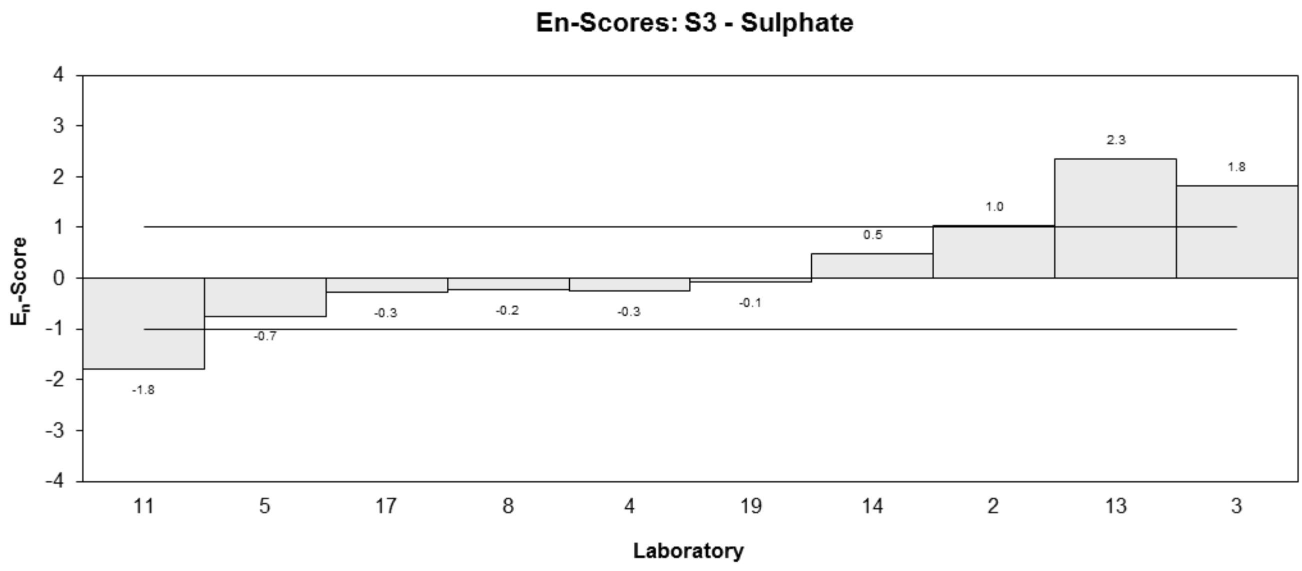

En-Scores: S3 - Sulphate
E_n-Score
Laboratory
-1.8
-0.7
-0.3
-0.2
-0.3
-0.1
0.5
1.0
2.3
1.8
11
5
17
8
4
19
14
2
13
3

Figure 53

Table 64

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	TKN
Units	mg/kg

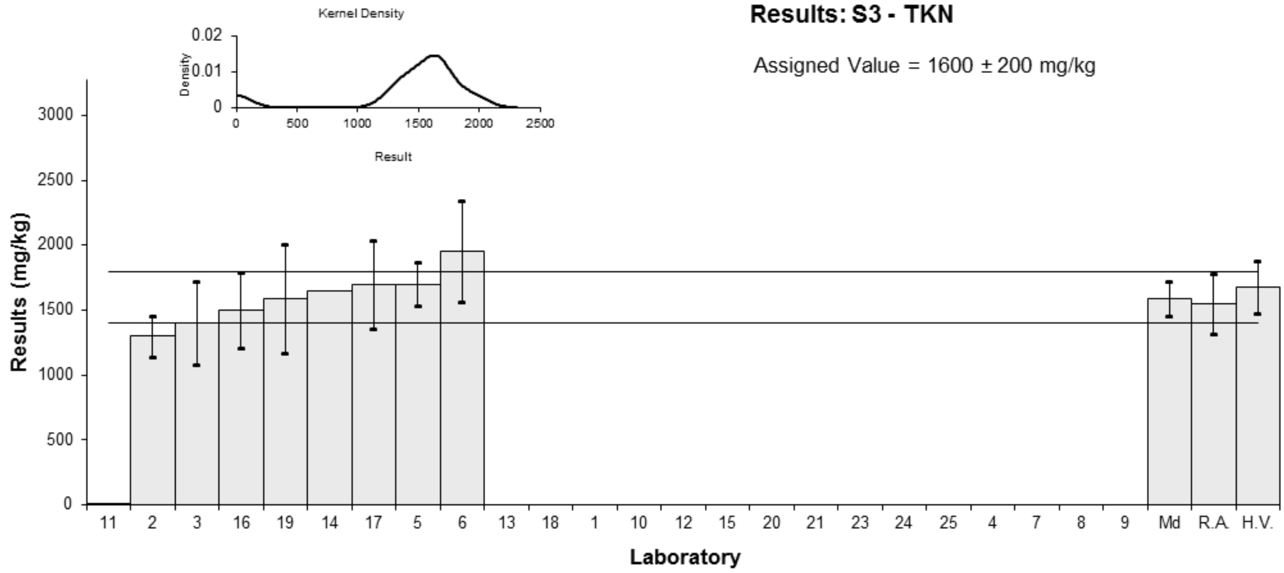
Participant Results

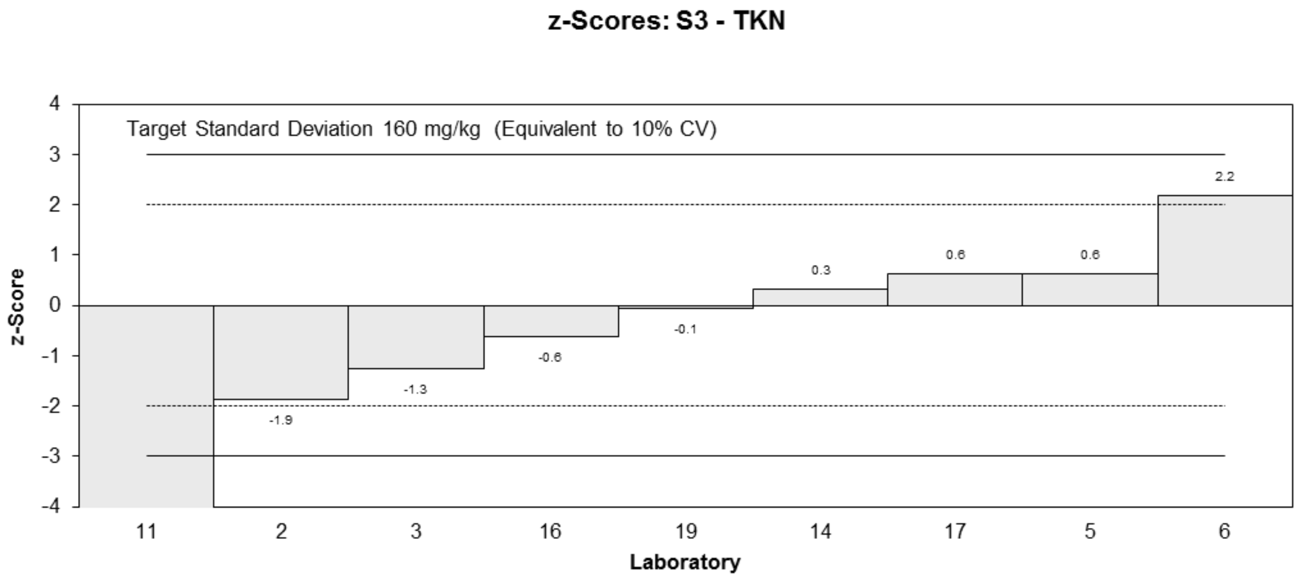
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	1300	160	-1.88	-1.17
3	1400	322	-1.25	-0.53
4	NT	NT		
5	1700	170	0.62	0.38
6	1950	390	2.19	0.80
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	0.15	NR	-10.00	-8.00
12	NT	NT		
13	NR	NR		
14	1650	NR	0.31	0.25
15	NT	NT		
16	1500	290	-0.62	-0.28
17	1700	340	0.62	0.25
18	NR	NR		
19	1590	420	-0.06	-0.02
20	NT	NT		
21	NT	NT		
23	NT	NT		
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value*	1600	200
Spike	Not Spiked	
Homogeneity Value	1680	200
Robust Average	1550	230
Median	1590	130
Mean	1421	
N	9	
Max.	1950	
Min.	0.15	
Robust SD	220	
Robust CV	14%	

*Robust Average excluding Laboratory 11.


Results: S3 - TKN
Assigned Value = 1600 ± 200 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
11 2 3 16 19 14 17 5 6 13 18 1 10 12 15 20 21 23 24 25 4 7 8 9 Md R.A. H.V.
Laboratory


z-Scores: S3 - TKN
Target Standard Deviation 160 mg/kg (Equivalent to 10% CV)
z-Score
-1.9
-1.3
-0.6
-0.1
0.3
0.6
0.6
2.2
11 2 3 16 19 14 17 5 6
Laboratory

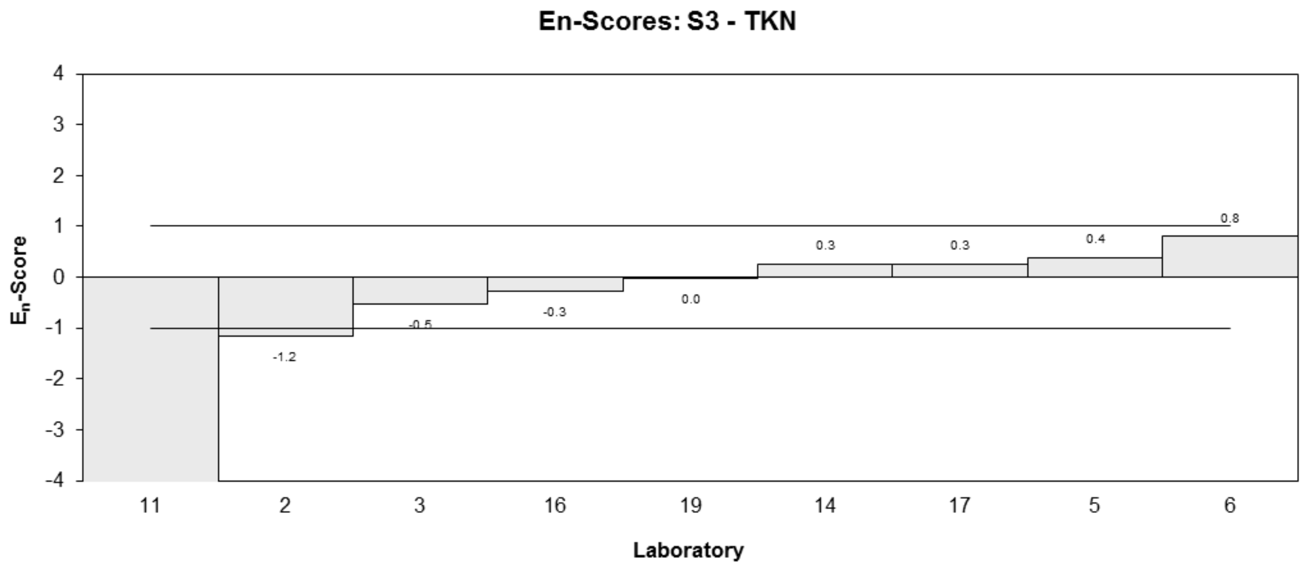

En-Scores: S3 - TKN
En-Score
-1.2
-0.5
-0.3
0.0
0.3
0.3
0.4
0.8
11 2 3 16 19 14 17 5 6
Laboratory

Figure 54

7 DISCUSSION OF RESULTS

7.1 Assigned Value and Traceability

Assigned Values of the inorganic analytes in the study samples S1, S2 and S3 were the robust average of participants' results. The robust averages used as assigned values and their associated expanded uncertainties were calculated using the procedure described in 'Statistical methods for use in proficiency testing by interlaboratory comparisons, ISO13528:2015(E)'. Results less than 50% and more than 150% of the robust average were investigated and then removed before calculation of the assigned value.[8] Appendix 2 sets out the calculation for the robust average of As in Sample S2 and its associated uncertainty.

No assigned value was set for Cs, Ga, Rb or Se in S1 because the reported results for these elements were too few. No assigned value was also set for Sb in S2 because the results were too variable.

Traceability The assigned value is not traceable to any external reference; it is traceable to the consensus of participants' results deriving from a variety of measurement methods and (presumably) a variety of calibrators. So although expressed in SI units, the metrological traceability of the assigned values has not been established.

7.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded measurement uncertainty associated with their results. Of 748 numerical results, 615 (82%) were reported with an expanded measurement uncertainty. The participants used a wide variety of procedures to estimate the expanded measurement uncertainty. These are presented in Tables 9 and 10.

Approaches to estimating measurement uncertainty include: standard deviation of replicate analysis, Horwitz formula, professional judgement, bottom up approach, top down approach using precision and estimates of method and laboratory bias and top down approach using only the reproducibility from inter-laboratory comparisons studies.[11–17]

Proficiency tests allow a check of the reasonableness of uncertainty estimates. Results and the expanded MU are presented in the bar charts for each analyte (Figure 2 to 54). In this study, the reported expanded measurement uncertainty has been over-estimated in some cases (e.g. Lab 23 for Ba in S1 or Lab 25 for Be in S1) or under-estimated (e.g. Lab 7 for As, Be, Ca, Co, Cr or Ni in S1). As a simple rule of thumb, when the uncertainty estimate is either smaller than the assigned uncertainty value or larger than the uncertainty of the assigned value plus twice the target standard deviation then this should be reviewed as suspect.

Overestimation of the precision and/or laboratory or method bias is the most common error seen in the laboratories' estimated uncertainty budgets. According to NATA According to General Accreditation Guidance, Estimating and reporting measurement uncertainty of chemical test results[14] and to NORDTEST TR 537,[12] the most common sources used to estimate the precision component are from:

- Stable control samples that cover the whole analytical process (including extraction) and **have a matrix similar** to the samples; **or**
- Stable control samples **and** duplicate analyses if control samples do not cover whole analytical process (e.g. the control sample is a synthetic sample - we have to take into consideration uncertainties arising from different matrices); **or**
- When control samples are not stable, from analysis of natural duplicates (gives within-day variation for sampling and measurement) and long-term uncertainty component from the variation in the instrument calibration ; **or**

- Replicate analyses performed on the same sample at different times to obtain estimates of intermediate precision; within-batch replication provides estimates of repeatability only.

The most common sources for estimating the method bias component for the measurement uncertainty calculation are from:

- Certified reference material recoveries; **or**
- Participation in PT studies (laboratory bias from at least 6 successful PT studies) ; **or**
- From sample spike recoveries.

When a laboratory has successfully participated in at least 6 proficiency testing studies, the standard deviation from proficiency testing studies alone, can also be used to estimate the uncertainty of their measurement results.[12, 14]

Laboratories 2, 6, 8, 21, 24 and 25 attached estimates of the expanded measurement uncertainty to results reported as less than their limit of detection. An estimate of uncertainty expressed as a value cannot be attached to a result expressed as a range.[11]

Laboratory 25 reported an estimate of expanded uncertainty for their Se and Sn measurement result larger than the result itself.

In some cases the results were reported with an inappropriate number of significant figures. The recommended format is to write uncertainty to no more than two significant figures and then to write the result with the corresponding number of decimal places. For example, instead of 64.3 ± 12.86 mg/kg, it is better to report 64 ± 13 mg/kg or instead of 9910 ± 1486.50 mg/kg, it is better to report 9910 ± 1500 mg/kg.[11]

7.3 E_n-score

E_n-score should be interpreted only in conjunction with z-scores. The E_n-score indicates how closely a result agrees with the assigned value taking into account the respective uncertainties. An unsatisfactory E_n score for an analyte can either be caused by an inappropriate measurement, an inappropriate estimation of measurement uncertainty, or both.

The dispersal of participants' E_n-scores is graphically presented in Figure 55. 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.

Of 721 results for which E_n-scores were calculated, 538 (75%) returned a satisfactory score of $|E_n| \leq 1$ indicating agreement of the participants' results with the assigned values within their respective expanded measurement uncertainties.

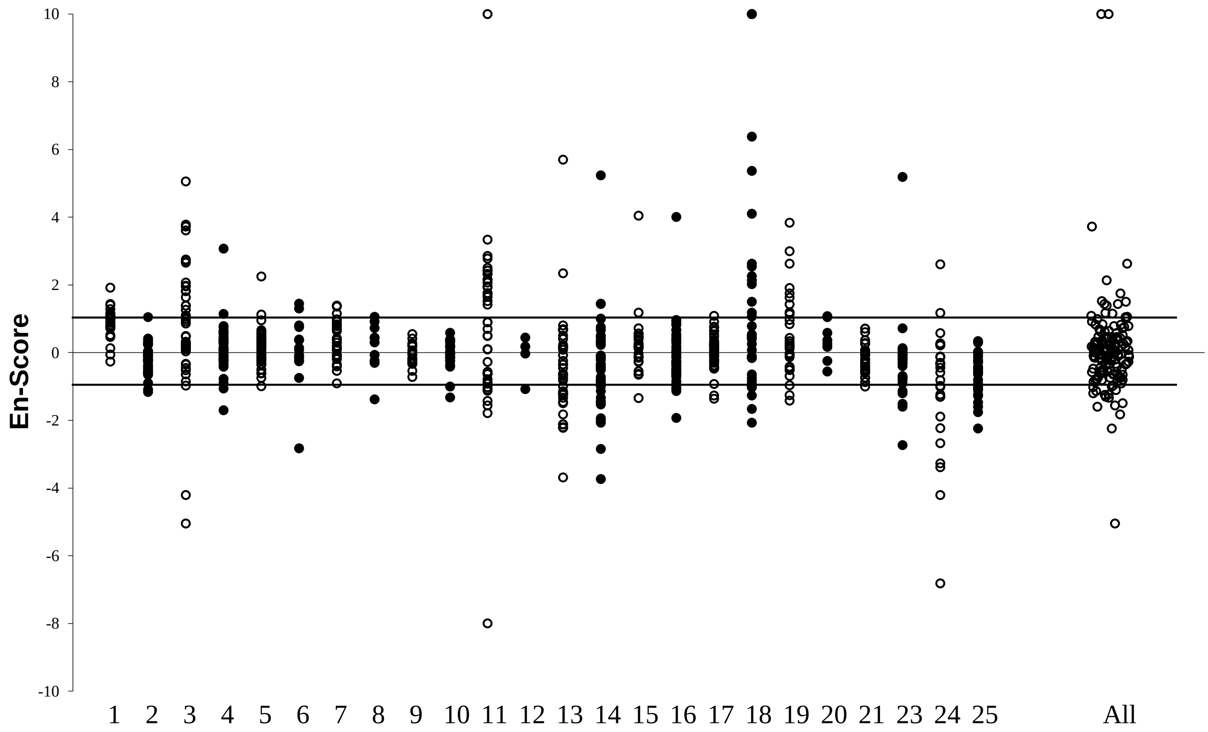
7.4 z-Score

The z-score compares the participant's deviation from the assigned value with the target standard deviation set for proficiency assessment.

The target standard deviation defines satisfactory performance in a proficiency test. Target standard deviations equivalent to 10% to 20% PCV were used to calculate z-scores. Unlike the standard deviation based on between laboratories CV, setting the target standard deviation as a realistic, set value enables z-scores to be used as fixed reference value points for assessment of laboratory performance, independent of group performance.

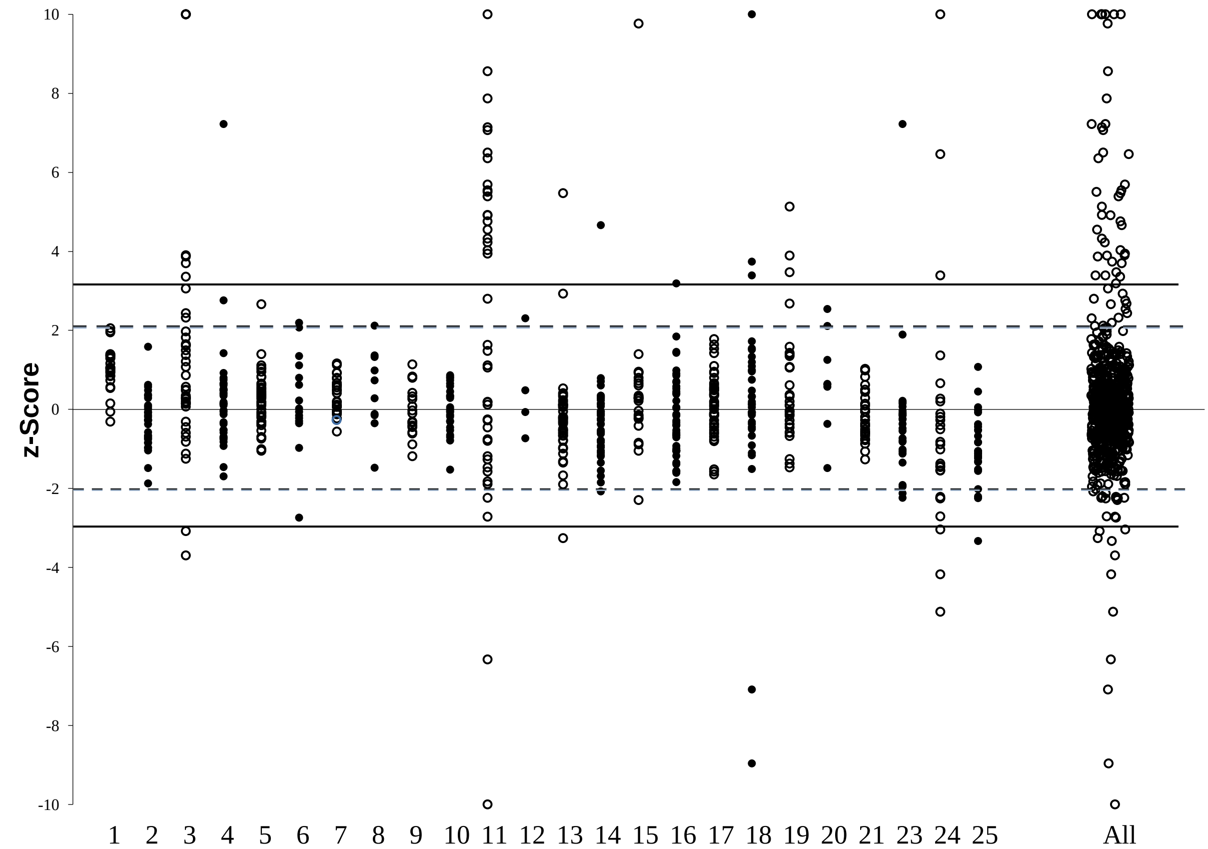
The between laboratory coefficient of variation predicted by the Thompson equation[9] and the participants' coefficient of variation resulted in this study are presented for comparison in Table 65.

The dispersal of participants' z-scores is presented in Figure 56 (by laboratory code) and in Figure 57 (by test). Of 721 results for which z-scores were calculated, 639 (89%) returned a satisfactory score of |z| ≤ 2 and 28 (4%) were questionable of 2 < |z| <3. Participants with multiple z-scores larger than 2 or smaller than -2 should check for laboratory bias.



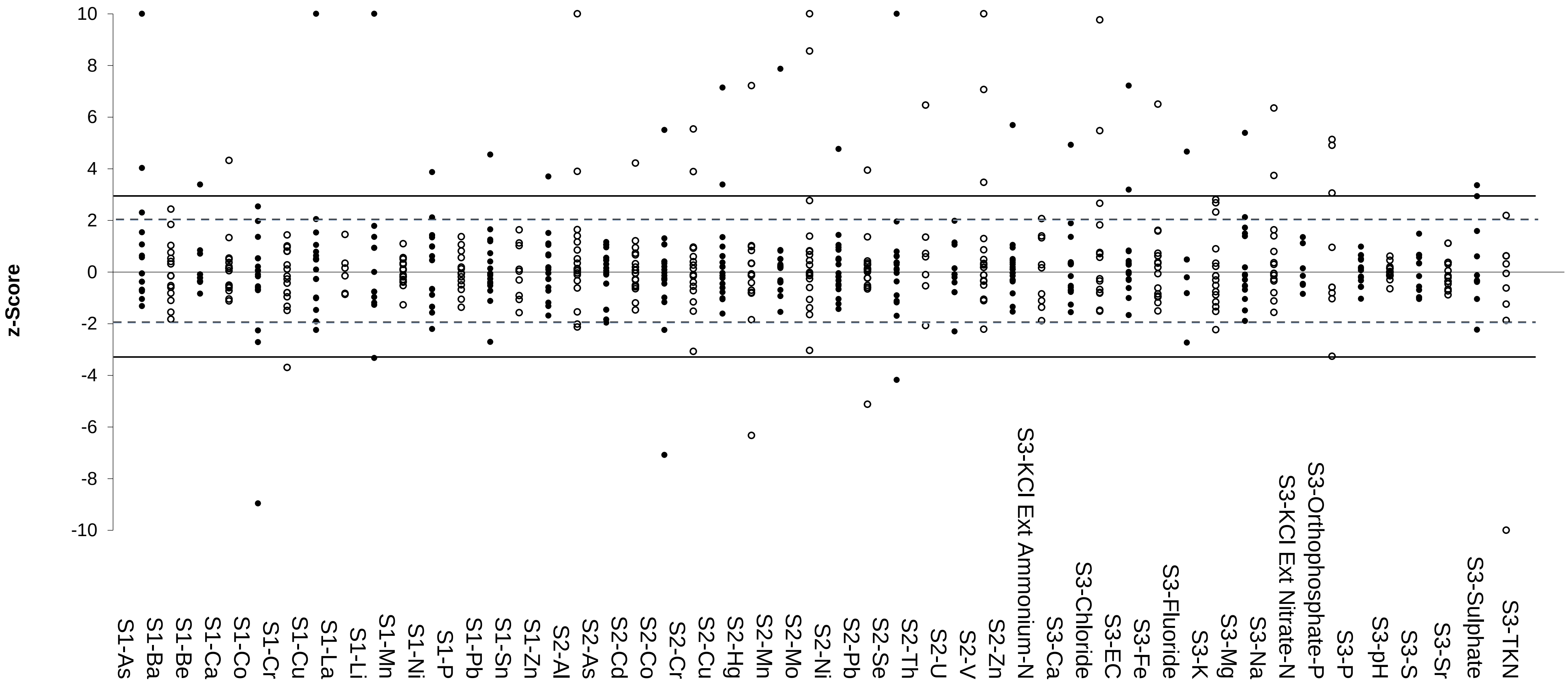
Scores of >10 or < -10 have been plotted as 10 or -10.

Figure 55 E_n-Score Dispersal by Laboratory



Scores of >10 or <-10 have been plotted as 10 or -10.

Figure 56 z-Score Dispersal by Laboratory



Scores of >10 or <-10 have been plotted as 10 or -10.

Figure 57 z-Score Dispersal by Test

Table 65 Between Laboratory CV of this Study, Thompson CV and Set Target SD

Sample	Test	Assigned value (mg/kg)	Between Laboratories CV	Thompson/ Horwitz CV	Target SD (as CV)
S1	As	2.12	15%	14%	15%
S1	Ba	89.3	12%	8%	10%
S1	Be	0.747	6.7%	17%	10%
S1	Ca	572	8%	6%	10%
S1	Co	6.46	13%	12%	10%
S1	Cr	12.5	15%	11%	15%
S1	Cs	Not Set	22%	Not Set	Not set
S1	Cu	6.68	13%	12%	10%
S1	Ga	Not Set	47%	Not Set	Not Set
S1	La	10.3	19%	11%	20%
S1	Li	2.36	26%	14%	20%
S1	Mn	377	4.5%	7%	10%
S1	Ni	5.77	22%	12%	15%
S1	P	161	6.2%	7%	10%
S1	Pb	10.1	9.8%	11%	15%
S1	Rb	Not Set	22%	Not Set	Not Set
S1	Se	Not Set	102%	Not Set	Not Set
S1	Sn	0.98	25%	16%	20%
S1	Zn	14.6	15%	11%	15%
S2	Al	8430	17%	4%	15%
S2	As	19.9	11%	10%	10%
S2	Cd	8.44	8.2%	12%	10%
S2	Co	9.03	8.4%	11%	10%
S2	Cr	20.2	14%	10%	15%
S2	Cu	24.5	8.9%	10%	10%
S2	Hg	8.71	7.7%	12%	10%
S2	Mn	343	6.1%	7%	10%
S2	Mo	8.62	11%	12%	10%
S2	Ni	14.0	13%	11%	15%
S2	Pb	28.7	3.5%	10%	10%
S2	Sb	Not Set	24%	12%	20%
S2	Se	8.05	14%	12%	15%
S2	Th	4.8	25%	13%	20%
S2	U	0.868	11%	16%	10%
S2	V	33.4	8.7%	9%	10%
S2	Zn	73.3	6.4%	8%	10%
S3	Ammonium-N	19.3	32%	10%	20%
S3	Ca	1830	11%	5%	10%
S3	Chloride	35.9	24%	9%	20%
S3	EC	288 (µS/cm)	6.9%	7%	10%
S3	Fe	3860	11%	5%	10%
S3	Fluoride	1.46	14%	15%	20%
S3	K	542	26%	6%	15%
S3	Mg	505	13%	6%	10%
S3	Na	70.9	19%	8%	20%
S3	Nitrate-N	65	19%	9%	20%
S3	Orthophosphate-P	2.27	24%	14%	20%
S3	P	276	6.5%	7%	10%
S3	pH	6.60	1.5%	12%	10%

Table 65 Between Laboratory CV of this study, Thompson CV and Set Target SD (continued)

Sample	Test	Assigned value (mg/kg)	Between Laboratories CV	Thompson/ Horwitz CV	Target SD (as CV)
S3	S	**244/245**	9%	7%	10%
S3	Sr	13.5	6.7%	11%	10%
S3	Sulphate	76	28%	8%	20%
S3	TKN	1600	14%	5%	10%

7.5 Participants' Results and Analytical Methods for Acid Extractable Elements

A summary of participants' results and performance is presented in Tables 66 and 67 and in Figures 56 and 57.

Low level Co was the test that presented analytical difficulty to participating laboratories in both study samples.

All the unsatisfactory results reported by Laboratory 11 were higher than the assigned value except for one. This is an indication of method or laboratory bias. The results from this laboratory were not included in the analyses of the extraction methods and instrumental techniques employed by participants.

Extraction Methods

The request was for acid extractable elements; NMI PT studies of metals in soil focus on 'pseudo-total' analyses of elements in soil rather than on true total metal content because when an assessment of the anthropogenic impact of the metal content in a soil sample is made, aggressive digestion regimes can lead to misleading conclusions – since metals can be extracted from the fraction naturally present in the soil matrix.[5, 18-21] While an aggressive digestion regime can produce high, misleading results, weak digestion regimes (low digestion temperature, reduced digestion time, diluted acids and/or a low ratio of acid to sample size) may extract just a fraction of the contaminants from the soil. There is no standardisation of methods for acid extractable elements. In general methods are conventionally defined by procedures involving extractions: with aqua regia or with various amounts of HNO_3, HCl, in combination or alone and most of these methods produce comparable results.[22-24]

The method descriptions provided by participants are presented in Table 1 while the instrumental conditions are presented in Appendix 4.

Laboratory 19 used an extraction regime which involved dilute acids and a digestion temperature of 170 ºC; all the unsatisfactory results reported by them were higher than the assigned value (Figure 56)

Laboratories 24 and 25 extracted their sample at 95 ºC for 30 min only; the majority of the results reported by them were lower than the assigned value (Figure 56).

Table 66 Summary of Participants' Results and Performance for Acid Extractable Elements in Sample S1

Lab Code	S1-As mg/kg	S1-Ba mg/kg	S1-Be mg/kg	S1-Ca mg/kg	S1-Co mg/kg	S1-Cr mg/kg	S1-Cs mg/kg	S1-Cu mg/kg	S1-Ga mg/kg	S1-La mg/kg
H.V.	2.02	93	0.70	580	6.35	12.3	0.70	6.93	2.93	10.8
A.V.	2.12	89.3	0.747	572	6.46	12.5	Not Set	6.68	Not Set	10.3
1	2.10	98.50	0.81	603.00	7.34	14.40	0.64	8.05	NT	10.60
2	2.0	88	0.74	530	6.5	12	NT	6.5	5	11
3	1.90	110.99	<5	508	7.73	5.57	NT	7.00	NT	NT
4	<3	96	<1	580	6	14	NT	7	NT	NT
5	2.32	92.1	0.74	593	6.35	12.2	NT	7.38	NT	NT
6	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
7	2.1	93	0.73	540	6.5	14	NT	7.2	NT	NT
8	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
9	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
10	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
11	3.4	73	<5	819	4.7	11	NT	5.7	NT	NT
12	2.85	NT	NT	NT	NT	NT	NT	7	NT	NT
13	1.7	84.6	0.73	537	6.8	12.7	NT	6.02	NT	NT
14	2.0	79.5	0.8	580	6.1	10.7	0.4	6.5	2.3	8.6
15	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
16	1.787	105.7	0.7182	511.8	6.598	15.18	NR	7.029	NR	13.29
17	1.9	94	0.72	600	6.8	14	0.62	7.7	2.9	10
18	2.61	NR	NR	648	0.67	14.3	NR	15.0	NR	NR
19	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
20	2.3	NT	<1	NT	8.1	9.7	NT	7.1	NT	NT
21	1.88	84	0.72	580	6.07	11.7	0.556	6.74	NT	8.5
23	<3	88	<1	584	6.4	11	NT	5.4	NT	NT
24	8	82	1	543	5	13	NR	6	NR	NR
25	2.46	75.4	0.684	575	6.02	10	NT	5.18	NT	NT

Shaded cells are results which returned a questionable or unsatisfactory z-score. A.V. = Assigned Value, H.V. = Homogeneity Value

Table 66 Summary of Participants' Results and Performance for Acid Extractable Elements in Sample S1 (continued)

Lab Code	S1-Li mg/kg	S1-Mn mg/kg	S1-Ni mg/kg	S1-P mg/kg	S1-Pb mg/kg	S1-Rb mg/kg	S1-Se mg/kg	S1-Sn mg/kg	S1-Zn mg/kg
H.V.	2.60	362	6.08	155	9.5	7.9	0.26	0.86	15.7
A.V.	2.36	377	5.77	161	10.1	Not Set	Not Set	0.98	14.6
1	2.80	398.00	6.93	178.00	11.20	7.17	NR	0.92	16.90
2	1.9	370	5.2	150	10	NT	<1	1.0	14
3	22.6	396	9.12	183	12.6	NT	<5	<5	22.7
4	2	390	7	160	9	NT	<4	<2	16
5	2.36	362	6.16	174	10.3	NT	0.16	1.18	14.8
6	NT	NT	NT	NT	NT	NT	NT	NT	NT
7	2.8	380	6.3	160	10	NT	0.15	1.2	15
8	NT	NT	NT	NT	NT	NT	NT	NT	NT
9	NT	NT	NT	NT	NT	NT	NT	NT	NT
10	NT	NT	NT	NT	NT	NT	NT	NT	NT
11	2	329	4.4	164	17	NT	<5	1.3	12
12	NT	NT	NT	NT	10	NT	NT	NT	13
13	<5	381	5.2	153	8.4	NT	<1	<5	14.5
14	1.8	380	4.6	160	9.3	4.9	<1	0.8	10.9
15	NT	NT	NT	NT	NT	NT	NT	NT	NT
16	NR	365.9	6.614	155.5	10.72	NR	0.1734	0.9852	16.09
17	3.2	380	7.0	170	9.9	NT	<0.5	0.67	17
18	NR	418	6.63	163	11.9	NR	0.87	NR	17.9
19	NT	NT	NT	NT	NT	NT	NT	NT	NT
20	NT	363	7.6	NT	12	NT	<1	<10	16
21	1.76	388	5.19	158	9.5	5.6	<2	0.77	13.3
23	NT	373	4.6	NT	9.7	NT	<3	<2	15
24	3	370	5	139	6	NR	1	NR	14
25	0.786	357	3.86	144	9.43	NT	<3	<3	11.7

Shaded cells are results which returned a questionable or unsatisfactory z-score. A.V. = Assigned Value, H.V. = Homogeneity Value

Table 67 Summary of Participants' Results and Performance for Acid Extractable Elements in Samples S2 and S3

Lab Code	S2-Al mg/kg	S2-As mg/kg	S2-Cd mg/kg	S2-Co mg/kg	S2-Cr mg/kg	S2-Cu mg/kg	S2-Hg mg/kg	S2-Mn mg/kg	S2-Mo mg/kg	S2-Ni mg/kg	S2-Pb mg/kg	S2-Sb mg/kg	S2-Se mg/kg
H.V.	7950	19.0	7.9	8.9	22.0	25.3	8.9	340	9.4	15.3	29.3	5.4	9.0
A.V.	8430	19.9	8.44	9.03	20.2	24.5	8.71	343	8.62	14.0	28.7	Not Set	8.05
1	10200.00	22.20	9.23	10.20	23.10	27.80	9.56	372.00	9.82	16.20	32.60	7.60	10.40
2	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
3	13362	20.2	9.46	10.0	10.86	23.4	9.01	352	71.3	15.8	29.5	19.8	59.99
4	8600	17	8	9	23	26	15	360	11	15	27	NR	6
5	9075	21.0	8.18	9.33	21.4	25.4	9.43	349	8.85	15.1	29.7	8.04	8.48
6	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
7	9900	21	8.2	9.1	23	26	8.6	350	9.2	15	28	NT	8.2
8	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
9	8000	19.7	7.95	8.62	21.0	23.0	9.00	329	9.31	13.3	29.9	9.07	8.15
10	8382.92	20.79	9.06	8.76	21.99	24.15	8.02	318.97	9.33	13.63	28.85	NT	8.40
11	22500	22	12	14	37	42	3.2	613	16	24	40	22	<2
12	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
13	8575	20	8.5	9.4	20.6	22.6	9	351	8.4	13.6	27	<10	8.5
14	6475	20.5	8.4	8.8	16.7	23.9	7.1	351	8.5	11.8	29.5	2.8	9
15	8857	21.8	8.71	9.22	23.0	24.4	8.34	353	9.31	14.6	28.0	8.71	8.77
16	9507	16.23	7.890	8.139	19.72	20.55	8.090	311.1	7.421	12.89	26.80	3.431	6.624
17	10500	19	8.0	9.4	23	25	8.6	360	7.2	16	29	NT	8.5
18	8495	19.0	8.60	2.63	19.9	32.8	NR	332	8.55	12.6	29.1	NR	6.95
19	NT	22	7.2	10	32	25	9.0	330	8.1	17	29	5.7	8.0
20	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
21	7640	19.8	8.4	8.79	18.5	26.9	9.6	371	9.0	13.9	28.7	5.0	8.8
23	5740	16	8.5	8.9	18	22	8	348	7.7	13	28	3	7.6
24	8293	17	9	7	19	25	NR	290	6	11	14	<1	3
25	5870	20.8	7.43	7.97	15.6	21.9	8.64	330	8.6	11.4	27.2	6.64	6.70

Shaded cells are results which returned a questionable or unsatisfactory z-score. A.V. = Assigned Value, H.V. = Homogeneity Value

Table 67 Summary of Participants' Results and Performance for Acid Extractable Elements in Samples S2 and S3 (continued)

Lab Code	S2-Th mg/kg	S2-U mg/kg	S2-V mg/kg	S2-Zn mg/kg	S3-Ca mg/kg	S3-Fe mg/kg	S3-K mg/kg	S3-Mg mg/kg	S3-Na mg/kg	S3-P mg/kg	S3-S mg/kg	S3-Sr mg/kg
H.V.	4.6	0.893	34.0	66	1730	3750	522	493	59.7	264	248	13.1
A.V.	4.8	0.868	33.4	73.3	1830	3860	542	505	70.9	276	244	13.5
1	NT	1.04	37.70	81.00	NT	NT	NT	NT	NT	NT	NT	NT
2	NT	NT	NT	NT	1700	4100	480	430	75	260	220	14
3	NT	<5	358	75.4	1720	4480	731	580	94.0	278	258	13.9
4	NT	NT	34	74	1900	3500	500	470	69	290	260	13
5	4.28	0.86	34.5	76.5	1700	3930	517	514	66	294	227	12.5
6	NT	NT	NT	NT	1800	4100	560	490	70	270	220	15
7	NT	0.86	35	73	NT	NT	NT	NT	NT	NT	NT	NT
8	NT	NT	NT	NT	2078	4142	NT	612	NT	303	NT	13.3
9	NT	0.967	32.3	71.0	NT	3400	NT	NT	NT	NT	NT	12.3
10	NT	NT	36.26	71.03	1889.63	3833.13	417.50	478.08	82.11	294.04	252.29	12.88
11	NT	NT	57	115	2730	6370	770	777	161	281	280	15
12	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
13	NT	0.85	32.2	74.7	1731	3484	432	409	59.5	267	NT	12.6
14	2.8	0.8	33	72.2	1890	3620	570	500	70	280	230	13.0
15	5.49	0.668	34.3	80.2	1883	3530	470	452	90.7	271	260	13.2
16	5.359	0.8324	29.85	63.38	1544	4008	615.1	498.6	48.66	247.4	218.3	13.57
17	4.7	0.88	34	76	1690	3990	530	500	67	260	260	14
18	NR	NR	NR	70.6	1888	3277	448	592	124	289	252	NR
19	6.1	0.96	45	74	1600	4470	760	575	76	260	240	14
20	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
21	NT	0.80	31.7	77	NT	NT	NT	NT	NT	NT	NT	NT
23	NT	NT	33	73	2175	NT	360	477	55	NT	NT	NT
24	11	NR	26	62	NT	NT	NT	NT	NT	NT	NT	NT
25	NT	NT	29.7	67.2	NT	NT	NT	NT	NT	NT	NT	NT

Shaded cells are results which returned a questionable or unsatisfactory z-score. A.V. = Assigned Value, H.V. = Homogeneity Value

Table 68 Summary of Participants' Results and Performance for Sample S3

Lab Code	S3-Cl^- mg/kg	S3-F^- mg/kg	S3-PO_4^{3-}-P mg/kg	S3-SO_4^{2-} mg/kg	S3-pH	S3-EC µS/cm	S3-TKN mg/kg	S3-KCl Ext NH_4^+-N mg/kg	S3-KCl Ext NO_3^--N mg/kg
H.V.	28.1	1.67	1.74	69.1	6.50	292	1680	NA	NA
A.V.	35.9	1.46	2.27	76	6.60	288	1600	19.3	65
1	NT	NT	NT	NT	NT	NT	NT	NT	NT
2	40	1.6	1.8	100	6.6	280	1300	16	54
3	49.0	1.22	3.66	127	6.73	279	1400	19.9	66.8
4	30	NT	NT	71	6.7	300	NT	NT	NT
5	55	1.4	2.7	60	6.54	259	1700	24.7	79.4
6	33.4	0.66	< 10	NT	6.61	311	1950	27.3	82.5
7	NT	NT	NT	NT	NT	NT	NT	NT	NT
8	25.3	NT	<10	70.7	6.52	296	NT	24.4	NT
9	NT	NT	NT	NT	6.58	312	NT	NT	NT
10	NT	NT	NT	NT	6.17	288	NT	NT	NT
11	34	NT	4.5	42	6.68	280	0.15	12	59
12	NT	NT	NT	NT	NT	NT	NT	NT	NT
13	75.17	NR	0.788	120.5	6.4	240	NR	NR	NR
14	30.2	2.82	2.0	85.1	6.69	297.7	1650	20.4	NT
15	106	NT	NT	NT	6.51	296	NT	NT	NT
16	41	<100	2	<100	6.9	380	1500	NR	NR
17	25	1.6	1.9	70	6.6	270	1700	<30	63
18	41.3	NR	NR	NR	6.51	286	NR	15.0	58.5
19	31	NT	4.6	74	7.0	280	1590	14	59
20	NT	NT	NT	NT	NT	NT	NT	NT	NT
21	NT	NT	NT	NT	NT	NT	NT	NT	NT
23	NT	NT	NT	NT	6.5	496	NT	NT	NT
24	NT	NT	NT	NT	NT	NT	NT	NT	NT
25	NT	NT	NT	NT	NT	NT	NT	NT	NT

Shaded cells are results which returned a questionable or unsatisfactory z-score. A.V. = Assigned Value, H.V. = Homogeneity Value, NA = Not Available

Individual Element Commentary

Aluminium is an element which is strongly dependent on digestion regime. The between-laboratory coefficient of variation for Al in Sample S2 was high (17%), and larger than that predicted by Thomson (4%).[9] Short extraction time (30 minutes) may explain some of the low results reported for Al in S2.

Plots of Al participants' performance versus instrumental technique used are presented in Figure 58.

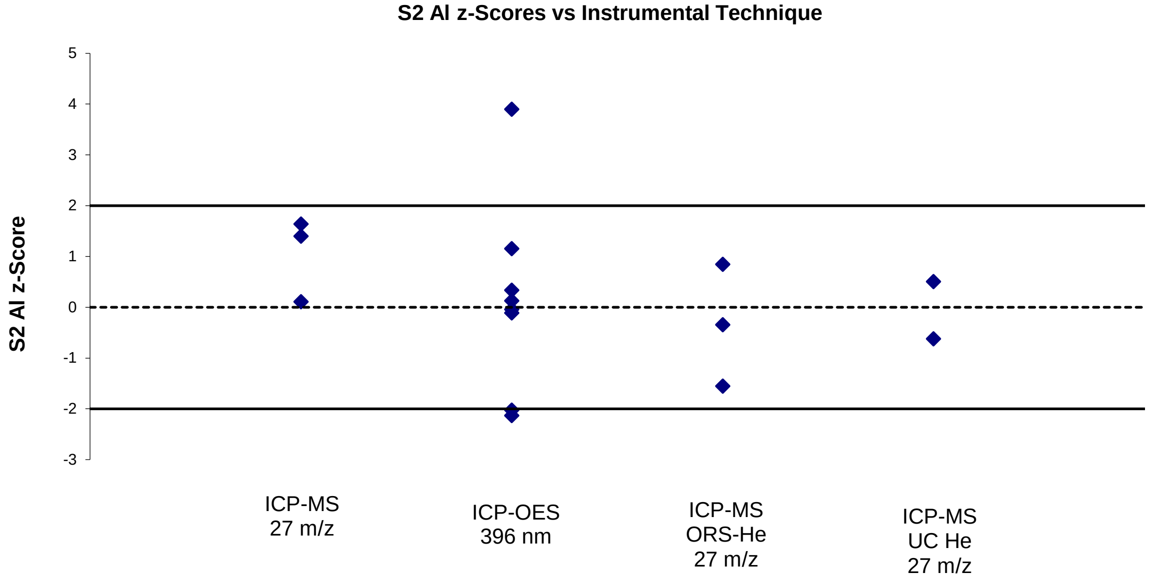
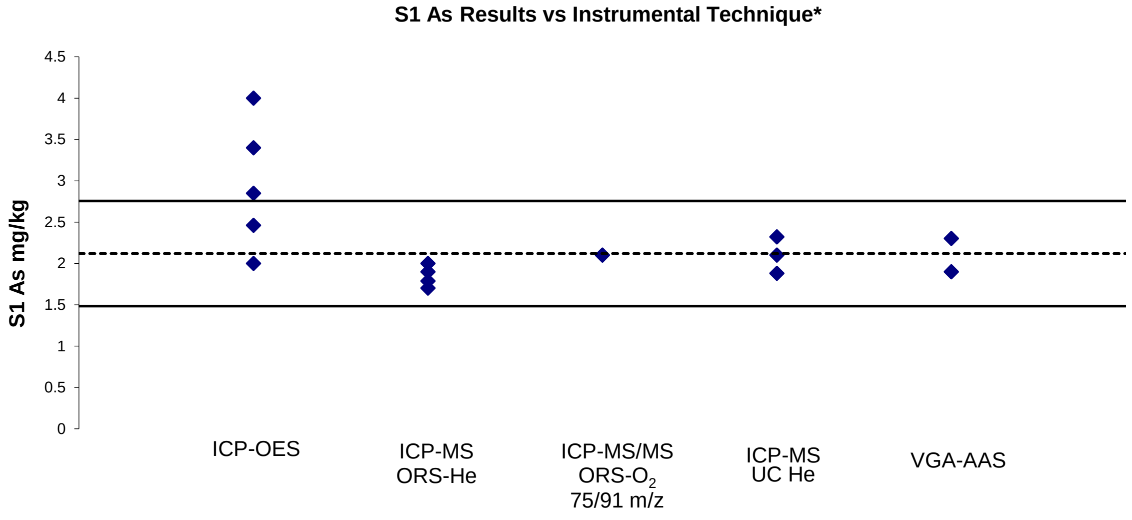


Figure 58 S2-Al z-Scores vs. Instrumental Technique

Arsenic measurements at a level of 2.12 mg/kg in soil Sample S1 presented difficulties to some participating laboratories. Of 18 participants, 16 reported results for As in S1 and 13 of them performed satisfactorily. Arsenic concentration in Sample S2 was 9.4 times higher than in Sample S1 (19.9 mg/kg) and all reported results returned satisfactory z-scores.

All unsatisfactory As results in S1 were from ICP-OES measurements (Figure 59).



*Result > 4 mg/kg has been plotted as 4 mg/kg.

Figure 59 S1-As Results vs. Instrumental Technique

Cobalt level in S1 and S2 was low, 6.46 mg/kg and 9.03 mg/kg respectively, and might have challenged participants who used ICP-OES. Plots of participants' performance versus instrumental technique are presented in Figure 60.

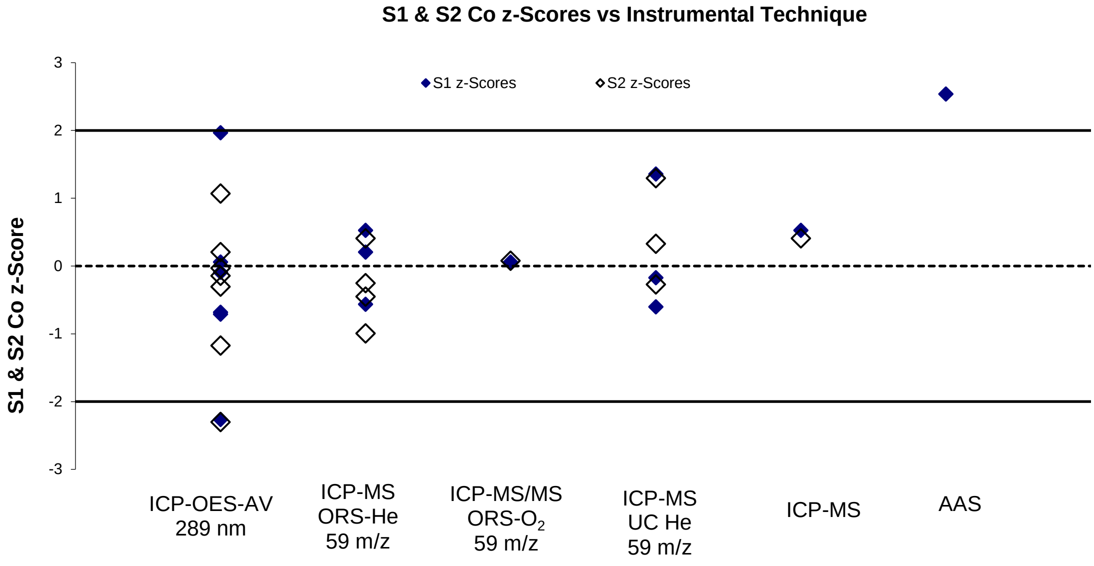


Figure 60 S1and S2 Co z-Scores vs. Instrumental Technique

Chromium and Nickel are two elements which are strongly dependent on extraction regime. The between-laboratory coefficient of variation for these elements was higher than that predicted by the Thomson value. Short digestion time (30 minutes) or high digestion temperature (170°C) might explain some of the low and high respectively unsatisfactory results.

Plots of participants' performance for Cr versus instrumental technique used are presented in Figure 61, while plots of participants' results for Ni versus instrumental technique are presented in Figure 62.

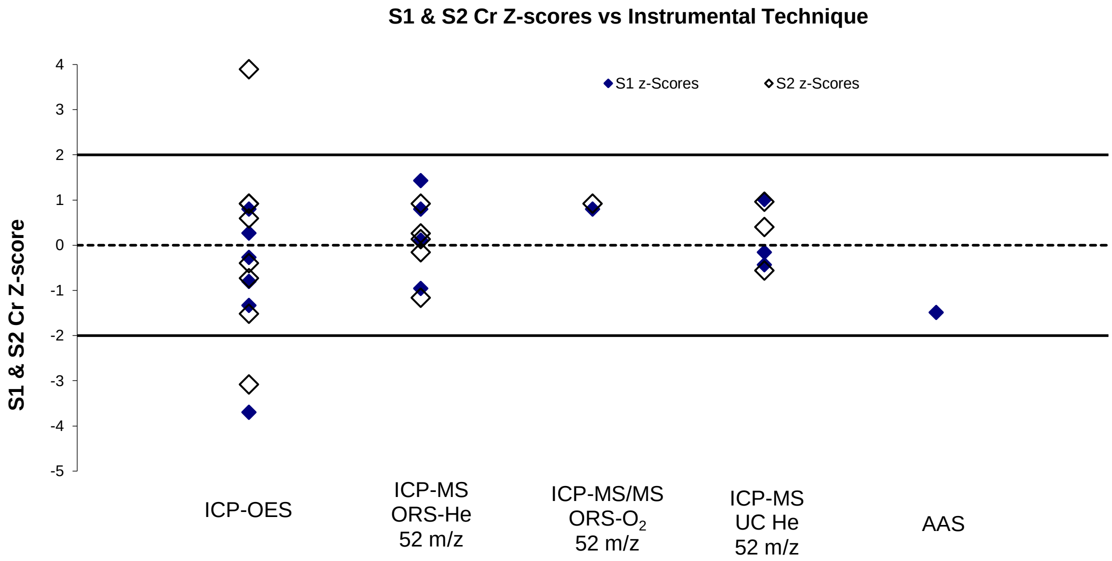


Figure 61 S1and S2 Cr z-Scores vs. Instrumental Technique

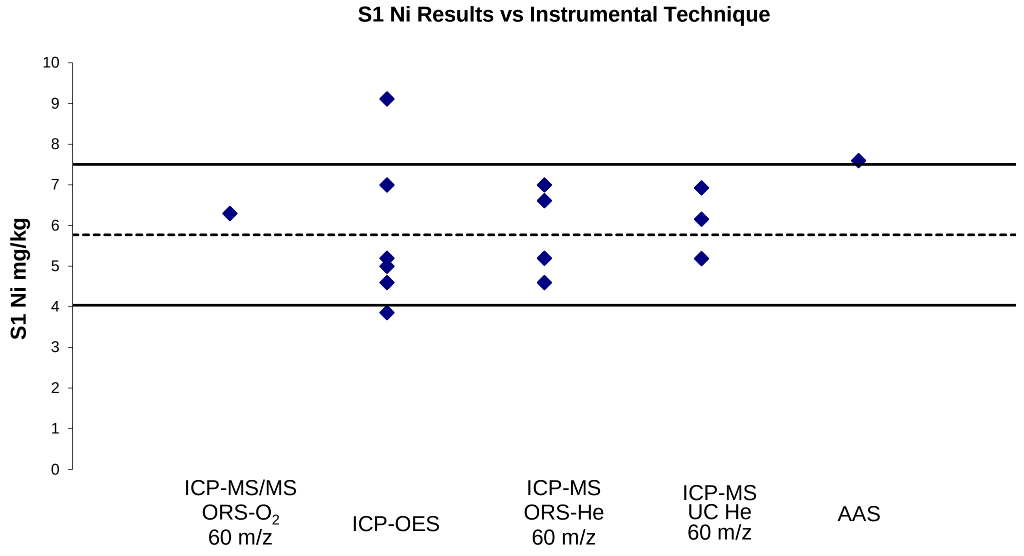


Figure 62 S1Ni Results vs. Instrumental Technique

Mercury Participants used a wide variety of instrumental techniques and all produced comparable results Figure 63.

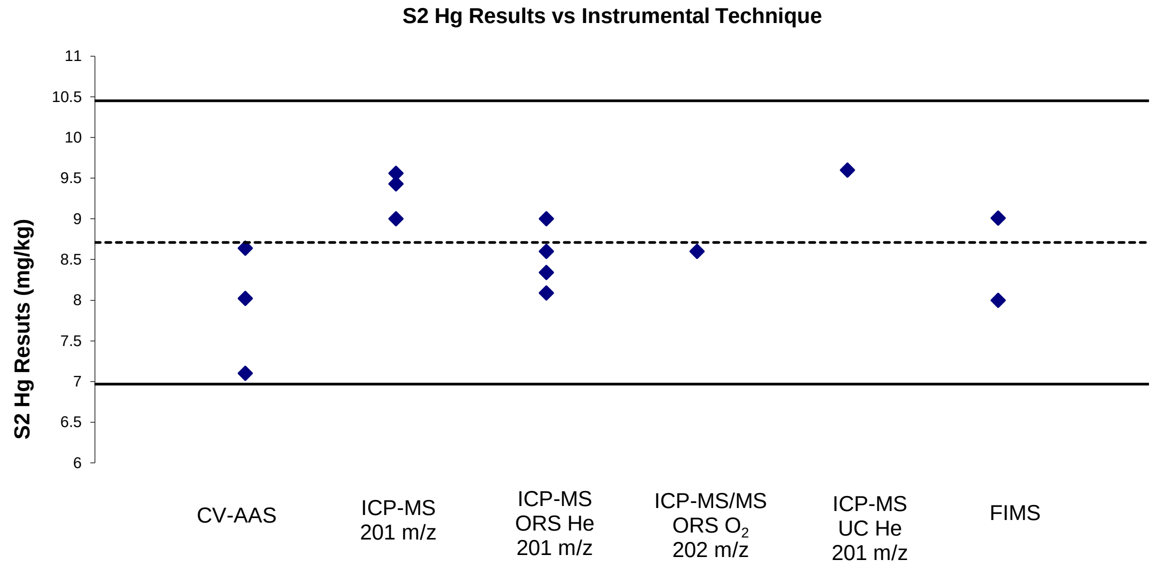
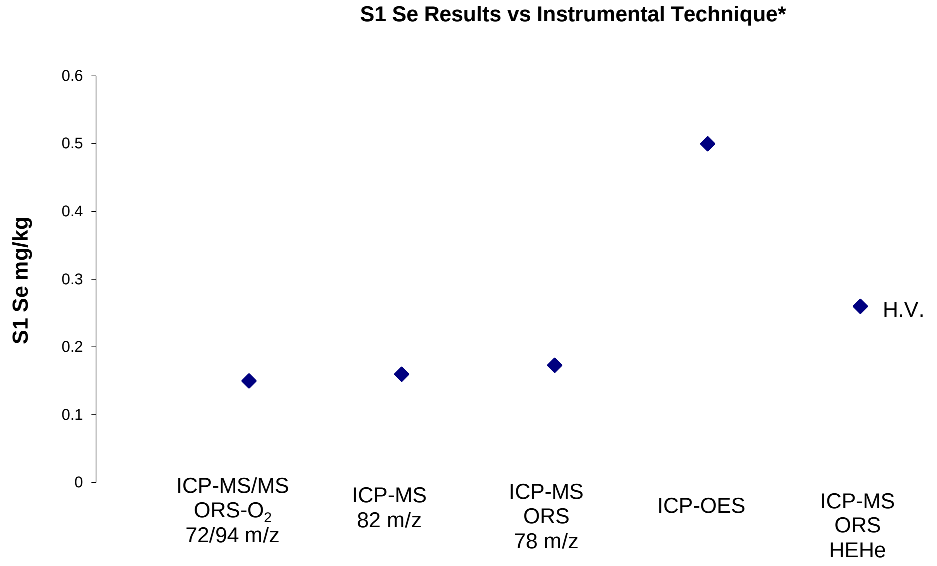


Figure 63 S2 Hg Results vs. Instrumental Technique

Selenium level in Sample S1 was below the reporting level of most participating laboratories. Only five results were reported for this test. Plots of participants results versus instrumental techniques used are presented in Figure 64.

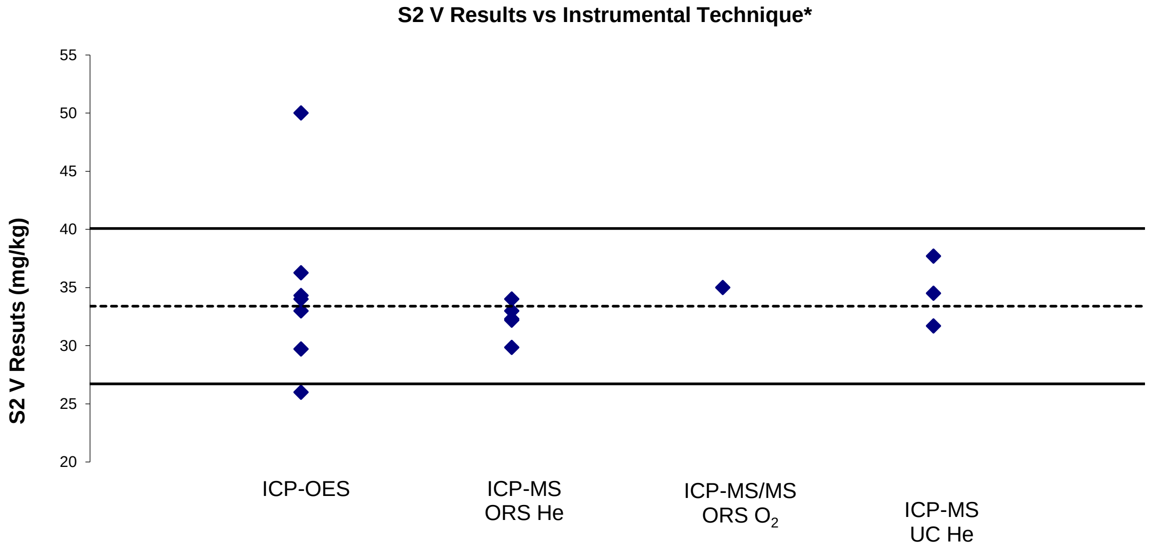
Vanadium measurements presented difficulties to participating laboratories. Of 18 reported results, 15 returned satisfactory z-scores. The high digestion temperature of 170°C might have been the cause of the high unsatisfactory V result.

Plots of participants performance versus instruments used for V measurements in S2 are presented in Figure 65.



*Result > 0.8 mg/kg has been plotted as 0.5 mg/kg. H.V. = Homogeneity Value

Figure 64 S1Se Results vs. Instrumental Technique



*Result > 50 mg/kg has been plotted as 50 mg/kg.

Figure 65 S2 V Results vs. Instrumental Technique

Potassium The between-laboratory coefficient of variation for K in S3 was higher than that predicted by the Thomson value. Participants used various instrumental techniques; these are presented against results in Figure 66.

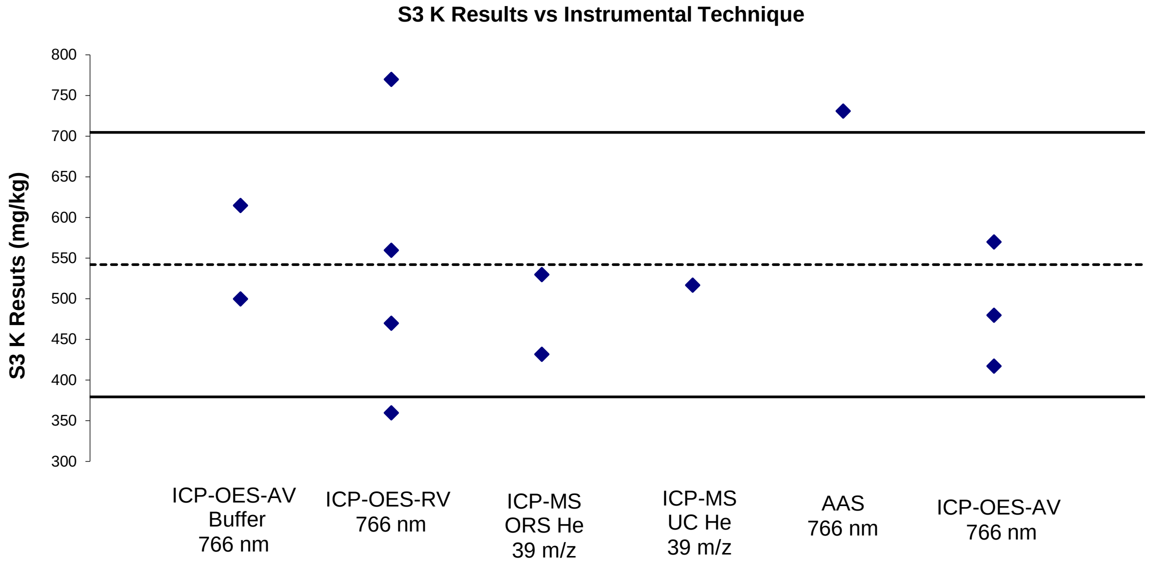


Figure 66 S3 K Results vs. Instrumental Technique

7.6 Participants' Within-Laboratory Reproducibility

Sample S3 was a soil material sample previously distributed as S3 of AQA 19-02. The same target standard deviation was used to calculate z-scores for analytes in both samples. This allowed evaluation of the within laboratory reproducibility of participants.

Of 18 laboratories who reported results in the present study in S3, 8 reported results in AQA 19-02 (Laboratories 4, 5, 6, 11, 14, 15, 17 and 19).

Bar charts of laboratories results in the two studies are presented in Figure 64. In some cases the participants results reported in the two studies are significantly different.

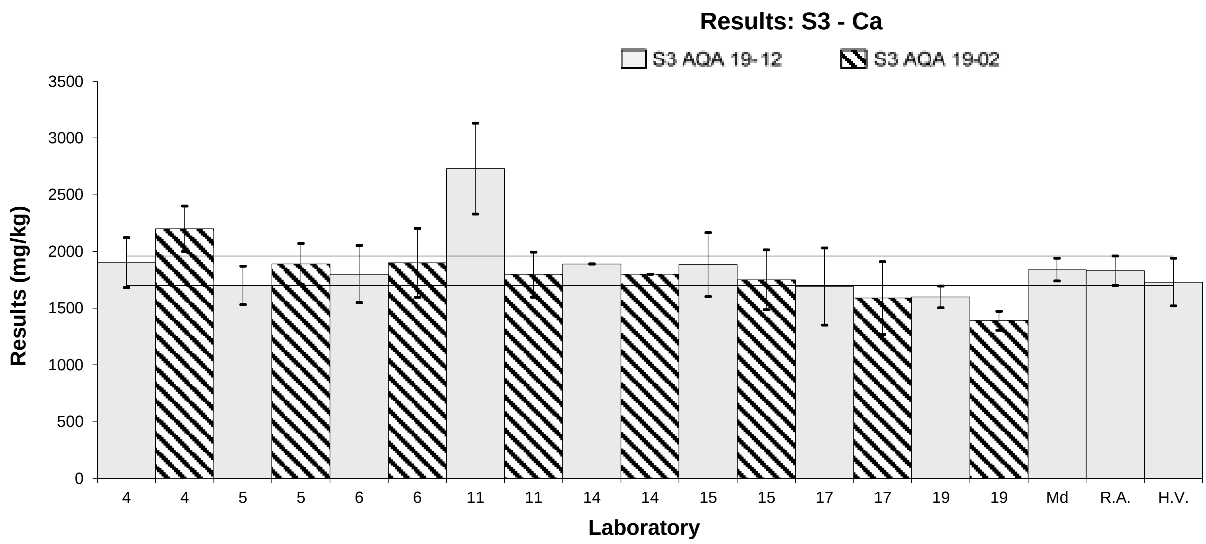
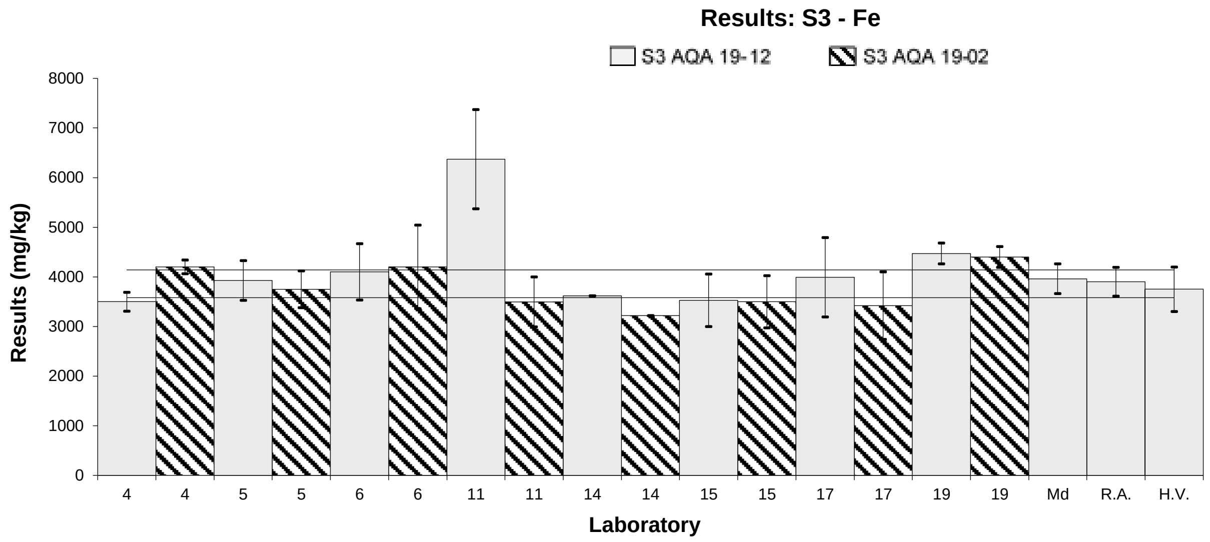
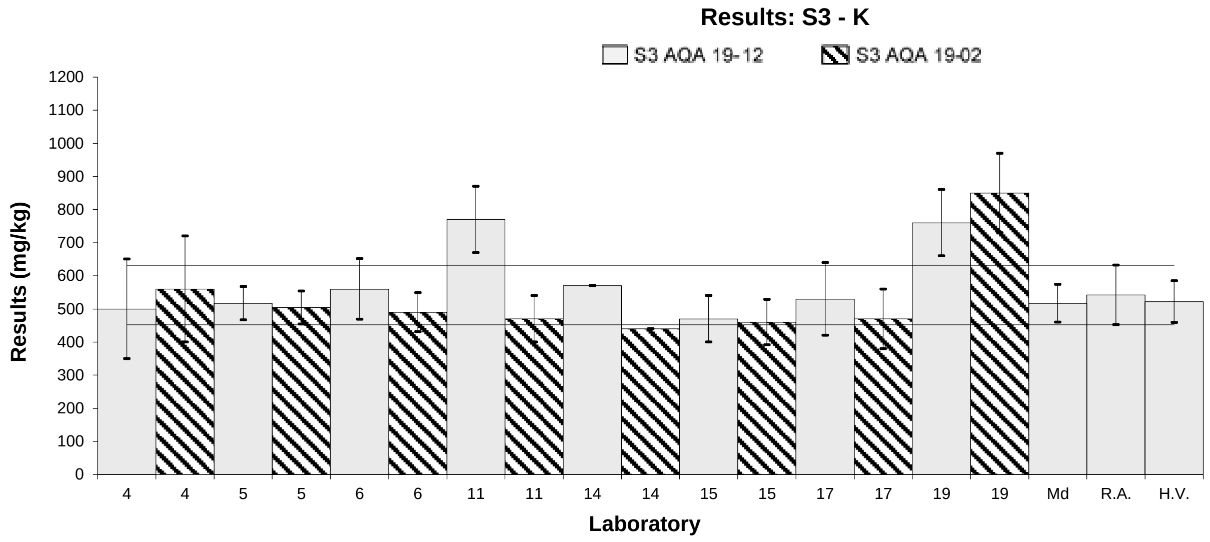


Figure 67 Bar charts of Results in S3 of AQA 19-12 and S3 of AQA 19-02





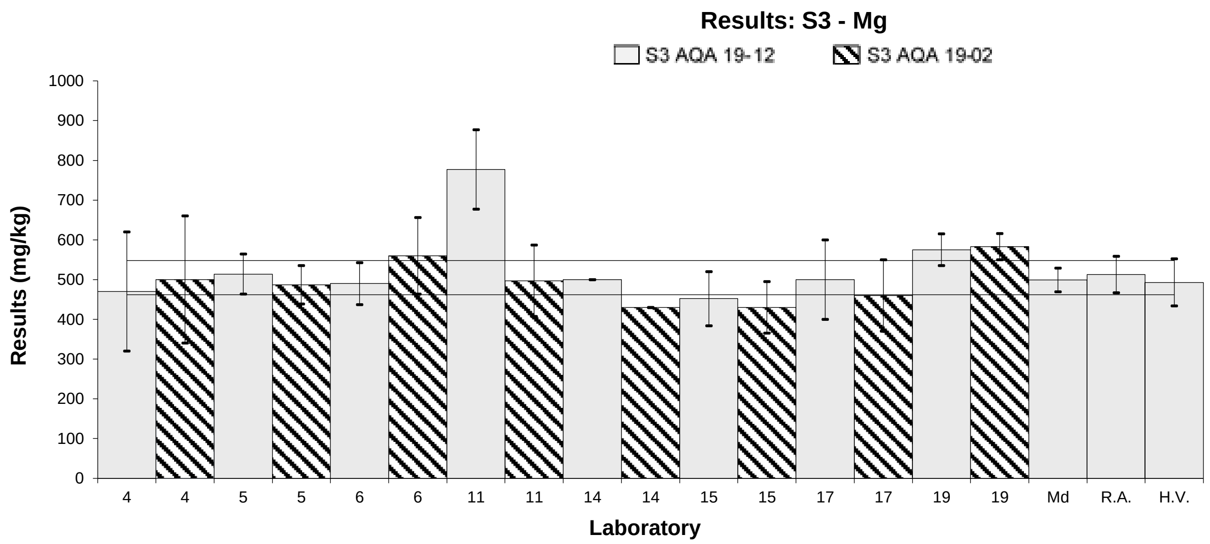
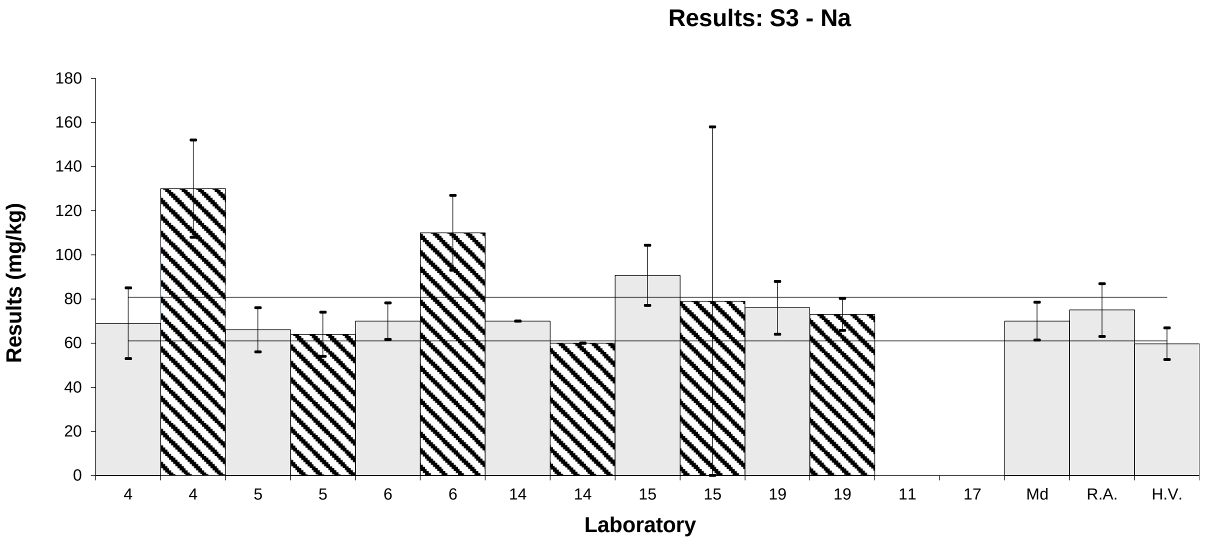
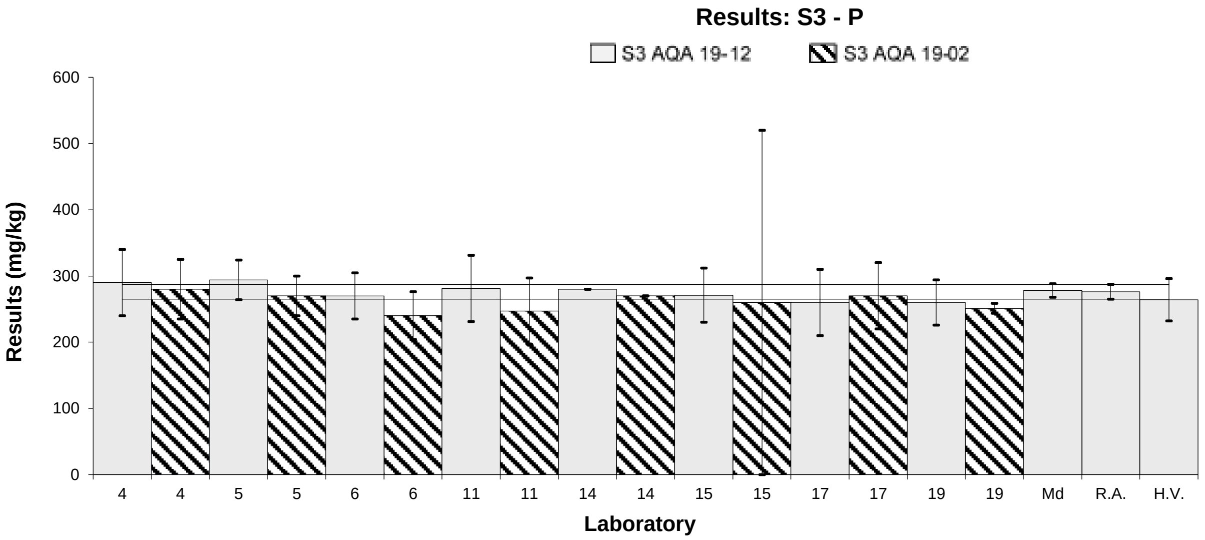


Figure 67 Bar charts of Results in S3 of AQA 19-12 and S3 of AQA 19-02 (continued)





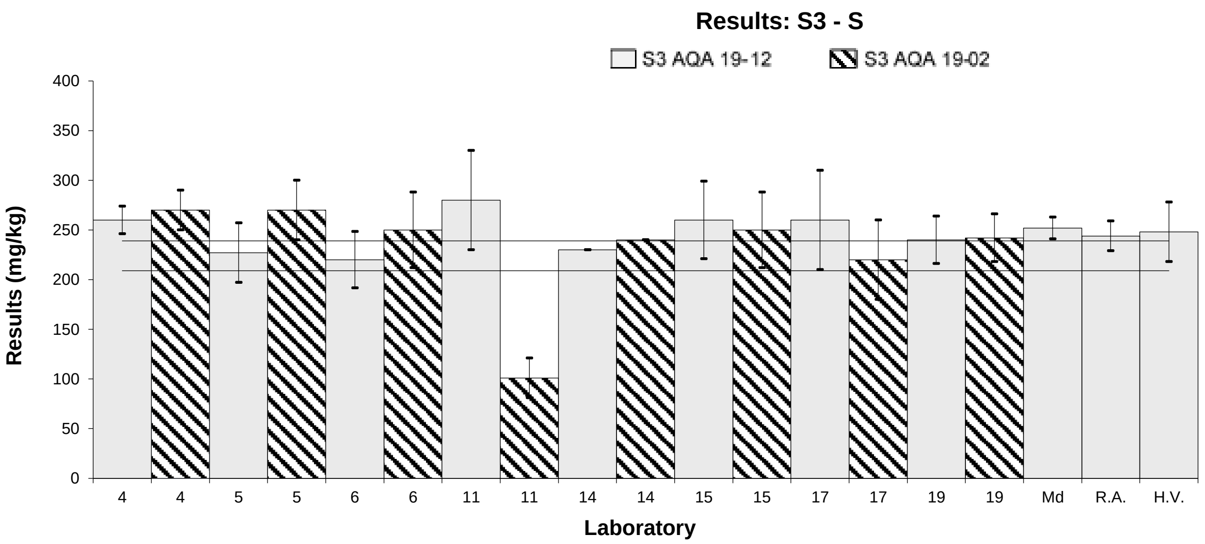
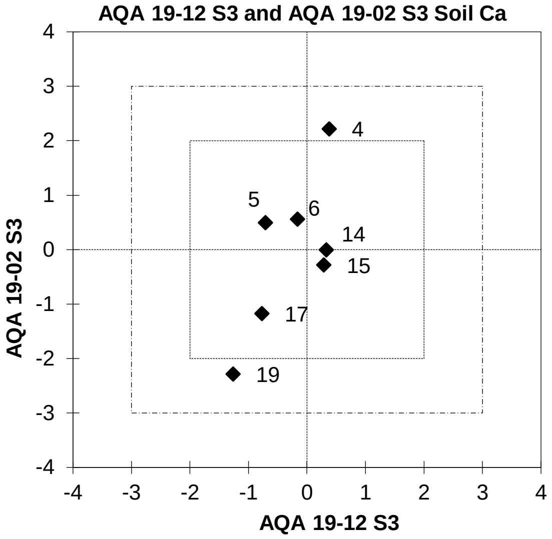
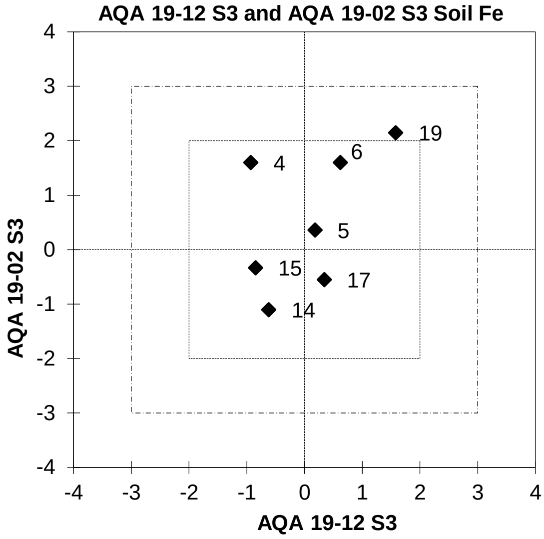


Figure 67 Bar charts of Results in S3 of AQA 19-12 and S3 of AQA 19-02 (continued)

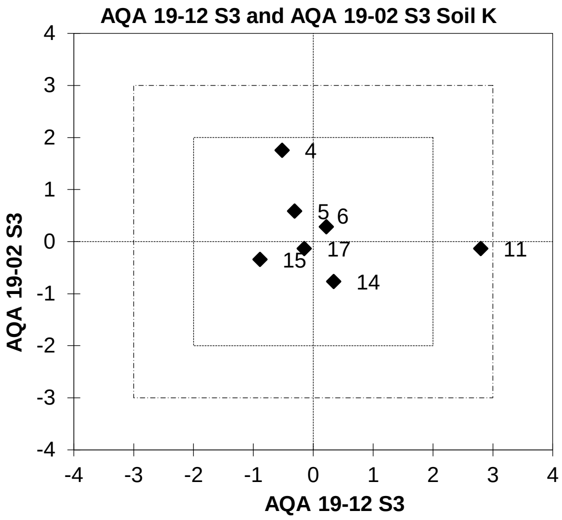
Figure 68 presents scatter plots of z-scores in Sample S3 of AQA 19-12 and S3 of AQA 19-02. Points close to the diagonal axis represent excellent reproducibility and points close to zero represent excellent reproducibility and accuracy.



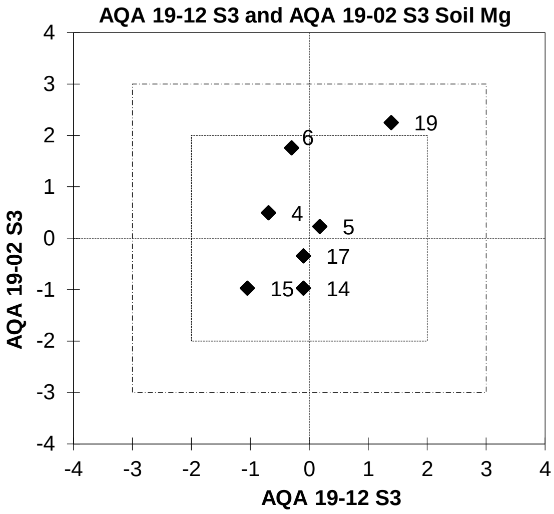
Laboratory 11 is off scale



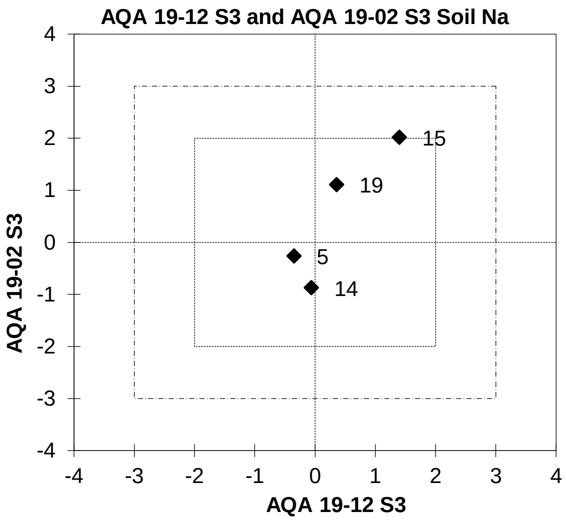
Laboratory 11 is off scale



Laboratory 19 is off scale



Laboratory 11 is off scale



Laboratories 4 and 6 are off scale

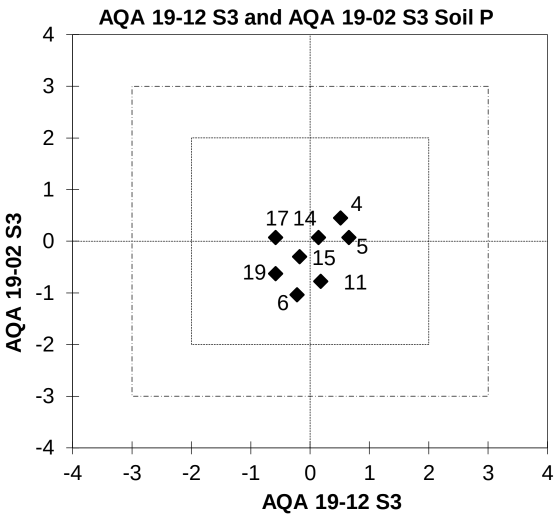
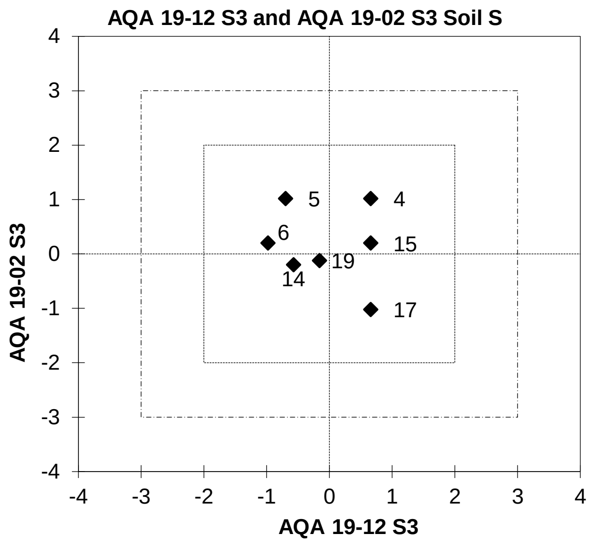


Figure 68 Scatter Plots of: z-Score in S3 of AQA 19-12 and S3 of AQA 19-02



Laboratory 11 is off scale

Figure 68 Scatter Plots of: z-Score in S3 of AQA 19-12 and S3 of AQA 19-02 (continued)

7.7 Participants' Results and Analytical Methods for 2M KCl Extractable Ammonium-N and Nitrate-N

Mineral nitrogen components, ammonium (NH_4^+), nitrite (NO_2^-) and nitrate (NO_3^-), are of particular interest when soil fertility is assessed. While water can extract NO_3^--N and NO_2^--N from a majority of soils, NH_4^+-N has to be displaced by another cation when the surface soil colloids are negatively charged.[25] The participating laboratories were asked to analyse the sample using their normal measurement technique but to follow the preparation procedure for the soil extract which involved: a soil/2M KCl ratio of 1:10 and a mixing time of one hour.

The method descriptions provided by participants are presented in Table 3. All participants used a soil/2M KCl ratio of 1:10.

2M KCl Extractable Ammonium-Nitrogen Plots of participants' results versus the analytical methods and instrumental technique used are presented in Figure 69. Although participants used various analytical methods and measurement technique most produced comparable results.

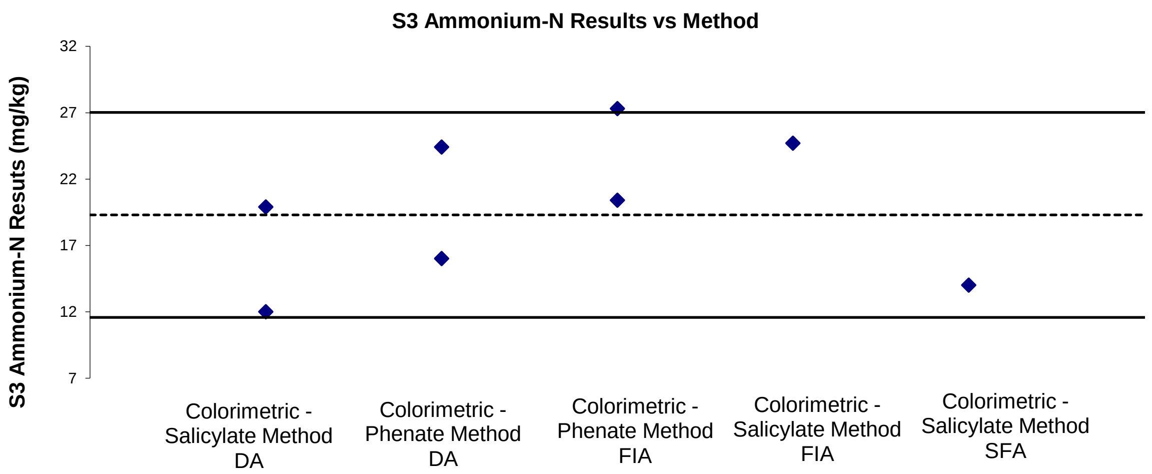


Figure 69 S3-NH_4^+-N Results vs. Analytical Method and Measurement Technique

2M KCl Extractable Nitrate-Nitrogen All participants performed satisfactorily for NO_3^--N, measurements. The measurement method used by most laboratories involved NO_3^--N reduction to NO_2^--N by passage of the clarified soil extract through a Cd-Cu reduction column followed by NO_x (the reduced NO_2^--N plus original NO_2^--N) measurements. NO_x was determined colorimetrically based on Griess-Ilosvay reaction and NO_3^--N calculated by subtracting NO_2^--N value (obtained by analysis without passing the sample through the Cd-Cu reduction column), from the NO_x value.

Two laboratories used trivalent V for NO_3^--N reduction to NO_2^--N (Figure 70).

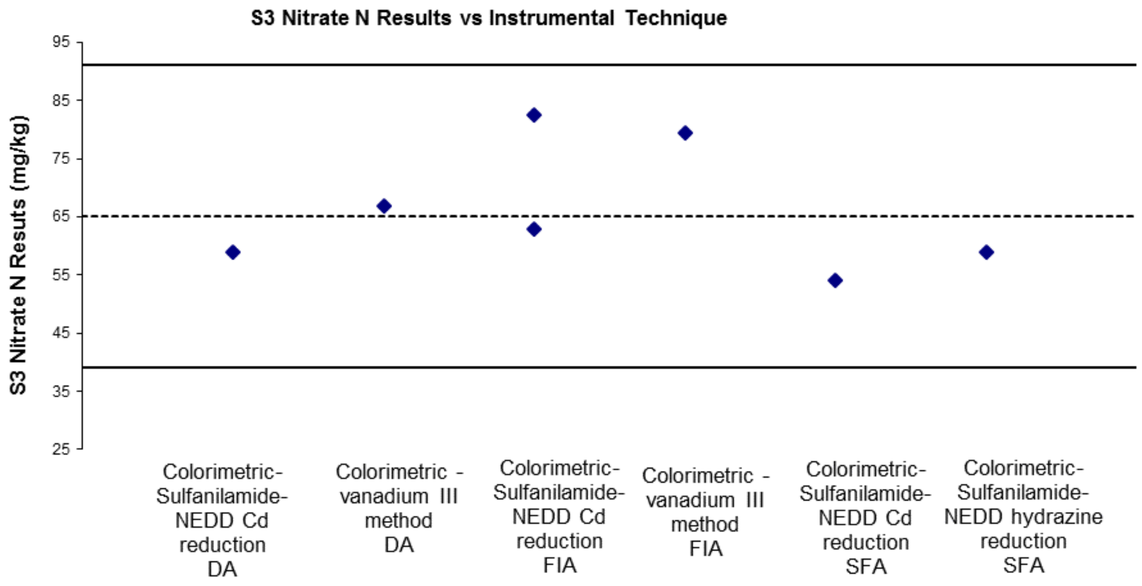
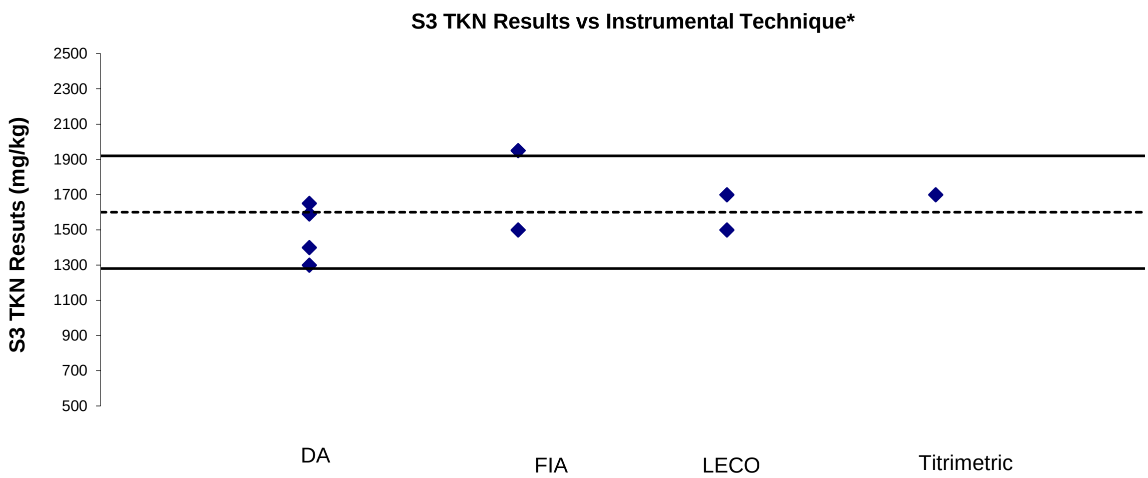


Figure 70: S3-NO_3^--N Results vs. Measurement Technique

7.8 Participants' Results and Analytical Methods for Total Kjeldahl Nitrogen

TKN assigned value was 1680 mg/kg. Laboratory 11 correctly measured TKN in S3 but reported it in the wrong units. Plots of participants' results versus analytical method and measurement technique are presented in Figure 71



*Results of 0.15 mg/kg has been plotted as 1500 mg/kg.

Figure 71 S3-TKN Results vs. Measurement Technique

7.9 Participants' Results and Analytical Methods for Water Soluble Anions

Measurement of water soluble anions in soil is an empirical measurement – where the method of extraction defines the measurand.[25, 26] With testing laboratories using different methods, each could be considered to be measuring a different measurand that is their version of 'water soluble anions in soil'. This lack of uniformity in the procedures can make the comparison of participants' results difficult.

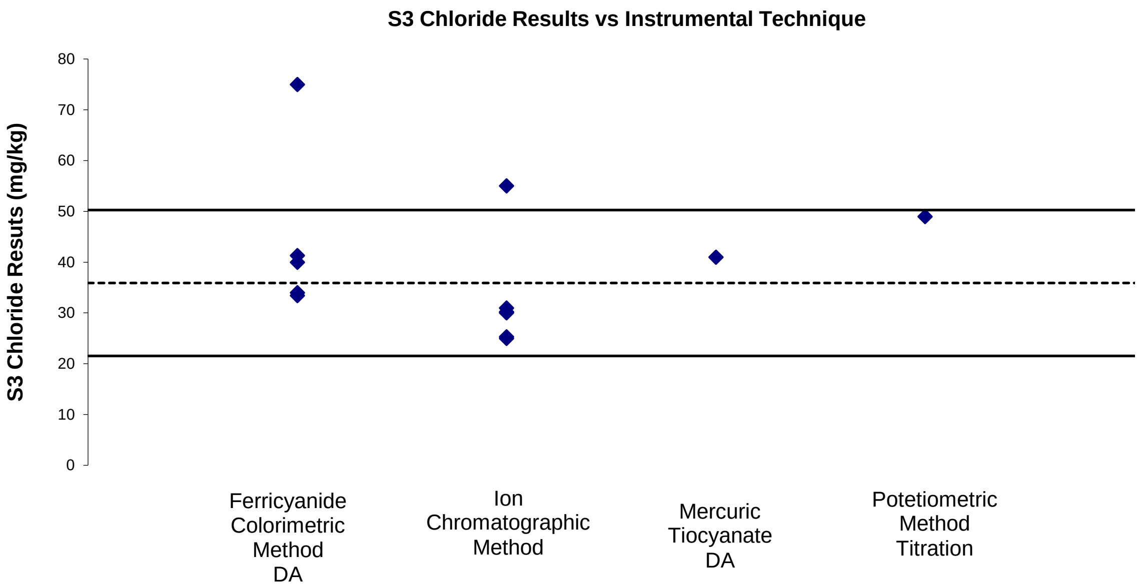
In the previous study of metals and anions in soil AQA 11-12, NMI conducted a study on water soluble anions content in soil using the same instrumental technique but two extraction procedures: one involved a soil/water ratio of 1: 5 and the other a soil/water ratio of 1:10. The fluoride, orthophosphate and sulphate results were found to change in direct proportion with the amount of water used in the extraction procedure.[27]

In the present study participating laboratories were asked to analyse the sample using their normal measurement technique but to follow the same preparation procedure for the soil extract which involved: a soil/water ratio of 1:5 and a mixing time of one hour.

The method descriptions and instrumental techniques provided by participants are presented in Tables 4 to 7. All participants used a soil/water ratio of 1 to 5.

Individual Water Soluble Anion Commentary

Chloride Fourteen participants reported results for chloride and 11 performed satisfactorily. Figure 72 presents a plot of participants' results versus measurement method and instrumental technique used for chloride analysis in S3.



*Results >75 mg/kg has been plotted as75 mg/kg.

Figure 72 S3-Chloride Results vs. Measurement Method

Fluoride Only six participants reported results for fluoride in S3**.**

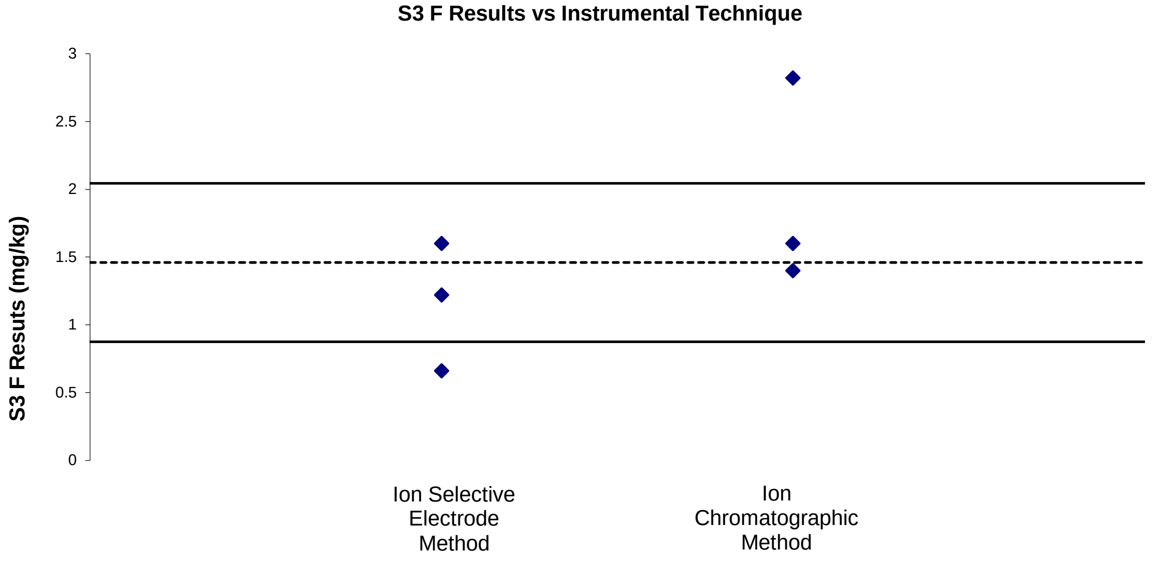
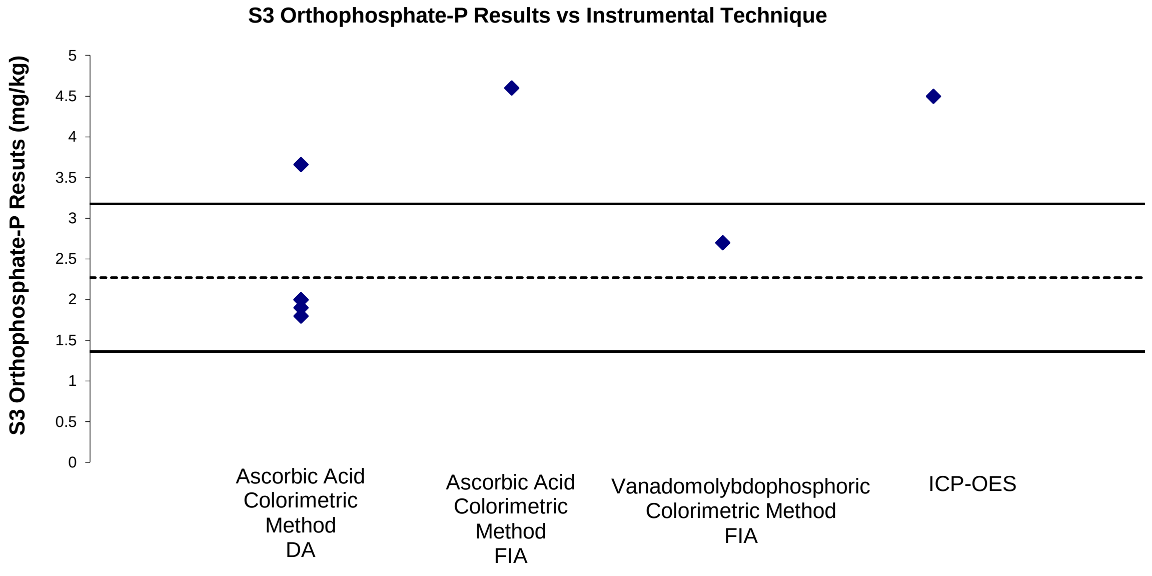


Figure 73 S3-Fluoride Results vs. Measurement Method

Orthophosphate-P. Participants used a wide variety of measurement methods and instrumental techniques (Figure 74). Some laboratories might have reported phosphate and not orthophosphate-P.



*Result >4 mg/kg has been plotted as 4 mg/kg.

Figure 74 S3-Orthophosphate-P Results vs. Method

Orthophosphates are phosphates that respond to colorimetric tests. Caution should be exercised when ICP-OES is used, as this instrument measures P in all forms of phosphates and not only in the orthophosphates.

Sulphate Plots of participants' results with the instrumental technique used are presented in Figure 75. Although most of the S in soil samples is from sulphate compounds, false positive results can be produced when this is measured by ICP-OES: this technique measures total S and not only S from sulphate compounds.

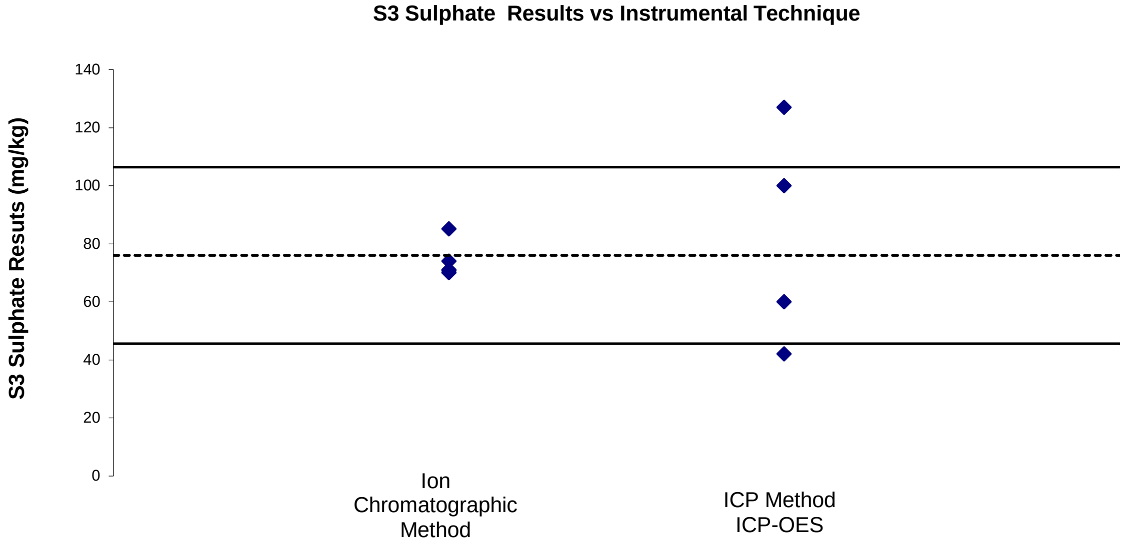


Figure 75 S3-Sulphate Results vs. Measurement Method and Instrumental Technique

pH measurements in soil did not present a difficulty to the participating laboratories**.** All the reported results were in agreement with the assigned value.

EC Of 17 reported results for EC in S3, 15 returned satisfactory z-scores.

7.10 Comparison with Previous NMI Proficiency Tests Studies of Metals in Soil

AQA 19-12 is the twenty-fifth NMI proficiency test of metals in soil. For most of the analytes the same fixed target standard deviation was used in the present study as in the previous studies of metals in soil. This allowed a comparison of participants' performance (z-score) over time and provided a benchmark for progressive improvement.

Participants' performance in measurement of metals in soil over time is presented in Figure 76. On average participants' performance remained fairly consistent.

Over time laboratories should expect at least 95% of its scores to lay with the range $|z| \leq 2$. Scores in the range $2 < |z| < 3$ occasionally can 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 are an indication of method or laboratory bias.

Individual performance history reports are emailed to each participant at the end of the study; the consideration of z-scores for an anlyte over time provides much more useful information than a single z-score.

7.11 Reference Materials and Certified Reference Materials

Participants reported whether control samples (spiked samples, certified reference materials-CRMs or matrix specific reference materials-RMs) had been used (Table 69).

Table 69 Control Samples Used by Participants

Lab. Code	Description of Control Samples
1	Reference Material
3	Spiked Sample
4	CRM 036

Table 69 Control Samples Used by Participants (continued)

Lab. Code	Description of Control Samples
5	AGAL 12
6	CRM
7	In House QC Samples
8	AGAL 10, AGAL 12
9	CRM
10	CRM
11	RM
12	CRM
13	AGAL 10
14	RM
15	Spiked Sample
16	Trace Metals - Sandy Loam - CRM026-50G
19	PACS2 Marine Sediment and NIST SRM 2704 Buffalo River Sediment
20	Previous AQA PT scheme samples
21	AGAL 10 Hawkesbury River Sediment
23	AGAL 10
24	CRM

Matrix matched control samples taken through all steps of the analytical process, are most valuable quality control tools for assessing the methods' performance.

Some laboratories reported using certified reference materials. These materials may not meet the internationally recognised definition of a Certified Reference Material:

> ***' a 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'*[28]

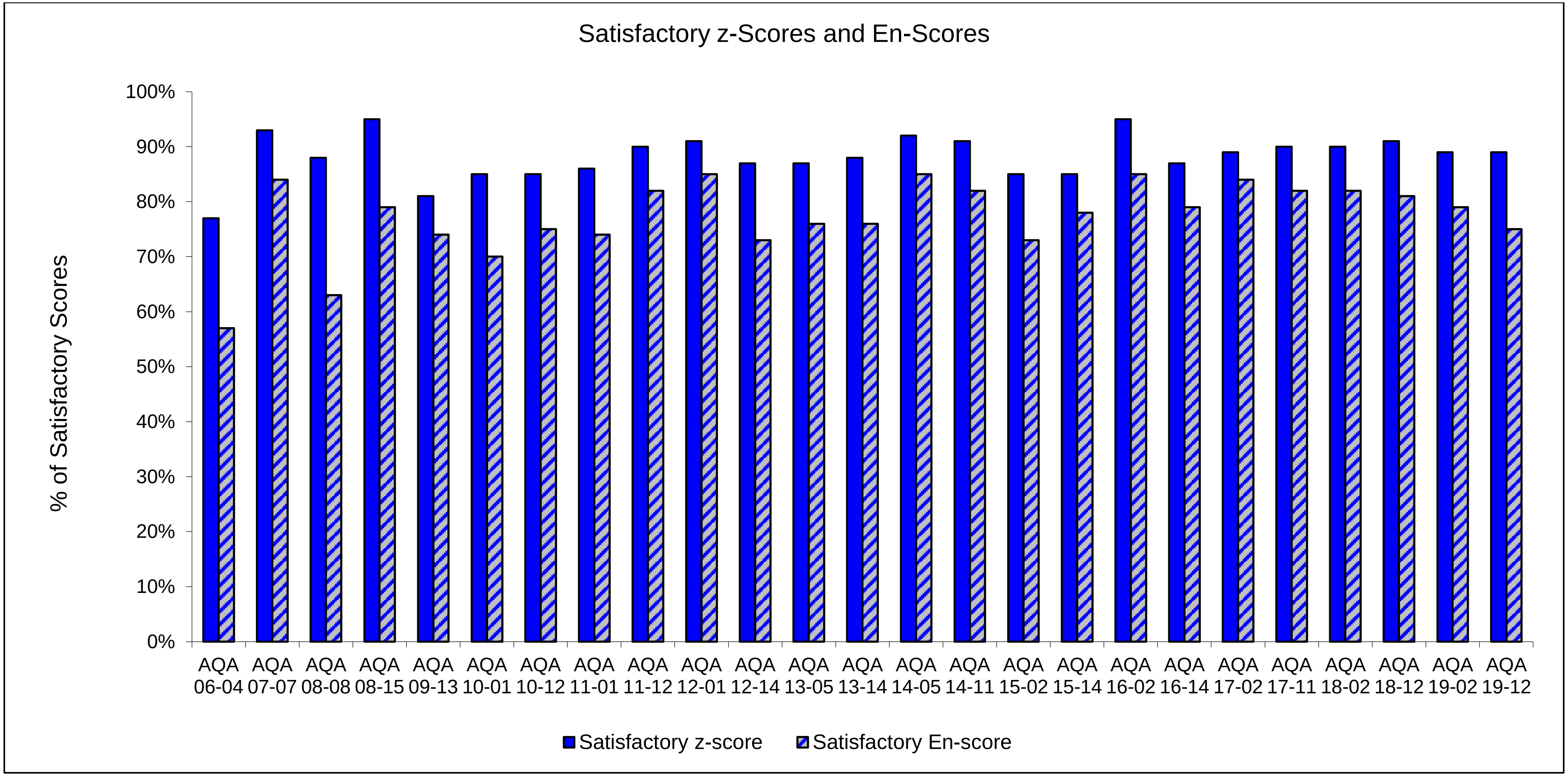


Figure 76 Participants' Performance over Time

8 REFERENCES

[1] ISO17043:2010, Conformity assessment – *General requirements for proficiency testing*.

[2] NMI 2016, *NMI Chemical Proficiency Testing Study Protocol,* viewed 7 November 2018, <http://www.measurement.gov.au>.

[3] NMI 2016, *NMI Chemical Proficiency Testing Statistical Manual,* viewed 7 November 2018, <http://www.measurement.gov.au>.

[4] Thompson, M, Ellison, S & Wood, R 2006, 'The international harmonized protocol for proficiency testing of (chemical) analytical laboratories', *Pure Appl. Chem*, vol 78, pp 145-196.

[5] National Environmental Protection Council, Schedule B(1) – *Guidelines on the Investigation Levels for Soil and Groundwater, viewed* November 2017, <http://www.ephc.gov.au/nepms>.

[6] NMI, (2012), *AQA 19-02 Metals in Soil,* <http://www.measurement.gov.au>.

[7] NMI (2014), *AQA 12-14 Metals in Soil*, <http://www.measurement.gov.au>.

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

[9] 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.

[10] ISO/IEC 17025:2018, *General requirements for the competence of testing and calibration laboratories*

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

[12] Betil, M, Naykki, T, Hovind, H & Krysell, M 2004, *Nordtest Report Handbook for Calculation of Measurement Uncertainty in Environmental Laboratories*, Nordest Tekniikantie, Finland, Esopo.

[13] Hibbert, B 2007, *Quality Assurance for the Analytical Chemistry Laboratory*, Oxford University Press.

[14] NATA 2018, *General Accreditation Guidance Estimating and Reporting Measurement Uncertainty of Chemical Test Results*

[15] ISO (2008), *Guide to the Expression of Uncertainty in Measurement (GUM)*, Geneva, Switzerland.

[16] Eurolab 2002, Technical Report No 1/2002 - *Measurement Uncertainty in Testing.*

[17] NMI, *Estimating Measurement Uncertainty for Chemists* – viewed March 2017, <www.measurement.gov.au>.

[18] Australian and New Zealand Environment and Conservation Council, and National Health and Medical Research Council -ANZECC/NMHRC 1992, *Australian and New Zealand Guidelines for the Assessment and Management of Contaminated Sites*.

[19] Australian Health Commission 1995, *Contaminated Sites Monograph No4: Trace Element Concentrations in Soils from Rural and Urban Areas of Australia*

[20] European Commission, DG Reasearch, Brussels Belgium 2002, *Methodologies for Soil and Sediment Fraction Studies.*

[21] Gaudino, S, Galas, C 2007, ‘The role of different soil sample digestion methods on trace element analysis: a comparison of ICP-MS and INAA measurement results’, *Accred Qual Assur* vol 12, pp 84-93.

[22] Chen, M, Ma, L 2001, ‘Comparison of Three Aqua Regia Digestion Methods for Twenty Florida Soil’, *Soil Sci. Soc. Am J*, vol 65, pp 491-499.

[23] Charun, Y 2006, ‘A comparative study of acid-extractable and total digestion methods for the determination of inorganic elements in peat material by inductively coupled plasma-optical emission spectrometry’, *Analytica Chmica Acta*, vol 557, pp 296-303.

[24] Roje, V 2010, ‘Multi-elemental analysis of marine sediment reference material MESS-3: one-step microwave digestion and determination by high resolution inductively coupled plasma-mass spectrometry (HR-ICP-MS)’, *Chemical papers* vol 64 (4), pp.409-414.

[25] Rayment, G. E.& Lyons, D. J 2011 *Soil Chemical Methods – Australasia*, CSIRO Publishing, Collingwood VIC Australia

[26] Afzal M., Yasin M, (2002), *Effect of soil to water ratios on chemical properties of saline-sodic and normal soil* Pakistan J. agric. Res. 17, 379-386.

[27] NMI (2014), *AQA 11-12 Metals in Soil*, <http://www.measurement.gov.au>.

[28] JCGM 200:2008, *International vocabulary of metrology – Basic and general concepts and associated terms (VIM)*, 3rd edition.

APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING

Sample Preparation

Samples S1 was a low-level contaminated soil. The material was a composite of soil samples submitted to NMI for metal analyses. The soil was autoclaved, dried at 105ºC, ground, sieved and divided into portions of 30 g each.

Sample S2 was sediment, a composite of sediment samples submitted to NMI for chemical analysis. The material was mixed, dried at 105ºC, ground, sieved and divided into portions of 25 g each.

Sample S3 was an agricultural soil material. This sample was previously distributed as sample S3 for AQA 19-02. The preparation procedure of this sample is provided in the report for AQA 19-02.

Sample Analysis and Homogeneity Testing

The same procedure was followed for Sample S1 and S2 preparation as in previous NMI PT studies.[5,6] Partial homogeneity testing was conducted for the elements of interest. Three bottles were analysed in duplicate and the average of the results was reported as the homogeneity value. Measurements were made under repeatability conditions in random order.

Although Sample S3 was formerly tested for partial homogeneity in AQA 19-02, a full homogeneity test was still conducted for this sample, with the exception of 2M KCl extractable ammonium nitrogen (NH_4^+-N) and 2M KCl extractable nitrate nitrogen (NO_3^-N). Sample S3 was demonstrated to be sufficiently homogeneous for the evaluation of participants' performance. Homogeneity testing was based on that described in the International Protocol. Seven sample bottles were selected at random. Duplicate test-portions were taken from each bottle and the concentration of all targeted analytes measured. Measurements were made under repeatability conditions in random order. Table 70 sets out an example for the testing of the homogeneity of Mg in Sample S3.

Table 70 Homogeneity Testing of Mg in Sample S3

BOTTLE	**A Mg (mg/kg)**	**B Mg (mg/kg)**
2	490	510
8	490	500
12	470	480
18	500	470
24	470	490
28	530	530
41	480	490

	Value	Critical	Result
Cochran	0.45	0.73	**Pass**
S_{an}/σ	0.24	0.50	**Pass**
S^2_{sam}	269	664	**Pass**

Sample Analysis for Acid Extractable Elements

A test portion of approximately 0.5 g of soil was weighed into a 50 mL graduated polypropylene centrifuge tube. The sample was digested using 3 mL of concentrated nitric acid and 3 mL of concentrated hydrochloric acid on a hot block at 95°C ± 5°C. After digestion, each sample was diluted to 40 mL with Milli-Q water and then further diluted as necessary.

The measurement instrument was calibrated using external standards for targeted analytes. A set of quality control samples consisting of blanks, blank matrix spike, and matrix matched reference materials, duplicates and sample matrix spikes, was carried through the same set of procedures and analysed at the same time as the samples. A summary of the instrument conditions used and the ion/wavelength monitored for each analyte is given in Table 71.

Table 71 Instrumental Technique used for Acid Extractable Elements

Analyte	Instrument	Internal Standard	Reaction/ Collision Cell (if applicable)	Cell Mode/Gas (if applicable)	S1Final Dilution Factor	S2/S3Final Dilution Factor	Ion (m/z)/ Wavelength (nm)
Al	ICP-MS	Rh	NA	NA	NA	800	27 m/z
As	ICP-MS	Rh	ORS	He	800	800	75 m/z
Ba	ICP-OES	Y	NA	NA	800	NA	445.403 nm
Be	ICP-MS	Rh	NA	NA	800	NA	9 m/z
Ca	ICP-OES	Y	NA	NA	800	800	422.673 nm
Cd	ICP-MS	Rh	NA	NA	NA	NA	111 m/z
Co	ICP-MS	Rh	ORS	He	800	800	59 m/z
Cr	ICP-MS	Rh	ORS	He	800	800	52 m/z
Cs	ICP-MS	Rh	ORS	He	800	NA	113 m/z
Cu	ICP-MS	Rh	ORS	He	800	800	65 m/z
Fe	ICP-MS	Rh	NA	NA	NA	800	56 m/z
Ga	ICP-MS	Rh	ORS	He	800	NA	71 m/z
Hg	ICP-MS	Rh	NA	NA	NA	800	201 m/z
K	ICP-MS	Rh	ORS	He	NA	800	39 m/z
La	ICP-MS	Rh	ORS	He	800	NA	139 m/z
Li	ICP-MS	Rh	ORS	He	800	NA	7 m/z
Mg	ICP-OES	Y	NA	NA	NA	800	280.270 nm
Mn	ICP-MS	Rh	ORS	He	800	800	55 m/z
Mo	ICP-MS	Rh	ORS	He	NA	800	95 m/z
Na	ICP-OES	Y	NA	NA	NA	800	588.995 nm
Ni	ICP-MS	Rh	ORS	He	800	800	60
P	ICP-OES	Rh	NA	NA	800	800	213.618
Pb	ICP-MS	Ir	NA	NA	800	800	Average of 206, 207, 208 m/z
Rb	ICP-MS	Rh	ORS	He	NA	800	85 m/z
S	ICP-OES	Y	NA	NA	NA	800	181.972 nm
Sb	ICP-MS	Rh	ORS	He	NA	800	212 m/z
Se	ICP-MS	Rh	ORS	HEHe	800	800	78 m/z
Sn	ICP-MS	Rh	NA	NA	800	NA	118 m/z
Sr	ICP-MS	Rh	ORS	He	NA	800	88 m/z
Th	ICP-MS	Ir	NA	NA	NA	800	205 m/z
U	ICP-MS	Ir	NA	NA	NA	800	238 m/z
V	ICP-MS	Rh	ORS	He	NA	800	51 m/z
Zn	ICP-MS	Rh	ORS	He	800	800	66 m/z

Sample Analysis for Water Soluble Anions

A test portion of 10 g was weighed into a 50 mL polypropylene container. The container was then filled with deionised water. The suspension was shaken, at room temperature for 1 h, centrifuged, and filtered through 0.45 µm filter. A summary of the measurement methods and instrumental techniques is presented in Table 72.

Table 72 Summary of the Measurement Methods and Instrumental Techniques used by NMI

Anion	Measurement Method	Instrument
Water Soluble Chloride	Ion Chromatographic Method	IC
Water Soluble Fluoride	Ion Chromatographic Method	IC
Water Soluble Nitrate-N	Colorimetric-Sulfanilamide-NEDD Cd reduction	FIA
Water Soluble Orthophosphate-P	Colorimetric, Ascorbic Acid Reduction	DA
Water Soluble Sulphate	Ion Chromatographic Method	IC

APPENDIX 2 - ASSIGNED VALUE, Z-SCORE AND E_N SCORE CALCULATION

The assigned value was calculated as the robust average using the procedure described in 'ISO13258:2015(E), Statistical methods for use in proficiency testing by interlaboratory comparisons – Annex C'[8] the uncertainty was estimated as:

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

where:

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

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

A worked example is set out below in Table 73.

Table 73 Uncertainty of Assigned Value for As in Sample S2

No. results (p)	19
Robust Average	19.9 mg/kg
$S_{rob\ av}$	2.08 mg/kg
$u_{rob\ av}$	0.60 mg/kg
k	2
$U_{rob\ av}$	1.2 mg/kg

The assigned value for **As** in Sample S2 is **19.9 ± 1.2 mg/kg**

z-Score and E_n-score

For each participant's result a z-score and E_n-score are calculated according to Equation 1 and Equation 2 respectively (see page 13).

A worked example is set out below in Table 82.

Table 74 z-Score and E_n-score for As Result Reported by Laboratory 5 in S2

As Result mg/kg	Assigned Value mg/kg	Set Target Standard Deviation	z-Score	E_n-Score
21.0 ± 2.2	19.9 ± 1.2	10% as CV or 0.10x19.9 = =1.99 mg/kg	$z = \frac{(21.0 - 19.9)}{1.99}$ z = 0.55	$En = \frac{(21.0 - 19.9)}{\sqrt{2.2^2 + 1.2^2}}$ E_n=0.44

APPENDIX 3 - USING PT DATA FOR UNCERTAINTY ESTIMATION

When a laboratory has successfully participated in at least 6 proficiency testing studies, the standard deviation from proficiency testing studies can also be used to estimate the uncertainty of their measurement results.[12, 14] An example is given. Between 2009 and 2019 NMI carried out 22 proficiency tests of metals in soil. These studies involved analyses of acid-extractable elements at low and high levels in dried soil, moist soil, biosoil, sediment and sludge. **Laboratory X** submitted results for As in all of these PTs. All reported results returned satisfactory z-scores. This data can usefully be separated into two ranges of results 0.5 to 10 mg/kg and 10 to 100 mg/kg.

Table 75 Laboratory X Reported Results for As at 0.5 to 10 mg/kg Level.

Study No.	Sample	Laboratory result mg/kg	Assigned value* mg/kg	Robust CV of all results (%)	Number of Results
AQA09-13	S1-biosoil	4.091	3.64	16	11
	S2-sludge	4.29	4.57	15	12
AQA11-01	S1-biosoil	3.54	3.57	19.7	18
AQA13-05	S1-soil	9.22	9.21	14	22
AQA14-11	S1-sediment	7.91	7.37	11.8	21
AQA15-02	S1-moist sludge	8.29	7.02	13	22
	S2-moist sludge	7.42	7.02	11.3	17
AQA15-14	S1-sedimentl	10	9.95	6.7	17
	S2-soil	4.53	4.47	6.4	14
AQA16-02	S2-agricultural soil	2.67	2.11	14	20
AQA 16-14	S1-clay	6.03	5.61	15	17
	S1 - soil	6.03	5.61	20	17
AQA 17-02	S1 – moist soil	3.71	3.76	10	13
AQA 18-02	S1 - compost	2.22	2.73	11	17
AQA 19-02	S1 – moist soil	2.83	2.65	11	24
AQA 19-12	S1 - soil	2.32	2.12	16	16
Average				12.5**	

* Expanded uncertainty at approximately 95% confidence. ** The mean value of Robust CV was used.

Table 76 Laboratory X Reported Results for As at 10 to 100 mg/kg Level.

Study No.	Sample	Laboratory result mg/kg	Assigned value* mg/kg	Robust CV of all results (%)	Number of Results
AQA10-12	S1-soil	16.6	14.4	8.5	19
AQA11-12	S1-moist sludge	25	21.6	15	13
AQA12-01	S1-sediment	18.4	17.3	8.1	21
AQA12-14	S2-soil	16.6	14.8	11	20
AQA13-14	S1-sandy soil	16.6	15.1	10.4	21
AQA14-05	S1-soil	13.2	12.3	7.8	25
AQA 17-11	S1 - sediment	18.1	17.4	11	22
AQA 18-12	S2 - soil	10.4	9.6	8	20
AQA 19-12	S2 - sediment	21	19.9	9	19
Average				9.9**	

* Expanded uncertainty at approximately 95% confidence. ** The mean value of Robust CV was used.

Taking the average of the robust CV over these PT samples for each concentration range gives estimates of the relative standard uncertainty of 13% and 10% respectively. Using a coverage factor of two gives relative expanded uncertainties of 26% and 20% respectively, at a level of confidence of approximately 95%. Table 75 and Table 76 sets out the expanded uncertainty for results of the measurement of As in soil, biosoil, clay, sediment, sludge, sandy soil, moist soil, compost and agricultural soil over the ranges 0.5 to 10 mg/kg and 10 to 30 mg/kg.

Table 77 Uncertainty of As Results Estimated Using PT Data.

Results mg/kg	Uncertainty mg/kg
1.00	0.26
5.0	1.3
20	4
75	15

The estimates of 26% and 20% relative passes the test of being reasonable, and the analysis of the 25 different PT samples over ten years can be assumed to include all the relevant uncertainty components (different matrices, operators, reagents, calibrators etc.), and so complies with ISO 17025:2018.[10]

APPENDIX 4 - INSTRUMENT DETAILS

Table 78 Instrument Conditions Al

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Dilution Factor	Dilution Factor	"Wavelength (nm)/
Ion(m/z)/Absorbance(nm)"							
1	ICP-MS	Sc	NA	NA	NA	250	
2					NA	NA	NA
3	ICP-OES-AV	NA	NA		NA	1000	396.152
4	ICP-OES-AV-buffer	Y			NA	100	
5	ICP-MS	Sc	UC	He	NA	625	27
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-OES-AV	Lu 219.556	NA	NA	N/A	80	Al 237.312
8					NA	NA	
9	ICP-MS	Sc 45	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	237.312
11	ICP-OES-RV	Y	NA	NA	NA	50	
12					NA	NA	
13	AGILENT-720	Y			NA		
14	ICP-MS	Sc	ORS	He	50	50	27
15	ICP-OES-RV	Y377	NA		NA		396.152 nm
16	ICP-MS	Sc	ORS	other/please type	NA	500	27
17	ICP-MS	Sc	NA	NA	NA	20	27
18					NA		
19					NA		
20					NA	NA	
21	ICP-MS	Sc	UC	He	NA	2000	27
23	ICP-OES-AV	NA			NA		308.215
24	ICP-AES	Y			NA		396.193
25	ICP-OES-AV	Y	NA	NA	NA	10	396.152

Table 79 Instrument Conditions As

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Rh	UC	NA	250	250	
2	ICP-OES-AV	Yb			50	NA	188.98
3	Hydrid generator on AAS	NA			20	200	193.7
4	ICP-OES-AV-buffer	Y			100	100	
5	ICP-MS	Ge	UC	He	625	625	75
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Rh103	ORS	O2	4000	4000	As 75/91
8					NA	NA	
9	ICP-MS	Rh 103	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	193.696
11	ICP-OES-AV	Y	NA	NA	50	50	
12	ICP-OES-AV					NA	
13	AGILENT -720	Y					
14	ICP-MS	Sc	ORS	He	50	50	75
15	ICP-MS	Ge 72	ORS		NA		75m/z
16	ICP-MS	Ge	ORS		500	500	75
17	ICP-MS	Rh	ORS	He	20	20	75
18							
19					NA		
20	AAS				366	NA	193.7
21	ICP-MS	Te	UC	He	200	200	75
23	ICP-OES-AV	NA					188.979
24	ICP-AES	Y					193.696
25	ICP-OES-AV	Y	NA	NA	1	1	188.98

Table 80 Instrument Conditions Ba

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	In	NA	NA	250	NA	
2	ICP-OES-AV	Yb			50	NA	455.403
3	ICP-OES-AV	NA			10	NA	455.403
4	ICP-OES-AV-buffer	Y			100	NA	
5	ICP-MS	Rh	NA	NA	625	NA	138
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Rh103	ORS	O2	4000	N/A	Ba 137
8					NA	NA	
9	ICP-MS	Rh 103	ORS	He		500	
10					NA	NA	
11	ICP-OES-AV	Y	NA	NA	50	NA	
12						NA	
13	AGILENT-720	Y				NA	
14	ICP-MS	Ir	ORS	He	50	NA	137
15					NA	NA	
16	ICP-MS	Ce	ORS		500	NA	137
17	ICP-MS	Rh	ORS	He	20	NA	134
18						NA	
19					NA	NA	
20						NA	
21	ICP-MS	Tb	UC	He	200	NA	137
23	ICP-OES-AV	NA				NA	233.527
24	ICP-AES	Y				NA	233.527
25	ICP-OES-AV	Y	NA	NA	1	NA	493.408

Table 81 Instrument Conditions Be

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Sc	NA	NA	250	NA	
2	ICP-OES-AV	Yb			50	NA	313.042
3	ICP-OES-AV	NA			10	NA	234.861
4	ICP-OES-AV-buffer	Y			100	NA	
5	ICP-MS	Sc	NA	NA	625	NA	9
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-OES-AV	Sc 45	ORS	N/A	80	N/A	Be 313.107
8					NA	NA	
9	ICP-MS	Sc 45				500	
10					NA	NA	
11	ICP-OES-AV	Y	NA	NA	50	NA	
12						NA	
13	AGILENT-720	Y				NA	
14	ICP-MS	Sc	NA		50	NA	9
15					NA	NA	
16	ICP-MS	Sc	ORS		500	NA	9
17	ICP-MS	Sc	NA	NA	20	NA	9
18						NA	
19					NA	NA	
20	AAS				366	NA	234.9
21	ICP-MS	Sc	UC	He	200	NA	9
23	ICP-OES-AV	NA				NA	313.107
24	ICP-AES	Y				NA	313.107
25	ICP-OES-AV	Y	NA	NA	1	NA	313.042

Table 82 Instrument Conditions Ca

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Sc	UC	He	250	NA	
2	ICP-OES-AV	Yb			50	50	373.69
3	AAS	NA	NA				422.7
4	ICP-OES-AV-buffer	Y			100	100	
5	ICP-MS	Sc	UC	He	625	625	44
6	ICP-OES-RV	Lu	NA	NA	NA	20	317.932
7	ICP-OES-AV	Eu 390.711			80	N/A	370.602
8					NA	NA	
9							
10	ICP-OES-AV	Lu 261.541			NA	50	315.887
11	ICP-OES-RV	Y	NA	NA	50	50	
12						NA	
13	AGILENT-720	Y					
14	ICP-OES-AV	Eu			50	50	316
15	ICP-OES-RV	Y377	NA		NA		317.933 nm
16	ICP-OES-AV-buffer		NA		500	500	317.933
17	ICP-MS	Rh	ORS	He	20	20	43
18							
19					NA		
20						NA	
21	ICP-MS	Sc			2000	NA	43
23	ICP-OES-RV	NA					317.933
24	ICP-AES	Y				NA	317.933
25	ICP-OES-AV	Y	NA	NA	1	NA	317.933

Table 83 Instrument Conditions Cd

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Rh	NA	NA	NA	250	
2					NA	NA	NA
3	ICP-OES-AV	NA			NA	10	226.502
4	ICP-OES-AV-buffer	Y			NA	100	
5	ICP-MS	Rh	NA	NA	NA	625	111
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Rh103	ORS	O2	4000	4000	Cd 111
8					NA	NA	
9	ICP-MS	Rh 103	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	214.439
11	ICP-OES-AV	Y	NA	NA	NA	50	
12					NA	NA	
13	AGILENT-720	Y			NA		
14	ICP-MS	Sc	ORS	He	NA	50	111
15	ICP-MS	Rh 103	ORS		NA		111m/z
16	ICP-MS	Rh	ORS		NA	500	111
17	ICP-MS	Rh	ORS	He	NA	20	111
18					NA		
19					NA		
20						NA	
21	ICP-MS	Rh	UC	He	NA	200	111
23	ICP-OES-AV	NA			NA		228.802
24	ICP-AES	Y			NA		228.802
25	ICP-OES-AV	Y	NA	NA	NA	1	228.802

Table 84 Instrument Conditions Co

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Rh	UC	He	250	250	
2	ICP-OES-AV	Yb			50	NA	230.786
3	ICP-OES-AV	NA			10	10	231.16
4	ICP-OES-AV-buffer	Y			100	100	
5	ICP-MS	Ge	UC	He	625	625	59
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Rh103	ORS	O2	4000	4000	Co 59
8					NA	NA	
9	ICP-MS	Sc 45	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	231.16
11	ICP-OES-AV	Y	NA	NA	50	50	
12						NA	
13	AGILENT -720	Y					
14	ICP-MS	Sc	ORS	He	50	50	58
15	ICP-OES-AV	Te214	NA		NA		228.615 nm
16	ICP-MS	Sc	ORS		500	500	59
17	ICP-MS	Rh	ORS	He	20	20	59
18							
19					NA		
20	AAS				9.3	NA	242.5
21	ICP-MS	Ga	UC	He	200	200	59
23	ICP-OES-AV	NA					228.616
24	ICP-AES	Y					228.616
25	ICP-OES-AV	Y	NA	NA	1	1	228.615

Table 85 Instrument Conditions Cr

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Sc	UC	He	250	250	
2	ICP-OES-AV	Yb			50	NA	206.158
3	ICP-OES-AV	NA			10	10	283.563
4	ICP-OES-AV-buffer	Y			100	100	
5	ICP-MS	Sc	UC	He	625	625	52
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Sc 45	ORS	O2	4000	4000	Cr 52
8					NA	NA	
9	ICP-MS	Sc 45	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	267.716
11	ICP-OES-AV	Y	NA	NA	50	50	
12						NA	
13	AGILENT -720	Y					
14	ICP-MS	Sc	ORS	He	50	50	52
15	ICP-OES-AV	Te214	NA		NA		267.716 nm
16	ICP-MS	Sc	ORS		500	500	52
17	ICP-MS	Rh	ORS	He	20	20	52
18							
19					NA		
20	AAS				9.3	NA	357.9
21	ICP-MS	Sc	UC	He	200	200	52
23	ICP-OES-AV	NA					267.716
24	ICP-AES	Y					267.716
25	ICP-OES-AV	Y	NA	NA	1	1	267.716

Table 86 Instrument Conditions Cs

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	In	NA	NA	250	NA	
2						NA	NA
3						NA	
4						NA	
5						NA	
6	NA	NA	NA	NA	NA	NA	NA
7	N/A	N/A	N/A	N/A	N/A		N/A
8					NA	NA	
9							
10					NA	NA	
11						NA	
12						NA	
13	AGILENT -720	Y				NA	
14	ICP-MS	Ir	ORS	He	50	NA	133
15					NA	NA	
16						NA	
17	ICP-MS	Rh	ORS	He	20	NA	133
18						NA	
19					NA	NA	
20						NA	
21	ICP-MS	Tb	UC	He	200	NA	133
23	NA	NA				NA	
24						NA	327.393
25	ICP-OES-AV	Y	NA	NA		NA	

Table 87 Instrument Conditions Cu

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Rh	UC	He	250	250	
2	ICP-OES-AV	Yb			50	NA	327.395
3	ICP-OES-AV	NA			10	10	324.754
4	ICP-OES-AV-buffer	Y			100	100	
5	ICP-MS	Ge	UC	He	625	625	63
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Rh103	ORS	O2	4000	4000	Cu 63
8					NA	NA	
9	ICP-MS	Sc 45	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	327.395
11	ICP-OES-AV	Y	NA	NA	50	50	
12	ICP-OES-AV					NA	
13	AGILENT-720	Y					
14	ICP-MS	Sc	ORS	He	50	50	63
15					NA		
16	ICP-MS	Sc	ORS		500	500	63
17	ICP-MS	Rh	ORS	He	20	20	63
18							
19					NA		
20	AAS				9.3	NA	327.4
21	ICP-MS	Ga	UC	He	200	200	63
23	ICP-OES-AV	NA					324.752
24	ICP-AES	Y					
25	ICP-OES-AV	Y	NA	NA	1	1	324.754

Table 88 Instrument Conditions Fe

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Sc	UC	He	NA	NA	
2	ICP-OES-AV	Yb			NA	50	238.204
3	ICP-OES-AV	NA			NA	100	238.204
4	ICP-OES-AV-buffer	Y			NA	100	
5	ICP-MS	Sc	UC	He	NA	625	56
6	ICP-MS	Rh	ORS	He	NA	20	56
7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
8	ICP-MS	Sc	ORS	He	NA	500	56
9	ICP-MS	Sc 45	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	100	238.204
11	ICP-OES-RV	Y	NA	NA	NA	50	
12					NA	NA	
13	AGILENT-720	Y			NA		
14	ICP-OES-AV	Eu			NA	50	259
15	ICP-OES-AV	Te214	NA		NA		259.940nm
16	ICP-MS	Sc	ORS		NA	500	56
17	ICP-MS	Rh	ORS	He	NA	20	56
18					NA		
19					NA		
20					NA	NA	
21					NA	NA	
23	NA	NA			NA		
24	ICP-AES	Y			NA	NA	278.209
25	ICP-OES-AV	Y	NA	NA	NA	NA	

Table 89 Instrument Conditions Ga

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS					NA	
2	ICP-MS	Sc	ORS	NA	50	NA	69
3						NA	
4						NA	
5						NA	
6	NA	NA	NA	NA	NA	NA	NA
7	N/A	N/A	N/A	N/A		NA	N/A
8					NA	NA	
9							
10					NA	NA	
11						NA	
12						NA	
13	AGILENT-720	Y				NA	
14	ICP-MS	Sc	ORS	He	50	NA	71
15					NA	NA	
16						NA	
17	ICP-MS	Rh	ORS	He	20	NA	71
18						NA	
19					NA	NA	
20						NA	
21	UC	He	200	200	59		
23	NA	NA				NA	
24						NA	
25	ICP-OES-AV	Y	NA	NA		NA	

Table 90 Instrument Conditions Hg

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Ir	NA	NA	NA	250	
2					NA	NA	NA
3	Cold Vapour FIMS	NA			NA		
4					NA		
5	ICP-MS	Ir	NA	NA	NA	625	201
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Ir 193	ORS	O2	N/A	4000	Hg 202
8					NA	NA	
9	ICP-MS	Lu 175	ORS	He		500	
10	CVAAS				NA	100	
11	VGA-ICP-OES	Y	NA	NA	NA	50	
12					NA	NA	
13	ELAN9000	Bi			NA		
14	AAS				NA	50	153
15	ICP-MS	Ir 193	ORS		NA		202m/z
16	ICP-MS	Rh	ORS		NA	500	202
17	ICP-MS	Ir	ORS	He	NA	20	202
18					NA		
19					NA		
20					NA	NA	
21	ICP-MS	Tb	UC	He	NA	200	201
23	FIMS	NA			NA		253.7
24					NA		
25	CVAAS	NA	NA	NA	NA		

Table 91 Instrument Conditions K

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Ir	NA	NA	NA	NA	
2	ICP-OES-AV	Yb			NA	50	766.491
3	AAS	NA			NA	10	766.5
4	ICP-OES-AV-buffer	Y			NA	100	
5	ICP-MS	Sc	UC	He	NA	625	39
6	ICP-OES-RV	Lu	NA	NA	NA	20	766.5
7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
8					NA	NA	
9							
10	ICP-OES-AV	Lu 261.541			NA	50	766.933
11	ICP-OES-RV	Y	NA	NA	NA	50	
12					NA	NA	
13	AGILENT-720	Y			NA		
14	ICP-OES-AV	Eu			NA	50	766
15	ICP-OES-RV	Y377	NA		NA		766.491nm
16	ICP-OES-AV-buffer		NA		NA	500	766.491
17	ICP-MS	Rh	ORS	He	NA	20	39
18					NA		
19					NA		
20					NA	NA	
21					NA	NA	
23	ICP-OES-RV	NA			NA		766.49
24	ICP-AES	Y			NA	NA	766.49
25	ICP-OES-AV	Y	NA	NA	NA	NA	

Table 92 Instrument Conditions La

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	In	NA	NA	250	NA	
2	ICP-MS	In	ORS	He	50	NA	139
3						NA	
4						NA	
5						NA	
6	NA	NA	NA	NA	NA	NA	NA
7	N/A	N/A	N/A	N/A		N/A	N/A
8					NA	NA	
9							
10					NA	NA	
11						NA	
12						NA	
13						NA	
14	ICP-MS	Rh	ORS	He	50	NA	139
15					NA	NA	
16	ICP-MS		ORS		500	NA	
17	ICP-MS	Rh	ORS	He	20	NA	139
18						NA	
19					NA	NA	
20						NA	
21	ICP-MS	Tb	UC	He	200	NA	139
23	NA	NA				NA	
24						NA	
25	ICP-OES-AV	Y	NA	NA		NA	

Table 93 Instrument Conditions Li

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Sc	NA	NA	250	NA	
2	ICP-OES-AV	Yb			50	NA	670.783
3	ICP-OES-AV	NA			10	NA	670.783
4	ICP-OES-AV-buffer	Y			100	NA	
5	ICP-MS	Sc	NA	NA	625	NA	7
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-OES-AV	Sc45	ORS	N/A	80	N/A	Li 670.783
8					NA	NA	
9							
10					NA	NA	
11	ICP-OES-AV	Y	NA	NA	50	NA	
12						NA	
13	AGILENT-720	Y				NA	
14	ICP-MS	Sc	NA		50	NA	7
15					NA	NA	
16			NA			NA	
17	ICP-MS	Sc	NA	NA	20	NA	7
18						NA	
19					NA	NA	
20						NA	
21	ICP-MS	Sc			2000	NA	7
23	NA	NA				NA	
24	ICP-AES	Y				NA	
25	ICP-OES-AV	Y	NA	NA	1	NA	670.783

Table 94 Instrument Conditions Mg

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Sc	UC	NA	NA	NA	
2	ICP-OES-AV	Yb			NA	50	383.829
3	AAS	NA			NA	10	285.2
4	ICP-OES-AV-buffer	Y			NA	100	
5	ICP-MS	Sc	UC	He	NA	625	25
6	ICP-OES-RV	Lu	NA	NA	NA	20	285.213
7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
8					NA	NA	
9							
10	ICP-OES-AV	Lu 261.541			NA	50	285.213
11	ICP-OES-RV	Y	NA	NA	NA	50	
12					NA	NA	
13	AGILENT-720	Y			NA		
14	ICP-OES-AV	Eu			NA	50	384
15	ICP-OES-RV	Y377	NA		NA		280.27 nm
16	ICP-OES-AV-buffer		NA		NA	500	285.213
17	ICP-MS	Rh	ORS	He	NA	20	24
18					NA		
19					NA		
20					NA	NA	
21					NA	NA	
23	ICP-OES-AV	NA			NA		279.077
24					NA	NA	285.213
25	ICP-OES-AV	Y	NA	NA	NA	NA	

Table 95 Instrument Conditions Mn

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Rh	UC	He	250	250	
2	ICP-OES-AV	Yb			50	NA	260.568
3	ICP-OES-AV	NA			10	10	294.921
4	ICP-OES-AV-buffer	Y			100	100	
5	ICP-MS	Sc	UC	He	625	625	55
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-OES-AV	Eu 271.700	NA	O2	80	80	Mn 257.610
8					NA	NA	
9	ICP-MS	Sc 45	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	257.61
11	ICP-OES-AV	Y	NA	NA	50	50	
12						NA	
13	AGILENT-720	Y					
14	ICP-MS	Sc	ORS	He	50	50	55
15	ICP-OES-AV	Te214	NA		NA		191.446 nm
16	ICP-MS	Sc	ORS		500	500	55
17	ICP-MS	Rh	ORS	He	20	20	55
18							
19					NA		
20	AAS				9.3	NA	279.5
21	ICP-MS	Sc	UC	He	2000	2000	55
23	ICP-OES-AV	NA					257.61
24	ICP-AES	Y					257.63
25	ICP-OES-AV	Y	NA	NA	1	1	257.61

Table 96 Instrument Conditions Mo

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Rh	NA	NA	NA	250	
2					NA	NA	NA
3	ICP-OES-AV	NA			NA	1	202.032
4	ICP-OES-AV-buffer	Y			NA	100	
5	ICP-MS	Rh	NA	NA	NA	625	95
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Rh103	NA	O2	N/A	4000	Mo 95
8					NA	NA	
9	ICP-MS	Rh 103	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	202.032
11	ICP-OES-AV	Y	NA	NA	NA	50	
12					NA	NA	
13	AGILENT-720	Y			NA		
14	ICP-MS	Rh	ORS	He	NA	50	95
15	ICP-OES-AV	Te214	NA		NA		204.598 nm
16	ICP-MS	Y	ORS		NA	500	95
17	ICP-MS	Rh	ORS	He	NA	20	95
18					NA		
19					NA		
20					NA	NA	
21	ICP-MS	Rh	UC	He	NA	200	98
23	ICP-OES-AV	NA			NA		202.031
24	ICP-AES	Y			NA		202.031
25	ICP-OES-AV	Y	NA	NA	NA	1	202.032

Table 97 Instrument Conditions Na

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS				NA	NA	
2	ICP-OES-AV	Yb			NA	50	589.592
3	AAS	NA			NA	1	330.2
4	ICP-OES-AV-buffer	Y			NA	100	
5	ICP-MS	Sc	UC	He	NA	625	23
6	ICP-OES-RV	Lu	NA	NA	NA	20	589.588
7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
8					NA	NA	
9							
10	ICP-OES-AV	Lu 261.541			NA	50	588.995
11	ICP-OES-RV	Y	NA	NA	NA	50	
12					NA	NA	
13	AGILENT -723	Y			NA		
14	ICP-OES-RV	Eu			NA	50	589
15	ICP-OES-RV	Y377	NA		NA		589.592 nm
16	ICP-OES-AV-buffer		ORS		NA	500	588.995
17	ICP-MS	Rh	ORS	He	NA	20	23
18					NA		
19					NA		
20					NA	NA	
21					NA	NA	
23	ICP-OES-RV	NA			NA		589.592
24					NA	NA	589.592
25	ICP-OES-AV	Y	NA	NA	NA	NA	

Table 98 Instrument Conditions Ni

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Rh	UC	He	250	250	
2	ICP-OES-AV	Yb			50	NA	231.604
3	ICP-OES-AV	NA			10	10	231.604
4	ICP-OES-AV-buffer	Y			100	100	
5	ICP-MS	Ge	UC	He	625	625	60
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Rh103	NA	O2	4000	4000	Ni 60
8					NA	NA	
9	ICP-MS	Sc 45	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	231.604
11	ICP-OES-AV	Y	NA	NA	50	50	
12						NA	
13	AGILENT-720	Y					
14	ICP-MS	Sc	ORS	He	50	50	60
15	ICP-OES-AV	Te214	NA		NA		216.555 nm
16	ICP-MS	Sc	ORS		500	500	60
17	ICP-MS	Rh	ORS	He	20	20	60
18							
19					NA		
20	AAS				9.3	NA	232
21	ICP-MS	Ga	UC	He	200	200	60
23	ICP-OES-AV	NA					231.604
24	ICP-AES	Y					231.604
25	ICP-OES-AV	Y	NA	NA	1	1	231.604

Table 99 Instrument Conditions P

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS					NA	
2	ICP-OES-AV	Yb			50	50	177.434
3	ICP-OES-AV	NA			10	10	213.618
4	ICP-OES-AV-buffer	Y			100	100	
5	ICP-MS	Sc	UC	He	625	625	31
6	ICP-OES-AV	Lu	NA	NA	NA	20	178.222
7	ICP-OES-AV	N/A			80	N/A	185.878
8	ICP-MS	Sc	ORS	He	NA	500	31
9							
10	ICP-OES-AV	Lu 261.541			NA	50	213.618
11	ICP-OES-AV	Y	NA	NA	50	50	
12						NA	
13	AGILENT-720	Y					
14	ICP-OES-AV	Eu			50	50	186
15	ICP-OES-AV	Te214	NA		NA		177.434 nm
16	ICP-OES-AV-buffer		NA		500	500	213.618
17	ICP-MS	Rh	ORS	HEHe	20	20	31
18							
19					NA		
20						NA	
21	ICP-MS	Sc			2000	NA	31
23	NA	NA					
24	ICP-AES	Y				NA	
25	ICP-OES-AV	Y	NA	NA		NA	213.618

Table 100 Instrument Conditions Pb

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Ir	NA	NA	250	250	
2	ICP-OES-AV	Yb			50	NA	220.353
3	ICP-OES-AV	NA			10	10	220.353
4	ICP-OES-AV-buffer	Y			100	100	
5	ICP-MS	Ir	NA	NA	625	625	206+207+208
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Ir 193	NA	O2	4000	4000	Pb 208
8					NA	NA	
9	ICP-MS	Lu 175	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	220.353
11	ICP-OES-AV	Y	NA	NA	50	50	
12	ICP-OES-AV					NA	
13	AGILENT-720	Y					
14	ICP-MS	Ir	ORS	He	50	50	208
15	ICP-OES-AV	Te214	NA		NA		220.353 nm
16	ICP-MS	Rh	ORS		500	500	
17	ICP-MS	Rh	ORS	He	20	20	207
18							
19					NA		
20	AAS				9.3	NA	283.3
21	ICP-MS	Tb	UC	He	200	200	206+207+208
23	ICP-OES-AV	NA					220.353
24							260.353
25	ICP-OES-AV	Y	NA	NA	1	1	220.353

Table 101 Instrument Conditions Rb

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS				250	NA	
2						NA	NA
3						NA	
4						NA	
5						NA	
6	NA	NA	NA	NA	NA	NA	NA
7	N/A	N/A	N/A	N/A		N/A	N/A
8					NA	NA	
9							
10					NA	NA	
11						NA	
12						NA	
13						NA	
14	ICP-MS	Sc	ORS	He	50	NA	85
15					NA	NA	
16						NA	
17	NA	NA	NA	NA	NA	NA	NA
18						NA	
19					NA	NA	
20						NA	
21	ICP-MS	Rh	UC	He	200	NA	85
23	NA	NA				NA	
24						NA	
25	ICP-OES-AV	Y	NA	NA		NA	

Table 102 Instrument Conditions S

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS				NA	NA	
2	ICP-OES-AV	Yb			NA	50	181.972
3	ICP-OES-AV	NA			NA	10	181.972
4	ICP-OES-AV-buffer	Y			NA	100	
5	ICP-OES-AV	Y	NA	NA	NA	62.5	181.975
6	ICP-OES-AV	Lu	NA	NA	NA	20	181.976
7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
8					NA	NA	
9							
10	ICP-OES-AV	Lu 261.541			NA	50	181.972
11	ICP-OES-AV	Y	NA	NA	NA	50	
12					NA	NA	
13					NA		
14	ICP-OES-AV	Eu			NA	50	182
15	ICP-OES-AV	Te214	NA		NA		180.669 nm
16	ICP-OES-AV-buffer		NA		NA	500	181.972
17	ICP-OES-RV	Y	NA	NA	NA	20	181.972
18					NA		
19					NA		
20					NA	NA	
21					NA	NA	
23	NA	NA			NA		
24					NA	NA	
25	ICP-OES-AV	Y	NA	NA	NA	NA	

Table 103 Instrument Conditions Sb

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS				NA	250	
2					NA	NA	NA
3	ICP-OES-AV	NA			NA	1	206.834
4	ICP-OES-AV-buffer	Y			NA	100	
5	ICP-MS	Rh	NA	NA	NA	625	121
6	NA	NA	NA	NA	NA	NA	NA
7	N/A	N/A	N/A	N/A	NA	N/A	N/A
8					NA	NA	
9	ICP-MS	Rh 103	ORS	He		500	
10					NA		
11	ICP-OES-AV	Y	NA	NA	NA	50	
12					NA	NA	
13	AGILENT-720	Y			NA		
14	ICP-MS	Rh	ORS	He	NA	50	121
15	ICP-MS	Rh 103			NA		
16	ICP-MS		ORS		NA	500	
17	NA	NA	NA	NA	NA	NA	NA
18					NA		
19					NA		
20					NA	NA	
21	ICP-MS	Rh	UC	He	NA	200	121
23	ICP-OES-AV	NA			NA		206.836
24					NA		206.836
25	ICP-OES-AV	Y	NA	NA	NA	1	206.834

Table 104 Instrument Conditions Se

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Rh	UC	He	250	250	
2	ICP-OES-AV	Yb			50	NA	196.026
3	ICP-OES-AV	NA				10	196.026
4	ICP-OES-AV-buffer	Y			100	100	
5	ICP-MS	Rh	NA	NA	625	625	82
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Rh103	ORS	O2	4000	4000	Se 78/94
8					NA	NA	
9	ICP-MS	Rh 103	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	196.026
11	ICP-OES-AV	Y	NA	NA	50	50	
12						NA	
13	AGILENT-720	Y					
14	ICP-MS	Sc	ORS	He	50	50	78
15	ICP-MS	Rh 103	ORS	HEHe	NA		78m/z
16	ICP-MS	Ge	ORS		500	500	78
17	ICP-MS	Rh	ORS	HEHe	20	20	78
18							
19					NA		
20	AAS				366	NA	196
21	ICP-MS	Te	UC	He	200	200	82
23	ICP-OES-AV	NA					196.026
24	ICP-AES	Y					196.026
25	ICP-OES-AV	Y	NA	NA	1	1	196.026

Table 105 Instrument Conditions Sn

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Rh	NA	NA	250	NA	
2	ICP-OES-AV	Yb			50	NA	189.925
3	ICP-OES-AV	NA				NA	189.925
4	ICP-OES-AV-buffer	Y			100	NA	
5	ICP-MS	Rh	NA	NA	625	NA	118
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Rh103	ORS	O2	4000	NA	Sn 118/134
8					NA	NA	
9	ICP-MS	Rh 103	ORS	He		500	
10					NA	NA	
11	ICP-OES-AV	Y	NA	NA	50	NA	
12						NA	
13	AGILENT-720	Y				NA	
14	ICP-MS	Rh	ORS	He	50	NA	118
15					NA	NA	
16	ICP-MS	Rh	ORS		500	NA	118
17	ICP-MS	Rh	ORS	He	20	NA	118
18						NA	
19					NA	NA	
20	AAS				366	NA	303.4
21	ICP-MS	Rh	UC	He	200	NA	120
23	ICP-OES-AV	NA				NA	189.927
24						NA	
25	ICP-OES-AV	Y	NA	NA	1	NA	189.925

Table 106 Instrument Conditions Sr

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Rh	NA	NA	NA	NA	
2	ICP-OES-AV	Yb			NA	50	407.771
3	ICP-OES-AV	NA			NA	1	407.771
4	ICP-OES-AV-buffer	Y			NA	100	
5	ICP-MS	Rh	NA	NA	NA	625	88
6	ICP-MS	Ge	ORS	He	NA	20	88
7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
8	ICP-MS	Rh	ORS	He	NA	500	88
9	ICP-MS	Rh 103	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	421.552
11	ICP-OES-AV	Y	NA	NA	NA	50	
12					NA	NA	
13	AGILENT-720	Y			NA		
14	ICP-MS	Rh	ORS	He	NA	50	88
15					NA		
16	ICP-MS	Y	ORS		NA	500	88
17	ICP-MS	Rh	ORS	He	NA	20	88
18					NA		
19					NA		
20					NA	NA	
21					NA	NA	
23	NA	NA			NA		
24					NA	NA	
25	ICP-OES-AV	Y	NA	NA	NA	NA	421.552

Table 107 Instrument Conditions Th

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS				NA	250	
2					NA	NA	NA
3					NA	1	
4					NA		
5	ICP-MS	Ir	NA	NA	NA	625	232
6	NA	NA	NA	NA	NA	NA	NA
7	N/A	N/A	N/A	N/A	N/A	N/A	N/A
8					NA	NA	
9							
10					NA		
11					NA		
12					NA	NA	
13					NA		
14	ICP-MS	Ir	ORS	He	NA	50	232
15	ICP-MS	Ir 193	ORS		NA		232 m/z
16	ICP-MS	Ir	ORS		NA	500	205
17	ICP-MS	Rh	ORS	He	NA	20	232
18					NA		
19					NA		
20					NA	NA	
21					NA	NA	
23	NA	NA			NA		
24					NA		
25	ICP-OES-AV	Y	NA	NA	NA		

Table 108 Instrument Conditions U

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Ir	NA	NA	NA	250	
2					NA	NA	NA
3	ICP-OES-AV	NA			NA	10	385.957
4					NA		
5	ICP-MS	Ir	NA	NA	NA	625	238
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Ir 193	ORS	HEHe	NA	4000	238 m/z
8					NA	NA	
9	ICP-MS	Lu 175	ORS	He		500	
10					NA		
11					NA		
12					NA	NA	
13	ELAN9000	Bi			NA		
14	ICP-MS	Ir	ORS	He	NA	50	238
15	ICP-MS	Ir 193	ORS		NA		238m/z
16	ICP-MS	Ir	ORS		NA	500	238
17	ICP-MS	Rh	ORS	He	NA	20	238
18					NA		
19					NA		
20					NA	NA	
21	ICP-MS	Tb	UC	He	NA	200	238
23	NA	NA			NA		
24					NA		
25	ICP-OES-AV	Y	NA	NA	NA		

Table 109 Instrument Conditions V

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Sc	UC	He	NA	250	
2					NA	NA	NA
3	ICP-OES-AV	NA			NA	10	292.401
4	ICP-OES-AV-buffer	Y			NA	100	
5	ICP-MS	Sc	UC	He	NA	625	51
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Rh 103	ORS	O2	NA	4000	67 m/z
8					NA	NA	
9	ICP-MS	Sc 45	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	290.881
11	ICP-OES-AV	Y	NA	NA	NA	50	
12					NA	NA	
13	AGILENT-720	Y			NA		
14	ICP-MS	Sc	ORS	He	NA	50	51
15	ICP-OES-RV	Y371	NA		NA		310.229 nm
16	ICP-MS	Sc	ORS		NA	500	51
17	ICP-MS	Rh	ORS	He	NA	20	51
18					NA		
19					NA		
20					NA	NA	
21	ICP-MS	Sc	UC	He	NA	200	51
23	ICP-OES-AV	NA			NA		292.402
24	ICP-AES	Y			NA		292.464
25	ICP-OES-AV	Y	NA	NA	NA	1	292.401

Table 110 Instrument Conditions Zn

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absorbance(nm)
1	ICP-MS	Rh	UC	He	250	250	
2	ICP-OES-AV	Yb			50	NA	213.857
3	ICP-OES-AV	NA			10	10	206.2
4	ICP-OES-AV-buffer	Y			100	100	
5	ICP-MS	Ge	UC	He	625	625	66
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS/MS	Rh103	NA	O2	4000	4000	Zn 66
8					NA	NA	
9	ICP-MS	Sc 45	ORS	He		500	
10	ICP-OES-AV	Lu 261.541			NA	50	206.2
11	ICP-OES-AV	Y	NA	NA	50	50	
12	ICP-OES-AV					NA	
13	AGILENT-720	Y					
14	ICP-MS	Ir	ORS	He	50	50	66
15	ICP-OES-AV	Te214	NA		NA		202.548 nm
16	ICP-MS	Sc	ORS		500	500	66
17	ICP-MS	Rh	ORS	He			64
18							
19					NA		
20	AAS				9.3	NA	213.9
21	ICP-MS	Ga	UC	He	200	200	66
23	ICP-OES-AV	NA					213.857
24	ICP-AES	Y					206.2
25	ICP-OES-AV	Y	NA	NA	1	1	213.857

END OF REPORT