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Proficiency Test Report AQA 18-02 Metals, Nutrients and Exchangeable Bases in Soil

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1 SUMMARY

This report presents the results of the proficiency test AQA 18-02, metals, nutrients and exchangeable bases in soil (compost) and agricultural soil. The study focused on the measurement of the following acid extractable elements: Ag, Al, As, B, Ba, Bi, Ca, Cd, Co, Cr, Cu, Cs, Fe, Hg, K, La, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Rb, S, Se, Sn, Sr, Th, U, V and Zn. Measurement of total carbon (TC), total organic carbon (TOC), total nitrogen (TN), colwell P, electrical conductivity (EC), pH of 1:5 soil / 0.01 M $CaCl_2$ extract and exchangeable bases (Ca^{2+}, Mg^{2+}, Na^{+}, K^{+}) in 1M NH_4Cl extract was also included in the program.

The sample set consisted of two dried compost samples and one dried agricultural soil sample.

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 476 results, 428 (90%) returned a satisfactory score of $|z| \leq 2$.

Of 476 E_n-scores, 389 (82%) were satisfactory with $|E_n| \leq 1$.

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

Low level Cd and Cs were the tests that presented the most analytical difficulty to participating laboratories. ICP-MS was shown to be a much better analytical technique, especially for difficult analytes such as Pb, B, Cd & As, than ICP-OES especially at lower levels.

iii. compare the performance of participant laboratories with their past performance;

Despite different matrices, analytes and analyte concentrations, on average participants' performance remained consistent.

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

Of 497 numerical results, 496 were reported with an expanded measurement uncertainty. While everyone is reporting an expanded measurement uncertainty the En-scores do still demonstrate that not everyone's estimation of uncertainty is realistic. An example of estimating measurement uncertainty using only proficiency testing data is given in Appendix 3.

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

The study samples were checked for homogeneity and stability and are well characterised, both by in-house testing and from the results of the proficiency round. Surplus test samples are available for sale.

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 18-02 is the 22nd 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 the determination of inorganic analytes in soil;
- compare the performance of participant laboratories with their past performance;
- develop the practical application of traceability and measurement uncertainty; and
- produce materials that can be used in method validation and as control samples.

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 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 forty-five tests were selected from those for which an investigation level is published in the Guidelines 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

Sixteen laboratories participated and fifteen submitted results.

The timetable of the study was:

Invitations issued:	03 February 2018
Samples dispatched:	26 February 2018
Results due:	23 March 2018
Interim report issued:	28 March 2018

3.3 Test Material Specification

Three samples were provided for analysis:

Samples S1 and S2 were 25 g of dried, unfortified, compost (soil, leaves, grass and roots).

Sample S3 was 50 g of dried agricultural soil.

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 except La as the same preparation procedure was followed as in previous studies.[1] The results from the partial homogeneity testing are reported in the present study as the homogeneity value.

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 for 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 test samples were stored at ambient temperature prior to dispatch.

The samples were dispatched by courier on 26 February 2018.

The following items were packaged with the samples:

- a covering letter which included a description of the test samples and instructions for participants; and
- a form to confirm the receipt and condition of 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.
- Take care when adding acid to Samples S1 and S2 as these samples are high in carbonates and may froth and bubble vigorously.

- For Sample S3 for determination of the exchangeable bases (Ca^{2+}, Mg^{2+}, Na^{+}, K^{+}) - 1M NH_4Cl extract, participants are asked to use the Method 15A1 as defined by Rayment, G.E. and David, J. L in "Soil Chemical Methods-Australia".
- For S1 and S2 report results for acid extractable elements on as received basis in units of mg/kg.
- For S3 report results on as received basis in units of (cmol(+)/kg) for exchangeable bases (Ca^{2+}, Mg^{2+}, Na^{+}, K^{+}) - 1M NH_4Cl extract. Except for EC, for all the other tests, report results on as received basis in units of mg/kg. EC results are to be reported in units of µS/cm.

SAMPLE S1		SAMPLE S2		SAMPLE S3	
Test acid extractable	Approximate Conc. Range mg/kg	Test acid extractable	Approximate Conc. Range mg/kg	Test	Approximate Conc. Range mg/kg
As	0.5-10	Ag	1-20	Ba (acid extractable)	50-1250
Cd	0.2-4	Al	750-15000	Ca (acid extractable)	1500-30000
Cr	5-100	B	2-40	Colwell P	250-5000
Cu	25-500	Ba	50-1000	EC	200-800 µS/cm
Hg	0.2-4	Bi	0.2-4	Exchangeable Ca-1MNH_4Cl extract*	>5 cmol(+)/kg
Mn	50-1000	Co	0.500-10	Exchangeable Mg-1MNH_4Cl extract*	>0.25 cmol(+)/kg
Mo	0.5-10	Cs	0.2-4	Exchangeable Na-1MNH_4Cl extract*	>0.25 cmol(+)/kg
Ni	5-100	La	0.5-10	Exchangeable K-1MNH_4Cl extract*	>0.25cmol(+)/kg
Pb	5-100	Li	0.5-10	Fe (acid extractable)	1500-30000
Se	0.5-10	Rb	0.5-10	K (acid extractable)	250-5000
Sn	2-40	Sr	25-5	Mg (acid extractable)	250-5000
V	2-40	Th	0.2-4	Na (acid extractable)	25-500
Zn	50-1000	U	0.2-4	P (acid extractable)	250-5000
				pH of 1:5soil/0.01M$CaCl_2$ extract	2-8
				S (acid extractable)	75-1500
				Sr (acid extractable)	20-400
				Total Carbon	5000-100000
				Total Organic Carbon	500-10000
				Total Nitrogen	50-1250

*Method 15A1 as defined by Rayment, G.E. and David, J. L in "Soil Chemical Methods-Australia". Results to be reported in units of (cmol(+)/kg).

- Report results as you would report to a client.
- Please send us all the requested details regarding the test method.
- Return the completed results sheet by 23 March 2018.

3.9 Interim Report

An interim report was emailed to participants on 28 March 2018.

4 PARTICIPANT LABORATORY INFORMATION

4.1 Test Method Summaries

Summaries of test methods are transcribed in Tables 1 to 7. The instruments and settings reported by participants are presented in Appendix 5.

Table 1 Methodology for Acid Extractable Elements

Lab. Code	Method Reference	Sample Mass (g)	Temp. (°C)	Time (min)	Vol. HNO_3 (mL)	Vol. HCl (mL)	Other
1	In House Method	1	95	120	8	4	
2	USEPA 3050/6010/6020/200.7/200.8	1	90-98	60	3	3	
3	USEPA 3051A (Modification)	1	170	15	8 (1:1)	2	
4	In House S6 - referencing APHA 3125	0.4	120	60	2.5	7.5	
5*	USEPA Method 200.2 Revision 2.8	1	95	60	2 (1:1)		2 mL H_2O_2, Cl from 32% HCl stock
6	EPA (Environmental Protection Agency) 1994 Method 200.8	2	109	60	800	400	
7		0.5-1	95-110	120	3	3	
8	USEPA 200.7	1	95	60	2	2	
9	In House Method	5	90	90	5	7	
10	USEPA Method 200.7/200.8/3050/6020/6010	1	95	90	3	3	
11	AS 4479.2-1997, AS 4479.4-1999	0.5	95	120	1	3	
12		2	95	120	2	6	
13	USEPA 3050	3	85	120	10	5	10 mL HNO_3 (1:1)
14*	EPA3050B, 6020B	2	90-95	60	4	12	
16	USEPA 3050/6010/6020/200.7/200.8	1	90-98	90	3	3	
17	US EPA 200.7, 3051, 6010C and 7471B	2	100	60	4	12	
18	In-house based on APHA 3125	0.5	95	30	1	1	
19	US EPA 200.8	1.2	110	30	1	1	

* See Additional Information for Methodology in Table 2

Table 2 Additional information for Acid Extractable Elements

Lab. Code	Additional Information
5	For S1 and S2: All results from ICP-MS (except Sr- possible interference, and aluminium and boron- not reportable on ICP-MS, from ICP-OES). Hg from FIMS. For acid extractable elements: Results from 1 g soil is digested in 2 ml 50% HNO3 and 10ml 20% HCl for 30 minutes on hot block at 95 degree, followed by 2ml 30% H_2O_2 and digested for another 30 minutes before bulking up to 50ml with UHP water.
14	For S2: Li in No Gas Mode

Table 3 Methodology for Total Carbon

Lab. Code	Method Reference	Test Method	Measurement Technique
4	In House S4a (TC) – based on Rayment and Lyons Method 6B3	High Temperature Oxidation	LECO
7		High Temperature Oxidation	Combustion
10	Lyons, D. J. & raiment G.E., 2011. Total soil C-Dumas high temperature combustion	High Temperature Oxidation	Dispersive Infra-Red Detection
15	Total Carbon (6B2b)	High Temperature Oxidation	IR-LECO
17	Rayment, GE & Lyons, DJ 2001 soil chemical method Australasia.	High Temperature Oxidation	Combustion Method

Table 4 Methodology for Total Organic Carbon

Lab. Code	Method Reference	Test Method	Measurement Technique	Additional Information
4	In House S15b (TOC) - based on Rayment and Lyons Method 6B3		LECO	For TOC sample is digested with Sulfurous acid to remove inorganic carbonates
7		High Temperature Oxidation	Combustion	
10	Lyons, D. J. & raiment G.E., 2011. Total soil C-Dumas high temperature combustion	High Temperature Oxidation		Determination of TOC and TC in soils and sediment via combustion and a Non-Dispersive Infra-Red Detector (NDIR)
15	TOC (6B3)	High Temperature Oxidation		Sulfurous acid (H_2SO_3) was used to treat the sample prior to analysis for TOC
16	A. Walkley and I.A. Black, Soil Science, 37, 29-38 (1934)	Chemical Oxidation (Ag_2SO_4 added)	Titration	Walkley-Black method
17	Rayment, GE & Lyons, DJ 2001 soil chemical method Australasia.	High Temperature Oxidation	Combustion Method	

Table 5 Methodology for Colwell P

Lab. Code	Method Reference	Sample Mass (g)	Extraction Solution 0.5 M $NaHCO_3$ Volume (mL)	Shake time (hours)	Measurement Technique
4	In House S3B	0.4	40	16	ICP-OES
7		0.5	50	16	ICP-OES
13	Rayment and Lyons 9B1	1	100	16	Discrete Analyser
15	9B2	1	100	16	FIA

Table 6 Methodology for Total Nitrogen

Lab. Code	Method Reference	Test Method	Measurement Method	Instrument	Additional Information
2		Digestion	Colorimetric – phenate method	Discrete Analyser	TN is calculated from measured TKN and NOx
4	In House S4a - based on Rayment and Lyons Method 6B2b	Combustion	Dumas -High temperature combustion	LECO	
5	Total Kjeldahl Nitrogen: APHA 4500 Norg A & D with Jirka modification 1976	Digestion, $TN=TKN+NO_x$	Colorimetric – salicylate method	Discrete Analyser	NOx is determined from 1:5 soil:water leach using vanadium chloride reduction method. Used $CuSO_4$ as catalyst for TKN digestion.
7		Digestion, Distillation, $TN=TKN+NO_x$	Titrimetric method	Flow Injection Analyser	NOx use FIA, TKN with titrimetric method
10	APHA Latest ED.4500-Norg-D	$TN+TKN+NO_x$	Colorimetric – salicylate method	Discrete Analyser	
13	ASTM D2216-98	Digestion, $TK=TKN+NO_x$	Colorimetric – salicylate method	Discrete Analyser	
15	7A5	Combustion, Dumas	Dumas -High temperature combustion	LECO	
17	Lachat TKN quikchem method 10-107-06-2-H	Digestion, $TK=TKN+NO_x$	Colorimetric – salicylate method	Flow Injection Analyser	

Table 7 Methodology for Exchangeable Bases

Lab. Code	Sample Mass (g)	Shake time (hrs)	Extraction Solution	Extraction Solution Vol. (mL)	Additional Information
2	2.5		1M NH_4Cl	50	
4	2	1	1M NH_4Cl	20	
5	2.5	1	1M NH_4Cl	50	
7	2	2	1M NH_4Cl	40	
10	2.5	1	1M NH_4Cl	50	
12	5	1	1M NH_4Cl	100	
13	5	1	1M NH_4Cl	100	Method Reference: Rayment and Lyons 15D3 & 15N1
15	5	1	1M NH_4Cl	100	
16	2.5	1	1M NH_4Cl	50	
17	5	2.5	1M NH_4Cl	100	in house method based on Method 15B1 and 15B2 as defined by Rayment, G.E. and David, J. L in "Soil Chemical Methods-Australia"

4.2 Basis of Participants' Measurement Uncertainty Estimates

Participants were requested to provide information about the basis of their uncertainty estimates (Tables 8 and 9).

Table 8 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	Top Down - precision and estimates of the method and laboratory bias	Duplicate Analysis	CRM	NATA Technical Note 33
2	Top Down - precision and estimates of the method and laboratory bias	Control Sample-SS		NATA Technical Note 33
3	Top Down - precision and estimates of the method and laboratory bias	Control Sample-CRM Duplicate Analysis	CRM	
4	Top Down - precision and estimates of the method and laboratory bias	Control Sample-RM Duplicate Analysis	CRM Standard Purity	
5	Top Down - precision and estimates of the method and laboratory bias	Control Sample-RM Duplicate Analysis	Recoveries of SS Instrument Calibration	ISO/GUM
6	SD of replicate analyses multiplied by 2 or 3	Control Sample-RM Duplicate Analysis	Recoveries of SS	
7	Top Down - precision and estimates of the method and laboratory bias	Control Sample Duplicate Analysis	Instrument Calibration Recoveries of SS Variance in sample moisture content	Nordtest Report TR537
8*	Top Down - precision and estimates of the method and laboratory bias	Duplicate Analysis Control Sample	CRM Standard Purity Laboratory Bias from PT Studies	NATA Technical Note 33
9	Top Down - precision and estimates of the method and laboratory bias	Control Samples-RM	CRM	NATA Technical Note 33
10	Top Down - precision and estimates of the method and laboratory bias	Control Samples-RM	Recoveries of SS Instrument Calibration	NATA Technical Note 33
11	Calculated from Standard deviation and concentration of long term in house QC samples	Control Sample-RM Duplicate Analysis Instrument Calibration	CRM Standard Purity Instrument Calibration Laboratory Bias from PT Studies	NATA Technical Note 33
12	Bottom Up (ISO/GUM, fish bone/ cause and effect diagram)	Duplicate Analysis Control Sample-CRM Instrument Calibration		ISO/GUM
13	Top Down - precision and estimates of the method and laboratory bias	Control Sample Duplicate Analysis Instrument Calibration	CRM Instrument Calibration	NATA Technical Note 33
14*	See 'Additional Information' section below	Control Sample-CRM Duplicate Analysis Instrument Calibration	CRM	See 'Additional Information' section below

15	Top Down - reproducibility (standard deviation) from PT studies used directly	Duplicate Analysis Control Sample-CRM Instrument Calibration	CRM Laboratory Bias from PT Studies	NATA Technical Note 33
16	Top Down - precision and estimates of the method and laboratory bias	Control Sample-SS	Recoveries of SS	NATA Technical Note 33
17*	See 'Additional Information' section below	Control Sample-CRM	CRM Instrument Calibration Recoveries of SS Laboratory Bias from PT Studies	See 'Additional Information' section below
18	SD of replicate analyses multiplied by 2 or 3	Duplicate Analysis		IANZ technical guide
19	Top Down – reproducibility (standard deviation) from PT studies used directly			NATA Technical Note 33

[a] RM = Reference Material, CRM = Certified Reference Material, SS =Spiked samples. *Additional Information in Table 9.

Table 9 Additional Information for Measurement Uncertainty

Lab. Code	Additional Information
8	We are participating in this trial as a validation procedure
14	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.
17	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.

4.3 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 10.

Table 10 Participants' Comments

Participants' Comments	Study Co-ordinator's Response
The two samples appear to have exactly the same concentrations, we ended up digesting them twice to confirm this. This was a little confusing for us as we thought it was a lab error.	Often our studies include samples prepared from the same material. When the samples have tests in common, this study design allows us to give a feedback to our participants on their within-laboratory repeatability.
For Total Organic Carbon Analysis I think it is important to specify the Exact Method used by the lab. The acid type and the treatment procedure can affect the final TOC results. Current national carbon accounting method specify to use Sulfurous (H_2SO_3) acid to remove carbonates. But if HCl or H_3PO4 is used then the TOC results can be significantly different.	The major problem when acid treatment is used for TOC analysis is uncertainty about the completeness of inorganic carbon removal. Introduction of a pretesting step to establish the right amount of sample to be taken for analyses and the right type and concentration of acid to be used for inorganic carbon removal can help avoid these problems (see subchapter 7.9). Questions regarding the acid used for treatment were included in our previous studies (AQA10-01, AQA10-12, AQA 11-01, AQA 12-01 and AQA 13-05). Because no significant difference was noticed between the participants' results and the acids used by participants, we decided to simplify the questionnaire. More questions regarding the method used for TOC will be included in some of our future studies. Thank you for your feedback.

5 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

5.1 Results Summary

Participant results are listed in Tables 11 to 54 with resultant 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 45.

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

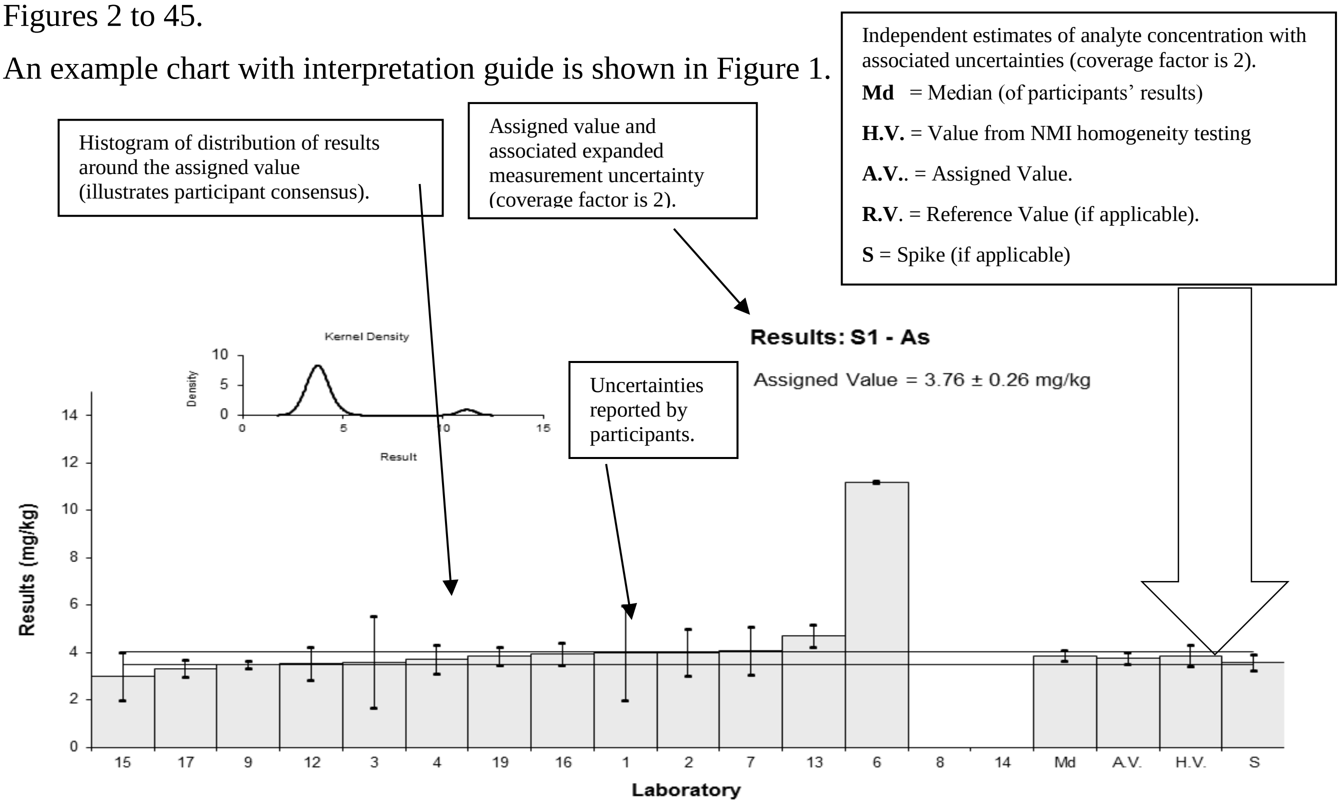


Figure 1 Guide to Presentation of Results

5.2 Assigned Value

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

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 inter-laboratory comparisons, ISO 13528:2015(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 ISO 13528:2015(E).[8]

5.5 Target Standard Deviation

The target standard deviation (σ) is used in the calculation of z-scores and provides scaling for laboratory deviation from the assigned value. It is important to note that the target standard deviation for this study is a fixed value established by the study coordinator and is not the standard deviation of participants' results. The fixed value set for the target standard deviation 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, and is backed up by mathematical models such as Thompson Horwitz equation.[9] By setting a fixed and realistic value for the performance standard deviation, the participant's performance (z-score) does not depend on other participants' performance and can be compared from study to study and against achievable performance. This provides a benchmark for progressive improvement.

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 1 below:

$$z = \frac{(\chi - X)}{\sigma} \qquad \text{Equation 1}$$

where:

- z is z-score
- χ is participant result
- X is the study assigned value
- σ is the target standard deviation from Equation 1

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

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

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 2 below:

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

where:

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

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

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

5.8 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025:2017[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 11

Sample Details

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

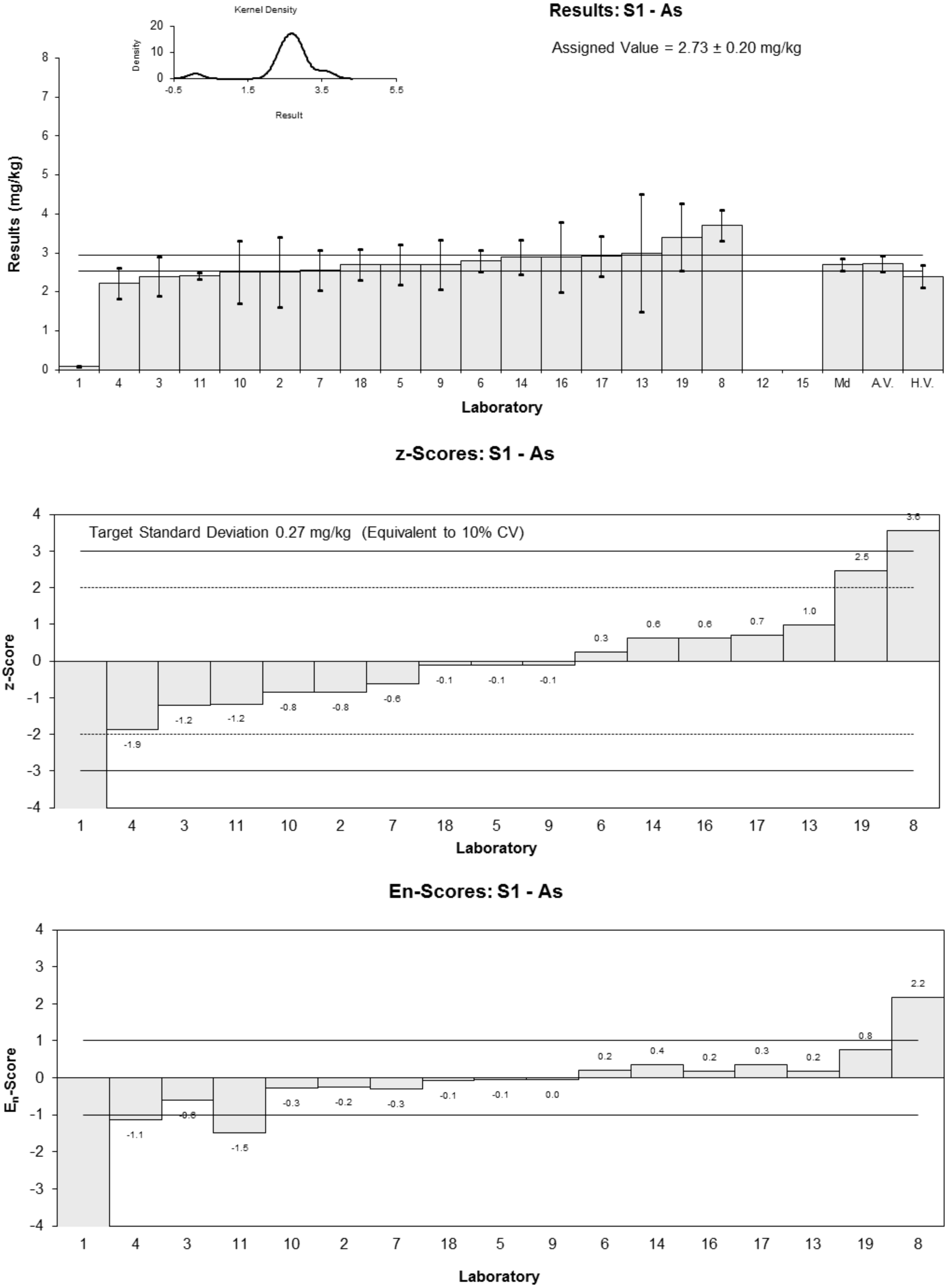
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.08	0.008	-9.71	-13.24
2	2.5	0.9	-0.84	-0.25
3	2.4	0.5	-1.21	-0.61
4	2.22	0.4	-1.87	-1.14
5	2.7	0.51	-0.11	-0.05
6	2.80	0.28	0.26	0.20
7	2.56	0.51	-0.62	-0.31
8	3.7	0.4	3.55	2.17
9	2.7	0.64	-0.11	-0.04
10	2.5	0.8	-0.84	-0.28
11	2.41	0.08	-1.17	-1.49
12	NR	NR		
13	3	1.5	0.99	0.18
14	2.9	0.44	0.62	0.35
15	NR	NR		
16	2.9	0.9	0.62	0.18
17	2.92	0.51	0.70	0.35
18	2.70	0.40	-0.11	-0.07
19	3.4	0.86	2.45	0.76

Statistics

Assigned Value*	2.73	0.20
Spike	Not Spiked	
Homogeneity Value	2.40	0.29
Robust Average	2.70	0.22
Median	2.70	0.15
Mean	2.61	
N	17	
Max.	3.7	
Min.	0.08	
Robust SD	0.32	
Robust CV	12%	

*Robust Average excluding Laboratory 1.



Results: S1 - As
Assigned Value = 2.73 ± 0.20 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
1 4 3 11 10 2 7 18 5 9 6 14 16 17 13 19 8 12 15 Md A.V. H.V.
z-Scores: S1 - As
Target Standard Deviation 0.27 mg/kg (Equivalent to 10% CV)
z-Score
-1.9 -1.2 -1.2 -0.8 -0.8 -0.6 -0.1 -0.1 -0.1 0.3 0.6 0.6 0.7 1.0 2.5 3.6
Laboratory
1 4 3 11 10 2 7 18 5 9 6 14 16 17 13 19 8
En-Scores: S1 - As
E_n-Score
-1.1 -0.6 -1.5 -0.3 -0.2 -0.3 -0.1 -0.1 0.0 0.2 0.4 0.2 0.3 0.2 0.8 2.2
Laboratory
1 4 3 11 10 2 7 18 5 9 6 14 16 17 13 19 8

Figure 2

Table 12

Sample Details

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

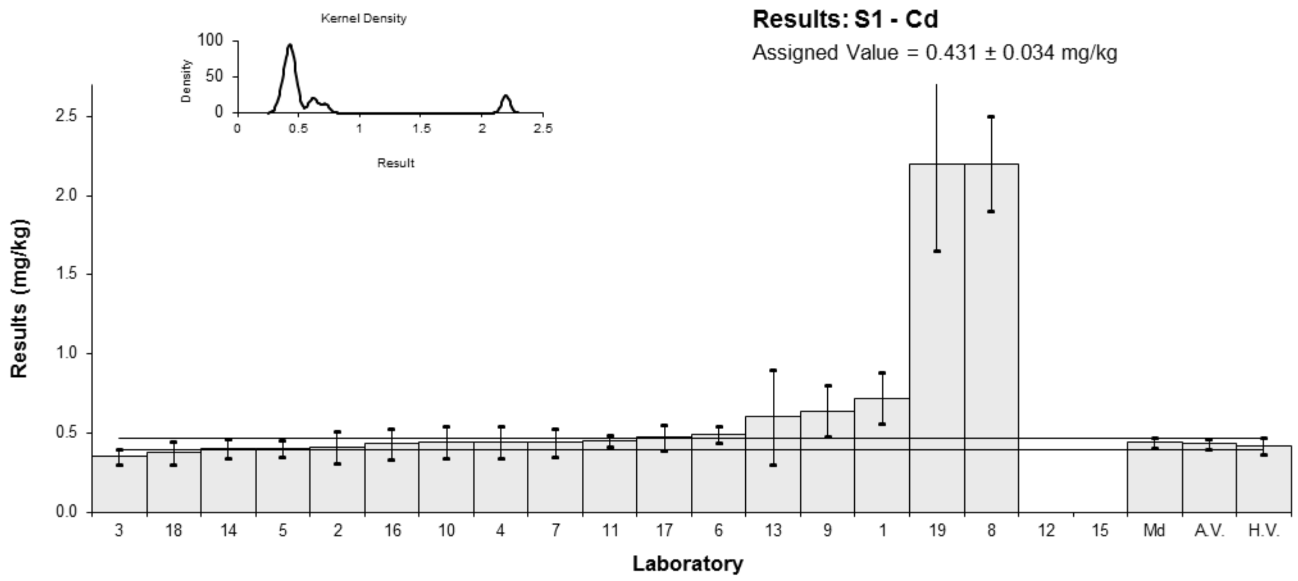
Participant Results

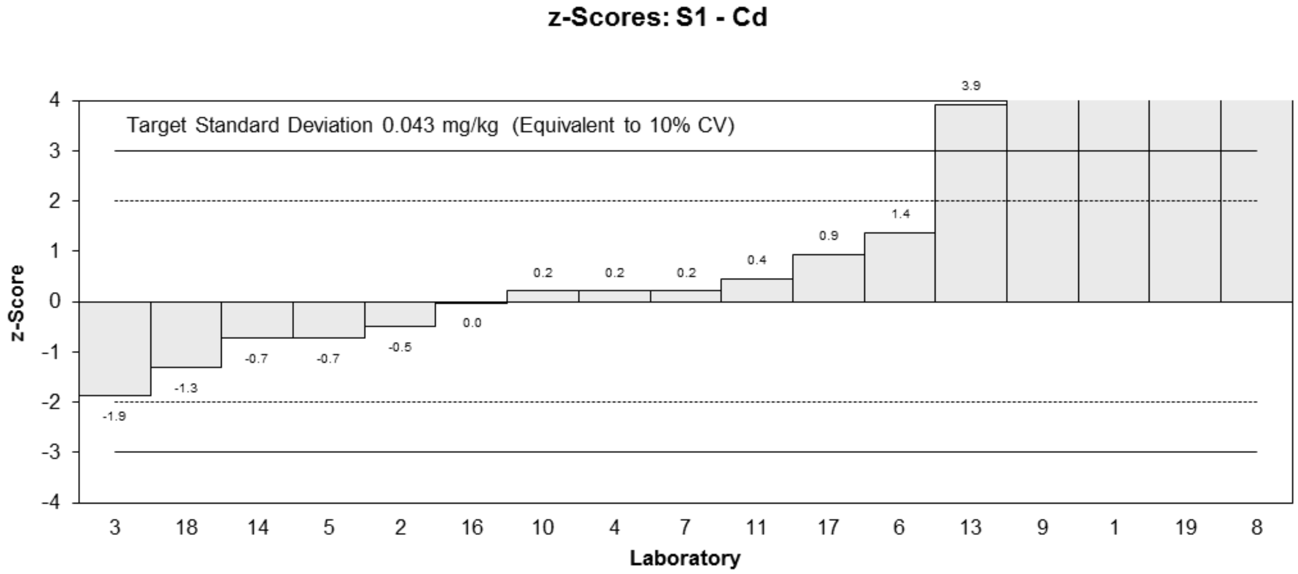
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.72	0.16	6.71	1.77
2	0.41	0.1	-0.49	-0.20
3	0.35	0.05	-1.88	-1.34
4	0.44	0.1	0.21	0.09
5	0.4	0.05	-0.72	-0.51
6	0.49	0.049	1.37	0.99
7	0.44	0.09	0.21	0.09
8	2.2	0.3	41.04	5.86
9	0.64	0.16	4.85	1.28
10	0.44	0.1	0.21	0.09
11	0.45	0.04	0.44	0.36
12	NR	NR		
13	0.6	0.3	3.92	0.56
14	0.40	0.06	-0.72	-0.45
15	NR	NR		
16	0.43	0.1	-0.02	-0.01
17	0.471	0.08	0.93	0.46
18	0.374	0.071	-1.32	-0.72
19	2.2	0.55	41.04	3.21

Statistics

Assigned Value	0.431*	0.034
Spike	Not Spiked	
Homogeneity Value	0.416	0.050
Robust Average	0.497	0.084
Median	0.440	0.031
Mean	0.674	
N	17	
Max.	2.2	
Min.	0.35	
Robust SD	0.048	
Robust CV	9.7%	

*Robust Average excluding Laboratories 1, 8, 9 and 19.


Results: S1 - Cd
Assigned Value = 0.431 ± 0.034 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
3 18 14 5 2 16 10 4 7 11 17 6 13 9 1 19 8 12 15 Md A.V. H.V.


z-Scores: S1 - Cd
Target Standard Deviation 0.043 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-1.9 -1.3 -0.7 -0.7 -0.5 0.0 0.2 0.2 0.2 0.4 0.9 1.4 3.9
3 18 14 5 2 16 10 4 7 11 17 6 13 9 1 19 8

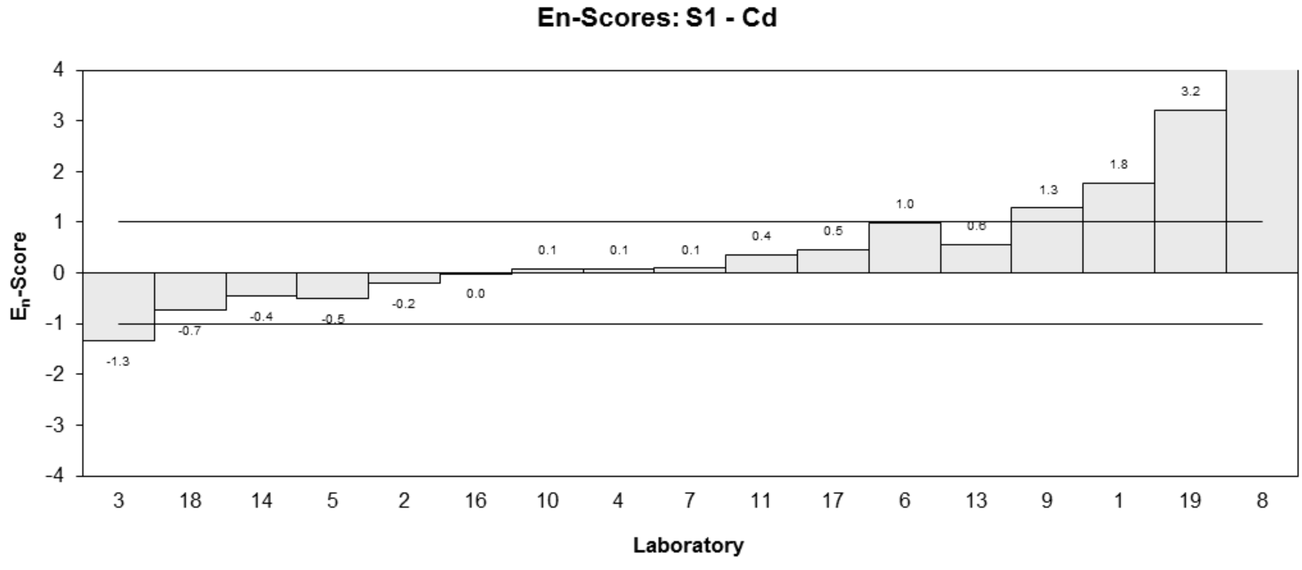

En-Scores: S1 - Cd
E_n-Score
Laboratory
-1.3 -0.7 -0.4 -0.5 -0.2 0.0 0.1 0.1 0.1 0.4 0.5 1.0 0.6 1.3 1.8 3.2
3 18 14 5 2 16 10 4 7 11 17 6 13 9 1 19 8

Figure 3

Table 13

Sample Details

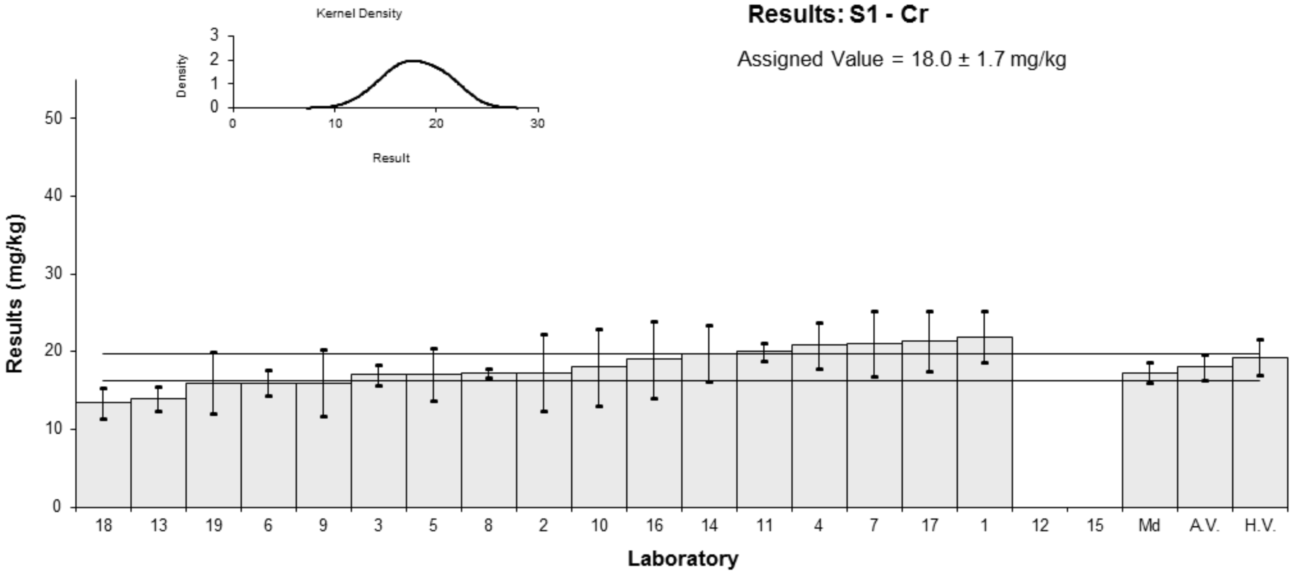
Sample No.	S1
Matrix.	Soil
Analyte.	Cr
Units	mg/kg

Participant Results

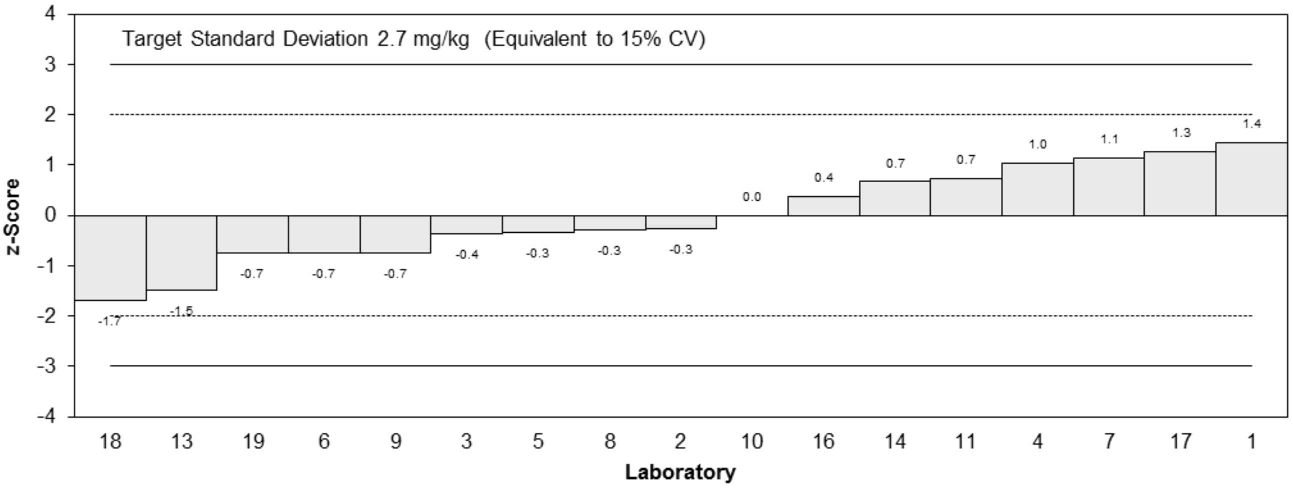
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	21.9	3.28	1.44	1.06
2	17.3	5	-0.26	-0.13
3	17	1.3	-0.37	-0.47
4	20.8	3.0	1.04	0.81
5	17.1	3.4	-0.33	-0.24
6	16.0	1.60	-0.74	-0.86
7	21.1	4.2	1.15	0.68
8	17.2	0.6	-0.30	-0.44
9	16	4.3	-0.74	-0.43
10	18	5	0.00	0.00
11	20.0	1.2	0.74	0.96
12	NR	NR		
13	14	1.54	-1.48	-1.74
14	19.8	3.56	0.67	0.46
15	NR	NR		
16	19	5	0.37	0.19
17	21.4	3.9	1.26	0.80
18	13.4	2.0	-1.70	-1.75
19	16	4.0	-0.74	-0.46

Statistics

Assigned Value	18.0	1.7
Spike	Not Spiked	
Homogeneity Value	19.3	2.3
Robust Average	18.0	1.7
Median	17.3	1.3
Mean	18.0	
N	17	
Max.	21.9	
Min.	13.4	
Robust SD	2.9	
Robust CV	16%	



z-Scores: S1 - Cr



En-Scores: S1 - Cr

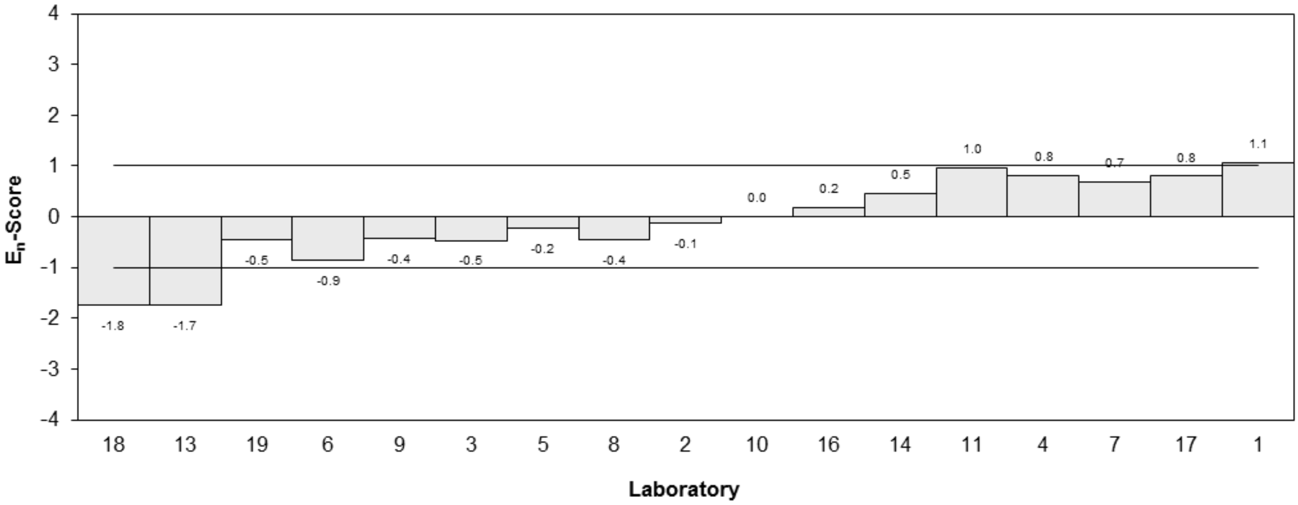


Figure 4

Table 14

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	142	28.4	0.76	0.34
2	123	40	-0.68	-0.22
3	124	8.8	-0.61	-0.67
4	139	15	0.53	0.41
5	119	13.6	-0.98	-0.82
6	142	14.2	0.76	0.61
7	133	27	0.08	0.04
8	151	4.8	1.44	2.04
9	150	25	1.36	0.69
10	120	40	-0.91	-0.29
11	130	10.4	-0.15	-0.15
12	NR	NR		
13	120	14.4	-0.91	-0.73
14	121	25	-0.83	-0.42
15	NR	NR		
16	127	40	-0.38	-0.12
17	151	28	1.44	0.65
18	123	18	-0.68	-0.46
19	130	32	-0.15	-0.06

Statistics

Assigned Value	132	8
Spike	Not Spiked	
Homogeneity Value	130	16
Robust Average	132	8
Median	130	7
Mean	132	
N	17	
Max.	151	
Min.	119	
Robust SD	13	
Robust CV	9.8%	

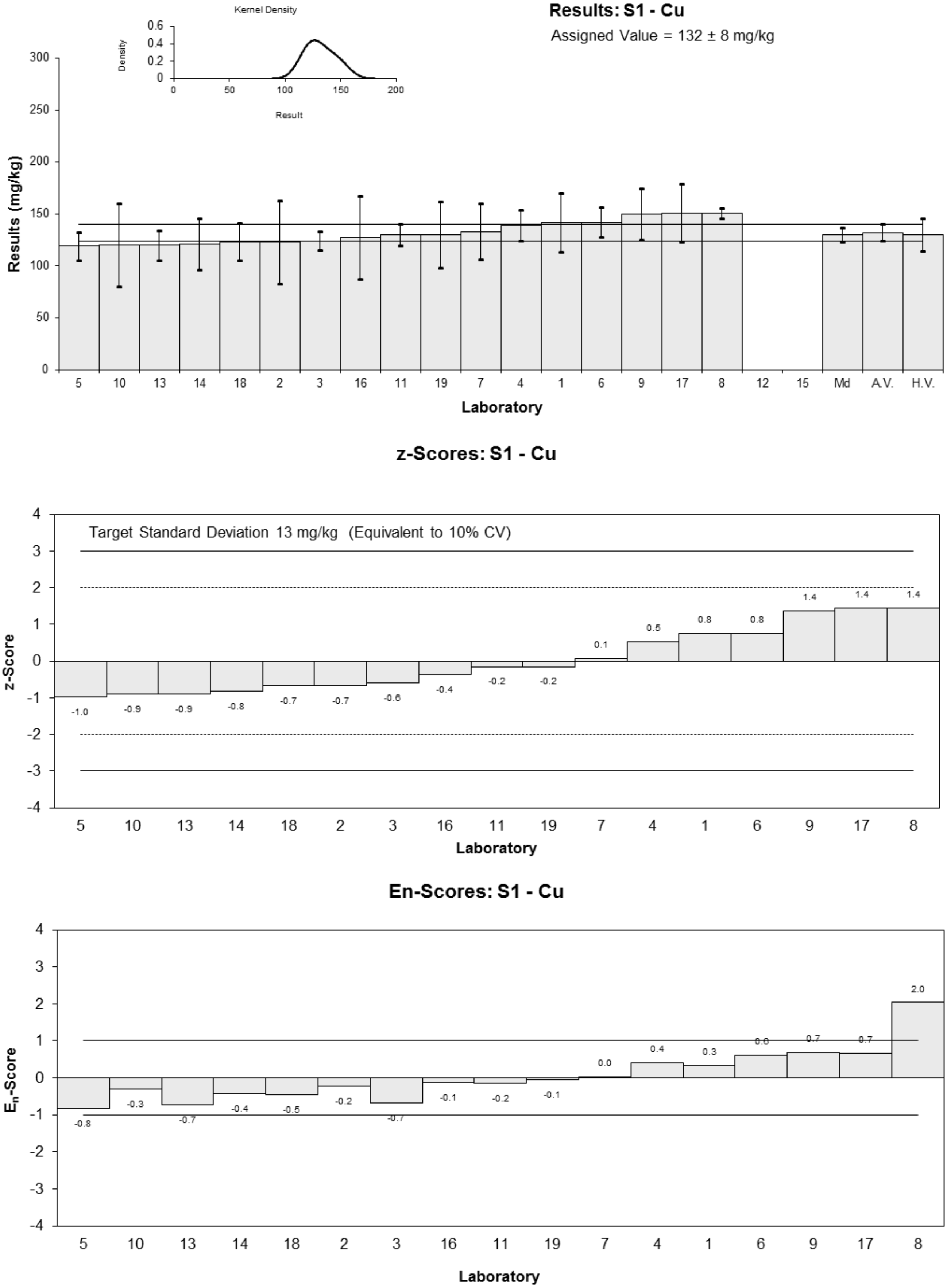

Results: S1 - Cu
Assigned Value = 132 ± 8 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
5 10 13 14 18 2 3 16 11 19 7 4 1 6 9 17 8 12 15 Md A.V. H.V.
z-Scores: S1 - Cu
Target Standard Deviation 13 mg/kg (Equivalent to 10% CV)
z-Score
-1.0 -0.9 -0.9 -0.8 -0.7 -0.7 -0.6 -0.4 -0.2 -0.2 0.1 0.5 0.8 0.8 1.4 1.4 1.4
5 10 13 14 18 2 3 16 11 19 7 4 1 6 9 17 8
Laboratory
En-Scores: S1 - Cu
En-Score
-0.8 -0.3 -0.7 -0.4 -0.5 -0.2 -0.7 -0.1 -0.2 -0.1 0.0 0.4 0.3 0.6 0.7 0.7 2.0
5 10 13 14 18 2 3 16 11 19 7 4 1 6 9 17 8
Laboratory

Figure 5

Table 15

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	0.47	0.1	-0.47	-0.22
3	0.53	0.03	0.75	0.76
4	0.53	0.1	0.75	0.35
5	0.4	0.05	-1.89	-1.48
6	0.50	0.050	0.14	0.11
7	0.49	0.10	-0.06	-0.03
8	NT	NT		
9	0.55	0.23	1.16	0.24
10	0.42	0.1	-1.48	-0.68
11	0.53	0.04	0.75	0.67
12	NR	NR		
13	0.54	0.081	0.95	0.53
14	0.55	0.10	1.16	0.53
15	NR	NR		
16	0.53	0.1	0.75	0.35
17	0.506	0.095	0.26	0.13
18	0.445	0.076	-0.97	-0.56
19	0.35	0.087	-2.90	-1.51

Statistics

Assigned Value	0.493	0.038
Spike	Not Spiked	
Homogeneity Value	0.445	0.053
Robust Average	0.493	0.038
Median	0.506	0.028
Mean	0.489	
N	15	
Max.	0.55	
Min.	0.35	
Robust SD	0.059	
Robust CV	12%	

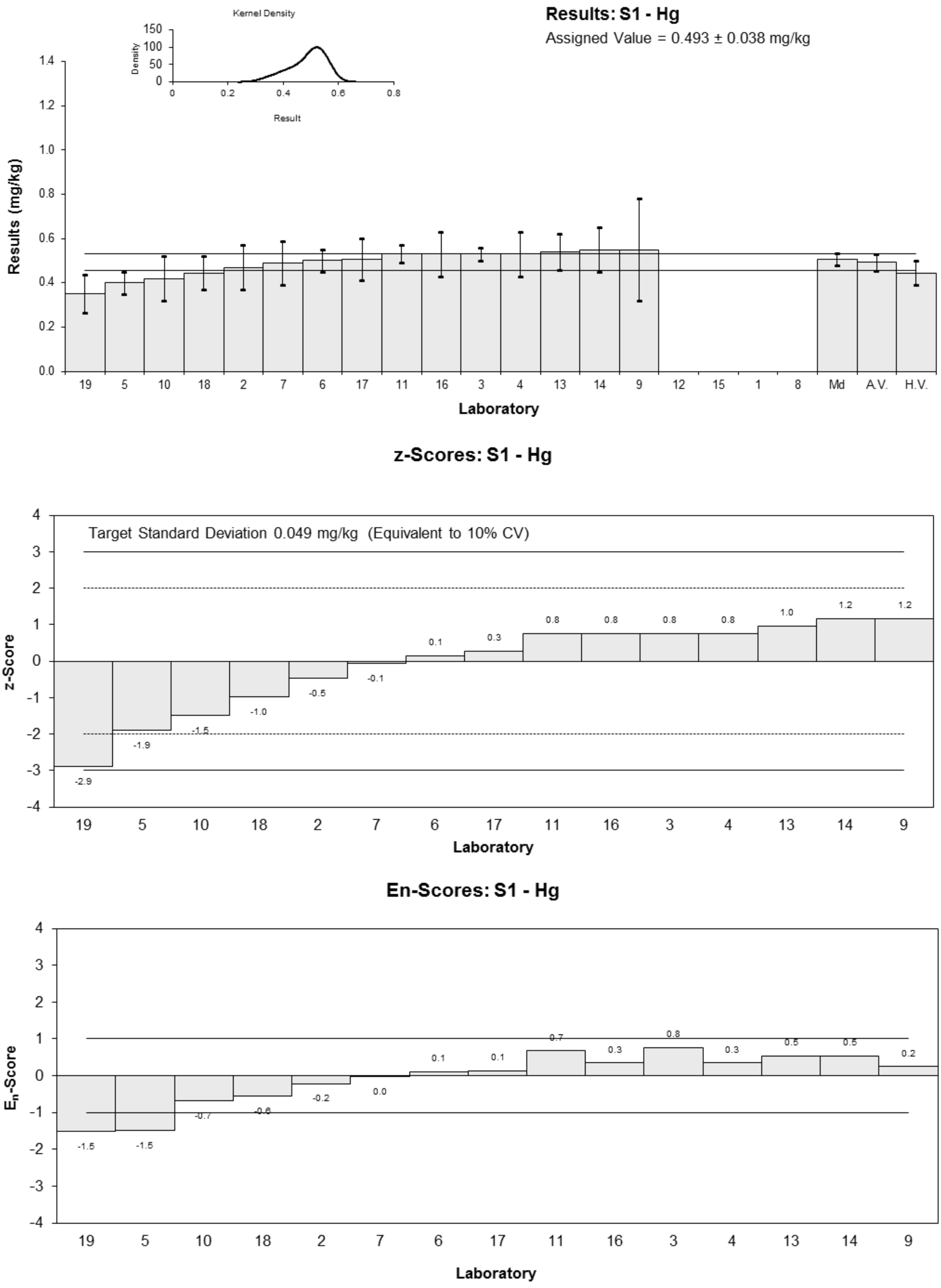

Results: S1 - Hg
Assigned Value = 0.493 ± 0.038 mg/kg
Kernel Density
Results (mg/kg)
Laboratory
z-Scores: S1 - Hg
Target Standard Deviation 0.049 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
En-Scores: S1 - Hg
En-Score
Laboratory

Figure 6

Table 16

Sample Details

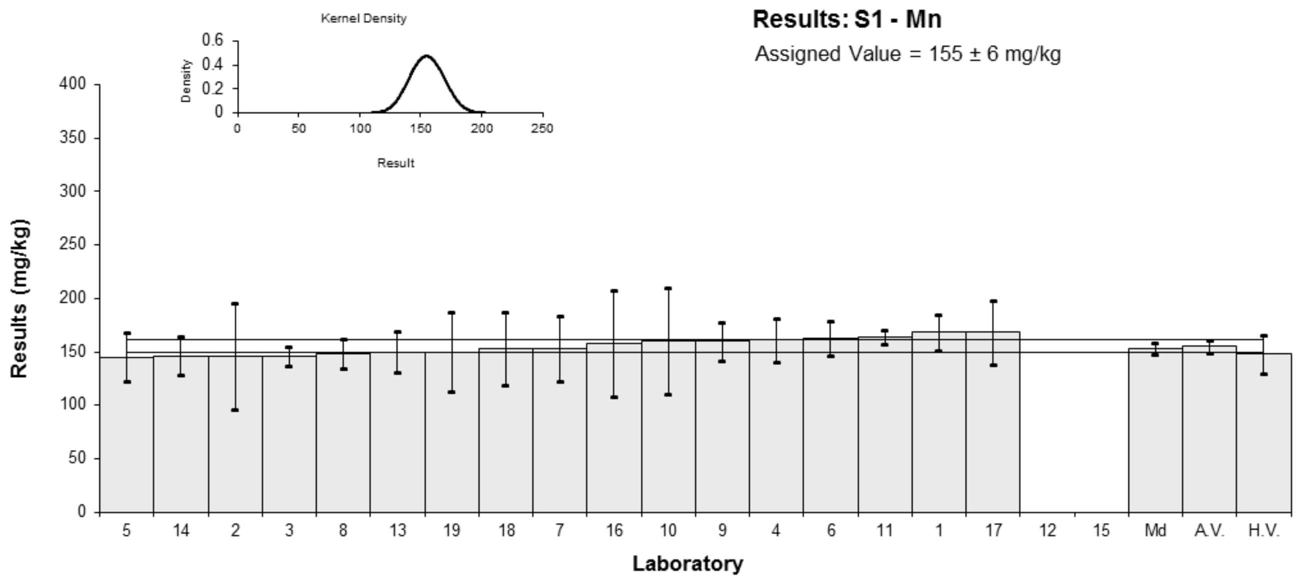
Sample No.	S1
Matrix.	Soil
Analyte.	Mn
Units	mg/kg

Participant Results

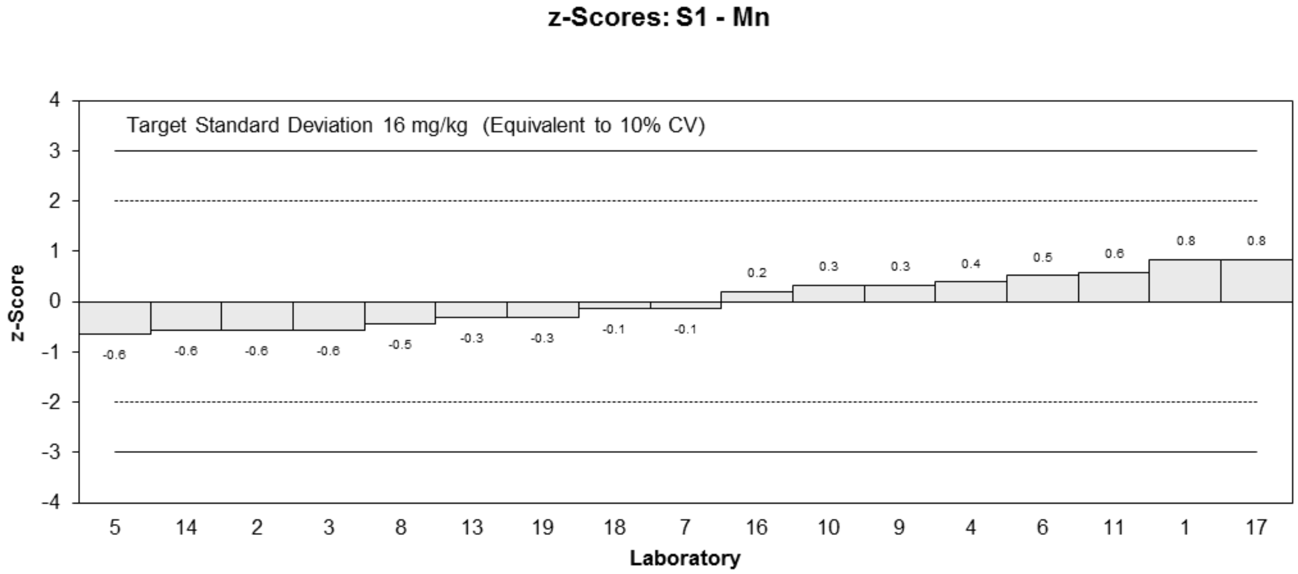
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	168	16.8	0.84	0.73
2	146	50	-0.58	-0.18
3	146	9.3	-0.58	-0.81
4	161	20	0.39	0.29
5	145	22.6	-0.65	-0.43
6	163	16.3	0.52	0.46
7	153	31	-0.13	-0.06
8	148	14	-0.45	-0.46
9	160	18	0.32	0.26
10	160	50	0.32	0.10
11	164	6.5	0.58	1.02
12	NR	NR		
13	150	19	-0.32	-0.25
14	146	17.8	-0.58	-0.48
15	NR	NR		
16	158	50	0.19	0.06
17	168	30	0.84	0.42
18	153	34	-0.13	-0.06
19	150	37	-0.32	-0.13

Statistics

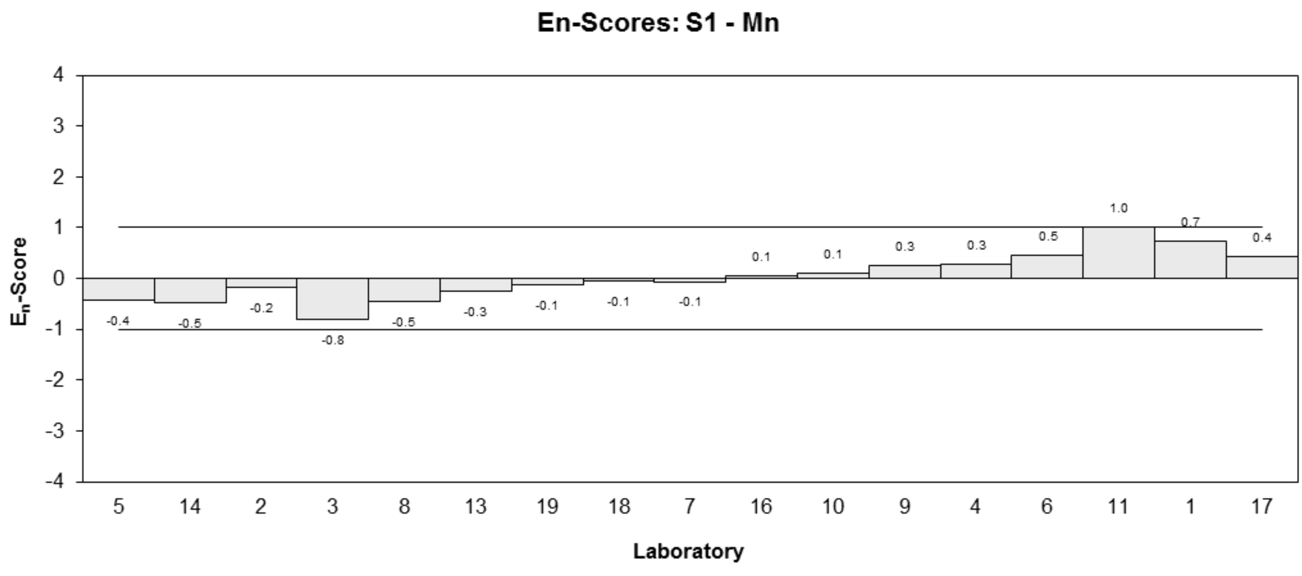
Assigned Value	155	6
Spike	Not Spiked	
Homogeneity Value	148	18
Robust Average	155	6
Median	153	5
Mean	155	
N	17	
Max.	168	
Min.	145	
Robust SD	9.1	
Robust CV	5.9%	



Results: S1 - Mn
Assigned Value = 155 ± 6 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
5
14
2
3
8
13
19
18
7
16
10
9
4
6
11
1
17
12
15
Md
A.V.
H.V.



z-Scores: S1 - Mn
Target Standard Deviation 16 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-0.6
-0.6
-0.6
-0.6
-0.5
-0.3
-0.3
-0.1
-0.1
0.2
0.3
0.3
0.4
0.5
0.6
0.8
0.8



En-Scores: S1 - Mn
En-Score
Laboratory
-0.4
-0.5
-0.2
-0.8
-0.5
-0.3
-0.1
-0.1
-0.1
0.1
0.1
0.3
0.3
0.5
1.0
0.7
0.4

Figure 7

Table 17

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1.72	0.17	2.84	1.98
2	1.3	0.4	-0.30	-0.10
3	1.3	0.05	-0.30	-0.39
4	1.44	0.3	0.75	0.32
5	1.4	0.23	0.45	0.24
6	1.46	0.146	0.90	0.70
7	1.38	0.28	0.30	0.14
8	NT	NT		
9	<5	NR		
10	1.3	0.4	-0.30	-0.10
11	1.39	0.06	0.37	0.46
12	NR	NR		
13	1	0.5	-2.54	-0.67
14	< 5	0.92		
15	NR	NR		
16	1.3	0.4	-0.30	-0.10
17	<5	1		
18	1.30	0.22	-0.30	-0.17
19	1.1	0.29	-1.79	-0.79

Statistics

Assigned Value	1.34	0.09
Spike	Not Spiked	
Homogeneity Value	1.28	0.15
Robust Average	1.34	0.09
Median	1.30	0.08
Mean	1.34	
N	13	
Max.	1.72	
Min.	1	
Robust SD	0.13	
Robust CV	9.7%	

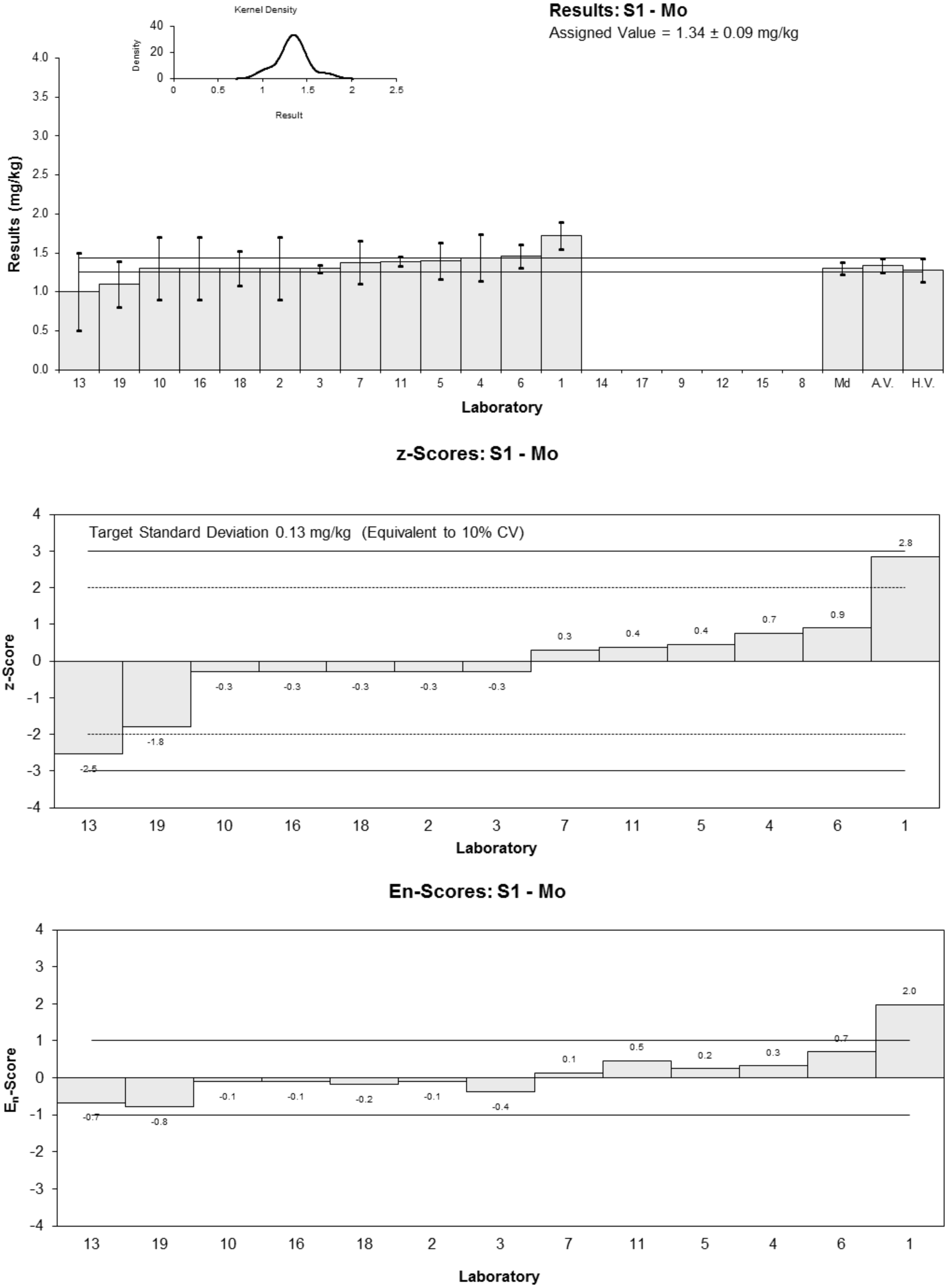

Results: S1 - Mo
Assigned Value = 1.34 ± 0.09 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
13 19 10 16 18 2 3 7 11 5 4 6 1 14 17 9 12 15 8 Md A.V. H.V.
Laboratory
z-Scores: S1 - Mo
Target Standard Deviation 0.13 mg/kg (Equivalent to 10% CV)
z-Score
-2.5 -1.8 -0.3 -0.3 -0.3 -0.3 -0.3 0.3 0.4 0.4 0.7 0.9 2.8
13 19 10 16 18 2 3 7 11 5 4 6 1
Laboratory
En-Scores: S1 - Mo
En-Score
-0.7 -0.8 -0.1 -0.1 -0.2 -0.1 -0.4 0.1 0.5 0.2 0.3 0.7 2.0
13 19 10 16 18 2 3 7 11 5 4 6 1
Laboratory

Figure 8

Table 18

Sample Details

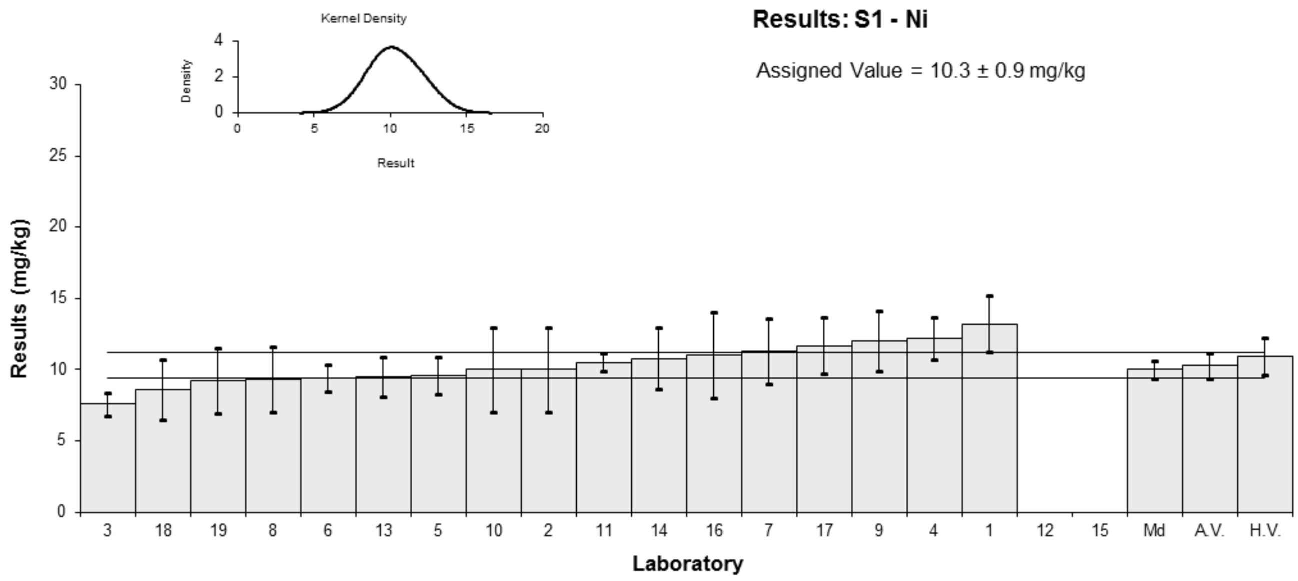
Sample No.	S1
Matrix.	Soil
Analyte.	Ni
Units	mg/kg

Participant Results

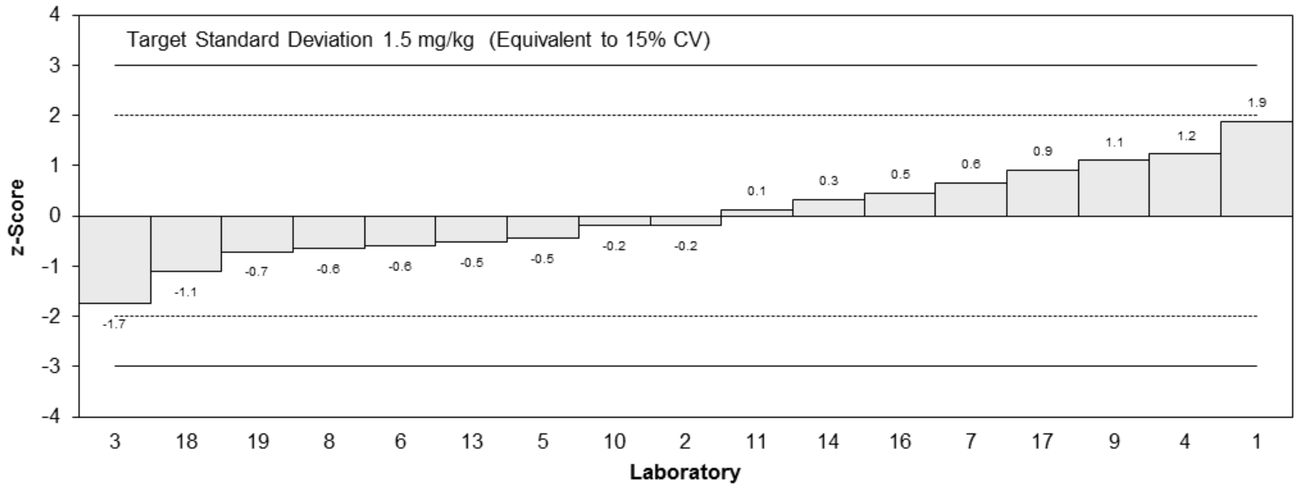
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	13.2	1.98	1.88	1.33
2	10	3	-0.19	-0.10
3	7.6	0.81	-1.75	-2.23
4	12.2	1.5	1.23	1.09
5	9.6	1.3	-0.45	-0.44
6	9.38	0.938	-0.60	-0.71
7	11.3	2.3	0.65	0.40
8	9.3	2.3	-0.65	-0.40
9	12	2.1	1.10	0.74
10	10	3	-0.19	-0.10
11	10.5	0.63	0.13	0.18
12	NR	NR		
13	9.5	1.4	-0.52	-0.48
14	10.8	2.13	0.32	0.22
15	NR	NR		
16	11	3	0.45	0.22
17	11.7	2	0.91	0.64
18	8.58	2.10	-1.11	-0.75
19	9.2	2.3	-0.71	-0.45

Statistics

Assigned Value	10.3	0.9
Spike	Not Spiked	
Homogeneity Value	10.9	1.3
Robust Average	10.3	0.9
Median	10.0	0.6
Mean	10.3	
N	17	
Max.	13.2	
Min.	7.6	
Robust SD	1.5	
Robust CV	15%	



z-Scores: S1 - Ni



En-Scores: S1 - Ni

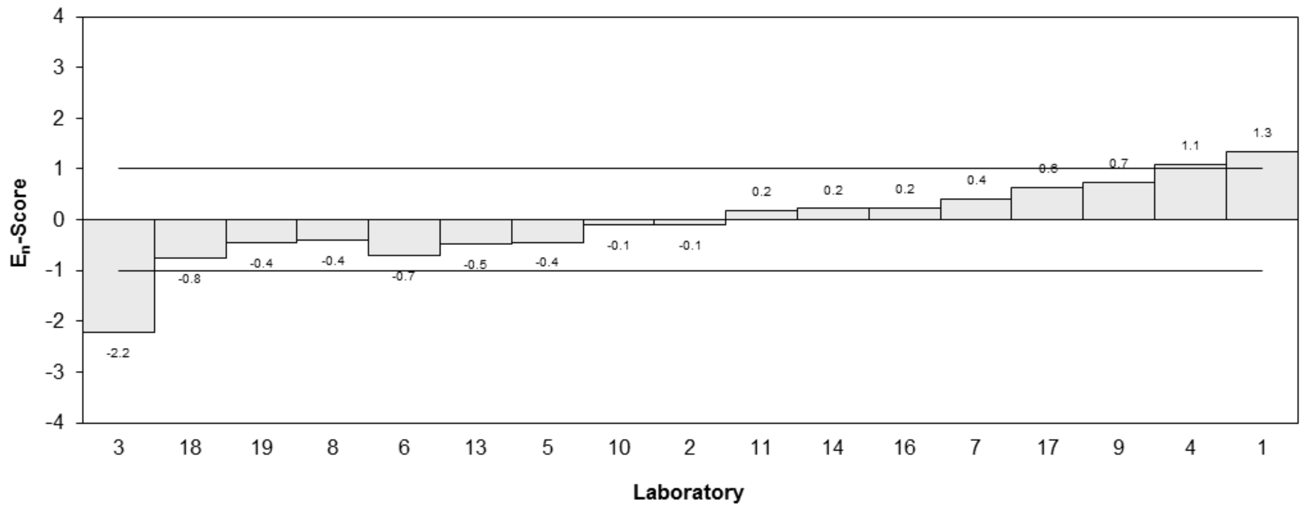


Figure 9

Table 19

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	22.3	3.34	2.82	1.42
2	16	5	-0.80	-0.28
3	16	0.93	-0.80	-1.08
4	17.2	2.0	-0.11	-0.09
5	16.9	2.7	-0.29	-0.18
6	18.4	1.84	0.57	0.49
7	17.8	3.6	0.23	0.11
8	26.8	2.2	5.40	3.95
9	19	3.2	0.92	0.48
10	16	5	-0.80	-0.28
11	17.5	0.88	0.06	0.08
12	NR	NR		
13	16	2.35	-0.80	-0.56
14	18.1	3.13	0.40	0.21
15	NR	NR		
16	16	5	-0.80	-0.28
17	19.6	3.5	1.26	0.61
18	17.0	2.70	-0.23	-0.14
19	18	4.4	0.34	0.13

Statistics

Assigned Value	17.4*	0.9
Spike	Not Spiked	
Homogeneity Value	17.3	2.1
Robust Average	17.6	1.0
Median	17.5	1.1
Mean	18.2	
N	17	
Max.	26.8	
Min.	16	
Robust SD	1.5	
Robust CV	8.5%	

*Robust Average excluding Laboratory 8.

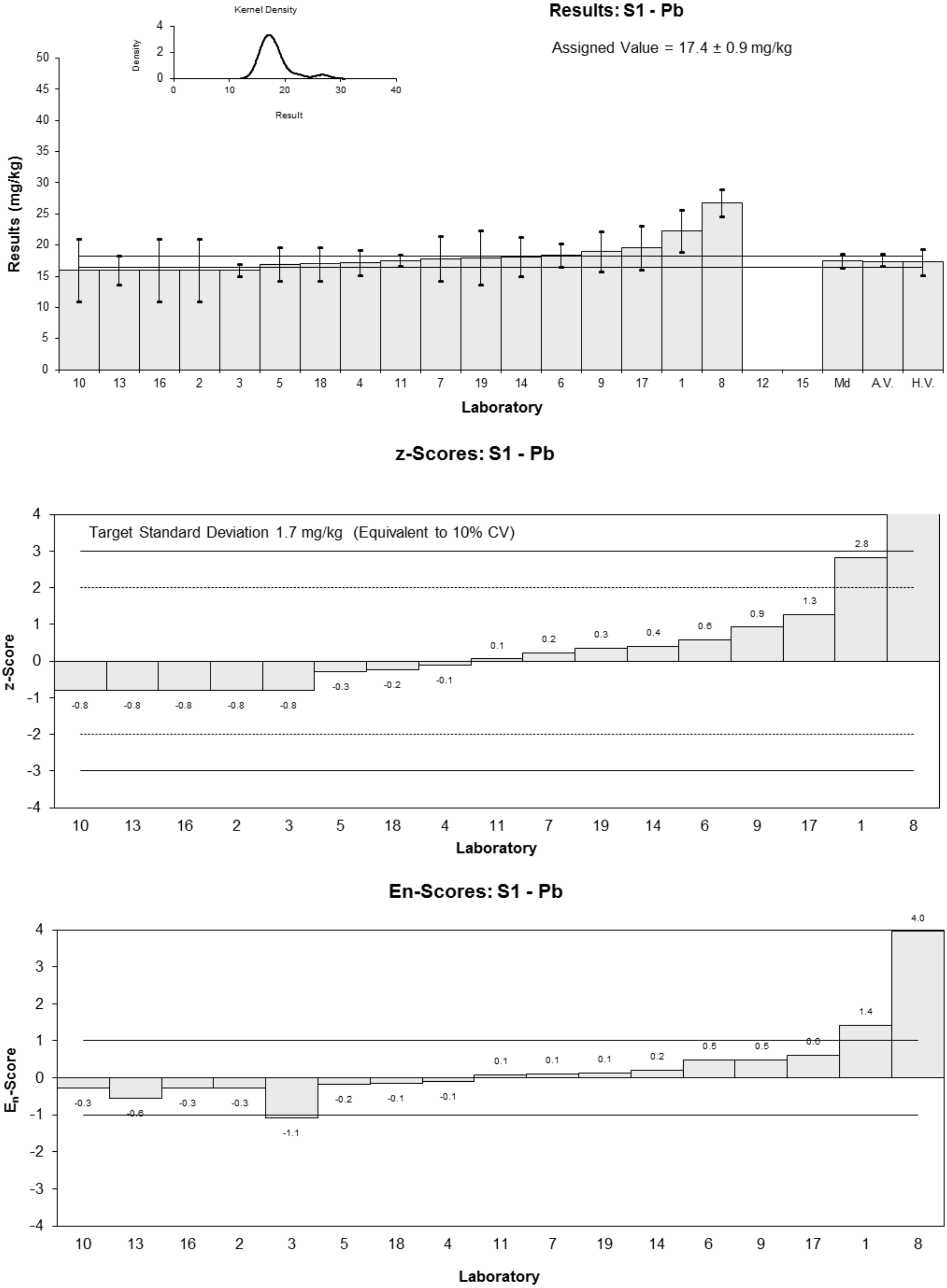

Results: S1 - Pb
Assigned Value = 17.4 ± 0.9 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
10 13 16 2 3 5 18 4 11 7 19 14 6 9 17 1 8 12 15 Md A.V. H.V.
z-Scores: S1 - Pb
Target Standard Deviation 1.7 mg/kg (Equivalent to 10% CV)
z-Score
-0.8 -0.8 -0.8 -0.8 -0.8 -0.3 -0.2 -0.1 0.1 0.2 0.3 0.4 0.6 0.9 1.3 2.8
10 13 16 2 3 5 18 4 11 7 19 14 6 9 17 1 8
Laboratory
En-Scores: S1 - Pb
En-Score
-0.3 -0.6 -0.3 -0.3 -1.1 -0.2 -0.1 -0.1 0.1 0.1 0.1 0.2 0.5 0.5 0.6 1.4 4.0
10 13 16 2 3 5 18 4 11 7 19 14 6 9 17 1 8
Laboratory

Figure 10

Table 20

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	<1	NR		
2	0.85	0.3	0.30	0.08
3	0.7	0.06	-1.52	-1.16
4	0.87	0.2	0.55	0.21
5	<1	0.28		
6	NT	NT		
7	0.84	0.17	0.18	0.08
8	3.0	0.5	26.36	4.28
9	<1	NR		
10	0.99	0.3	2.00	0.53
11	0.72	0.04	-1.27	-1.08
12	NR	NR		
13	<3	NR		
14	< 2	0.28		
15	NR	NR		
16	0.86	0.3	0.42	0.11
17	<2	0.4		
18	0.784	0.140	-0.50	-0.25
19	1.5	0.38	8.18	1.73

Statistics

Assigned Value	0.825*	0.089
Spike	Not Spiked	
Homogeneity Value	0.786	0.118
Robust Average	0.900	0.160
Median	0.860	0.110
Mean	1.11	
N	10	
Max.	3	
Min.	0.7	
Robust SD	0.1	
Robust CV	11%	

*Robust Average excluding Laboratories 8 and 19.

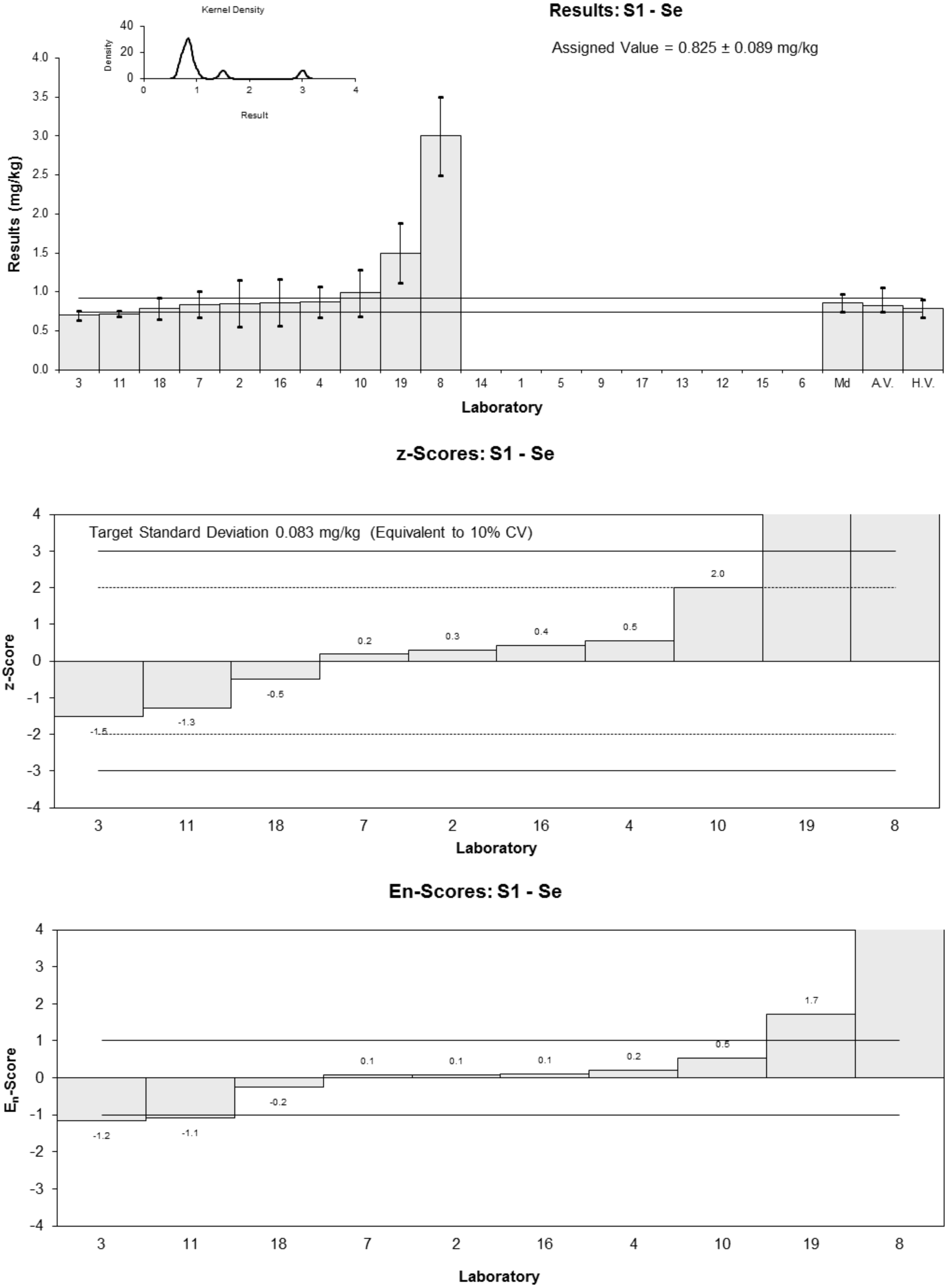

Results: S1 - Se
Assigned Value = 0.825 ± 0.089 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
3 11 18 7 2 16 4 10 19 8 14 1 5 9 17 13 12 15 6 Md A.V. H.V.
z-Scores: S1 - Se
Target Standard Deviation 0.083 mg/kg (Equivalent to 10% CV)
z-Score
-1.5 -1.3 -0.5 0.2 0.3 0.4 0.5 2.0
Laboratory
3 11 18 7 2 16 4 10 19 8
En-Scores: S1 - Se
En-Score
-1.2 -1.1 -0.2 0.1 0.1 0.1 0.2 0.5 1.7
Laboratory
3 11 18 7 2 16 4 10 19 8

Figure 11

Table 21

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	12	3	-0.83	-0.53
3	13	2.4	-0.34	-0.26
4	13.7	1.8	0.00	0.00
5	13.9	2.8	0.10	0.07
6	15.4	1.54	0.83	0.87
7	14.0	2.8	0.15	0.10
8	NT	NT		
9	15	7.3	0.63	0.18
10	10	3	-1.80	-1.15
11	14.3	1.4	0.29	0.33
12	NR	NR		
13	13	1.9	-0.34	-0.31
14	18.6	3.72	2.38	1.25
15	NR	NR		
16	11	3	-1.31	-0.84
17	16.2	3.2	1.22	0.73
18	13.2	4.60	-0.24	-0.11
19	13	3.3	-0.34	-0.20

Statistics

Assigned Value	13.7	1.2
Spike	Not Spiked	
Homogeneity Value	10.8	1.6
Robust Average	13.7	1.2
Median	13.7	0.6
Mean	13.8	
N	15	
Max.	18.6	
Min.	10	
Robust SD	1.9	
Robust CV	14%	

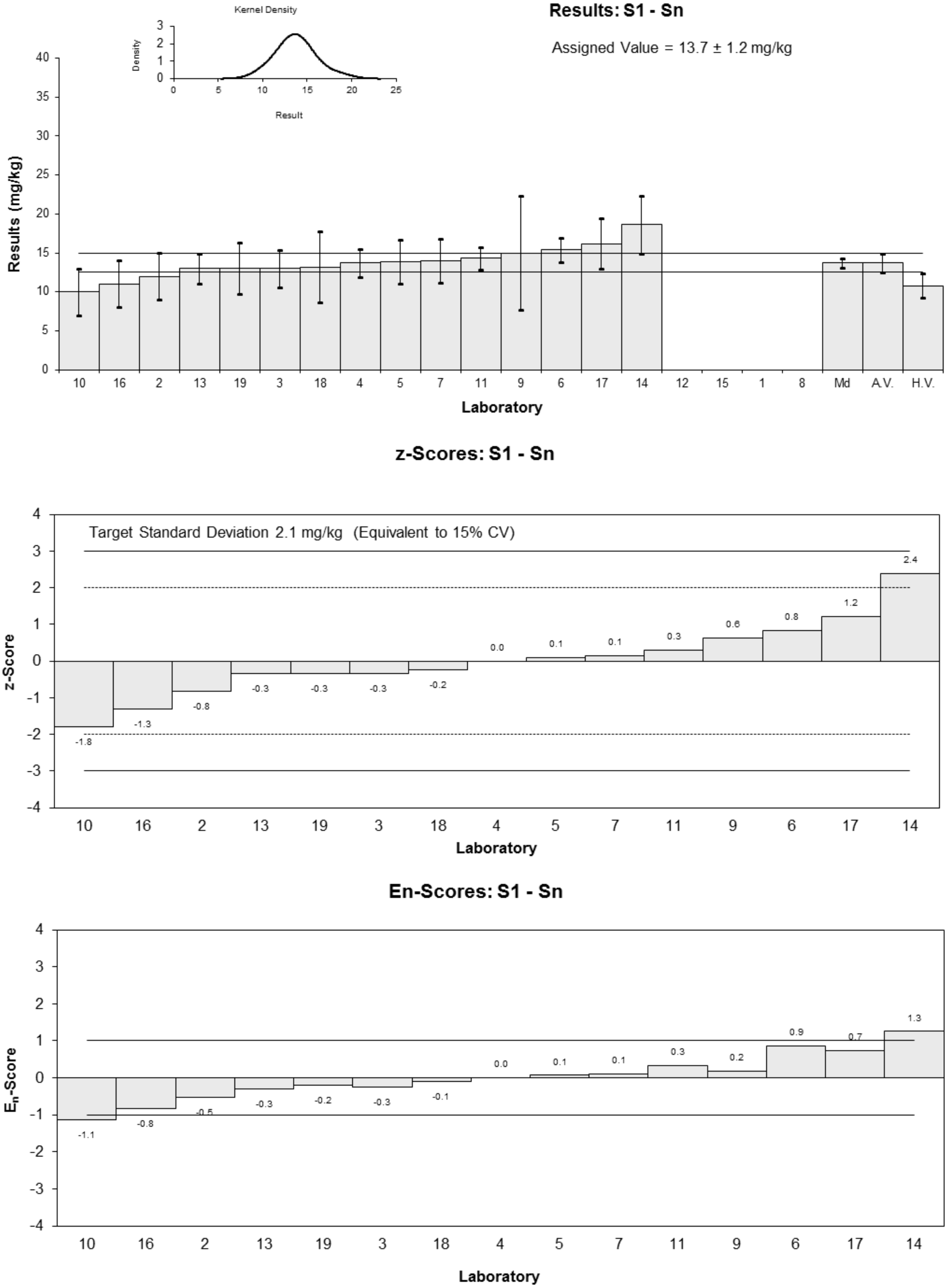

Kernel Density
Density
Result
Results: S1 - Sn
Assigned Value = 13.7 ± 1.2 mg/kg
Results (mg/kg)
Laboratory
10 16 2 13 19 3 18 4 5 7 11 9 6 17 14 12 15 1 8 Md A.V. H.V.
z-Scores: S1 - Sn
Target Standard Deviation 2.1 mg/kg (Equivalent to 15% CV)
z-Score
-1.8 -1.3 -0.8 -0.3 -0.3 -0.3 -0.2 0.0 0.1 0.1 0.3 0.6 0.8 1.2 2.4
10 16 2 13 19 3 18 4 5 7 11 9 6 17 14
Laboratory
En-Scores: S1 - Sn
E_n-Score
-1.1 -0.8 -0.5 -0.3 -0.2 -0.3 -0.1 0.0 0.1 0.1 0.3 0.2 0.9 0.7 1.3
10 16 2 13 19 3 18 4 5 7 11 9 6 17 14
Laboratory

Figure 12

Table 22

Sample Details

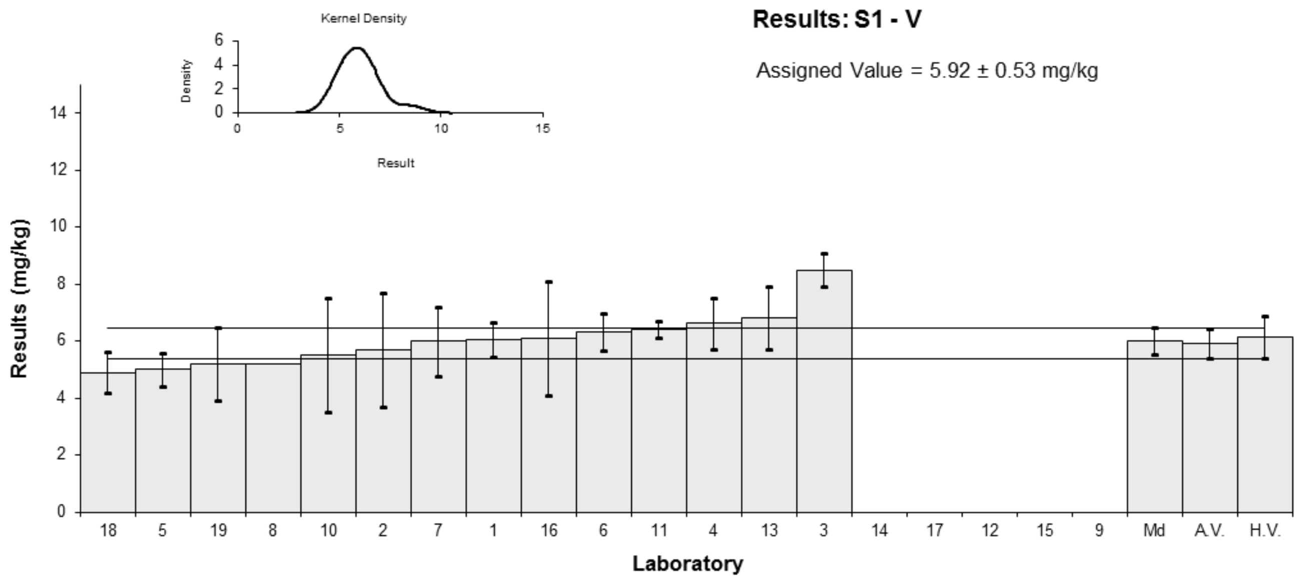
Sample No.	S1
Matrix.	Soil
Analyte.	V
Units	mg/kg

Participant Results

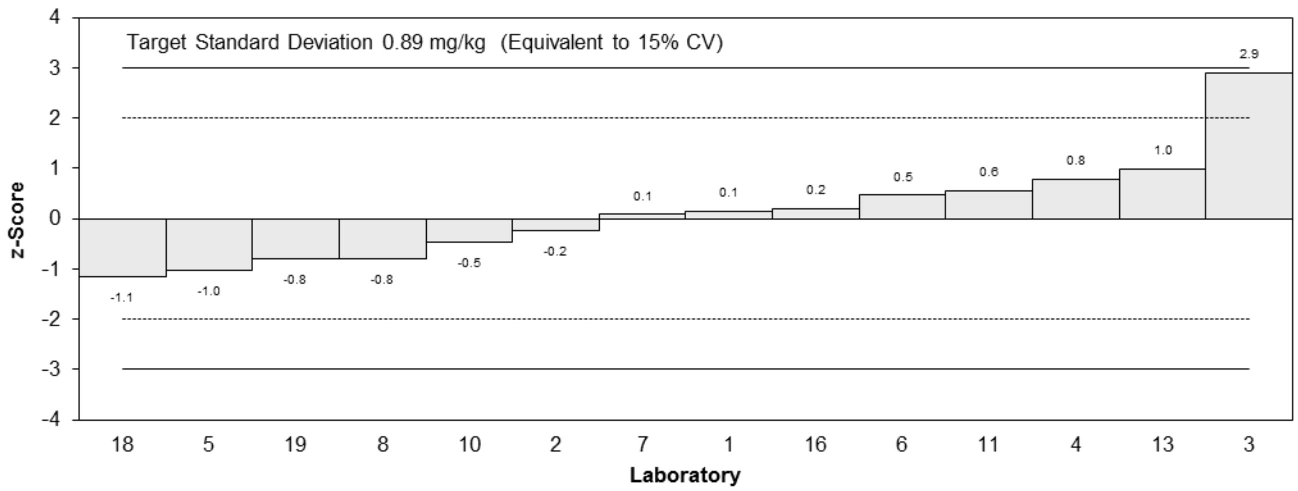
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	6.04	0.60	0.14	0.15
2	5.7	2	-0.25	-0.11
3	8.5	0.6	2.91	3.22
4	6.62	0.9	0.79	0.67
5	5	0.57	-1.04	-1.18
6	6.33	0.65	0.46	0.49
7	5.99	1.20	0.08	0.05
8	5.2	NR	-0.81	-1.36
9	NT	NT		
10	5.5	2	-0.47	-0.20
11	6.41	0.30	0.55	0.80
12	NR	NR		
13	6.8	1.1	0.99	0.72
14	< 10	2.0		
15	NR	NR		
16	6.1	2	0.20	0.09
17	<10	2		
18	4.90	0.73	-1.15	-1.13
19	5.2	1.3	-0.81	-0.51

Statistics

Assigned Value	5.92	0.53
Spike	Not Spiked	
Homogeneity Value	6.15	0.74
Robust Average	5.92	0.53
Median	6.02	0.48
Mean	6.02	
N	14	
Max.	8.5	
Min.	4.9	
Robust SD	0.79	
Robust CV	13%	



z-Scores: S1 - V



En-Scores: S1 - V

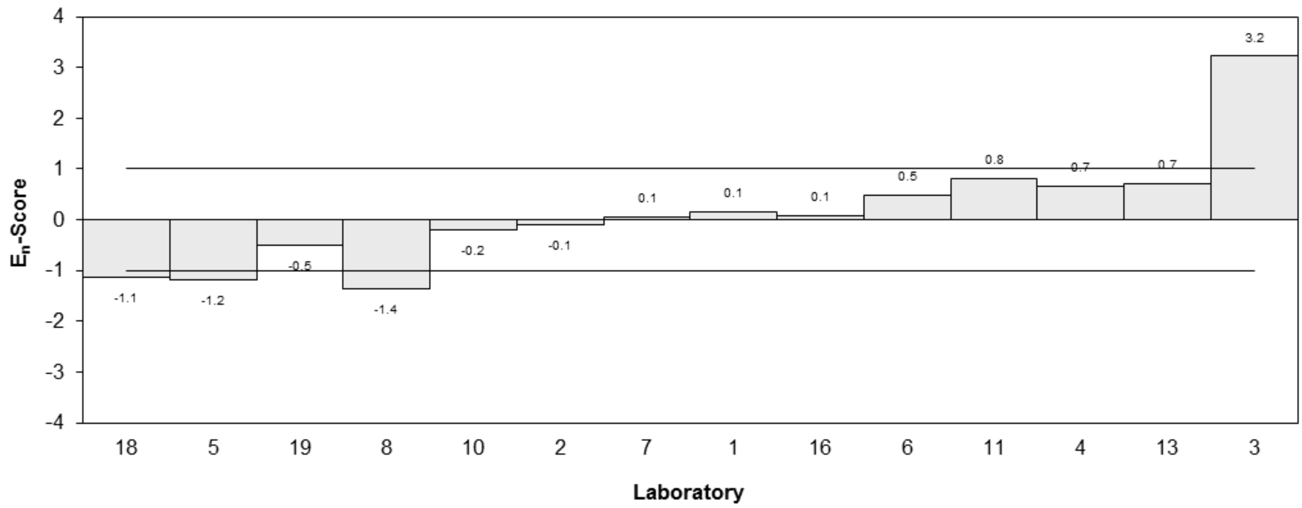


Figure 13

Table 23

Sample Details

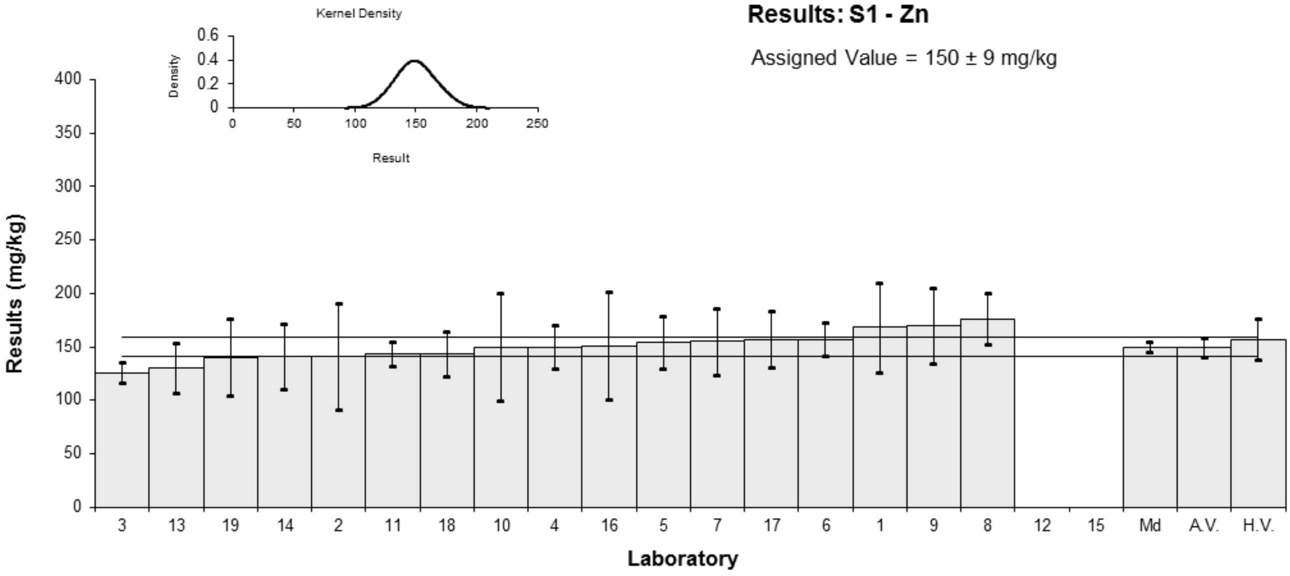
Sample No.	S1
Matrix.	Soil
Analyte.	Zn
Units	mg/kg

Participant Results

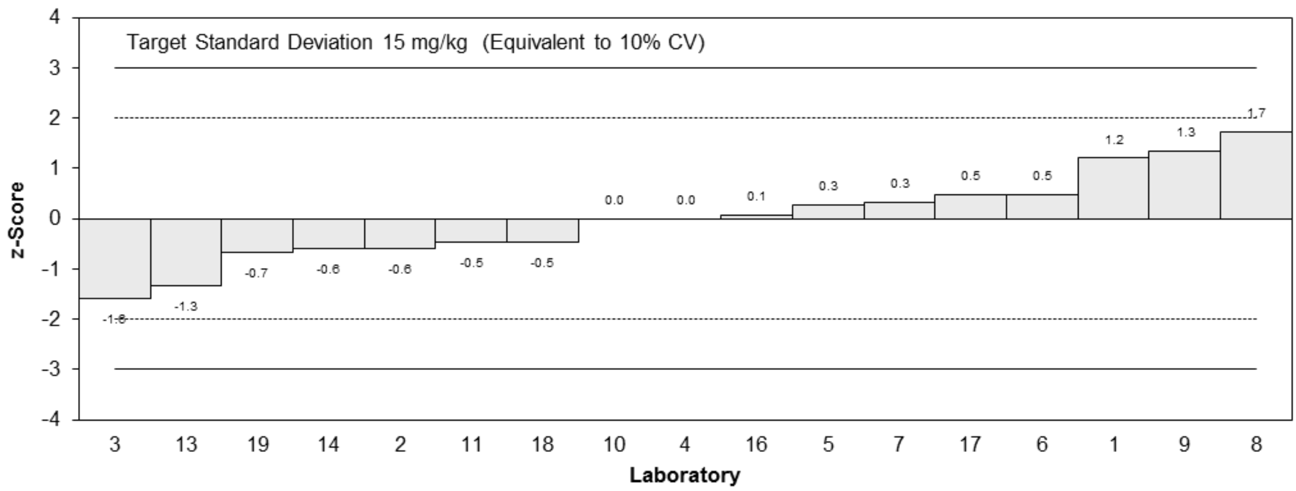
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	168	42	1.20	0.42
2	141	50	-0.60	-0.18
3	126	9.2	-1.60	-1.86
4	150	20	0.00	0.00
5	154	24.5	0.27	0.15
6	157	15.7	0.47	0.39
7	155	31	0.33	0.15
8	176	24	1.73	1.01
9	170	35	1.33	0.55
10	150	50	0.00	0.00
11	143	11.4	-0.47	-0.48
12	NR	NR		
13	130	23.4	-1.33	-0.80
14	141	30	-0.60	-0.29
15	NR	NR		
16	151	50	0.07	0.02
17	157	26	0.47	0.25
18	143	21	-0.47	-0.31
19	140	36	-0.67	-0.27

Statistics

Assigned Value	150	9
Spike	Not Spiked	
Homogeneity Value	157	19
Robust Average	150	9
Median	150	5
Mean	150	
N	17	
Max.	176	
Min.	126	
Robust SD	14	
Robust CV	9.3%	



z-Scores: S1 - Zn



En-Scores: S1 - Zn

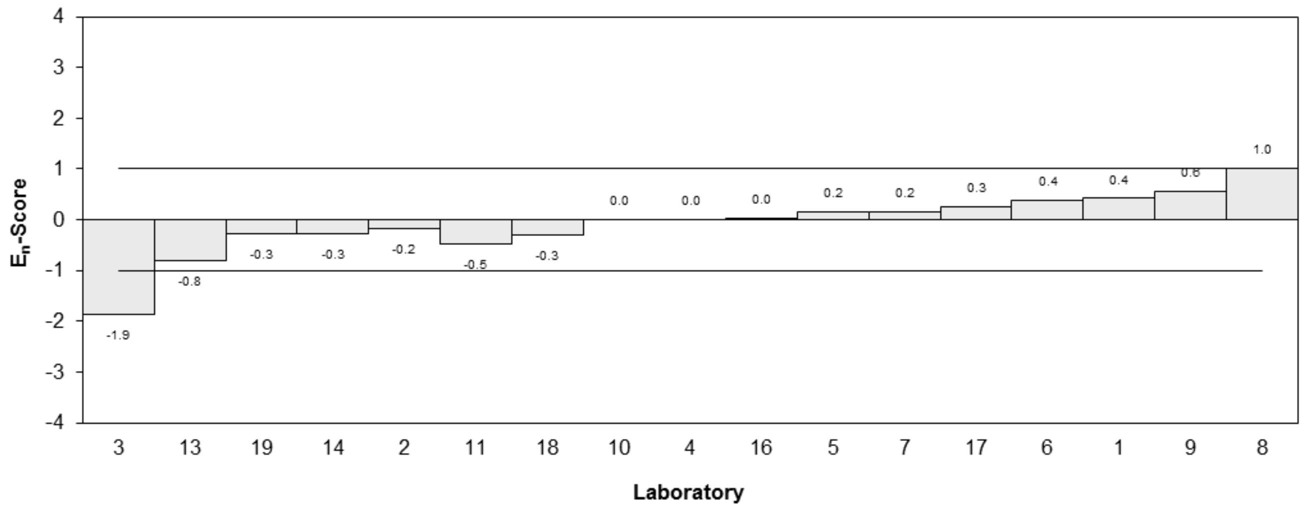


Figure 14

Table 24

Sample Details

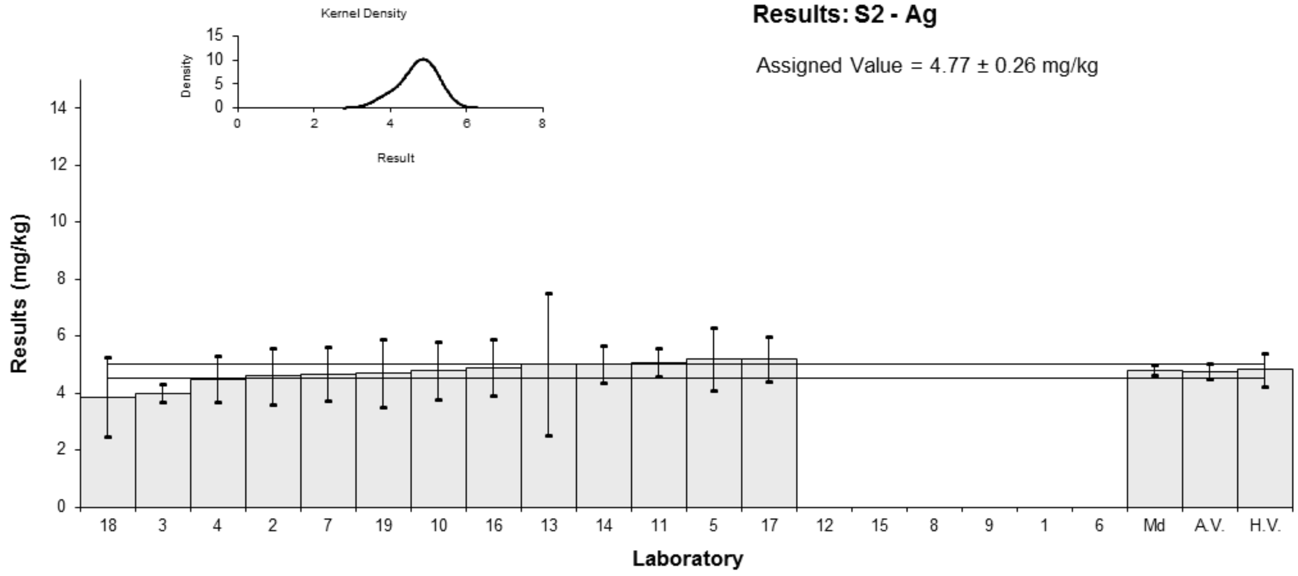
Sample No.	S2
Matrix.	Soil
Analyte.	Ag
Units	mg/kg

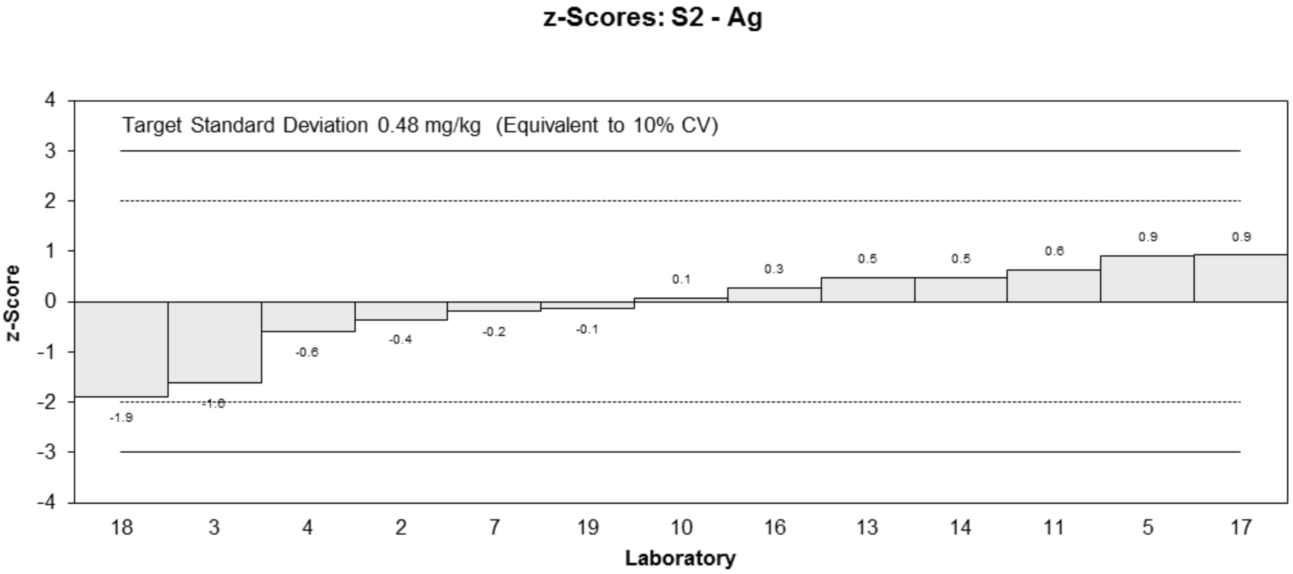
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	4.6	1	-0.36	-0.16
3	4.0	0.3	-1.61	-1.94
4	4.49	0.8	-0.59	-0.33
5	5.2	1.1	0.90	0.38
6	NT	NT		
7	4.68	0.94	-0.19	-0.09
8	NR	NR		
9	NR	NR		
10	4.8	1	0.06	0.03
11	5.07	0.50	0.63	0.53
12	NR	NR		
13	5	2.5	0.48	0.09
14	5.0	0.65	0.48	0.33
15	NR	NR		
16	4.9	1	0.27	0.13
17	5.21	0.78	0.92	0.54
18	3.86	1.40	-1.91	-0.64
19	4.7	1.2	-0.15	-0.06

Statistics

Assigned Value	4.77	0.26
Spike	Not Spiked	
Homogeneity Value	4.82	0.58
Robust Average	4.77	0.26
Median	4.80	0.18
Mean	4.73	
N	13	
Max.	5.21	
Min.	3.86	
Robust SD	0.38	
Robust CV	8%	


Results: S2 - Ag
Assigned Value = 4.77 ± 0.26 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
18 3 4 2 7 19 10 16 13 14 11 5 17 12 15 8 9 1 6 Md A.V. H.V.


z-Scores: S2 - Ag
Target Standard Deviation 0.48 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
18 3 4 2 7 19 10 16 13 14 11 5 17
-1.9 -1.6 -0.6 -0.4 -0.2 -0.1 0.1 0.3 0.5 0.5 0.6 0.9 0.9

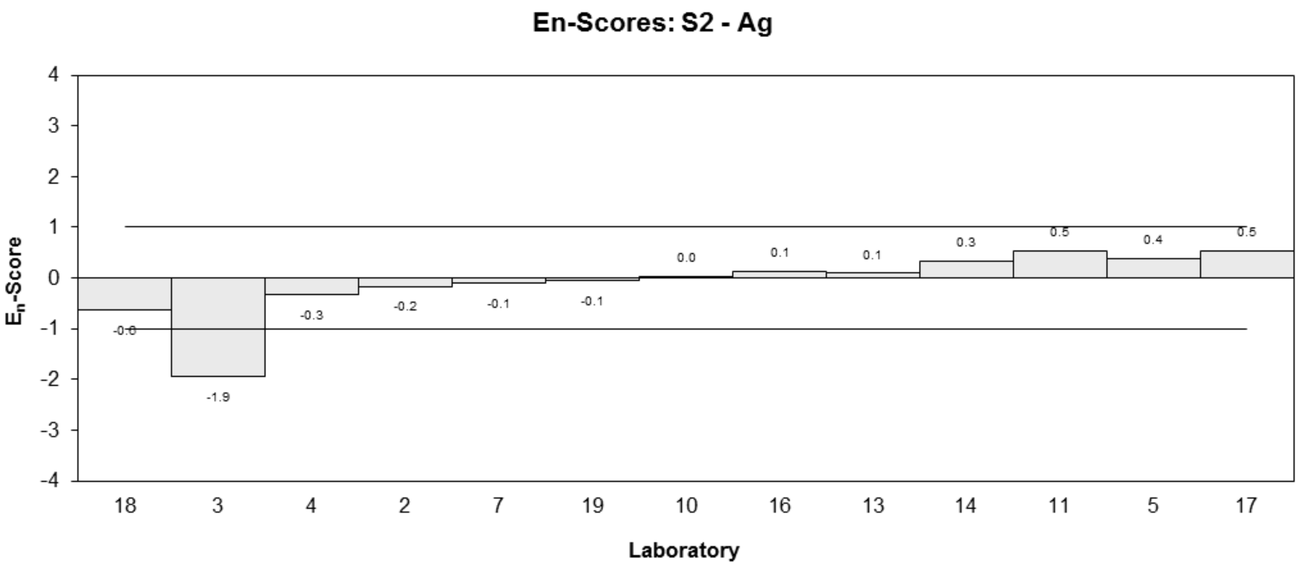

En-Scores: S2 - Ag
En-Score
Laboratory
18 3 4 2 7 19 10 16 13 14 11 5 17
-0.6 -1.9 -0.3 -0.2 -0.1 -0.1 0.0 0.1 0.1 0.3 0.5 0.4 0.5

Figure 15

Table 25

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	2100	525	-0.73	-0.40
2	2200	700	-0.45	-0.20
3	NR	NR		
4	2879	300	1.47	1.05
5	2160	334	-0.56	-0.39
6	NT	NT		
7	2840	570	1.36	0.69
8	NR	NR		
9	NR	NR		
10	2200	700	-0.45	-0.20
11	2800	140	1.24	1.06
12	NR	NR		
13	2800	605	1.24	0.61
14	2984	597	1.76	0.88
15	NR	NR		
16	2000	700	-1.02	-0.45
17	2630	530	0.76	0.41
18	1540	520	-2.32	-1.26
19	1600	400	-2.15	-1.36

Statistics

Assigned Value	2360	390
Spike	Not Spiked	
Homogeneity Value	2820	420
Robust Average	2360	390
Median	2200	540
Mean	2364	
N	13	
Max.	2984	
Min.	1540	
Robust SD	353	
Robust CV	15%	

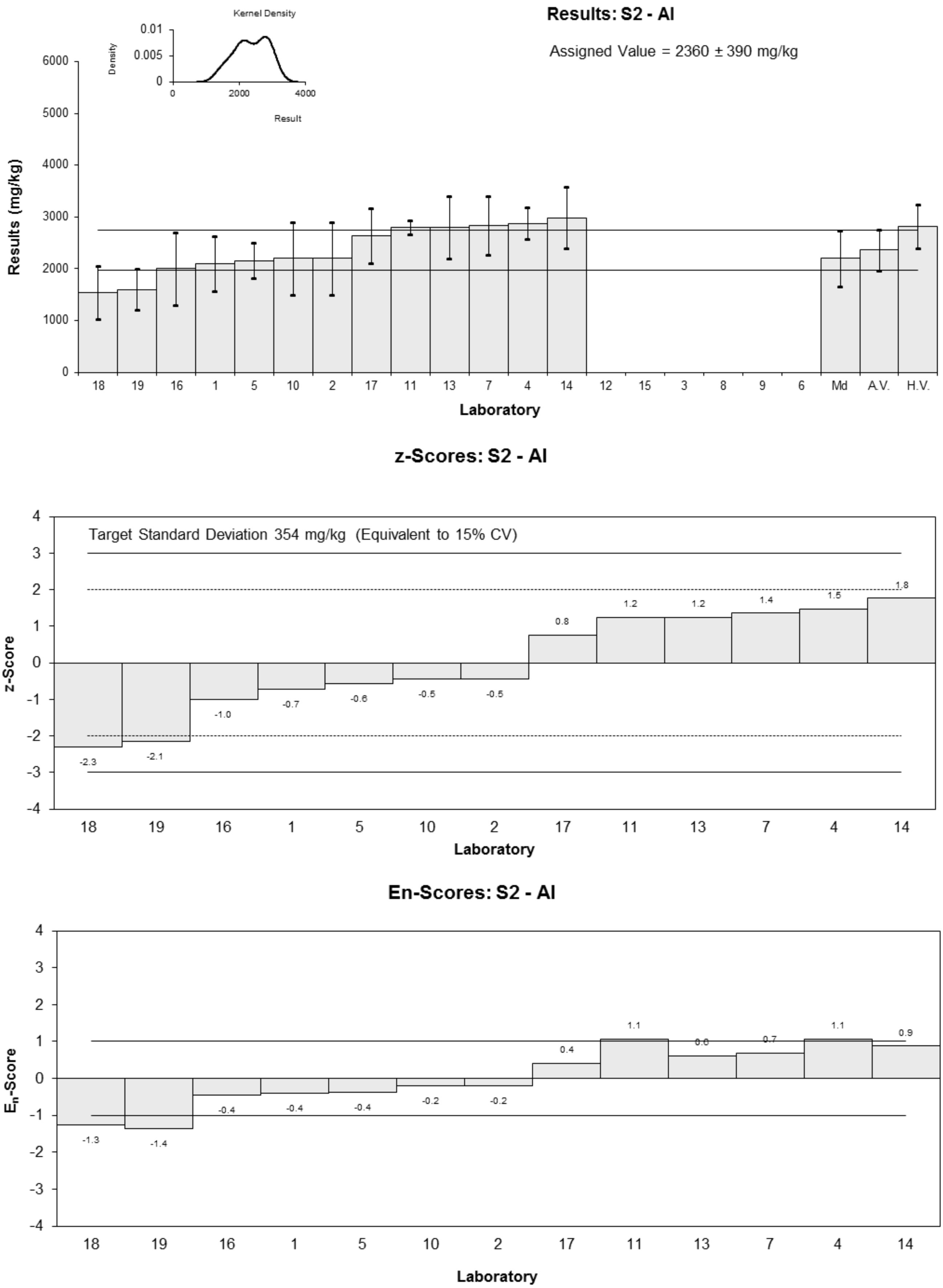

Results: S2 - Al
Assigned Value = 2360 ± 390 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
z-Scores: S2 - Al
Target Standard Deviation 354 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
En-Scores: S2 - Al
E_n-Score
Laboratory

Figure 16

Table 26

Sample Details

Sample No.	S2
Matrix.	Soil
Analyte.	B
Units	mg/kg

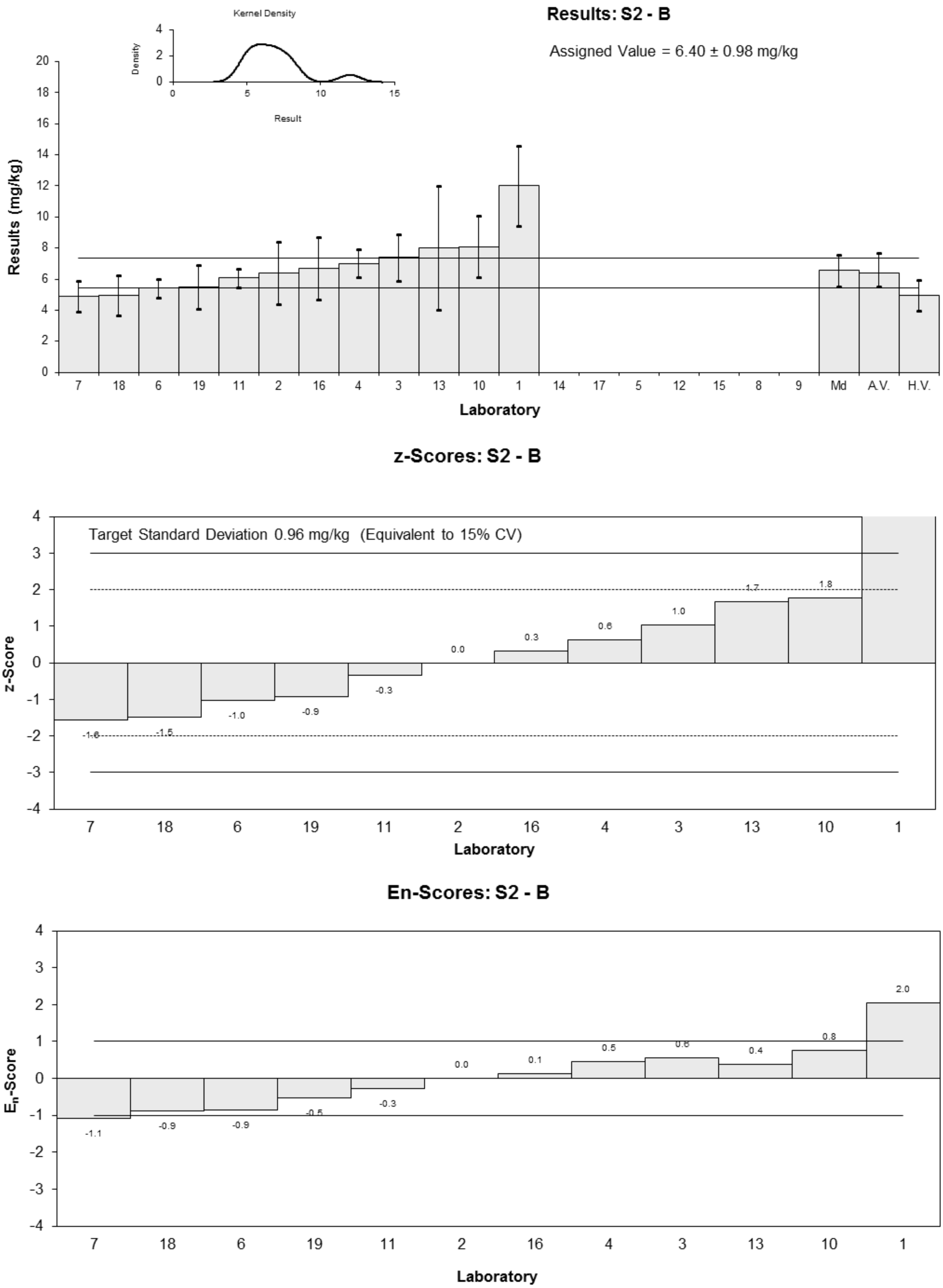
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	12	2.56	5.83	2.04
2	6.4	2	0.00	0.00
3	7.4	1.5	1.04	0.56
4	7.00	0.9	0.62	0.45
5	<50	16		
6	5.41	0.604	-1.03	-0.86
7	4.90	0.98	-1.56	-1.08
8	NR	NR		
9	NR	NR		
10	8.1	2	1.77	0.76
11	6.07	0.60	-0.34	-0.29
12	NR	NR		
13	8	4	1.67	0.39
14	<10	2.0		
15	NR	NR		
16	6.7	2	0.31	0.13
17	<10	2		
18	4.97	1.30	-1.49	-0.88
19	5.5	1.4	-0.94	-0.53

Statistics

Assigned Value	6.40*	0.98
Spike	Not Spiked	
Homogeneity Value	4.94	0.99
Robust Average	6.6	1.1
Median	6.6	1.0
Mean	6.87	
N	12	
Max.	12	
Min.	4.9	
Robust SD	1.3	
Robust CV	20%	

*Robust Average excluding Laboratory 1.



Results: S2 - B
Assigned Value = 6.40 ± 0.98 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
7 18 6 19 11 2 16 4 3 13 10 1 14 17 5 12 15 8 9 Md A.V. H.V.
z-Scores: S2 - B
Target Standard Deviation 0.96 mg/kg (Equivalent to 15% CV)
z-Score
-1.6 -1.5 -1.0 -0.9 -0.3 0.0 0.3 0.6 1.0 1.7 1.8
7 18 6 19 11 2 16 4 3 13 10 1
Laboratory
En-Scores: S2 - B
En-Score
-1.1 -0.9 -0.9 -0.5 -0.3 0.0 0.1 0.5 0.6 0.4 0.8 2.0
7 18 6 19 11 2 16 4 3 13 10 1
Laboratory

Figure 17

Table 27

Sample Details

Sample No.	S2
Matrix.	Soil
Analyte.	Ba
Units	mg/kg

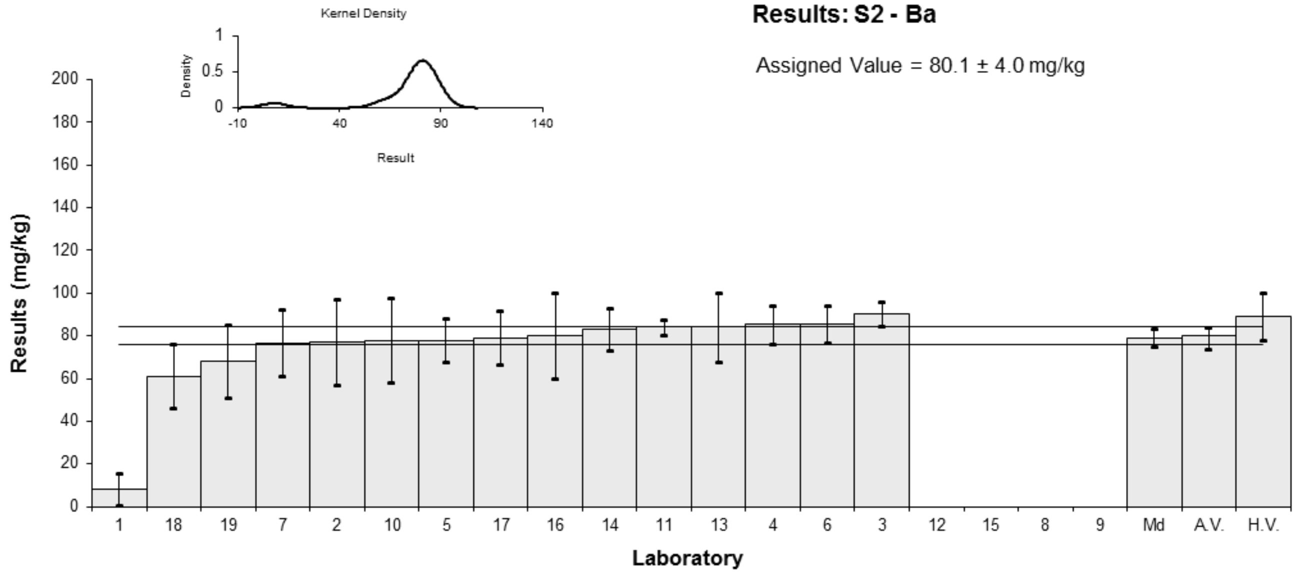
Participant Results

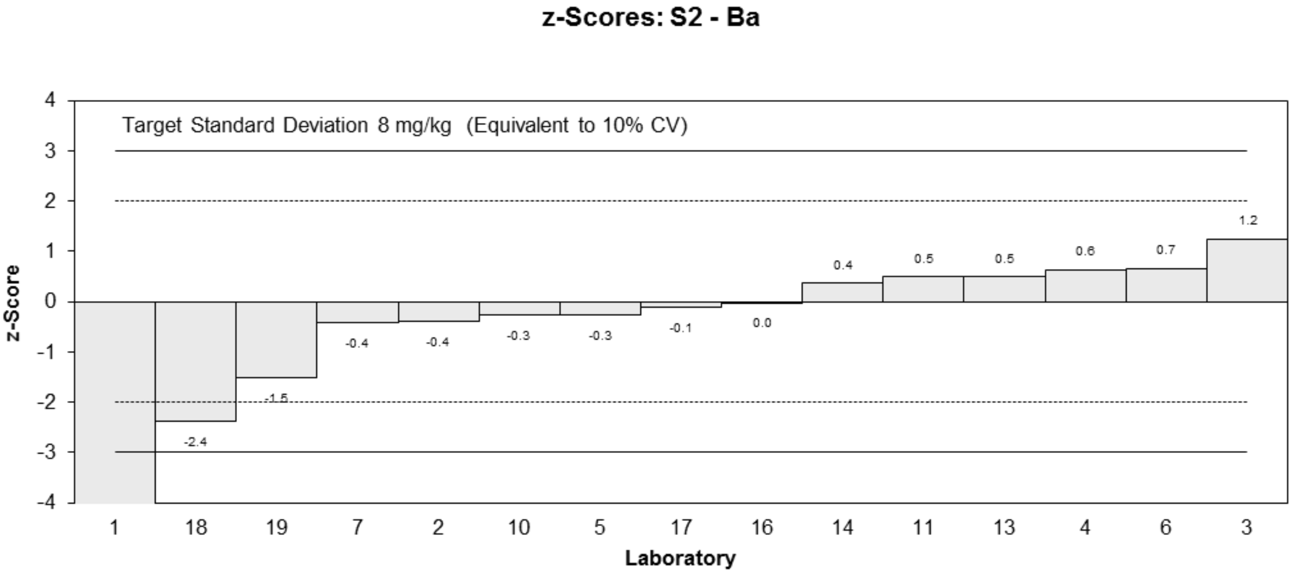
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	8	7.65	-9.00	-8.35
2	77	20	-0.39	-0.15
3	90	5.7	1.24	1.42
4	85.2	9.0	0.64	0.52
5	78	9.9	-0.26	-0.20
6	85.4	8.54	0.66	0.56
7	76.8	15.4	-0.41	-0.21
8	NR	NR		
9	NR	NR		
10	78	20	-0.26	-0.10
11	84.0	3.4	0.49	0.74
12	NR	NR		
13	84	16.1	0.49	0.24
14	83	10	0.36	0.27
15	NR	NR		
16	80	20	-0.01	0.00
17	79.1	12.7	-0.12	-0.08
18	61.1	15.0	-2.37	-1.22
19	68	17	-1.51	-0.69

Statistics

Assigned Value*	80.1	4.0
Spike	Not Spiked	
Homogeneity Value	89	11
Robust Average	78.8	5.2
Median	79.1	4.0
Mean	74.5	
N	15	
Max.	90	
Min.	8	
Robust SD	6.0	
Robust CV	7.6%	

*Robust Average excluding Laboratory 1.


Results: S2 - Ba
Assigned Value = 80.1 ± 4.0 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
1 18 19 7 2 10 5 17 16 14 11 13 4 6 3 12 15 8 9 Md A.V. H.V.
Laboratory


z-Scores: S2 - Ba
Target Standard Deviation 8 mg/kg (Equivalent to 10% CV)
z-Score
-2.4 -1.5 -0.4 -0.4 -0.3 -0.3 -0.1 0.0 0.4 0.5 0.5 0.6 0.7 1.2
1 18 19 7 2 10 5 17 16 14 11 13 4 6 3
Laboratory

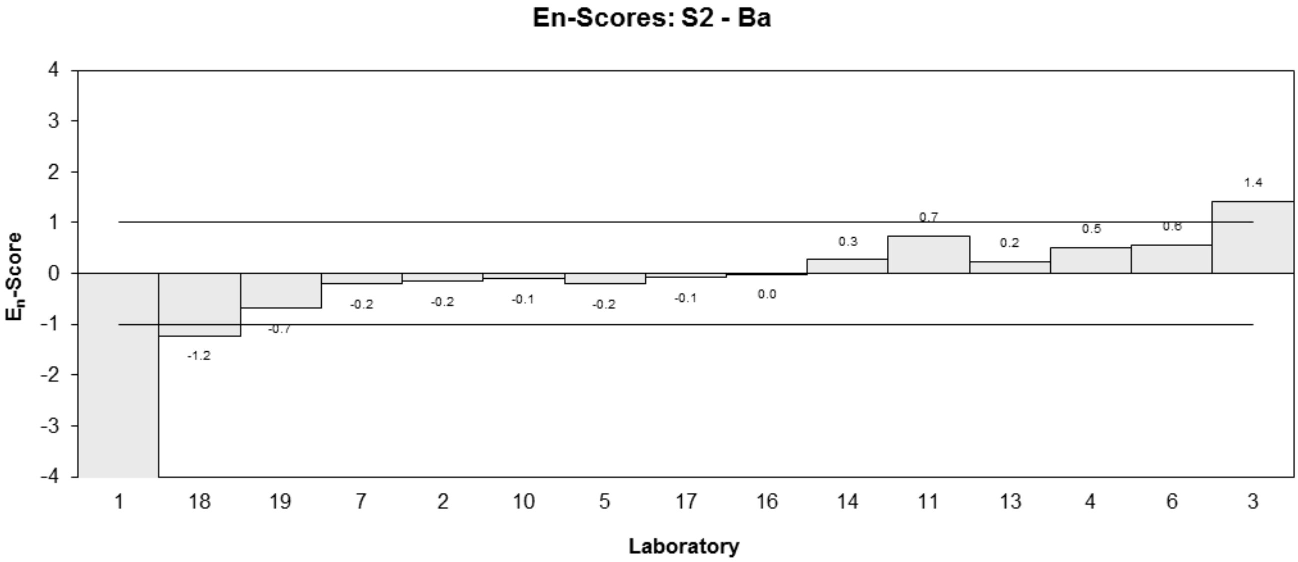

En-Scores: S2 - Ba
E_n-Score
-1.2 -0.7 -0.2 -0.2 -0.1 -0.2 -0.1 0.0 0.3 0.7 0.2 0.5 0.6 1.4
1 18 19 7 2 10 5 17 16 14 11 13 4 6 3
Laboratory

Figure 18

Table 28

Sample Details

Sample No.	S2
Matrix.	Soil
Analyte.	Bi
Units	mg/kg

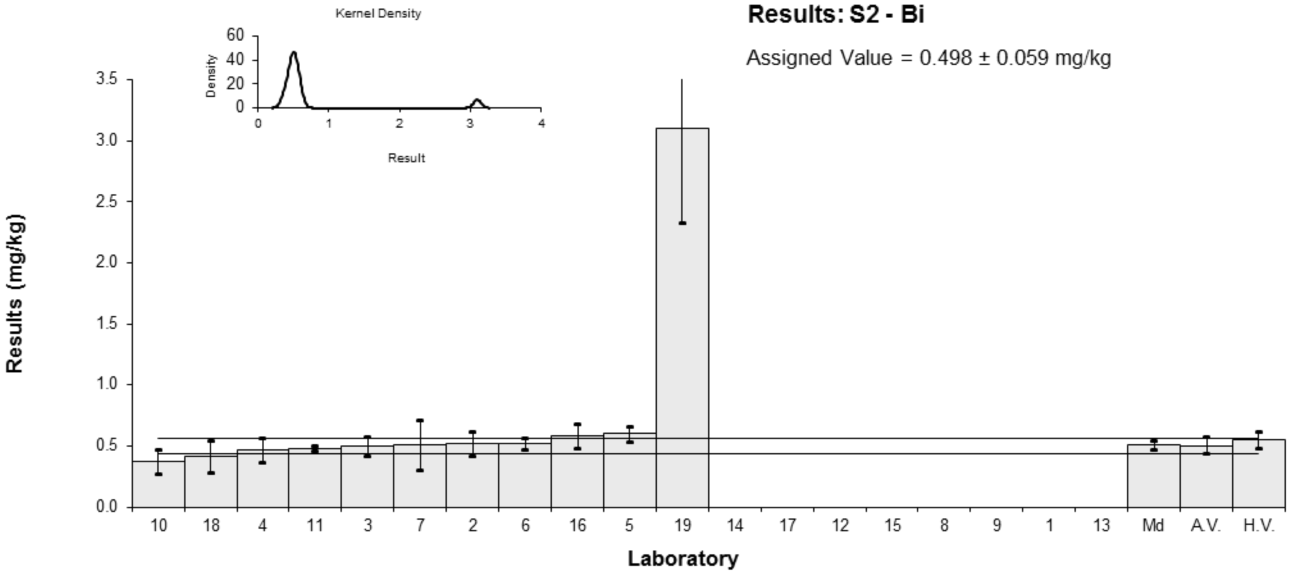
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	2	NT		
2	0.52	0.1	0.29	0.19
3	0.50	0.08	0.03	0.02
4	0.47	0.1	-0.37	-0.24
5	0.6	0.06	1.37	1.21
6	0.52	0.052	0.29	0.28
7	0.51	0.20	0.16	0.06
8	NR	NR		
9	NR	NR		
10	0.37	0.1	-1.71	-1.10
11	0.48	0.02	-0.24	-0.29
12	NR	NR		
13	NT	NT		
14	< 10	0.25		
15	NR	NR		
16	0.58	0.1	1.10	0.71
17	<10	2		
18	0.415	0.130	-1.11	-0.58
19	3.1	0.77	34.83	3.37

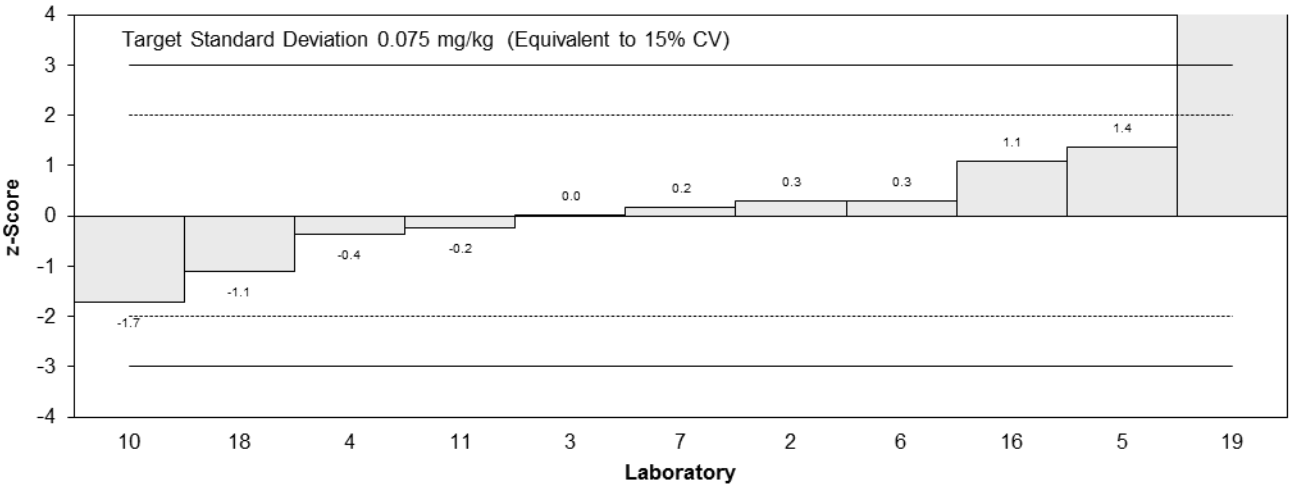
Statistics

Assigned Value*	0.498	0.059
Spike	Not Spiked	
Homogeneity Value	0.554	0.066
Robust Average	0.511	0.066
Median	0.510	0.040
Mean	0.733	
N	11	
Max.	3.1	
Min.	0.37	
Robust SD	0.074	
Robust CV	15%	

*Robust Average excluding Laboratory 19.



z-Scores: S2 - Bi



En-Scores: S2 - Bi

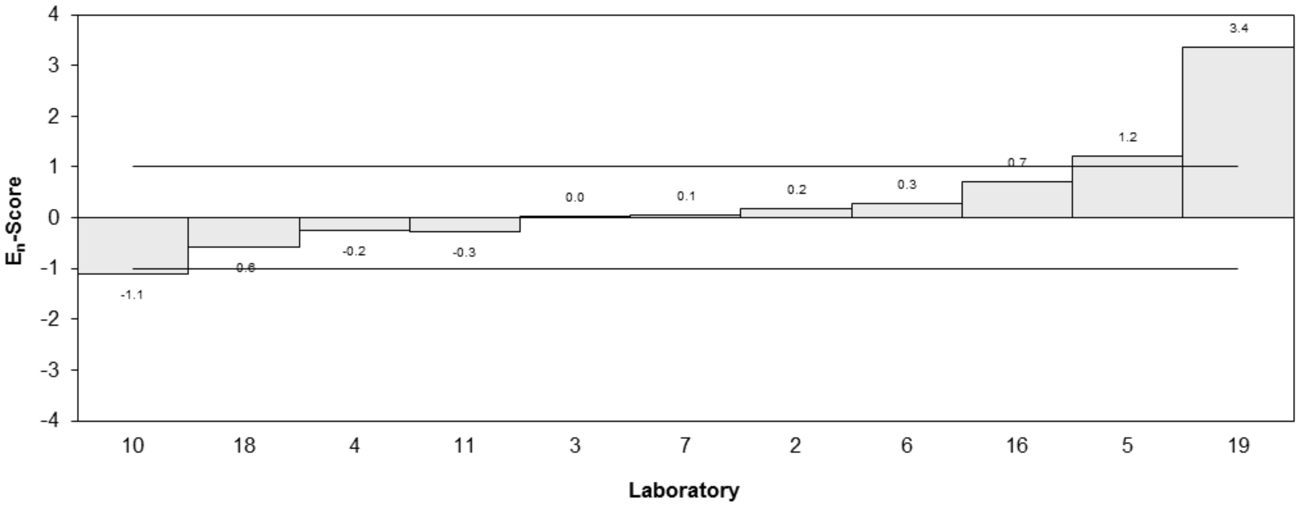


Figure 19

Table 29

Sample Details

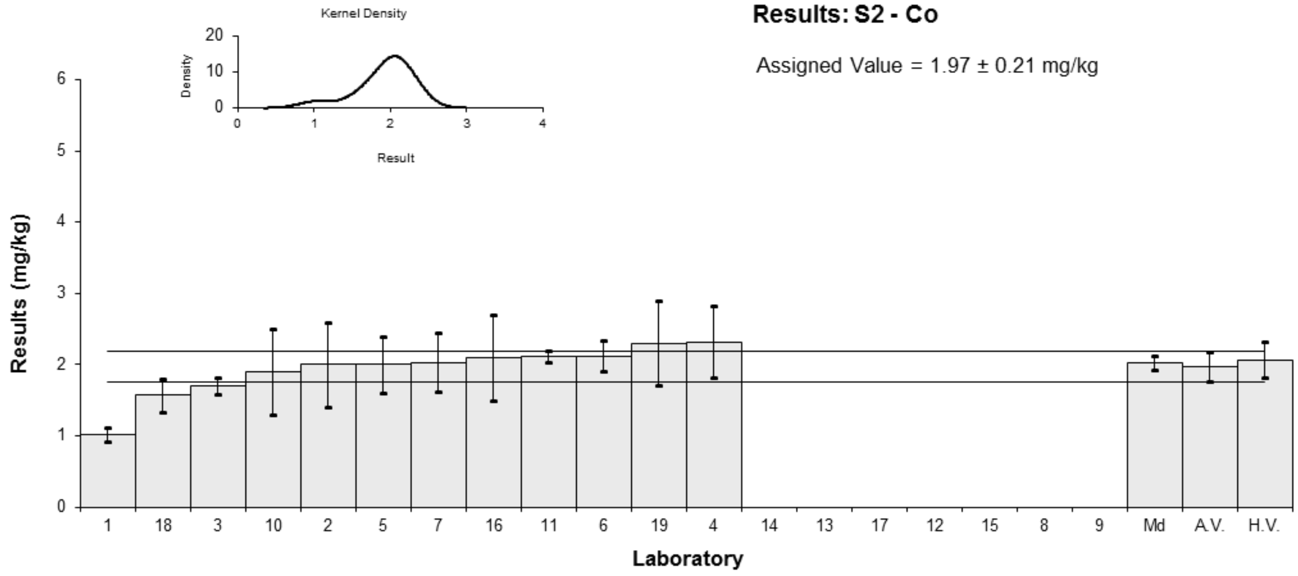
Sample No.	S2
Matrix.	Soil
Analyte.	Co
Units	mg/kg

Participant Results

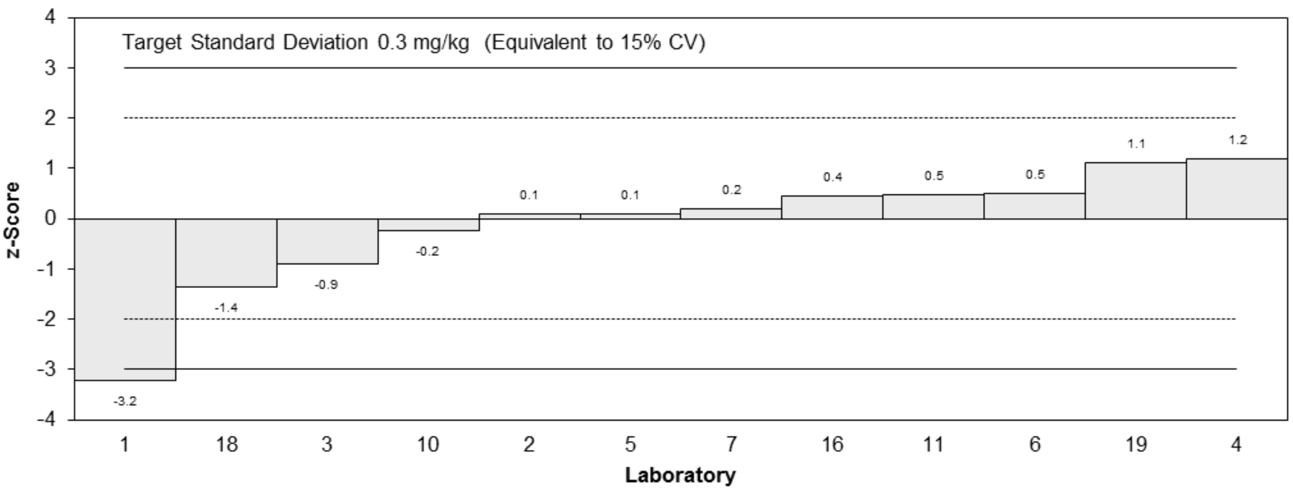
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1.02	0.102	-3.21	-4.07
2	2	0.6	0.10	0.05
3	1.7	0.12	-0.91	-1.12
4	2.32	0.5	1.18	0.65
5	2.0	0.4	0.10	0.07
6	2.12	0.212	0.51	0.50
7	2.03	0.41	0.20	0.13
8	NR	NR		
9	NR	NR		
10	1.9	0.6	-0.24	-0.11
11	2.11	0.08	0.47	0.62
12	NR	NR		
13	<0.5	NR		
14	< 5	0.73		
15	NR	NR		
16	2.1	0.6	0.44	0.20
17	<5	1		
18	1.57	0.23	-1.35	-1.28
19	2.3	0.59	1.12	0.53

Statistics

Assigned Value	1.97	0.21
Spike	Not Spiked	
Homogeneity Value	2.07	0.25
Robust Average	1.97	0.21
Median	2.02	0.10
Mean	1.93	
N	12	
Max.	2.32	
Min.	1.02	
Robust SD	0.29	
Robust CV	15%	



z-Scores: S2 - Co



En-Scores: S2 - Co

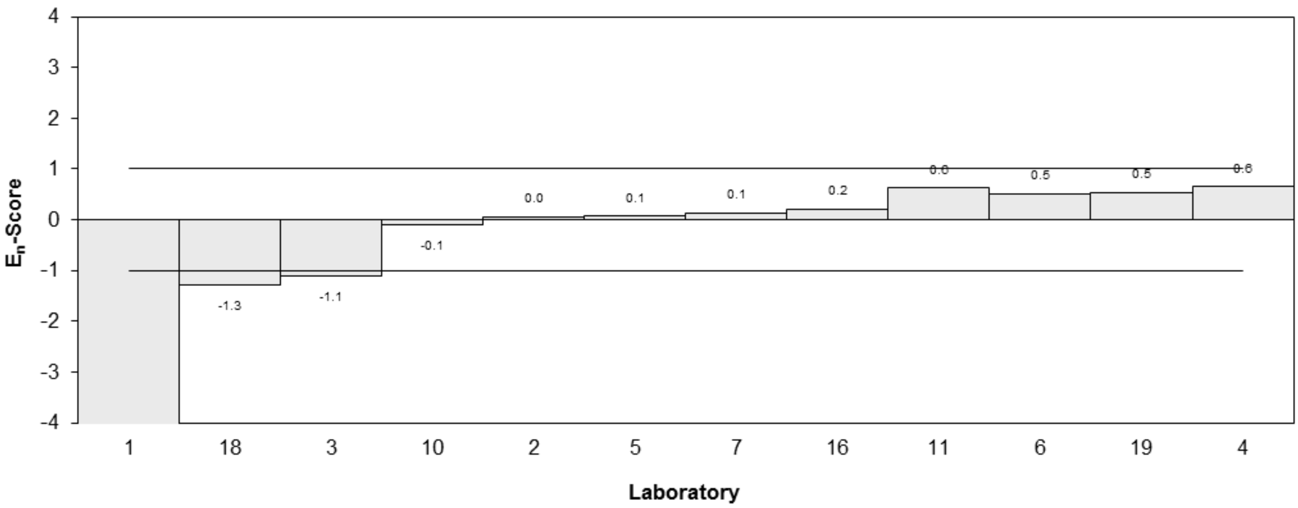


Figure 20

Table 30

Sample Details

Sample No.	S2
Matrix.	Soil
Analyte.	Cs
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	<1	NR
3	0.74	0.11
4	NT	NT
5	0.2	0.02
6	NT	NT
7	0.43	0.18
8	NR	NR
9	NR	NR
10	<1	NR
11	NT	NT
12	NR	NR
13	NT	NT
14	<10	2.0
15	NR	NR
16	NT	NT
17	NT	NT
18	0.256	0.038
19	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.485	0.097
Robust Average	0.410	0.340
Median	0.340	0.270
Mean	0.406	
N	4	
Max.	0.74	
Min.	0.2	
Robust SD	0.28	
Robust CV	68%	

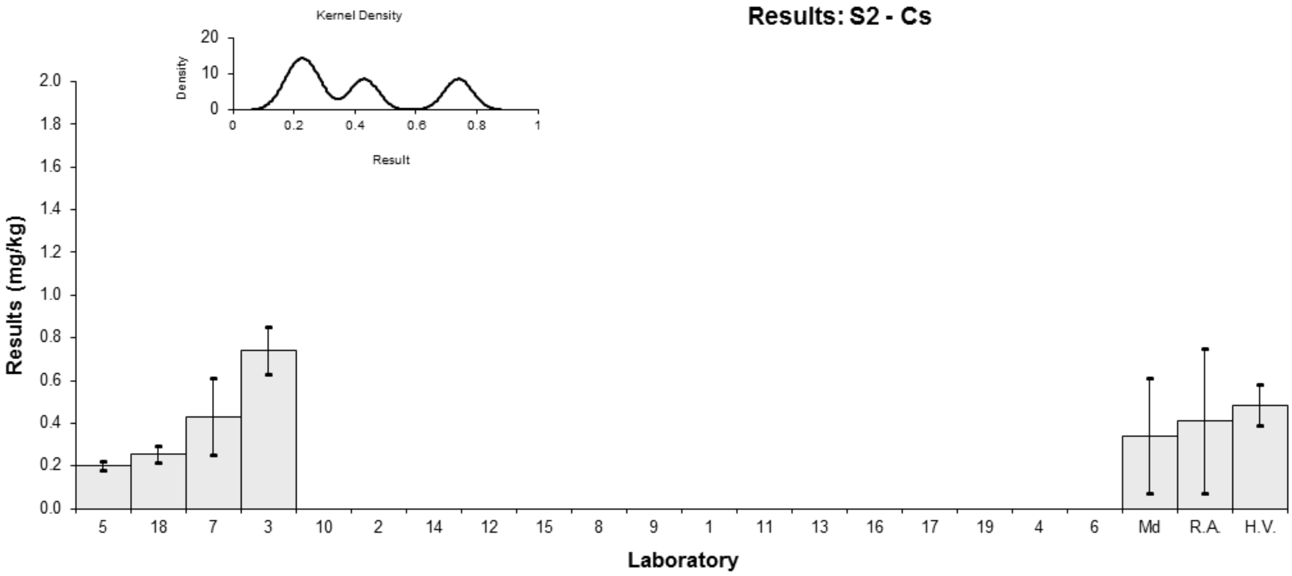

Results: S2 - Cs
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
5
18
7
3
10
2
14
12
15
8
9
1
11
13
16
17
19
4
6
Md
R.A.
H.V.

Figure 21

Table 31

Sample Details

Sample No.	S2
Matrix.	Soil
Analyte.	La
Units	mg/kg

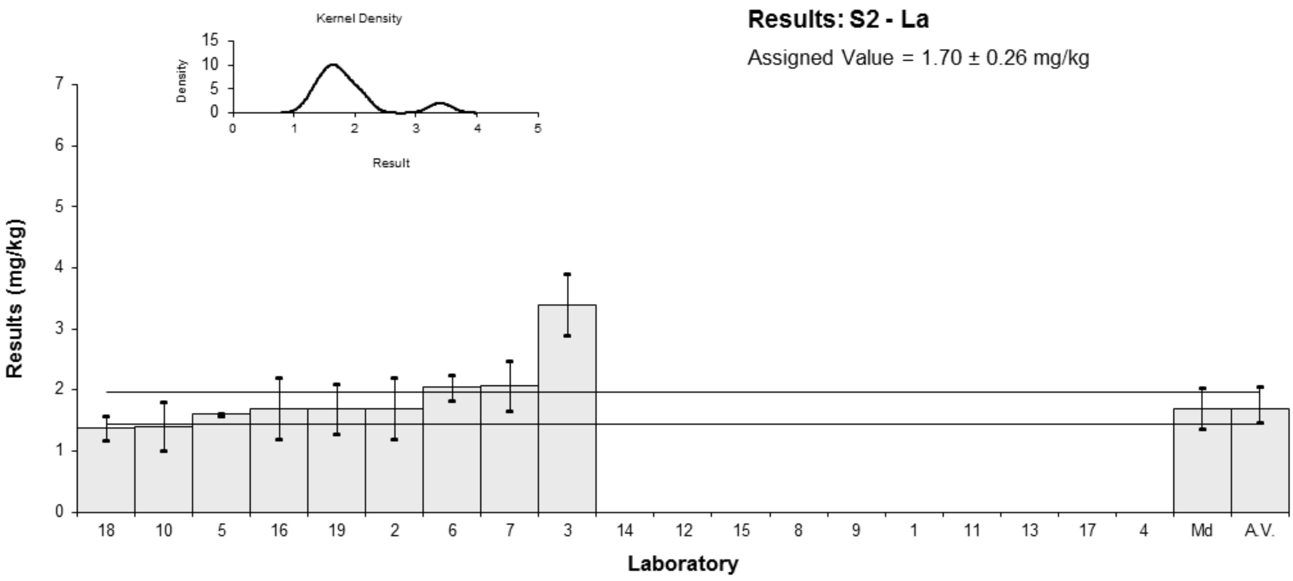
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	1.7	0.5	0.00	0.00
3	3.4	0.5	6.67	3.02
4	NT	NT		
5	1.6	0.02	-0.39	-0.38
6	2.04	0.204	1.33	1.03
7	2.07	0.41	1.45	0.76
8	NR	NR		
9	NR	NR		
10	1.4	0.4	-1.18	-0.63
11	NT	NT		
12	NR	NR		
13	NT	NT		
14	<10	2.0		
15	NR	NR		
16	1.7	0.5	0.00	0.00
17	NT	NT		
18	1.37	0.200	-1.29	-1.01
19	1.7	0.41	0.00	0.00

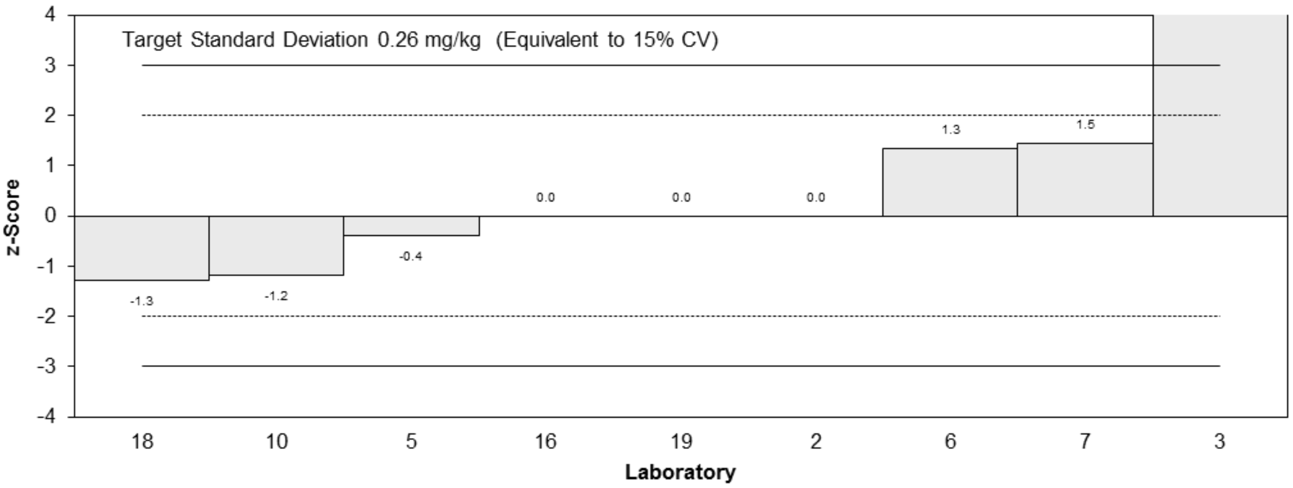
Statistics

Assigned Value*	1.70	0.26
Spike	Not Spiked	
Robust Average	1.76	0.30
Median	1.70	0.34
Mean	1.89	
N	9	
Max.	3.4	
Min.	1.37	
Robust SD	0.29	
Robust CV	17%	

*Robust Average excluding Laboratory 3.



z-Scores: S2 - La



En-Scores: S2 - La

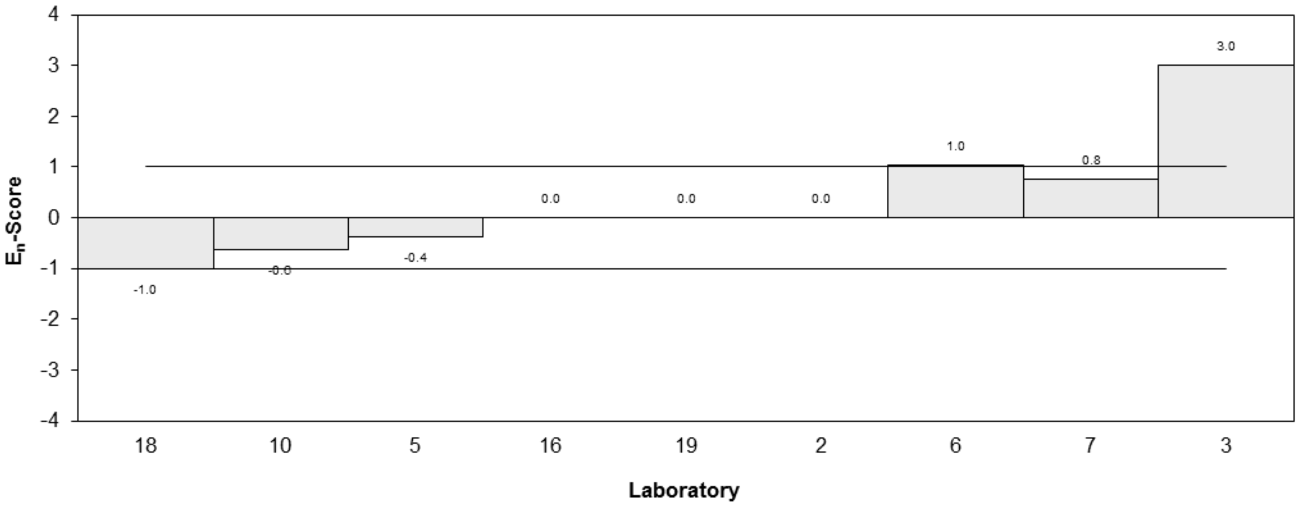


Figure 22

Table 32

Sample Details

Sample No.	S2
Matrix.	Soil
Analyte.	Li
Units	mg/kg

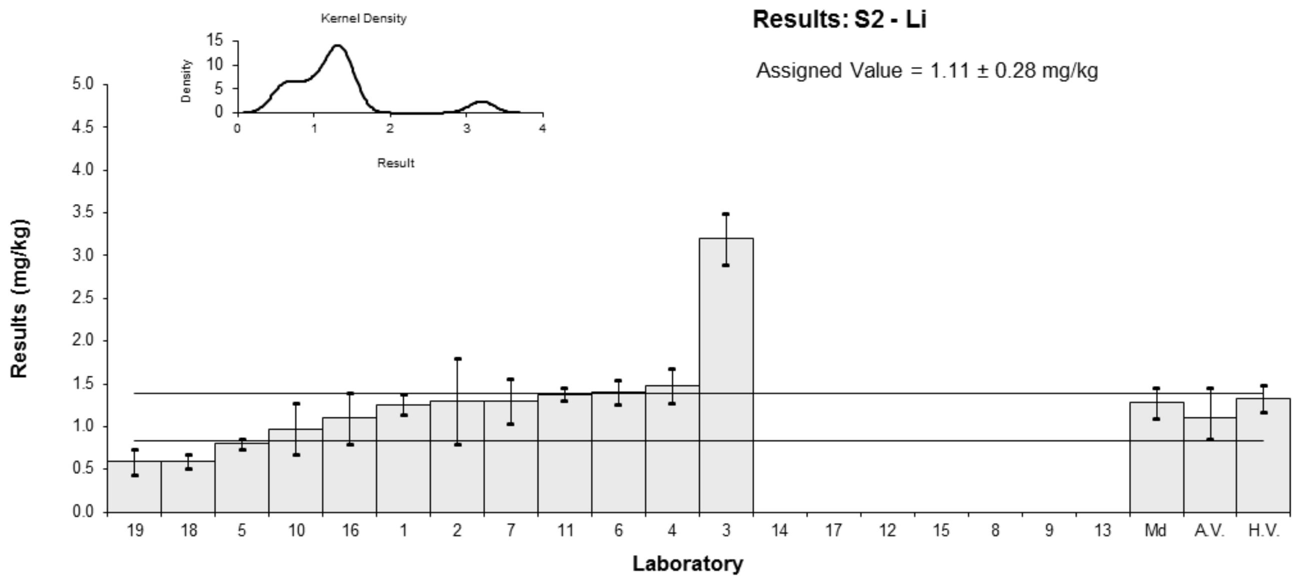
Participant Results

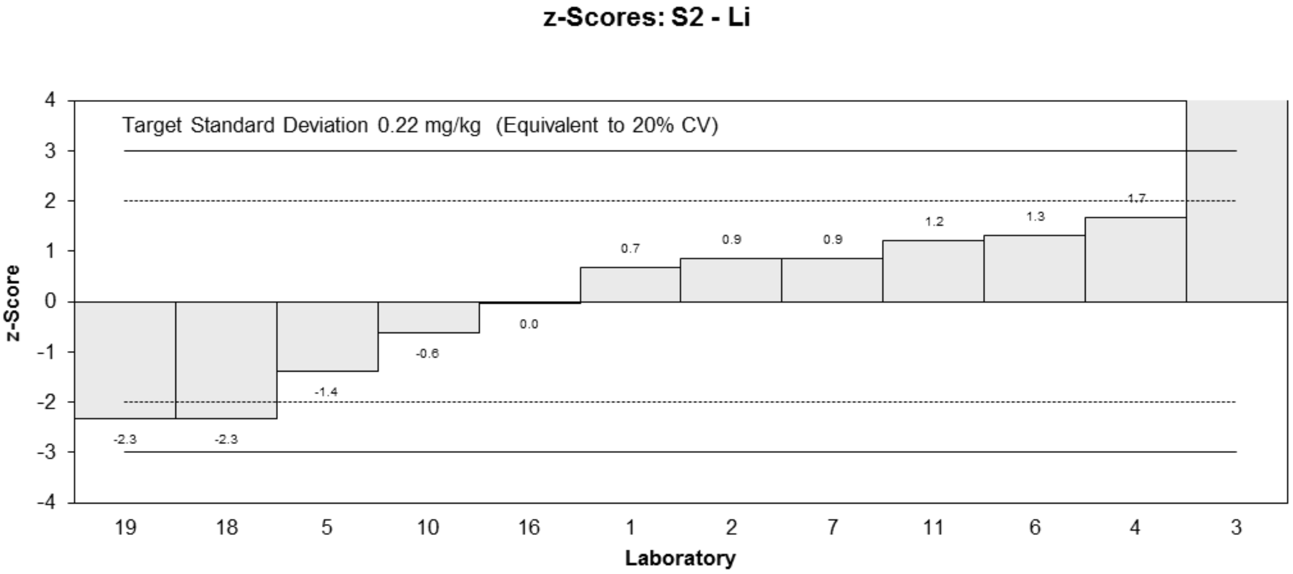
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1.26	0.126	0.68	0.49
2	1.3	0.5	0.86	0.33
3	3.2	0.3	9.41	5.09
4	1.48	0.2	1.67	1.08
5	0.8	0.06	-1.40	-1.08
6	1.40	0.140	1.31	0.93
7	1.30	0.26	0.86	0.50
8	NR	NR		
9	NR	NR		
10	0.97	0.3	-0.63	-0.34
11	1.38	0.07	1.22	0.94
12	NR	NR		
13	NT	NT		
14	< 5	1.0		
15	NR	NR		
16	1.1	0.3	-0.05	-0.02
17	<5	1		
18	0.593	0.089	-2.33	-1.76
19	0.59	0.15	-2.34	-1.64

Statistics

Assigned Value*	1.11	0.28
Spike	Not Spiked	
Homogeneity Value	1.33	0.16
Robust Average	1.16	0.30
Median	1.28	0.18
Mean	1.28	
N	12	
Max.	3.2	
Min.	0.59	
Robust SD	0.37	
Robust CV	32%	

*Robust Average excluding Laboratory 3.


Results: S2 - Li
Assigned Value = 1.11 ± 0.28 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
19
18
5
10
16
1
2
7
11
6
4
3
14
17
12
15
8
9
13
Md
A.V.
H.V.


z-Scores: S2 - Li
Target Standard Deviation 0.22 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
-2.3
-2.3
-1.4
-0.6
0.0
0.7
0.9
0.9
1.2
1.3
1.7

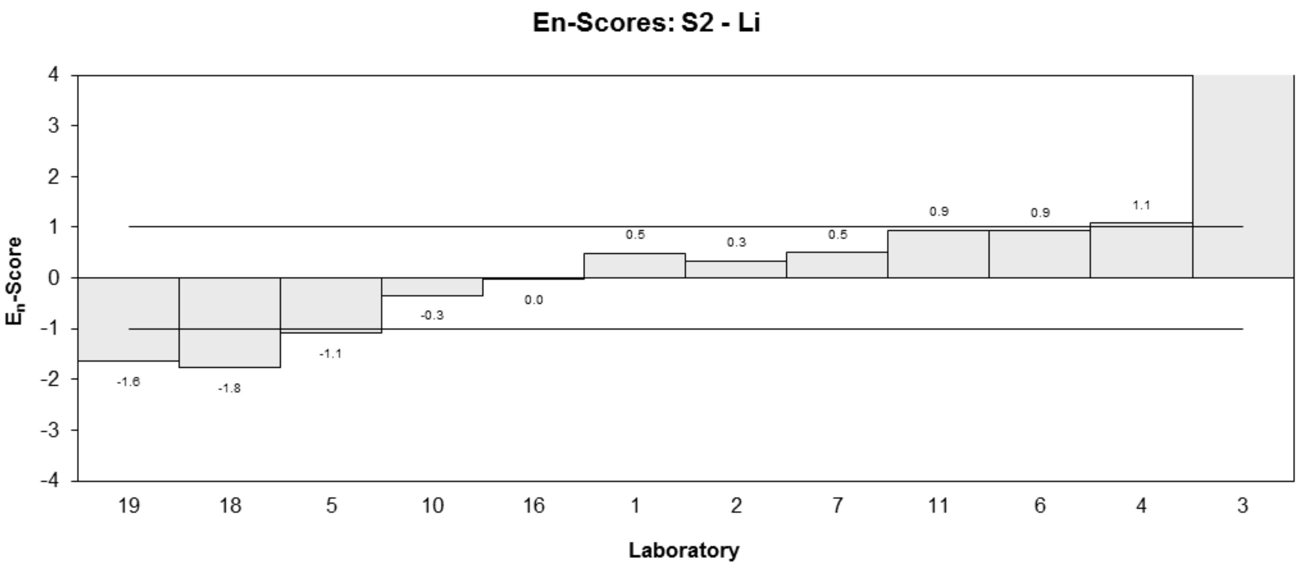

En-Scores: S2 - Li
En-Score
Laboratory
-1.6
-1.8
-1.1
-0.3
0.0
0.5
0.3
0.5
0.9
0.9
1.1

Figure 23

Table 33

Sample Details

Sample No.	S2
Matrix.	Soil
Analyte.	Rb
Units	mg/kg

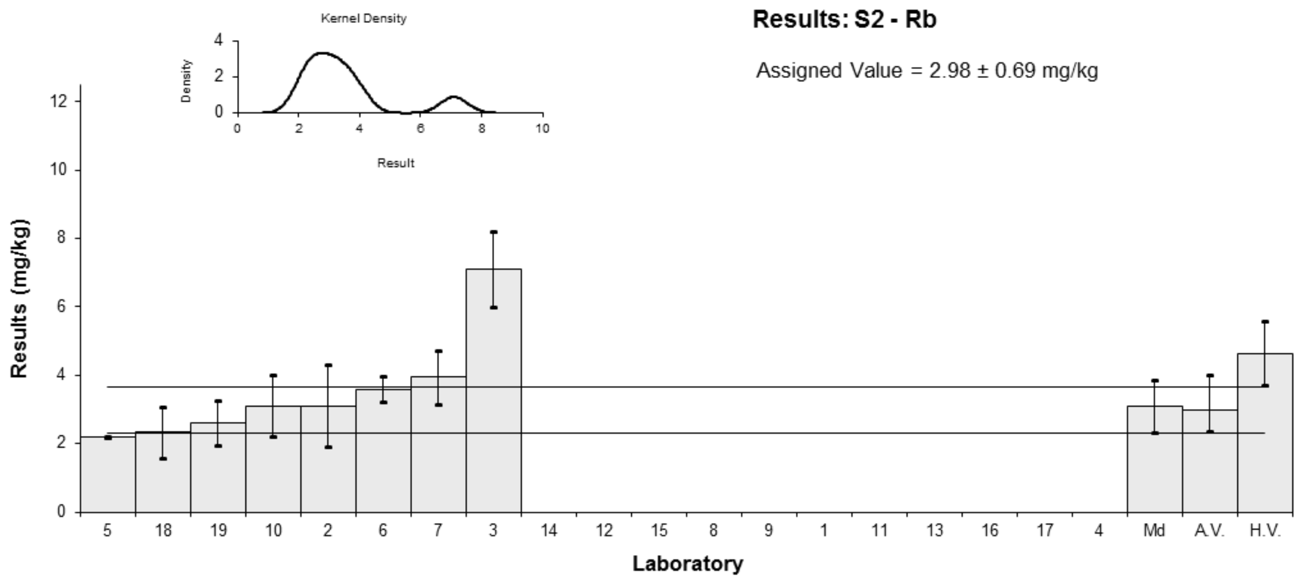
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	3.1	1.2	0.20	0.09
3	7.1	1.1	6.91	3.17
4	NT	NT		
5	2.2	0.02	-1.31	-1.13
6	3.60	0.360	1.04	0.80
7	3.94	0.79	1.61	0.92
8	NR	NR		
9	NR	NR		
10	3.1	0.9	0.20	0.11
11	NT	NT		
12	NR	NR		
13	NT	NT		
14	<10	2.0		
15	NR	NR		
16	NT	NT		
17	NT	NT		
18	2.34	0.75	-1.07	-0.63
19	2.6	0.65	-0.64	-0.40

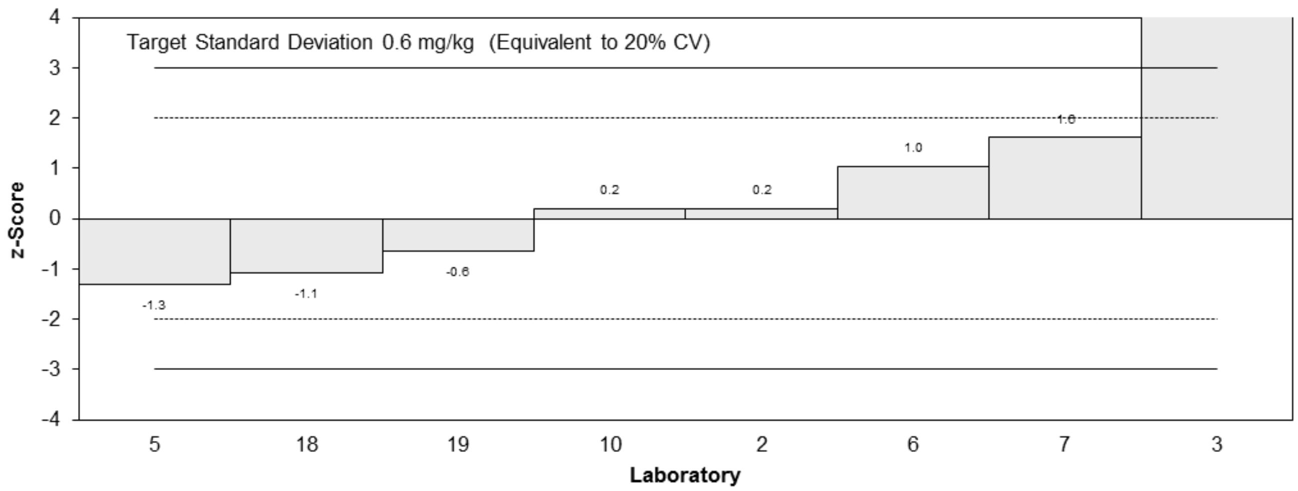
Statistics

Assigned Value*	2.98	0.69
Spike	Not Spiked	
Homogeneity Value	4.64	0.93
Robust Average	3.18	0.82
Median	3.10	0.78
Mean	3.50	
N	8	
Max.	7.1	
Min.	2.2	
Robust SD	0.73	
Robust CV	23%	

*Robust Average excluding Laboratory 3.



z-Scores: S2 - Rb



En-Scores: S2 - Rb

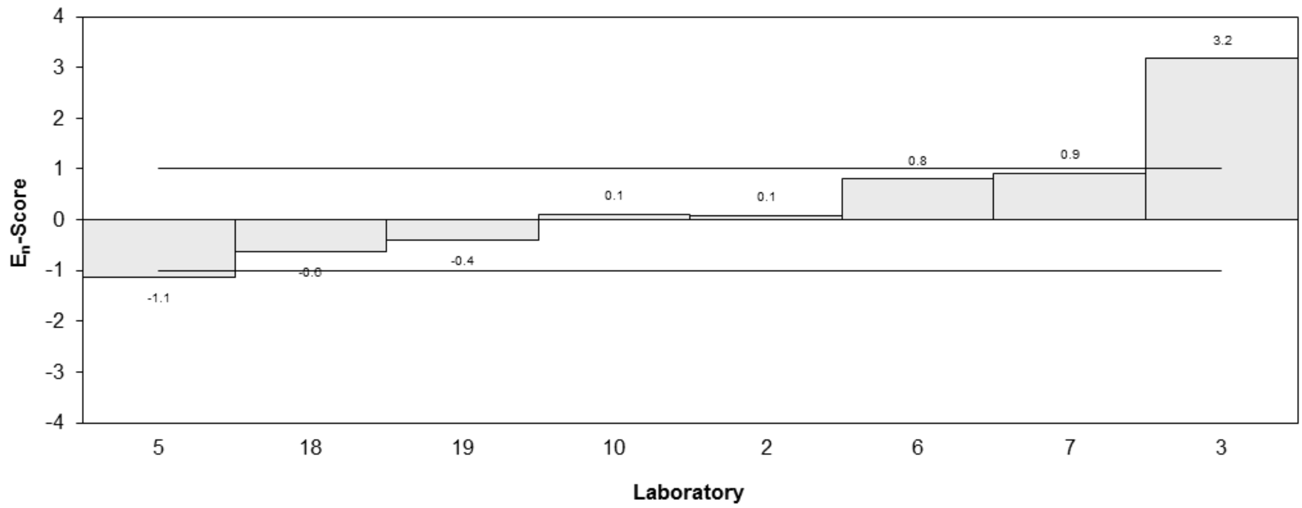


Figure 24

Table 34

Sample Details

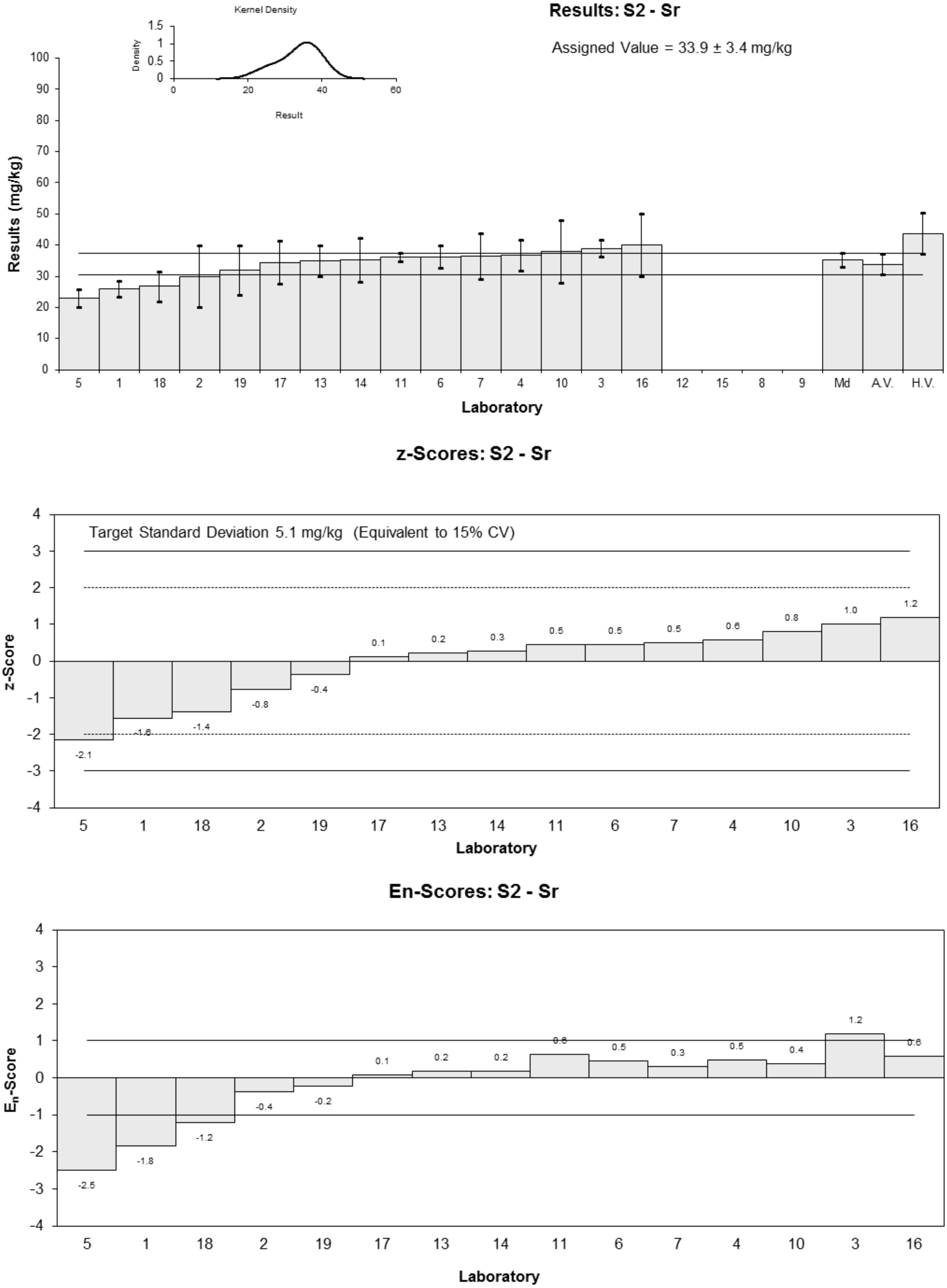
Sample No.	S2
Matrix.	Soil
Analyte.	Sr
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	26.0	2.6	-1.55	-1.85
2	30	10	-0.77	-0.37
3	39	2.6	1.00	1.19
4	36.8	5.0	0.57	0.48
5	23	2.76	-2.14	-2.49
6	36.2	3.62	0.45	0.46
7	36.4	7.3	0.49	0.31
8	NR	NR		
9	NR	NR		
10	38	10	0.81	0.39
11	36.2	1.4	0.45	0.63
12	NR	NR		
13	35	4.9	0.22	0.18
14	35.3	7.1	0.28	0.18
15	NR	NR		
16	40	10	1.20	0.58
17	34.5	6.9	0.12	0.08
18	26.8	4.8	-1.40	-1.21
19	32	8.0	-0.37	-0.22

Statistics

Assigned Value	33.9	3.4
Spike	Not Spiked	
Homogeneity Value	43.7	6.6
Robust Average	33.9	3.4
Median	35.3	2.2
Mean	33.7	
N	15	
Max.	40	
Min.	23	
Robust SD	5.3	
Robust CV	16%	



Results: S2 - Sr
Assigned Value = 33.9 ± 3.4 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
5 1 18 2 19 17 13 14 11 6 7 4 10 3 16 12 15 8 9 Md A.V. H.V.
z-Scores: S2 - Sr
Target Standard Deviation 5.1 mg/kg (Equivalent to 15% CV)
z-Score
-2.1 -1.6 -1.4 -0.8 -0.4 0.1 0.2 0.3 0.5 0.5 0.5 0.6 0.8 1.0 1.2
5 1 18 2 19 17 13 14 11 6 7 4 10 3 16
Laboratory
En-Scores: S2 - Sr
E_n-Score
-2.5 -1.8 -1.2 -0.4 -0.2 0.1 0.2 0.2 0.6 0.5 0.3 0.5 0.4 1.2 0.6
5 1 18 2 19 17 13 14 11 6 7 4 10 3 16
Laboratory

Figure 25

Table 35

Sample Details

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

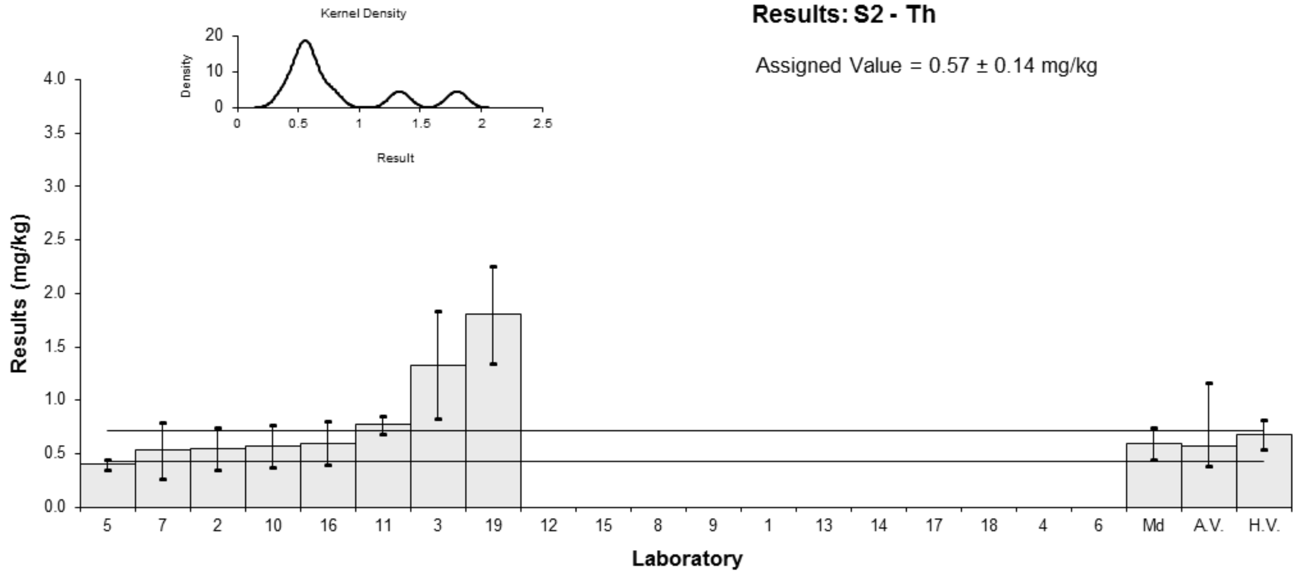
Participant Results

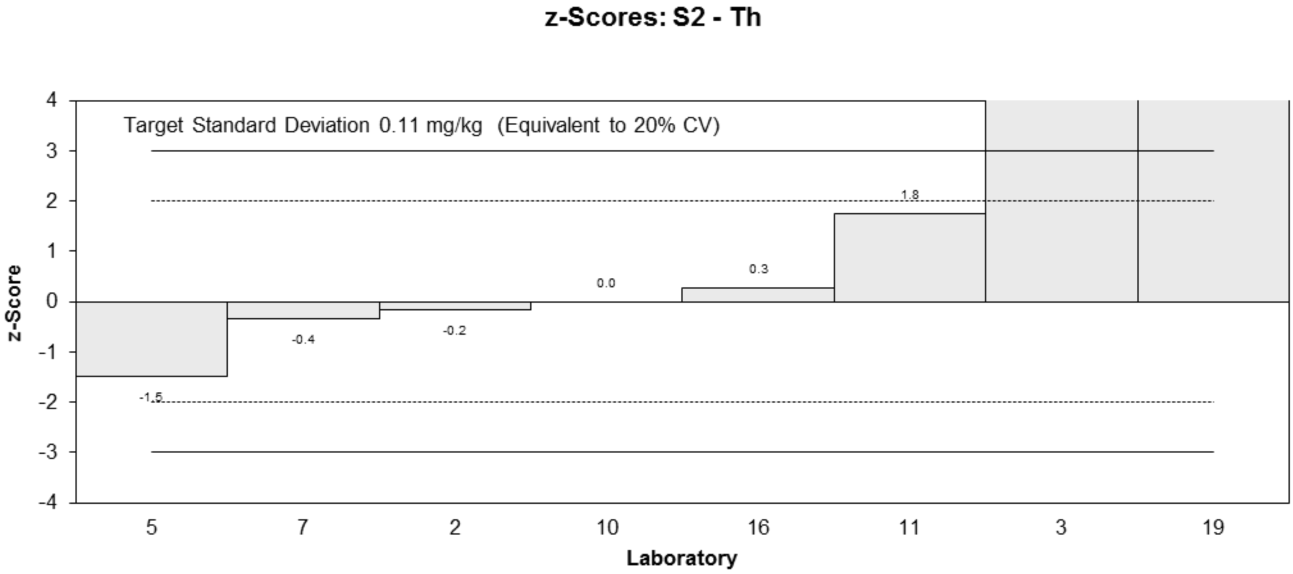
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	0.55	0.2	-0.18	-0.08
3	1.33	0.5	6.67	1.46
4	NT	NT		
5	0.4	0.05	-1.49	-1.14
6	NT	NT		
7	0.53	0.26	-0.35	-0.14
8	NR	NR		
9	NR	NR		
10	0.57	0.2	0.00	0.00
11	0.77	0.08	1.75	1.24
12	NR	NR		
13	NT	NT		
14	NT	NT		
15	NR	NR		
16	0.60	0.2	0.26	0.12
17	NT	NT		
18	NT	NT		
19	1.8	0.45	10.79	2.61

Statistics

Assigned Value*	0.57	0.14
Spike	Not Spiked	
Homogeneity Value	0.68	0.14
Robust Average	0.77	0.39
Median	0.59	0.15
Mean	0.82	
N	8	
Max.	1.8	
Min.	0.4	
Robust SD	0.14	
Robust CV	18%	

*Robust Average excluding Laboratories 3 and 19..


Results: S2 - Th
Assigned Value = 0.57 ± 0.14 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
5 7 2 10 16 11 3 19 12 15 8 9 1 13 14 17 18 4 6 Md A.V. H.V.


z-Scores: S2 - Th
Target Standard Deviation 0.11 mg/kg (Equivalent to 20% CV)
z-Score
-1.5
-0.4
-0.2
0.0
0.3
1.8
Laboratory
5 7 2 10 16 11 3 19

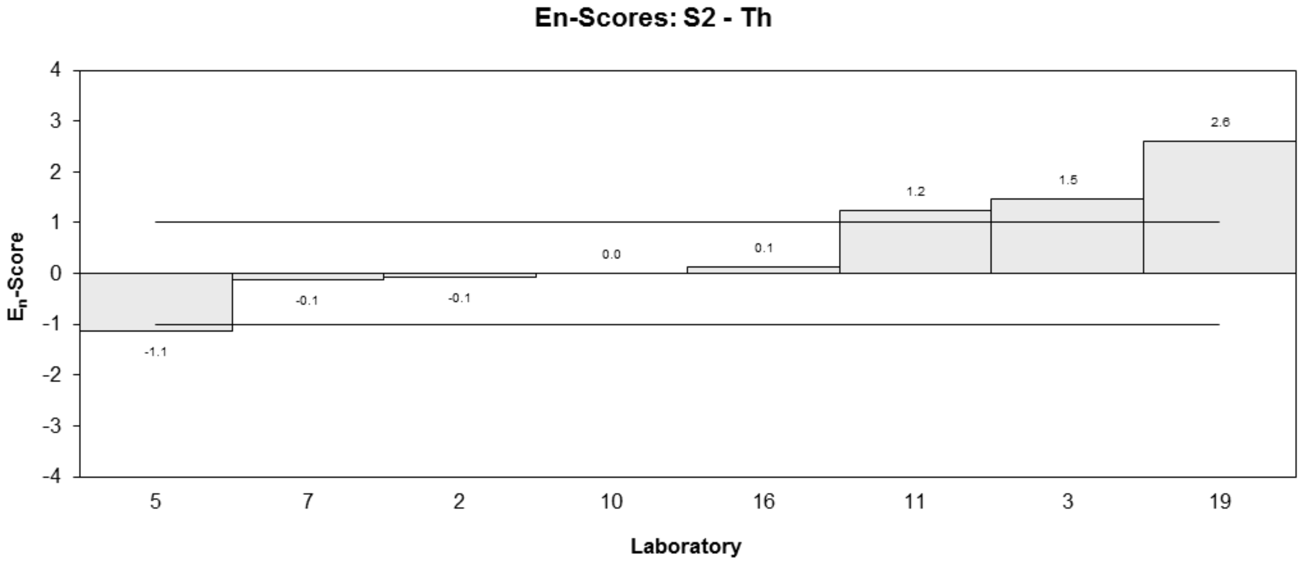

En-Scores: S2 - Th
E_n-Score
-1.1
-0.1
-0.1
0.0
0.1
1.2
1.5
2.6
Laboratory
5 7 2 10 16 11 3 19

Figure 26

Table 36

Sample Details

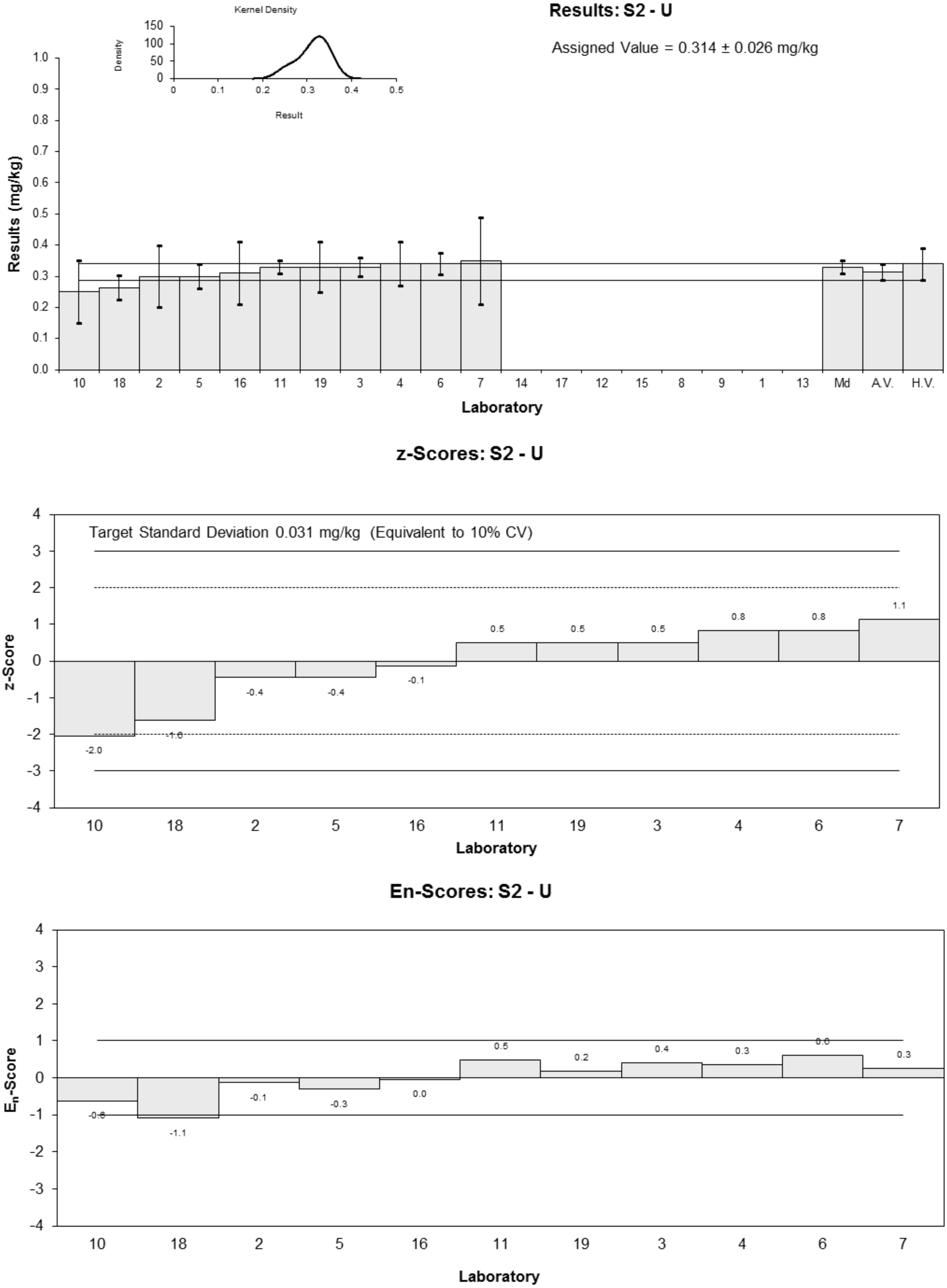
Sample No.	S2
Matrix.	Soil
Analyte.	U
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	0.3	0.1	-0.45	-0.14
3	0.33	0.03	0.51	0.40
4	0.34	0.07	0.83	0.35
5	0.3	0.04	-0.45	-0.29
6	0.34	0.034	0.83	0.61
7	0.35	0.14	1.15	0.25
8	NR	NR		
9	NR	NR		
10	0.25	0.1	-2.04	-0.62
11	0.33	0.02	0.51	0.49
12	NR	NR		
13	NT	NT		
14	< 1	0.20		
15	NR	NR		
16	0.31	0.1	-0.13	-0.04
17	<10	2		
18	0.263	0.039	-1.62	-1.09
19	0.33	0.08	0.51	0.19

Statistics

Assigned Value	0.314	0.026
Spike	Not Spiked	
Homogeneity Value	0.340	0.051
Robust Average	0.314	0.026
Median	0.330	0.020
Mean	0.313	
N	11	
Max.	0.35	
Min.	0.25	
Robust SD	0.034	
Robust CV	11%	



Results: S2 - U
Assigned Value = 0.314 ± 0.026 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
10 18 2 5 16 11 19 3 4 6 7 14 17 12 15 8 9 1 13 Md A.V. H.V.
z-Scores: S2 - U
Target Standard Deviation 0.031 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
En-Scores: S2 - U
En-Score
Laboratory

Figure 27

Table 37

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	230	70	0.09	0.03
3	NR	NR		
4	252	30	1.05	0.70
5	230	29	0.09	0.06
6	NR	NR		
7	210	42	-0.79	-0.40
8	NR	NR		
9	NR	NR		
10	250	80	0.96	0.27
11	NR	NR		
12	200	42	-1.23	-0.62
13	230	44.2	0.09	0.04
14	NR	NR		
15	NR	NR		
16	213	70	-0.66	-0.21
17	241	39	0.57	0.31
18	NR	NR		
19	NR	NR		

Statistics

Assigned Value	228	17
Spike	Not Spiked	
Homogeneity Value	233	28
Robust Average	228	17
Median	230	19
Mean	228	
N	9	
Max.	252	
Min.	200	
Robust SD	20	
Robust CV	8.8%	

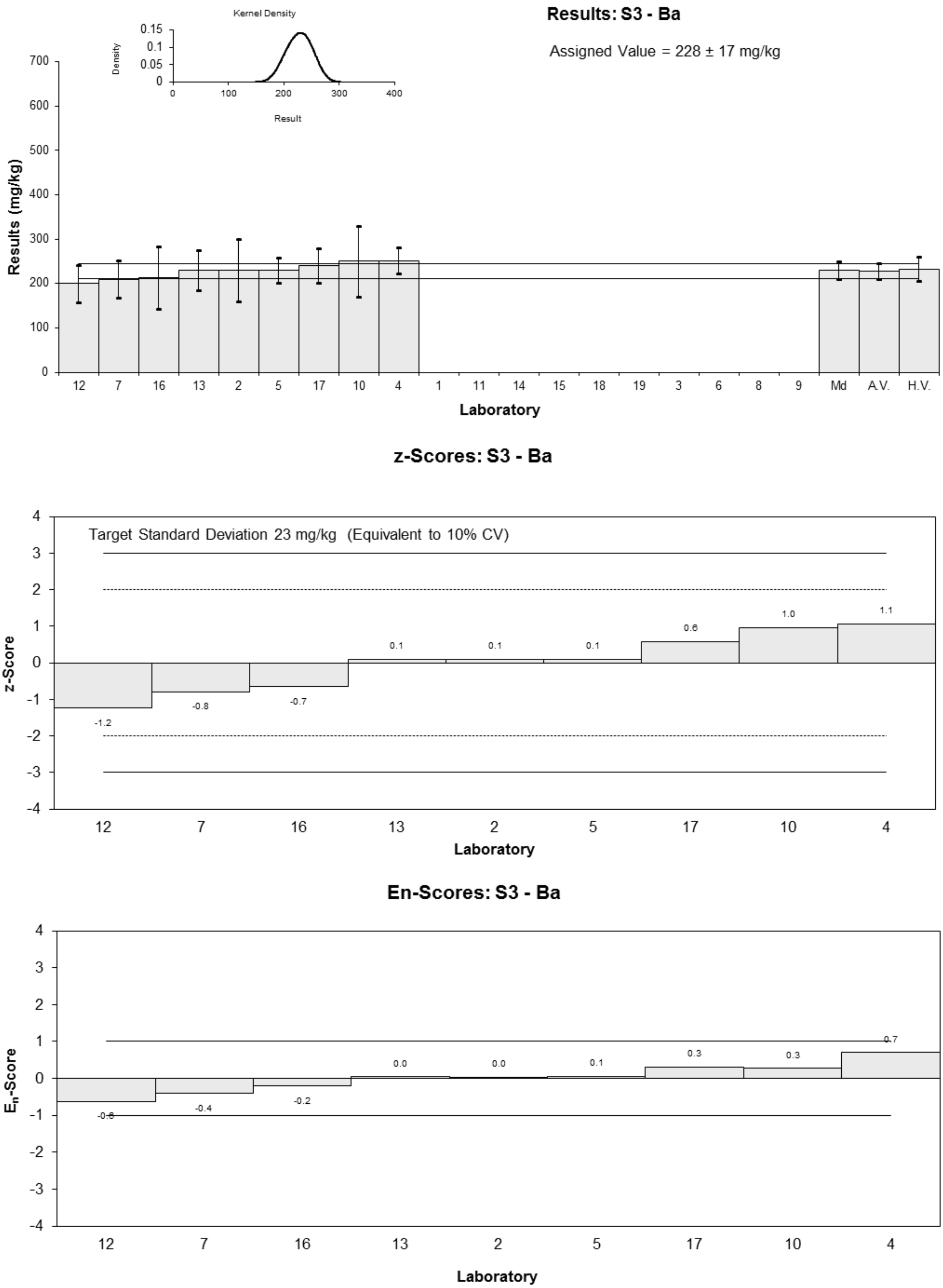

Results: S3 - Ba
Assigned Value = 228 ± 17 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
12 7 16 13 2 5 17 10 4 1 11 14 15 18 19 3 6 8 9 Md A.V. H.V.
z-Scores: S3 - Ba
Target Standard Deviation 23 mg/kg (Equivalent to 10% CV)
z-Score
-1.2 -0.8 -0.7 0.1 0.1 0.1 0.6 1.0 1.1
Laboratory
12 7 16 13 2 5 17 10 4
En-Scores: S3 - Ba
En-Score
-0.6 -0.4 -0.2 0.0 0.0 0.1 0.3 0.3 0.7
Laboratory
12 7 16 13 2 5 17 10 4

Figure 28

Table 38

Sample Details

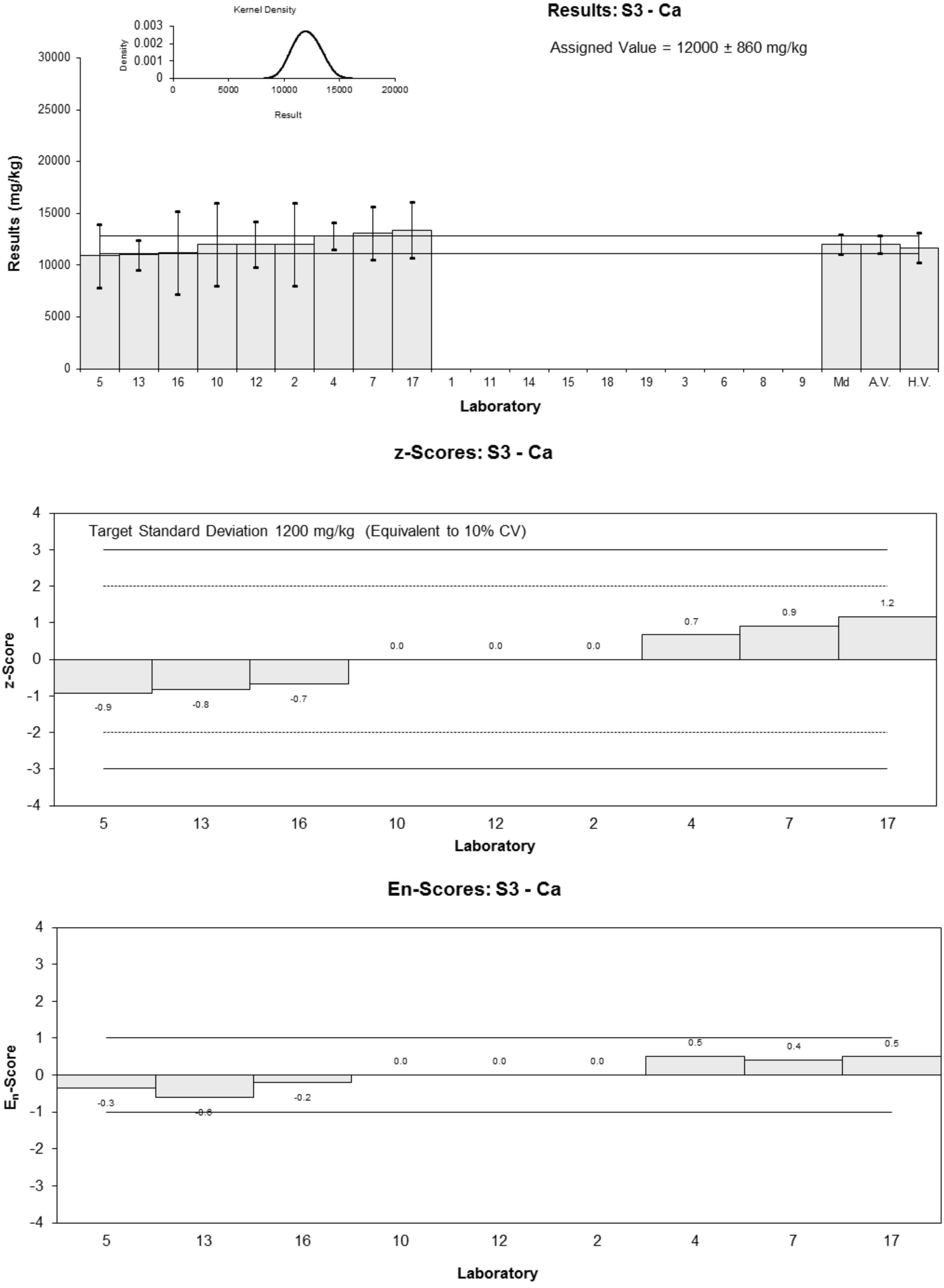
Sample No.	S3
Matrix.	Soil
Analyte.	Ca
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	12000	4000	0.00	0.00
3	NR	NR		
4	12800	1300	0.67	0.51
5	10900	3040	-0.92	-0.35
6	NR	NR		
7	13100	2600	0.92	0.40
8	NR	NR		
9	NR	NR		
10	12000	4000	0.00	0.00
11	NR	NR		
12	12000	2200	0.00	0.00
13	11000	1430	-0.83	-0.60
14	NR	NR		
15	NR	NR		
16	11180	4000	-0.68	-0.20
17	13400	2700	1.17	0.49
18	NR	NR		
19	NR	NR		

Statistics

Assigned Value	12000	860
Spike	Not Spiked	
Homogeneity Value	11700	1400
Robust Average	12000	860
Median	12000	930
Mean	12042	
N	9	
Max.	13400	
Min.	10900	
Robust SD	1000	
Robust CV	8.3%	



Results: S3 - Ca
Assigned Value = 12000 ± 860 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
z-Scores: S3 - Ca
Target Standard Deviation 1200 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
En-Scores: S3 - Ca
En-Score
Laboratory

Figure 29

Table 39

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	NR	NR
2	NT	NT
3	NR	NR
4	751	75
5	NT	NT
6	NR	NR
7	737	147
8	NR	NR
9	NR	NR
10	NT	NT
11	NR	NR
12	NT	NT
13	430	130
14	NR	NR
15	510	16
16	NT	NT
17	NT	NT
18	NR	NR
19	NR	NR

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	710	85
Robust Average	607	229
Median	624	284
Mean	607	
N	4	
Max.	751	
Min.	430	
Robust SD	180	
Robust CV	30%	

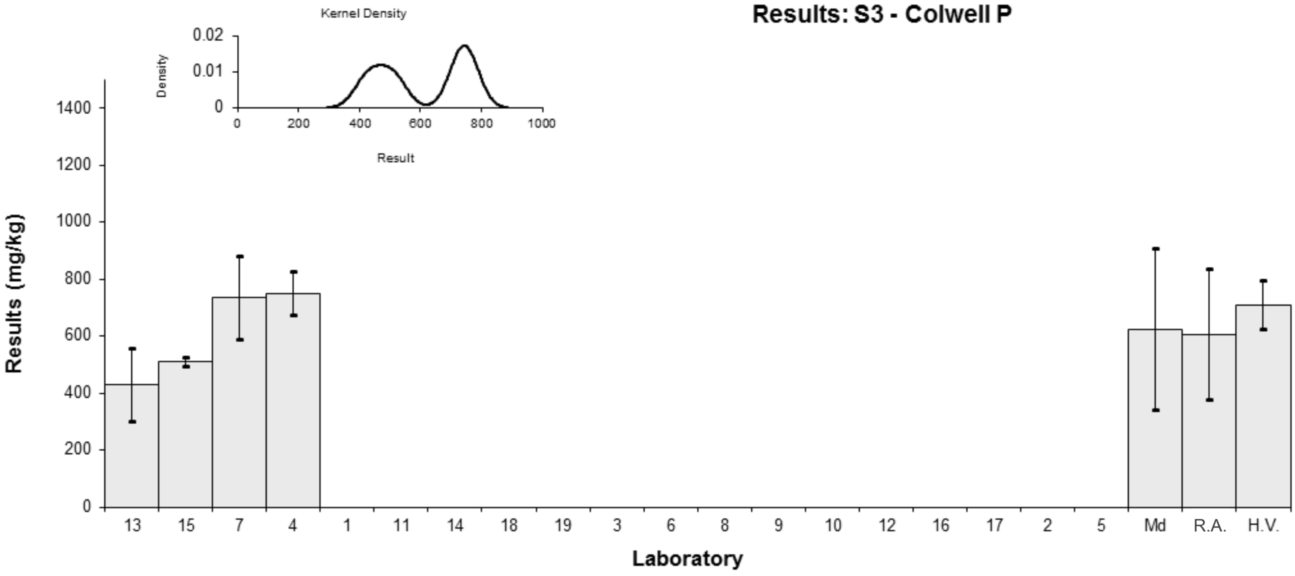

Results: S3 - Colwell P
Kernel Density
Density
0.02
0.01
0
0
200
400
600
800
1000
Result
Results (mg/kg)
1400
1200
1000
800
600
400
200
0
13
15
7
4
1
11
14
18
19
3
6
8
9
10
12
16
17
2
5
Md
R.A.
H.V.
Laboratory

Figure 30

Table 40

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	480	50	0.39	0.33
3	NR	NR		
4	452	45	-0.22	-0.20
5	464	33	0.04	0.05
6	NR	NR		
7	500	50	0.82	0.71
8	NR	NR		
9	NR	NR		
10	490	100	0.61	0.27
11	NR	NR		
12	460	64	-0.04	-0.03
13	450	20	-0.26	-0.42
14	NR	NR		
15	446	6	-0.35	-0.77
16	457	50	-0.11	-0.09
17	400	60	-1.34	-0.98
18	NR	NR		
19	NR	NR		

Statistics

Assigned Value	462	20
Spike	Not Spiked	
Homogeneity Value	400	30
Robust Average	462	20
Median	459	11
Mean	460	
N	10	
Max.	500	
Min.	400	
Robust SD	25	
Robust CV	5.4%	

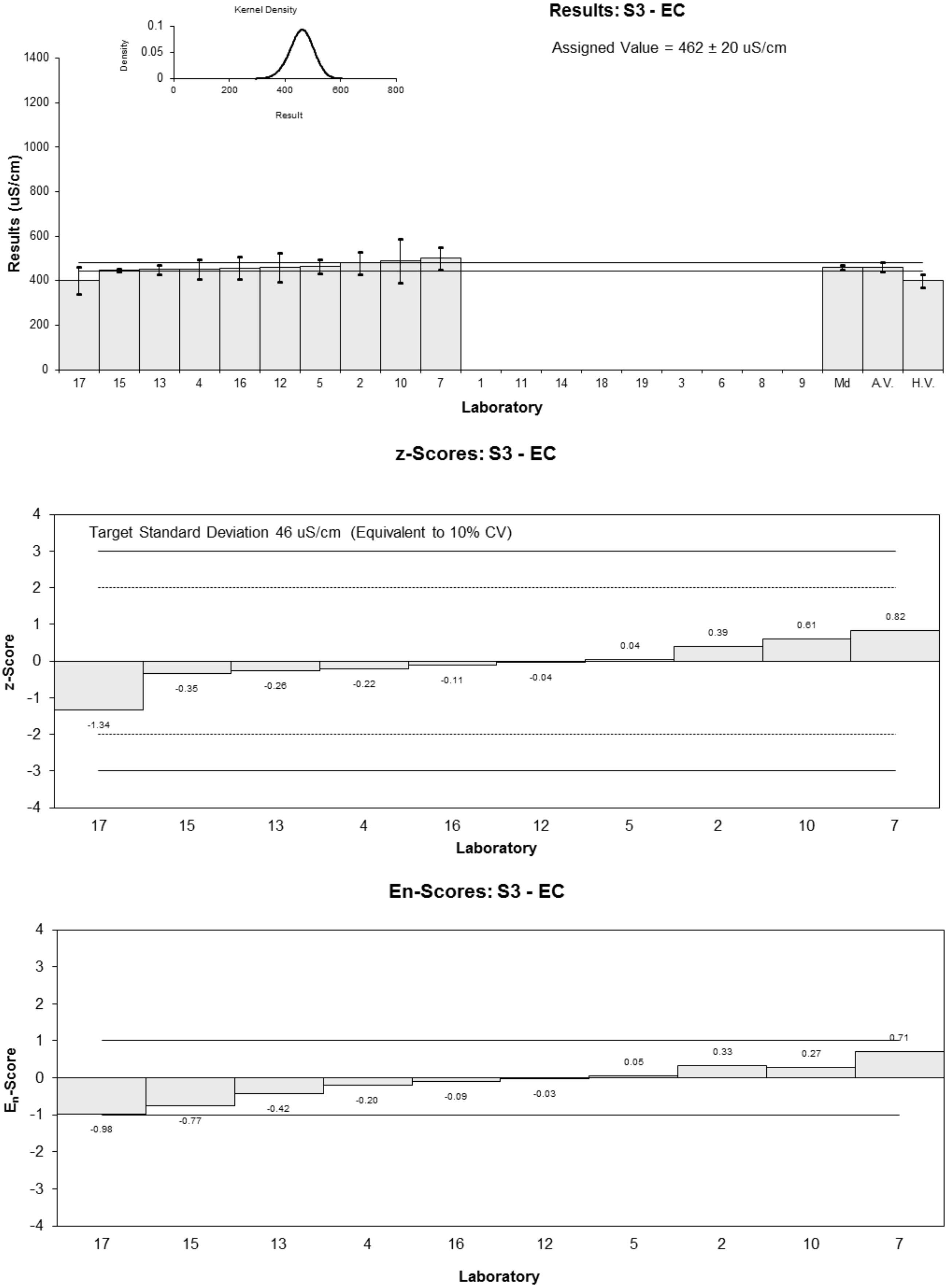

Results: S3 - EC
Assigned Value = 462 ± 20 uS/cm
Kernel Density
Density
Result
Results (uS/cm)
Laboratory
17 15 13 4 16 12 5 2 10 7 1 11 14 18 19 3 6 8 9 Md A.V. H.V.
z-Scores: S3 - EC
Target Standard Deviation 46 uS/cm (Equivalent to 10% CV)
z-Score
-1.34 -0.35 -0.26 -0.22 -0.11 -0.04 0.04 0.39 0.61 0.82
Laboratory
En-Scores: S3 - EC
En-Score
-0.98 -0.77 -0.42 -0.20 -0.09 -0.03 0.05 0.33 0.27 0.71
Laboratory

Figure 31

Table 41

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	Exchangeable Ca^{2+}
Units	cmol(+)/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	28	8	0.62	0.27
3	NR	NR		
4	20.7	2.5	-1.28	-1.08
5	20.4	3.9	-1.35	-0.95
6	NR	NR		
7	25.3	5.1	-0.08	-0.05
8	NR	NR		
9	NR	NR		
10	27	8	0.36	0.16
11	NR	NR		
12	3.4	0.8	-5.78	-5.72
13	42	12.6	4.27	1.25
14	NR	NR		
15	25.34	0.56	-0.07	-0.07
16	26	8	0.10	0.05
17	32.9	6.6	1.90	0.96
18	NR	NR		
19	NR	NR		

Statistics

Assigned Value*	25.6	3.8
Spike	Not Spiked	
Homogeneity Value	21.7	4.6
Robust Average	25.7	5.3
Median	25.7	3.9
Mean	25.1	
N	10	
Max.	42	
Min.	3.4	
Robust SD	5.5	
Robust CV	21%	

*Robust Average excluding Laboratories 12 and 13.

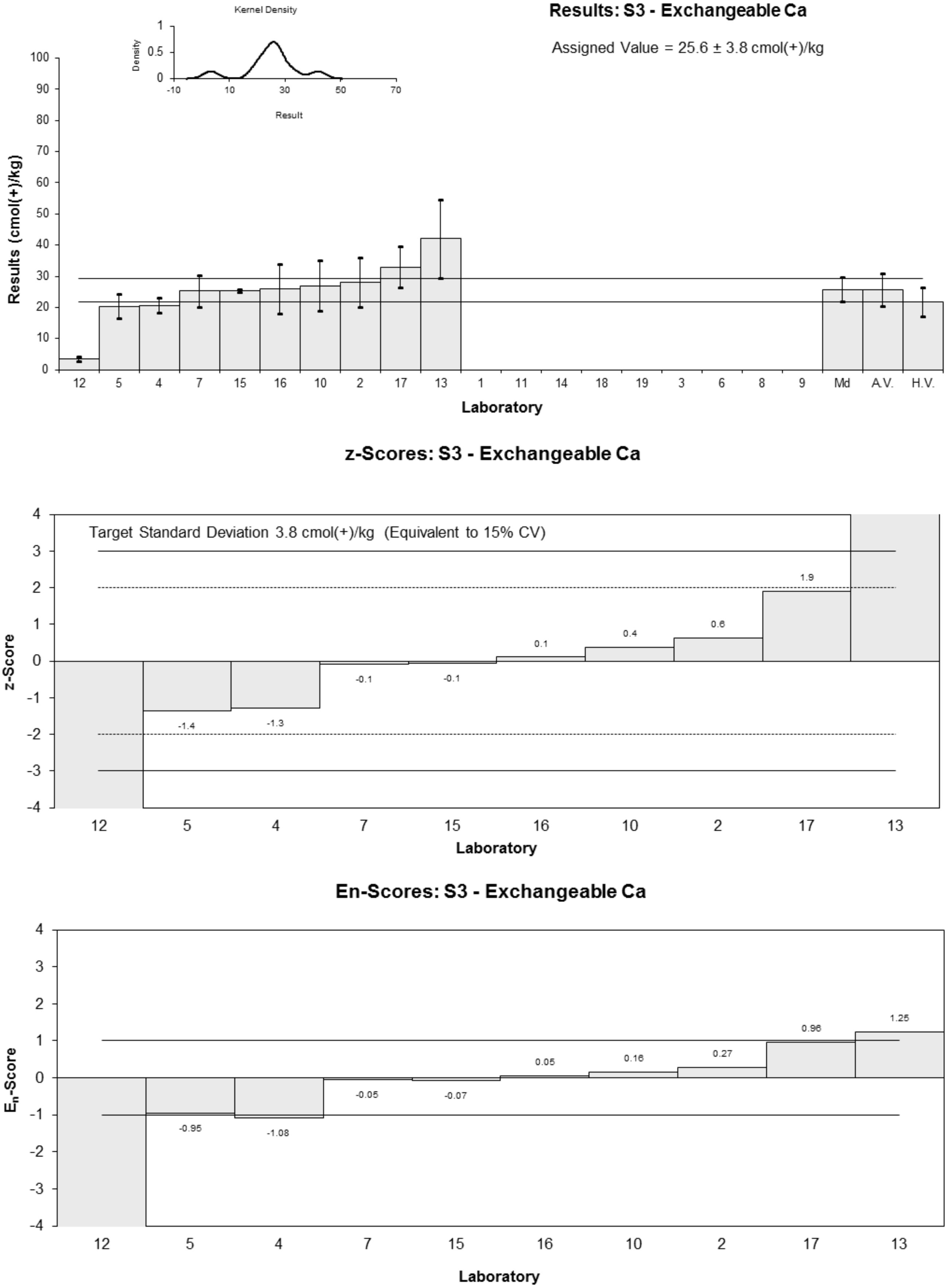

Results: S3 - Exchangeable Ca
Assigned Value = 25.6 ± 3.8 cmol(+)/kg
Kernel Density
Density
Result
Results (cmol(+)/kg)
12 5 4 7 15 16 10 2 17 13 1 11 14 18 19 3 6 8 9 Md A.V. H.V.
Laboratory
z-Scores: S3 - Exchangeable Ca
Target Standard Deviation 3.8 cmol(+)/kg (Equivalent to 15% CV)
z-Score
-1.4
-1.3
-0.1
-0.1
0.1
0.4
0.6
1.9
12 5 4 7 15 16 10 2 17 13
Laboratory
En-Scores: S3 - Exchangeable Ca
E_n-Score
-0.95
-1.08
-0.05
-0.07
0.05
0.16
0.27
0.96
1.25
12 5 4 7 15 16 10 2 17 13
Laboratory

Figure 32

Table 42

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	Exchangeable K^+
Units	cmol(+)/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	2.0	0.5	0.38	0.24
3	NR	NR		
4	1.97	0.2	0.30	0.29
5	1.3	0.2	-1.51	-1.48
6	NR	NR		
7	2.24	0.45	1.02	0.69
8	NR	NR		
9	NR	NR		
10	1.6	0.5	-0.70	-0.44
11	NR	NR		
12	3.0	0.68	3.06	1.52
13	3.6	1.1	4.68	1.52
14	NR	NR		
15	2.18	0.07	0.86	0.98
16	1.6	0.5	-0.70	-0.44
17	1.97	0.39	0.30	0.22
18	NR	NR		
19	NR	NR		

Statistics

Assigned Value*	1.86	0.32
Spike	Not Spiked	
Homogeneity Value	2.20	0.44
Robust Average	2.09	0.51
Median	1.99	0.34
Mean	2.15	
N	10	
Max.	3.6	
Min.	1.3	
Robust SD	0.37	
Robust CV	18%	

*Robust Average excluding Laboratories 12 and 13.

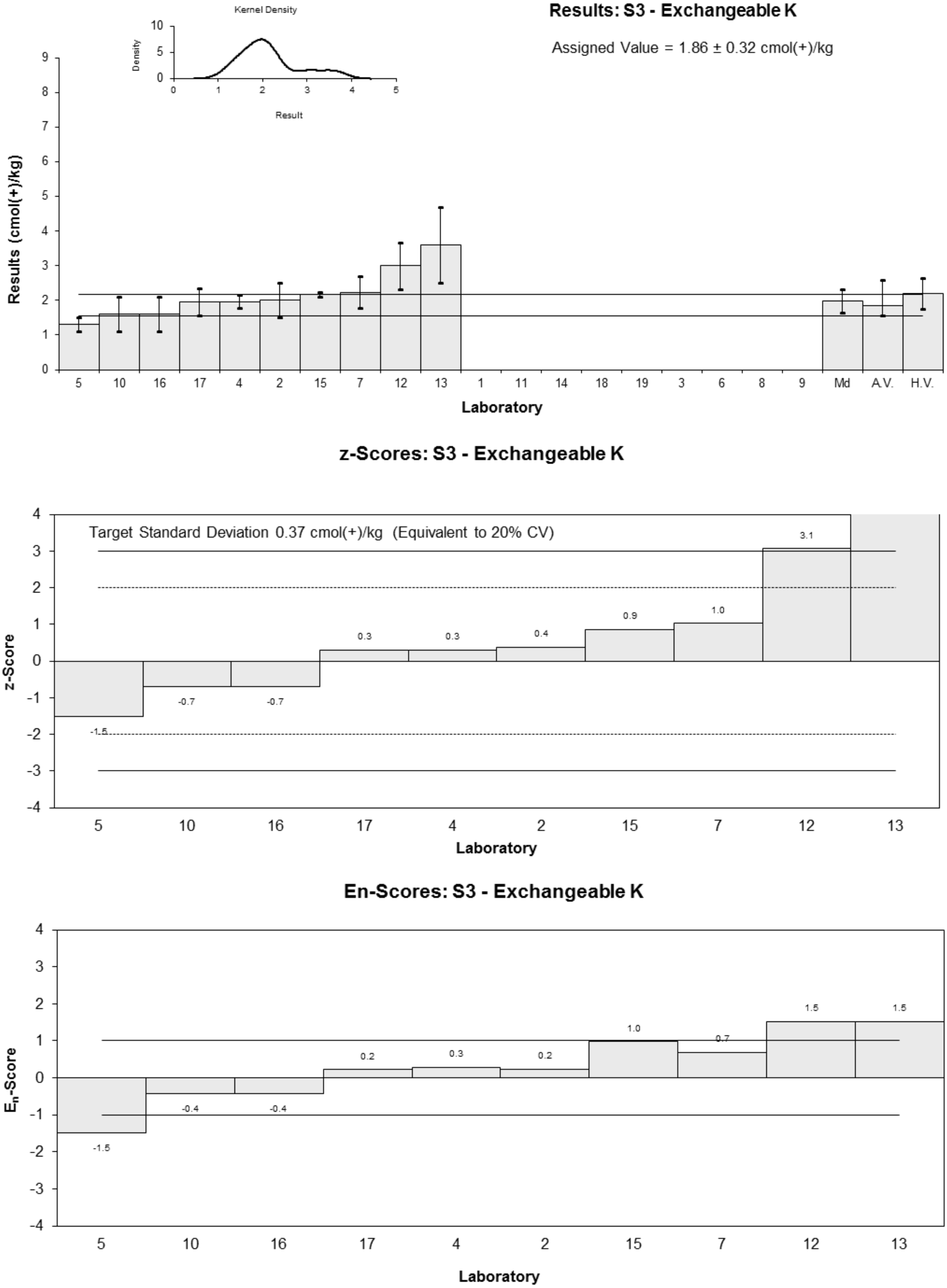

Results: S3 - Exchangeable K
Assigned Value = 1.86 ± 0.32 cmol(+)/kg
Kernel Density
Density
Result
Results (cmol(+)/kg)
5 10 16 17 4 2 15 7 12 13 1 11 14 18 19 3 6 8 9 Md A.V. H.V.
Laboratory
z-Scores: S3 - Exchangeable K
Target Standard Deviation 0.37 cmol(+)/kg (Equivalent to 20% CV)
z-Score
-1.5 -0.7 -0.7 0.3 0.3 0.4 0.9 1.0 3.1
5 10 16 17 4 2 15 7 12 13
Laboratory
En-Scores: S3 - Exchangeable K
E_n-Score
-1.5 -0.4 -0.4 0.2 0.3 0.2 1.0 0.7 1.5 1.5
5 10 16 17 4 2 15 7 12 13
Laboratory

Figure 33

Table 43

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	Exchangeable Mg^{2+}
Units	cmol(+)/kg

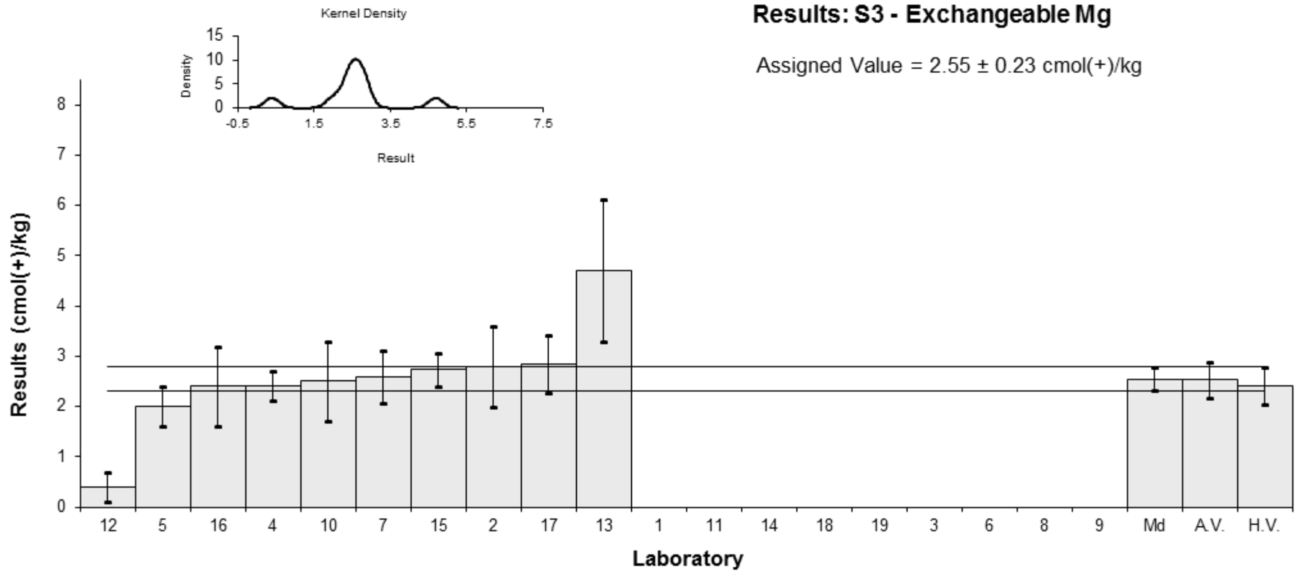
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	2.8	0.8	0.98	0.30
3	NR	NR		
4	2.41	0.3	-0.55	-0.37
5	2.0	0.4	-2.16	-1.19
6	NR	NR		
7	2.59	0.52	0.16	0.07
8	NR	NR		
9	NR	NR		
10	2.5	0.8	-0.20	-0.06
11	NR	NR		
12	0.4	0.3	-8.43	-5.69
13	4.7	1.41	8.43	1.50
14	NR	NR		
15	2.73	0.34	0.71	0.44
16	2.4	0.8	-0.59	-0.18
17	2.84	0.57	1.14	0.47
18	NR	NR		
19	NR	NR		

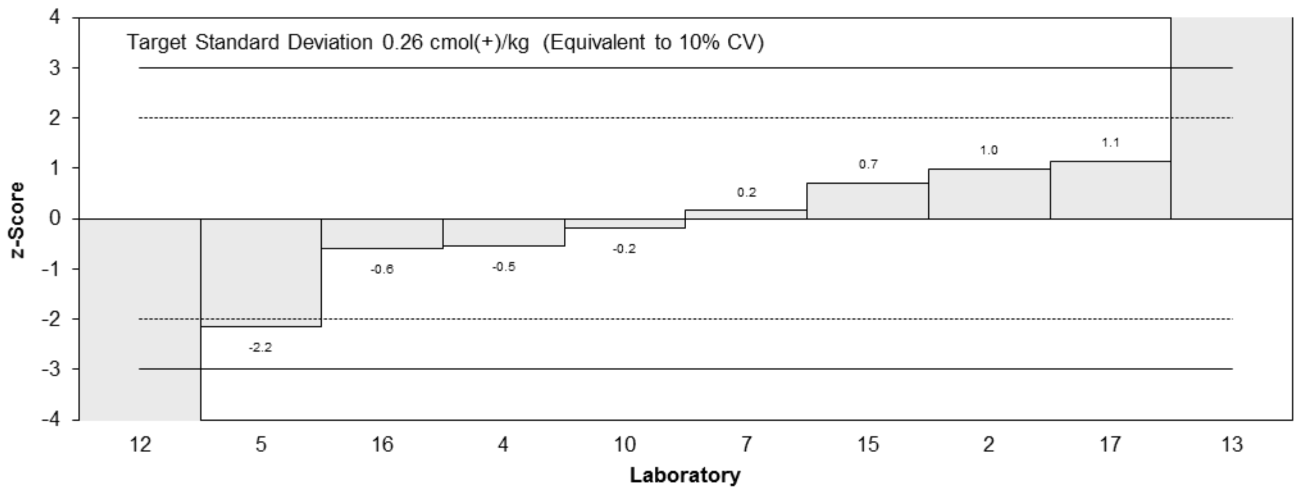
Statistics

Assigned Value*	2.55	0.23
Spike	Not Spiked	
Homogeneity Value	2.41	0.36
Robust Average	2.53	0.36
Median	2.55	0.23
Mean	2.54	
N	10	
Max.	4.7	
Min.	0.4	
Robust SD	0.26	
Robust CV	10%	

*Robust Average excluding Laboratories 12 and 13.



z-Scores: S3 - Exchangeable Mg



En-Scores: S3 - Exchangeable Mg

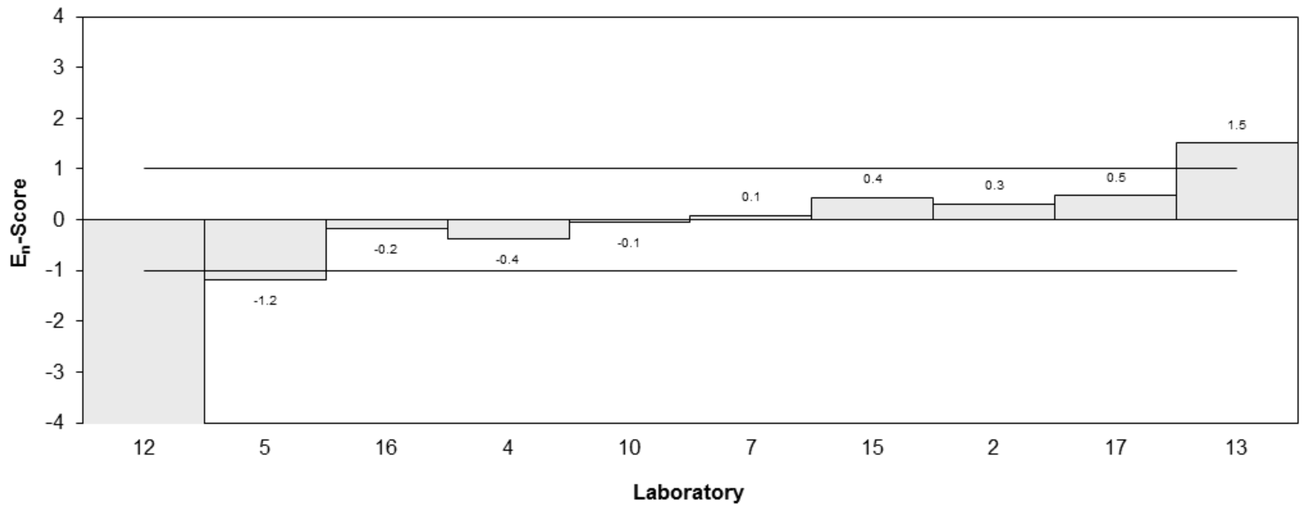


Figure 34

Table 44

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	Exchangeable Na^{+}
Units	cmol(+)/kg

Participant Results

Lab Code	Result	Uncertainty
1	NR	NR
2	0.3	0.1
3	NR	NR
4	0.092	0.01
5	<0.1	0.2
6	NR	NR
7	0.13	0.05
8	NR	NR
9	NR	NR
10	0.1	0.03
11	NR	NR
12	28	7.5
13	0.12	0.036
14	NR	NR
15	0.1	0.18
16	0.2	0.03
17	<0.5	0.1
18	NR	NR
19	NR	NR

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.136	0.027
Robust Average	0.172	0.097
Median	0.125	0.036
Mean	3.630	
N	8	
Max.	28	
Min.	0.092	
Robust SD	0.110	
Robust CV	64%	

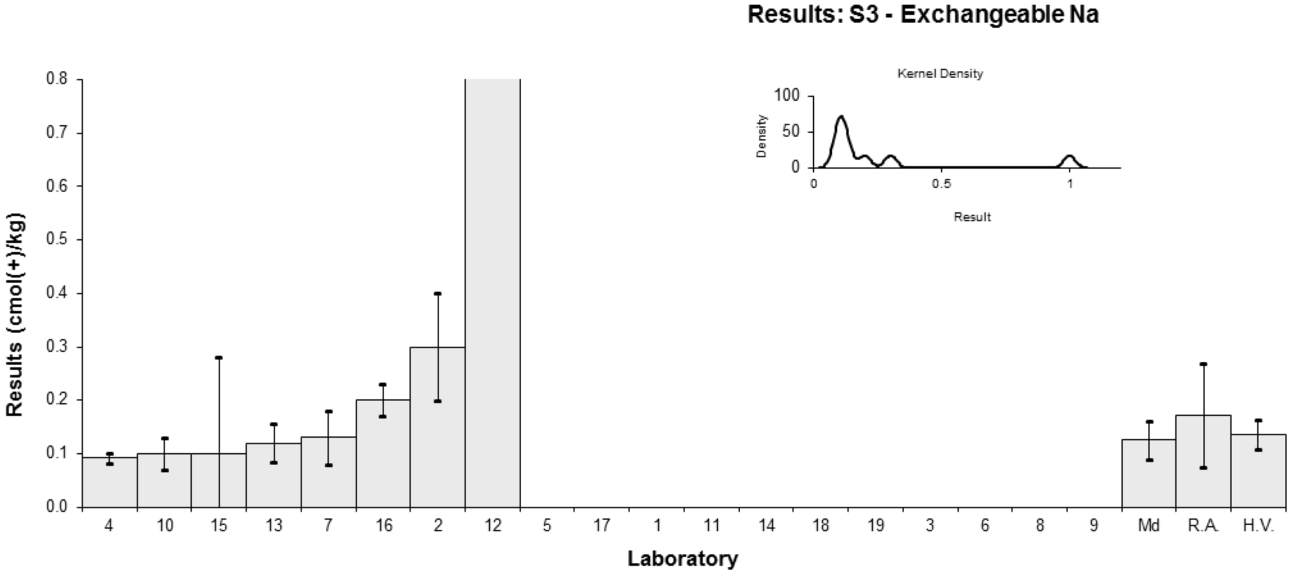

Results: S3 - Exchangeable Na
Kernel Density
Density
Result
Results (cmol(+)/kg)
Laboratory
4
10
15
13
7
16
2
12
5
17
1
11
14
18
19
3
6
8
9
Md
R.A.
H.V.

Figure 35

Table 45

Sample Details

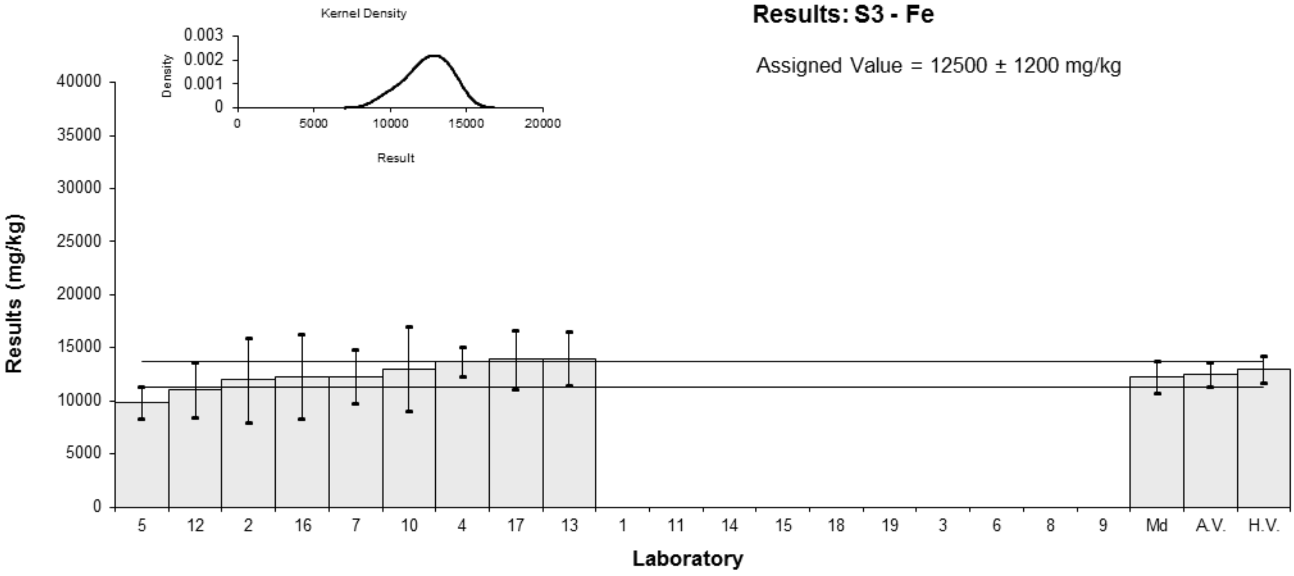
Sample No.	S3
Matrix.	Soil
Analyte.	Fe
Units	mg/kg

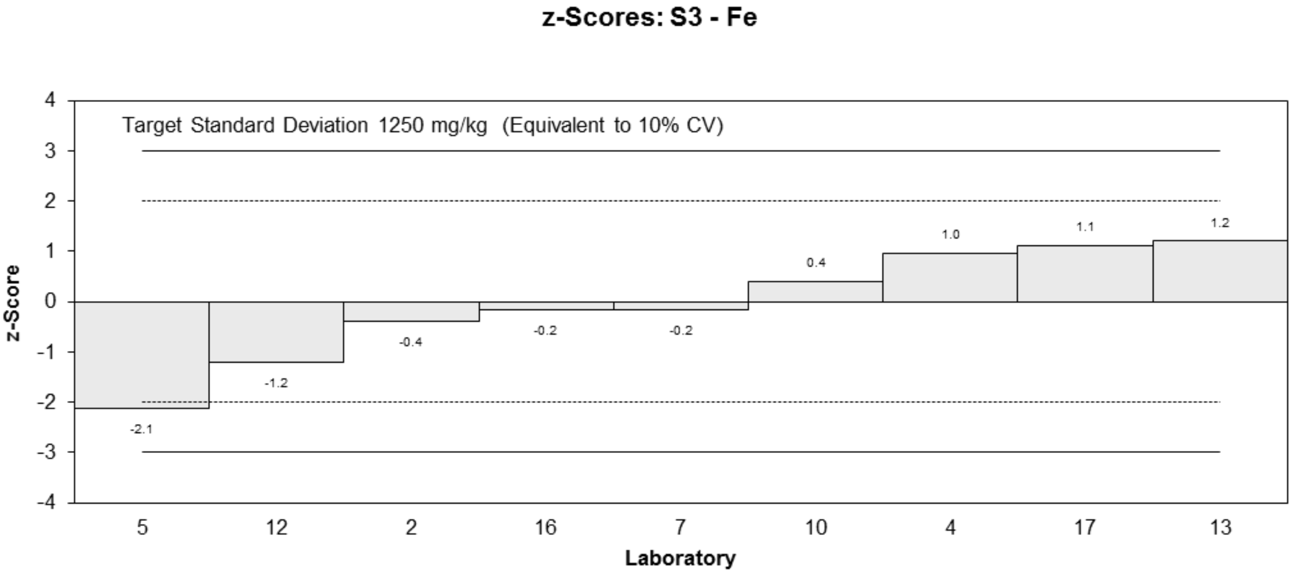
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	12000	4000	-0.40	-0.12
3	NR	NR		
4	13700	1400	0.96	0.65
5	9830	1490	-2.14	-1.40
6	NR	NR		
7	12300	2500	-0.16	-0.07
8	NR	NR		
9	NR	NR		
10	13000	4000	0.40	0.12
11	NR	NR		
12	11000	2600	-1.20	-0.52
13	14000	2505	1.20	0.54
14	NR	NR		
15	NR	NR		
16	12300	4000	-0.16	-0.05
17	13900	2800	1.12	0.46
18	NR	NR		
19	NR	NR		

Statistics

Assigned Value	12500	1200
Spike	Not Spiked	
Homogeneity Value	13000	1300
Robust Average	12500	1200
Median	12300	1500
Mean	12448	
N	9	
Max.	14000	
Min.	9830	
Robust SD	1500	
Robust CV	12%	


Results: S3 - Fe
Assigned Value = 12500 ± 1200 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
5
12
2
16
7
10
4
17
13
1
11
14
15
18
19
3
6
8
9
Md
A.V.
H.V.


z-Scores: S3 - Fe
Target Standard Deviation 1250 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-2.1
-1.2
-0.4
-0.2
-0.2
0.4
1.0
1.1
1.2

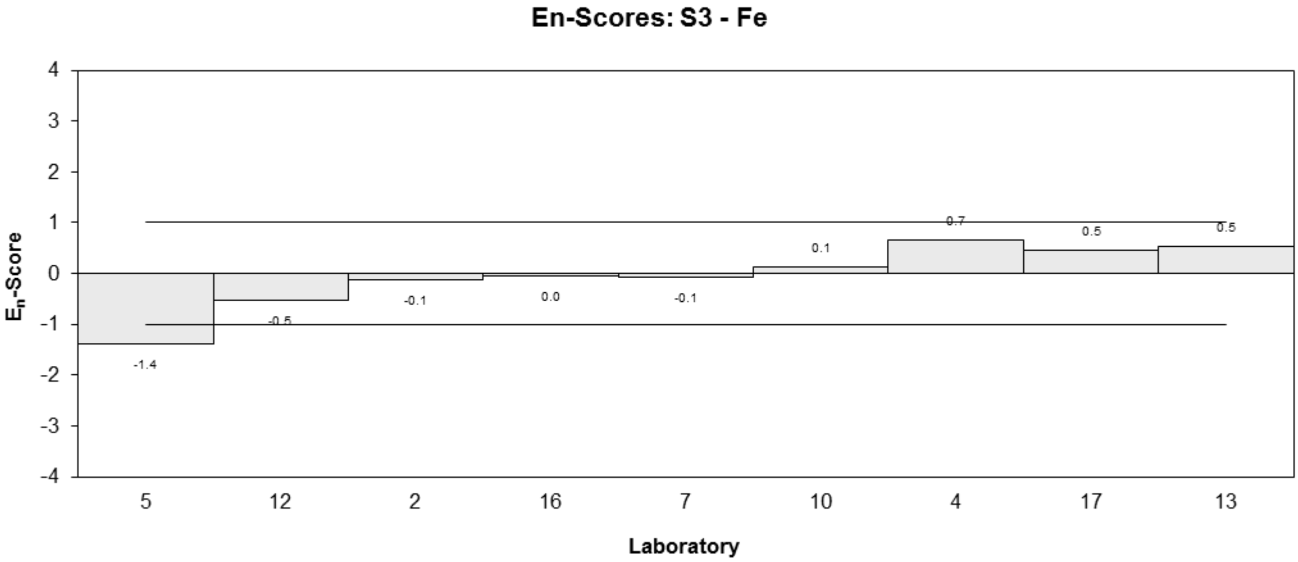

En-Scores: S3 - Fe
En-Score
Laboratory
-1.4
-0.5
-0.1
0.0
-0.1
0.1
0.7
0.5
0.5

Figure 36

Table 46

Sample Details

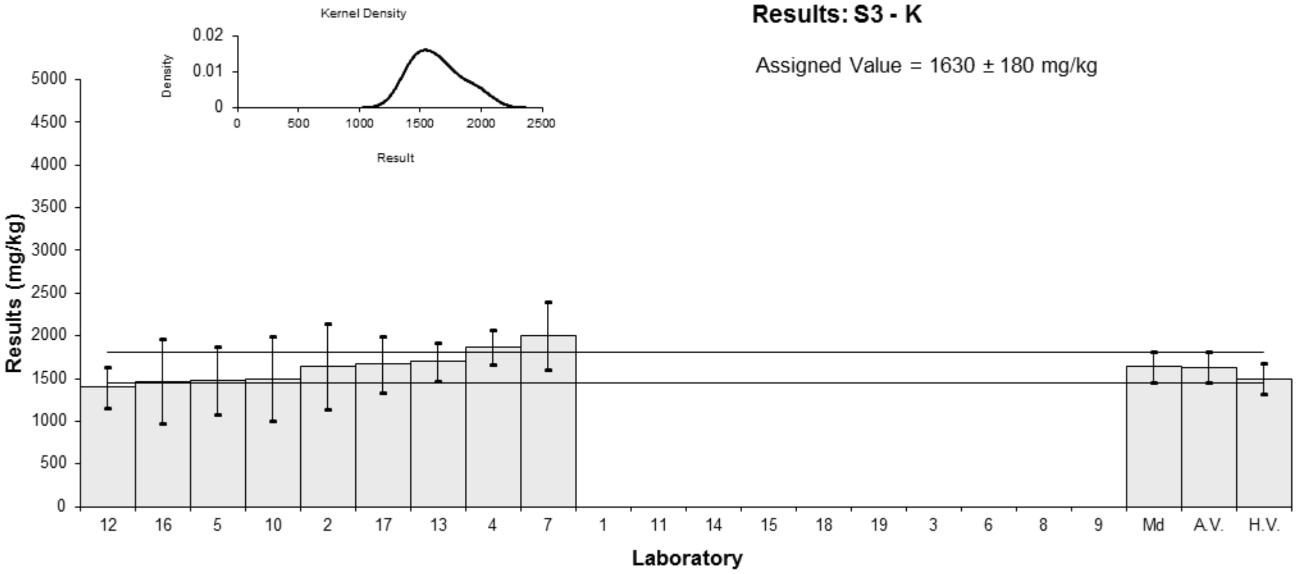
Sample No.	S3
Matrix.	Soil
Analyte.	K
Units	mg/kg

Participant Results

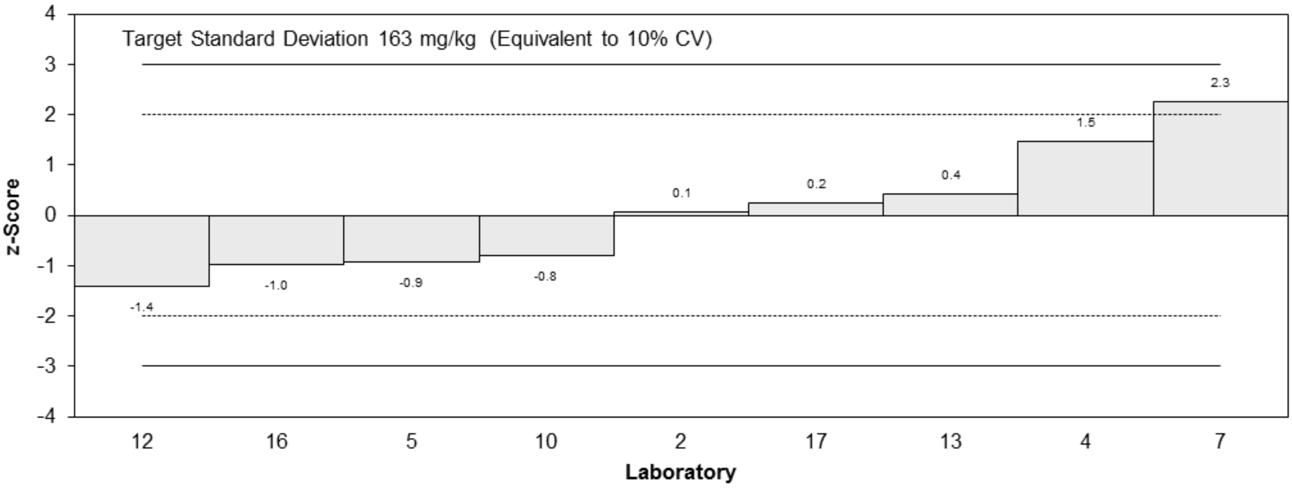
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	1640	500	0.06	0.02
3	NR	NR		
4	1870	200	1.47	0.89
5	1480	393	-0.92	-0.35
6	NR	NR		
7	2000	400	2.27	0.84
8	NR	NR		
9	NR	NR		
10	1500	500	-0.80	-0.24
11	NR	NR		
12	1400	240	-1.41	-0.77
13	1700	226	0.43	0.24
14	NR	NR		
15	NR	NR		
16	1470	500	-0.98	-0.30
17	1670	330	0.25	0.11
18	NR	NR		
19	NR	NR		

Statistics

Assigned Value	1630	180
Spike	Not Spiked	
Homogeneity Value	1500	180
Robust Average	1630	180
Median	1640	180
Mean	1637	
N	9	
Max.	2000	
Min.	1400	
Robust SD	210	
Robust CV	13%	



z-Scores: S3 - K



En-Scores: S3 - K

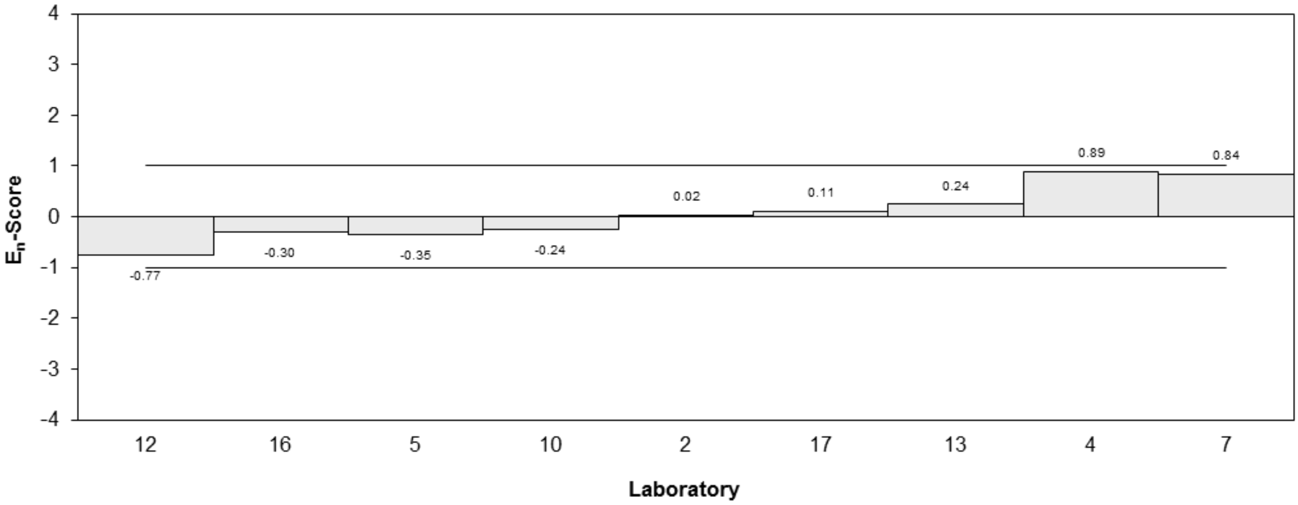


Figure 37

Table 47

Sample Details

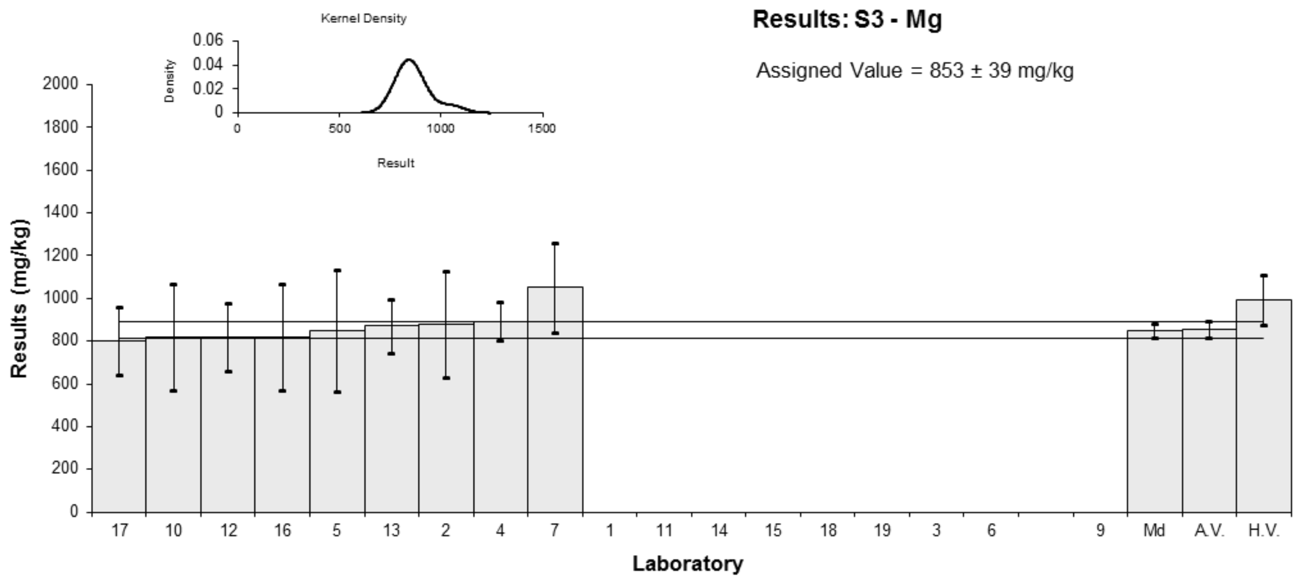
Sample No.	S3
Matrix.	Soil
Analyte.	Mg
Units	mg/kg

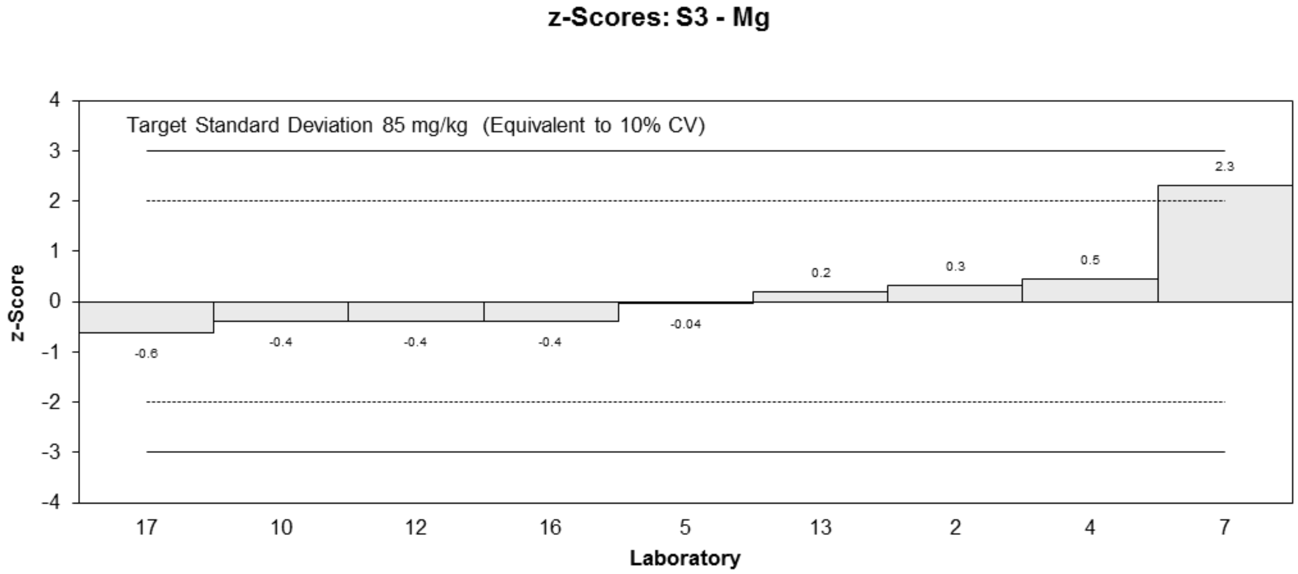
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	880	250	0.32	0.11
3	NR	NR		
4	892	90	0.46	0.40
5	850	284	-0.04	-0.01
6	NR	NR		
7	1050	210	2.31	0.92
8	NR	NR		
9	NR	NR		
10	820	250	-0.39	-0.13
11	NR	NR		
12	820	160	-0.39	-0.20
13	870	124	0.20	0.13
14	NR	NR		
15	NR	NR		
16	820	250	-0.39	-0.13
17	800	160	-0.62	-0.32
18	NR	NR		
19	NR	NR		

Statistics

Assigned Value	853	39
Spike	Not Spiked	
Homogeneity Value	993	120
Robust Average	853	39
Median	850	34
Mean	867	
N	9	
Max.	1050	
Min.	800	
Robust SD	46	
Robust CV	5.4%	


Results: S3 - Mg
Assigned Value = 853 ± 39 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
17 10 12 16 5 13 2 4 7 1 11 14 15 18 19 3 6 9 Md A.V. H.V.


z-Scores: S3 - Mg
Target Standard Deviation 85 mg/kg (Equivalent to 10% CV)
z-Score
-0.6 -0.4 -0.4 -0.4 -0.04 0.2 0.3 0.5 2.3
17 10 12 16 5 13 2 4 7
Laboratory

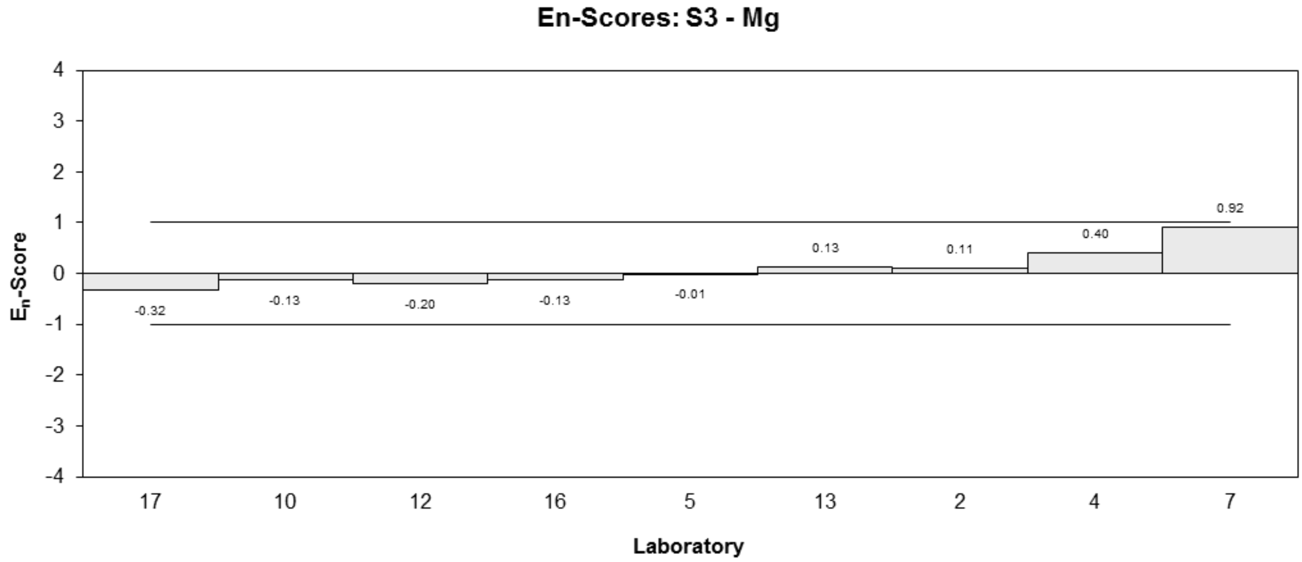

En-Scores: S3 - Mg
En-Score
-0.32 -0.13 -0.20 -0.13 -0.01 0.13 0.11 0.40 0.92
17 10 12 16 5 13 2 4 7
Laboratory

Figure 38

Table 48

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	110	40	-0.76	-0.21
3	NR	NR		
4	134	15	1.26	0.73
5	120	54.1	0.08	0.02
6	NR	NR		
7	137	27	1.51	0.59
8	NR	NR		
9	NR	NR		
10	120	40	0.08	0.02
11	NR	NR		
12	NT	NT		
13	86	19.4	-2.77	-1.38
14	NR	NR		
15	NR	NR		
16	124	40	0.42	0.12
17	109	22	-0.84	-0.38
18	NR	NR		
19	NR	NR		

Statistics

Assigned Value	119	14
Spike	Not Spiked	
Homogeneity Value	133	20
Robust Average	119	14
Median	120	13
Mean	118	
N	8	
Max.	137	
Min.	86	
Robust SD	15	
Robust CV	13%	

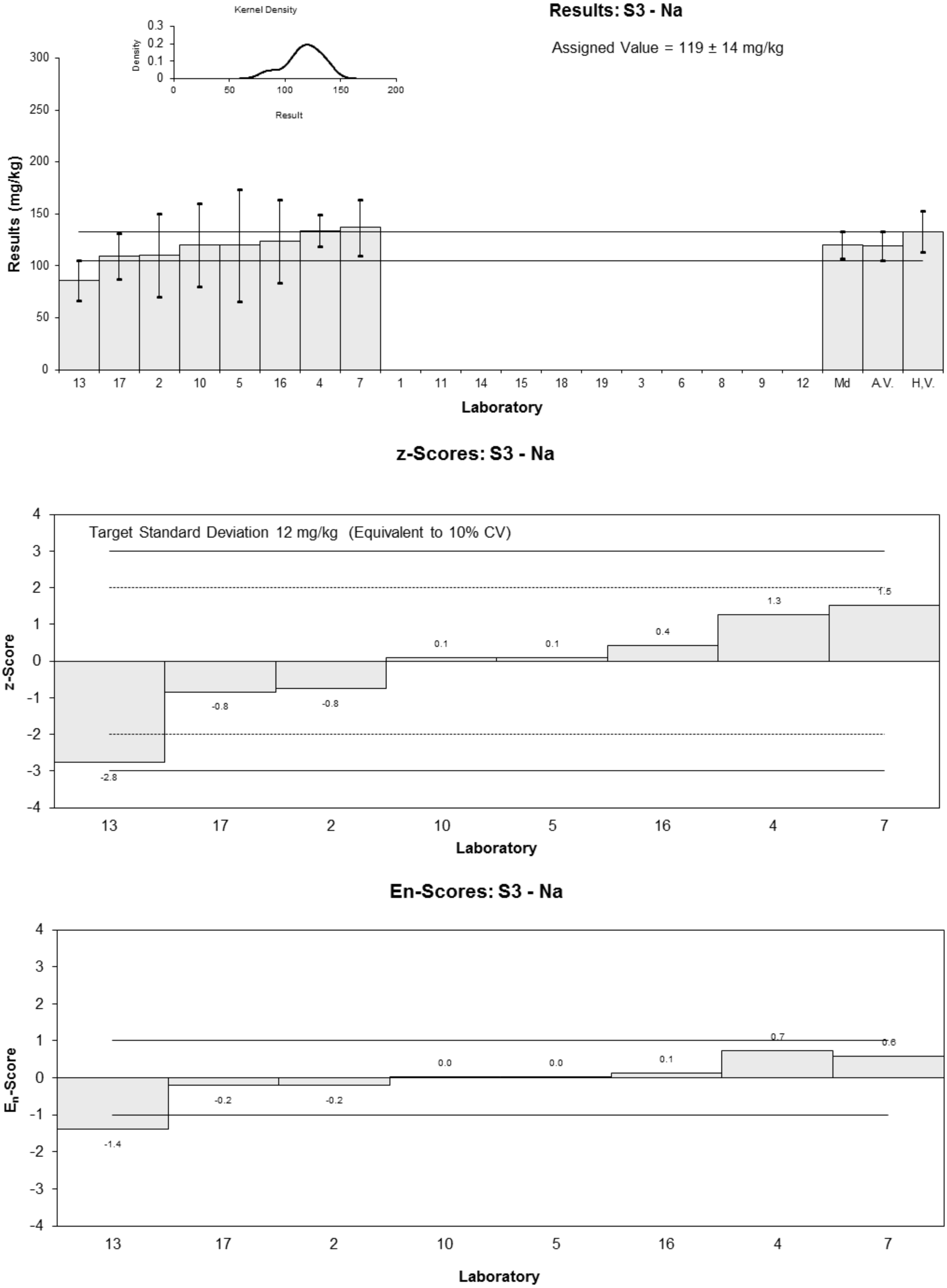

Results: S3 - Na
Assigned Value = 119 ± 14 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
13 17 2 10 5 16 4 7 1 11 14 15 18 19 3 6 8 9 12 Md A.V. H,V.
Laboratory
z-Scores: S3 - Na
Target Standard Deviation 12 mg/kg (Equivalent to 10% CV)
z-Score
-2.8 -0.8 -0.8 0.1 0.1 0.4 1.3 1.5
13 17 2 10 5 16 4 7
Laboratory
En-Scores: S3 - Na
En-Score
-1.4 -0.2 -0.2 0.0 0.0 0.1 0.7 0.6
13 17 2 10 5 16 4 7
Laboratory

Figure 39

Table 49

Sample Details

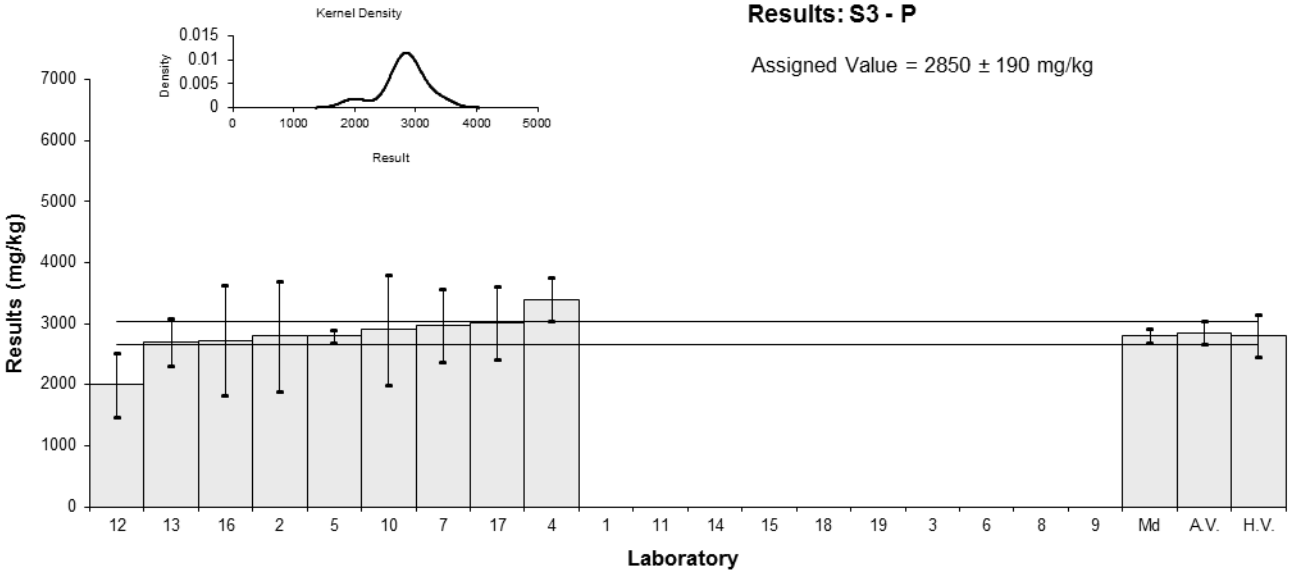
Sample No.	S3
Matrix.	Soil
Analyte.	P
Units	mg/kg

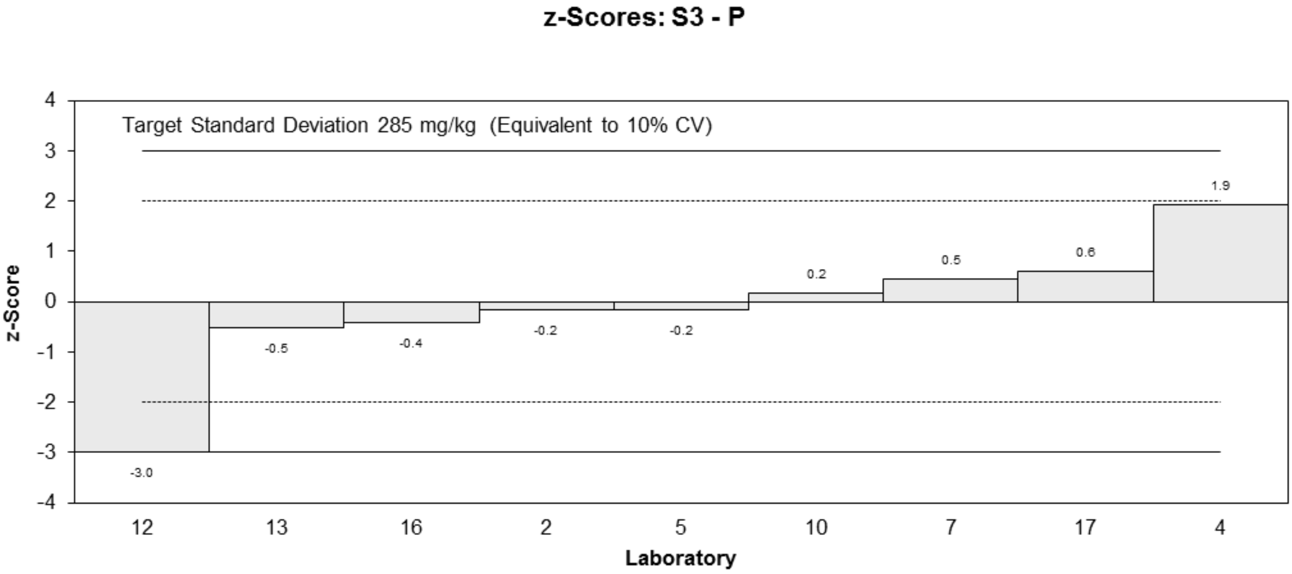
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	2800	900	-0.18	-0.05
3	NR	NR		
4	3400	350	1.93	1.38
5	2800	105	-0.18	-0.23
6	NR	NR		
7	2980	600	0.46	0.21
8	NR	NR		
9	NR	NR		
10	2900	900	0.18	0.05
11	NR	NR		
12	2000	530	-2.98	-1.51
13	2700	397	-0.53	-0.34
14	NR	NR		
15	NR	NR		
16	2730	900	-0.42	-0.13
17	3020	600	0.60	0.27
18	NR	NR		
19	NR	NR		

Statistics

Assigned Value	2850	190
Spike	Not Spiked	
Homogeneity Value	2800	340
Robust Average	2850	190
Median	2800	110
Mean	2814	
N	9	
Max.	3400	
Min.	2000	
Robust SD	230	
Robust CV	8.1%	


Results: S3 - P
Assigned Value = 2850 ± 190 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
12 13 16 2 5 10 7 17 4 1 11 14 15 18 19 3 6 8 9 Md A.V. H.V.


z-Scores: S3 - P
Target Standard Deviation 285 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
12 13 16 2 5 10 7 17 4
-3.0 -0.5 -0.4 -0.2 -0.2 0.2 0.5 0.6 1.9

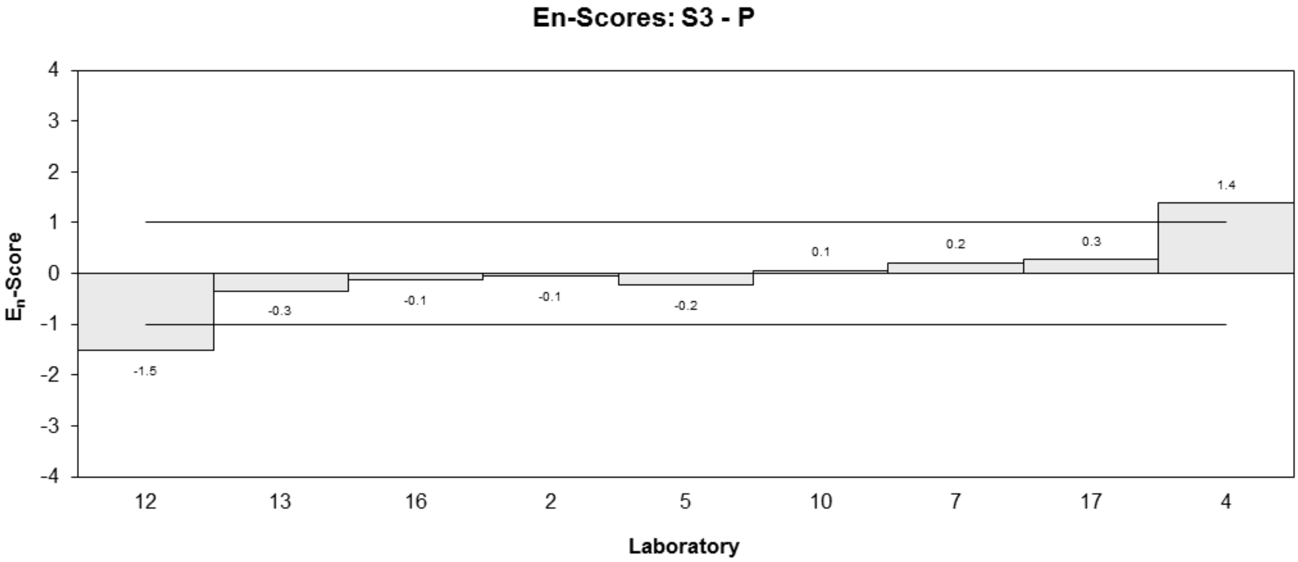

En-Scores: S3 - P
En-Score
Laboratory
12 13 16 2 5 10 7 17 4
-1.5 -0.3 -0.1 -0.1 -0.2 0.1 0.2 0.3 1.4

Figure 40

Table 50

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	pH

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	6.3	0.2	-0.16	-0.37
3	NR	NR		
4	6.41	0.1	0.02	0.05
5	NT	NT		
6	NR	NR		
7	6.2	0.2	-0.31	-0.74
8	NR	NR		
9	NR	NR		
10	6.3	0.5	-0.16	-0.19
11	NR	NR		
12	6.1	0.2	-0.47	-1.11
13	6.7	0.33	0.47	0.80
14	NR	NR		
15	6.49	0.082	0.14	0.46
16	6.49	0.5	0.14	0.17
17	6.6	0.66	0.31	0.29
18	NR	NR		
19	NR	NR		

Statistics

Assigned Value	6.40	0.18
Spike	Not Spiked	
Homogeneity Value	6.10	0.15
Robust Average	6.40	0.18
Median	6.41	0.13
Mean	6.40	
N	9	
Max.	6.7	
Min.	6.1	
Robust SD	0.22	
Robust CV	3.4%	

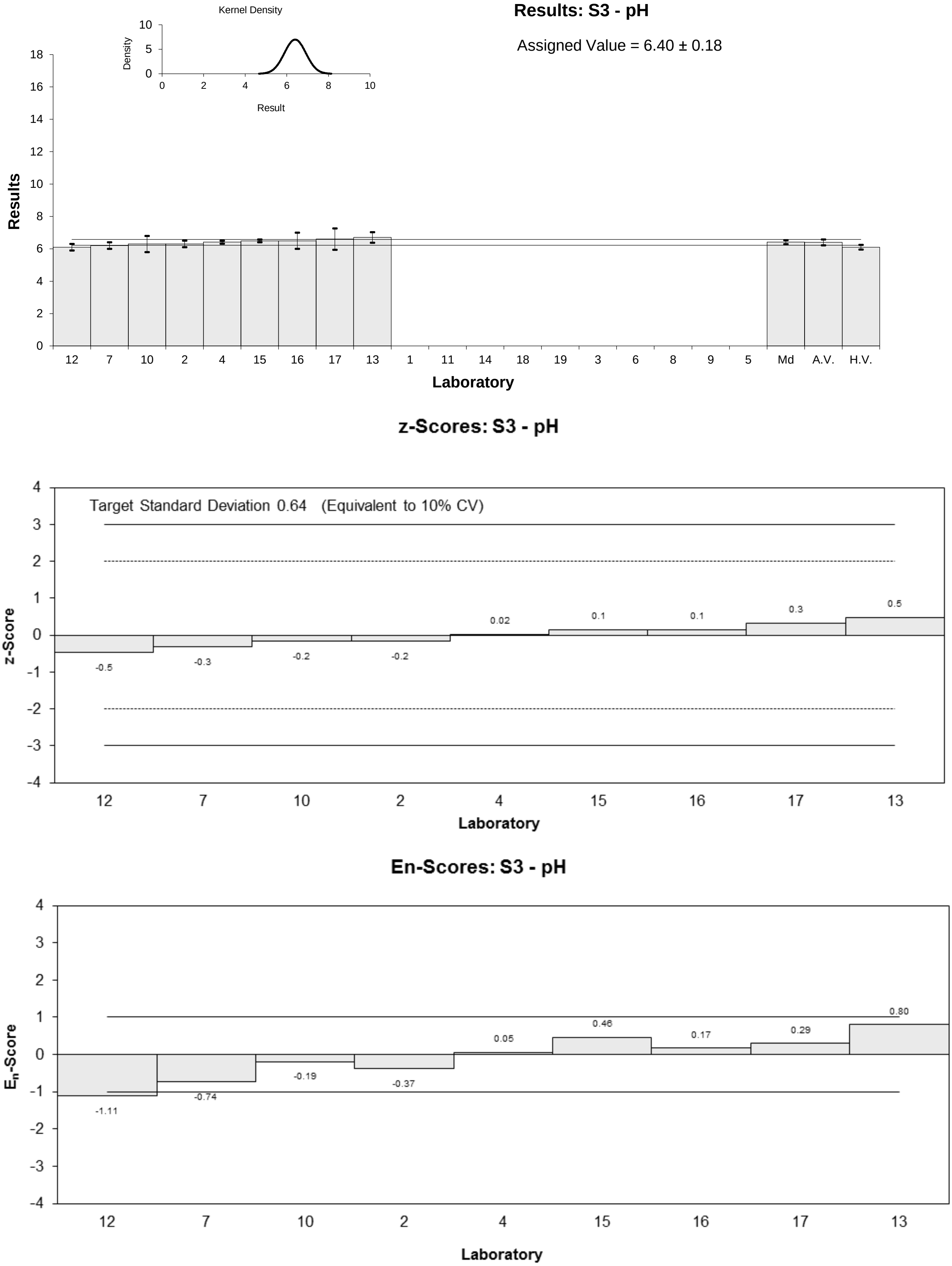

Results: S3 - pH
Assigned Value = 6.40 ± 0.18
Kernel Density
Density
Result
Results
Laboratory
12 7 10 2 4 15 16 17 13 1 11 14 18 19 3 6 8 9 5 Md A.V. H.V.
z-Scores: S3 - pH
Target Standard Deviation 0.64 (Equivalent to 10% CV)
z-Score
-0.5 -0.3 -0.2 -0.2 0.02 0.1 0.1 0.3 0.5
Laboratory
12 7 10 2 4 15 16 17 13
En-Scores: S3 - pH
E_n-Score
-1.11 -0.74 -0.19 -0.37 0.05 0.46 0.17 0.29 0.80
Laboratory
12 7 10 2 4 15 16 17 13

Figure 41

Table 51

Sample Details

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

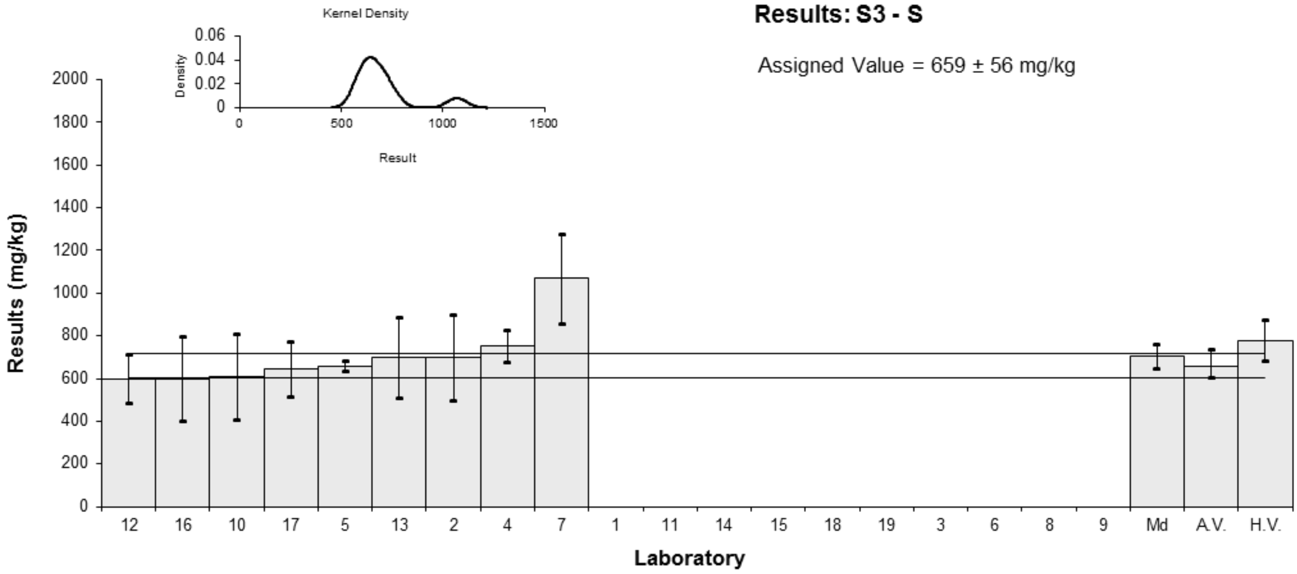
Participant Results

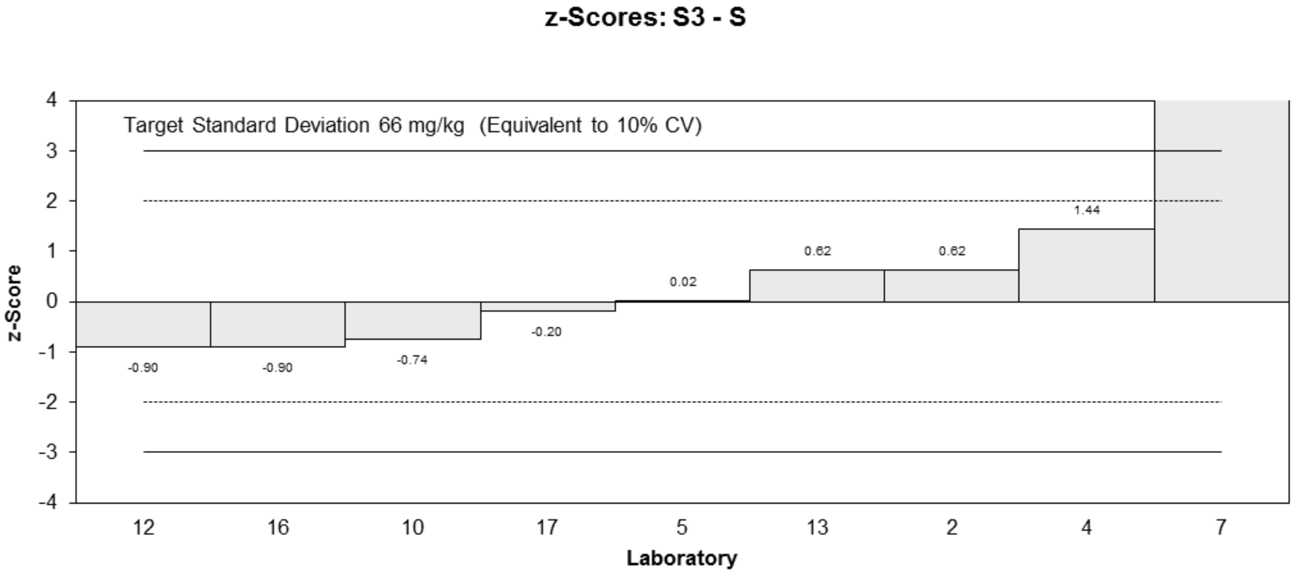
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	700	200	0.62	0.20
3	NR	NR		
4	754	75	1.44	1.01
5	660	22	0.02	0.02
6	NR	NR		
7	1070	210	6.24	1.89
8	NR	NR		
9	NR	NR		
10	610	200	-0.74	-0.24
11	NR	NR		
12	600	114	-0.90	-0.46
13	700	190	0.62	0.21
14	NR	NR		
15	NR	NR		
16	600	200	-0.90	-0.28
17	646	129	-0.20	-0.09
18	NR	NR		
19	NR	NR		

Statistics

Assigned Value*	659	56
Spike	Not Spiked	
Homogeneity Value	780	94
Robust Average	673	64
Median	704	57
Mean	704	
N	9	
Max.	1070	
Min.	600	
Robust SD	63	
Robust CV	9.4%	

*Robust Average excluding Laboratory 7.


Results: S3 - S
Assigned Value = 659 ± 56 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
12 16 10 17 5 13 2 4 7 1 11 14 15 18 19 3 6 8 9 Md A.V. H.V.


z-Scores: S3 - S
Target Standard Deviation 66 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-0.90 -0.90 -0.74 -0.20 0.02 0.62 0.62 1.44
12 16 10 17 5 13 2 4 7

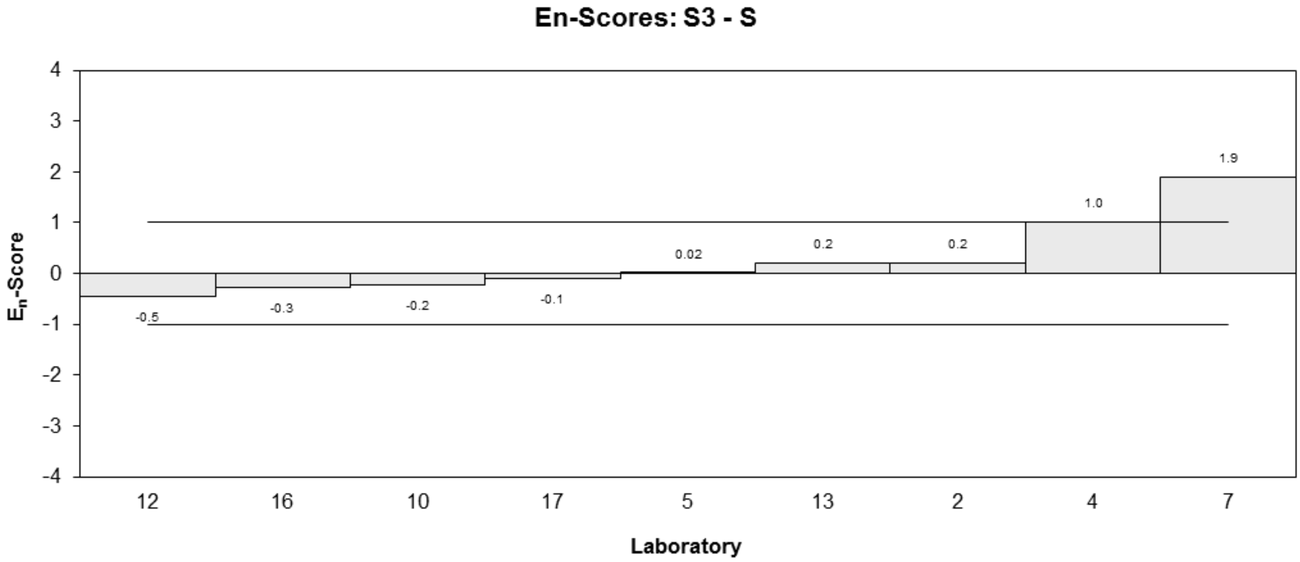

En-Scores: S3 - S
En-Score
Laboratory
-0.5 -0.3 -0.2 -0.1 0.02 0.2 0.2 1.0 1.9
12 16 10 17 5 13 2 4 7

Figure 42

Table 52

Sample Details

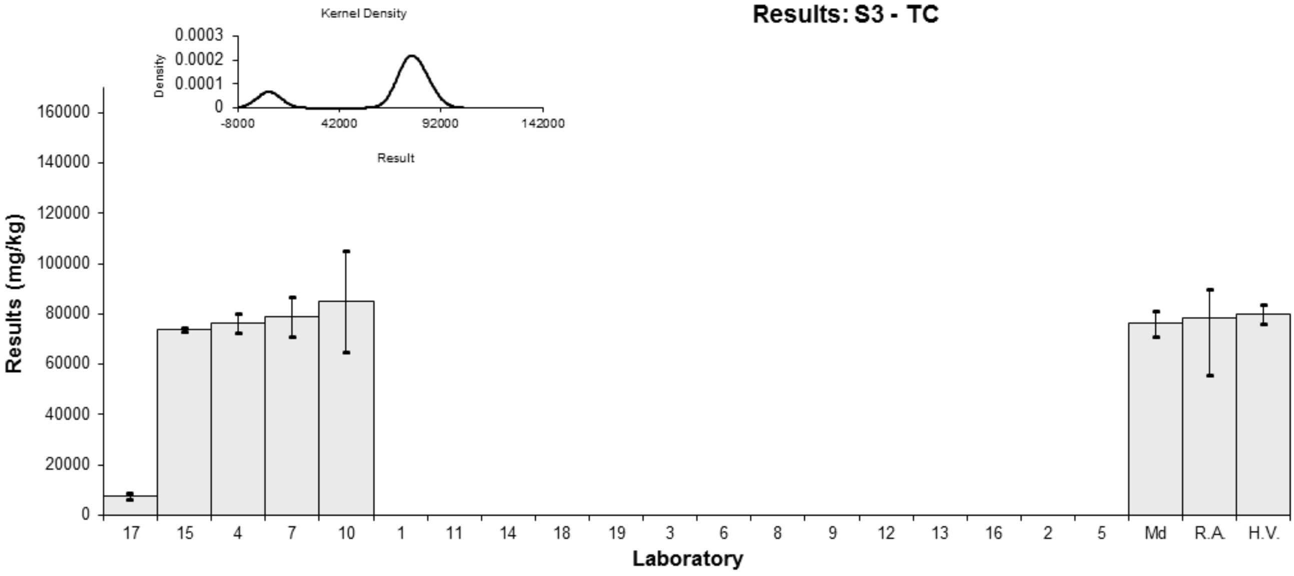
Sample No.	S3
Matrix.	Soil
Analyte.	TC
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	NR	NR
2	NT	NT
3	NR	NR
4	76200	3800
5	NT	NT
6	NR	NR
7	79000	7900
8	NR	NR
9	NR	NR
10	85000	20000
11	NR	NR
12	NT	NT
13	NT	NT
14	NR	NR
15	73992	800
16	NT	NT
17	7470	1195
18	NR	NR
19	NR	NR

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	80000	4000
Robust Average	72900	17000
Median	76200	5200
Mean	64332	
N	5	
Max.	85000	
Min.	7470	
Robust SD	15000	
Robust CV	21%	



Results: S3 - TC
Kernel Density
Density
0.0003
0.0002
0.0001
0
-8000
42000
92000
142000
Result
Results (mg/kg)
160000
140000
120000
100000
80000
60000
40000
20000
0
17
15
4
7
10
1
11
14
18
19
3
6
8
9
12
13
16
2
5
Md
R.A.
H.V.
Laboratory

Figure 43

Table 53

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	5800	1800	-0.02	-0.01
3	NR	NR		
4	5900	600	0.15	0.14
5	5956	1900	0.25	0.08
6	NR	NR		
7	6100	920	0.50	0.31
8	NR	NR		
9	NR	NR		
10	5300	2000	-0.88	-0.25
11	NR	NR		
12	NT	NT		
13	<0.01	NR		
14	NR	NR		
15	5850	100	0.07	0.15
16	NT	NT		
17	5621	836	-0.33	-0.22
18	NR	NR		
19	NR	NR		

Statistics

Assigned Value	5810	240
Spike	Not Spiked	
Homogeneity Value	6300	760
Robust Average	5810	240
Median	5850	150
Mean	5790	
N	7	
Max.	6100	
Min.	5300	
Robust SD	250	
Robust CV	4.3%	

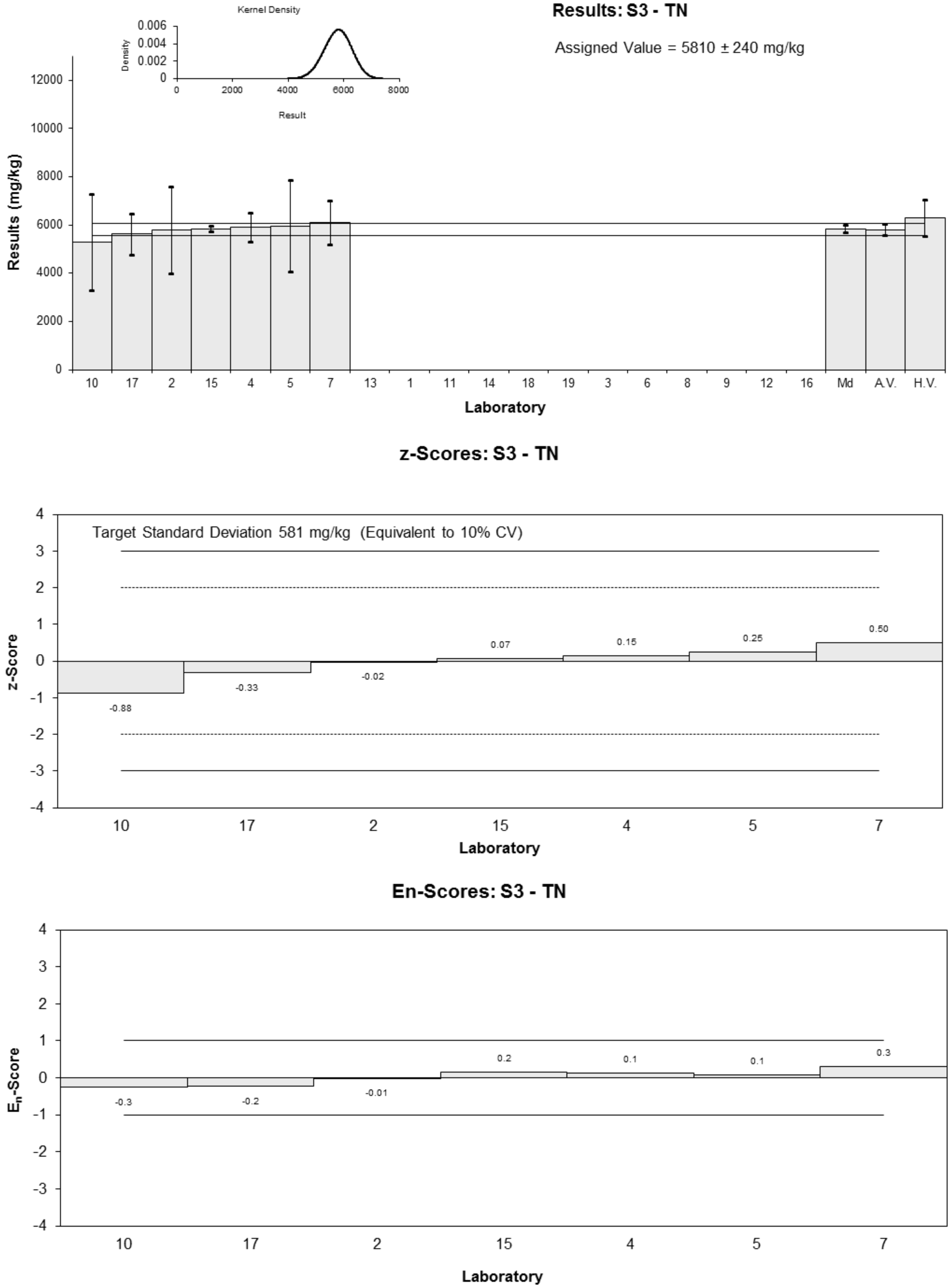

Results: S3 - TN
Assigned Value = 5810 ± 240 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
10 17 2 15 4 5 7 13 1 11 14 18 19 3 6 8 9 12 16 Md A.V. H.V.
z-Scores: S3 - TN
Target Standard Deviation 581 mg/kg (Equivalent to 10% CV)
z-Score
-0.88 -0.33 -0.02 0.07 0.15 0.25 0.50
10 17 2 15 4 5 7
Laboratory
En-Scores: S3 - TN
En-Score
-0.3 -0.2 -0.01 0.2 0.1 0.1 0.3
10 17 2 15 4 5 7
Laboratory

Figure 44

Table 54

Sample Details

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

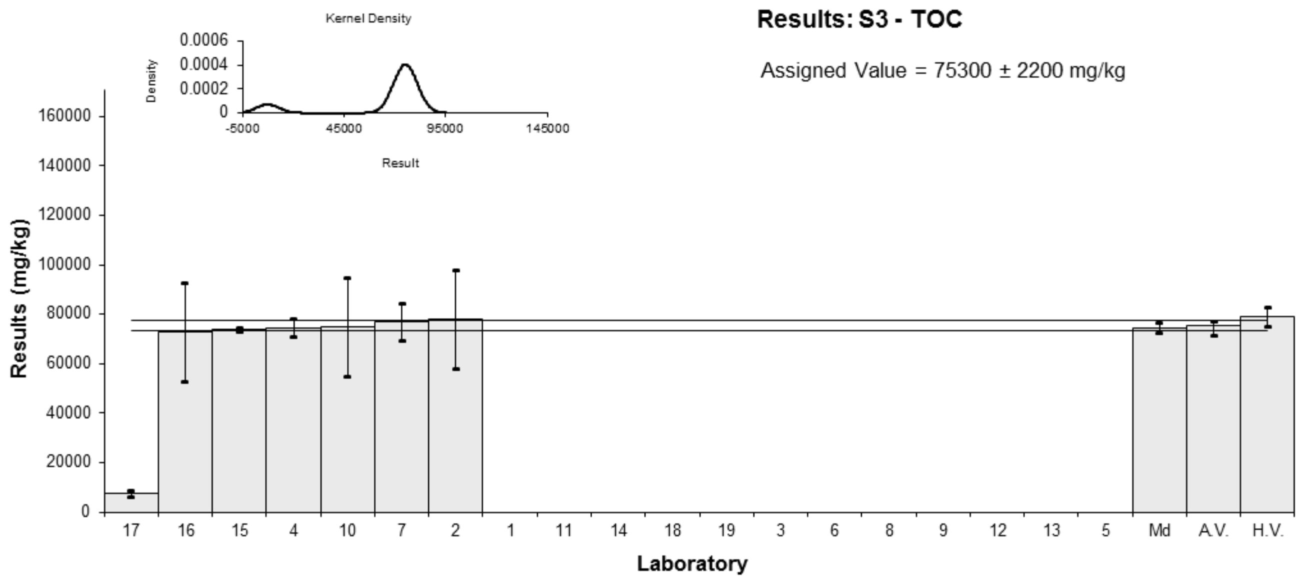
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NR	NR		
2	78000	20000	0.36	0.13
3	NR	NR		
4	74600	3800	-0.09	-0.16
5	NT	NT		
6	NR	NR		
7	77000	7700	0.23	0.21
8	NR	NR		
9	NR	NR		
10	75000	20000	-0.04	-0.01
11	NR	NR		
12	NT	NT		
13	NT	NT		
14	NR	NR		
15	73919	853	-0.18	-0.59
16	73000	20000	-0.31	-0.11
17	7470	1195	-9.01	-27.09
18	NR	NR		
19	NR	NR		

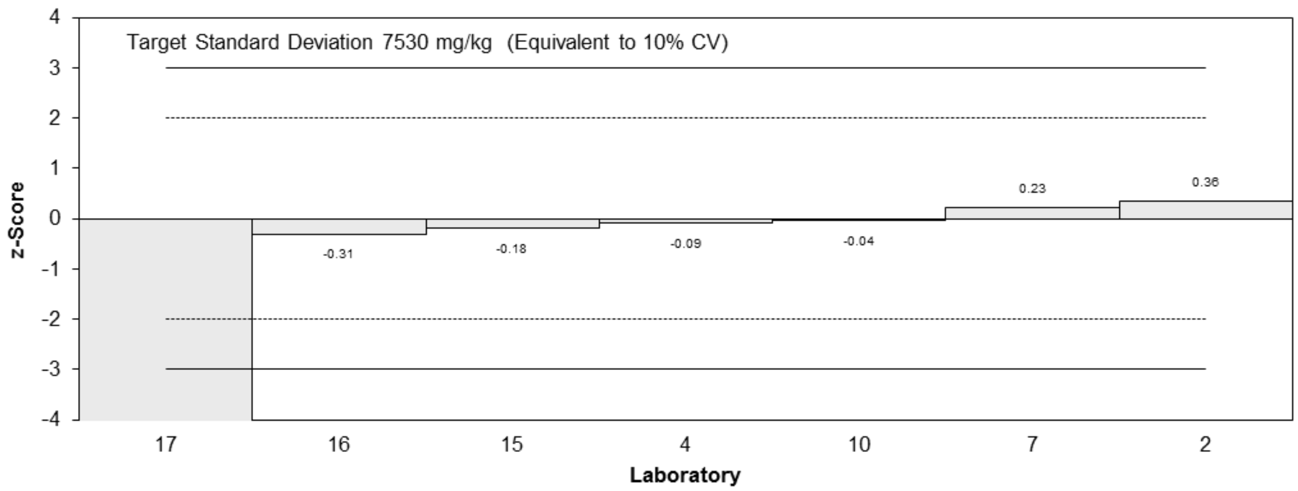
Statistics

Assigned Value*	75300	2200
Spike	Not Spiked	
Homogeneity Value	79000	4000
Robust Average	74500	2800
Median	74600	2200
Mean	65570	
N	7	
Max.	78000	
Min.	7470	
Robust SD	2100	
Robust CV	2.8%	

*Robust Average excluding Laboratory 17.



z-Scores: S3 - TOC



En-Scores: S3 - TOC

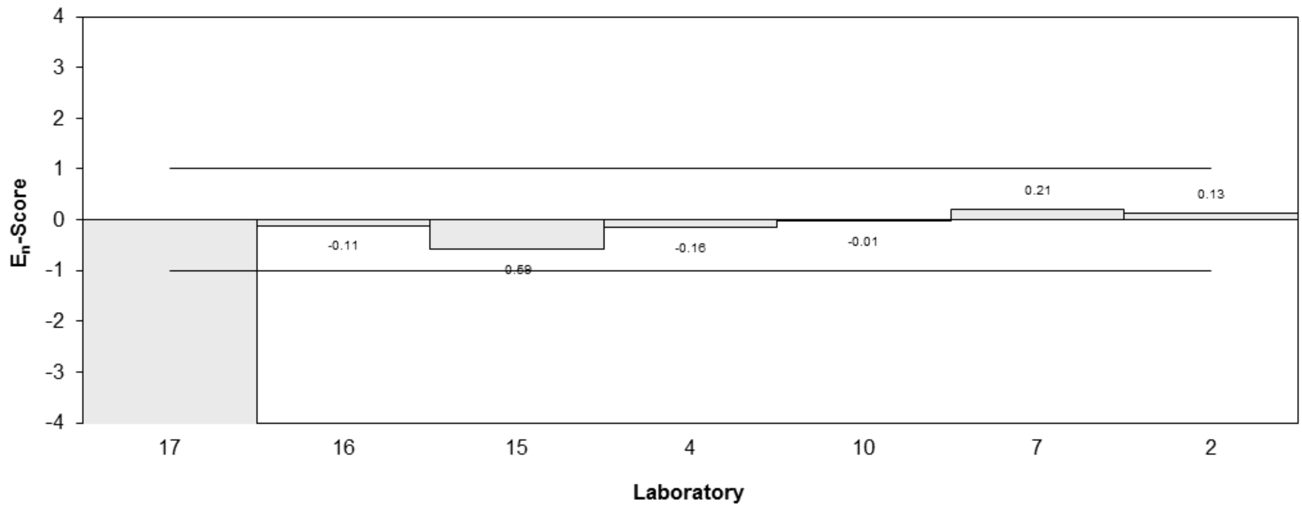


Figure 45

7 DISCUSSION OF RESULTS

7.1 Assigned Value

Samples S1 and S2 were dried soil samples prepared from the same material (compost: soil, leaves, grass and roots). The analytes of interest in these samples were incurred.

Sample S3 was dried agricultural soil.

Assigned values for the 45 tests in the study samples were the robust averages of participants' results. The robust averages used as assigned values and their associated expanded uncertainties were calculated using the procedures described in 'ISO 13528:2015(E). Results less than 50% and more than 150% of the robust average were removed before calculation of each assigned value.[8] Appendix 2 sets out the calculation for the robust average of Mn in Sample S1 and its associated uncertainty.

No assigned value was set for Cs in S2 and Colwell P and Exchangeable Na^+ in S3 because too few results were reported for these tests.

Traceability The assigned values are not traceable to any external reference; they are traceable to the consensus of participants' results derived 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 497 numerical results, 496 were reported with an expanded measurement uncertainty, indicating that all laboratories have addressed this requirement of ISO 17025.[10] The participants used a wide variety of procedures to estimate the expanded measurement uncertainty. These are presented in Tables 8 and 9.

Approaches to estimating measurement uncertainty include: standard deviation of replicate analysis, long term reproducibility, professional judgement, 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.[11–18]

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 45). In this study, the reported expanded measurement uncertainty has been over-estimated in some cases (e.g. Lab 13 for As and Cd in S1 and Ag and B in S2, Lab 9 for Hg and Sn in S1 or Lab 5 for Na in S3) or under-estimated (e.g. Lab 11 for As in S1 or Lab 8 for Cr in S1). As a simple rule of thumb, when the uncertainty estimate is 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.

Double counting the precision uncertainty components and overestimation of the laboratory or method bias are the most common errors seen in the laboratories' estimated uncertainty budgets. According to NATA Technical Note 33[14] and to NORDTEST TR 537,[12] the most common experimental data used for estimating the precision component for the measurement uncertainty calculation in the top down approach 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 only, can also be used to estimate the uncertainty of their measurement results.[12, 14] An example of estimating measurement uncertainty using proficiency testing data only is given in Appendix 3.

Laboratories 14 and 17 attached estimates of the expanded measurement uncertainty for 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]

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 0.6 ± 0.06 mg/kg, it is better to report 0.60 ± 0.06 mg/kg or instead of 84 ±16.1 mg/kg, it is better to report 84 ±16 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 46. 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 476 results for which E_n-scores were calculated, 389 (82%) 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

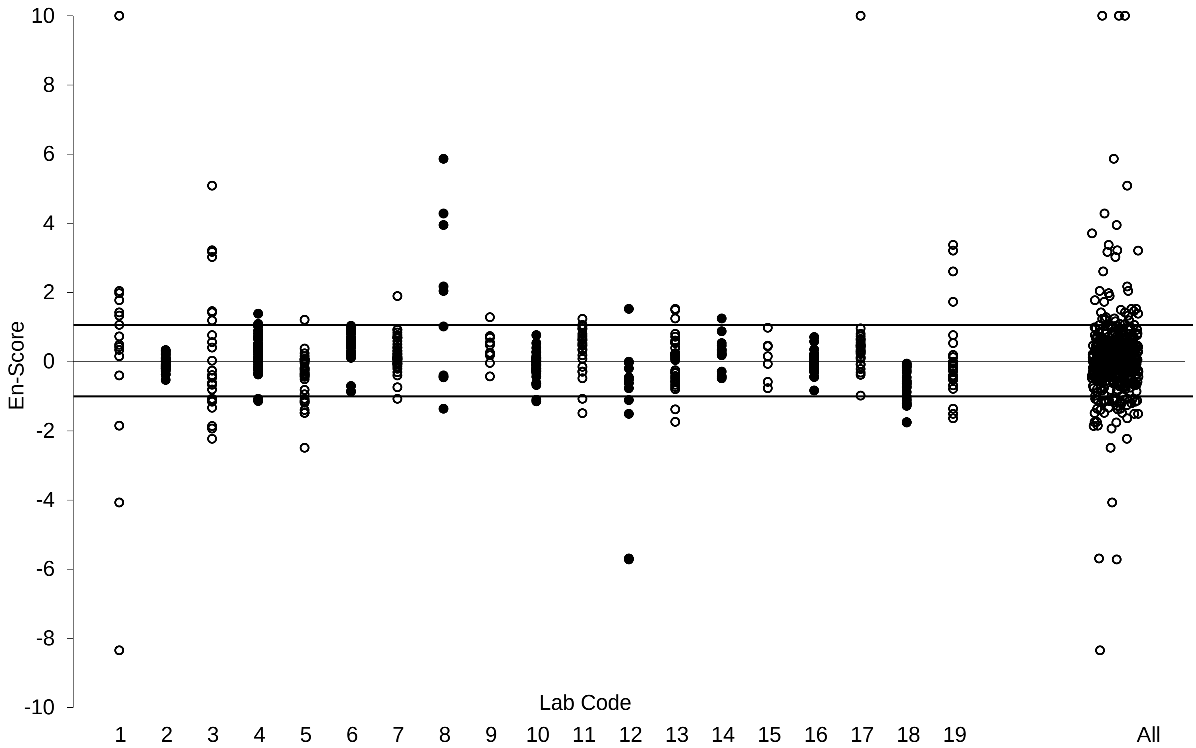
The z-score compares participant's deviation from the assigned value with the target standard deviation set for proficiency assessment.

The target standard deviation defines satisfactory performance in a proficiency test. Target standard deviations equivalent to 10%, 15% and 20% CV 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 a fixed reference value point for assessment of laboratory performance, independent of group performance.

The between laboratory coefficient of variation predicted by the Thompson equation[9] and the between laboratory coefficient of variation resulted in this study are presented for comparison in Table 55. The dispersal of participants' z-scores is presented in Figure 47 (by laboratory code) and in Figure 48 (by test). Of 476 results for which z-scores were calculated, 428 (90%)

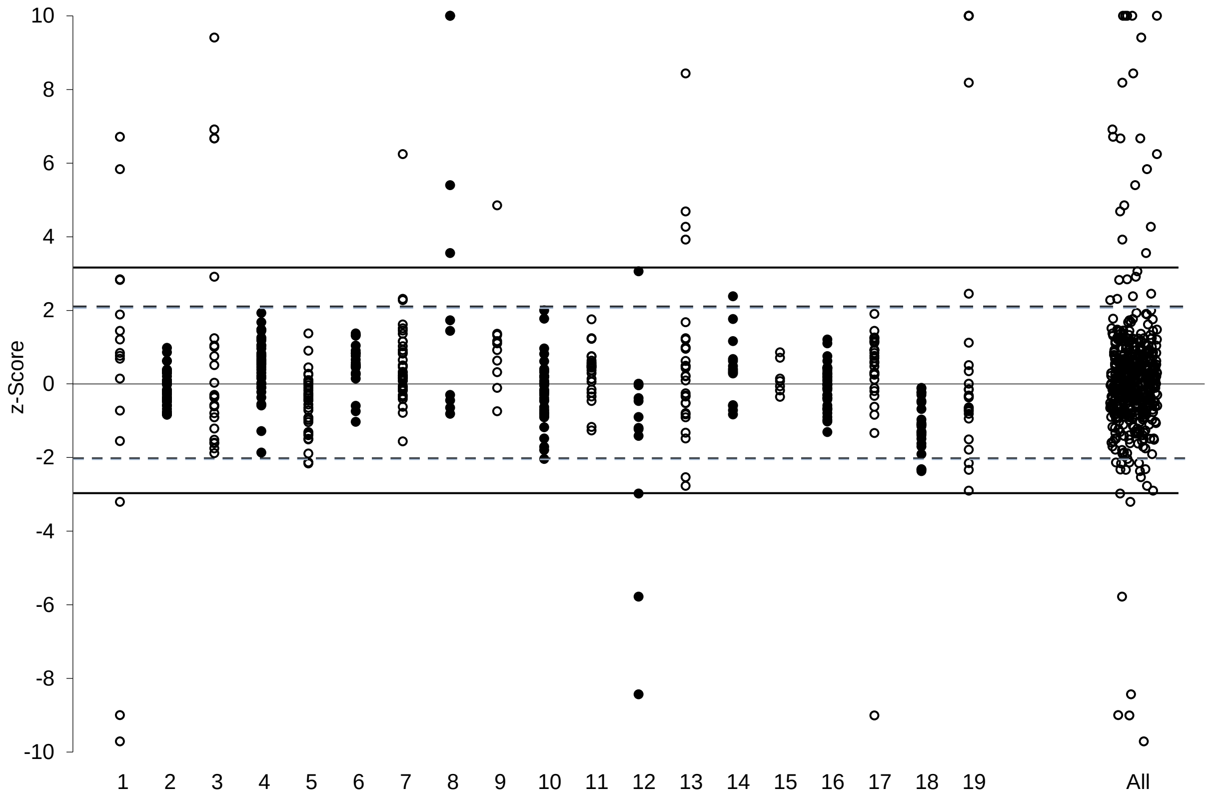
returned a satisfactory score of |z| ≤ 2 and 27 (6%) were questionable of 2 < |z| ≤ 3. Participants with multiple z-scores larger than 2 or smaller than -2 should check for laboratory bias.

Laboratories 2, 4, 6, 11, 15 and 16 returned satisfactory z-scores for all analytes reported.



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

Figure 46 E_n-Score Dispersal by Laboratory



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

Figure 47 z-Score Dispersal by Laboratory

Table 55 Between-Laboratory CV of this Study, Thompson CV and Set Target CV

Sample	Test	Assigned value mg/kg	Between Laboratories CV	Thompson/ Horwitz CV	Target SD (as CV)
S1	As	2.73	12%	14%	10%
S1	Cd	0.431	9.7%	18%	10%
S1	Cr	18.0	16%	10%	15%
S1	Cu	132	9.8%	8%	10%
S1	Hg	0.493	12%	18%	10%
S1	Mn	155	5.9%	7%	10%
S1	Mo	1.34	9.7%	15%	10%
S1	Ni	10.3	15%	11%	15%
S1	Pb	17.4	8.5%	10%	10%
S1	Se	0.825	11%	16%	10%
S1	Sn	13.7	14%	11%	15%
S1	V	5.92	13%	12%	15%
S1	Zn	150	9.3%	8%	10%
S2	Ag	4.77	8.0%	13%	10%
S2	Al	2360	15%	5%	15%
S2	B	6.40	20%	12%	15%
S2	Ba	80.1	7.6%	8%	10%
S2	Bi	0.498	15%	18%	15%
S2	Co	1.97	15%	14%	15%
S2	Cs	Not Set	68%	18%	Not Set
S2	La	1.70	17%	15%	15%
S2	Li	1.11	32%	16%	20%
S2	Rb	2.98	23%	14%	20%
S2	Sr	33.9	16%	9%	15%
S2	Th	0.57	18%	17%	20%
S2	U	0.314	11%	19%	10%
S3	Ba	228	8.8%	7%	10%
S3	Ca	12000	8.3%	4%	10%
S3	Colwell P	Not Set	30%	6%	Not Set
S3	EC (µS/com)	462	5.4%	6%	10%
S3	Exchangeable Ca^{2+} (cmol(+)/kg)	25.6	21%	10%	15%
S3	Exchangeable K^{+} (cmol(+)/kg)	1.86	18%	15%	20%
S3	Exchangeable Mg^{2+} (cmol(+)/kg)	2.55	10%	14%	10%
S3	Exchangeable Na^{+} (cmol(+)/kg)	Not Set	64%	21%	Not Set
S3	Fe	12500	12%	4%	10%
S3	K	1630	13%	5%	10%
S3	Mg	853	5.4%	6%	10%
S3	Na	119	13%	8%	10%
S3	P	2850	8.1%	5%	10%
S3	pH	6.40	3.4%	12%	10%
S3	S	659	9.4%	6%	10%
S3	TC	Not Set	21%	3%	Not Set
S3	TN	5810	4.3%	4%	10%
S3	TOC	75300	2.8%	3%	10%

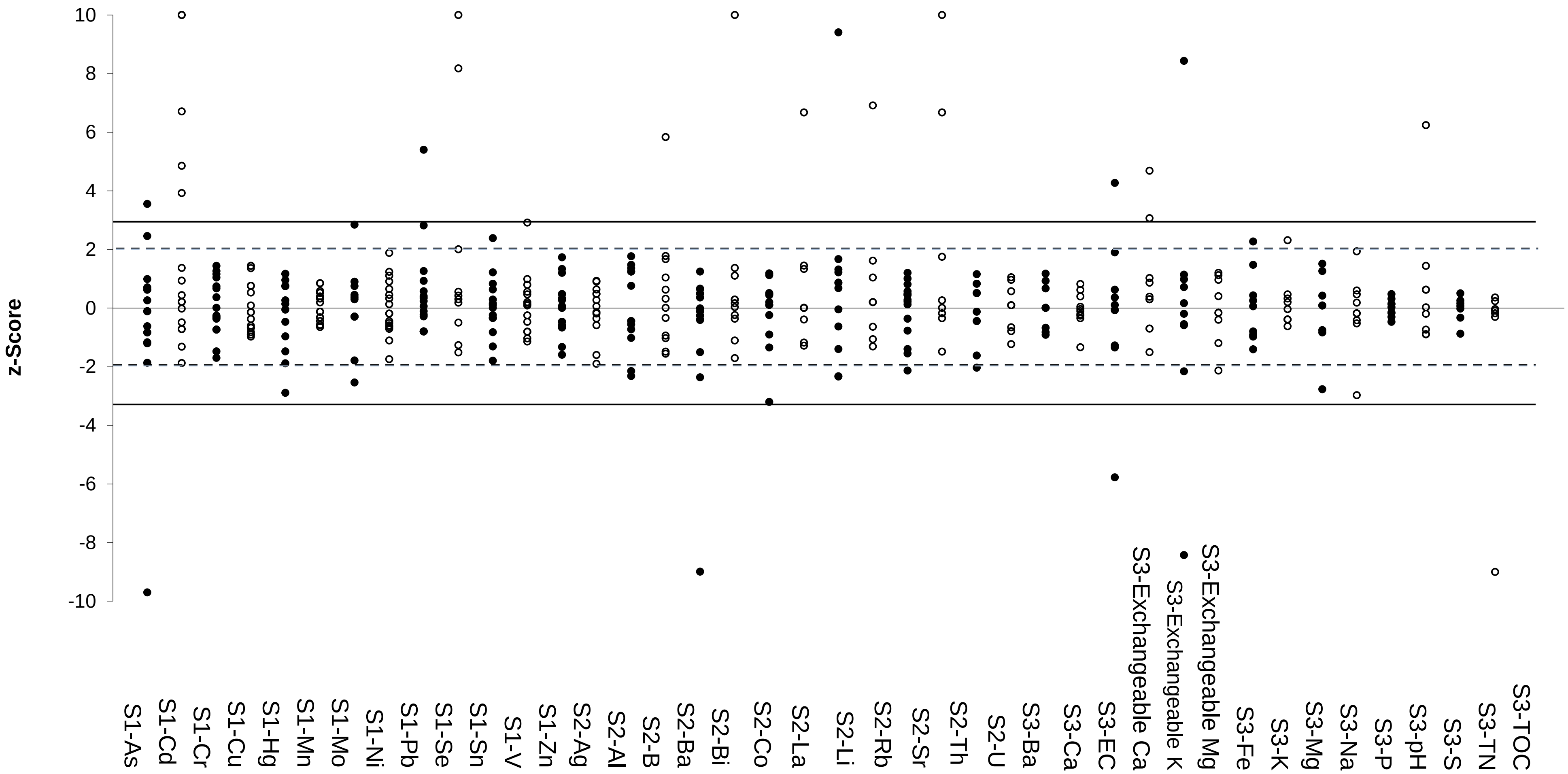


Figure 48 z-Score Dispersal by Test

Table 56 Summary of Participants' Results and Performance for Sample S1

Lab Code	S1-As mg/kg	S1-Cd mg/kg	S1-Cr mg/kg	S1-Cu mg/kg	S1-Hg mg/kg	S1-Mn mg/kg	S1-Mo mg/kg	S1-Ni mg/kg	S1-Pb mg/kg	S1-Se mg/kg	S1-Sn mg/kg	S1-V mg/kg	S1-Zn mg/kg
H.V.	2.40	0.416	19.3	130	0.445	148	1.28	10.9	17.3	0.786	10.8	6.15	157
A.V.	2.73	0.431	18.0	132	0.493	155	1.34	10.3	17.4	0.825	13.7	5.92	150
1	0.08	0.72	21.9	142	NT	168	1.72	13.2	22.3	<1	NT	6.04	168
2	2.5	0.41	17.3	123	0.47	146	1.3	10	16	0.85	12	5.7	141
3	2.4	0.35	17	124	0.53	146	1.3	7.6	16	0.7	13	8.5	126
4	2.22	0.44	20.8	139	0.53	161	1.44	12.2	17.2	0.87	13.7	6.62	150
5	2.7	0.4	17.1	119	0.4	145	1.4	9.6	16.9	<1	13.9	5	154
6	2.80	0.49	16.0	142	0.50	163	1.46	9.38	18.4	NT	15.4	6.33	157
7	2.56	0.44	21.1	133	0.49	153	1.38	11.3	17.8	0.84	14.0	5.99	155
8	3.7	2.2	17.2	151	NT	148	NT	9.3	26.8	3.0	NT	5.2	176
9	2.7	0.64	16	150	0.55	160	<5	12	19	<1	15	NT	170
10	2.5	0.44	18	120	0.42	160	1.3	10	16	0.99	10	5.5	150
11	2.41	0.45	20.0	130	0.53	164	1.39	10.5	17.5	0.72	14.3	6.41	143
12	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
13	3	0.6	14	120	0.54	150	1	9.5	16	<3	13	6.8	130
14	2.9	0.40	19.8	121	0.55	146	< 5	10.8	18.1	< 2	18.6	< 10	141
15	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
16	2.9	0.43	19	127	0.53	158	1.3	11	16	0.86	11	6.1	151
17	2.92	0.471	21.4	151	0.506	168	<5	11.7	19.6	<2	16.2	<10	157
18	2.70	0.374	13.4	123	0.445	153	1.30	8.58	17.0	0.784	13.2	4.90	143
19	3.4	2.2	16	130	0.35	150	1.1	9.2	18	1.5	13	5.2	140

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

Table 57 Summary of Participants' Results and Performance for Sample S2

Lab Code	S2-Ag mg/kg	S2-Al mg/kg	S2-B mg/kg	S2-Ba mg/kg	S2-Bi mg/kg	S2-Co mg/kg	S2-Cs mg/kg	S2-La mg/kg	S2-Li mg/kg	S2-Rb mg/kg	S2-Sr mg/kg	S2-Th mg/kg	S2-U mg/kg
H.V.	4.82	2820	4.94	89.0	0.554	2.07	0.485	Not Set	1.33	4.64	43.7	0.68	0.340
A.V.	4.77	2360	6.40	80.1	0.498	1.97	Not Set	1.70	1.11	2.98	33.9	0.57	0.314
1	NT	2100	12	8	NT	1.02	NT	NT	1.26	NT	26.0	NT	NT
2	4.6	2200	6.4	77	0.52	2	<1	1.7	1.3	3.1	30	0.55	0.3
3	4.0	NR	7.4	90	0.50	1.7	0.74	3.4	3.2	7.1	39	1.33	0.33
4	4.49	2879	7.00	85.2	0.47	2.32	NT	NT	1.48	NT	36.8	NT	0.34
5	5.2	2160	<50	78	0.6	2.0	0.2	1.6	0.8	2.2	23	0.4	0.3
6	NT	NT	5.41	85.4	0.52	2.12	NT	2.04	1.40	3.60	36.2	NT	0.34
7	4.68	2840	4.90	76.8	0.51	2.03	0.43	2.07	1.30	3.94	36.4	0.53	0.35
8	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
9	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
10	4.8	2200	8.1	78	0.37	1.9	<1	1.4	0.97	3.1	38	0.57	0.25
11	5.07	2800	6.07	84.0	0.48	2.11	NT	NT	1.38	NT	36.2	0.77	0.33
12	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
13	5	2800	8	84	NT	<0.5	NT	NT	NT	NT	35	NT	NT
14	5.0	2984	<10	83	< 10	< 5	<10	<10	< 5	<10	35.3	NT	< 1
15	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
16	4.9	2000	6.7	80	0.58	2.1	NT	1.7	1.1	NT	40	0.60	0.31
17	5.21	2630	<10	79.1	<10	<5	NT	NT	<5	NT	34.5	NT	<10
18	3.86	1540	4.97	61.1	0.415	1.57	0.256	1.37	0.593	2.34	26.8	NT	0.263
19	4.7	1600	5.5	68	3.1	2.3	NT	1.7	0.59	2.6	32	1.8	0.33

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

Table 58 Summary of Participants' Results and Performance for Sample S3

Lab Code	S3-Ba mg/kg	S3-Ca mg/kg	S3-Colwell P mg/kg	S3-EC (µS/com)	Exchangeable Ca^{2+} (cmol(+)/kg)	Exchangeable K^{+} (cmol(+)/kg)	Exchangeable Mg^{2+} (cmol(+)/kg)	Exchangeable Na^{+} (cmol(+)/kg)	S3-Fe mg/kg
H.V.	233	11700	710	400	21.7	2.20	2.41	0.136	13000
A.V.	228	12000	Not Set	462	25.6	1.86	2.55	Not Set	12500
1	NR	NR	NR	NR	NR	NR	NR	NR	NR
2	230	12000	NT	480	28	2.0	2.8	0.3	12000
3	NR	NR	NR	NR	NR	NR	NR	NR	NR
4	252	12800	751	452	20.7	1.97	2.41	0.092	13700
5	230	10900	NT	464	20.4	1.3	2.0	<0.1	9830
6	NR	NR	NR	NR	NR	NR	NR	NR	NR
7	210	13100	737	500	25.3	2.24	2.59	0.13	12300
8	NR	NR	NR	NR	NR	NR	NR	NR	NR
9	NR	NR	NR	NR	NR	NR	NR	NR	NR
10	250	12000	NT	490	27	1.6	2.5	0.1	13000
11	NR	NR	NR	NR	NR	NR	NR	NR	NR
12	200	12000	NT	460	3.4	3.0	0.4	28	11000
13	230	11000	430	450	42	3.6	4.7	0.12	14000
14	NR	NR	NR	NR	NR	NR	NR	NR	NR
15	NR	NR	510	446	25.34	2.18	2.73	0.1	NR
16	213	11180	NT	457	26	1.6	2.4	0.2	12300
17	241	13400	NT	400	32.9	1.97	2.84	<0.5	13900
18	NR	NR	NR	NR	NR	NR	NR	NR	NR
19	NR	NR	NR	NR	NR	NR	NR	NR	NR

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

Table 58 Summary of Participants' Results and Performance for Sample S3 (Continued)

Lab Code	S3-K mg/kg	S3-Mg mg/kg	S3-Na mg/kg	S3-P mg/kg	S3-pH	S3-S mg/kg	S3-TC mg/kg	S3-TN mg/kg	S3-TOC mg/kg
H.V.	1500	993	133	2800	6.10	780	80000	6300	79000
A.V.	1630	853	119	2850	6.40	659	Not Set	5810	75300
1	NR	NR	NR	NR	NR	NR	NR	NR	NR
2	1640	880	110	2800	6.3	700	NT	5800	78000
3	NR	NR	NR	NR	NR	NR	NR	NR	NR
4	1870	892	134	3400	6.41	754	76200	5900	74600
5	1480	850	120	2800	NT	660	NT	5956	NT
6	NR	NR	NR	NR	NR	NR	NR	NR	NR
7	2000	1050	137	2980	6.2	1070	79000	6100	77000
8	NR	NR	NR	NR	NR	NR	NR	NR	NR
9	NR	NR	NR	NR	NR	NR	NR	NR	NR
10	1500	820	120	2900	6.3	610	85000	5300	75000
11	NR	NR	NR	NR	NR	NR	NR	NR	NR
12	1400	820	NT	2000	6.1	600	NT	NT	NT
13	1700	870	86	2700	6.7	700	NT	<0.01	NT
14	NR	NR	NR	NR	NR	NR	NR	NR	NR
15	NR	NR	NR	NR	6.49	NR	73992	5850	73919
16	1470	820	124	2730	6.49	600	NT	NT	73000
17	1670	800	109	3020	6.6	646	7470	5621	7470
18	NR	NR	NR	NR	NR	NR	NR	NR	NR
19	NR	NR	NR	NR	NR	NR	NR	NR	NR

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

7.5 Participants' Results and Analytical Methods for Acid Extractable Elements

A summary of participants' results and performance is presented in Tables 56 to 58 and in Figures 46 and 47.

Cd was the element with the most unsatisfactory z-scores.

Manganese was the element that presented the least analytical difficulty to participating laboratories (the between laboratory CV was 5.9%).

All the unsatisfactory results reported by Laboratory 8 were greater than the assigned value. This is an indication of method or laboratory bias.

The method descriptions provided by participants are presented in Tables 1 and 2 while the instrumental conditions are presented in Appendix 5.

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, 19-21] 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]

Participating laboratories used a wide variety of extraction techniques and most produced comparable results for all analytes.

One laboratory used an extraction regime which involved dilute acids and a digestion temperature of 170 ºC, which was much higher than other laboratories; all the unsatisfactory results reported by them were higher than the assigned value.

Two laboratories 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 47).

One laboratory reported using a staggered digestion: the sample was first digested in 2 mL 50% HNO_3 and 10 mL 20% HCl for 30 minutes and then in 2 mL 30% H_2O_2 for another 30 minutes.

Individual Element Commentary

Aluminium is an element strongly dependent on the digestion regime. The between laboratory coefficient of variation for Al in Sample S2 was high (15%), higher than predicted by Thomson (5%).[9] Short extraction time (30 min) may explain the low results reported by Laboratories 18 and 19.

Plots of Al results versus instrumental technique used are presented in Figure 49.

Arsenic All unsatisfactory As results were from ICP-OES measurements as shown in Figure 50.

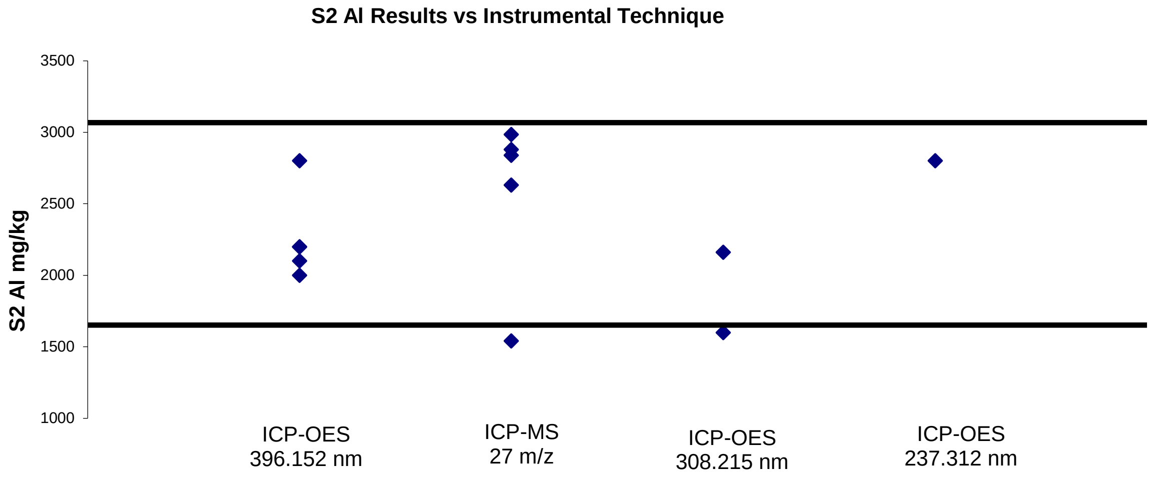


Figure 49 Al Results vs. Instrumental Technique

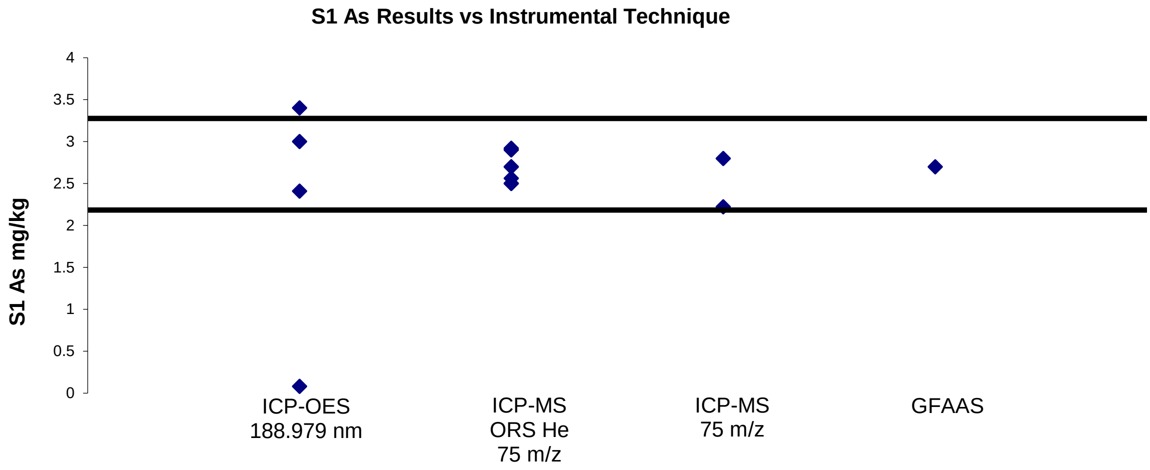


Figure 50 As Results vs. Instrumental Technique

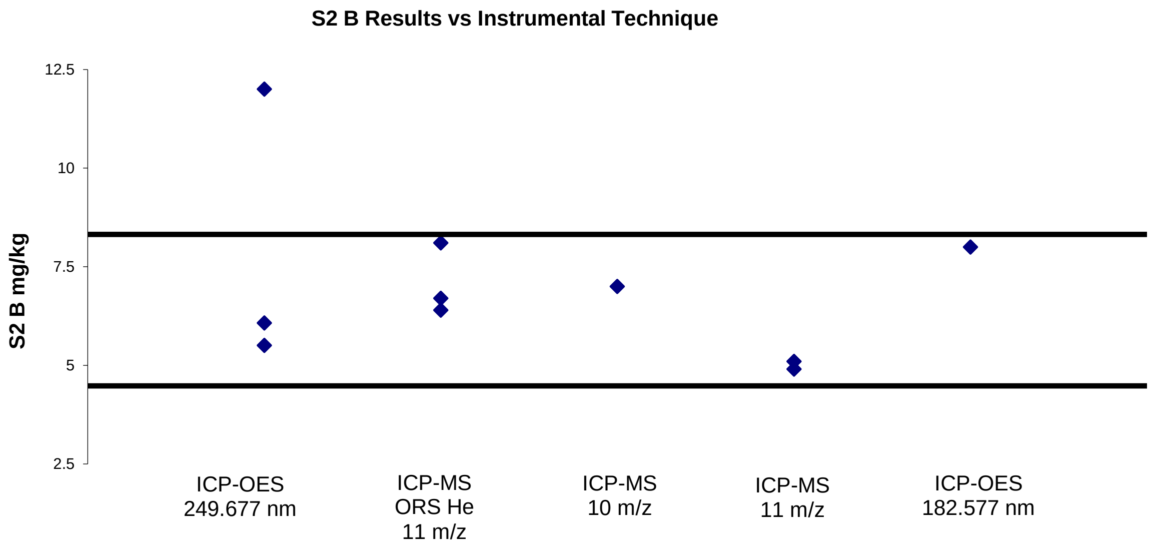


Figure 51 B Results vs. Instrumental Technique

Boron level in sample S3 was low and this might have presented difficulties to some laboratories; the between laboratory coefficient of variation was high (20%). Boron is an element prone to contamination. The sampling system should be cleaned before low level B determination.

Boron measured at 249.7 nm can have significant interferences from Fe 249.771 nm if on-line inter-element correction is not used. Plots of participants' results versus instrumental technique used are presented in Figure 51.

Caesium No assigned value was set for Cs in S2 as only 4 participants reported results for this element. The four results reported were not in agreement with each other; The low Cs results were from the digestion regime which involved dilute acid or short extraction time while the high Cs result was from a laboratory that used dilute acids but high digestion temperature (170°C).

Cadmium Measurement of low level Cd presented difficulty to participating laboratories, of 17 results reported for Cd, only 12 returned satisfactory z-scores. A significant relationship between the Cd results and the instrumental technique used was found, with low results coming from ICP-MS technique and high results coming from ICP-OES or AAS technique. The distribution graph of participants' results and instrumental techniques is presented in Figures 52.

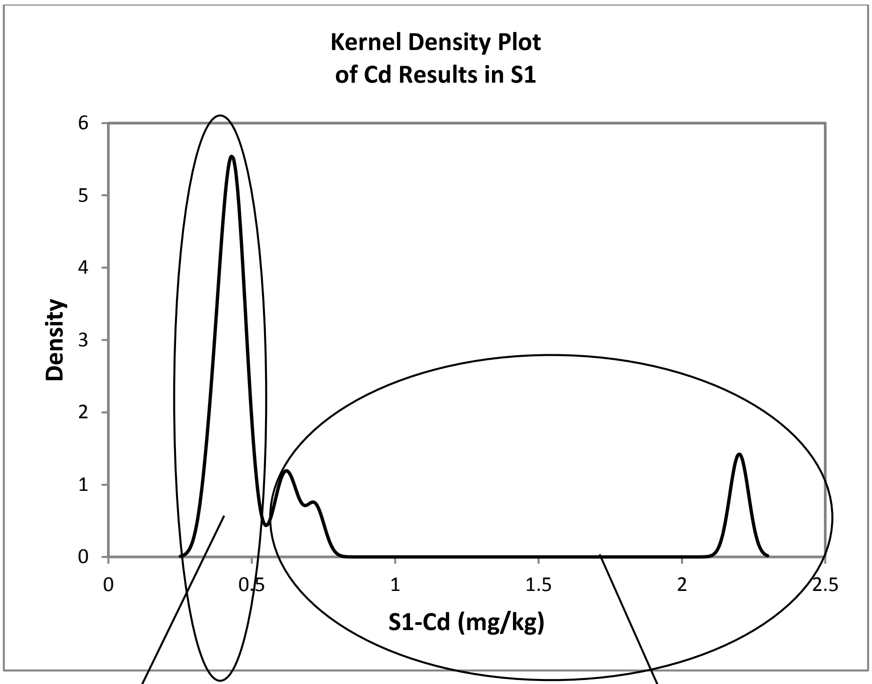


Figure 52 Kernel Density Plot of Participants' Results for Cd in Sample S1

Lab code	Results mg/kg	Instrumental Techniques
18	0.374	ICP-MS/ 114 m/z
2, 14, 5	0.41-0.4	ICP-MS/ 111 m/z
16	0.43	ICP-MS/ 111 m/z
10, 7, 4	0.44	ICP-MS/ 111 m/z
11	0.45	ICP-OES 226.502 nm
17	0.471	ICP-MS/ 111 m/z
6	0.49	ICP-MS

Lab code	Results mg/kg	Instrumental Techniques
13	0.6	ICP-OES/214.439 nm
9	0.64	AAS
1	0.72	ICP-OES / 214.44 nm
8	2.2	ICP-AES

Cd 228.802 nm and Cd 214.439 nm can have significant spectral interferences from Fe 228.804 nm and Fe 214.445 respectively. If ICP-OES is used then online inter-element correction may be required for measurements of low level Cd in soil samples, which usually is rich in Fe.

Chromium and Nickel are two elements strongly dependent on the extraction regime. The between laboratories coefficient of variation for these elements was higher than predicted by Thomson value. Weak extraction conditions (small amounts of acids or dilute HNO_3 and dilute HCl) might explain the low results.

Plots of participants' results versus instrumental technique used are presented in Figures 53 and 54.

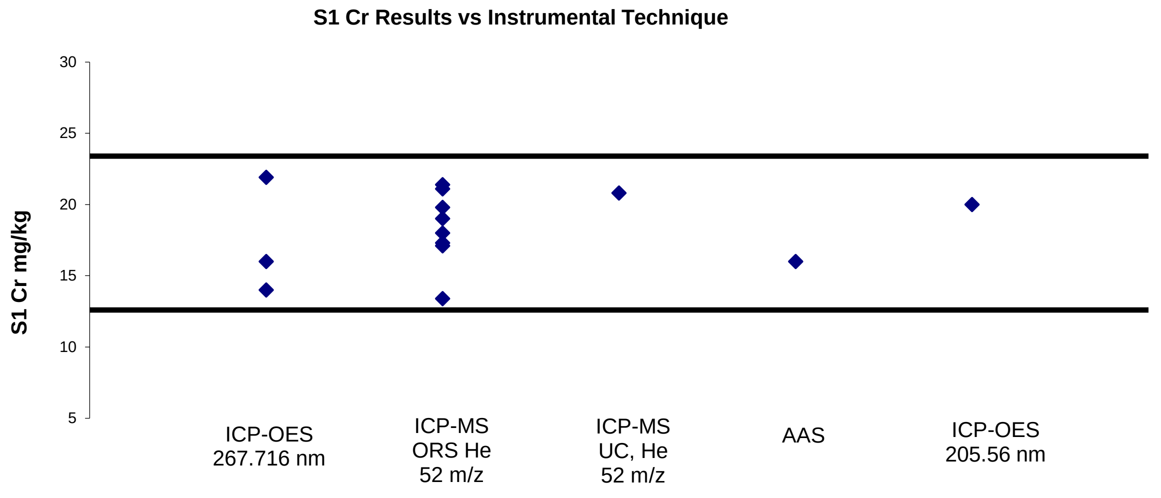


Figure 53 Cr Results vs. Instrumental Technique

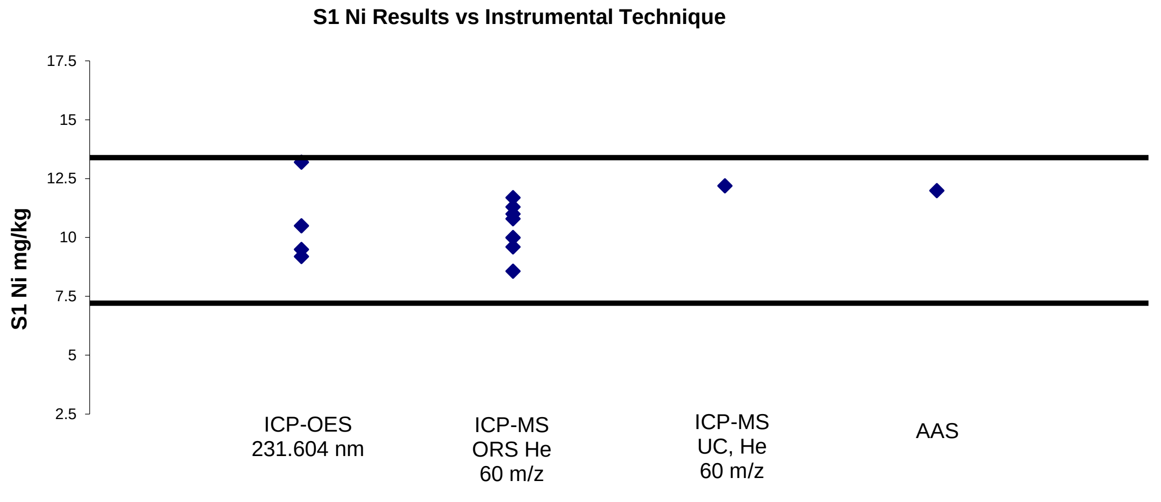


Figure 54 Ni Results vs. Instrumental Technique

Lead Caution should be exercised when low level Pb in soil is measured by ICP-OES. Soil usually has a high Fe content and line 220.353 nm has direct overlap interference on Pb line 220.353 nm.

Lithium The results reported for Li were variable (between laboratories CV was 20%). Short digestion time may explain the low, unsatisfactory results reported for Li in S2.

Lanthanum, Rubidium and Thorium A small number of participants reported results for La, Rb and Th in S2. Participants used various digestion regimes for these elements and this might explain the big variation between the reported results (the between laboratories CV was from 15% to 23%).

All participants reported using ICP-MS for La, Rb and Th measurements in S2.

Mercury level in S1 was low, 0.445 mg/kg. 15 participants reported results for Hg in S1 and 14 performed satisfactorily. Plots of participants' results versus instrumental technique used are presented in Figure 55.

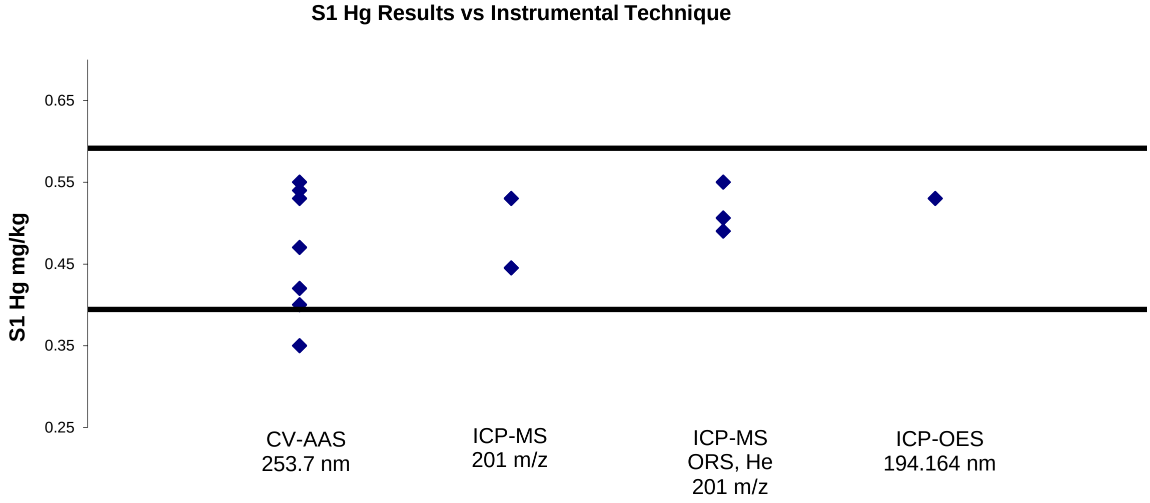


Figure 55 Hg Results vs. Instrumental Technique

Potassium, Magnesium, and Sodium Plots of K, Mg and Na results versus instrumental technique used are presented in Figures 56 to 58. ICP-OES-AV with buffer or correction equation was the preferred measurement technique for these elements.

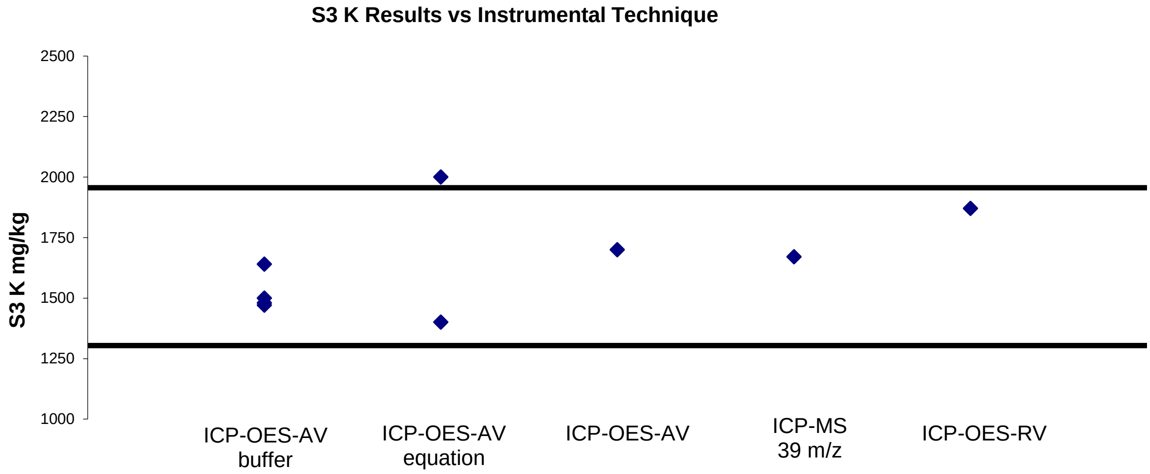


Figure 56 K Results vs. Instrumental Technique

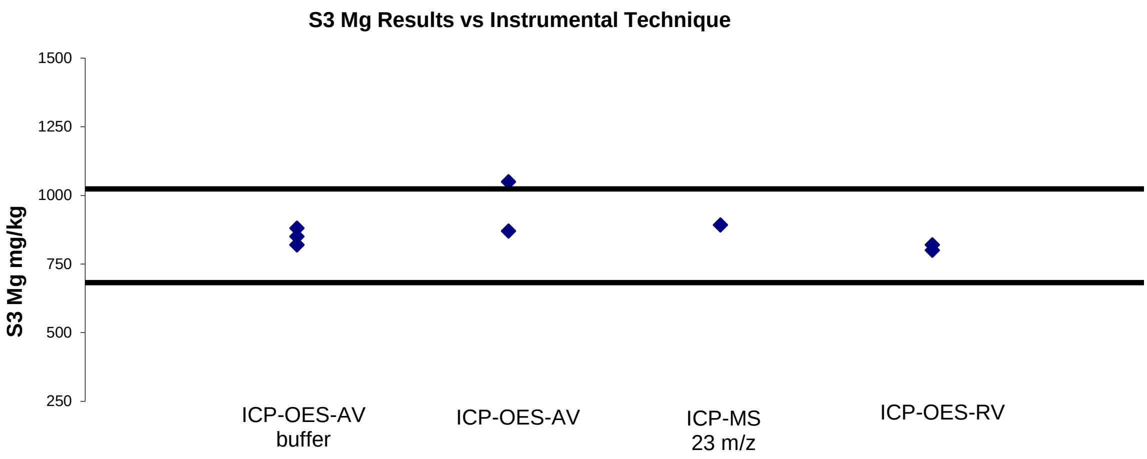


Figure 57 Mg Results vs. Instrumental Technique

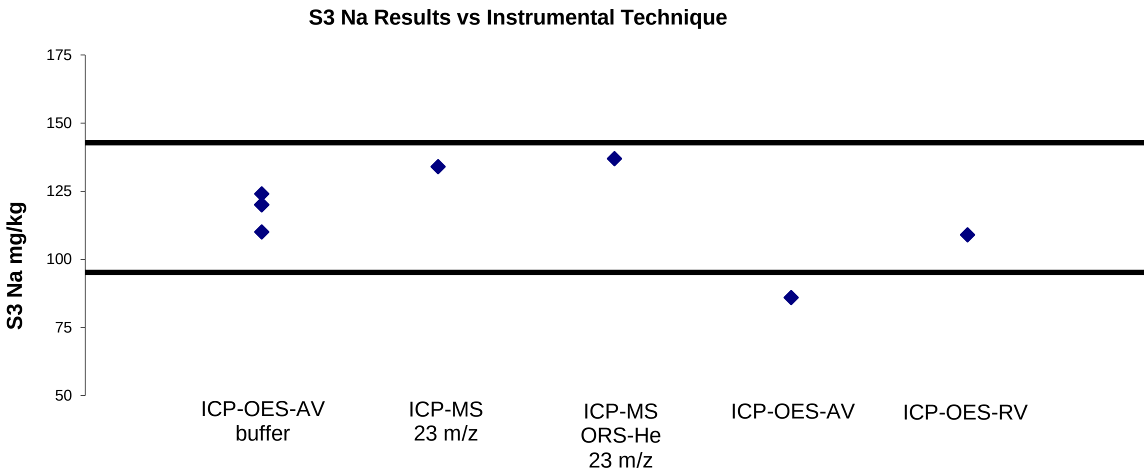
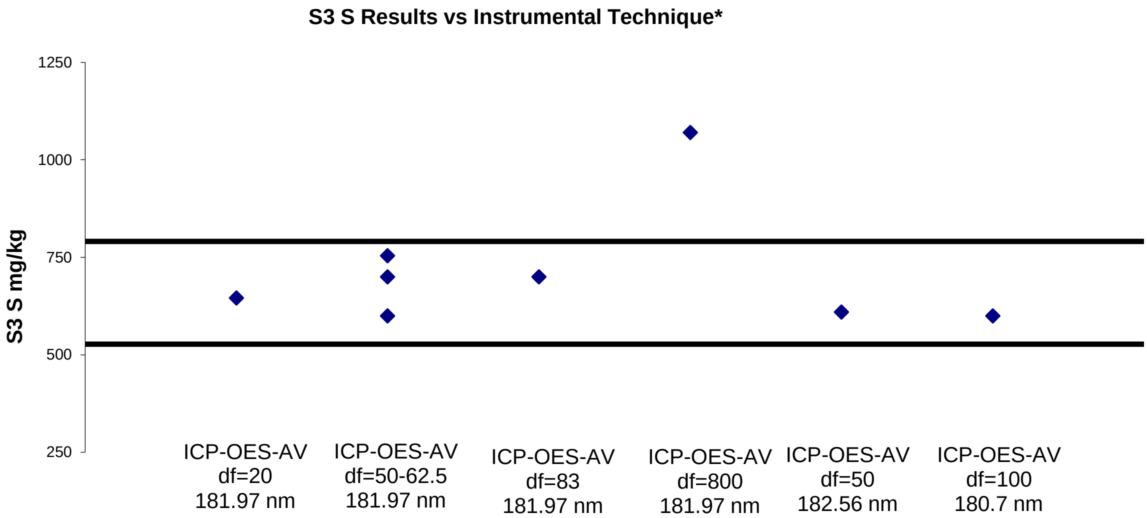


Figure 58 Na Results vs. Instrumental Technique



*df =dilution factor

Figure 59 S Results vs. Instrumental Technique

Sulphur Participants used ICP-OES with various wavelengths and dilution factors for S measurements (Figure 59).

Selenium Participants' results versus the instrumental techniques used for Se measurement in S1 are presented in Figure 60. ICP-OES may not be the right technique for measurements of Se in soil at parts per billion level.

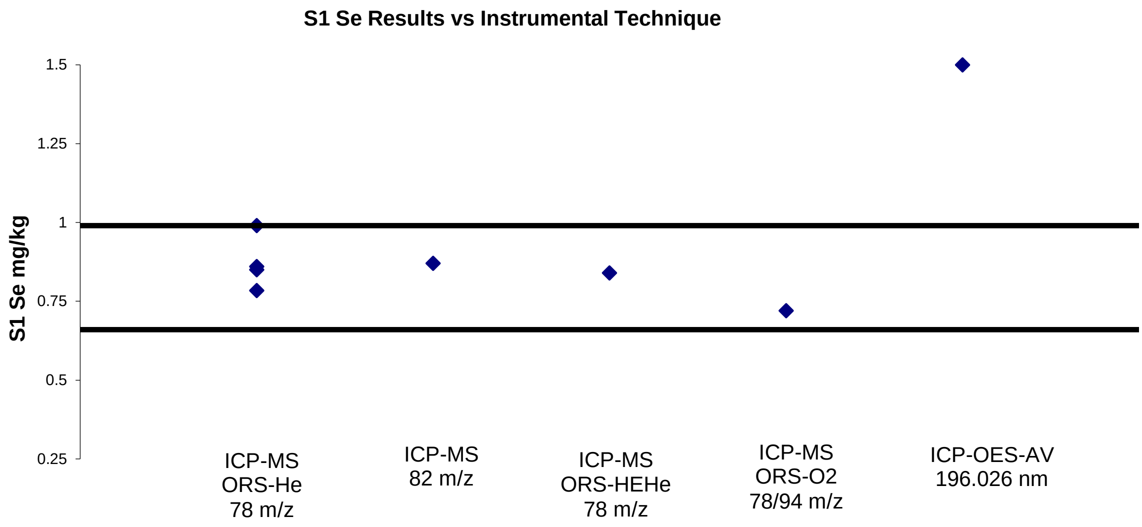


Figure 60 Se z-Scores vs. Instrumental Technique

7.6 Participants' Results and Analytical Methods for Exchangeable Cations

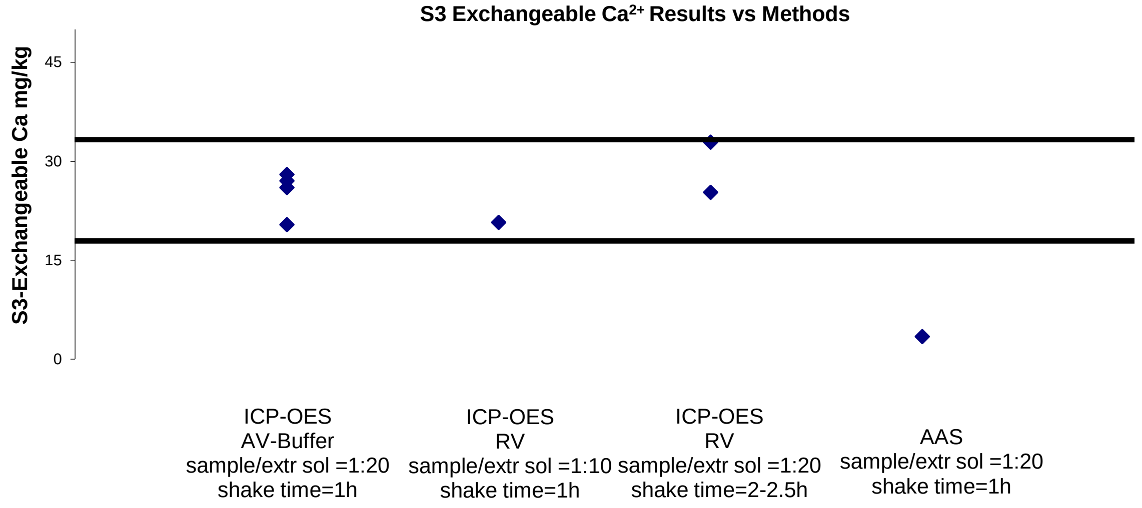
Measurement of exchangeable bases in soil is an empirical measurement – where the method of extraction defines the measurand. The participating laboratories were asked to analyse the sample using their normal measurement technique but to use the same preparation procedure the Method 15A1 as defined by Rayment, G.E. and David, J. L in "Soil Chemical Methods-Australia". [25]

The method descriptions provided by participants are presented in Table 7 and the instrumental techniques are presented in Appendix 5.

No assigned value could be set for Exchangeable Na^+ in S3 because the reported results were too variable (between laboratories CV was 68%).

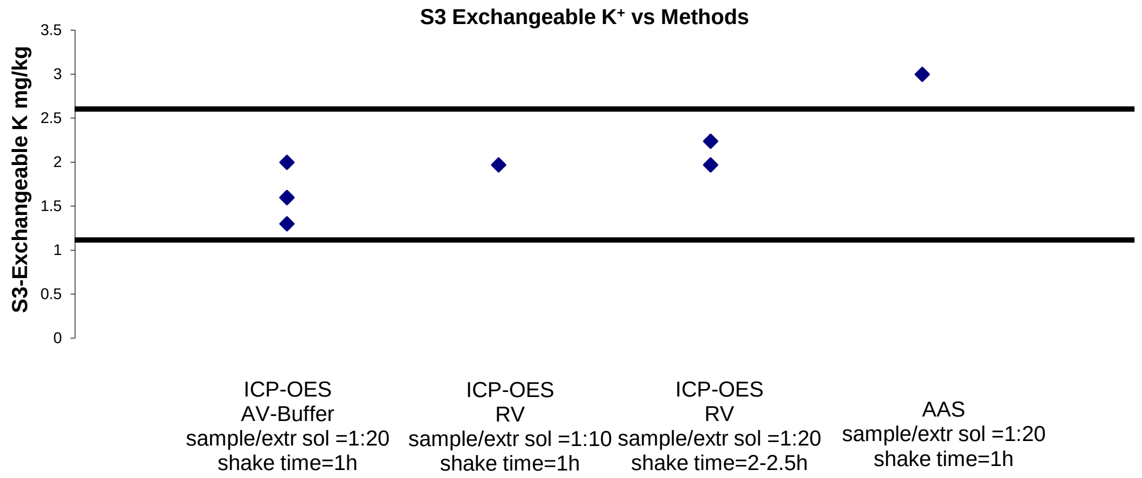
A systematic error was observed for Laboratory 13. All the results reported by them for the exchangeable bases in S3 were higher than the assigned value by approximatively the same factor (2). This suggests a problem with the dilution and/or calculation procedure. These results were not included in the analyses of the extraction methods and instrumental techniques employed by participants.

Plots of participants' results versus the analytical methods used for the exchangeable bases measurement are presented in Figures 61 to 64.



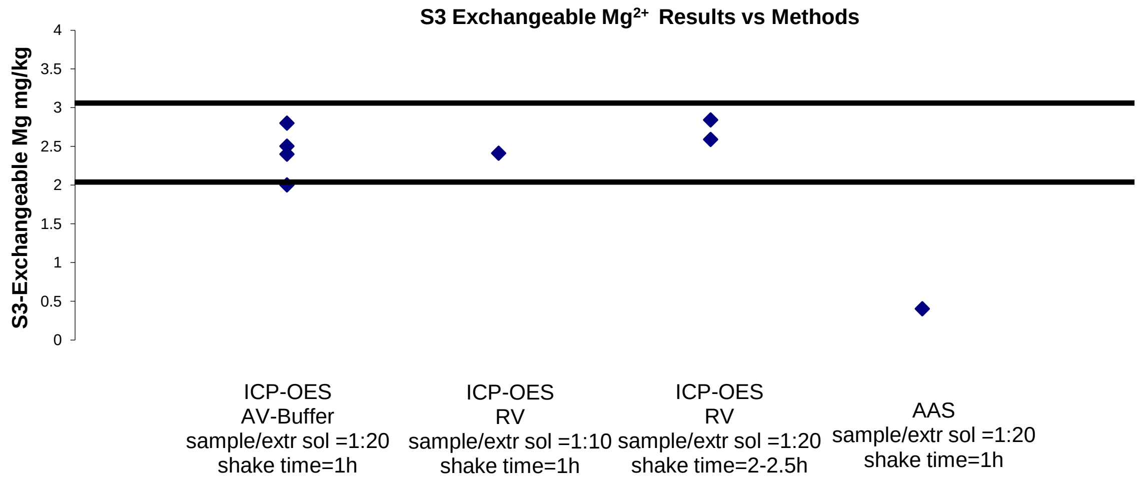
Horizontal lines on chart are the results corresponding to z-scores of 2 and -2; extr. sol. = extraction solution

Figure 61 Exchangeable Ca^{2+} Results vs. Analytical Methods



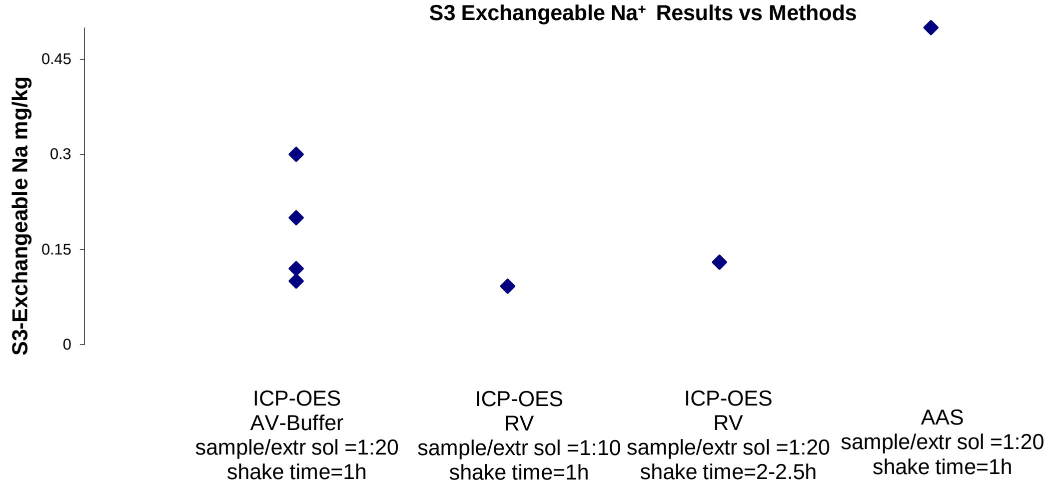
Horizontal lines on chart are the results corresponding to z-scores of 2 and -2; extr. sol. = extraction solution

Figure 62 Exchangeable K^{+} Results vs. Analytical Methods



Horizontal lines on chart are the results corresponding to z-scores of 2 and -2; extr. sol. = extraction solution

Figure 63 Exchangeable Mg^{2+} Results vs. Analytical Methods



Result > 0.5 mg/kg has been plotted as 0.5 mg/kg. sol. = extraction solution

Figure 64 Exchangeable Na^+ Results vs. Analytical Methods

7.7 Participants' Results and Analytical Methods for Colwell P

Colwell P Only 4 results were report for Colwell P in S3 and all were in agreement with each other, centred on 607 mg/kg. All participants shook the sample for 16 hours and used a ratio of sample mass/extraction solution of 1 to 100 (Table 5). Plots of participants' results versus the instrumental technique used are presented in Figure 65.

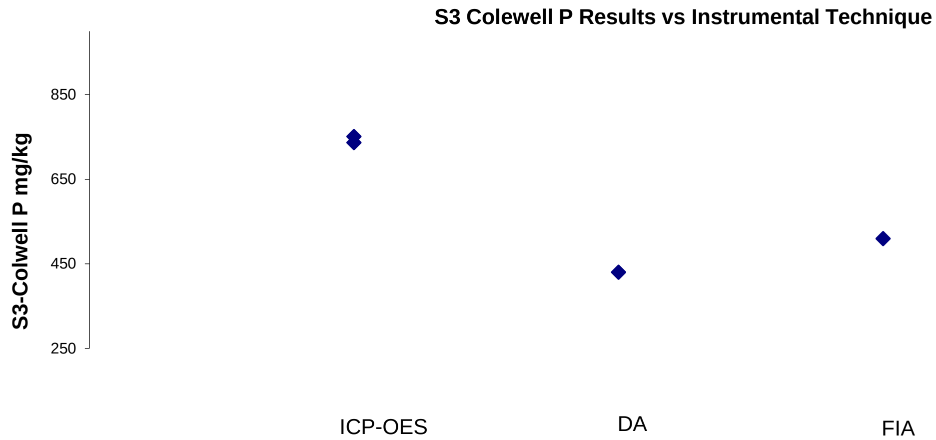
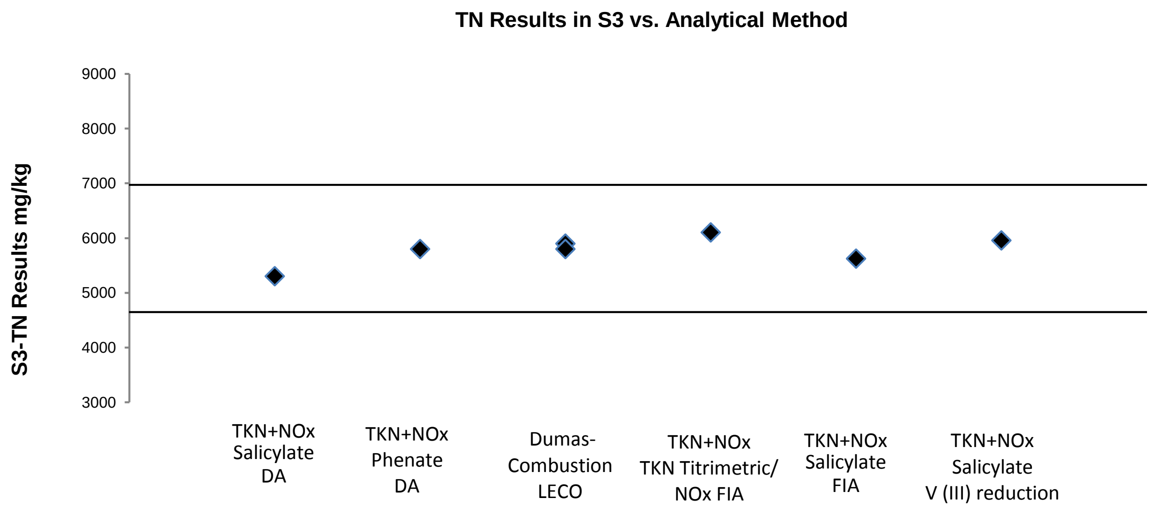


Figure 65 Colwell P Results vs. Instrumental Technique

7.8 Participants' Results and Analytical Methods for Total Nitrogen

All results reported for TN returned satisfactory z-scores. No significant difference was found between the TN results from combustion and TN results calculated from TKN and NOx. The method descriptions provided by participants are presented in Table 6. A plot of participants' results versus analytical method and measurement technique used for TN analysis in S3 is presented in Figure 66.



Horizontal lines on chart are the results corresponding to z-scores of 2 and -2

Figure 66 TN Results vs. Analytical Method

7.9 Participants' Results and Analytical Methods for Total Carbon and Total Organic Carbon

Participants were free to choose an appropriate method and were given no guidance apart from the instruction to: "Quantitatively analyse the samples using your normal test method." The method descriptions provided by participants for TC and TOC analyses are presented in Tables 3 and 4.

Total Carbon No assigned value could be set for TC in S3 because only five participants reported results for this test. Except for one all reported results were in good agreement with each other, centred on 79000 mg/kg.

Total Organic Carbon assigned value was 75300 mg/kg.

Total organic carbon (TOC) measurements should involve measurement of both volatile organic carbon (VOC) and of non–purgeable organic carbon (NPOC). As the loss of VOC is considered negligible when compared to the content of NPOC in soil sample, all the NPOC reported results in sample S3 have been considered as TOC.[27, 28]

Five participants used a high temperature oxidation method and one a chemical oxidation method based on the "Walkley-Black" method. No significant difference was noticed between the results coming from the two methods.

The chemical oxidation method based on the "Walkley-Black" method is easy and cheap. Its major limitation is that only the most active OC is oxidised. The quantities of OC recovered depend on the soil type and nature of the organic material therein. [27, 28]

The high temperature oxidation method for organic carbon determination can be rapid and reliable when inorganic carbon is removed prior to combustion. The separation of organic carbon from inorganic carbon can be achieved by ashing or acid treatment. When ashing is used, good knowledge of the nature of soil is required to choose the right ashing temperature. The major problem when acid treatment is used is uncertainty about the completeness of inorganic carbon removal. Introduction of a pretesting step to establish the right amount of sample to be taken for analyses and the right type and concentration of acid to be used for inorganic carbon removal can help avoid these problems.[28, 29]

7.10 Comparison with Previous NMI Proficiency Tests of Metals in Soil

AQA 18-02 is the twenty-second NMI proficiency test of metals in soil.

Participants' performance in measurement of metals in soil over time is presented in Figure 67. Despite different matrices, analytes and analytes' concentrations, on average participants' performance has remained consistent over the last 10 years, with the percentage of satisfactory z-scores ranging from 83% to 97% and satisfactory E_n scores from 72% to 89%.

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 59).

Table 59 Control Samples Used by Participants

Lab. Code	Description of Control Samples
1	CRM
2	Spiked Sample
3	Certified Reference Material
4	Certified Reference Material/Reference Material – AGAL 12, In house AG 1.20
5	Reference Material - Several internal reference materials
6	Reference Material
7	agal10, agal12
8	AQA17-11
9	Certified Reference Material/Reference Material – Metals in soil/clay/sediment – various samples
10	Reference Material
11	Reference Material – AQA 17-11 soil samples
12	Certified Reference Material
13	Certified reference Material
14	Certified Reference Material – AGAL 10 and AGAL 12
15	Certified Reference Material – ASPAC 3192 and ASPAC 3202 and ASPAC 7098-C1
16	Spiked Sample
17	Certified Reference Material – AGAL-10

Matrix matched control samples taken through all steps of the analytical process, are the most valuable quality control tools for assessing a 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'*[30]

Surplus test samples from this study are available from NMI.

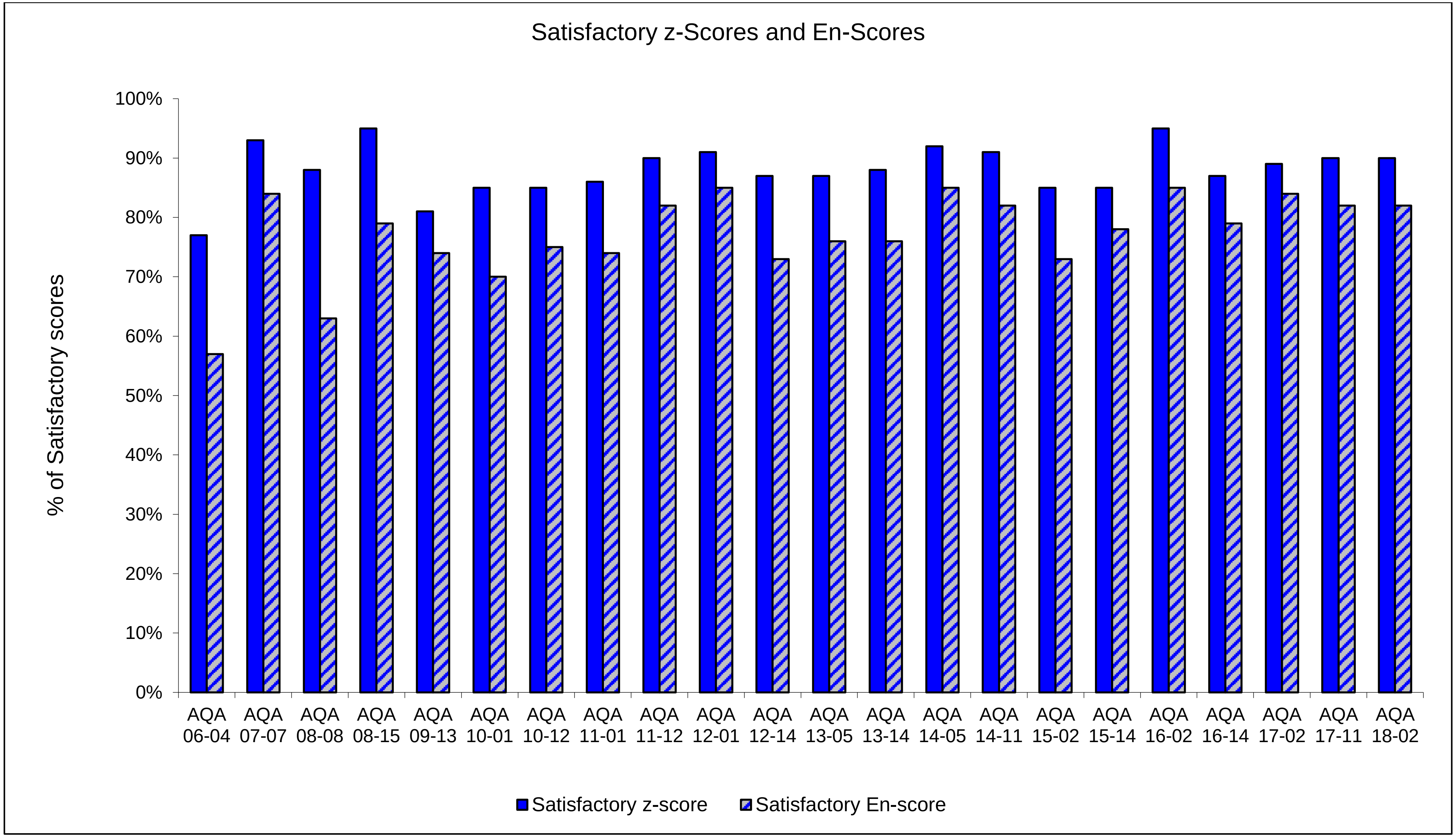


Figure 67 Participants' Performance over Time (2006-2018)

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APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING

Sample Preparation

Samples S1 and S2 were dried compost samples (soil, leaves, grass and roots) prepared from the same material, a composite of compost samples submitted to NMI for metal analysis. The compost material was blended, dried, ground and passed through a 212 µm sieve, mixed and divided into portions of 25 g each. The analytes' level in these samples was the incurred level.

Sample S3 was an unfortified, dried, agricultural soil material. It was ground and sieved through 212 µm sieve, further mixed and divided in portions of approximately 50 g each.

Sample Analysis and Homogeneity Testing

A partial homogeneity test was conducted for all tests except La. Three bottles were analysed in duplicate and the average of the results was reported as the homogeneity value.

Sample Analysis for Acid Extractable Elements

Measurements for acid extractable elements were made using NMI method: NT2.49.[31] NMI holds third party (NATA) accreditation for this method. Testing using NMI Method NT2.49 involve solubilisation of metals and metal complexes using a mixture of nitric acid and hydrochloric acid. Metals were then measured using ICP-MS and ICP-OES.

A test portion of approximately 1 g for the soil (compost) samples and 0.8 g for the agricultural soil sample was weighed into a 50 mL graduated polypropylene centrifuge tube. The samples were 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 for ICP-MS or ICP-OES determination.

The measurement instrument was calibrated using external standards for targeted analytes. A set of quality control samples consisting of blanks, blank matrix spike, 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 condition used and the ion/wavelength monitored for each analyte is given in Table 60.

Table 60 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)
Ag	ICP-MS	Rh	NA	NA	NA	800	107 m/z
Al	ICP-MS	Rh	NA	NA	NA	800	27 m/z
As	ICP-MS	Rh	ORS	He	800	NA	75 m/z
B	ICP-MS	Rh	NA	NA	NA	800	11 m/z
Ba	ICP-OES	Y	NA	NA	NA	800	445.403 nm
Bi	ICP-MS	Ir	NA	NA	NA	800	209 m/z
Ca	ICP-OES	Y	NA	NA	NA	800	422.673 nm
Cd	ICP-MS	Rh	NA	NA	800	800	111 m/z
Co	ICP-MS	Rh	ORS	He	NA	800	59 m/z
Cr	ICP-MS	Rh	ORS	He	800	NA	52 m/z
Cu	ICP-MS	Rh	ORS	He	800	NA	65 m/z
Cs	ICP-MS	Rh	ORS	He	NA	800	113 m/z
Hg	ICP-MS	Rh	NA	NA	800	NA	201
K	ICP-MS	Rh	ORS	He	NA	800	39 m/z
Li	ICP-MS	Rh	ORS	He	NA	800	7 m/z

Table 60 Instrumental Technique used for Acid Extractable Elements (continued)

Analyte	Instrument	Internal Standard	Reaction/ Collision Cell (if applicable)	Cell Mode/Gas (if applicable)	S1Final Dilution Factor	S2/S3 Final Dilution Factor	Ion (m/z)/ Wavelength (nm)
Mg	ICP-OES	Y	NA	NA	NA	800	280.270 nm
Mn	ICP-MS	Rh	ORS	He	800	NA	55 m/z
Mo	ICP-MS	Rh	ORS	He	800	NA	95 m/z
Na	ICP-OES	Y	NA	NA	NA	800	588.995 nm
Ni	ICP-MS	Rh	ORS	He	800	NA	60
P	ICP-OES	Rh	NA	NA	NA	800	213.618
Pb	ICP-MS	Ir	NA	NA	800	NA	Average of 206, 207, 208 m/z
Rb	ICP-MS	Rh	ORS	He	NA	800	85 m/z
S	ICP-MS	Y	NA	NA	NA	800	191.972 nm
Se	ICP-MS	Rh	ORS	HEHe	800	NA	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	800	NA	51 m/z
Zn	ICP-MS	Rh	ORS	He	800	NA	66 m/z

Sample Analysis for Exchangeable Bases

A test portion of 5 g was weighed into a 100 mL polypropylene container. The container was then filled with 100 mL NH_4Cl. The suspension was shaken, at room temperature for 1 h, centrifuged, and filtered through 0.45 µm filter. A summary of the measurement techniques used is presented in Table 61.

Table 61 Instrumental Technique used for Exchangeable Bases

Analyte	Instrument	Internal Standard	Final Dilution Factor	Wavelength nm
Exchangeable Ca^{2+}	ICP-OES	Y	40	315.887
Exchangeable Mg^{2+}	ICP-OES	Y	40	279.8
Exchangeable Na^{+}	ICP-OES	Y	40	588.995
Exchangeable K^{+}	ICP-OES	Y	40	766.491

Sample Analysis for Total Carbon and Total Organic Carbon

The measurements for TC and TOC were made using NMI Method NWS15.[32] For TOC measurements a portion of sample weighing 0.25 g was reacted for 12 hours with 20 mL diluted hydrochloric acid to remove inorganic carbon. The sample was further purged with nitrogen gas to remove the inorganic carbon in solution and further diluted with 20 mL Milli-Q water. The insoluble part was then filtered and collected on a filter, dried and analysed as total carbon (TC). The TOC was calculated as the sum of the TOC from the insoluble part and the dissolved organic carbon (DOC) from liquid solution.

Sample Analysis for Total Nitrogen

Total Nitrogen in Sample S3 was measured as the sum of TKN +NOx.
The measurements for TKN were made using NMI Method NWS9.[34] Organic nitrogen from a test portion of 1 g was converted to ammonia with 50 mL digestion reagent (potassium sulfate, sulfuric acid and cupric sulfate) on a block digester at 400 °C ± 5 °C for 4 hours. The digested solution was then made alkaline with sodium hydroxide solution, distilled into a steam distillation analyser unit and automatically titrated with standard hydrochloric acid to the end point. The amount of ammonia nitrogen was then calculated.

Measurements for NOx in soil were made using NMI Method NW_B19.[35] A test portion of 10 g was weighed into a 100 mL polypropylene container. The container was then filled with 50 mL deionised water. The suspension was shaken, at room temperature for 1 h, centrifuged, and filtered through 0.45 µm filter. NO_3^--N was further measured by cadmium reduction to NO_2^--N followed by NO_x (the reduced NO_2^--N plus original NO_2^--N) measurements by FIA.

Sample Analysis for Colwell P

The measurements for Colwell P in S3 were made using NMI method NT2_59.[36] A test portion of approximately 1 g of dried soil were weighed into a 125 mL plastic container and shooked with 100 mL of extracting solution (0.5 M sodium bicarbonate at pH 8.5) for 16 hours. Colwell P was then measured in the extracting solution as P by ICP-OES.

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 'ISO 13258:2015(E)[8]; the uncertainty was estimated as:

$$u_{rob\ av} = 1.25 * S_{rob\ av} / \sqrt{p} \qquad \text{Equation 3}$$

where:

$u_{rob\ av}$ robust average standard uncertainty
$S_{rob\ mean}$ 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 62.

Table 62 Uncertainty of Assigned Value for Mn in Sample S1

No. results (p)	17
Robust Average	155 mg/kg
$S_{rob\ av}$	9.1 mg/kg
$u_{rob\ av}$	2.8 mg/kg
k	2
$U_{rob\ av}$	5.5 mg/kg

The assigned value for **Mn** in Sample S1 is **155 ± 6 mg/kg**

z-Score and E_n-score

For each participant's result 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 63.

Table 63 z-Score and E_n-score for Mn result reported by Laboratory 1 in S1

Mn Result mg/kg	Assigned Value mg/kg	Set Target Standard Deviation	z-Score	E_n-Score
168±16.8	155±6	10% as CV or 0.10x155 = =15.5 mg/kg	$z = \frac{(168-155)}{15.5}$ z = 0.84	$En = \frac{(168-155)}{\sqrt{16.8^2+6^2}}$ E_n=0.73

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 2016 NMI carried out fourteen 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 thirteen 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. Results are presented in Tables 64 and 65.

Table 64 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
AQA 09-13	Biosoil	4.091 ± 0.41	3.64 ± 0.43	16	11
	Soil	4.29 ± 0.43	4.57 ± 0.50	15	12
AQA 11-01	Biosoil	3.54 ± 0.35	3.57 ± 0.26	20	18
AQA 13-05	Soil	9.22 ± 1.4	9.21 ± 0.68	14	22
AQA 14-11	Sediment	7.91 ± 1.2	7.37 ± 0.32	12	21
AQA 15-02	Sludge	8.29 ± 1.2	7.02 ± 0.29	13	22
	Sludge	7.42 ± 1.1	7.02 ± 0.29	11	17
AQA 15-14	Sediment	10 ± 1.5	9.95 ± 0.40	6.7	17
	Soil	4.53 ± 0.9	4.47 ± 0.19	6.4	14
AQA 16-02	Agricultural Soil	2.67 ± 0.4	2.11 ± 0.17	14	20
Average				13**	

* Expanded uncertainty at approximately 95% confidence.
** The mean value of Robust CV was used. The pooled standard deviation could also be used. In this case the pooled standard deviation is 13%. Using a coverage factor of 2 gives an estimate of 26%.

Table 65 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
AQA 10-12	Soil	16.6 ± 1.66	14.4 ± 0.7	8.5	19
AQA 11-12	Moist Soil	25 ± 3.6	21.6 ± 2.2	15	13
AQA 12-01	Sediment	18.4 ± 2.7	17.3 ± 0.8	8.1	21
AQA 12-14	Soil	16.6 ± 2.4	14.8 ± 0.9	11	20
AQA 13-14	Sandy Soil	16.6 ± 2.4	15.1 ± 0.9	10	21
AQA 14-05	Soil	13.2 ± 1.9	12.3 ± 0.5	7.8	25
Average				10**	

* Expanded uncertainty at approximately 95% confidence.
** The mean value of Robust CV was used. The pooled standard deviation could also be used. In this case the pooled standard deviation is 10%. Using a coverage factor of 2 gives an estimate of 20%.

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 66 sets out the expanded uncertainty for results of the measurement of As in soil, biosoil, sediment, sludge, sandy soil, moist soil and agricultural soil over the ranges 0.5 to 10 mg/kg and 10 to 30 mg/kg.

Table 66 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 16 different PT samples over seven years can be assumed to include all the relevant uncertainty components (different matrices, operators, reagents, calibrators etc.), and so complies with ISO 17025.[10]

APPENDIX 4 - ACRONYMS AND ABBREVIATIONS

APHA	American Public Health Association
A.V.	Assigned Value
CRI	Collision Reaction Interface
CRM	Certified Reference Material
CV	Coefficient of Variation
CV-AAS	Cold Vapour-Atomic Absorption Spectrometry
CV-AFS	Cold Vapour-Atomic Florescence Spectrometry
DA	Discreet Analyser
DRC	Dynamic Reaction Cell
FIA	Flow Injection Analyser
HEHe	High energy He mode
H.V.	Homogeneity Value
ICP-MS	Quadrupole - Inductively Coupled Plasma - Mass Spectrometry
ICP-OES-AV	Inductively Coupled Plasma - Optical Emission Spectrometry- axial view
ICP-OES-AV-buffer	Inductively Coupled Plasma - Optical Emission Spectrometry- axial view with buffer
ICP-OES-AV-eq	Inductively Coupled Plasma - Optical Emission Spectrometry- axial view with correction equation
ICP-OES-RV	Inductively Coupled Plasma - Optical Emission Spectrometry- radial view
IC	Ion chromatograph
IR	Infrared Detector
ISE	Ion selective electrode
Max	Maximum value in a set of results
Md	Median
Min	Minimum value in a set of results
NEPC	National Environmental Protection Council
NMI	National Measurement Institute (of Australia)
NR	Not Reported
NT	Not Tested
ORS	Octopole Reaction System
PT	Proficiency Test
RM	Reference Material
Robust CV	Robust Coefficient of Variation
Robust SD	Robust Standard Deviation
S	Spiked or formulated concentration of a PT sample
SS	Spiked sample
SI	The International System of Units
s^2_{sam}	Sampling variance
s_a/σ	Analytical standard deviation divided by the target standard deviation
SFA	Segment Flow Analyser
SRM	Standard Reference Material (Trademark of NIST)
Target SD	Target standard deviation
σ	Target standard deviation
UC	Universal Cell
UV-Vis	Ultraviolet and Visible Spectroscopy

APPENDIX 5 - INSTRUMENT DETAILS

Table 67 Instrument Conditions Ag

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					N/A		
2	ICP-OES-AV-buffer	Lu			N/A	1000	328.068
3					N/A		
4	ICP-MS	Rh	NA	NA	N/A	625	109
5	ICP-MS	Sc, Ir, Rh	ORS	He	N/A		107 m/z
6					N/A		
7	ICP-MS	Rh	ORS	He	N/A	800	107
8					N/A	N/A	
9					N/A	N/A	
10	ICP-MS	In	ORS		N/A	2.5	107
11	ICP-OES-AV-buffer	Eu390.711	NA	NA	N/A	80	Ag328.068
12					N/A	N/A	
13	ICP-OES-AV	Lu			N/A	83	328.068
14	ICP-MS	Rh	ORS	He	N/A	200	107
15					N/A	N/A	
16	ICP-OES-AV-buffer	Lu			N/A	2.5	328.068
17	ICP-MS	Rh	ORS	He	N/A	20	107
18	ICP-MS	Rh	ORS	He	N/A	5000	107
19	ICP-OES-AV-buffer	Yttrium	NA	NA	N/A	1	328.068

Table 68 Instrument Conditions Al

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-OES-AV-equation				N/A	1000	396.153
2	ICP-OES-AV-buffer	Lu			N/A	50	396.152
3					N/A		
4	ICP-MS	Sc	UC	He	N/A	625	27
5	ICP-OES-AV-buffer	Cs, Eu			N/A		308.215 nm
6					N/A		
7	ICP-MS	Sc		none	N/A	800	27
8					N/A	N/A	
9					N/A	N/A	
10	ICP-OES-AV-buffer	Lu			N/A	50	396.152
11	ICP-OES-AV-buffer	Eu 412.972	NA	NA	N/A	80	Al 237.312
12					N/A	N/A	
13	ICP-OES-AV	Lu			N/A	833	396.152
14	ICP-MS	Sc	ORS	He	N/A	200	27
15					N/A	N/A	
16	ICP-OES-AV-buffer	Lu			N/A	50	396.152
17	ICP-MS	Sc	ORS	He	N/A	20	27
18	ICP-MS	Sc	NA		N/A	1000	27
19	ICP-OES-AV-buffer	Yttrium	NA	NA	N/A	1	308.215

Table 69 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)/Absor bance(nm)
1	ICP-OES-AV-equation				10	N/A	188.979
2	ICP-MS	Ge	ORS	He	1000	N/A	75
3						N/A	
4	ICP-MS	Rh	NA	NA	625	N/A	75
5	ICP-MS	Sc, Ir, Rh	ORS	He		N/A	75 m/z
6	ICP-MS	Rh	NA	He	250	N/A	
7	ICP-MS	Rh	ORS	He	N/A	800	75
8						N/A	
9	GFAAS					N/A	
10	ICP-MS	Ge	ORS	He	2.5	N/A	75
11	ICP-OES-AV-buffer	Lu 219.556	NA	NA	80	N/A	As 188.980
12					N/A	N/A	
13	ICP-OES-AV	Lu			83	N/A	188.98
14	ICP-MS	Rh	ORS	He	200	N/A	75
15					N/A	N/A	
16	ICP-MS	Ge	ORS	He	2.5	N/A	75
17	ICP-MS	Ge	ORS	He	20	N/A	75
18	ICP-MS	Rh	ORS	He	5000	N/A	75
19	ICP-OES-AV-buffer	Yttrium	NA	NA	1	N/A	188.979

Table 70 Instrument Conditions B

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1 Final Dilution Factor	S2/S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/Absor bance(nm)
1	ICP-OES-AV-equation				N/A	100	249.677
2	ICP-MS	Li6	ORS	He	N/A	1000	11
3					N/A		
4	ICP-MS	Sc	NA	NA	N/A	625	10
5	ICP-OES-AV-buffer	Cs, Eu			N/A		249.772 nm
6	ICP-MS	Sc	NA	NA	N/A	250	
7	ICP-MS	Sc		none	N/A	800	11
8					N/A	N/A	
9					N/A	N/A	
10	ICP-MS	Ge	ORS		N/A	2.5	11
11	ICP-OES-AV-buffer	Eu 271.700	NA	NA	N/A	80	B 249.678
12					N/A	N/A	
13	ICP-OES-AV	Lu			N/A	83	182.577
14	ICP-MS	Sc	ORS	He	N/A	200	11
15					N/A	N/A	
16	ICP-MS	Li6	ORS	He	N/A	2.5	11
17	ICP-MS	Sc	ORS	No gas	N/A	20	11
18	ICP-MS	Y	NA		N/A	100	11
19	ICP-OES-AV-buffer	Yttrium	NA	NA	N/A	1	249.678

Table 71 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-OES-AV-equation				N/A	100	233.527
2	ICP-MS	Rh	ORS	He	N/A	1000	138
3					N/A		
4	ICP-MS	Ir	NA	NA	N/A	625	138
5	ICP-MS	Sc, Ir, Rh	ORS	He	N/A		137 m/z
6	ICP-MS	In	NA	NA	N/A	250	
7	ICP-MS	Rh	ORS	He	N/A	800	137
8					N/A	N/A	
9					N/A	N/A	
10	ICP-MS	Lu	ORS		N/A	2.5	137
11	ICP-OES-AV-buffer	Eu 290.667	NA	NA	N/A	80	Ba 455.403
12	ICP-OES-RV	Y			N/A	100	493.4
13	ICP-OES-AV	Lu			N/A	83	493.408
14	ICP-MS	Rh	ORS	He	N/A	200	135
15					N/A		
16	ICP-MS	Rh	ORS	He	N/A	2.5	138
17	ICP-MS	Rh	ORS	He	N/A	20	137
18	ICP-MS	Tb	ORS	He	N/A	1000	137
19	ICP-OES-AV-buffer	Yttrium	NA	NA	N/A	1	455.403

Table 72 Instrument Conditions Bi

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					N/A		
2	ICP-MS	Rh	ORS	He	N/A	1000	209
3					N/A		
4	ICP-MS	Ir	NA	NA	N/A	625	209
5	ICP-MS	Sc, Ir, Rh	ORS	He	N/A		209 m/z
6	ICP-MS	Ir	NA	NA	N/A	250	
7	ICP-MS	Ir	ORS	He	N/A	800	209
8					N/A	N/A	
9					N/A	N/A	
10	ICP-MS	Lu	ORS		N/A	2.5	209
11	ICP-MS	Ir 193	ORS	O2	N/A	8000	Bi 209
12					N/A	N/A	
13	ICP-OES-AV	Lu			N/A	N/A	N/A
14	ICP-MS	Lu	ORS	He	N/A	200	209
15					N/A	N/A	
16	ICP-MS	Rh	ORS	He	N/A	2.5	209
17	ICP-MS	Lu	ORS	He	N/A	20	209
18	ICP-MS	Tb	NA		N/A	1000	209
19	ICP-OES-AV-buffer	Yttrium	NA	NA	N/A	1	190.171

Table 73 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					N/A	N/A	
2	ICP-OES-AV-buffer	Lu			N/A	50	315.933
3					N/A	N/A	
4	ICP-MS	Sc	NA	NA	N/A	625	43
5	ICP-OES-AV-buffer	Cs, Eu			N/A		370.602 nm
6							
7	ICP-OES-RV	Y			N/A	800	315.887
8					N/A	N/A	
9					N/A	N/A	
10	ICP-OES-AV-buffer	Lu			N/A	2500	315.887
11							
12	ICP-OES-RV	Y			N/A	4000	393.3
13	ICP-OES-AV	Lu			N/A	83	317.933
14					N/A	N/A	
15					N/A		
16	ICP-OES-AV-buffer	Lu			N/A	2500	315.933
17	ICP-OES-RV	Lu	N/A	N/A	N/A	200	317.941
18	ICP-MS	Sc	ORS	H2	N/A	N/A	40
19	NA	NA	NA	NA	N/A	N/A	N/A

Table 74 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-OES-AV-equation				100	N/A	214.44
2	ICP-MS	Rh	ORS	He	1000	N/A	111
3						N/A	
4	ICP-MS	Rh	NA	NA	625	N/A	111
5	ICP-MS	Sc, Ir, Rh	ORS	He		N/A	111 m/z
6	ICP-MS	Rh	NA	NA	250	N/A	
7	ICP-MS	Rh	ORS	He	N/A	800	111
8						N/A	
9	AAS					N/A	
10	ICP-MS	In	ORS	He	2.5	N/A	111
11	ICP-OES-AV-equation	Lu 291.136	NA	NA	80	N/A	Cd 226.502
12					N/A	N/A	
13	ICP-OES-AV	Lu			83	N/A	214.439
14	ICP-MS	Rh	ORS	He	200	N/A	111
15					N/A	N/A	
16	ICP-MS	Rh	ORS	He	2.5	N/A	111
17	ICP-MS	Rh	ORS	He	20	N/A	111
18	ICP-MS	Rh	NA		1000	N/A	111
19	NA	NA	NA	NA	N/A	N/A	N/A

Table 75 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-OES-AV-equation				N/A	100	228.616
2	ICP-MS	Ge	ORS	He	N/A	1000	59
3					N/A		
4	ICP-MS	Ge	UC	He	N/A	625	59
5	ICP-MS	Sc, Ir, Rh	ORS	He	N/A		59 m/z
6	ICP-MS	Rh	NA	He	N/A	250	
7	ICP-MS	Rh	ORS	He	N/A	800	59
8					N/A	N/A	
9					N/A	N/A	
10	ICP-MS	Ge	ORS	He	N/A	2.5	59
11	ICP-OES-AV-buffer	Eu 271.700	NA	NA	N/A	80	Co 228.615
12					N/A	N/A	
13	ICP-OES-AV	Lu			N/A	83	230.786
14	ICP-MS	Sc	ORS	He	N/A	200	59
15					N/A	N/A	
16	ICP-MS	Ge	ORS	He	N/A	2.5	59
17	ICP-MS	Sc	ORS	He	N/A	20	59
18	ICP-MS	Rh	ORS	He	N/A	1000	59
19	ICP-OES-AV-buffer	Yttrium	NA	NA	N/A	1	228.615

Table 76 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-OES-AV-equation				100	N/A	267.716
2	ICP-MS	Ge	ORS	He	50	N/A	52
3						N/A	
4	ICP-MS	Sc	UC	He	625	N/A	52
5	ICP-MS	Sc, Ir, Rh	ORS	He		N/A	52 m/z
6	ICP-MS	Sc	NA	He	250	N/A	
7	ICP-MS	Rh	ORS	He	N/A	800	52
8						N/A	
9	AAS					N/A	
10	ICP-MS	Ge	ORS	He	2.5	N/A	52
11	ICP-OES-AV-buffer	Eu 271.700	NA	NA	80	N/A	Cr 205.560
12					N/A	N/A	
13	ICP-OES-AV	Lu			83	N/A	267.716
14	ICP-MS	Sc	ORS	He	200	N/A	52
15					N/A	N/A	
16	ICP-MS	Ge	ORS	He	2.5	N/A	52
17	ICP-MS	Sc	ORS	He	20	N/A	52
18	ICP-MS	Ge	ORS	He	5000	N/A	52
19	ICP-OES-AV-buffer	Yttrium	NA	NA	1	N/A	267.716

Table 77 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-OES-AV-equation				100	N/A	327.393
2	ICP-MS	Ge	ORS	He	1000	N/A	63
3						N/A	
4	ICP-MS	Ge	UC	He	625	N/A	63
5	ICP-MS	Sc, Ir, Rh	ORS	He		N/A	63 m/z
6	ICP-MS	Rh	NA	He	250	N/A	
7	ICP-MS	Rh	ORS	He	N/A	800	63
8						N/A	
9	AAS					N/A	
10	ICP-MS	Ge	ORS	He	2.5	N/A	63
11	ICP-OES-AV-buffer	Eu 271.700	NA	NA	80	N/A	Cu 327.395
12					N/A	N/A	
13	ICP-OES-AV	Lu			83	N/A	327.395
14	ICP-MS	Sc	ORS	He	200	N/A	63
15					N/A	N/A	
16	ICP-MS	Ge	ORS	He	2.5	N/A	63
17	ICP-MS	Ge	ORS	He	20	N/A	63
18	ICP-MS	Rh	ORS	He	5000	N/A	63
19	ICP-OES-AV-buffer	Yttrium	NA	NA	1	N/A	327.395

Table 78 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					N/A		
2	ICP-MS	Rh			N/A	1000	133
3					N/A		
4					N/A		
5	ICP-MS	Sc, Ir, Rh	ORS	He	N/A		133 m/z
6	ICP-MS	In	NA	NA	N/A	250	
7	ICP-MS	Rh	ORS	He	N/A	800	133
8					N/A	N/A	
9					N/A	N/A	
10	ICP-MS	In	ORS		N/A	2.5	133
11							
12					N/A	N/A	
13	ICP-OES-AV	Lu			N/A	N/A	N/A
14	ICP-MS	Rh	ORS	He	N/A	200	133
15					N/A	N/A	
16					N/A	2.5	
17	N/A	N/A	N/A	N/A	N/A	N/A	N/A
18	ICP-MS	Tb	ORS	He	N/A	1000	133
19	NA	NA	NA	NA	N/A	N/A	N/A

Table 79 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					N/A	N/A	
2	ICP-OES-AV-buffer	Lu			N/A	50	259.94
3					N/A	N/A	
4	ICP-MS	Ge	NA	NA	N/A	625	54
5	ICP-OES-AV-buffer	Cs, Eu			N/A		370.792 nm
6							
7	ICP-OES-RV	Y			N/A	800	261.187
8					N/A	N/A	
9					N/A	N/A	
10	ICP-OES-AV-buffer	Ge			N/A	2500	261.187
11							
12	ICP-OES-AV-equation	Y			N/A	100	238.2
13	ICP-OES-AV	Lu			N/A	83	238.204
14	ICP-MS	Sc	ORS	He	N/A	N/A	56
15					N/A		
16	ICP-OES-AV-buffer	Lu			N/A	2500	259.94
17	ICP-MS	Sc	ORS	He	N/A	20	56
18	ICP-MS	Rh	ORS	H2	N/A	N/A	56
19	NA	NA	NA	NA	N/A	N/A	N/A

Table 80 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						N/A	
2	AAS	--			500	N/A	253.7
3						N/A	
4	ICP-MS	Ir	NA	NA	625	N/A	201
5	AAS					N/A	253.7 nm
6	ICP-MS	Ir	NA	NA	250	N/A	
7	ICP-MS	Ir	ORS	He	N/A	800	201
8						N/A	
9	MHAAS					N/A	
10	AAS	N/A			1000	N/A	253.7
11	ICP-OES-AV-buffer	Lu 291.139	NA	NA	80	N/A	Hg 194.164
12					N/A	N/A	
13	Cetac Hg Analyser				83	N/A	253.7
14	ICP-MS	Lu	ORS	He	200	N/A	202
15					N/A	N/A	
16	AAS	--			1000	N/A	253.7
17	ICP-MS	Lu	ORS	He	20	N/A	201
18	ICP-MS	Ir	NA		5000	N/A	202
19	AAS	NA	NA	NA	1	N/A	253

Table 81 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					N/A	N/A	
2	ICP-OES-AV-buffer	Lu			N/A	50	766.491
3					N/A	N/A	
4	ICP-MS	Sc	NA	NA	N/A	625	39
5	ICP-OES-AV-buffer	Cs, Eu			N/A		769.897 nm
6							
7	ICP-OES-RV	Y			N/A	800	39
8					N/A	N/A	
9					N/A	N/A	
10	ICP-OES-AV-buffer	Cs			N/A	50	766.941
11							
12	ICP-OES-AV-equation	Y			N/A	100	769.8
13	ICP-OES-AV	Lu			N/A	83	769.897
14					N/A	N/A	
15					N/A		
16	ICP-OES-AV-buffer	Lu			N/A	50	766.491
17	ICP-OES-RV	Lu	N/A	N/A	N/A	20	766.502
18	ICP-MS	Sc	ORS	He	N/A	N/A	39
19	NA	NA	NA	NA	N/A	N/A	N/A

Table 82 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					N/A		
2	ICP-MS	Lu			N/A	2.5	139
3					N/A		
4					N/A		
5	ICP-MS	Sc, Ir, Rh	ORS	He	N/A		139 m/z
6	ICP-MS	In	NA	NA	N/A	250	
7	ICP-MS	Rh	ORS	He	N/A	800	139
8					N/A	N/A	
9					N/A	N/A	
10	ICP-MS	Lu	ORS	He	N/A	2.5	139
11							
12					N/A	N/A	
13	ICP-OES-AV	Lu			N/A	N/A	N/A
14	ICP-MS	Rh	ORS	He	N/A	200	139
15					N/A	N/A	
16	ICP-MS	Lu			N/A	2.5	139
17	N/A	N/A	N/A	N/A	N/A	N/A	N/A
18	ICP-MS	Tb	ORS	He	N/A	5000	139
19	ICP-MS		NA	NA	N/A	1	

Table 83 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-OES-AV-equation				N/A	100	670.784
2	ICP-OES-AV-buffer	Lu			N/A	50	670.3
3					N/A		
4	ICP-MS	Sc	NA	NA	N/A	625	7
5	ICP-MS	Sc, Ir, Rh	ORS	He	N/A		7 m/z
6	ICP-MS	Sc	NA	NA	N/A	250	
7	ICP-MS	Sc		none	N/A	800	7
8					N/A	N/A	
9					N/A	N/A	
10	ICP-OES-AV-buffer	Lu			N/A	50	670.783
11	ICP-MS	Sc45	ORS		N/A	8000	Li7
12					N/A	N/A	
13	ICP-OES-AV	Lu			N/A	N/A	N/A
14	ICP-MS	Sc	ORS	No GAS	N/A	200	7
15					N/A	N/A	
16	ICP-OES-AV-buffer	Lu			N/A	50	670.3
17	ICP-MS	Li 6	ORS	No gas	N/A	20	7
18	ICP-MS	Sc	ORS	H2	N/A	1000	7
19	ICP-OES-AV-buffer	Yttrium	NA	NA	N/A	1	670.783

Table 84 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					N/A	N/A	
2	ICP-OES-AV-buffer	Lu			N/A	50	279.8
3					N/A	N/A	
4	ICP-MS	Sc	NA	NA	N/A	625	24
5	ICP-OES-AV-buffer	Cs, Eu			N/A		383.829 nm
6	ICP-MS	Sc	NA	He	N/A	N/A	
7	ICP-OES-RV	Y			N/A	800	279
8					N/A	N/A	
9					N/A	N/A	
10	ICP-OES-AV-buffer	Lu			N/A	50	279.8
11							
12	ICP-OES-RV	Y			N/A	100	279.5
13	ICP-OES-AV	Lu			N/A	83	383.829
14					N/A	N/A	
15					N/A		
16	ICP-OES-AV-buffer	Lu			N/A	50	279.8
17	ICP-OES-RV	Lu	N/A	N/A	N/A	20	285.217
18	ICP-MS	Sc	ORS	H2	N/A	N/A	24
19	NA	NA	NA	NA	N/A	N/A	N/A

Table 85 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-OES-AV-equation				100	N/A	257.61
2	ICP-MS	Ge	ORS	He	1000	N/A	55
3						N/A	
4	ICP-MS	Ge	NA	NA	625	N/A	55
5	ICP-MS	Sc, Ir, Rh	ORS	He		N/A	55 m/z
6	ICP-MS	Rh	NA	He	250	N/A	
7	ICP-MS	Rh	ORS	He	N/A	800	55
8						N/A	
9	AAS					N/A	
10	ICP-OES-AV-buffer	Lu			50	N/A	257.61
11	ICP-OES-AV-buffer	Eu 271.700	NA	NA	80	N/A	Mn 257.610
12					N/A	N/A	
13	ICP-OES-AV	Lu			83	N/A	260.568
14	ICP-MS	Sc	ORS	He	200	N/A	55
15					N/A	N/A	
16	ICP-MS	Ge	ORS	He	50	N/A	55
17	ICP-MS	Sc	ORS	He	20	N/A	55
18	ICP-MS	Rh	NA		5000	N/A	55
19	ICP-OES-AV-buffer	Yttrium	NA	NA	1	N/A	257.61

Table 86 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-OES-AV-equation				100	N/A	202.036
2	ICP-MS	Rh	ORS	He	1000	N/A	95
3						N/A	
4	ICP-MS	Rh	NA	NA	625	N/A	98
5	ICP-MS	Sc, Ir, Rh	ORS	He		N/A	95 m/z
6	ICP-MS	Rh	NA	NA	250	N/A	
7	ICP-MS	Rh	ORS	He	N/A	800	95
8						N/A	
9	GFAAS					N/A	
10	ICP-MS	Rh	ORS	He	2.5	N/A	95
11	ICP-OES-AV-buffer	Eu 271.700	NA	NA	80	N/A	Mo 202.032
12					N/A	N/A	
13	ICP-OES-AV	Lu			83	N/A	202.032
14	ICP-MS	Rh	ORS	He	200	N/A	95
15					N/A	N/A	
16	ICP-MS	Rh	ORS	He	2.5	N/A	95
17	ICP-MS	Rh	ORS	He	20	N/A	95
18	ICP-MS	Y	ORS	He	1000	N/A	95
19	ICP-OES-AV-buffer	Yttrium	NA	NA	1	N/A	202.032

Table 87 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					N/A	N/A	
2	ICP-OES-AV-buffer	Lu			N/A	50	588.995
3					N/A	N/A	
4	ICP-MS	Sc	NA	NA	N/A	625	23
5	ICP-OES-AV-buffer	Cs, Eu			N/A		589.592 nm
6	ICP-MS	Sc	NA	He	N/A	N/A	
7	ICP-MS	Rh	ORS	He	N/A	800	23
8					N/A	N/A	
9					N/A	N/A	
10	ICP-OES-AV-buffer	Cs			N/A	50	588.995
11							
12					N/A		
13	ICP-OES-AV	Lu			N/A	83	589.592
14					N/A	N/A	
15					N/A		
16	ICP-OES-AV-buffer	Lu			N/A	50	588.995
17	ICP-OES-RV	Lu	N/A	N/A	N/A	20	589.624
18	ICP-MS	Sc	ORS	H2	N/A	N/A	23
19	NA	NA	NA	NA	N/A	N/A	N/A

Table 88 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-OES-AV-equation				100	N/A	231.604
2	ICP-MS	Ge	ORS	He	1000	N/A	60
3						N/A	
4	ICP-MS	Ge	UC	He	625	N/A	60
5	ICP-MS	Sc, Ir, Rh	ORS	He		N/A	60 m/z
6	ICP-MS	Rh	NA	He	250	N/A	
7	ICP-MS	Rh	ORS	He	N/A	800	60
8						N/A	
9	AAS					N/A	
10	ICP-MS	Ge	ORS	He	2.5	N/A	60
11	ICP-OES-AV-buffer	Eu 271.700	NA	NA	80	N/A	Ni 231.604
12					N/A	N/A	
13	ICP-OES-AV	Lu			83	N/A	231.604
14	ICP-MS	Sc	ORS	He	200	N/A	60
15					N/A	N/A	
16	ICP-MS	Ge	ORS	He	2.5	N/A	60
17	ICP-MS	Ge	ORS	He	20	N/A	60
18	ICP-MS	Rh	ORS	He	5000	N/A	60
19	ICP-OES-AV-buffer	Yttrium	NA	NA	1	N/A	231.604

Table 89 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					N/A	N/A	
2	ICP-OES-AV-buffer	Lu			N/A	50	213.618
3					N/A	N/A	
4	ICP-MS	Sc	NA	NA	N/A	625	31
5	ICP-OES-AV-buffer	Cs, Eu			N/A		185.827 nm
6	ICP-MS	Sc	NA	He	N/A	N/A	
7	ICP-OES-RV	Y			N/A	800	213.618
8					N/A	N/A	
9					N/A	N/A	
10	ICP-OES-AV-buffer	Lu			N/A	50	213.618
11							
12					N/A		
13	ICP-OES-AV	Lu			N/A	83	178.222
14					N/A	N/A	
15					N/A		
16	ICP-OES-AV-buffer	Lu			N/A	50	213.618
17	ICP-OES-RV	Lu	N/A	N/A	N/A	200	178.221
18	ICP-MS	Ge	ORS	He	N/A	N/A	31
19	NA	NA	NA	NA	N/A	N/A	N/A

Table 90 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-OES-AV-equation				100	N/A	220.353
2	ICP-MS	Rh	ORS	He	1000	N/A	206+207+208
3						N/A	
4	ICP-MS	Ir	NA	NA	625	N/A	206+207+208
5	ICP-MS	Sc, Ir, Rh	ORS	He		N/A	208 m/z
6	ICP-MS	Ir	NA	NA	250	N/A	
7	ICP-MS	Ir	ORS	He	N/A	800	207
8						N/A	
9	AAS					N/A	
10	ICP-OES-AV-buffer	Lu			50	N/A	220.353
11	ICP-OES-AV-buffer	Eu 271.700	NA	NA	80	N/A	Pb 220.353
12					N/A	N/A	
13	ICP-OES-AV	Lu			83	N/A	220.353
14	ICP-MS	Lu	ORS	He	200	N/A	208
15					N/A	N/A	
16	ICP-MS	Rh	ORS	He	50	N/A	206+207+208
17	ICP-MS	Lu	ORS	He	20	N/A	208
18	ICP-MS	Tb	NA		5000	N/A	208
19	ICP-OES-AV-buffer	Yttrium	NA	NA	1	N/A	220.353

Table 91 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					N/A		
2	ICP-MS	Ge	ORS	He	N/A	1000	85
3					N/A		
4					N/A		
5	ICP-MS	Sc, Ir, Rh	ORS	He	N/A		85 m/z
6	ICP-MS	Rh	NA	NA	N/A	250	
7	ICP-MS	Rh	ORS	He	N/A	800	85
8					N/A	N/A	
9					N/A	N/A	
10	ICP-MS	Ge	ORS		N/A	2.5	85
11							
12					N/A	N/A	
13	ICP-OES-AV	Lu			N/A	N/A	N/A
14	ICP-MS	Rh	ORS	He	N/A	200	85
15					N/A	N/A	
16					N/A		
17	N/A	N/A	N/A	N/A	N/A	N/A	N/A
18	ICP-MS	Y	ORS	He	N/A	1000	85
19	ICP-MS				N/A	1	

Table 92 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					N/A	N/A	
2	ICP-OES-AV-buffer	Lu			N/A	50	181.972
3					N/A	N/A	
4	ICP-OES-AV-equation	Y	NA	NA	N/A	62.5	181.975
5	ICP-OES-AV-buffer	Cs, Eu			N/A		181.972 nm
6	ICP-MS						
7	ICP-OES-RV	Y			N/A	800	181.972
8					N/A	N/A	
9					N/A	N/A	
10	ICP-OES-AV-buffer	Lu			N/A	50	182.562
11							
12	ICP-OES-AV-equation	Y			N/A	100	180.7
13	ICP-OES-AV	Lu			N/A	83	181.972
14					N/A	N/A	
15					N/A		
16	ICP-OES-AV-buffer	Lu			N/A	50	181.972
17	ICP-OES-RV	Lu	N/A	N/A	N/A	20	181.977
18			NA		N/A	N/A	
19	NA	NA	NA	NA	N/A	N/A	N/A

Table 93 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-OES-AV-equation				10	N/A	196.026
2	ICP-MS	Ge	ORS	H2	1000	N/A	78
3						N/A	
4	ICP-MS	Rh	NA	NA	625	N/A	82
5	ICP-MS	Sc, Ir, Rh	ORS	He		N/A	78 m/z
6	ICP-MS	Rh	NA	He	250	N/A	
7	ICP-MS	Rh	ORS	HEHe	N/A	800	78
8						N/A	
9	GFAAS					N/A	
10	ICP-MS	Ge	ORS	H2	2.5	N/A	78
11	ICP-MS	Rh103	ORS	O2	8000	N/A	Se78/94
12					N/A	N/A	
13	ICP-OES-AV	Lu			83	N/A	196.026
14	ICP-MS	Rh	ORS	He	200	N/A	78
15					N/A	N/A	
16	ICP-MS	Ge	ORS	H2	2.5	N/A	78
17	ICP-MS	Ge	ORS	HEHe	20	N/A	78
18	ICP-MS	Ge	ORS	H2	1000	N/A	78
19	ICP-OES-AV-buffer	Yttrium	NA	NA	1	N/A	196.026

Table 94 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						N/A	
2	ICP-MS	Rh			1000	N/A	118
3						N/A	
4	ICP-MS	Ge	NA	NA	625	N/A	118
5	ICP-MS	Sc, Ir, Rh	ORS	He		N/A	118 m/z
6	ICP-MS	Rh	NA	NA	250	N/A	
7	ICP-MS	Rh	ORS	He	N/A	800	118
8						N/A	
9	GFAAS					N/A	
10	ICP-MS	Rh	ORS	He	2.5	N/A	118
11	ICP-OES-AV-buffer	Lu 219.556	NA	NA	80	N/A	Sn 189.927
12					N/A	N/A	
13	ICP-OES-AV	Lu			83	N/A	189.925
14	ICP-MS	Rh	ORS	He	200	N/A	118
15					N/A	N/A	
16	ICP-MS	Rh			2.5	N/A	118
17	ICP-MS	Rh	ORS	He	20	N/A	118
18	ICP-MS	Rh	ORS	He	5000	N/A	120
19	ICP-OES-AV-buffer	Yttrium	NA	NA	1	N/A	189.925

Table 95 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-OES-AV-equation				N/A		421.552
2	ICP-MS	Lu			N/A	1000	407.771
3					N/A		
4	ICP-MS	Rh	NA	NA	N/A	625	88
5	ICP-OES-AV-buffer	Cs, Eu			N/A		430.544 nm
6	ICP-MS	Rh	NA	NA	N/A	250	
7	ICP-MS	Rh	ORS	He	N/A	800	88
8					N/A	N/A	
9					N/A	N/A	
10	ICP-OES-AV-buffer	Lu			N/A	50	407.771
11	ICP-OES-AV-buffer	Lu 219.556	NA	NA	N/A	80	Sr 216.596
12					N/A	N/A	
13	ICP-OES-AV	Lu			N/A	83	407.771
14	ICP-MS	Rh	ORS	He	N/A	200	88
15					N/A	N/A	
16	ICP-MS	Lu			N/A	50	407.771
17	ICP-MS	Ge	ORS	He	N/A	20	88
18	ICP-MS	Rh	ORS	He	N/A	5000	88
19	ICP-OES-AV-buffer	Yttrium	NA	NA	N/A	1	407.771

Table 96 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					N/A		
2	ICP-MS	Lu			N/A	1000	232
3					N/A		
4					N/A		
5	ICP-MS	Sc, Ir, Rh	ORS	He	N/A		232 m/z
6	ICP-MS						
7	ICP-MS	Ir	ORS	He	N/A	800	232
8					N/A	N/A	
9					N/A	N/A	
10	ICP-MS	Lu	ORS		N/A	2.5	232
11	ICP-MS	Ir 193	ORS	O2	N/A	8000	Th232
12					N/A	N/A	
13	ICP-OES-AV	Lu			N/A	N/A	N/A
14					N/A	N/A	
15					N/A	N/A	
16	ICP-MS	Lu			N/A	2.5	232
17	N/A	N/A	N/A	N/A	N/A	N/A	N/A
18			ORS	He	N/A		
19	ICP-MS				N/A	1	

Table 97 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					N/A		
2	ICP-MS	Lu			N/A	1000	238
3					N/A		
4	ICP-MS	Ir	NA	NA	N/A	625	238
5	ICP-MS	Sc, Ir, Rh	ORS	He	N/A		238 m/z
6	ICP-MS	Ir	NA	NA	N/A	250	
7	ICP-MS	Ir	ORS	He	N/A	800	238
8					N/A	N/A	
9					N/A	N/A	
10	ICP-MS	Lu	ORS		N/A	2.5	238
11	ICP-MS	Ir 193	ORS	He	N/A	8000	U238
12					N/A	N/A	
13	ICP-OES-AV	Lu			N/A	N/A	N/A
14	ICP-MS	Lu	ORS	He	N/A	200	238
15					N/A	N/A	
16	ICP-MS	Lu			N/A	2.5	238
17	ICP-MS	Lu	ORS	He	N/A	20	238
18	ICP-MS	Tb	NA		N/A	1000	238
19	ICP-MS				N/A	1	

Table 98 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-OES-AV-equation				100	N/A	311.071
2	ICP-MS	Ge			1000	N/A	51
3						N/A	
4	ICP-MS	Ge	NA	NA	625	N/A	51
5	ICP-MS	Sc, Ir, Rh	ORS	He		N/A	51 m/z
6	ICP-MS	Sc	NA	He	250	N/A	
7	ICP-MS	Rh	ORS	He	N/A	800	51
8						N/A	
9						N/A	
10	ICP-MS	Ge	ORS	He	2.5	N/A	51
11	ICP-OES-AV-buffer	Eu 272.778	NA	NA	80	N/A	V 292.401
12					N/A	N/A	
13	ICP-OES-AV	Lu			83	N/A	292.401
14	ICP-MS	Sc	ORS	He	200	N/A	51
15					N/A	N/A	
16	ICP-MS	Ge			2.5	N/A	51
17	ICP-MS	Sc	ORS	He	20	N/A	51
18	ICP-MS	Ge	ORS	He	5000	N/A	51
19	ICP-OES-AV-buffer	Yttrium	NA	NA	1	N/A	292.401

Table 99 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-OES-AV-equation				100	N/A	206.2
2	ICP-MS	Ge			1000	N/A	66
3						N/A	
4	ICP-MS	Ge	UC	He	625	N/A	66
5	ICP-MS	Sc, Ir, Rh	ORS	He		N/A	68 m/z
6	ICP-MS	Rh	NA	He	250	N/A	
7	ICP-MS	Rh	ORS	He	N/A	800	66
8						N/A	
9	AAS					N/A	
10	ICP-OES-AV-buffer	Lu			50	N/A	206.2
11	ICP-OES-AV-buffer	Eu 272.778	NA	NA	80	N/A	Zn 206.200
12					N/A	N/A	
13	ICP-OES-AV	Lu			83	N/A	213.857
14	ICP-MS	Sc	ORS	He	200	N/A	66
15					N/A	N/A	
16	ICP-MS	Ge			50	N/A	66
17	ICP-MS	Ge	ORS	He	20	N/A	66
18	ICP-MS	Rh	ORS	He	5000	N/A	66
19	ICP-OES-AV-buffer	Yttrium	NA	NA	1	N/A	213.857

Table 100 Instrument Conditions Exchangeable Ca^{2+}

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S3 Final Dilution Factor	Wavelength (nm)/
2	ICP-OES-AV-buffer				100	
4	ICP-OES-RV		NA	NA	10	317.933
5	ICP-OES-AV-buffer	Cs, Eu			25	370.602 nm
7	ICP-OES-RV	Y			80	315.887
10	ICP-OES-AV-buffer	NA	NA		100	315.887
12	AAS	NA	NA			
13	ICP-OES-AV-buffer	Lu				317.933
16	ICP-OES-AV-buffer	NA	NA		100	315.933
17	ICP-OES-RV	Lu	N/A	N/A	200	317.941

Table 101 Instrument Conditions Exchangeable K^{+}

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S3 Final Dilution Factor	Wavelength (nm)/
2	ICP-OES-AV-buffer				100	
4	ICP-OES-RV		NA	NA	10	766.49
5	ICP-OES-AV-buffer	Cs, Eu			25	769.897 nm
7	ICP-OES-RV	Y			80	766.491
10	ICP-OES-AV-buffer	NA	NA		100	766.941
12	AAS	NA	NA			
13	ICP-OES-AV-buffer	Lu				769.897
16	ICP-OES-AV-buffer	NA	NA		100	766.491
17	ICP-OES-RV	Lu	N/A	N/A	200	766.502

Table 102 Instrument Conditions Exchangeable Mg^{2+}

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S3 Final Dilution Factor	Wavelength (nm)/
2	ICP-OES-AV-buffer				100	
4	ICP-OES-RV		NA	NA	10	285.213
5	ICP-OES-AV-buffer	Cs, Eu			25	383.829 nm
7	ICP-OES-RV	Y			80	279.8
10	ICP-OES-AV-buffer	NA	NA		100	279.8
12	AAS	NA	NA			
13	ICP-OES-AV-buffer	Lu				383.829
16	ICP-OES-AV-buffer	NA	NA		100	279.8
17	ICP-OES-RV	Lu	N/A	N/A	200	285.217

Table 103 Instrument Conditions Exchangeable Na^+

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S3 Final Dilution Factor	Wavelength (nm)/
2	ICP-OES-AV-buffer				100	
4	ICP-OES-RV		NA	NA	10	589.592
5	ICP-OES-AV-buffer	Cs, Eu			25	589.592 nm
7	ICP-OES-RV	Y			80	588.995
10	ICP-OES-AV-buffer	NA	NA		100	588.995
12	AAS	NA	NA			
13	ICP-OES-AV-buffer	Lu				589.592
16	ICP-OES-AV-buffer	NA	NA		100	588.995
17	ICP-OES-RV	Lu	N/A	N/A	200	589.624

END OF REPORT