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Proficiency Test Report AQA 21-01 Metals, Nutrients and Exchangeable Bases in Soil

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

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Accredited for compliance with ISO/IEC 17043

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SUMMARY

This report presents the results of the proficiency test AQA 21-01, metals, nutrients and exchangeable bases in soil. The study focused on the measurement of the following acid extractable elements: Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cs, Cu, Fe, Hg, K, La, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Rb, S, Sb, Se, Sn, Sr, Th, Tl, U, V and Zn. Measurement of total P, P buffer index (with Colwell P)- PBI_{+ColP} , calcium chloride-extractable B, total carbon (TC), total organic carbon (TOC), total nitrogen (TN), Colwell P, Colwell K, EC, pH of 1:5 soil / 0.01 M $CaCl_2$ extract, exchangeable bases (Ca^{2+} , Mg^{2+} , Na^+ , K^+) - 1M NH_4Cl extract and moisture content was also included in the program.

The sample set consisted of one dried sediment sample, one moist sludge sample and one 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.

23 laboratories enrolled and reported 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 722 results, 645 (89%) returned a satisfactory score of $|z| \leq 2.0$.

Of 722 E_n -scores, 564 (78%) were satisfactory with $|E_n| \leq 1.0$.

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

The tests that presented the most analytical difficulty to participating laboratories were: B and Sb.

- 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 782 numerical results, 749 were reported with an expanded measurement uncertainty. An example of estimating measurement uncertainty using only proficiency testing data is given in Appendix 3.

A large number of participants are still reporting results in the wrong format; they attach an estimate of uncertainty expressed as a *value* to a result expressed as a *range* (e.g. less than the level of reporting).

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

Surplus test samples from this study are available for sale.

1 INTRODUCTION

1.1 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 21-01 is the 28th NMI proficiency study of inorganic analytes in soil.

1.2 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.

1.3 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 17043¹ and The International Harmonized Protocol for Proficiency Testing of (Chemical) Analytical Laboratories.⁴

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

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

2 STUDY INFORMATION

2.1 Selection of Matrices and Inorganic Analytes

The 61 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.

2.2 Participation

Twenty-three laboratories participated and all submitted results.

The timetable of the study was:

Invitations issued:	11 January 2021
Samples dispatched:	15 February 2021
Results due:	19 March 2021
Interim report issued:	23 March 2021

2.3 Test Material Specification

Three samples were provided for analysis:

Sample S1 was 25 g of dried sediment;

Sample S2 was 100 g of wet sludge; and

Sample S3 was 75 g of dried agricultural soil.

2.4 Laboratory Code

All participant laboratories were assigned a confidential code number.

2.5 Sample Preparation, Analysis and Homogeneity Testing

Test samples from previous studies have been demonstrated to be sufficiently homogeneous for the evaluation of participants' performance. Therefore, only a partial homogeneity test was conducted for all analytes with the exception of calcium chloride-extractable B, colwell K, EC, pH, PBI, TC, Mg, S, Sr and exchangeable bases, as the same preparation procedure was followed in previous studies.¹ The results from the partial homogeneity testing for these samples 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.

2.6 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.

2.7 Sample Storage, Dispatch and Receipt

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

The samples were dispatched by courier on 15 February 2021.

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.

2.8 Instructions to Participants

Participants were instructed as follows:

- Quantitatively analyse the samples using your normal test method.
- Sample S2, the sludge sample, should be thoroughly mixed before removing a test portion. To avoid loss of moisture, do not leave the sample uncovered.

- For Sample S3 for the determination of calcium chloride- extractable B, exchangeable bases (Ca^{2+} , Mg^{2+} , Na^+ , K^+) - 1M NH_4Cl extract and of P buffer index (with Colwell P)- $\text{PBI}_{+\text{ColP}}$, participants are asked to use the methods defined by Rayment, G.E. and David, J. L in “Soil Chemical Methods-Australasia”.
- For S1 report results for acid extractable elements on as received basis in units of mg/kg.
- For S2 report results for moisture content in % (g/100g). For acid extractable elements in S2 results are to be reported on dry weight bases (corrected for moisture content) and in units of mg/kg.
- For S3 report results on as received basis in units of $\text{cmol}(+)/\text{kg}$ 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 $\mu\text{S}/\text{cm}$.

SAMPLE S1		SAMPLE S2		SAMPLE S3	
Test acid extractable	Approximate Conc. Range (as received basis) mg/kg	Test acid extractable	Approximate Conc. Range (dry weight basis) mg/kg	Test	Approximate Conc. Range (as received basis) mg/kg
As	1-20	Ag	1-20	Ca (acid extractable)	250-5000
B	1-20	As	1-20	Calcium chloride –extractable B ¹	Not Available
Ba	50-250	Al	1000-20000	Colwell P	5-100
Be	0.2-4	Ba	25-500	Colwell K	5-100
Cd	0.2-4	Bi	0.1-5	EC	>100 $\mu\text{S}/\text{cm}$
Cr	5-100	Cd	0.2-4	Exchangeable Ca-1M NH_4Cl extract ²	>0.5 $\text{cmol}(+)/\text{kg}$
Cu	25-500	Co	1-20	Exchangeable Mg-1M NH_4Cl extract ²	>0.25 $\text{cmol}(+)/\text{kg}$
Hg	0.2-4	Cr	5-100	Exchangeable Na-1M NH_4Cl extract ²	>0.05 $\text{cmol}(+)/\text{kg}$
Li	1-20	Cs	0.2-4	Exchangeable K-1M NH_4Cl extract ²	>0.015 $\text{cmol}(+)/\text{kg}$
Mn	25-500	Cu	25-500	Fe (acid extractable)	500-10000
Ni	5-100	Hg	0.2-4	K (acid extractable)	Not Available
Pb	5-100	La	5-50	Mg (acid extractable)	Not Available
Rb	1-20	Mo	0.2-4	Na (acid extractable)	Not Available
Sb	5-100	Ni	5-50	P (acid extractable)	50-1250
Se	0.5-20	Pb	5-100	P (total)	Not Available
Sn	1-20	Se	0.5-20	P buffer index (with Colwell P)- $\text{PBI}_{+\text{ColP}}$ ³	Not Available
Th	1-20	Tl	0.02-0.4	pH of 1:5soil/0.01M CaCl_2 extract	Not Available
V	5-100	U	0.2-4	S (acid extractable)	10-200
Zn	25-500	Zn	25-500	Sr (acid extractable)	5-100
		Moisture Content	10-60%	Total Carbon	5000-50000
				Total Organic Carbon	5000-50000
				Total Nitrogen	250-5000

¹Method 12C, ²Method 15A1, ³Method 9I2 as defined by Rayment, G.E. and David, J. L in “Soil Chemical Methods-Australasia” (2011).

- Report results using the electronic results sheet emailed to you:
- Report results as you would report to a client. For each analyte, report the expanded measurement uncertainty.
- Please send us all the requested details regarding the test method.

2.9 Interim Report

An interim report was emailed to participants on 23 March 2021.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Method Summaries

Summaries of test methods are transcribed in Tables 1 to 10. 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)	Vol. HNO ₃ (1:1) (mL)	Vol. HCl (1:1) (mL)	Vol. H ₂ O ₂ (mL)	Other (mL)
1	EPA Method 3050B Acid Digestion of Sediments, Sludges and Soils	0.5	85	240	5	5				
3	EPA Method 3050B	2	100	60	4	12				4 (H ₂ O)
4	In-house	2.2	95	90	5	5			5	
5	USEPA 3050	2.5	90	120	2	6				
6	200.2 Revision 2.8	1	95 ± 5	60	2	10			2	
8	EPA3050B, 6020B	2	95-105	60	4	12				
9*	USEPA 3050	3	85	120	10	5	10		6	
11*	Soil Chemical Methods - Australasia (Raymond and Lyons) - Method 17B1	3	95	120	22.5	7.5				
12	In house method S6 - referencing APHA 3125	0.4	120	60	2.5	7.5				
13*	USEPA 200.2 rev 2.8	0.8-1.2	95	30	2	10			2	
14	USEPA 3050	1	95	60	10				6	
15	US EPA	1	95	150	5	5				
16	USEPA 3050B	0.5	95	120	3	3				
17			95-100	120	3	3				
19	US-EPA Method 200.2	1	95	50	2	2				10 (H ₂ O)
21*	US EPA 200.8	0.5	95	120	2	2				10 (H ₂ O)

* See Additional Information for Methodology in Table 2

Table 2 Additional Information for Acid Extractable Elements

Lab Code	Additional Information
9	Instrument for Hg: Hg Analyser.
11	Ca, Mg, Na and K analysed by AAS, Fe, S and Sr analysed by ICP-OES; pH of 1:5 soil/0.1M CaCl ₂ and EC @ 25°C. Exchangeable Ca, Mg, Na and K in 1M NH ₄ Cl extract
13	S1: These results have been moisture corrected S2: Moisture Corrected S3: These results have been moisture corrected
21	Mercury analysis by Cold Vapour Atomic Fluorescence. S3: S has been reported as S-SO ₄

Table 3 Methodology for Total Carbon

Lab. Code	Method Reference	Test Method	Measurement Technique	Additional Information
1	Iso 10694-1995	High Temperature Oxidation	IR detector	
6		High Temperature Oxidation	Eltra CS2000	
12	In house	High Temperature Oxidation	LECO	
14	In-House	High Temperature Oxidation	Combustion Analyser	
15		High Temperature Oxidation		
16		High Temperature Oxidation	Infrared Gas Analysis	
17		High temp combustion		
20	6B2b	High Temperature Oxidation	IR-LECO	

Table 4 Methodology for Total Organic Carbon

Lab. Code	Method Reference	Test Method	Measurement Technique	Additional Information
1	Iso 10694-1995	High Temperature Oxidation		
6		High Temperature Oxidation	Eltra CS2000	
7	Soil Chemical Methods – Australasia, method 6B1. G. E. Rayment and D. J. Lyons, 2011	Chemical Oxidation with Ag ₂ SO ₄	Manual UV-Vis at 600 nm	
9	Rayment & Higginson 6B1	Chemical Oxidation no Ag ₂ SO ₄	DA	
11	Soil Chemical Methods – Australasia (Rayment & Lyons) – Method 6A1	Chemical Oxidation no Ag ₂ SO ₄	Supernatant is analysed by Colourimetry at an absorbance of 600 nm	
12	In house	High Temperature Oxidation	LECO	Sample digested with sulphurous acid prior to analysis on LECO
14	In-House	High Temperature Oxidation	Combustion Analyser	Performed after heating sample with conc HCl
15		High Temperature Oxidation		
16		High Temperature Oxidation	Infrared Gas Analysis	
17		High temp combustion		
20	6B3	High Temperature Oxidation	LECO	Sample was Fizz test with 4 M HCl and no Fizzing observed. Therefore, no acid treatment was carried for TOC

Table 5 Methodology for Colwell P and Colwell K

Lab. Code	Method Reference	Sample Mass (g)	Extraction Solution 0.5 M NaHCO ₃ Volume (mL)	Shake time (hours)	Final Dilution Factor (Colwell K)	Final Dilution Factor (Colwell P)	Measurement Technique (Colwell K))	Measurement Technique (Colwell P)
6	Rayment & Lyons 9B1 & 18A1	1	100	16	100	100	ICP-OES 404.721 nm, 766.491 nm	UV-Vis 882 nm
9	Rayment and Lyons 9B1	1	100	16		50		DA 880 nm
11	Soil Chemical Methods- Australasia – Rayment & Lyons – method 9B (Colwell P) and 18A1 (Colwell K)	1	100	16	2	5	AAS 766.5 nm	DA 880 nm
12	Colwell P 9B2, Colwell K 18A1	0.4	40	16	3280	328	ICPMS 31 m/z	FIA 880 nm
15	Rayment and Lyons 9B1	1.2	120	16				DA
17		0.5	50	16				ICP-OES
20		10	100	17				FIA

Table 6 Methodology for P Buffer Index – PBI_{ColP}

Lab. Code	Method Reference*	Sample Mass (g)	Extraction Solution (P equilibrating Solution) Volume (mL)	Shake time (hours)	Instrument	Final Dilution Factor	Wavelength (nm) / Absorbance (nm)
11	9I2	10	100	17	DA	100	880
12	9I2b	2	20	16	ICP-OES	10	213.617
20	9I2b	7	70	17	ICP-OES	10	178.24

*9I2 as defined by Rayment, G.E. and David, J. L. in “Soil Chemical Methods-Australasia”

Table 7 Methodology for Total P

Lab. Code	Method
6	Total P by APHA 4500 Norg-D with Jirka modification followed by DA finish
7	Total P by Kjeldahl digestion and Ascorbic Acid Colorimetric detection by Discrete Analyser
9	Total P by kjeldahl digestion and DA
11	In-house method – Soil digested as per Acid extractable metals (reverse aqua regia as above) then digest analysed on DA
13	Total P by Kjeldahl digestion + DA

Table 8 Methodology for Calcium Chloride Extractable B

Lab. Code	Method Reference*	Sample Mass (g)	Extraction Solution (0.01 M CaCl ₂) Volume (mL)	Reflux Time (min)	Instrument	Final Dilution Factor	Wavelength (nm) / Absorbance (nm)
6	12C	10	20	10	ICP-OES	2	249.773
12	12C2	10	20	10	ICP-OES	2	208.889

*12C as defined by Rayment, G.E. and David, J. L. in “Soil Chemical Methods-Australasia”

Table 9 Methodology for Total Nitrogen

Lab. Code	Method Reference	Test Method	Measurement Method	Instrument
1	AOAC 990.03	Combustion Digestion Distillation TN=TKN+NO _x	Dumas – High temperature combustion	LECO
6	APHA 22 nd edition 4500 Norg A & D with Jirka Modification-Jirka et al. (1976) and the appropriate Discrete Analyser method	Digestion TN=TKN+NO _x	Colorimetric – phenate method	DA
7	Soil Chem Methods Australasia (Rayment and Lyons) and USEPA Methods 351.2 and 365.3	Digestion	Colorimetric – salicylate method	DA
9	ASTM D2216-98	Digestion TN=TKN+NO _x	Colorimetric – salicylate method	DA
11	In-house method based on APHA 23 rd Edition 4500 – Norg B	Digestion Distillation TN=TKN	Colorimetric – salicylate method	DA
12	In house S4a – Dumas combustion	Combustion	Dumas – High temperature combustion	LECO
13	TKN: APHA 4500 – Norg. A & D, with Jirka Modification 1976; NO _x : This method is based on Thermo Scientific Method D08727 and NEMI (National Environmental Method Index) METHOD ID: 9171	Digestion TN=TKN+NO _x	Colorimetric – phenate method	DA
14	APHA 4500B	Digestion Distillation TN=TKN	Titrimetric	Manual Analysis
15		Combustion	Dumas – High temperature combustion	LECO
16	Inhouse	Digestion TN=TKN	Colorimetric – phenate method	FIA
17		Digestion Distillation TN=TKN+NO _x	Titrimetric Method	Manual Analysis
20		Dumas	Dumas – High temperature combustion	LECO

Table 10 Methodology for Exchangeable Bases

Lab. Code	Method Reference*	Sample Mass (g)	Shake time (hrs)	Extraction Solution	Extraction Solution Vol. (mL)	Additional Information
1	15A1	2	2	1M NH ₄ OAc	20	
6	15A1	2.5	1	1M NH ₄ Cl	50	
9	Rayment and Lyons 15D3 & 15N1	5	1	1M NH ₄ Cl	100	
11	15A1	5	1	1M NH ₄ Cl	100	
12	15A1	1	1	1M NH ₄ Cl	20	
14	In-House	1	1	Silver Thiourea	25	Solubles measured separately and subtracted from Available extract results
17	15A1	2	2	1M NH ₄ Cl	40	
20	15A1	2	1	1M NH ₄ Cl	40	
21	15A1	2	1	1 M NH ₄ Cl	40	

*15A1 as defined by Rayment, G.E. and David, J. L. in “Soil Chemical Methods-Australasia”

3.2 Basis of Participants' Measurement Uncertainty Estimates

Participants were requested to provide information about the basis of their uncertainty estimates:

Table 11 Basis of Uncertainty Estimate

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation ^a		Guide Document for Estimating MU
		Precision	Method Bias	
1	Top Down - precision and estimates of the method and laboratory bias	Control Samples Duplicate Analysis	Laboratory Bias from PT Studies	NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33)
2	Top Down - precision and estimates of the method and laboratory bias	Control Samples Duplicate Analysis Instrument Calibration	CRM	NMI Uncertainty Course
3		Control Samples Duplicate Analysis	CRM	ASTM E2554-13
4	Top Down - precision and estimates of the method and laboratory bias	Control Samples - RM	CRM	NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33)
5	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM Duplicate Analysis	CRM Instrument Calibration Variation in Sample Moisture Content Laboratory Bias from PT Studies Recoveries of SS Standard Purity	NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33)
6	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM Duplicate Analysis	Instrument Calibration	Eurachem/CITAC Guide
7	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration Recoveries of SS	NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33)
8		Control Samples Duplicate Analysis Instrument Calibration	CRM	ASTM E2554-13
9	Top Down - precision and estimates of the method and laboratory bias	Control Samples	Instrument Calibration Variation in Sample Moisture Content	
10	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples - RM	Recoveries of SS	
11	Top Down - precision and estimates of the method and laboratory bias	Control Samples - SS Duplicate Analysis	Recoveries of SS	NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33)
12	Top Down - precision and estimates of the method and laboratory bias	Control Samples - RM Duplicate Analysis	Instrument Calibration Standard Purity	Nordtest Report TR537
13	Bottom Up (ISO/GUM, fish bone/ cause and effect diagram)	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration Laboratory Bias from PT Studies Recoveries of SS	NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33)
14	Top Down Approach	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration Variation in Sample	Eurochem Guide 2007

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation ^a		Guide Document for Estimating MU
		Precision	Method Bias	
			Moisture Content Recoveries of SS	
15	Top Down - precision and estimates of the method and laboratory bias	Standard deviation from PT studies only		NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33)
		Control Samples - RM Duplicate Analysis Instrument Calibration	CRM Laboratory Bias from PT Studies	
16	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples Duplicate Analysis		
17	Top Down - precision and estimates of the method and laboratory bias	Control Samples - RM Duplicate Analysis	Instrument Calibration Variation in Sample Moisture Content Recoveries of SS	Nordtest Report TR537
18	Top Down - precision and estimates of the method and laboratory bias	Control Samples - RM Duplicate Analysis	CRM	NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33)
19	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples - CRM Duplicate Analysis	CRM Instrument Calibration Laboratory Bias from PT Studies	Eurachem/CITAC Guide
20	Top Down - reproducibility (standard deviation) from PT studies used directly	Control Samples - CRM Duplicate Analysis Instrument Calibration	Laboratory Bias from PT Studies	NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33)
21	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM Duplicate Analysis	CRM Recoveries of SS	NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33)
22	Top Down - precision and estimates of the method and laboratory bias	Control Samples - SS	Recoveries of SS	ISO/GUM
23	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM	CRM Recoveries of SS	NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33)

^a RM = Reference Material, CRM = Certified Reference Material, SS = Spiked samples.

3.3 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 12.

Table 12 Participants' Comments

Participants' Comments	Study Co-ordinator's Response
Study was reasonably priced, and just enough time to complete the testing based on our current workloads. Reporting phase fairly lengthy, so extra week was handy. All in all very useful.	Thank you! Your feedback is important to us.
I just wonder whether it is possible to submit the results by two different methods (example: TN and TOC Predicted by MIR and LECO). This will help us to evaluate the accuracy and precision of both methods. Pooling the results from NIR, MIR and LECO may increase the Std of the Mean values.	Yes, you can report more than one set of results from different methods and/or analysts. Please ask the study coordinator for extra lab code numbers in the enrolment form.

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 13 to 71 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 60.

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

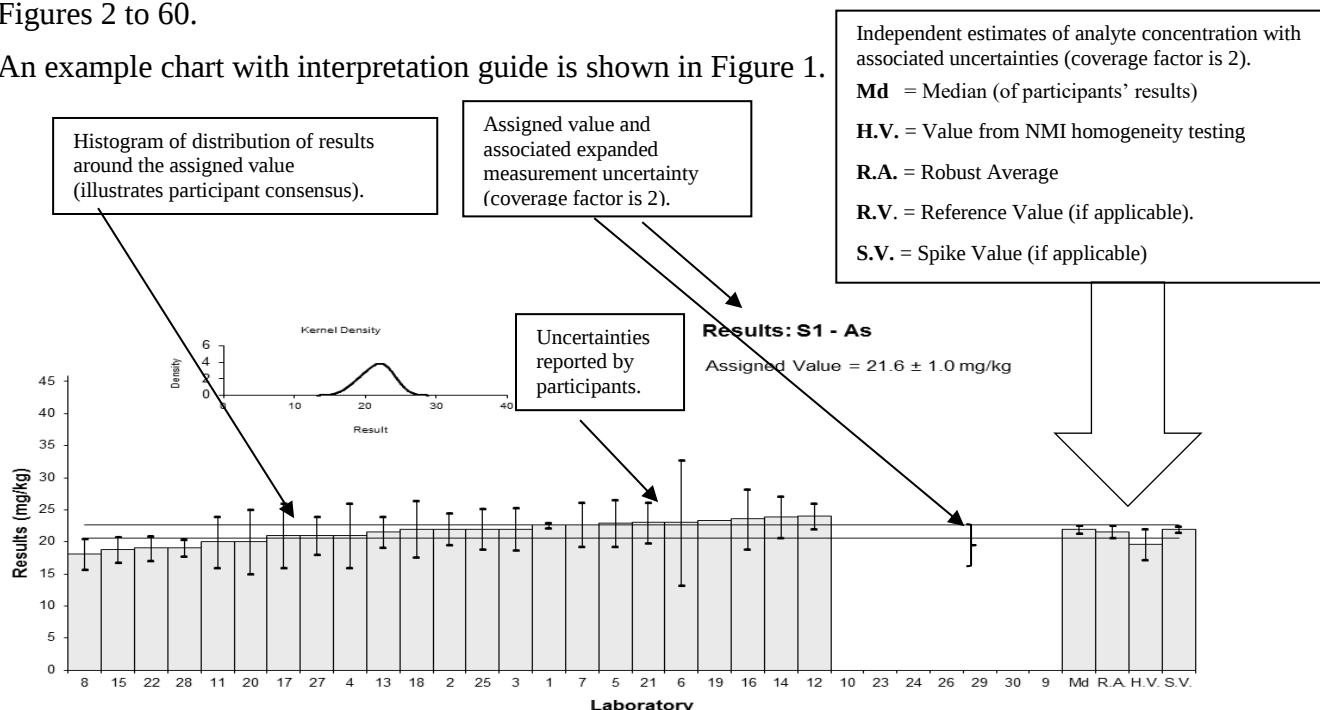


Figure 1 Guide to Presentation of Results

4.2 Assigned Value

An example of the assigned value calculation using data from the present study is given in Appendix 2. The assigned value is defined as: ‘the value attributed to a particular property of a proficiency test item.’¹ In this study the property is the mass fraction of analyte. Assigned values were the robust average of participants’ results; the expanded uncertainties were estimated from the associated robust standard deviations (results less than 50% and greater than 150% of the robust average were removed before the calculation)^{3,4}

4.3 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)’.⁶

4.4 Robust Between-Laboratory Coefficient of Variation

The robust between-laboratory coefficient of variation (robust CV) is a measure of the variability of participants’ results and was calculated using the procedure described in ISO13528:2015(E).⁶

4.5 Target Standard Deviation

The target standard deviation (σ) is the product of the assigned value (X) and the performance coefficient of variation (PCV) as presented in Equation 1. This value is used for calculation of participant z-score and provides scaling for laboratory deviation from the assigned value.

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

It is important to note that the PCV is a fixed value and is not the standard deviation of participants' results. The fixed value set for PCV is based on the existing regulation, the acceptance criteria indicated by the methods, the matrix, the concentration level of analyte and on experience from previous studies. It is backed up by mathematical models such as Thompson Horwitz equation.⁷ By setting a fixed and realistic value for the PCV, the participants' performance does not depend on other participants' performance and can be compared from study to study and against achievable performance.

4.6 z-Score

An example of z-score calculation using data from the present study is given in Appendix 2. For each participants' result a z-score is calculated according to Equation 2 below:

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

where:

- z is z-score
- χ is participants' 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.

4.7 E_n-Score

An example of E_n-score calculation using data from the present study is given in Appendix 2. The E_n-score is complementary to the z-score in assessment of laboratory performance. E_n-score includes measurement uncertainty and is calculated according to Equation 3 below:

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

where:

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

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

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

4.8 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025:2018⁸ 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.⁹

5 TABLES AND FIGURES

Table 13

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	5.84	0.9	-0.67	-0.45
2	6.8	0.4	0.86	1.14
3	6.8	0.95	0.86	0.55
4	6.3	1.1	0.06	0.04
5	6.45	0.97	0.30	0.19
6	5.8	0.9	-0.73	-0.49
7	NT	NT		
8	6	0.87	-0.42	-0.29
9	6	0.80	-0.42	-0.31
10	4.32	0.432	-3.10	-3.89
11	NT	NT		
12	7.02	1.1	1.21	0.67
13	6.3	0.12	0.06	0.14
14	6.0	1.5	-0.42	-0.17
15	<25	NR		
16	6.29	1.26	0.05	0.02
17	5.7	1.1	-0.89	-0.50
18	6.56	0.656	0.48	0.43
19	6.2	1.6	-0.10	-0.04
20	NT	NT		
21	6.7	0.9	0.70	0.47
22	5.8	1.16	-0.73	-0.39
23	6.2	NR	-0.10	-0.24

Statistics*

Assigned Value	6.26	0.25
Spike	7.01	0.40
Homogeneity Value	6.57	0.79
Robust Average	6.26	0.25
Median	6.25	0.21
Mean	6.26	
N	18	
Max.	7.02	
Min.	5.7	
Robust SD	0.43	
Robust CV	6.9%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

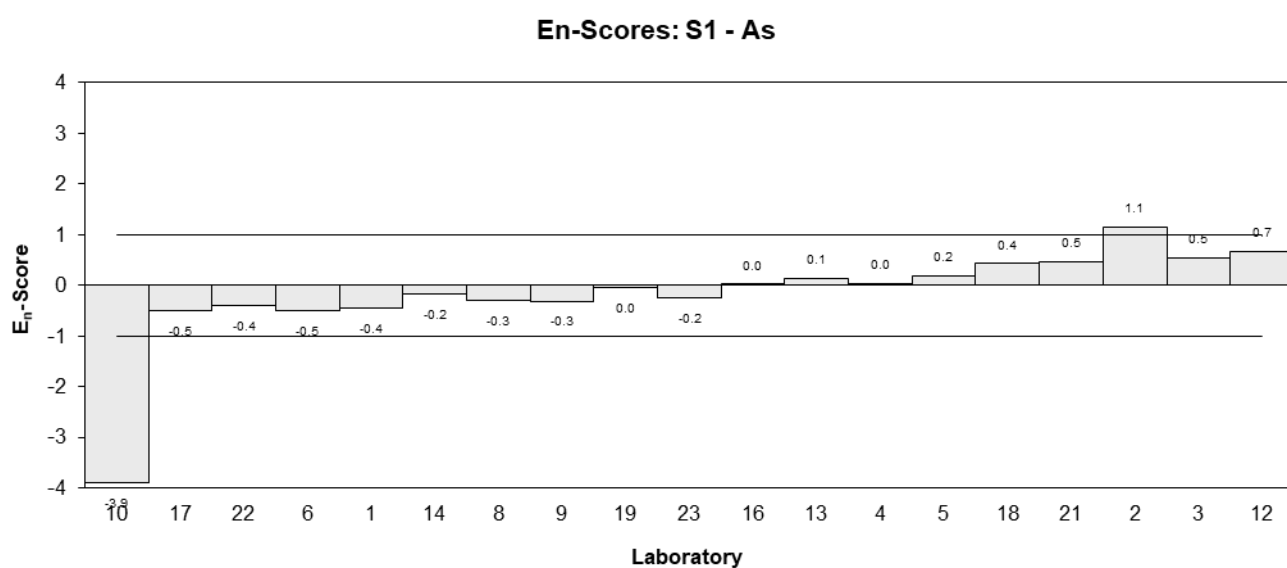
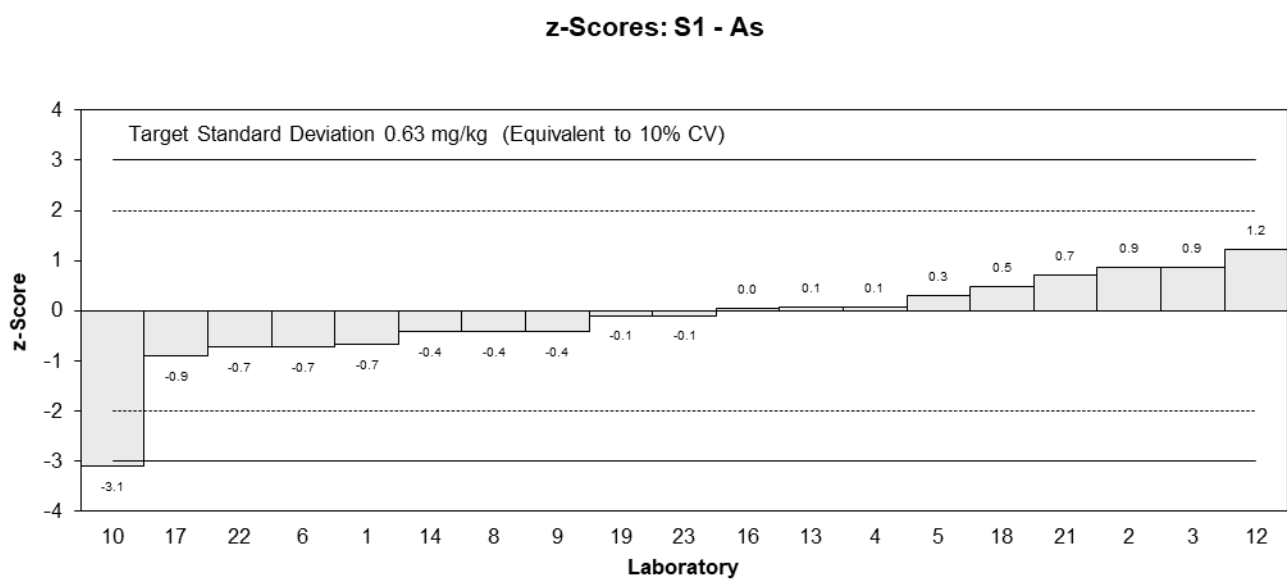
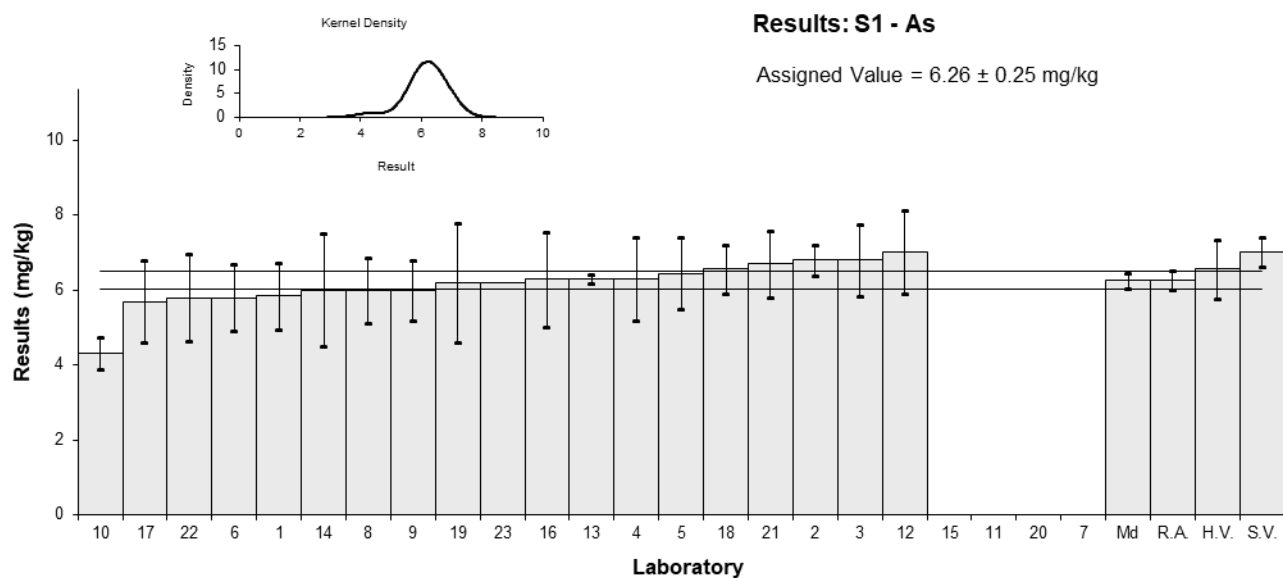


Figure 2

Table 14

Sample Details

Sample No.	S1
Matrix.	Sediment
Analyte.	B
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	6.16	0.9
2	9.7	0.97
3	< 10	NR
4	NT	NT
5	NT	NT
6	<50	NR
7	NT	NT
8	<10	2
9	<5	5
10	3.73	0.373
11	NT	NT
12	8.12	1.5
13	<50	32
14	4.8	1.4
15	<5	NR
16	5.55	1.17
17	7.0	1.4
18	4.95	0.495
19	< 20	3.6
20	NT	NT
21	NT	NT
22	<10	2
23	14	NR

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	5.53	0.66
Robust Average	7.0	2.1
Median	6.6	2.0
Mean	7.5	
N	8	
Max.	14	
Min.	4.8	
Robust SD	2.3	
Robust CV	33%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

Results: S1 - B

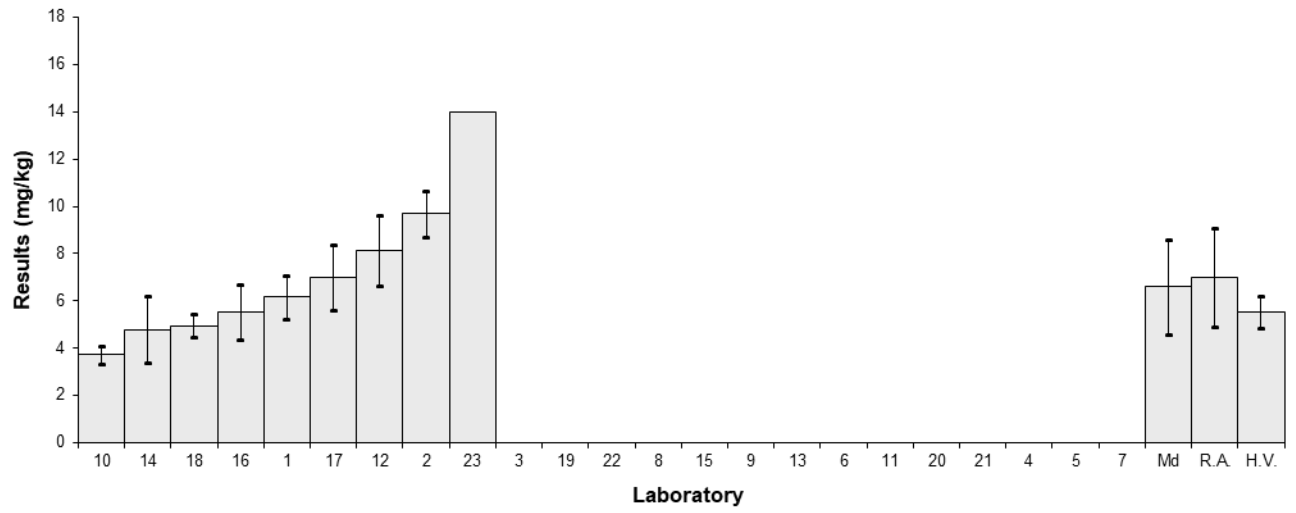


Figure 3

Table 15

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	79.2	12	-0.20	-0.12
2	92	6	1.39	1.41
3	73	9.5	-0.97	-0.72
4	NT	NT		
5	74.79	11.22	-0.74	-0.49
6	70.0	12.0	-1.34	-0.83
7	NT	NT		
8	86	13	0.64	0.37
9	86	10.58	0.64	0.44
10	142	14.2	7.57	4.05
11	NT	NT		
12	82.1	8.5	0.16	0.13
13	73	5.96	-0.97	-0.99
14	76	21	-0.59	-0.22
15	89	6	1.01	1.03
16	75.4	15.1	-0.67	-0.34
17	73	15	-0.97	-0.49
18	114	11.4	4.11	2.65
19	84.0	5.1	0.40	0.44
20	NT	NT		
21	98	17.6	2.13	0.94
22	76	15.2	-0.59	-0.30
23	77	NR	-0.47	-0.73

Statistics*

Assigned Value	80.8	5.2
Spike	Not Spiked	
Homogeneity Value	75.9	9.1
Robust Average	80.8	5.2
Median	78.1	3.8
Mean	82.1	
N	18	
Max.	114	
Min.	70	
Robust SD	8.9	
Robust CV	11%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

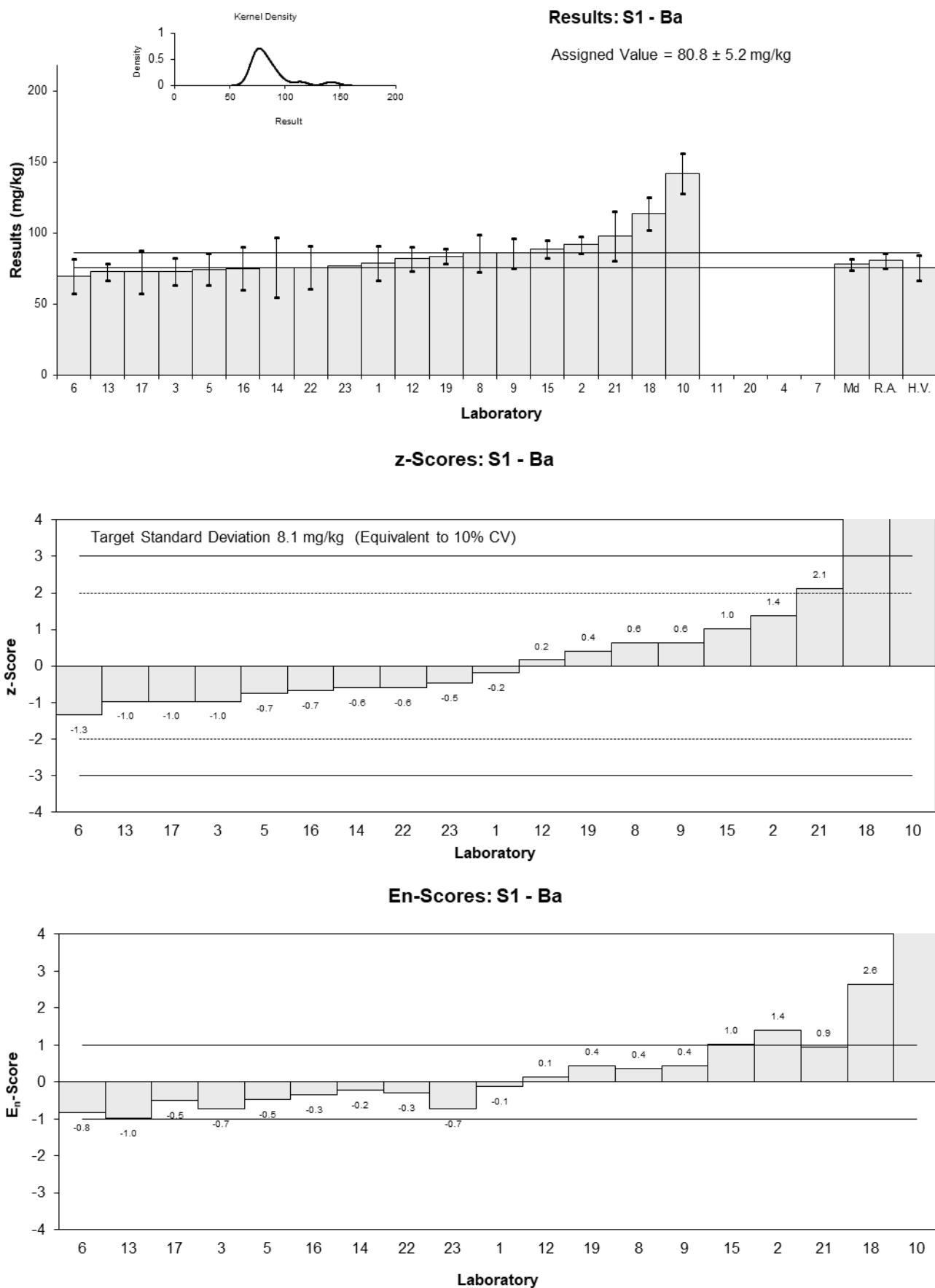


Figure 4

Table 16

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.62	0.1	0.19	0.15
2	0.76	0.3	1.74	0.52
3	< 2	NR		
4	NT	NT		
5	0.61	0.09	0.08	0.07
6	0.5	0.1	-1.14	-0.92
7	NT	NT		
8	<2	0.4		
9	0.6	0.3	-0.03	-0.01
10	0.73	0.073	1.40	1.44
11	NT	NT		
12	0.71	0.1	1.18	0.96
13	0.6	0.04	-0.03	-0.05
14	NT	NT		
15	<1	NR		
16	0.596	0.131	-0.08	-0.05
17	0.56	0.11	-0.48	-0.36
18	0.63	0.063	0.30	0.34
19	0.59	0.17	-0.14	-0.07
20	NT	NT		
21	0.61	0.06	0.08	0.09
22	<2	0.4		
23	0.5	NR	-1.14	-2.10

Statistics*

Assigned Value	0.603	0.049
Spike	Not Spiked	
Homogeneity Value	0.578	0.069
Robust Average	0.603	0.049
Median	0.600	0.018
Mean	0.607	
N	13	
Max.	0.76	
Min.	0.5	
Robust SD	0.071	
Robust CV	12%	

**Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

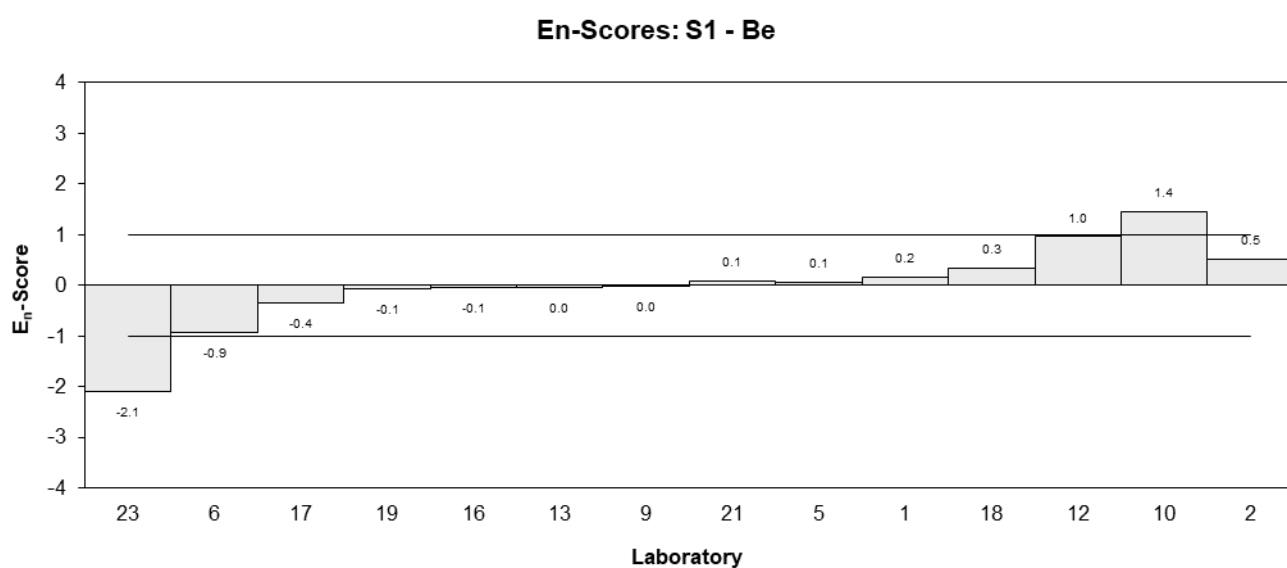
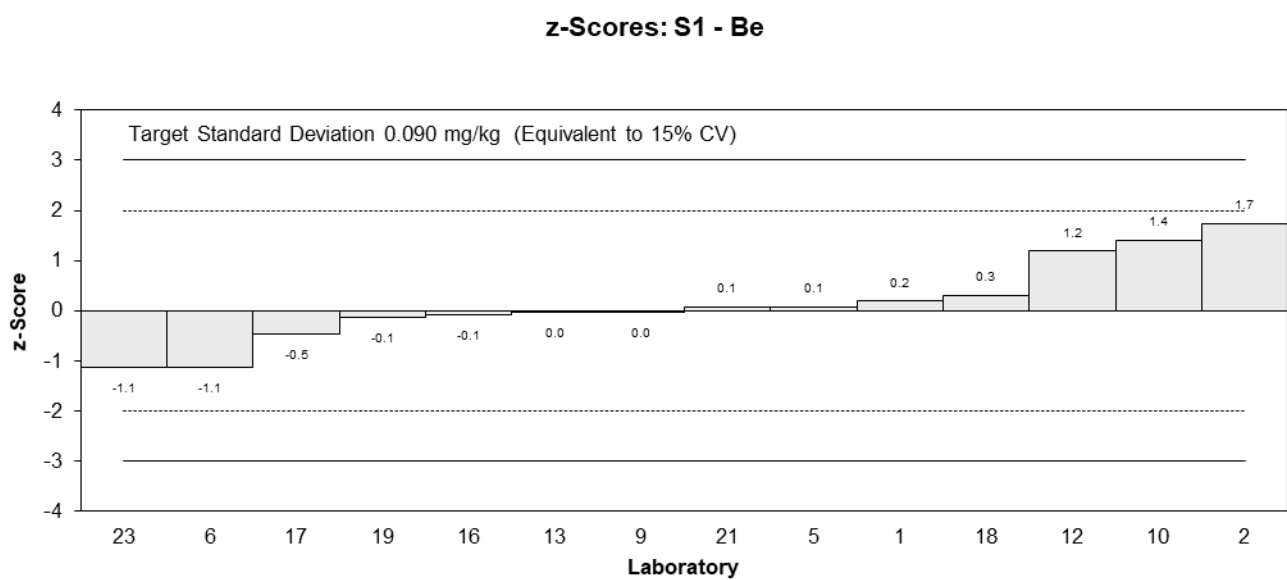
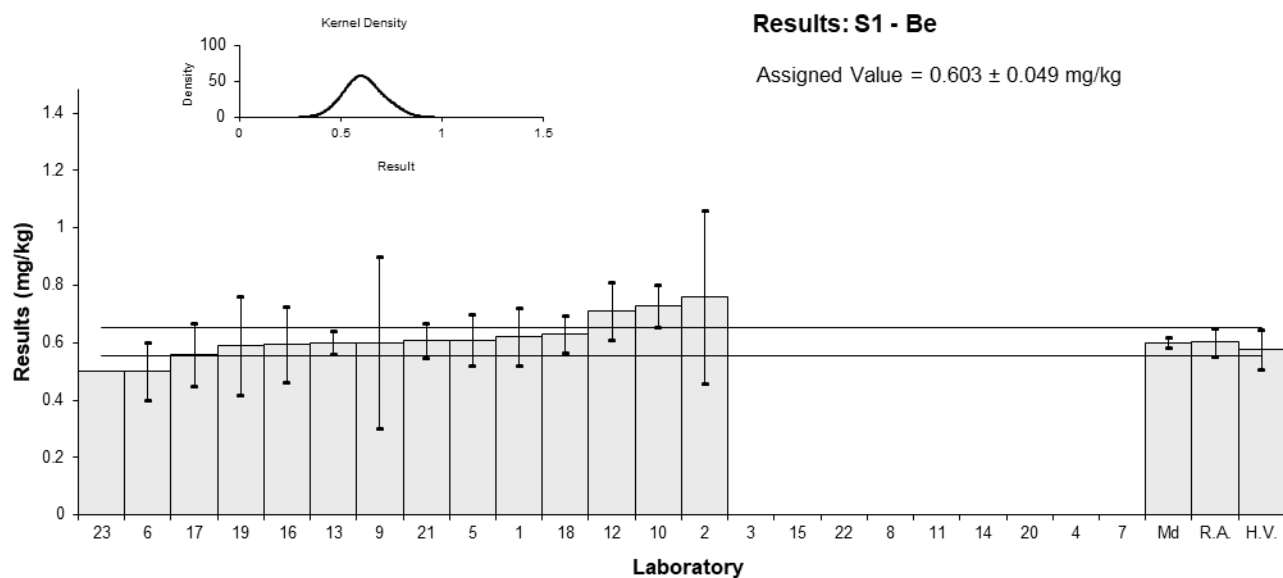


Figure 5

Table 17

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	2.19	0.3	1.12	0.71
2	1.9	0.2	-0.36	-0.32
3	2.0	0.24	0.15	0.12
4	1.9	0.44	-0.36	-0.16
5	2.31	0.35	1.73	0.95
6	1.9	0.2	-0.36	-0.32
7	NT	NT		
8	2	0.27	0.15	0.11
9	1.9	0.28	-0.36	-0.24
10	0.79	0.079	-5.99	-10.50
11	NT	NT		
12	2.02	0.3	0.25	0.16
13	2	0.249	0.15	0.11
14	1.8	0.4	-0.86	-0.42
15	1.8	0.4	-0.86	-0.42
16	2.01	0.43	0.20	0.09
17	1.8	0.36	-0.86	-0.46
18	1.95	0.195	-0.10	-0.09
19	2.07	0.30	0.51	0.32
20	NT	NT		
21	2.2	0.1	1.17	1.80
22	1.8	0.36	-0.86	-0.46
23	2.0	NR	0.15	0.38

Statistics*

Assigned Value	1.97	0.08
Spike	1.96	0.08
Homogeneity Value	2.10	0.25
Robust Average	1.97	0.08
Median	2.00	0.07
Mean	1.98	
N	19	
Max.	2.31	
Min.	1.8	
Robust SD	0.15	
Robust CV	7.4%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

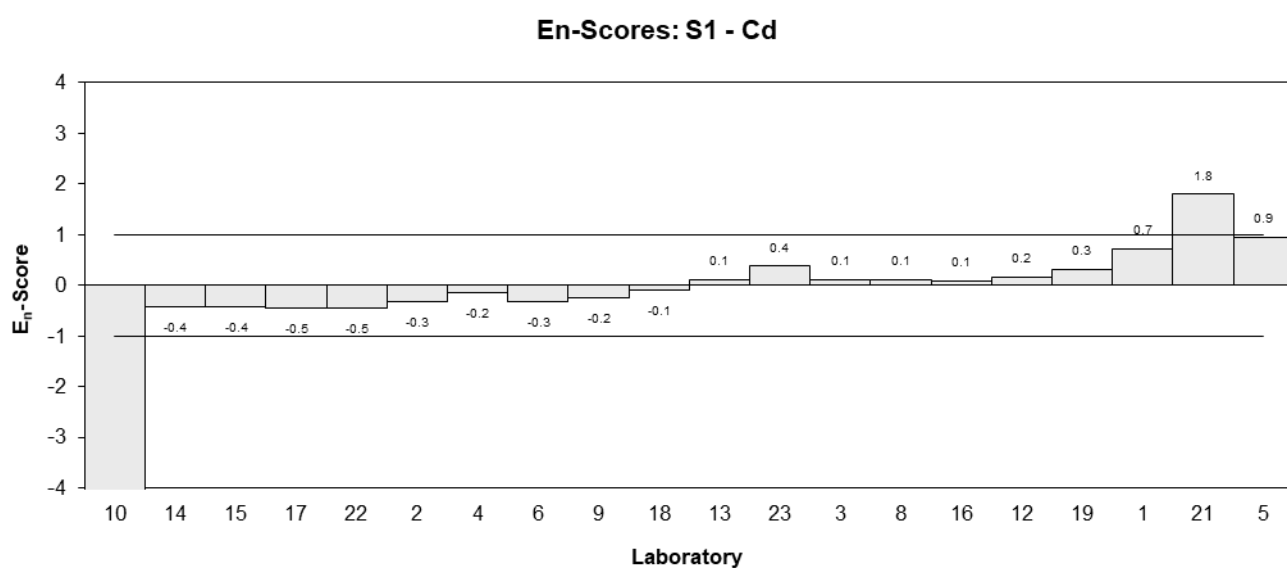
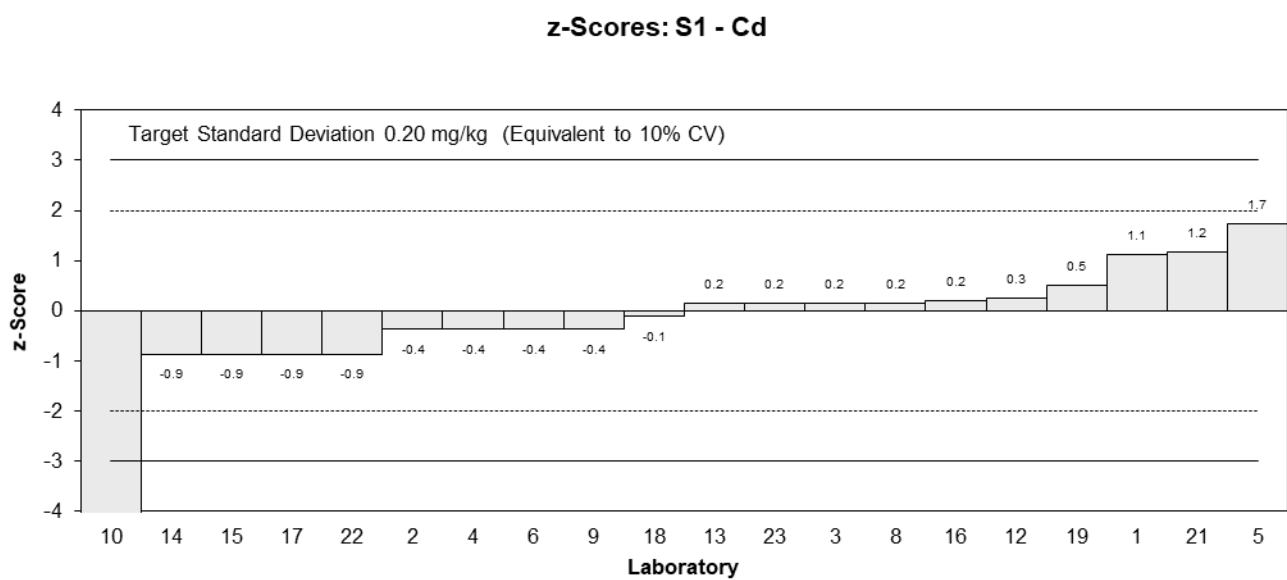
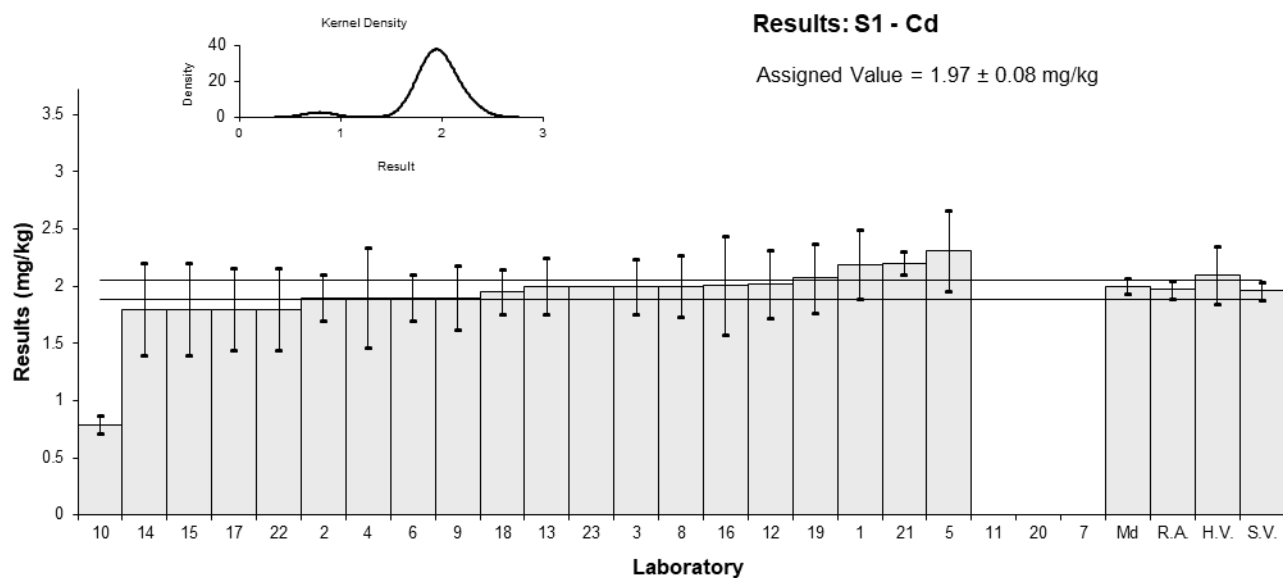


Figure 6

Table 18

Sample Details

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

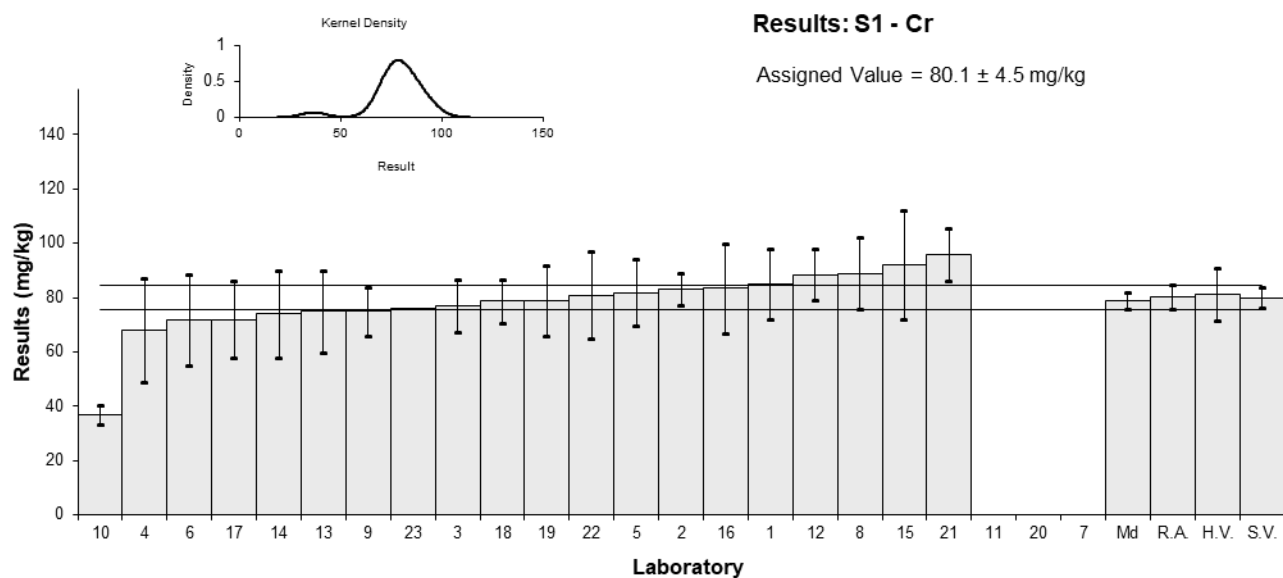
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	85.2	13	0.64	0.37
2	83	6	0.36	0.39
3	77	9.6	-0.39	-0.29
4	68	19	-1.51	-0.62
5	81.75	12.26	0.21	0.13
6	71.7	16.8	-1.05	-0.48
7	NT	NT		
8	89	13	1.11	0.65
9	75	8.93	-0.64	-0.51
10	36.9	3.69	-5.39	-7.42
11	NT	NT		
12	88.5	9.5	1.05	0.80
13	75	15.08	-0.64	-0.32
14	74	16	-0.76	-0.37
15	92	20	1.49	0.58
16	83.4	16.7	0.41	0.19
17	72	14	-1.01	-0.55
18	78.7	7.87	-0.17	-0.15
19	79	13	-0.14	-0.08
20	NT	NT		
21	96	9.6	1.99	1.50
22	81	16.2	0.11	0.05
23	76	NR	-0.51	-0.91

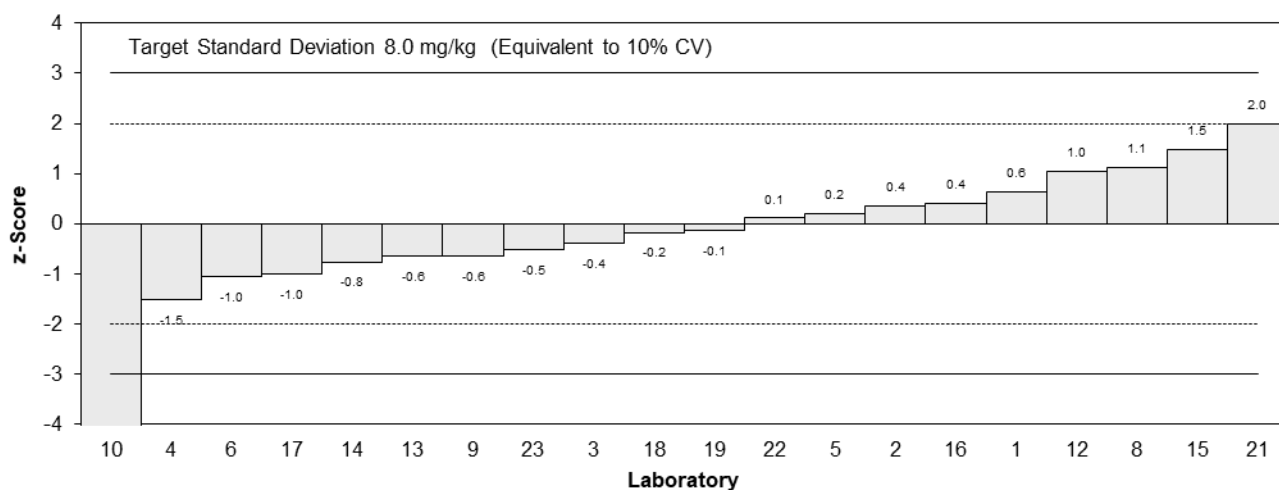
Statistics*

Assigned Value	80.1	4.5
Spike	80.0	3.7
Homogeneity Value	81.2	9.7
Robust Average	80.1	4.5
Median	79.0	3.1
Mean	80.3	
N	19	
Max.	96	
Min.	68	
Robust SD	7.9	
Robust CV	9.9%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.



z-Scores: S1 - Cr



En-Scores: S1 - Cr

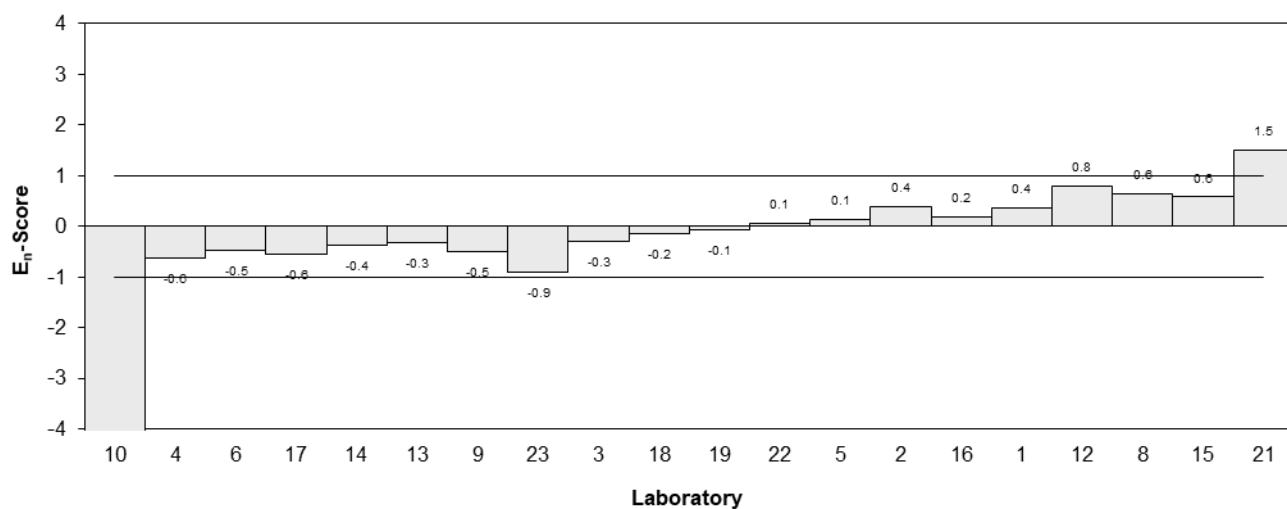


Figure 7

Table 19

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	68.3	10	0.03	0.02
2	57	3	-1.63	-2.94
3	66	8.7	-0.31	-0.23
4	67	16	-0.16	-0.07
5	68.09	10.21	0.00	0.00
6	67.3	10.7	-0.12	-0.07
7	NT	NT		
8	66	11	-0.31	-0.19
9	67	8.38	-0.16	-0.13
10	170	17	14.96	5.94
11	NT	NT		
12	74.1	8.0	0.88	0.72
13	67	9.091	-0.16	-0.12
14	68	14	-0.01	-0.01
15	84	17	2.33	0.93
16	68.8	13.8	0.10	0.05
17	64	13	-0.60	-0.31
18	65.5	6.55	-0.38	-0.37
19	66.8	9.2	-0.19	-0.14
20	NT	NT		
21	75	10.5	1.01	0.64
22	65	13	-0.46	-0.23
23	78	NR	1.45	4.30

Statistics*

Assigned Value	68.1	2.3
Spike	70.5	2.6
Homogeneity Value	66.8	8.0
Robust Average	68.1	2.3
Median	67.0	0.9
Mean	68.6	
N	19	
Max.	84	
Min.	57	
Robust SD	4.1	
Robust CV	6.0%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

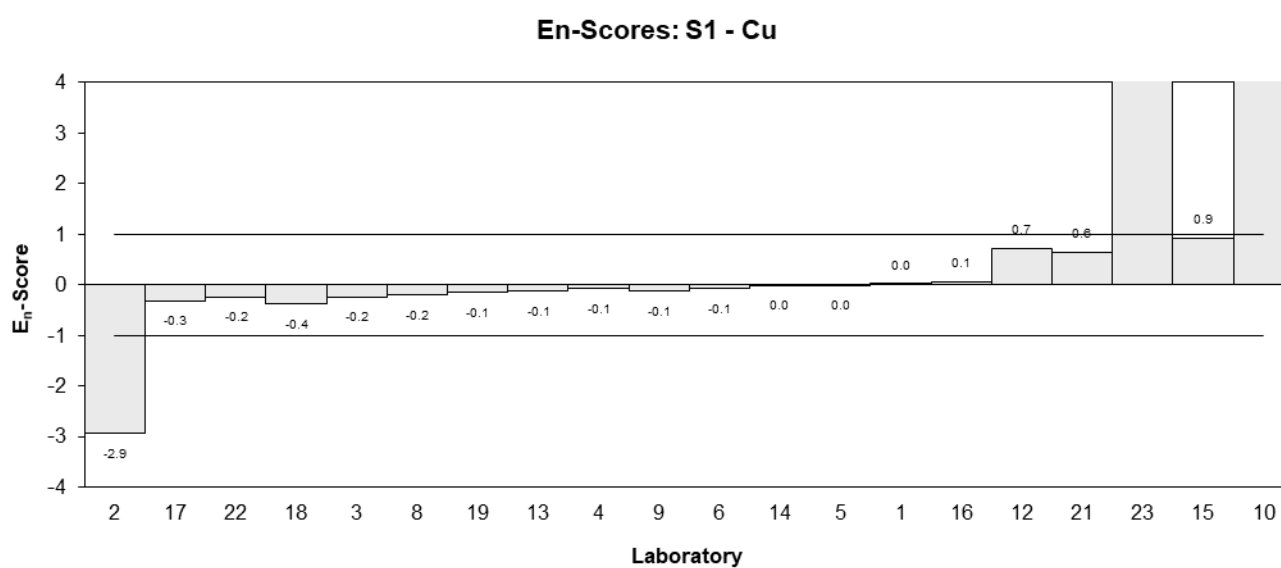
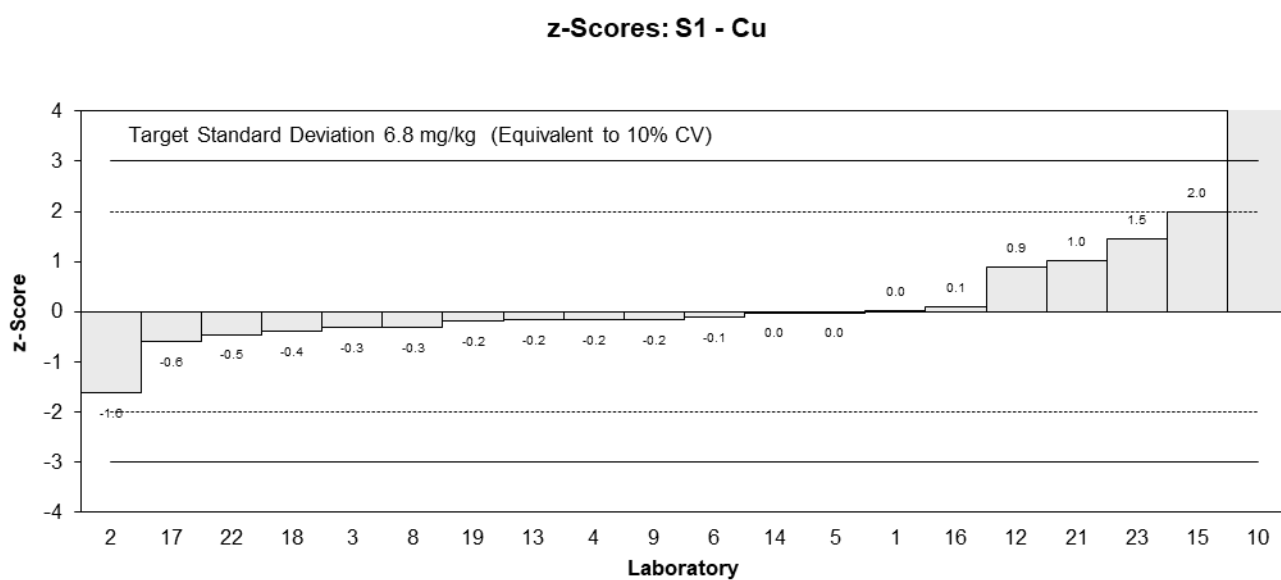
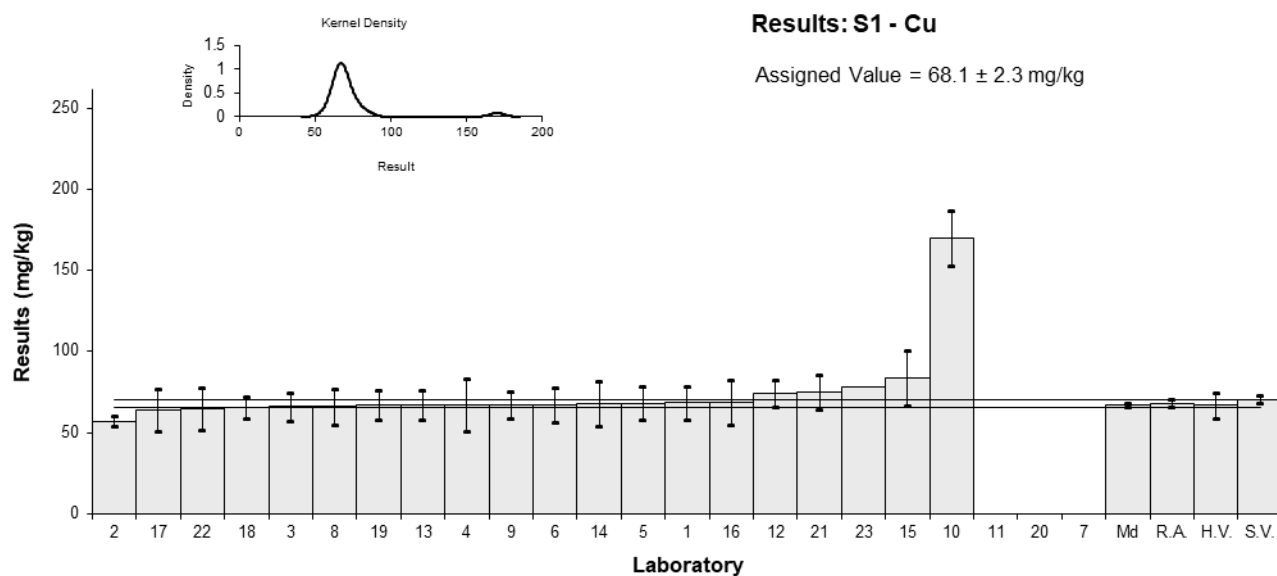


Figure 8

Table 20

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.73	0.1	0.91	0.76
2	0.61	0.03	-0.33	-0.49
3	0.65	0.08	0.08	0.08
4	0.78	0.24	1.43	0.56
5	0.68	0.10	0.39	0.33
6	0.6	0.1	-0.44	-0.36
7	NT	NT		
8	0.66	0.11	0.19	0.14
9	0.79	0.13	1.54	1.04
10	0.57	0.057	-0.75	-0.89
11	NT	NT		
12	0.71	0.1	0.71	0.59
13	0.6	0.1	-0.44	-0.36
14	0.40	0.08	-2.51	-2.45
15	0.57	0.1	-0.75	-0.62
16	0.633	0.083	-0.09	-0.09
17	0.61	0.12	-0.33	-0.24
18	0.755	0.076	1.17	1.18
19	0.62	0.12	-0.23	-0.17
20	NT	NT		
21	0.5	0.015	-1.47	-2.37
22	0.7	0.14	0.60	0.38
23	0.515	NR	-1.32	-2.19

Statistics*

Assigned Value	0.642	0.058
Spike	0.703	0.014
Homogeneity Value	0.640	0.077
Robust Average	0.642	0.058
Median	0.633	0.045
Mean	0.638	
N	19	
Max.	0.79	
Min.	0.4	
Robust SD	0.10	
Robust CV	16%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

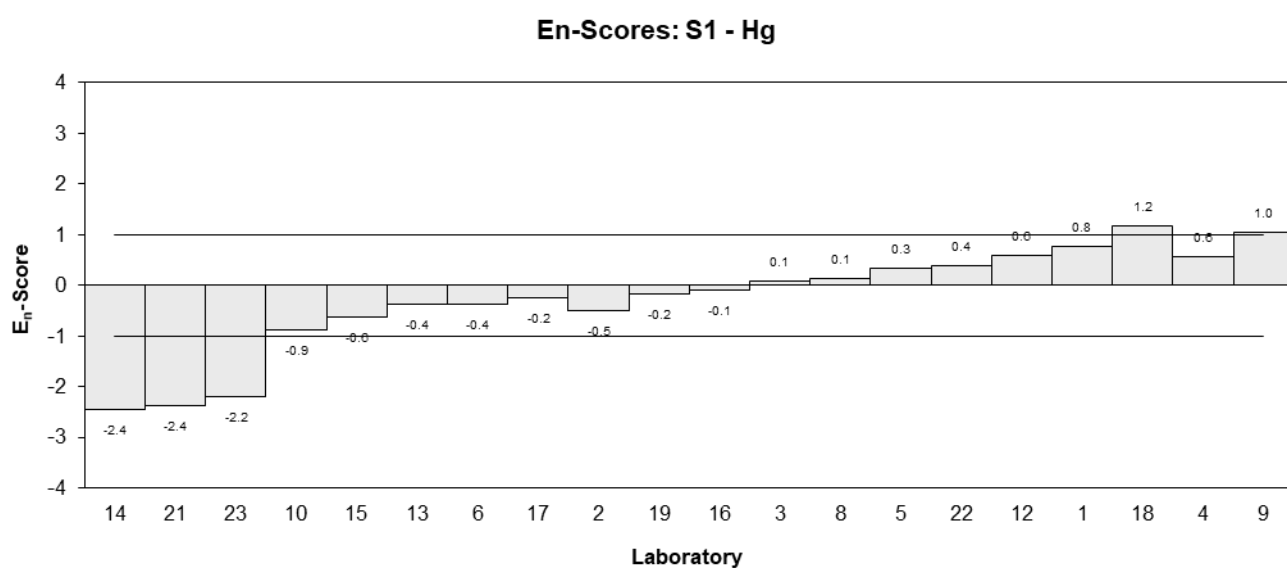
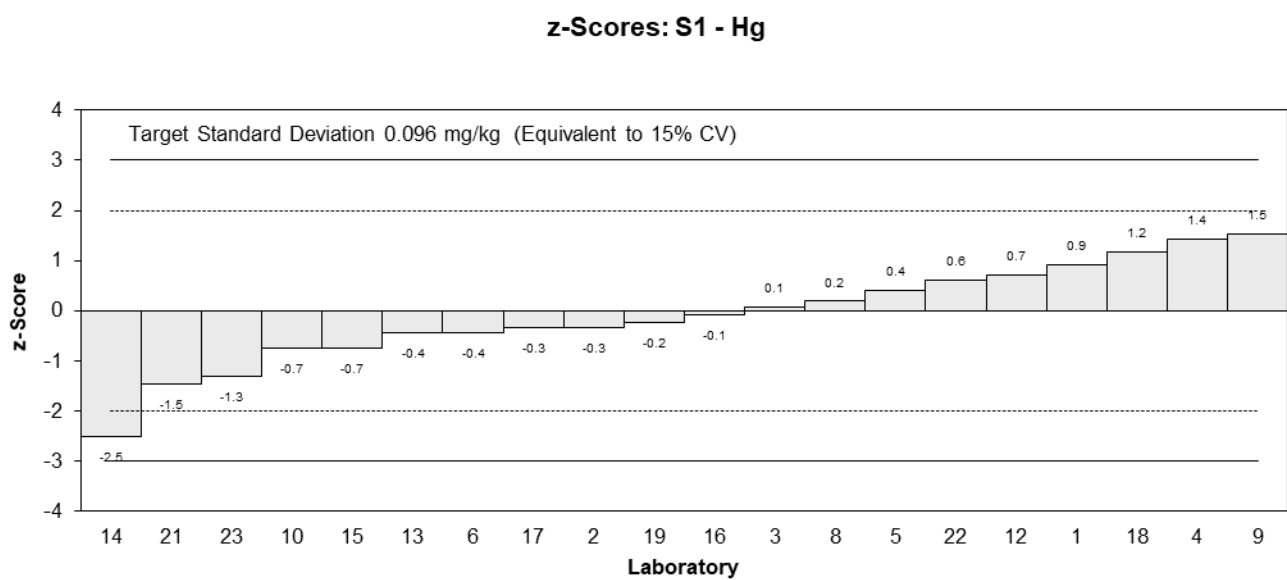
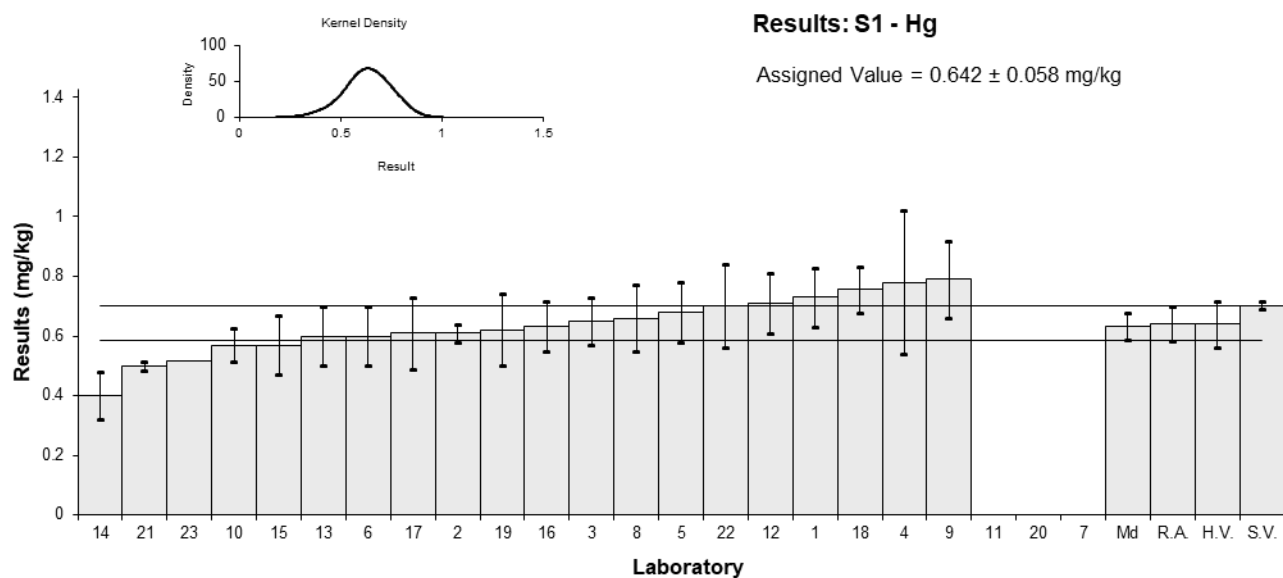


Figure 9

Table 21

Sample Details

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

Participant Results

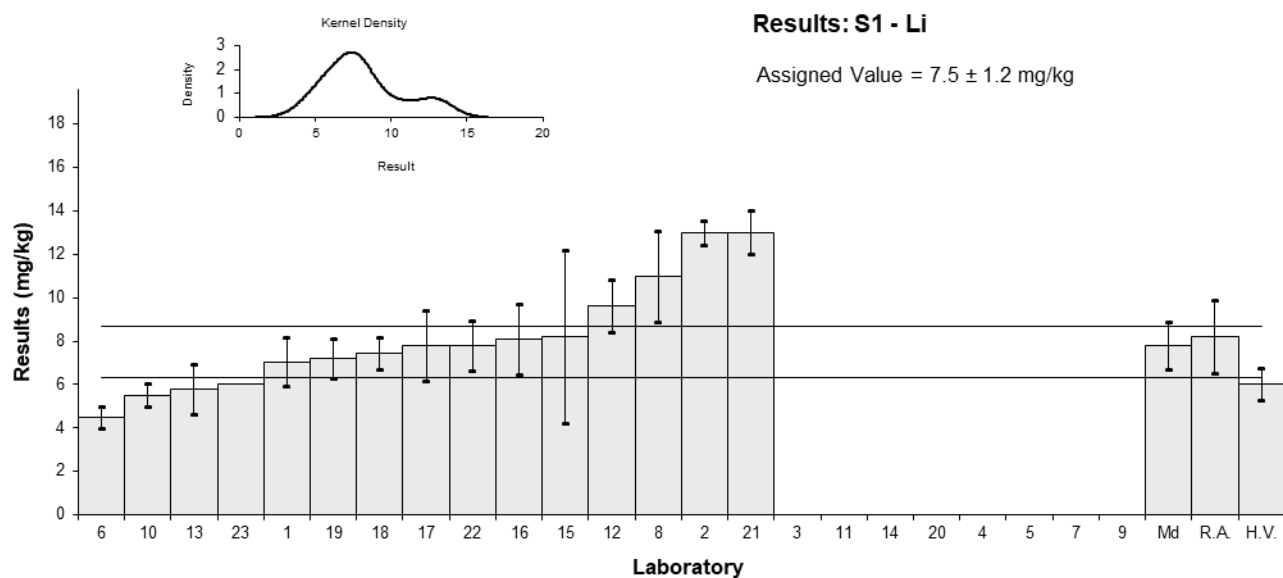
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	7.05	1.1	-0.30	-0.28
2	13	0.55	3.67	4.17
3	NR	NR		
4	NT	NT		
5	NT	NT		
6	4.5	0.5	-2.00	-2.31
7	NT	NT		
8	11	2.1	2.33	1.45
9	NT	NT		
10	5.52	0.552	-1.32	-1.50
11	NT	NT		
12	9.61	1.2	1.41	1.24
13	5.8	1.16	-1.13	-1.02
14	NT	NT		
15	8.2	4	0.47	0.17
16	8.08	1.62	0.39	0.29
17	7.8	1.6	0.20	0.15
18	7.46	0.746	-0.03	-0.03
19	7.19	0.91	-0.21	-0.21
20	NT	NT		
21	13	1	3.67	3.52
22	7.8	1.17	0.20	0.18
23	6.0	NR	-1.00	-1.25

Statistics*

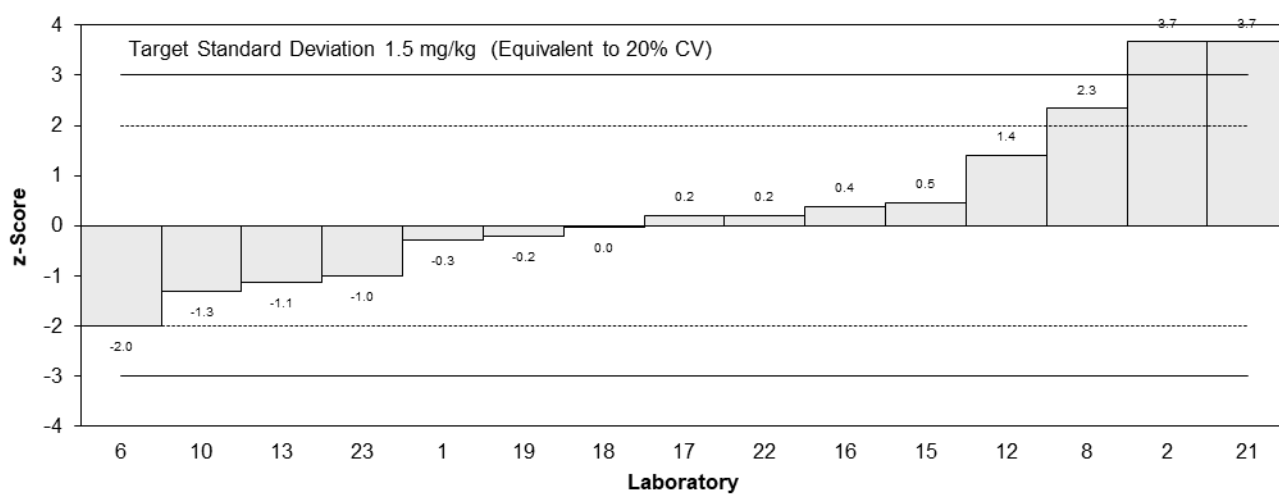
Assigned Value**	7.5	1.2
Spike	Not Spiked	
Homogeneity Value	6.03	0.72
Robust Average	8.2	1.7
Median	7.8	1.1
Mean	8.3	
N	14	
Max.	13	
Min.	4.5	
Robust SD	2.6	
Robust CV	31%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

**Robust Average excluding Laboratories 2 and 21.



z-Scores: S1 - Li



En-Scores: S1 - Li

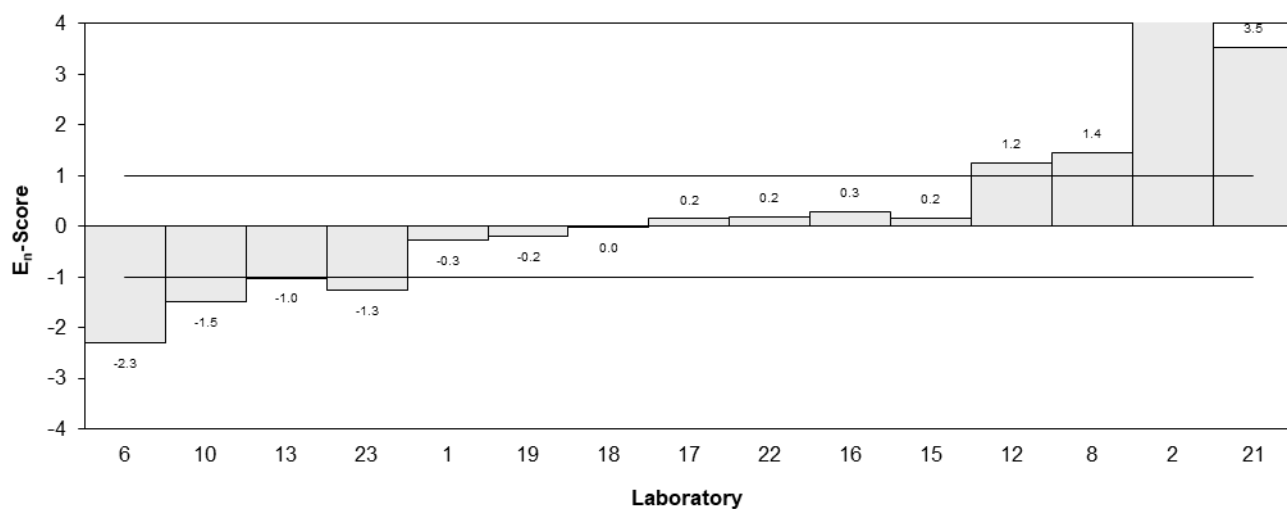


Figure 10

Table 22

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	273	41	0.66	0.40
2	232	15	-0.94	-1.21
3	240	29	-0.62	-0.50
4	NT	NT		
5	277.36	41.60	0.83	0.49
6	231	39	-0.98	-0.61
7	NT	NT		
8	260	38	0.16	0.10
9	250	28	-0.23	-0.19
10	524	52.4	10.47	4.96
11	NT	NT		
12	261	25	0.20	0.18
13	280	43.73	0.94	0.53
14	240	45	-0.62	-0.34
15	278	40	0.86	0.52
16	267	54	0.43	0.20
17	220	44	-1.41	-0.78
18	260	26.0	0.16	0.14
19	269	27	0.51	0.43
20	NT	NT		
21	280	42	0.94	0.55
22	250	37.5	-0.23	-0.15
23	229	NR	-1.05	-2.08

Statistics*

Assigned Value	256	13
Spike	Not Spiked	
Homogeneity Value	217	26
Robust Average	256	13
Median	260	13
Mean	255	
N	18	
Max.	280	
Min.	220	
Robust SD	22	
Robust CV	8.6%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

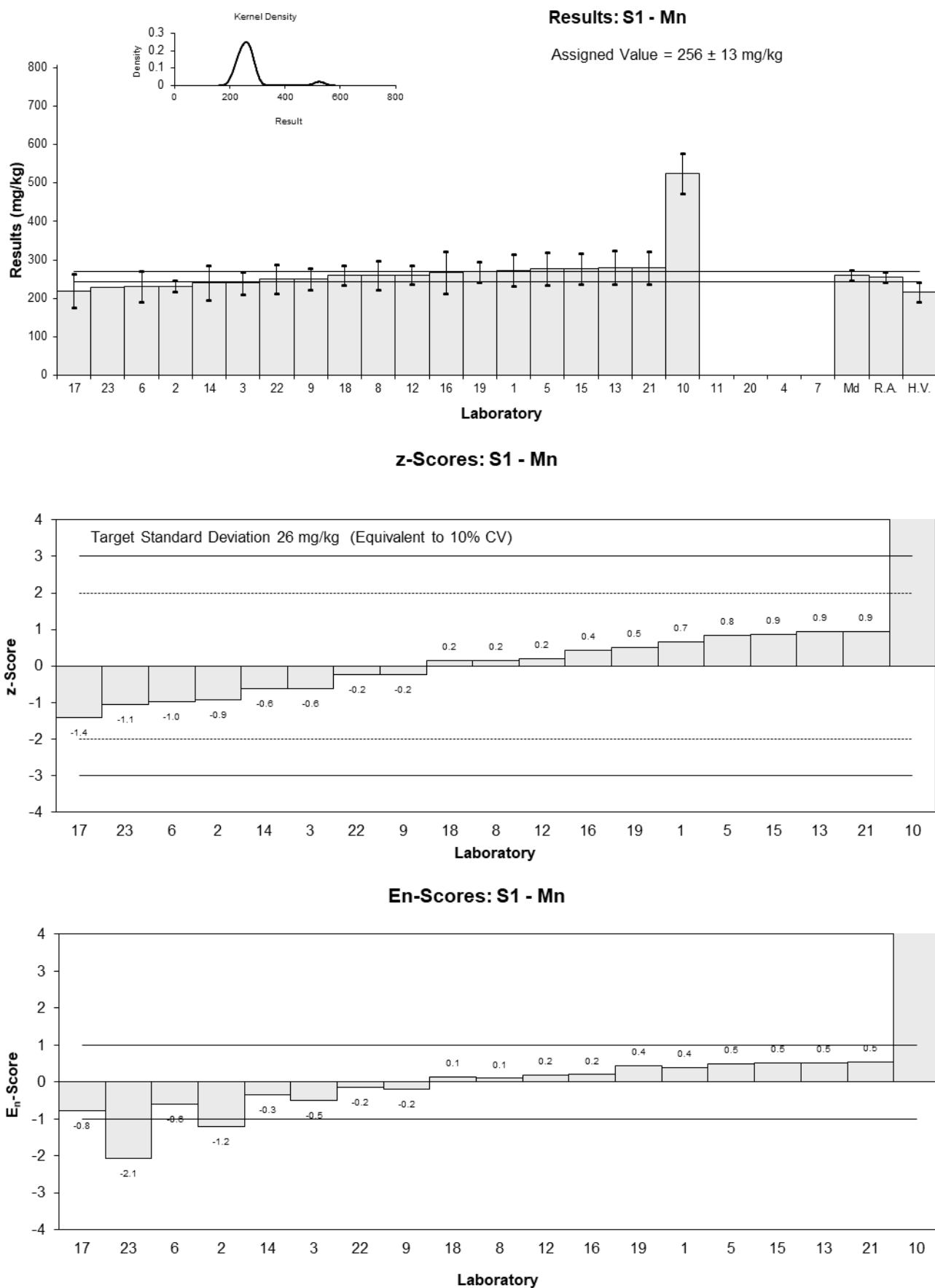


Figure 11

Table 23

Sample Details

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

Participant Results

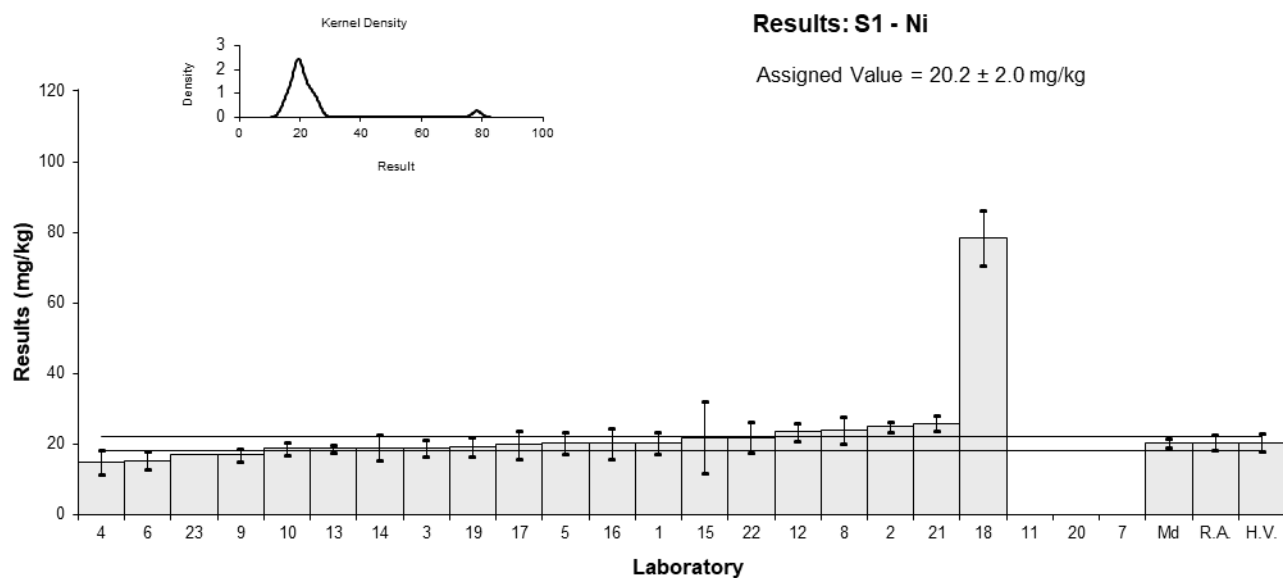
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	20.4	3.1	0.10	0.05
2	25	1.5	2.38	1.92
3	19	2.3	-0.59	-0.39
4	15	3.4	-2.57	-1.32
5	20.29	3.04	0.04	0.02
6	15.4	2.5	-2.38	-1.50
7	NT	NT		
8	24	3.9	1.88	0.87
9	17	1.84	-1.58	-1.18
10	18.8	1.88	-0.69	-0.51
11	NT	NT		
12	23.5	2.5	1.63	1.03
13	18.8	1.21	-0.69	-0.60
14	19	3.6	-0.59	-0.29
15	22	10	0.89	0.18
16	20.3	4.3	0.05	0.02
17	20	4.0	-0.10	-0.04
18	78.31	7.83	28.77	7.19
19	19.2	2.8	-0.50	-0.29
20	NT	NT		
21	26	2.3	2.87	1.90
22	22	4.4	0.89	0.37
23	17	NR	-1.58	-1.60

Statistics*

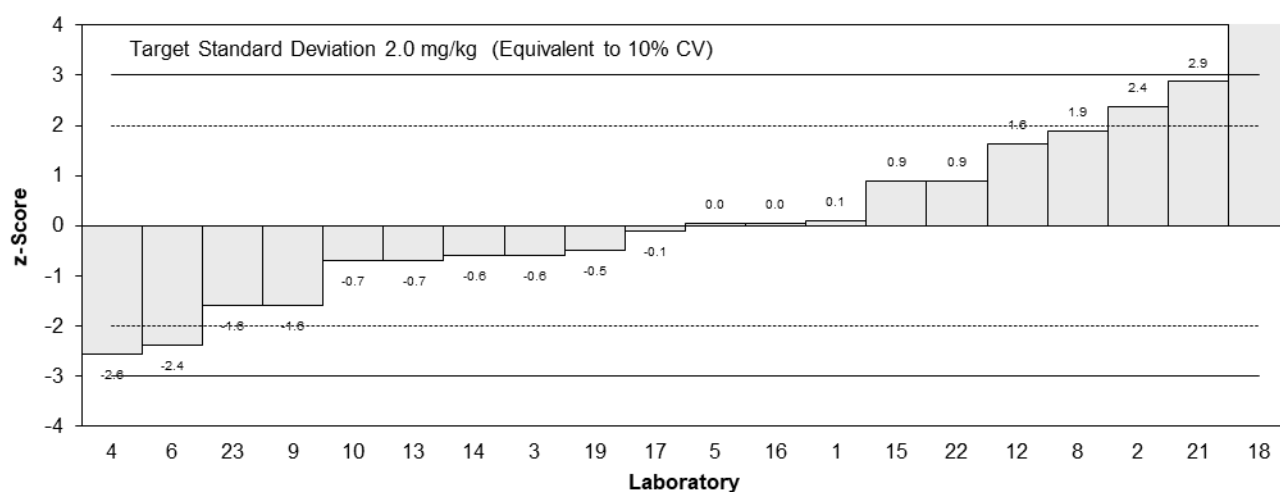
Assigned Value**	20.2	2.0
Spike	Not Spiked	
Homogeneity Value	20.5	2.5
Robust Average	20.5	2.2
Median	20.3	1.2
Mean	23.3	
N	19	
Max.	78.31	
Min.	15	
Robust SD	3.8	
Robust CV	18%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

**Robust Average excluding Laboratory 18.



z-Scores: S1 - Ni



En-Scores: S1 - Ni

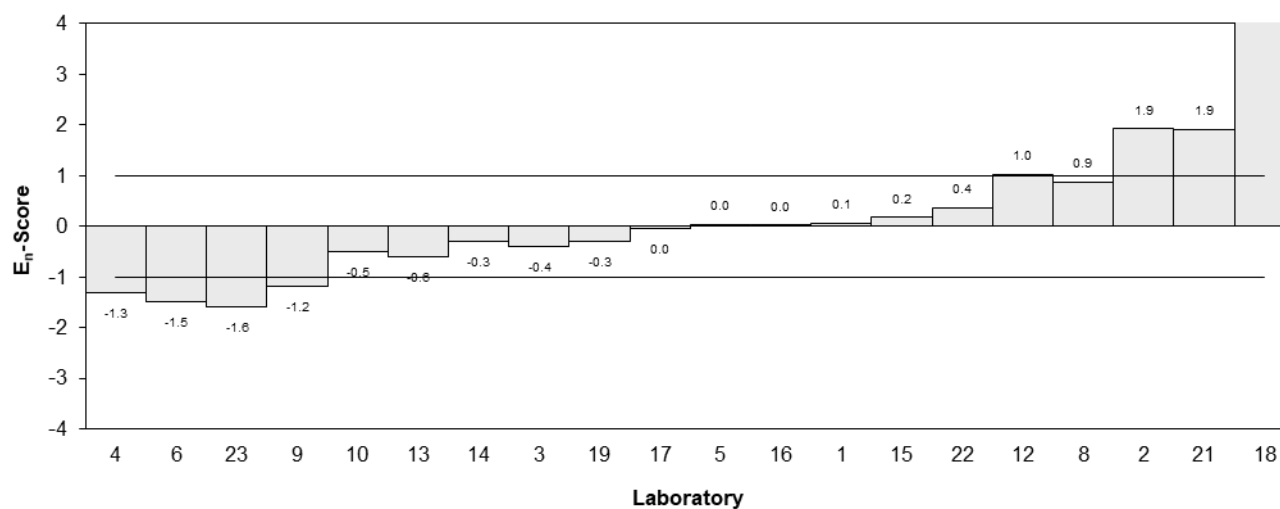


Figure 12

Table 24

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	41.9	6.3	0.72	0.43
2	39	2	-0.03	-0.04
3	39	5.1	-0.03	-0.02
4	33	6.3	-1.56	-0.93
5	40.98	6.15	0.48	0.29
6	42.7	8.2	0.92	0.43
7	NT	NT		
8	39	6	-0.03	-0.02
9	40	5.44	0.23	0.16
10	35.9	3.59	-0.82	-0.81
11	NT	NT		
12	39.3	4.5	0.05	0.04
13	38	5.959	-0.28	-0.18
14	36	7.5	-0.79	-0.40
15	44	10	1.25	0.48
16	40.0	8.0	0.23	0.11
17	35	7.0	-1.05	-0.57
18	36.57	3.66	-0.65	-0.63
19	41.0	6.2	0.49	0.30
20	NT	NT		
21	43	4.3	1.00	0.84
22	37	7.4	-0.54	-0.28
23	37	NR	-0.54	-1.24

Statistics*

Assigned Value	39.1	1.7
Spike	40.0	2.3
Homogeneity Value	33.8	4.1
Robust Average	39.1	1.7
Median	39.0	1.4
Mean	39.1	
N	19	
Max.	44	
Min.	33	
Robust SD	3.0	
Robust CV	7.8%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

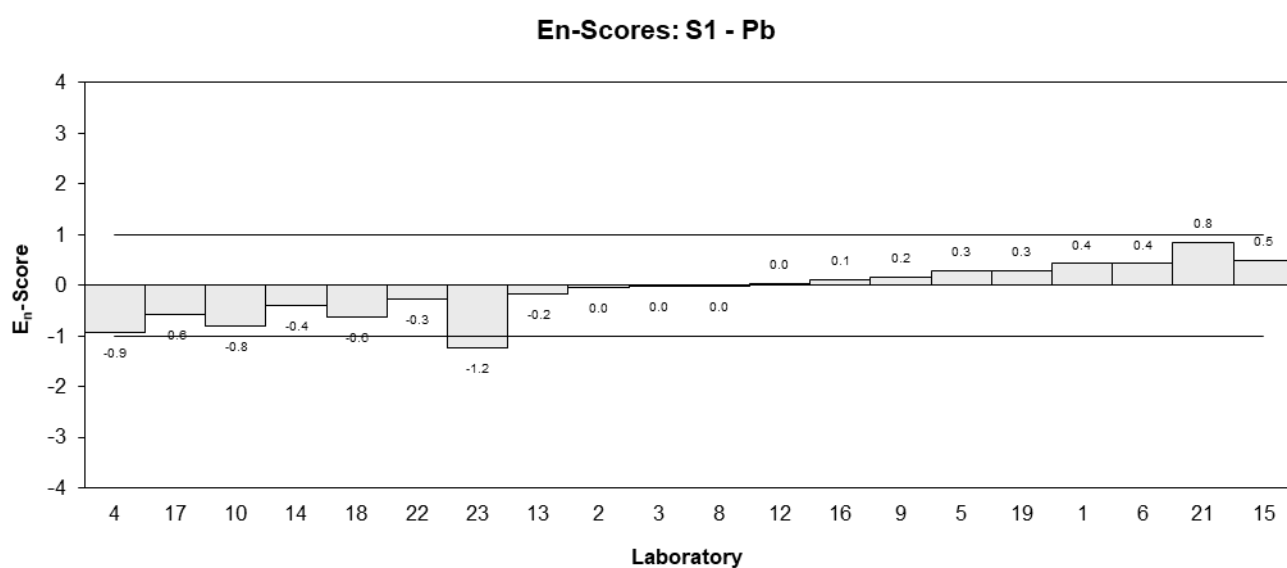
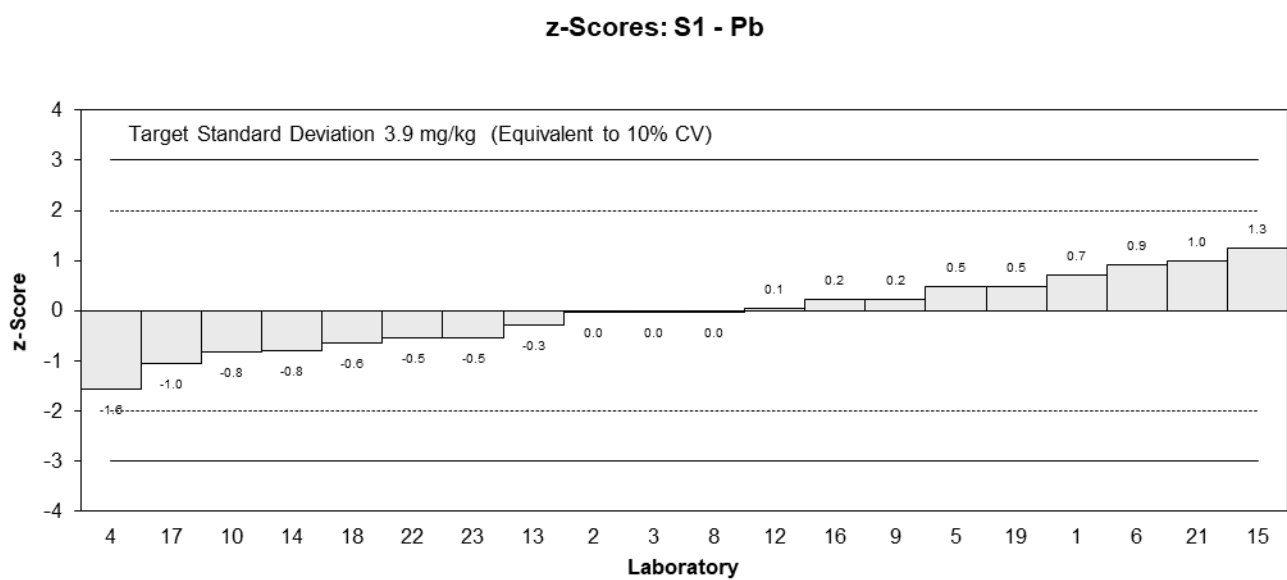
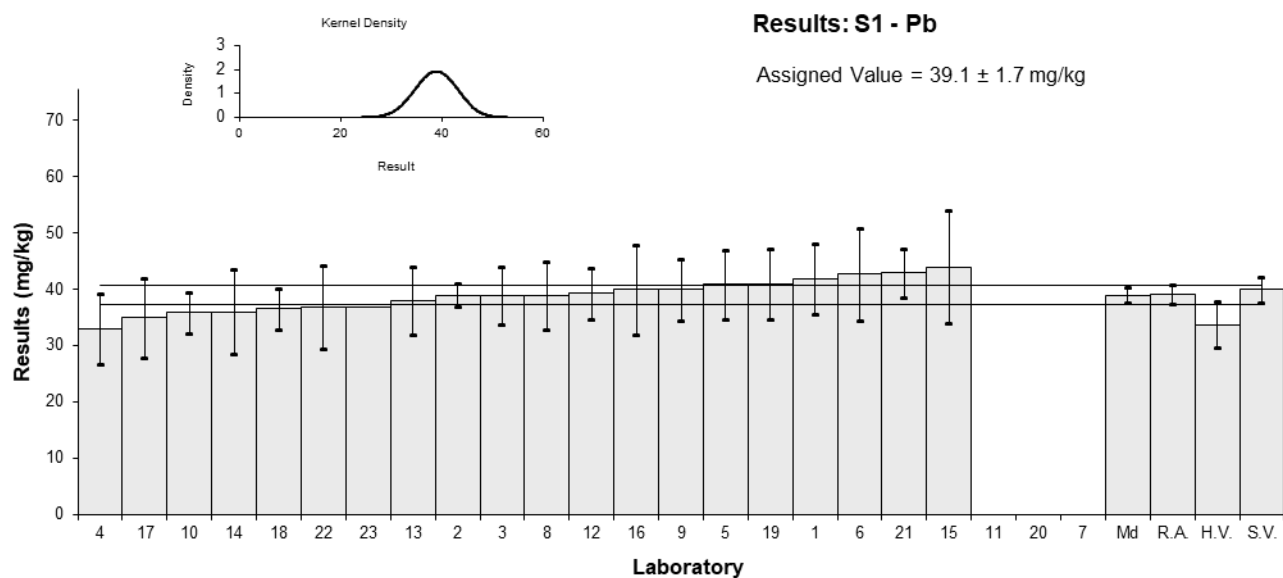


Figure 13

Table 25

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	22	3.3
3	NR	NR
4	NT	NT
5	NT	NT
6	7.6	0.8
7	NT	NT
8	NT	NT
9	NT	NT
10	18.3	1.83
11	NT	NT
12	NT	NT
13	9.2	1.84
14	NT	NT
15	NR	NR
16	NR	NR
17	12	2.4
18	NT	NT
19	12.8	1.4
20	NT	NT
21	NT	NT
22	NT	NT
23	NT	NT

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	10.8	1.3
Robust Average	12.7	7.1
Median	12.0	5.2
Mean	12.7	
N	5	
Max.	22	
Min.	7.6	
Robust SD	6.3	
Robust CV	50%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

Results: S1 - Rb

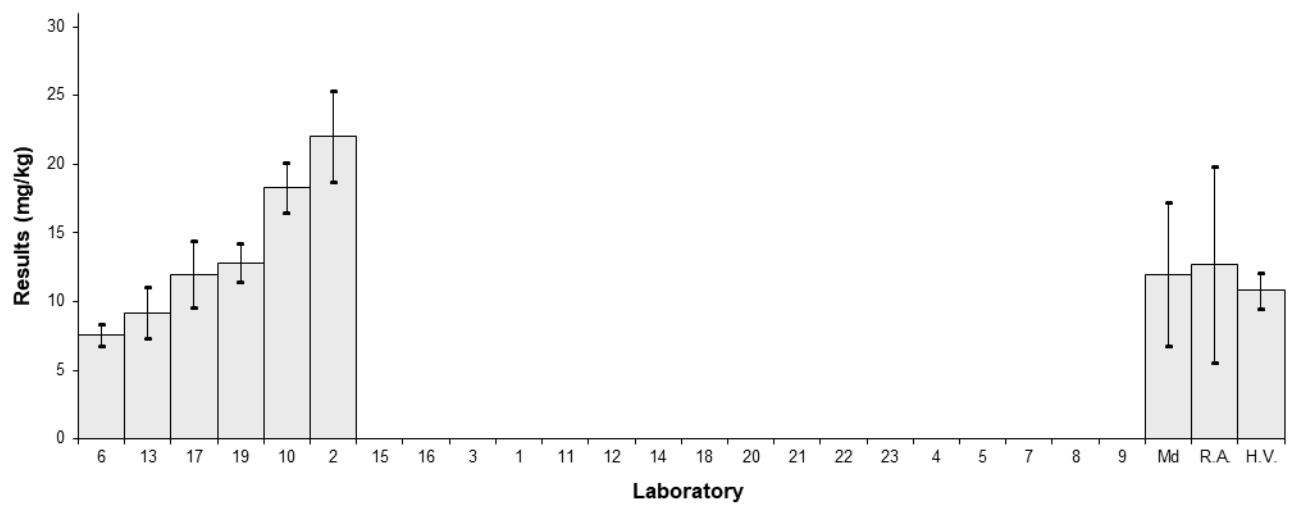


Figure 14

Table 26

Sample Details

Sample No.	S1
Matrix.	Sediment
Analyte.	Sb
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	35.8	5.4
2	18	1.8
3	35	5.0
4	NT	NT
5	34.28	5.14
6	11.9	3.0
7	NT	NT
8	33	4.7
9	8	1.90
10	1.32	0.132
11	NT	NT
12	33.4	3.8
13	21	6.505
14	22	5.0
15	<100	NR
16	37.0	15.2
17	24	4.8
18	NT	NT
19	21.5	3.9
20	NT	NT
21	30	2.7
22	33	6.6
23	6.5	NR

Statistics*

Assigned Value	Not Set	
Spike	40.2	0.8
Homogeneity Value	23.8	2.9
Robust Average	25.5	7.0
Median	27.0	5.4
Mean	25.3	
N	16	
Max.	37	
Min.	6.5	
Robust SD	11	
Robust CV	44%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

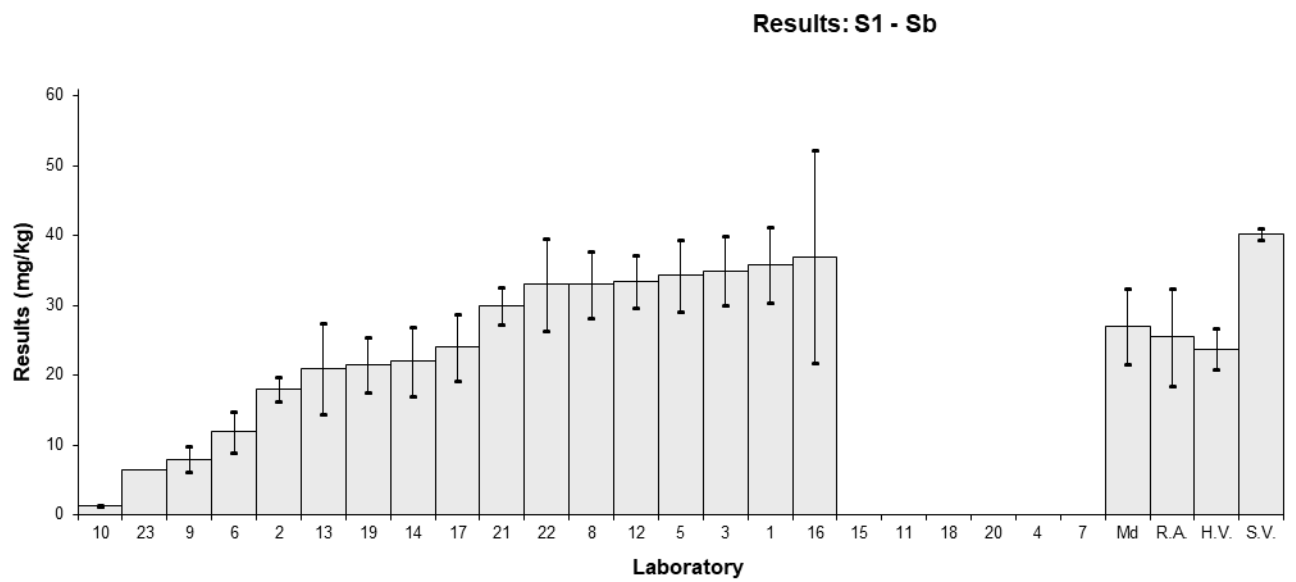


Figure 15

Table 27

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	7.43	1.1	-0.54	-0.53
2	9.8	0.8	1.42	1.77
3	7.5	1.0	-0.48	-0.51
4	NT	NT		
5	7.16	1.07	-0.76	-0.76
6	9	2	0.76	0.44
7	NT	NT		
8	8.4	1.4	0.26	0.21
9	6	1.45	-1.72	-1.34
10	2.29	0.229	-4.78	-9.72
11	NT	NT		
12	8.71	1.0	0.52	0.55
13	9	2.492	0.76	0.36
14	8.5	2.0	0.35	0.2
15	<100	NR		
16	7.97	2.56	-0.09	-0.04
17	7.5	1.5	-0.48	-0.36
18	8.38	0.838	0.25	0.3
19	< 20	14		
20	NT	NT		
21	7.8	0.7	-0.23	-0.31
22	7.7	1.16	-0.31	-0.3
23	NT	NT		

Statistics*

Assigned Value	8.08	0.55
Spike	10.6	0.2
Homogeneity Value	8.07	0.97
Robust Average	8.08	0.55
Median	7.97	0.44
Mean	8.06	
N	15	
Max.	9.8	
Min.	6	
Robust SD	0.85	
Robust CV	11%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

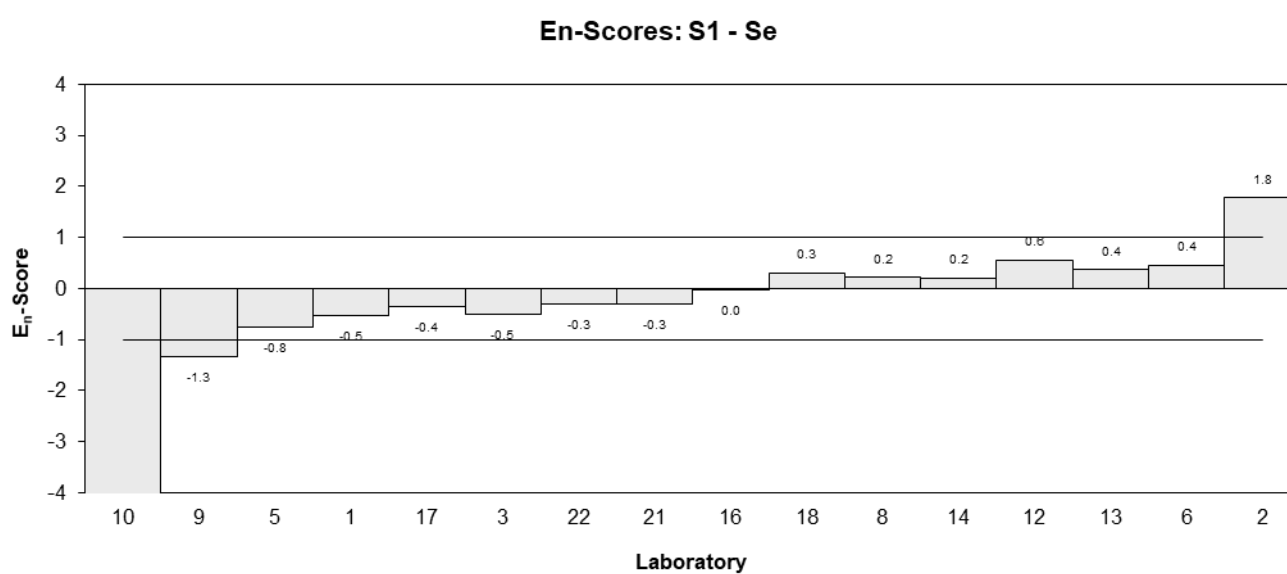
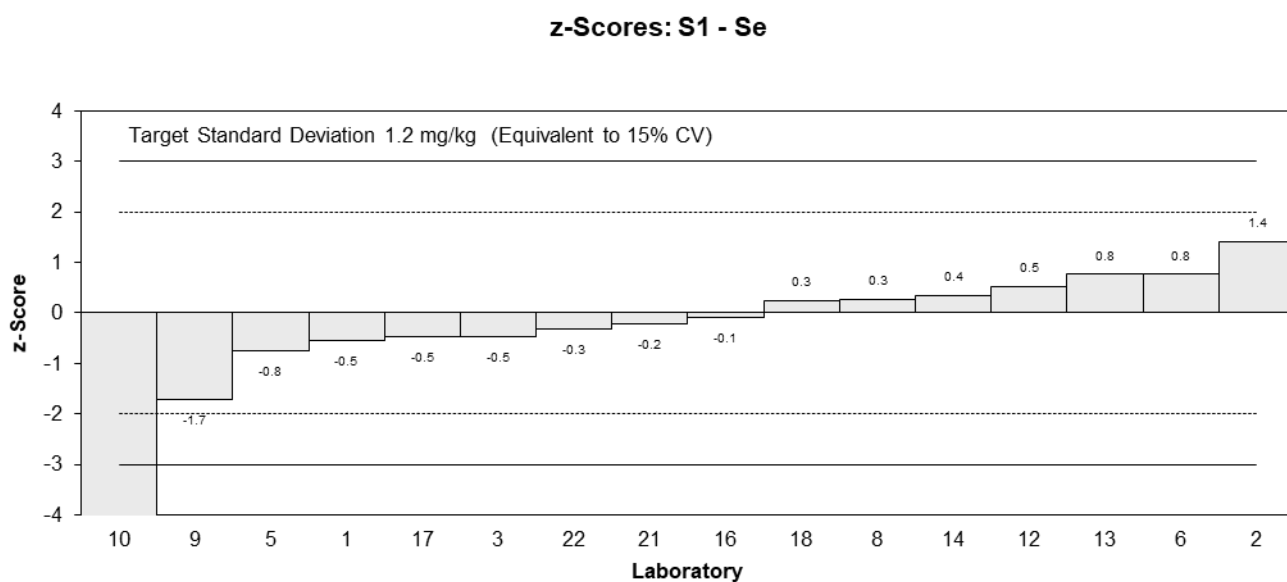
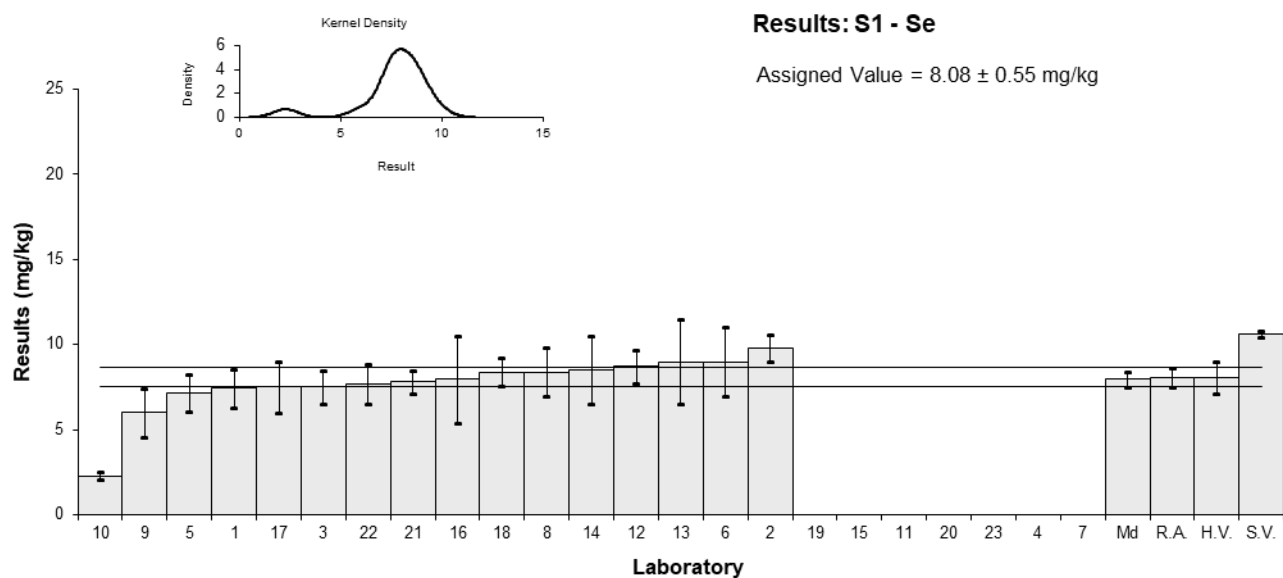


Figure 16

Table 28

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	1.47	0.2	0.21	0.13
2	1.6	0.3	1.11	0.50
3	< 10	NR		
4	NT	NT		
5	1.51	0.23	0.49	0.27
6	1.2	0.4	-1.67	-0.57
7	NT	NT		
8	<10	2		
9	<3	3		
10	17.8	1.78	113.61	9.17
11	NT	NT		
12	1.68	0.3	1.67	0.74
13	1.4	0.28	-0.28	-0.13
14	1.5	1.0	0.42	0.06
15	<50	NR		
16	1.41	0.34	-0.21	-0.08
17	1.2	0.24	-1.67	-0.89
18	3.77	0.377	16.18	5.89
19	1.47	0.71	0.21	0.04
20	NT	NT		
21	1.4	0.1	-0.28	-0.26
22	<10	2		
23	NT	NT		

Statistics*

Assigned Value**	1.44	0.12
Spike	Not Spiked	
Homogeneity Value	1.23	0.15
Robust Average	1.47	0.13
Median	1.47	0.07
Mean	1.63	
N	12	
Max.	3.77	
Min.	1.2	
Robust SD	0.19	
Robust CV	13%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

**Robust Average excluding Laboratory 18.

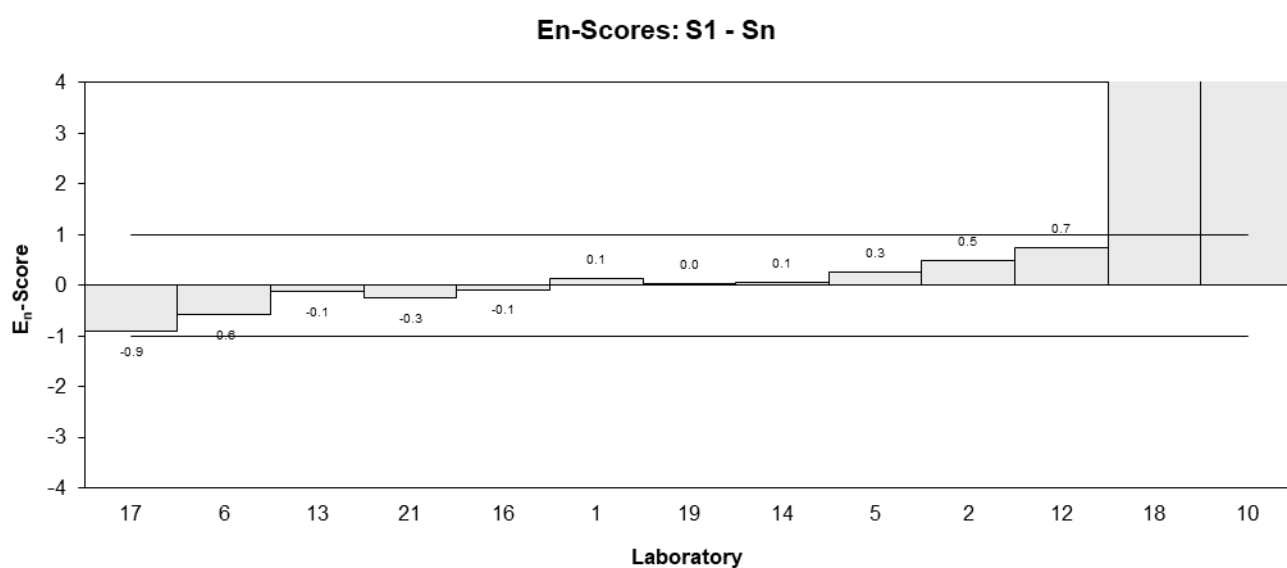
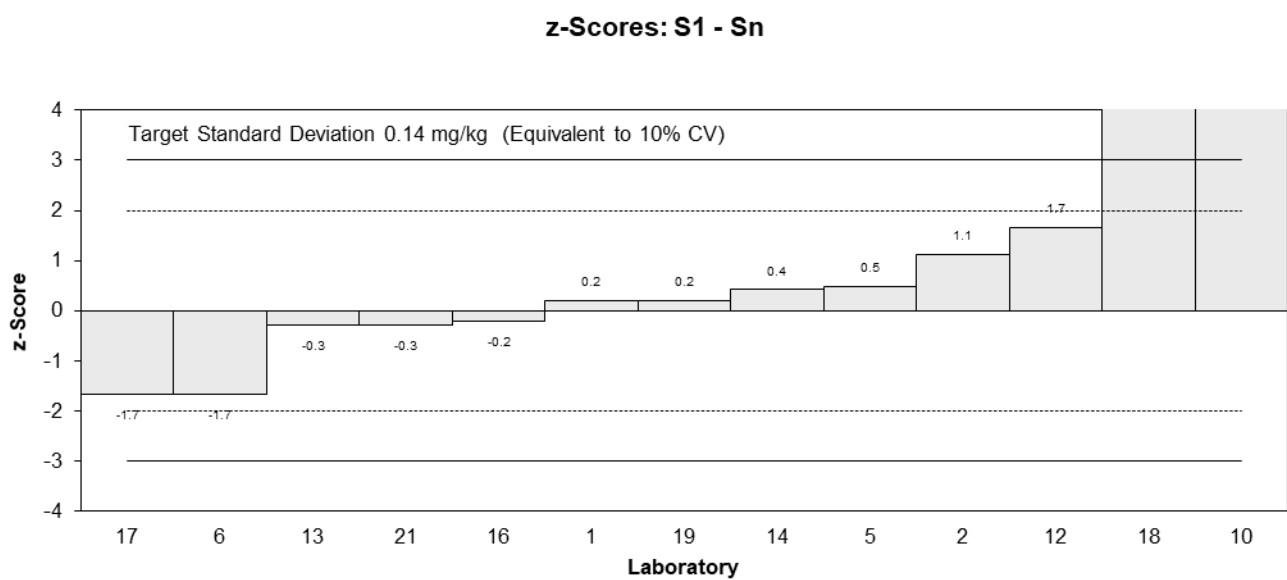
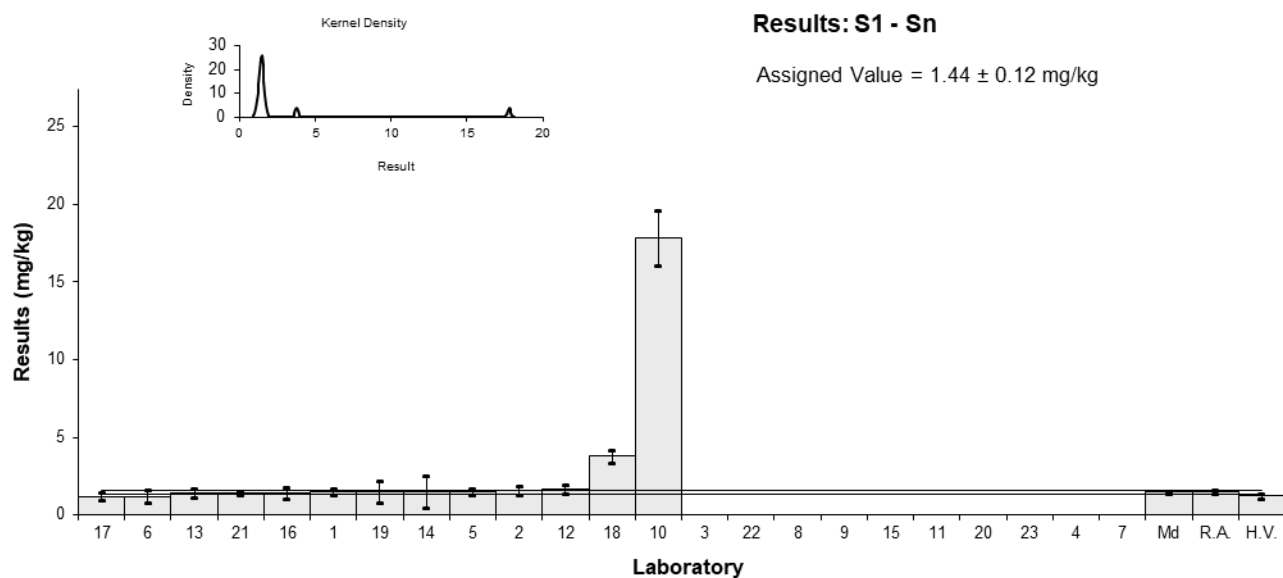


Figure 17

Table 29

Sample Details

Sample No.	S1
Matrix.	Sediment
Analyte.	Th
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	5.7	1.5
3	NR	NR
4	NT	NT
5	NT	NT
6	1.2	0.3
7	NT	NT
8	NT	NT
9	NT	NT
10	NT	NT
11	NT	NT
12	3.34	0.5
13	1.6	0.32
14	NT	NT
15	NR	NR
16	NR	NR
17	3.8	0.76
18	NT	NT
19	NT	NT
20	NT	NT
21	NT	NT
22	NT	NT
23	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	5.80	0.70
Robust Average	3.1	2.3
Median	3.3	3.2
Mean	3.1	
N	5	
Max.	5.7	
Min.	1.2	
Robust SD	2.1	
Robust CV	66%	

Results: S1 - Th

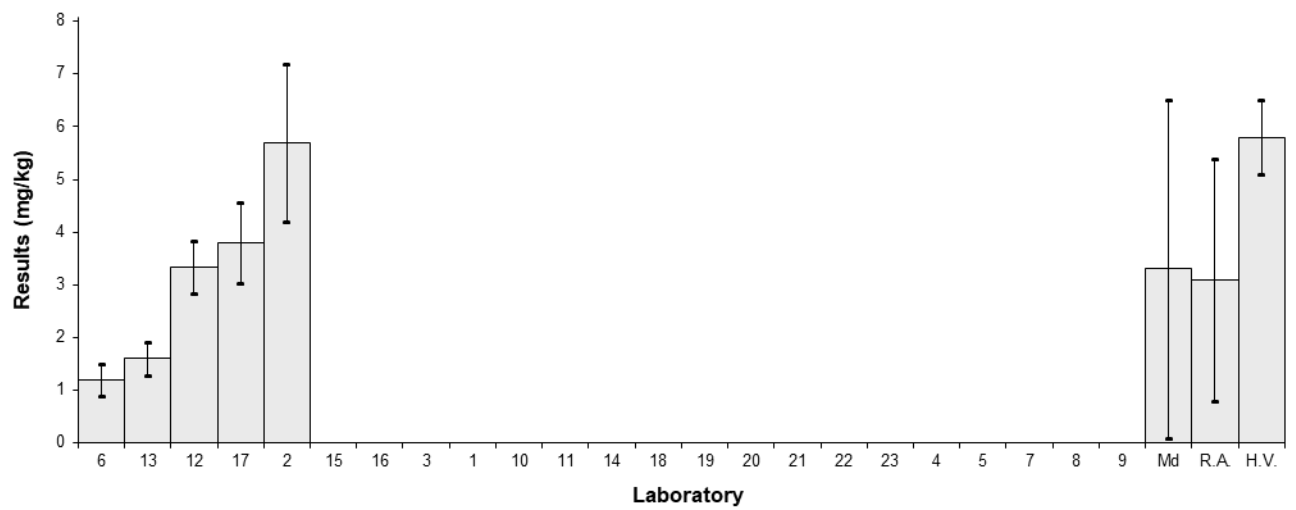


Figure 18

Table 30

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	39.1	6	0.51	0.30
2	38	2.7	0.22	0.23
3	36	4.7	-0.32	-0.23
4	NT	NT		
5	38.72	5.81	0.41	0.25
6	32	7	-1.40	-0.71
7	NT	NT		
8	42	6.2	1.29	0.73
9	34	3.4	-0.86	-0.80
10	33.2	3.32	-1.08	-1.02
11	NT	NT		
12	40.2	5.0	0.81	0.55
13	35	3.963	-0.59	-0.49
14	33	8.1	-1.13	-0.50
15	41	10	1.02	0.37
16	37.9	7.6	0.19	0.09
17	35	7.0	-0.59	-0.30
18	39.0	3.90	0.48	0.41
19	< 100	67		
20	NT	NT		
21	41	2	1.02	1.31
22	37	5.55	-0.05	-0.03
23	34	NR	-0.86	-1.52

Statistics*

Assigned Value	37.2	2.1
Spike	Not Spiked	
Homogeneity Value	35.5	4.3
Robust Average	37.2	2.1
Median	37.9	2.2
Mean	37.2	
N	17	
Max.	42	
Min.	32	
Robust SD	3.4	
Robust CV	9.2%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

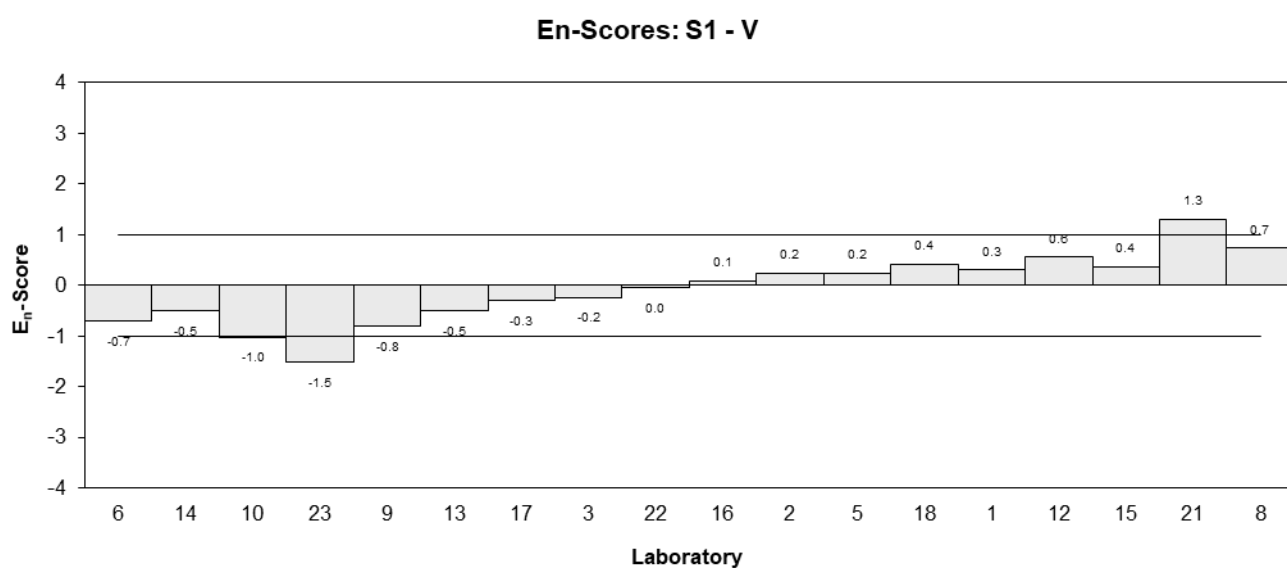
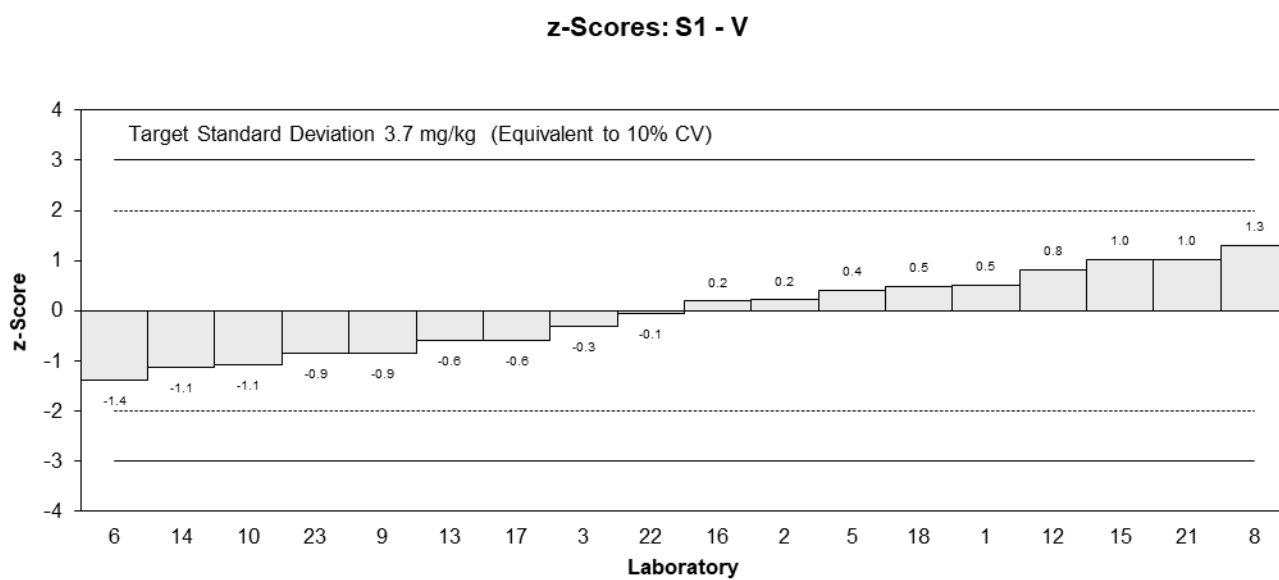
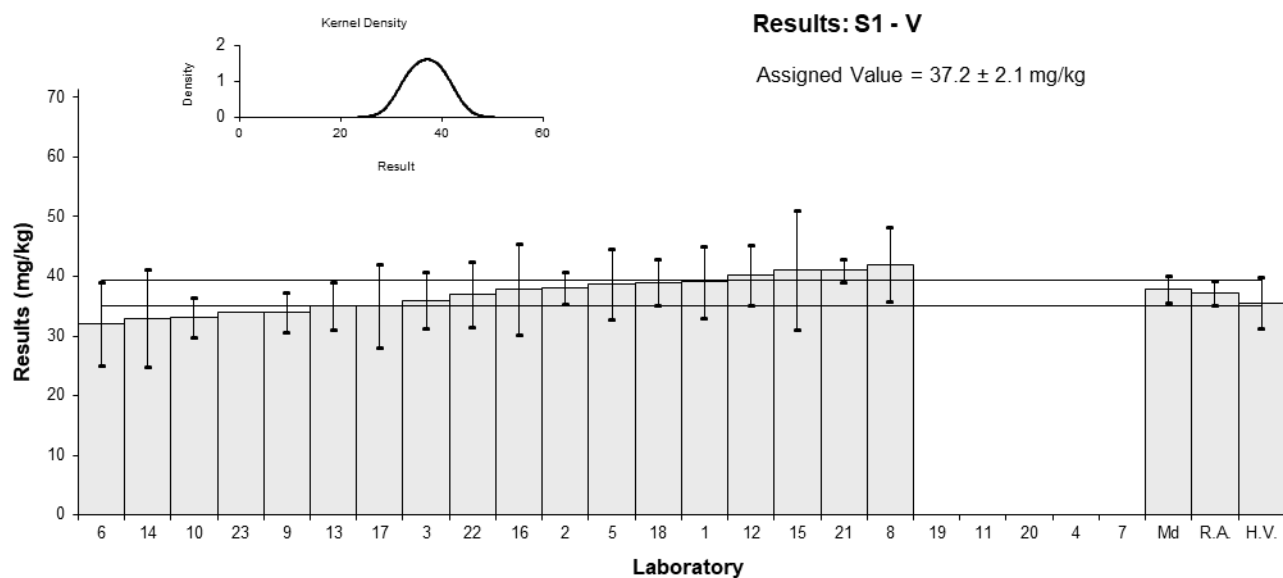


Figure 19

Table 31

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	102	15	0.96	0.58
2	95	7	0.20	0.24
3	89	11	-0.44	-0.35
4	86	26	-0.76	-0.27
5	97.47	14.62	0.47	0.29
6	83.6	14.1	-1.02	-0.65
7	NT	NT		
8	94	15	0.10	0.06
9	88	15.84	-0.55	-0.31
10	206	20.6	12.13	5.39
11	NT	NT		
12	102	10	0.96	0.83
13	95	15.105	0.20	0.12
14	85	14	-0.87	-0.56
15	98	10	0.53	0.46
16	94.8	20.0	0.18	0.08
17	88	18	-0.55	-0.28
18	93.2	9.32	0.01	0.01
19	94.9	7.2	0.19	0.22
20	NT	NT		
21	110	6.6	1.82	2.22
22	92	18.4	-0.12	-0.06
23	87	NR	-0.66	-1.61

Statistics*

Assigned Value	93.1	3.8
Spike	Not Spiked	
Homogeneity Value	91	11
Robust Average	93.1	3.8
Median	94.0	3.6
Mean	93.4	
N	19	
Max.	110	
Min.	83.6	
Robust SD	6.7	
Robust CV	7.2%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

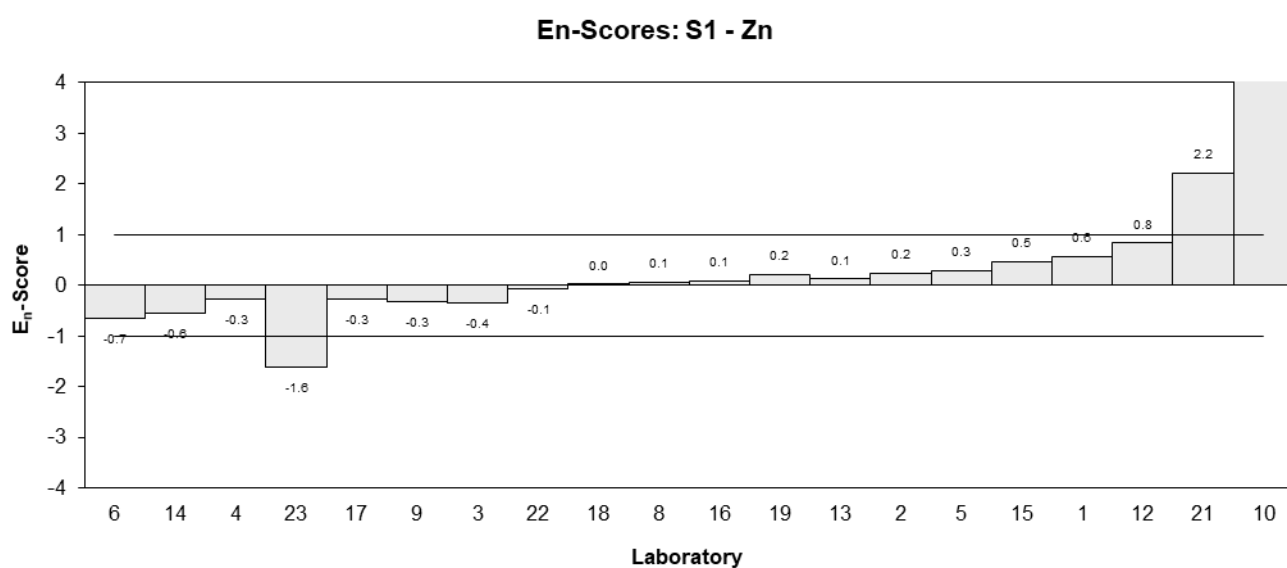
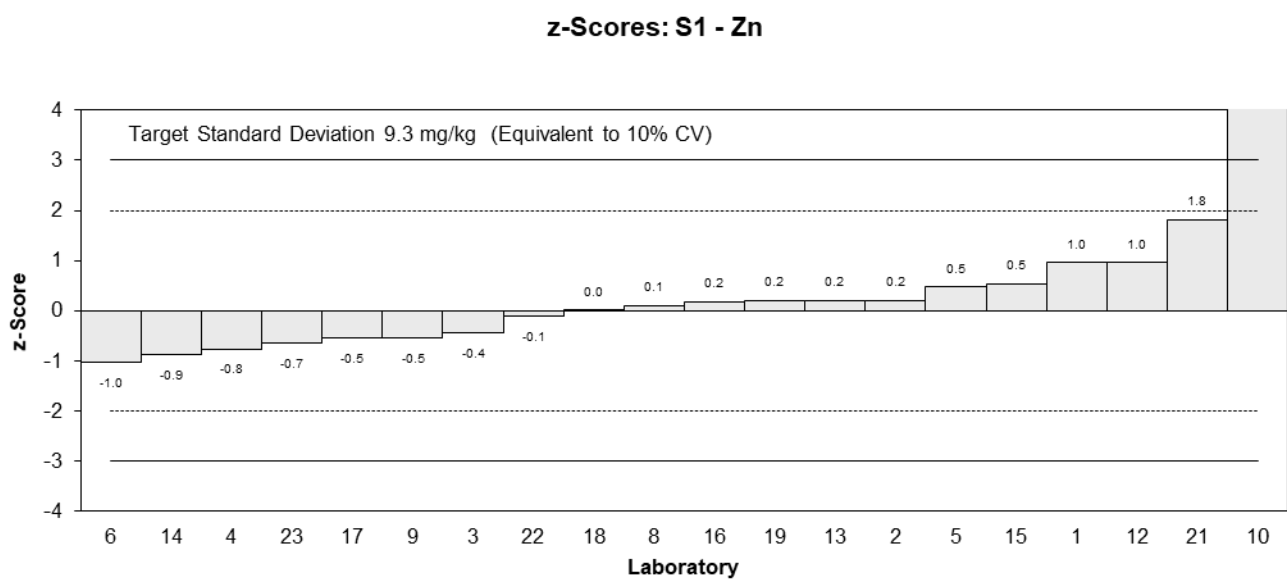
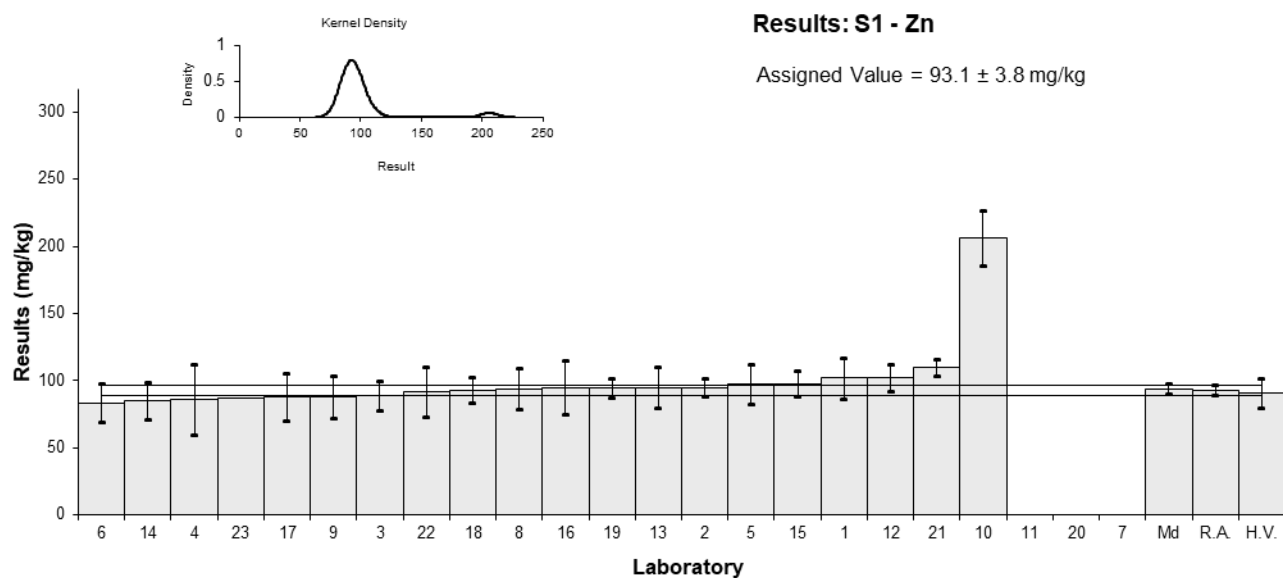


Figure 20

Table 32

Sample Details

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

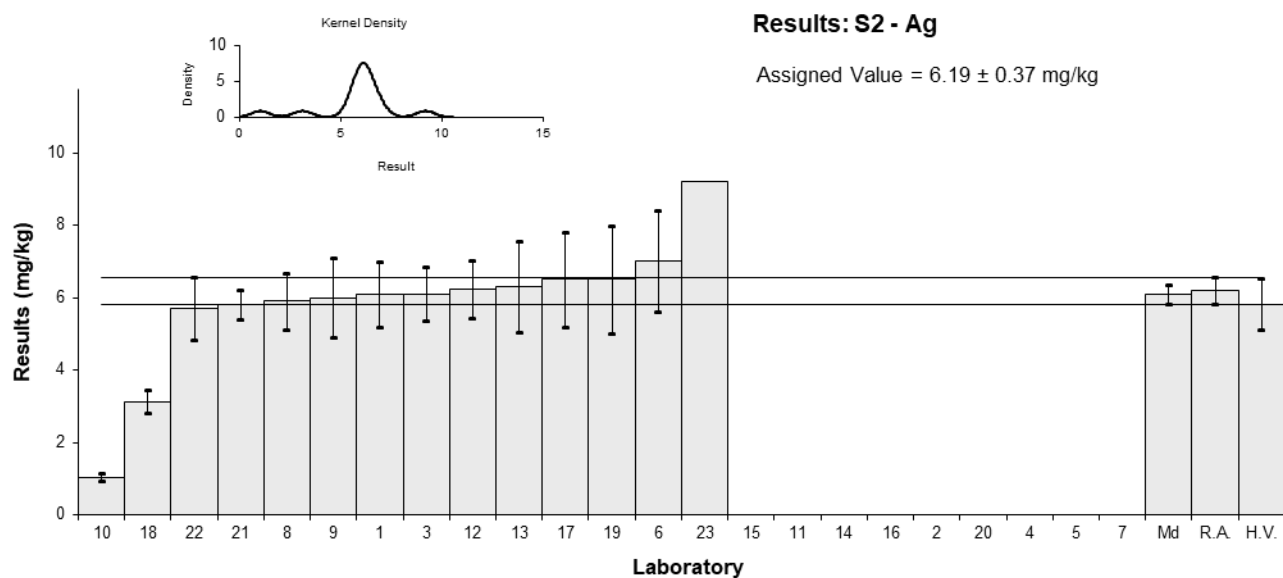
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	6.08	0.9	-0.18	-0.11
2	NT	NT		
3	6.1	0.74	-0.15	-0.11
4	NT	NT		
5	NT	NT		
6	7.0	1.4	1.31	0.56
7	NT	NT		
8	5.9	0.77	-0.47	-0.34
9	6	1.09	-0.31	-0.17
10	1.04	0.104	-8.32	-13.40
11	NT	NT		
12	6.23	0.8	0.06	0.05
13	6.3	1.26	0.18	0.08
14	NT	NT		
15	NR	NR		
16	NT	NT		
17	6.5	1.3	0.50	0.23
18	3.13	0.313	-4.94	-6.31
19	6.5	1.5	0.50	0.20
20	NT	NT		
21	5.8	0.41	-0.63	-0.71
22	5.7	0.86	-0.79	-0.52
23	9.2	NR	4.86	8.14

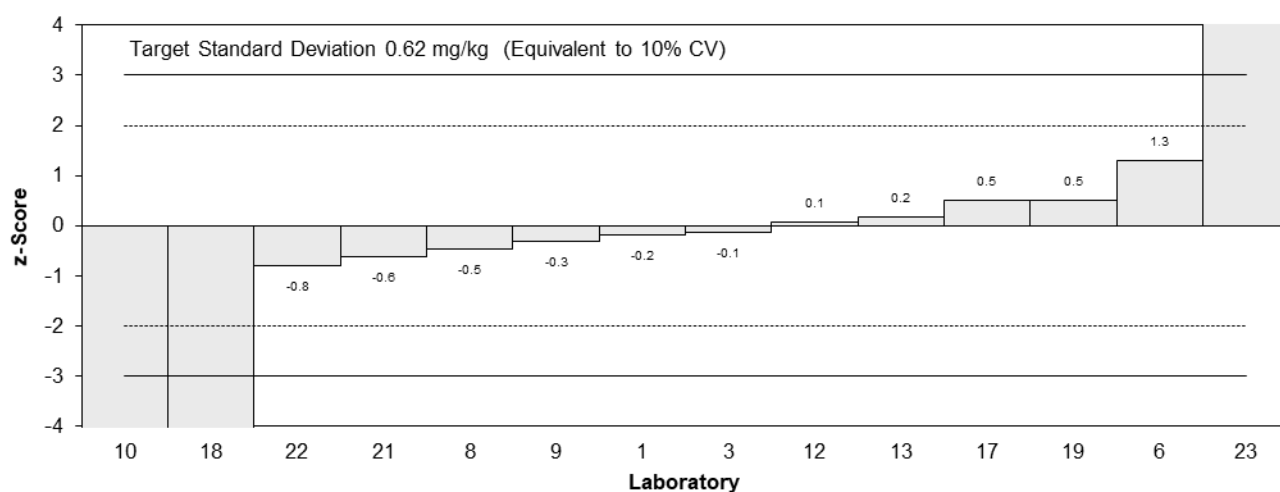
Statistics*

Assigned Value	6.19	0.37
Spike	Not Spiked	
Homogeneity Value	5.82	0.70
Robust Average	6.19	0.37
Median	6.10	0.27
Mean	6.19	
N	13	
Max.	9.2	
Min.	3.13	
Robust SD	0.53	
Robust CV	8.6%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.



z-Scores: S2 - Ag



En-Scores: S2 - Ag

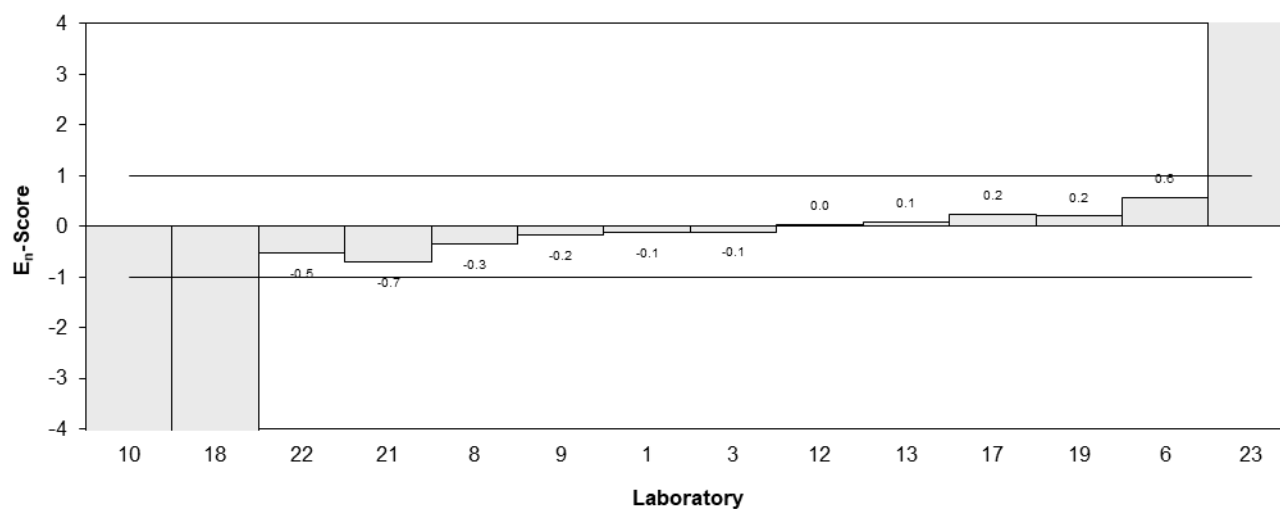


Figure 21

Table 33

Sample Details

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

Participant Results

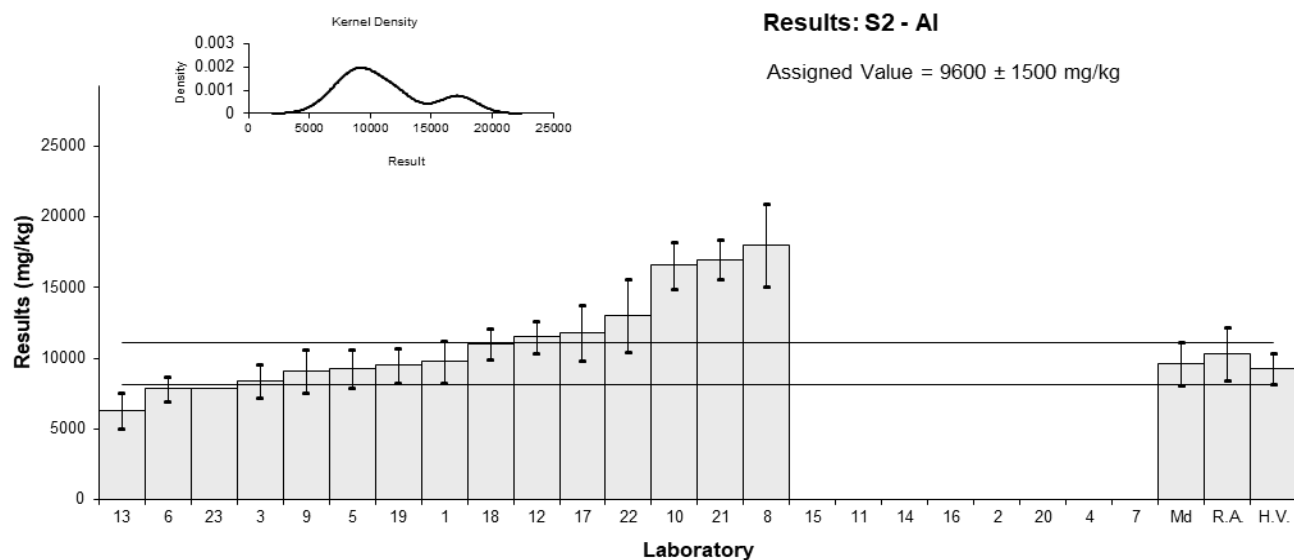
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	9750	1460	0.08	0.07
2	NT	NT		
3	8400	1200	-0.62	-0.62
4	NT	NT		
5	9254.31	1388.15	-0.18	-0.17
6	7830	902	-0.92	-1.01
7	NT	NT		
8	18000	2900	4.38	2.57
9	9100	1537.9	-0.26	-0.23
10	16580	1658	3.64	3.12
11	NT	NT		
12	11520	1150	1.00	1.02
13	6290	1258	-1.72	-1.69
14	NT	NT		
15	NR	NR		
16	NT	NT		
17	11800	2000	1.15	0.88
18	11000	1100	0.73	0.75
19	9500	1200	-0.05	-0.05
20	NT	NT		
21	17000	1360	3.85	3.65
22	13000	2600	1.77	1.13
23	7866	NR	-0.90	-1.16

Statistics*

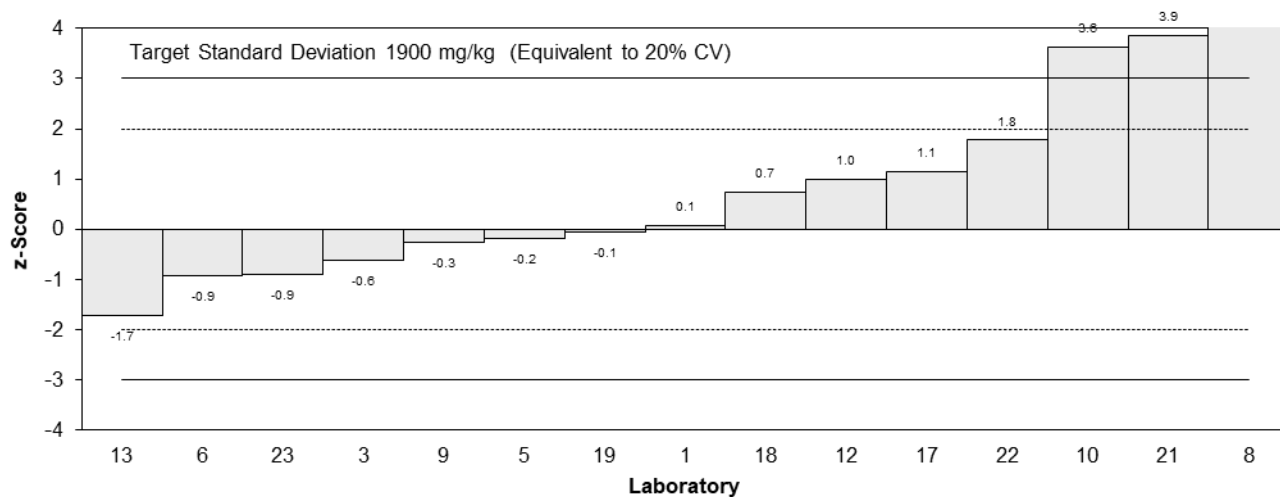
Assigned Value**	9600	1500
Spike	Not Spiked	
Homogeneity Value	9200	1100
Robust Average	10300	1900
Median	9600	1500
Mean	10700	
N	14	
Max.	18000	
Min.	6290	
Robust SD	2900	
Robust CV	28%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

**Robust Average excluding Laboratories 8 and 21.



z-Scores: S2 - Al



En-Scores: S2 - Al

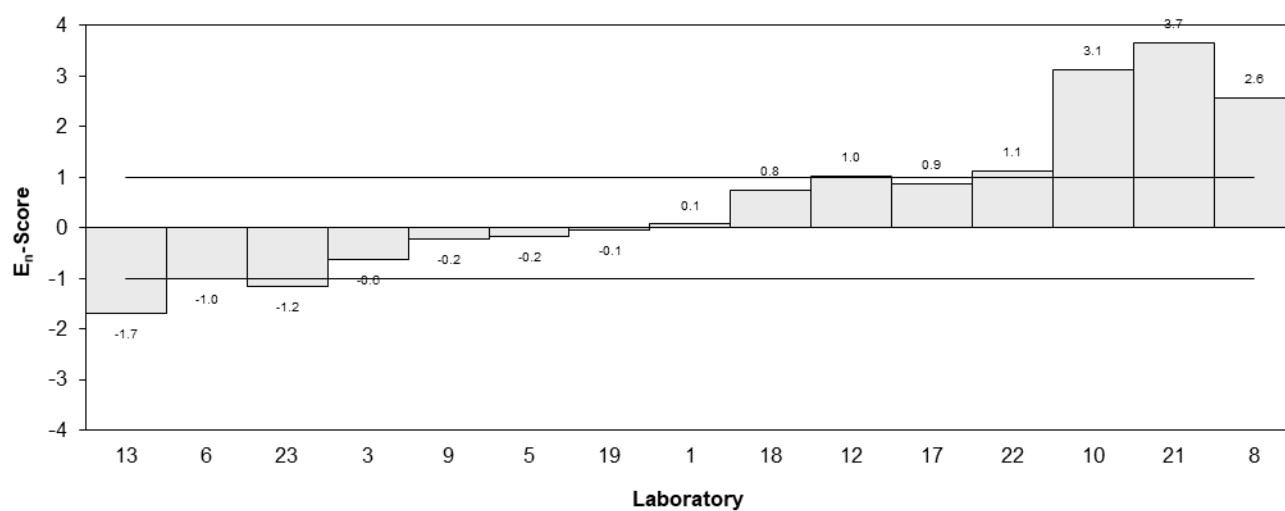


Figure 22

Table 34

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	3.30	0.5	-0.78	-0.48
2	NT	NT		
3	4.0	0.56	1.17	0.66
4	NT	NT		
5	3.99	0.60	1.15	0.61
6	3.6	0.6	0.06	0.03
7	NT	NT		
8	3.8	0.55	0.61	0.35
9	4	0.53	1.17	0.68
10	6.9	0.69	9.27	4.39
11	NT	NT		
12	3.99	0.6	1.15	0.61
13	3	0.6	-1.62	-0.86
14	NT	NT		
15	<25	NR		
16	NT	NT		
17	3.6	0.72	0.06	0.03
18	3.10	0.31	-1.34	-1.09
19	3.5	1.5	-0.22	-0.05
20	NT	NT		
21	<5	NR		
22	3.8	0.76	0.61	0.27
23	2.0	NR	-4.41	-5.10

Statistics*

Assigned Value	3.58	0.31
Spike	Not Spiked	
Homogeneity Value	3.99	0.48
Robust Average	3.58	0.31
Median	3.60	0.35
Mean	3.51	
N	13	
Max.	4	
Min.	2	
Robust SD	0.45	
Robust CV	13%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

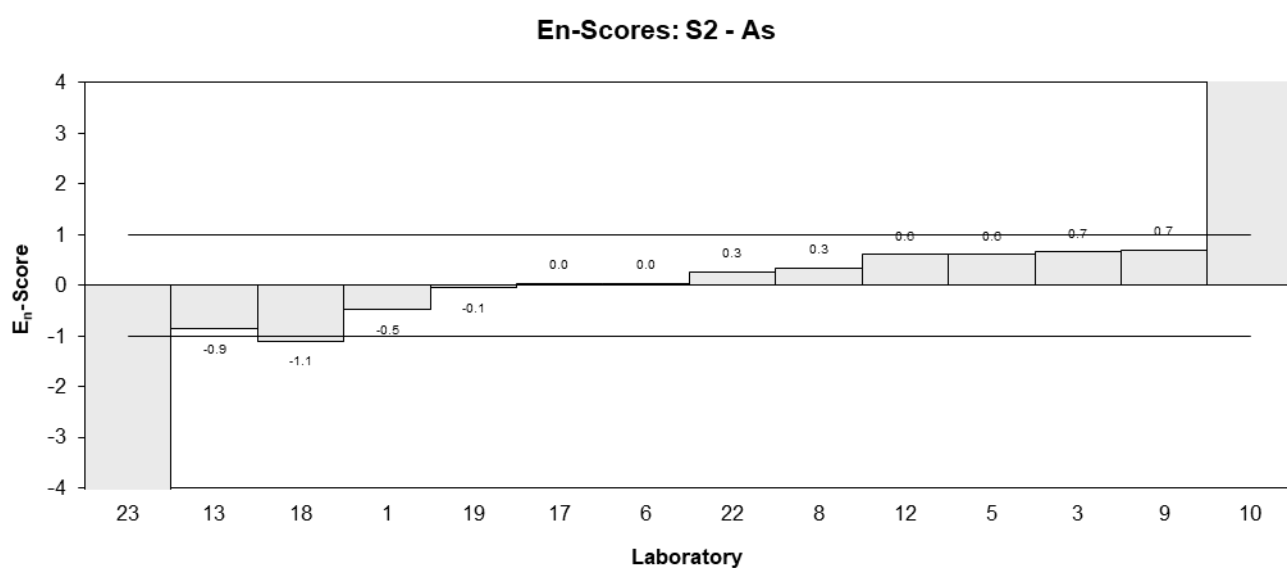
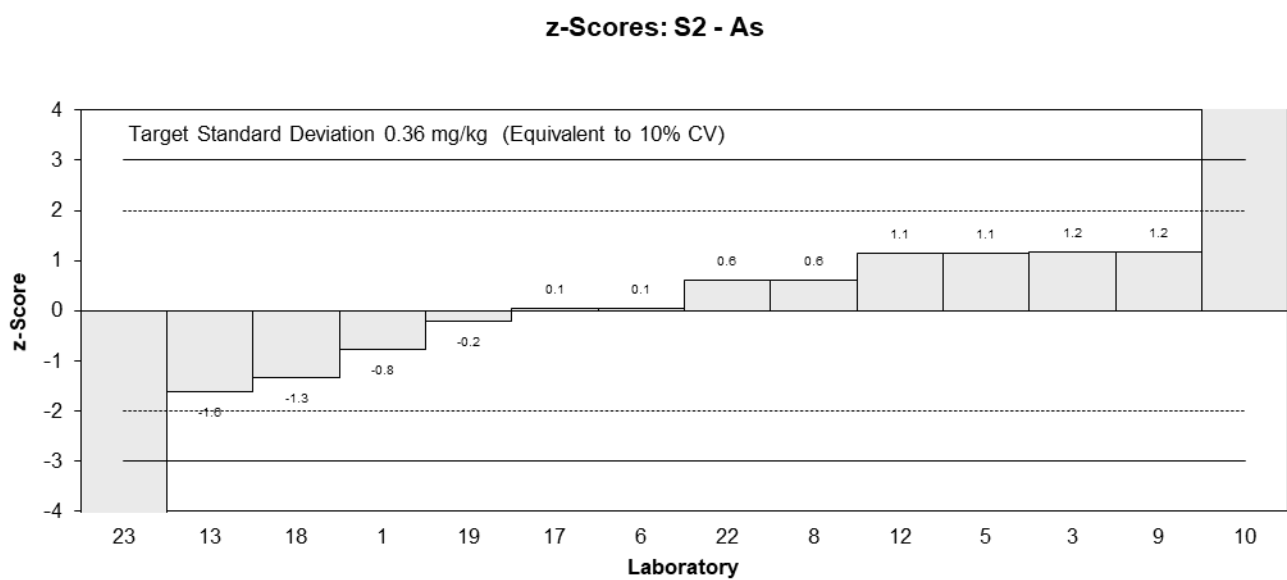
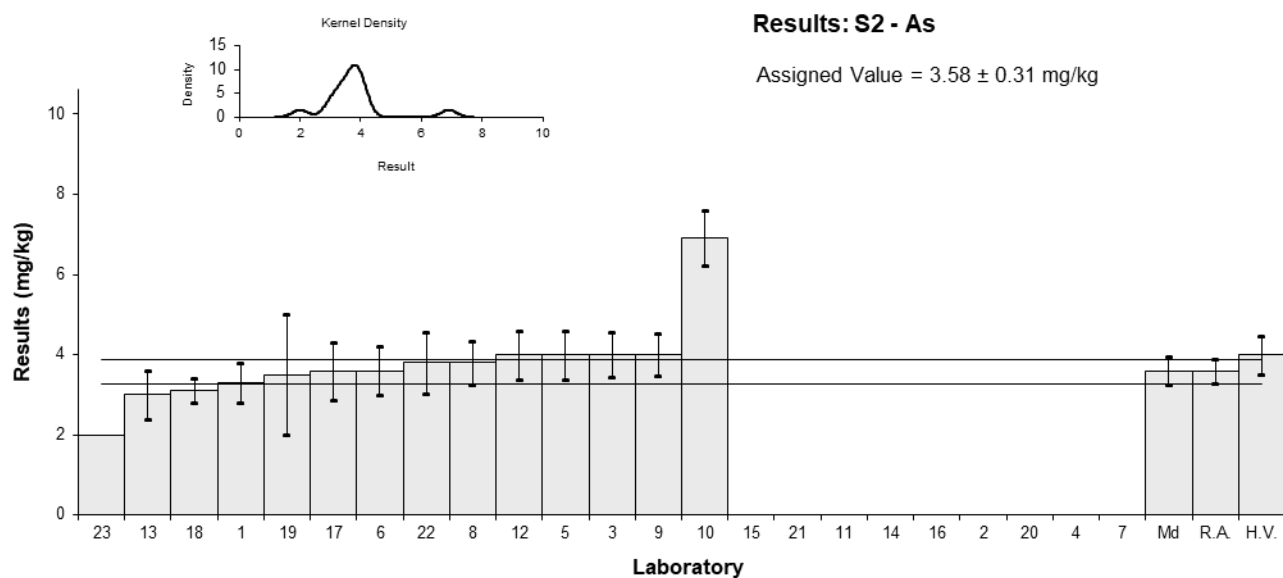


Figure 23

Table 35

Sample Details

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

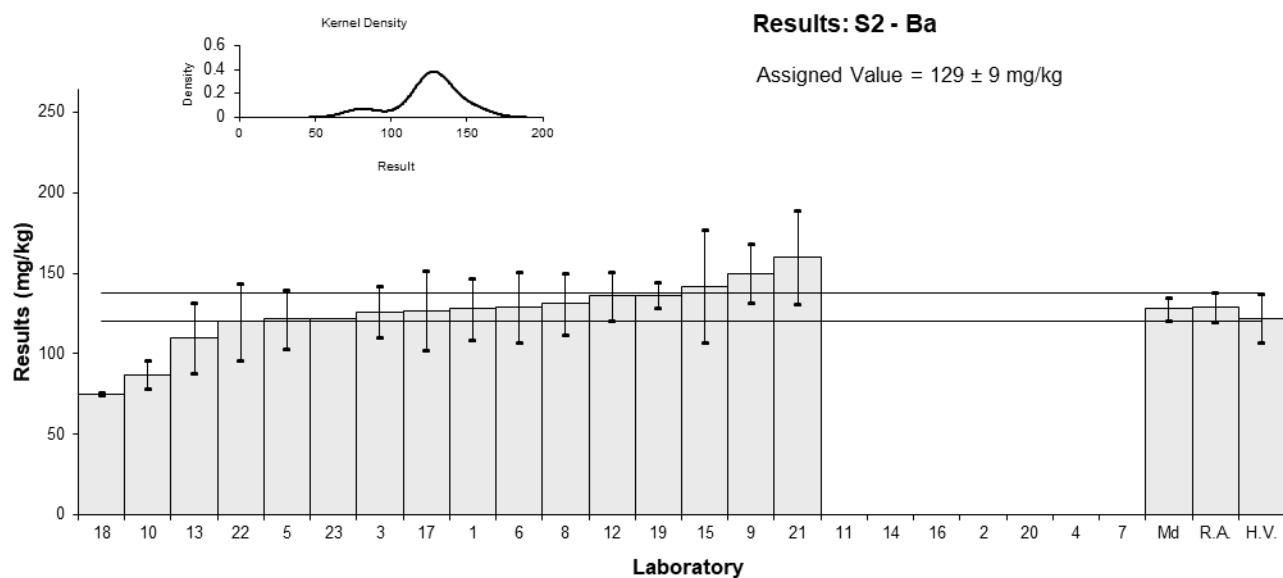
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	128	19	-0.08	-0.05
2	NT	NT		
3	126	16	-0.23	-0.16
4	NT	NT		
5	121.70	18.26	-0.57	-0.36
6	129	22	0.00	0.00
7	NT	NT		
8	131	19	0.16	0.09
9	150	18.45	1.63	1.02
10	87	8.7	-3.26	-3.34
11	NT	NT		
12	136	15	0.54	0.40
13	110	22	-1.47	-0.80
14	NT	NT		
15	142	35	1.01	0.36
16	NT	NT		
17	127	25	-0.16	-0.08
18	75	0.75	-4.19	-5.91
19	136.4	8.2	0.57	0.60
20	NT	NT		
21	160	28.8	2.40	1.03
22	120	24	-0.70	-0.35
23	122	NR	-0.54	-0.77

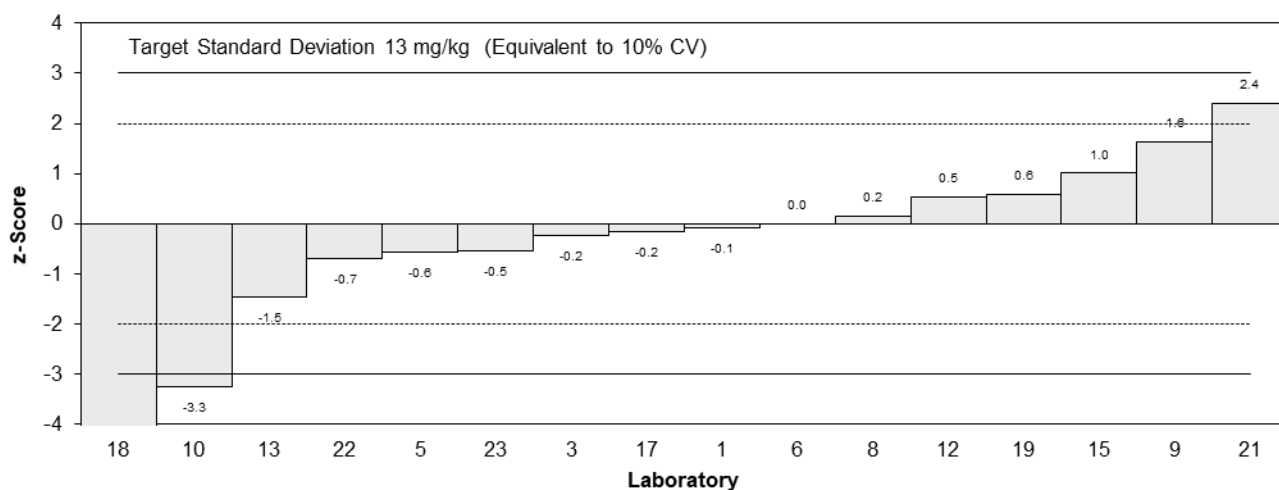
Statistics*

Assigned Value	129	9
Spike	Not Spiked	
Homogeneity Value	122	15
Robust Average	129	9
Median	128	7
Mean	128	
N	15	
Max.	160	
Min.	75	
Robust SD	14	
Robust CV	11%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.



z-Scores: S2 - Ba



En-Scores: S2 - Ba

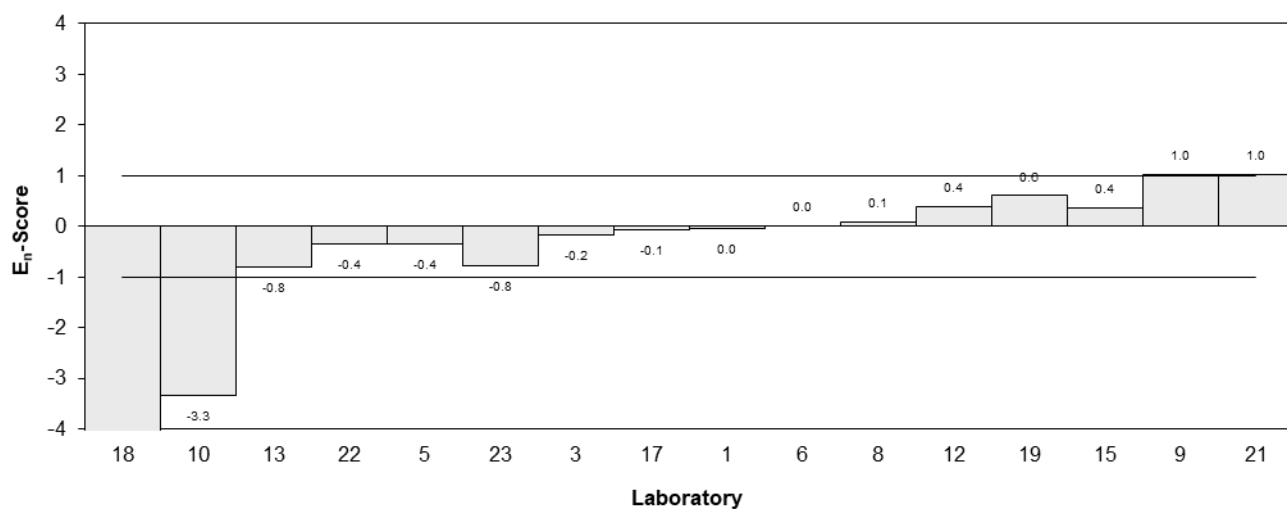


Figure 24

Table 36

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	1.81	0.3
2	NT	NT
3	<10	NR
4	NT	NT
5	NT	NT
6	0.9	0.3
7	NT	NT
8	<10	2
9	NT	NT
10	0.13	0.013
11	NT	NT
12	0.68	0.1
13	0.7	0.14
14	NT	NT
15	NR	NR
16	NT	NT
17	0.78	0.16
18	NT	NT
19	0.82	0.29
20	NT	NT
21	NT	NT
22	<10	2
23	1.8	NR

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.750	0.090
Robust Average	1.07	0.54
Median	0.82	0.16
Mean	1.07	
N	7	
Max.	1.81	
Min.	0.68	
Robust SD	0.58	
Robust CV	54%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

Results: S2 - Bi

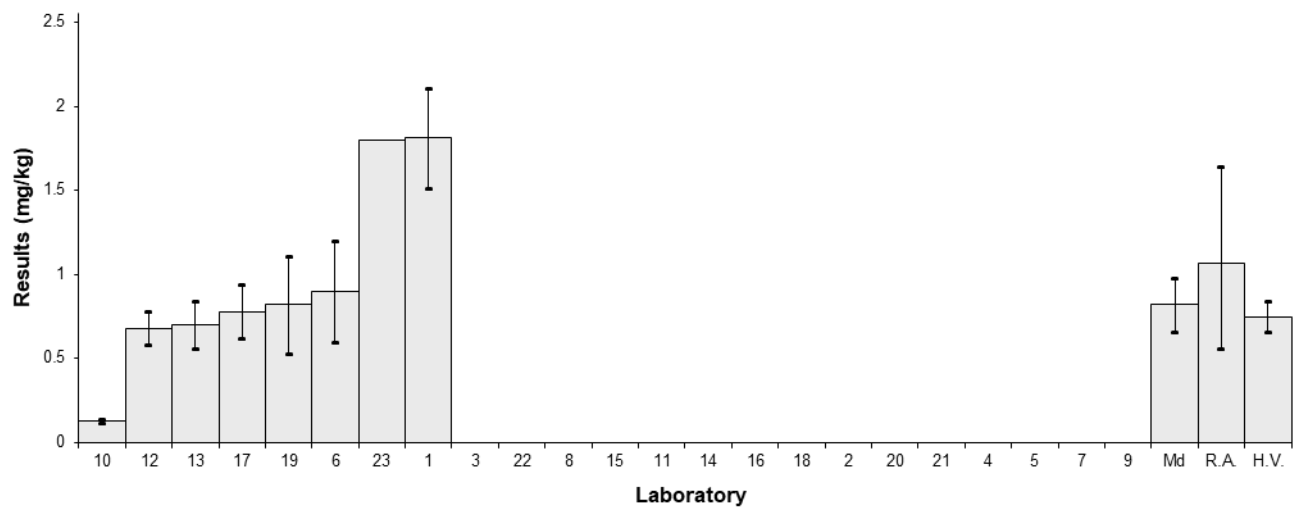


Figure 25

Table 37

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.86	0.1	1.98	1.20
2	NT	NT		
3	0.7	0.082	-0.25	-0.17
4	NT	NT		
5	1.10	0.17	5.32	2.11
6	0.8	0.1	1.14	0.69
7	NT	NT		
8	0.7	0.094	-0.25	-0.16
9	0.6	0.09	-1.64	-1.07
10	2.17	0.217	20.22	6.43
11	NT	NT		
12	0.78	0.1	0.86	0.52
13	0.6	0.2	-1.64	-0.56
14	NT	NT		
15	<1	NR		
16	NT	NT		
17	0.75	0.15	0.45	0.20
18	0.653	0.065	-0.91	-0.72
19	0.78	0.13	0.86	0.43
20	NT	NT		
21	<1	NR		
22	0.7	0.14	-0.25	-0.12
23	0.7	NR	-0.25	-0.29

Statistics*

Assigned Value**	0.718	0.063
Spike	Not Spiked	
Homogeneity Value	0.783	0.094
Robust Average	0.731	0.069
Median	0.700	0.072
Mean	0.748	
N	13	
Max.	1.1	
Min.	0.6	
Robust SD	0.10	
Robust CV	14%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

**Robust Average excluding Laboratory 5.

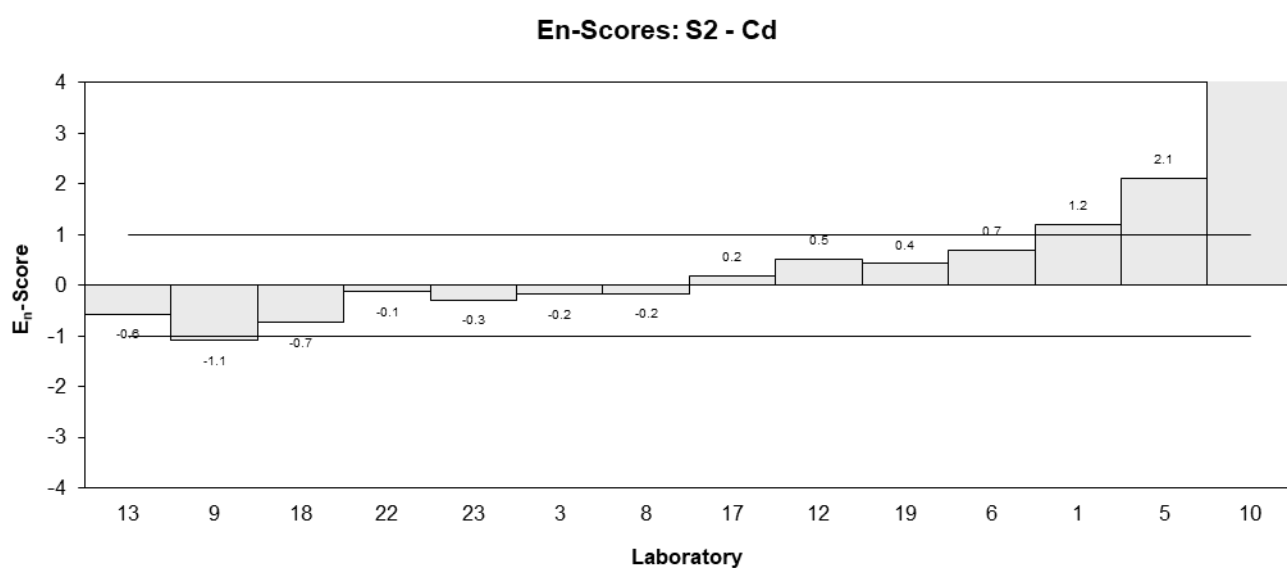
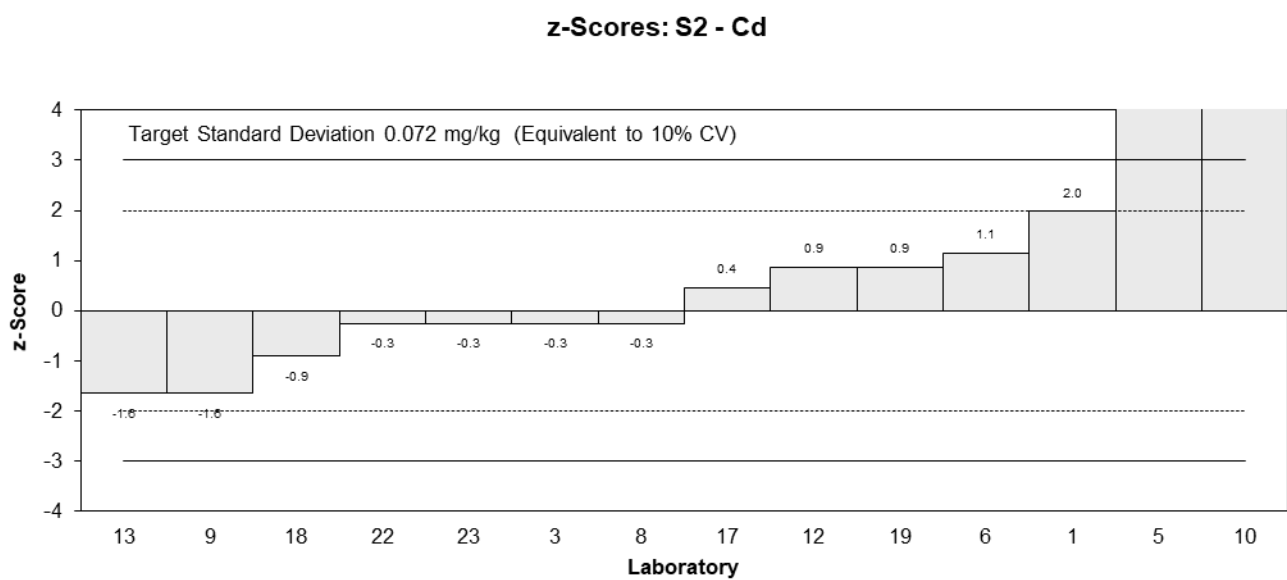
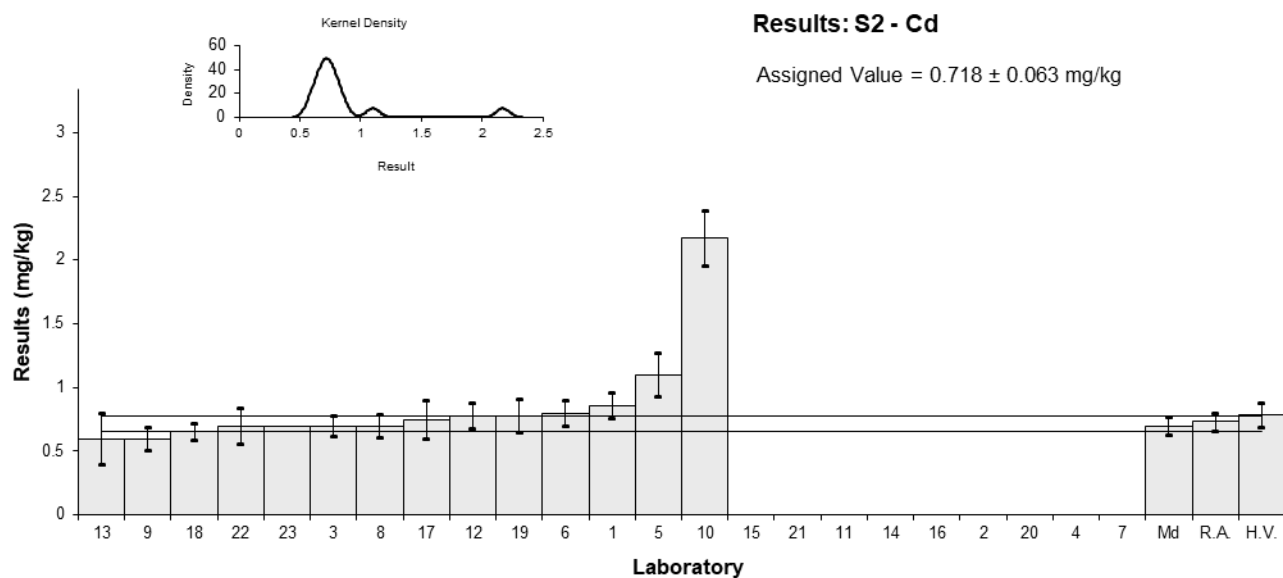


Figure 26

Table 38

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	8.31	1.2	-0.06	-0.04
2	NT	NT		
3	7.9	1.0	-0.55	-0.43
4	NT	NT		
5	7.92	1.19	-0.53	-0.35
6	8.4	1.3	0.05	0.03
7	NT	NT		
8	8.7	1.4	0.41	0.23
9	9.4	1.11	1.24	0.88
10	11.5	1.15	3.76	2.57
11	NT	NT		
12	8.96	0.9	0.72	0.61
13	6.8	10.3	-1.87	-0.15
14	NT	NT		
15	NR	NR		
16	NT	NT		
17	8.4	1.7	0.05	0.02
18	7.80	0.78	-0.67	-0.64
19	8.3	1.2	-0.07	-0.05
20	NT	NT		
21	9.8	NR	1.72	3.51
22	8.2	1.23	-0.19	-0.12
23	8.2	NR	-0.19	-0.39

Statistics*

Assigned Value	8.36	0.41
Spike	Not Spiked	
Homogeneity Value	7.58	0.91
Robust Average	8.36	0.41
Median	8.31	0.33
Mean	8.36	
N	14	
Max.	9.8	
Min.	6.8	
Robust SD	0.61	
Robust CV	7.3%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

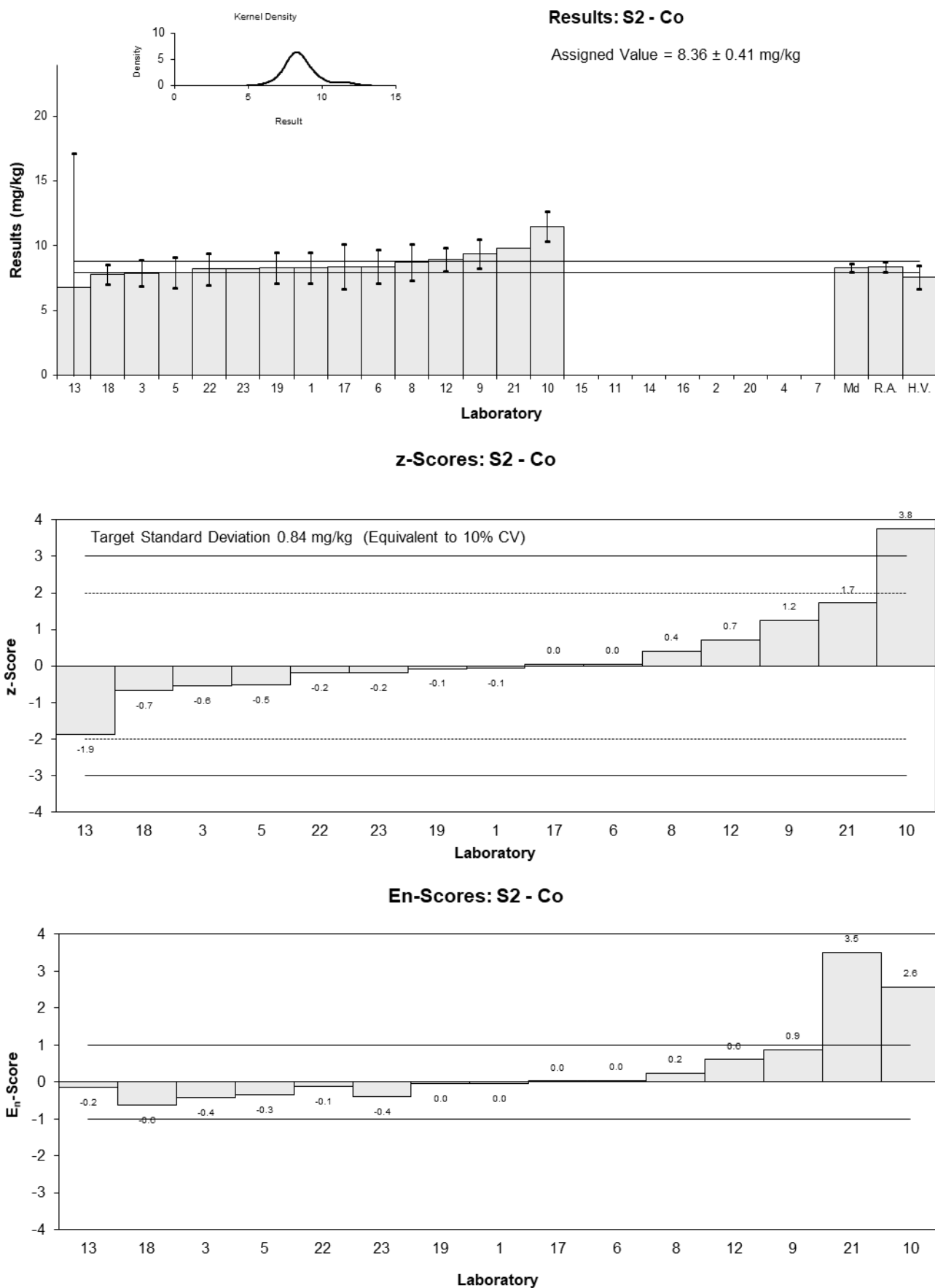


Figure 27

Table 39

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	33.1	5	0.09	0.06
2	NT	NT		
3	31	3.9	-0.55	-0.41
4	NT	NT		
5	32.45	4.87	-0.11	-0.07
6	32.7	7.7	-0.03	-0.01
7	NT	NT		
8	36	5.5	0.98	0.55
9	32	3.81	-0.24	-0.19
10	90.5	9.05	17.59	6.23
11	NT	NT		
12	35.3	3.5	0.76	0.62
13	27	5.4	-1.77	-1.01
14	NT	NT		
15	37	10	1.28	0.41
16	NT	NT		
17	32	6.4	-0.24	-0.12
18	27.4	2.74	-1.65	-1.59
19	31.4	5.1	-0.43	-0.26
20	NT	NT		
21	40	4	2.20	1.61
22	33	6.6	0.06	0.03
23	32	NR	-0.24	-0.40

Statistics*

Assigned Value	32.8	2.0
Spike	Not Spiked	
Homogeneity Value	33.2	4.0
Robust Average	32.8	2.0
Median	32.5	0.9
Mean	32.8	
N	15	
Max.	40	
Min.	27	
Robust SD	3.2	
Robust CV	9.7%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

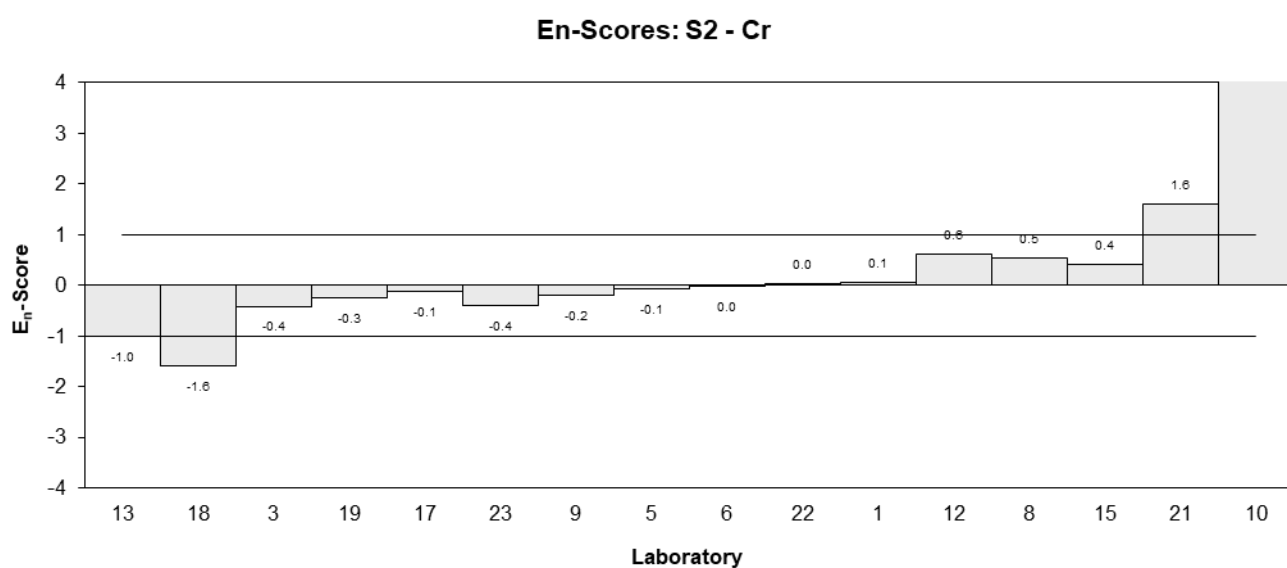
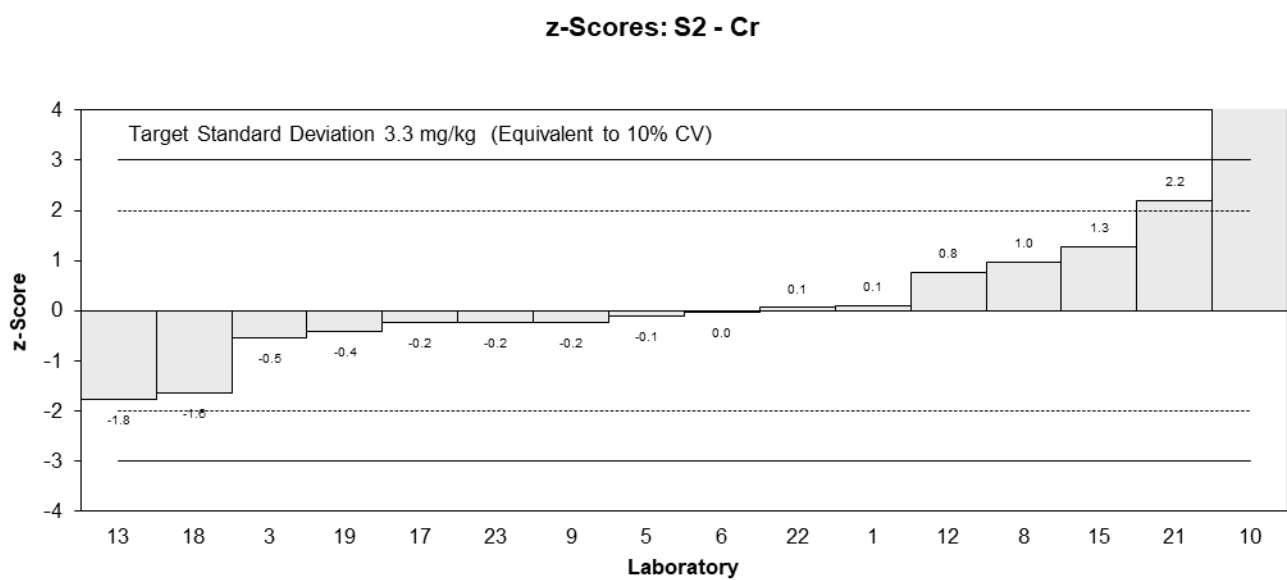
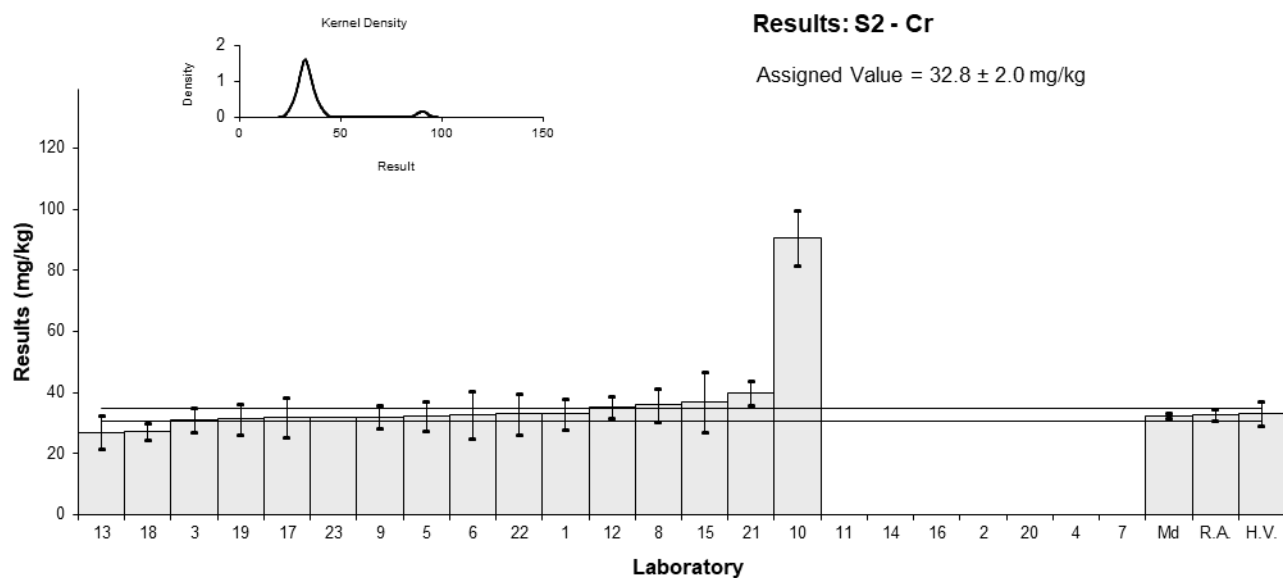


Figure 28

Table 40

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	NT	NT
3	NR	NR
4	NT	NT
5	NT	NT
6	0.6	0.1
7	NT	NT
8	NT	NT
9	NT	NT
10	1.35	0.135
11	NT	NT
12	NT	NT
13	0.5	0.1
14	NT	NT
15	NR	NR
16	NT	NT
17	1.1	0.22
18	NT	NT
19	0.82	0.16
20	NT	NT
21	NT	NT
22	NT	NT
23	NT	NT

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.84	0.10
Robust Average	0.76	0.38
Median	0.71	0.38
Mean	0.76	
N	4	
Max.	1.1	
Min.	0.5	
Robust SD	0.30	
Robust CV	40%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

Results: S2 - Cs

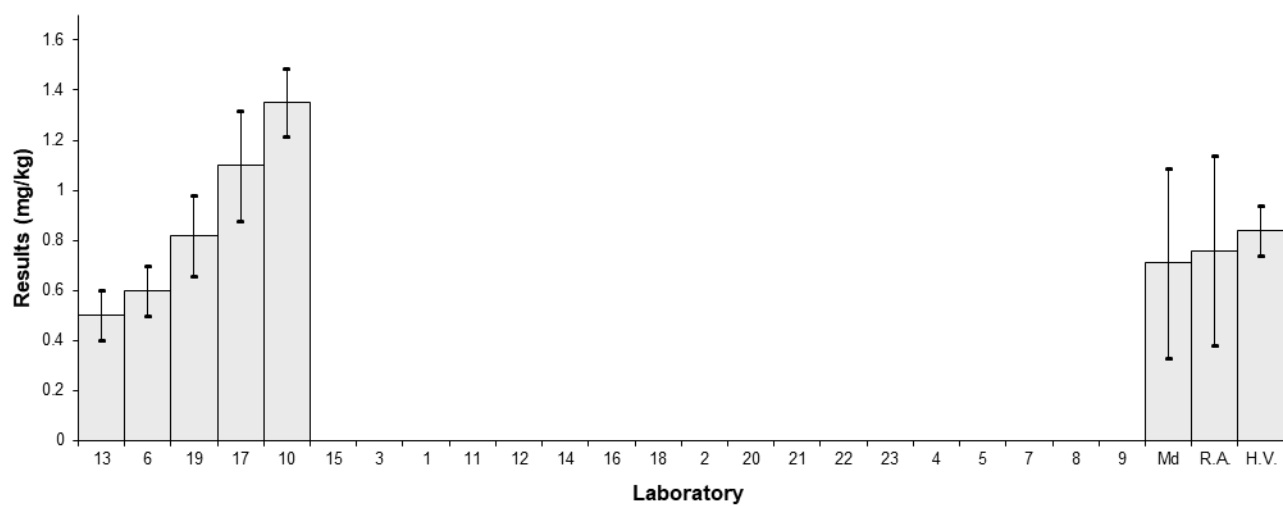


Figure 29

Table 41

Sample Details

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

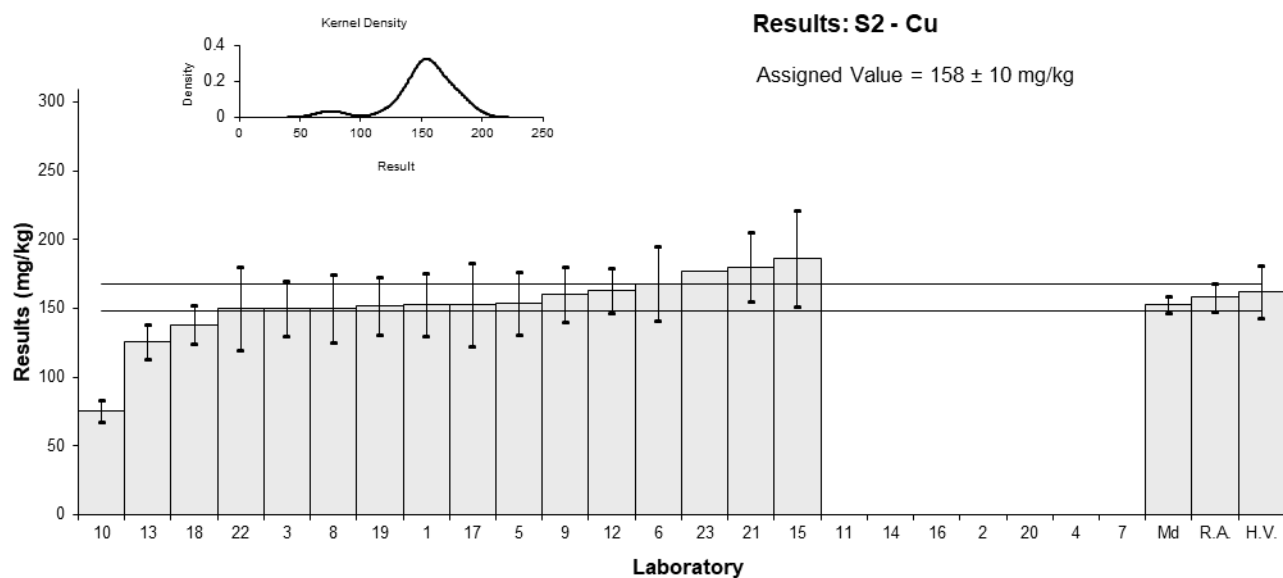
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	153	23	-0.32	-0.20
2	NT	NT		
3	150	20	-0.51	-0.36
4	NT	NT		
5	153.65	23.05	-0.28	-0.17
6	168	27	0.63	0.35
7	NT	NT		
8	150	25	-0.51	-0.30
9	160	20	0.13	0.09
10	75.5	7.55	-5.22	-6.58
11	NT	NT		
12	163	16	0.32	0.26
13	126	12.6	-2.03	-1.99
14	NT	NT		
15	186	35	1.77	0.77
16	NT	NT		
17	153	30	-0.32	-0.16
18	138.3	13.8	-1.25	-1.16
19	152	21	-0.38	-0.26
20	NT	NT		
21	180	25.2	1.39	0.81
22	150	30	-0.51	-0.25
23	177	NR	1.20	1.90

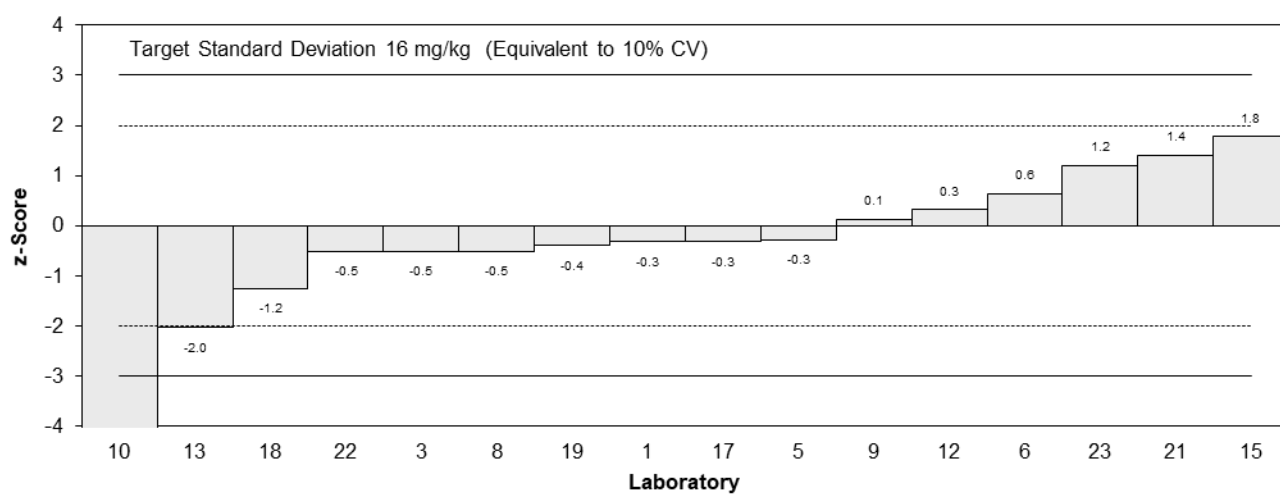
Statistics*

Assigned Value	158	10
Spike	Not Spiked	
Homogeneity Value	162	19
Robust Average	158	10
Median	153	6
Mean	157	
N	15	
Max.	186	
Min.	126	
Robust SD	16	
Robust CV	10%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.



z-Scores: S2 - Cu



En-Scores: S2 - Cu

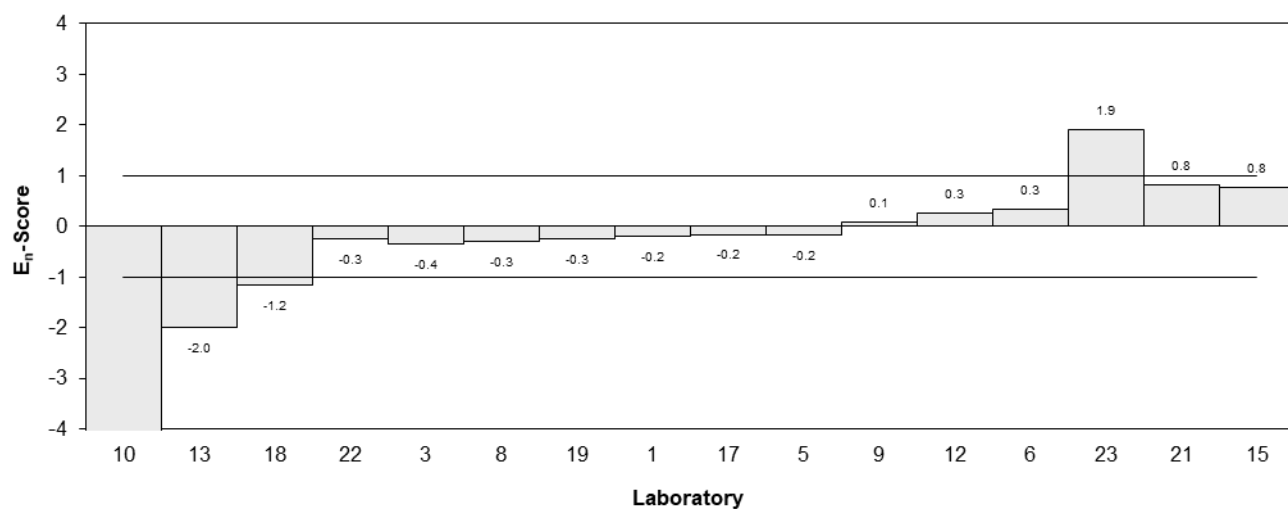


Figure 30

Table 42

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.54	0.1	0.93	0.58
2	NT	NT		
3	0.50	0.060	0.37	0.32
4	NT	NT		
5	0.47	0.07	-0.06	-0.05
6	0.6	0.1	1.77	1.11
7	NT	NT		
8	0.5	0.080	0.37	0.27
9	0.36	0.06	-1.60	-1.42
10	0.75	0.075	3.88	3.01
11	NT	NT		
12	0.58	0.08	1.49	1.10
13	0.4	0.08	-1.04	-0.77
14	NT	NT		
15	0.42	0.1	-0.76	-0.48
16	NT	NT		
17	0.47	0.09	-0.06	-0.04
18	0.505	0.05	0.44	0.43
19	0.50	0.11	0.37	0.21
20	NT	NT		
21	0.4	0.012	-1.04	-1.36
22	0.5	0.1	0.37	0.23
23	0.367	NR	-1.50	-2.02

Statistics*

Assigned Value	0.474	0.053
Spike	Not Spiked	
Homogeneity Value	0.467	0.056
Robust Average	0.474	0.053
Median	0.500	0.033
Mean	0.474	
N	15	
Max.	0.6	
Min.	0.36	
Robust SD	0.082	
Robust CV	17%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

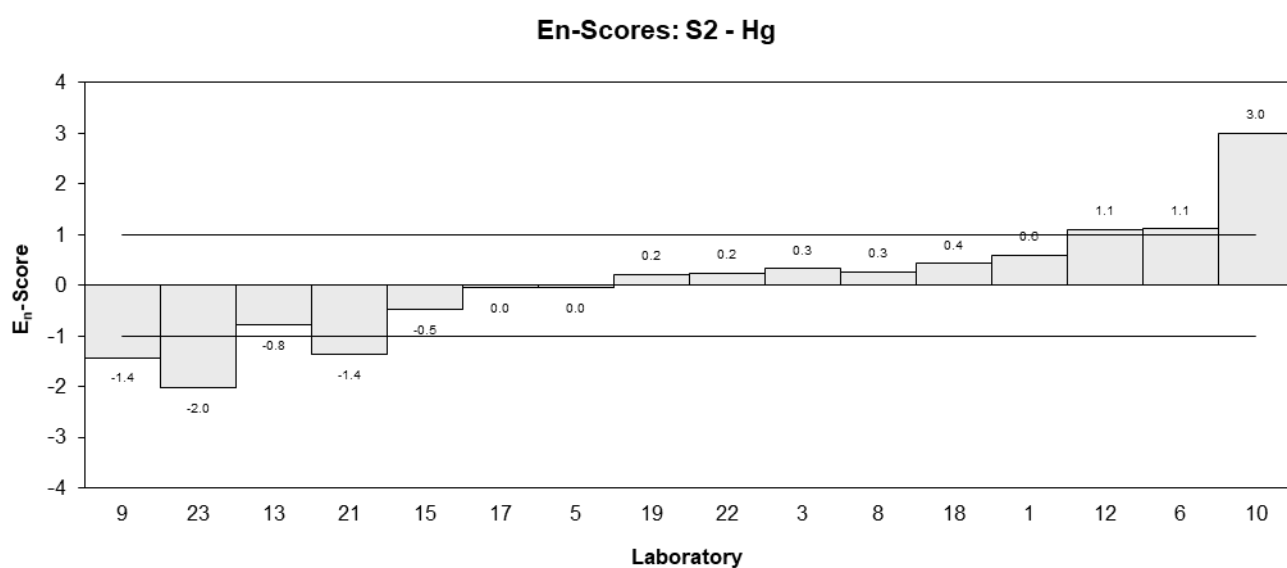
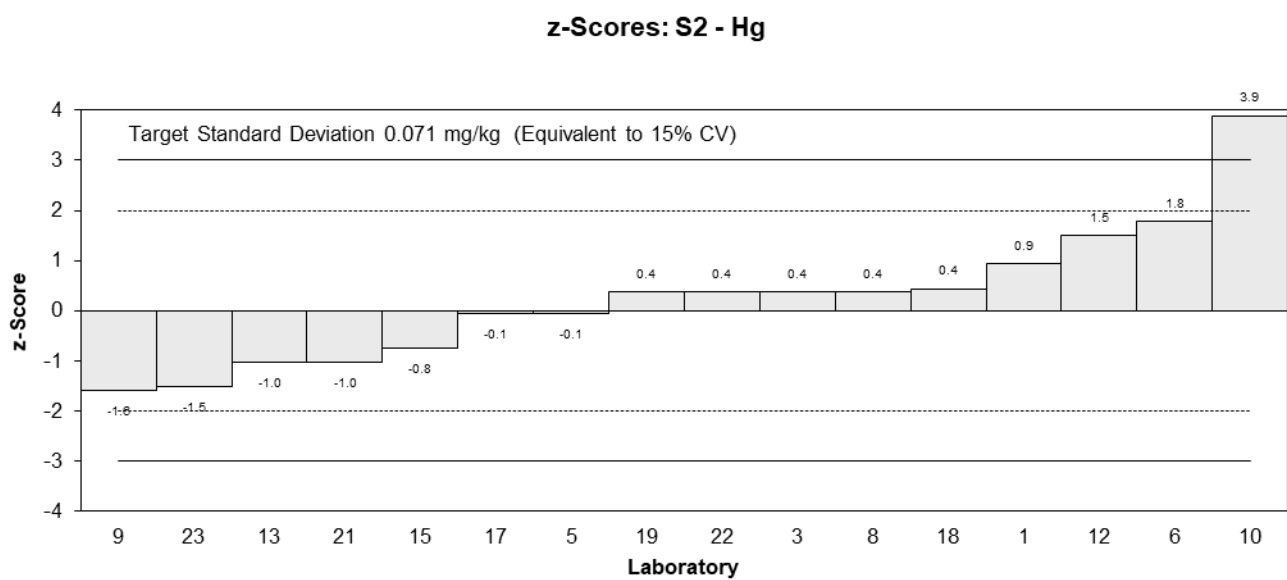
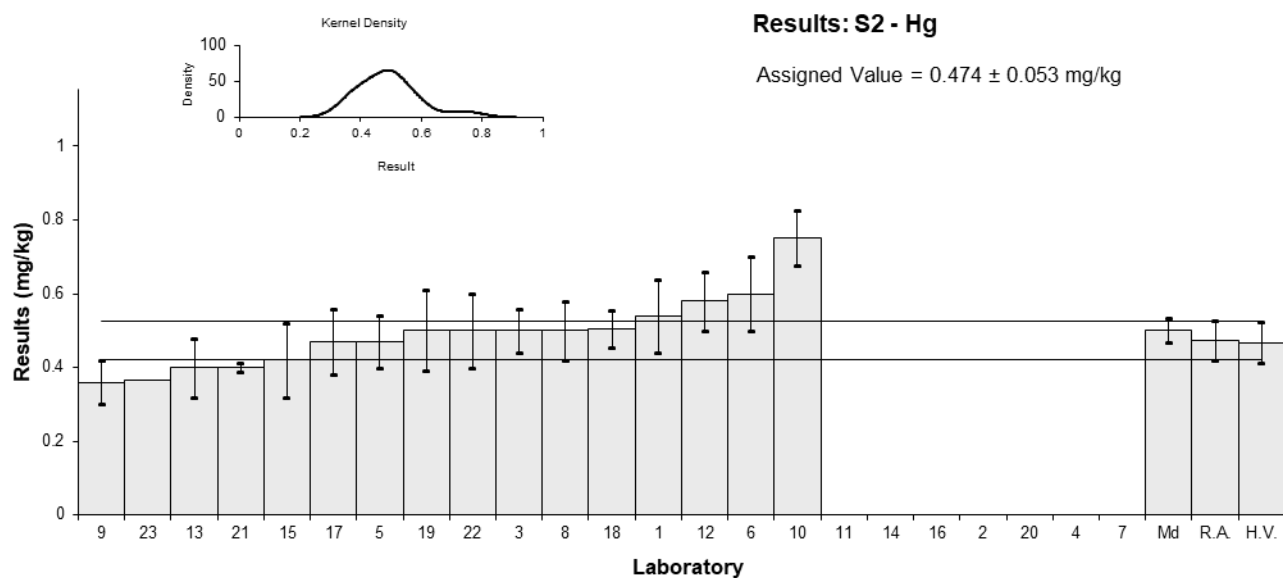


Figure 31

Table 43

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	13.4	2	1.10	0.58
2	NT	NT		
3	NR	NR		
4	NT	NT		
5	NT	NT		
6	11.0	1.6	-0.29	-0.16
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	18.9	1.89	4.29	2.30
11	NT	NT		
12	NT	NT		
13	10.1	2.02	-0.81	-0.43
14	NT	NT		
15	NR	NR		
16	NT	NT		
17	14	2.8	1.45	0.65
18	NT	NT		
19	12.4	1.0	0.52	0.32
20	NT	NT		
21	NT	NT		
22	NT	NT		
23	8.0	NR	-2.03	-1.35

Statistics*

Assigned Value	11.5	2.6
Spike	Not Spiked	
Homogeneity Value	12.5	1.5
Robust Average	11.5	2.6
Median	11.7	2.6
Mean	11.5	
N	6	
Max.	14	
Min.	8	
Robust SD	2.5	
Robust CV	22%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

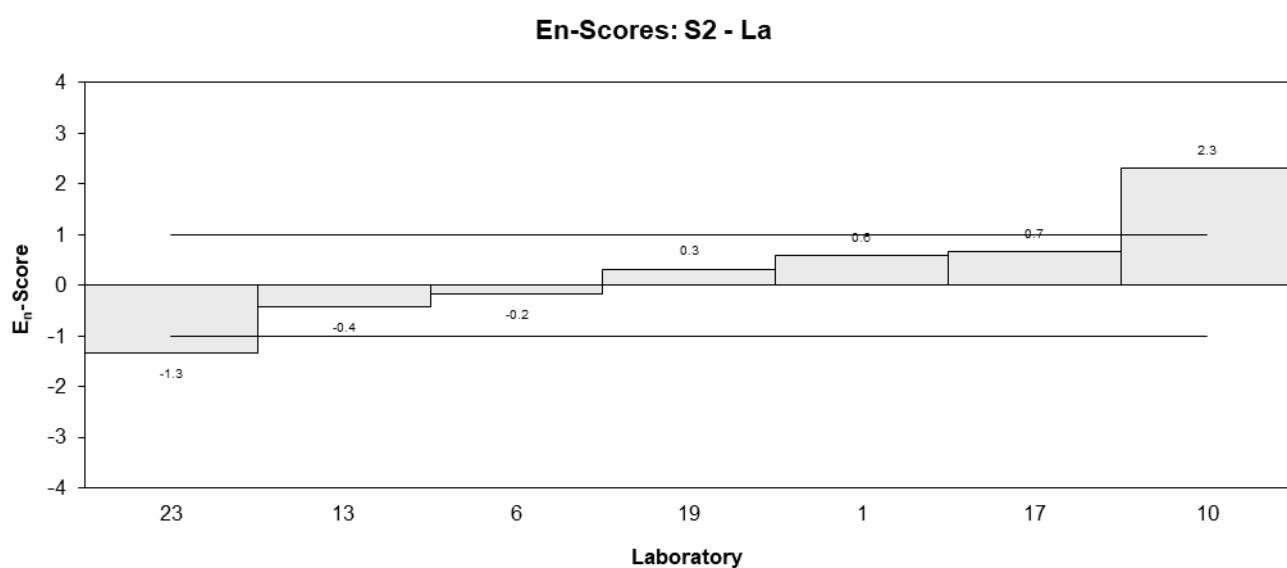
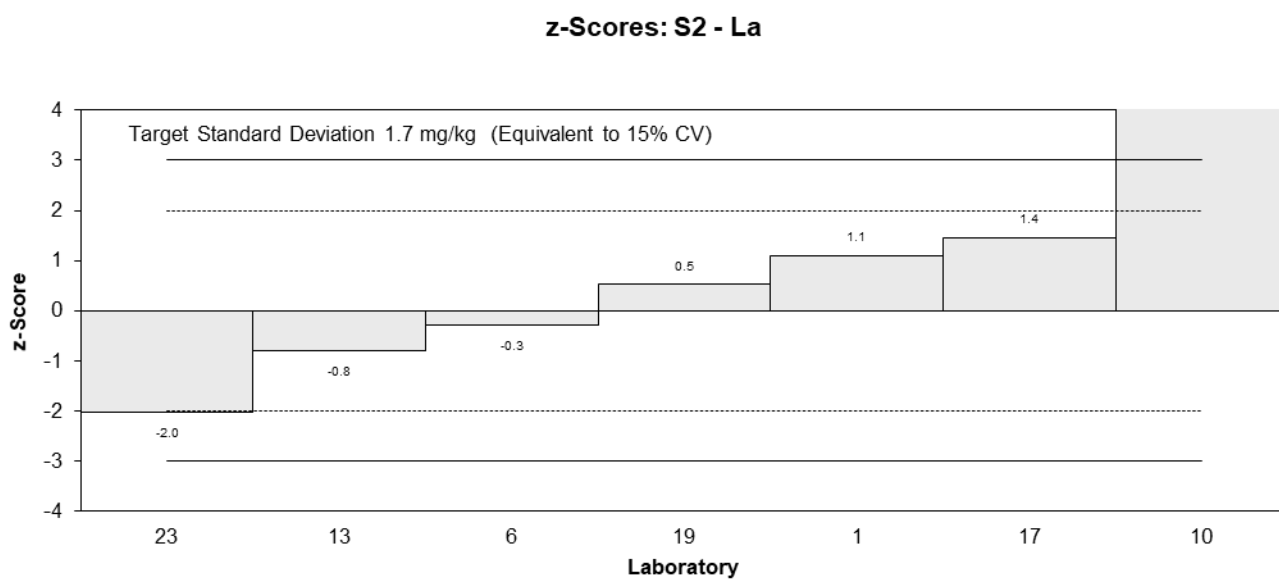
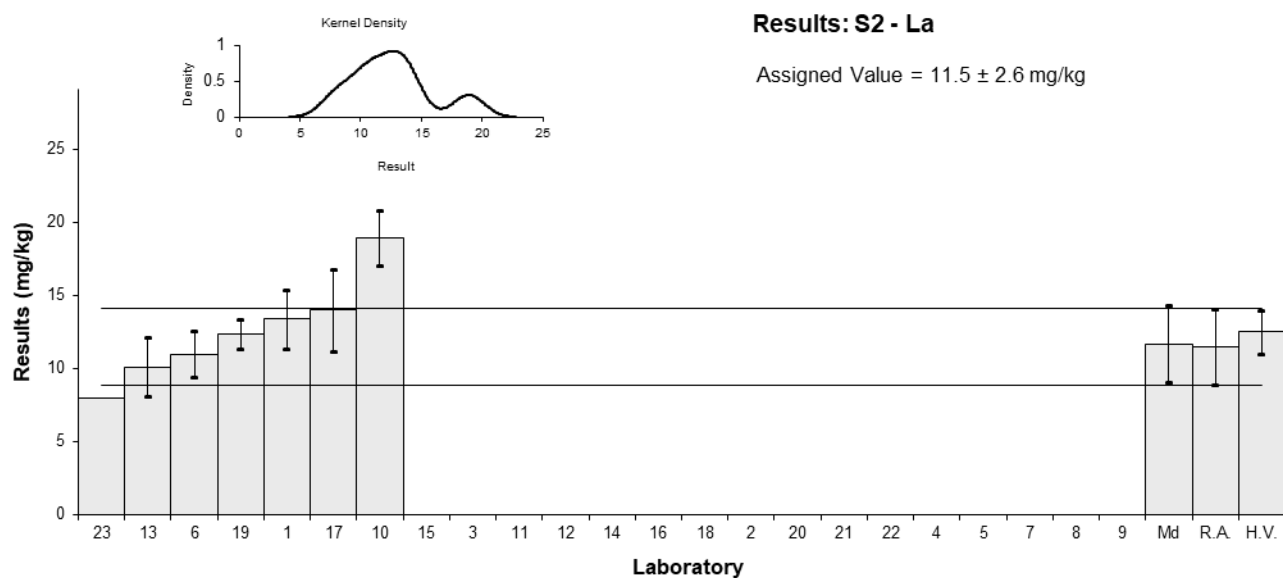


Figure 32

Table 44

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1.58	0.2	0.97	0.67
2	NT	NT		
3	< 5	NR		
4	NT	NT		
5	1.55	0.23	0.82	0.53
6	1.5	0.3	0.58	0.32
7	NT	NT		
8	<5	1		
9	1	0.13	-1.84	-1.49
10	0.99	0.099	-1.88	-1.62
11	NT	NT		
12	1.54	0.2	0.77	0.54
13	1.3	0.364	-0.39	-0.19
14	NT	NT		
15	NR	NR		
16	NT	NT		
17	1.5	0.30	0.58	0.32
18	0.851	0.085	-2.56	-2.24
19	1.48	0.37	0.48	0.23
20	NT	NT		
21	<5	NR		
22	<5	1		
23	0.34	NR	-5.02	-4.73

Statistics*

Assigned Value**	1.38	0.22
Spike	Not Spiked	
Homogeneity Value	1.37	0.16
Robust Average	1.31	0.28
Median	1.49	0.08
Mean	1.26	
N	10	
Max.	1.58	
Min.	0.851	
Robust SD	0.35	
Robust CV	27%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

**Robust Average excluding Laboratory 23.

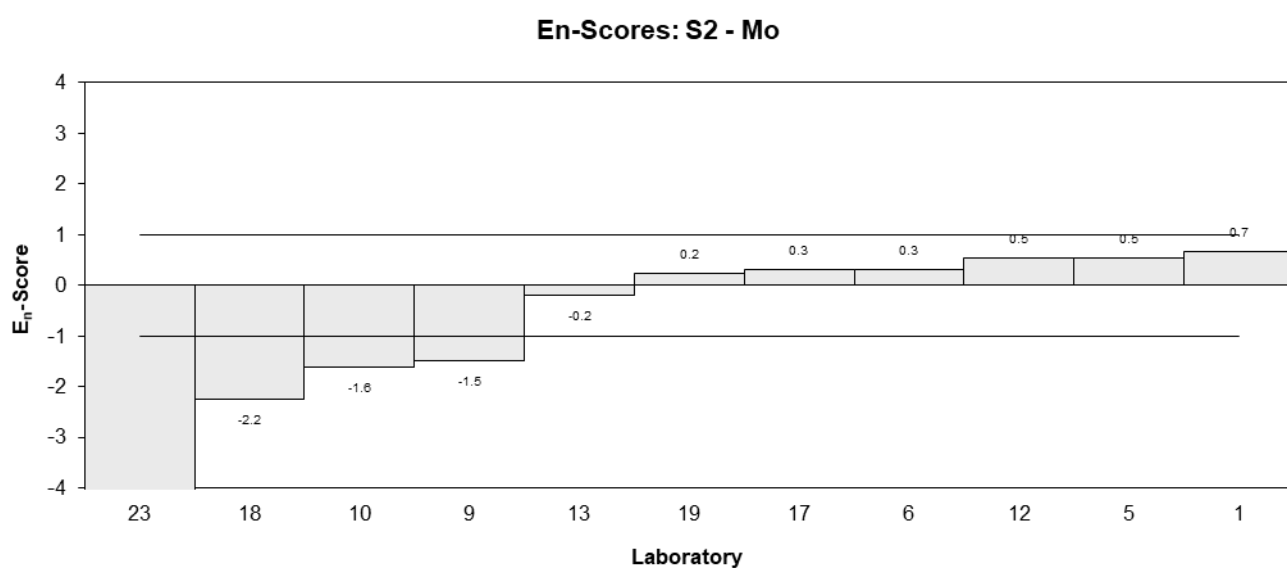
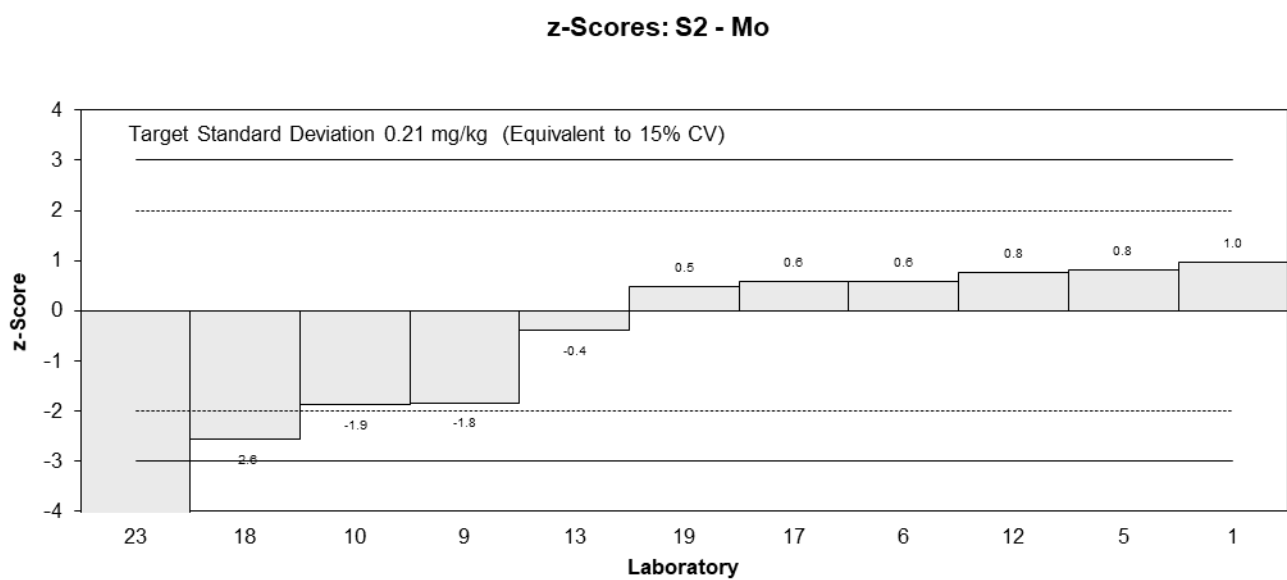
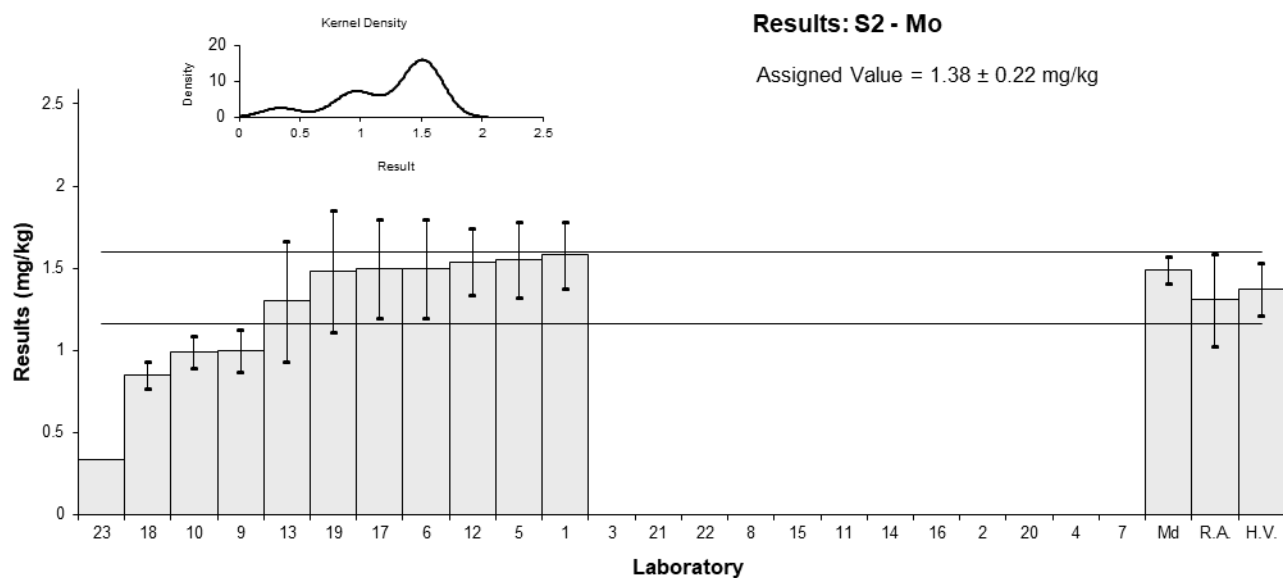


Figure 33

Table 45

Sample Details

Sample No.	S2
Matrix.	Sludge
Analyte.	Moisture Content
Units	%

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	41.2	5	-0.42	-0.34
2	NT	NT		
3	44	4.4	0.23	0.22
4	NT	NT		
5	48	7.20	1.16	0.68
6	43.0	2.0	0.00	0.00
7	NT	NT		
8	42	4.2	-0.23	-0.22
9	45	13.5	0.47	0.15
10	NT	NT		
11	NT	NT		
12	42.9	4.0	-0.02	-0.02
13	32.1	6.4	-2.53	-1.66
14	NT	NT		
15	43.5	35	0.12	0.01
16	NT	NT		
17	42.8	11	-0.05	-0.02
18	41.4	8.3	-0.37	-0.19
19	41	NR	-0.47	-1.33
20	NT	NT		
21	57	9	3.26	1.53
22	43	8.6	0.00	0.00
23	NR	NR		

Statistics

Assigned Value	43.0	1.5
Spike	42.0	0.8
Homogeneity Value	43.3	2.2
Robust Average	43.0	1.5
Median	43.0	1.1
Mean	43.4	
N	14	
Max.	57	
Min.	32.1	
Robust SD	2.2	
Robust CV	5.1%	

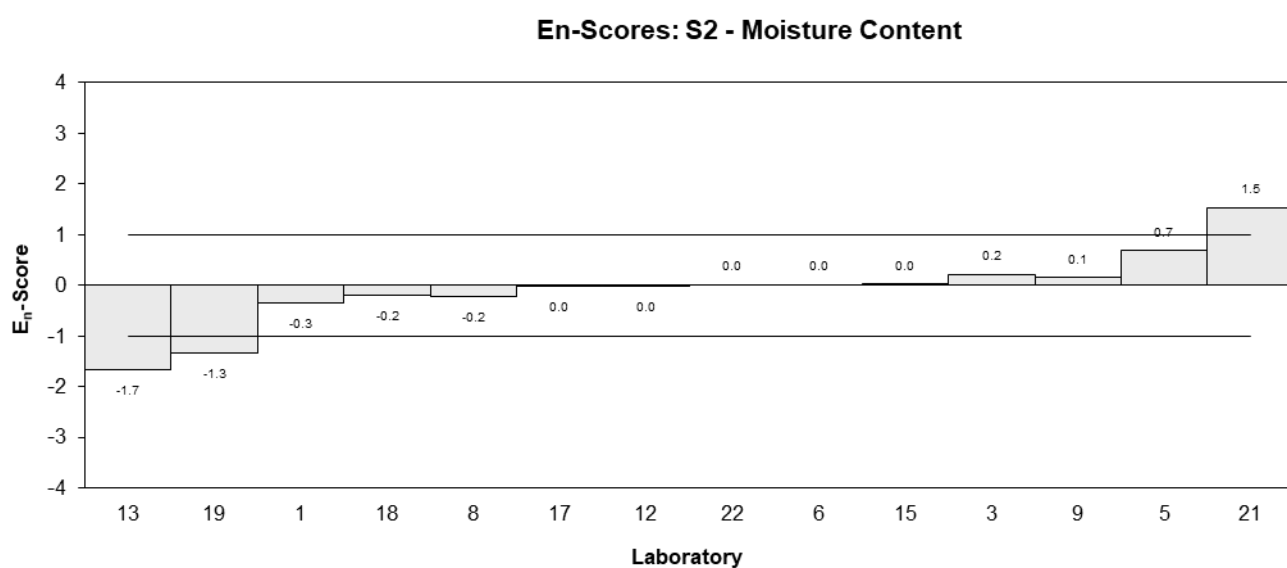
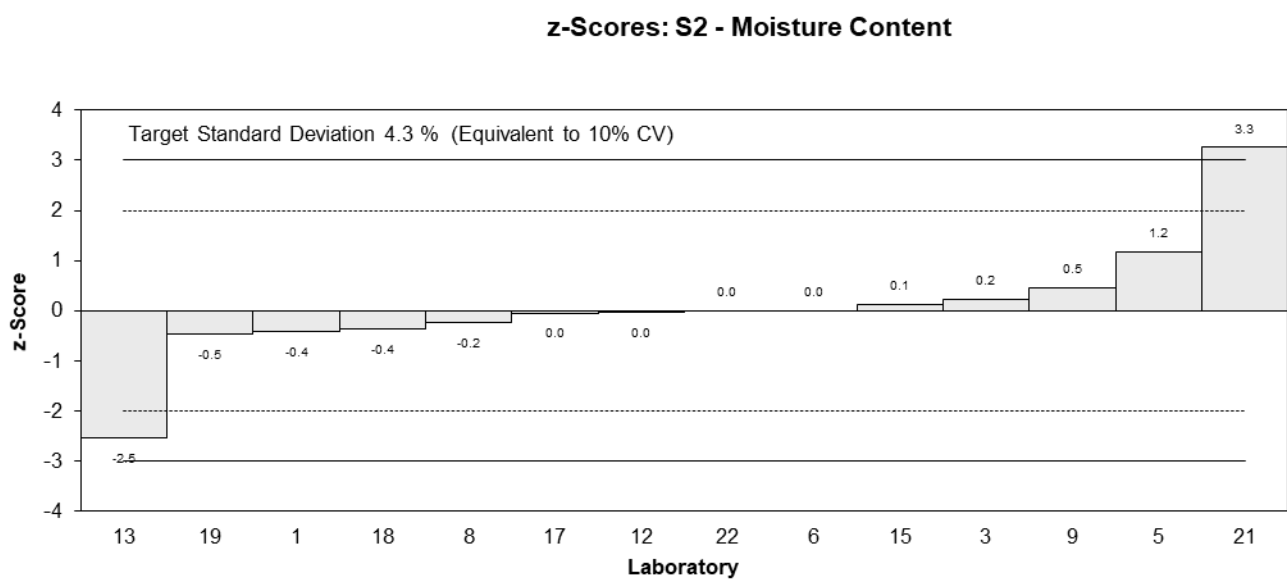
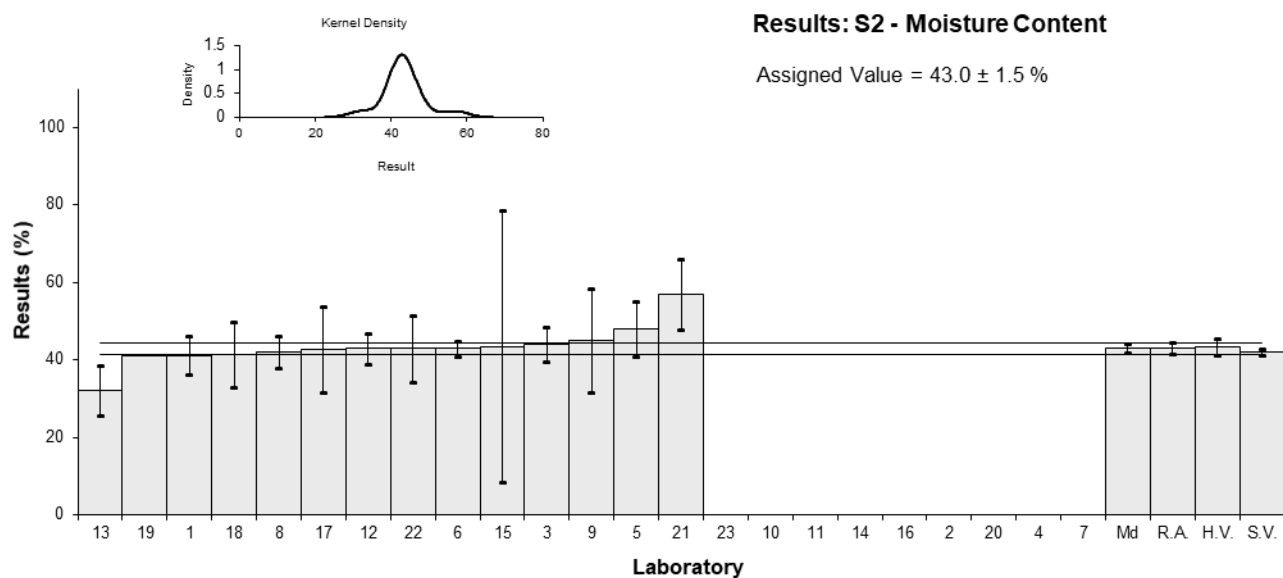


Figure 34

Table 46

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	15.5	2.3	-0.61	-0.37
2	NT	NT		
3	16	2.0	-0.30	-0.20
4	NT	NT		
5	15.19	2.28	-0.79	-0.49
6	15.4	2.5	-0.67	-0.38
7	NT	NT		
8	19	3.1	1.52	0.73
9	16	1.73	-0.30	-0.22
10	25.3	2.53	5.33	3.04
11	NT	NT		
12	17.5	2.0	0.61	0.41
13	13.6	0.87	-1.76	-1.76
14	NT	NT		
15	18	5	0.91	0.29
16	NT	NT		
17	18	3.6	0.91	0.39
18	71.4	7.14	33.27	7.55
19	15.1	2.4	-0.85	-0.50
20	NT	NT		
21	20	1.8	2.12	1.53
22	18	3.6	0.91	0.39
23	14	NR	-1.52	-1.79

Statistics*

Assigned Value	16.5	1.4
Spike	Not Spiked	
Homogeneity Value	17.0	2.0
Robust Average	16.5	1.4
Median	16.0	1.5
Mean	16.5	
N	14	
Max.	25.3	
Min.	13.6	
Robust SD	2.1	
Robust CV	13%	

*Laboratories 18 excluded from statistical calculation (extreme outlier). Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

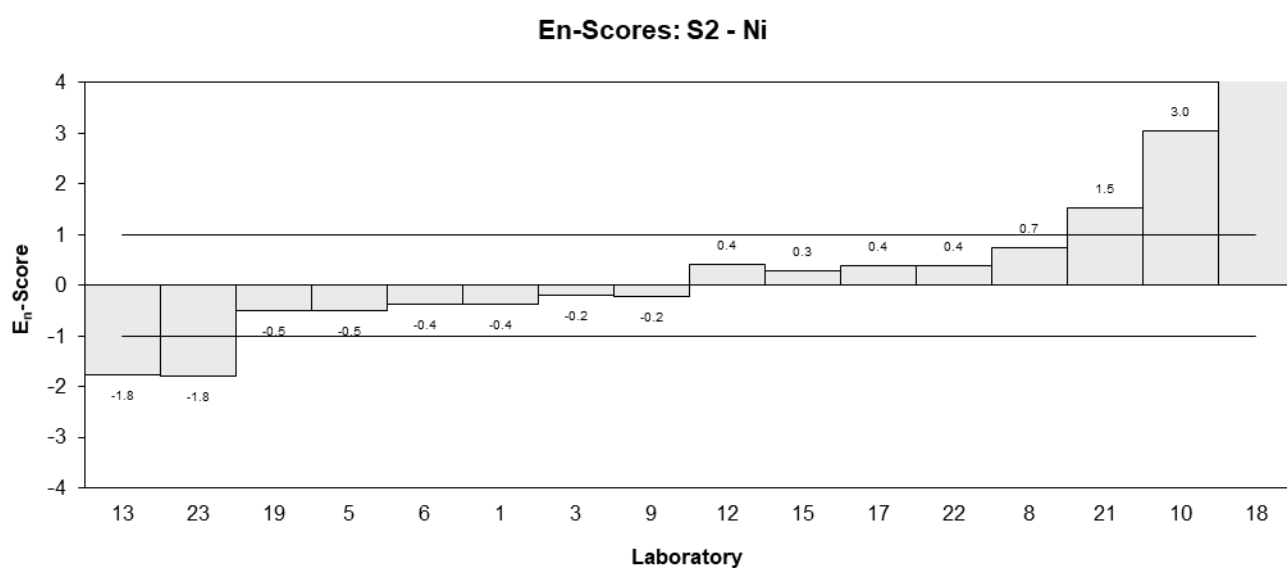
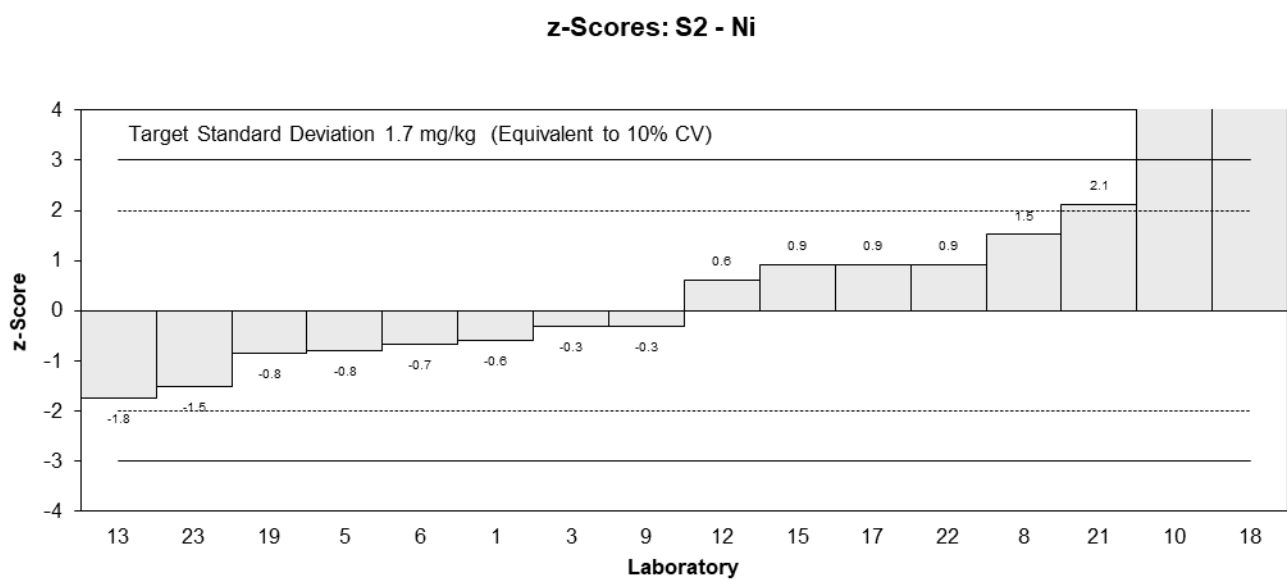
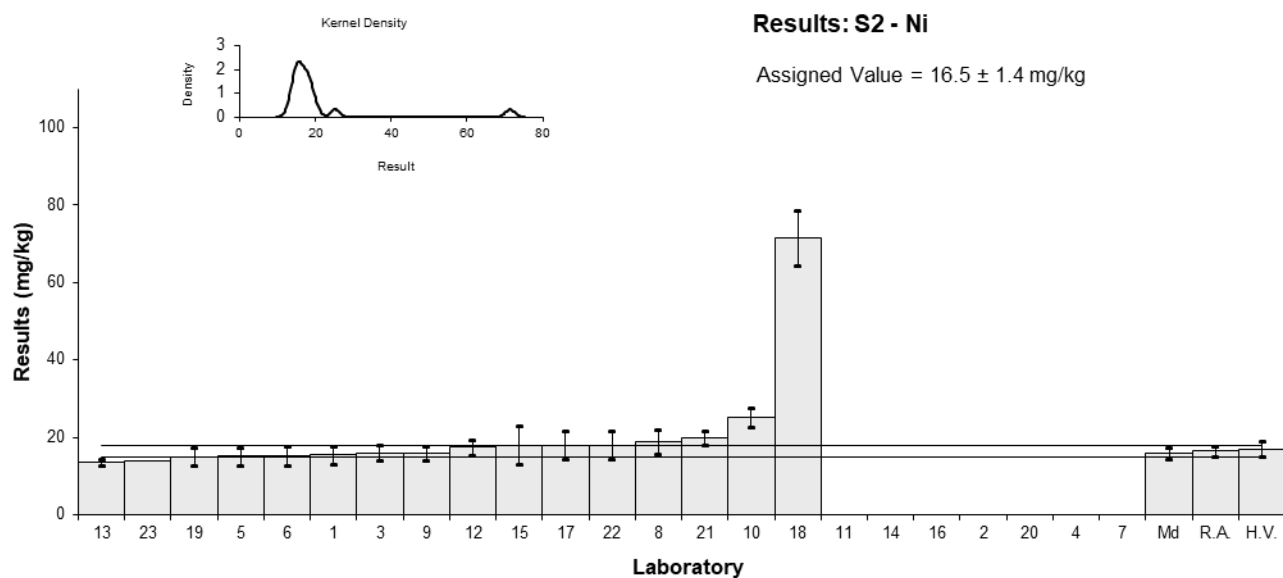


Figure 35

Table 47

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	32.7	5	0.22	0.13
2	NT	NT		
3	32	4.2	0.00	0.00
4	NT	NT		
5	32.9	4.94	0.28	0.17
6	38.2	7.4	1.94	0.81
7	NT	NT		
8	32	4.9	0.00	0.00
9	34	4.63	0.62	0.40
10	42.8	4.28	3.37	2.31
11	NT	NT		
12	31.6	3.5	-0.12	-0.10
13	25.8	1.52	-1.94	-2.55
14	NT	NT		
15	33	8	0.31	0.12
16	NT	NT		
17	31	6.2	-0.31	-0.15
18	28.02	2.80	-1.24	-1.18
19	32.9	5.0	0.28	0.17
20	NT	NT		
21	37	3.7	1.56	1.20
22	30	6	-0.62	-0.32
23	29	NR	-0.94	-1.58

Statistics*

Assigned Value	32.0	1.9
Spike	Not Spiked	
Homogeneity Value	26.7	3.2
Robust Average	32.0	1.9
Median	32.0	0.8
Mean	32.0	
N	15	
Max.	38.2	
Min.	25.8	
Robust SD	3.0	
Robust CV	9.3%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

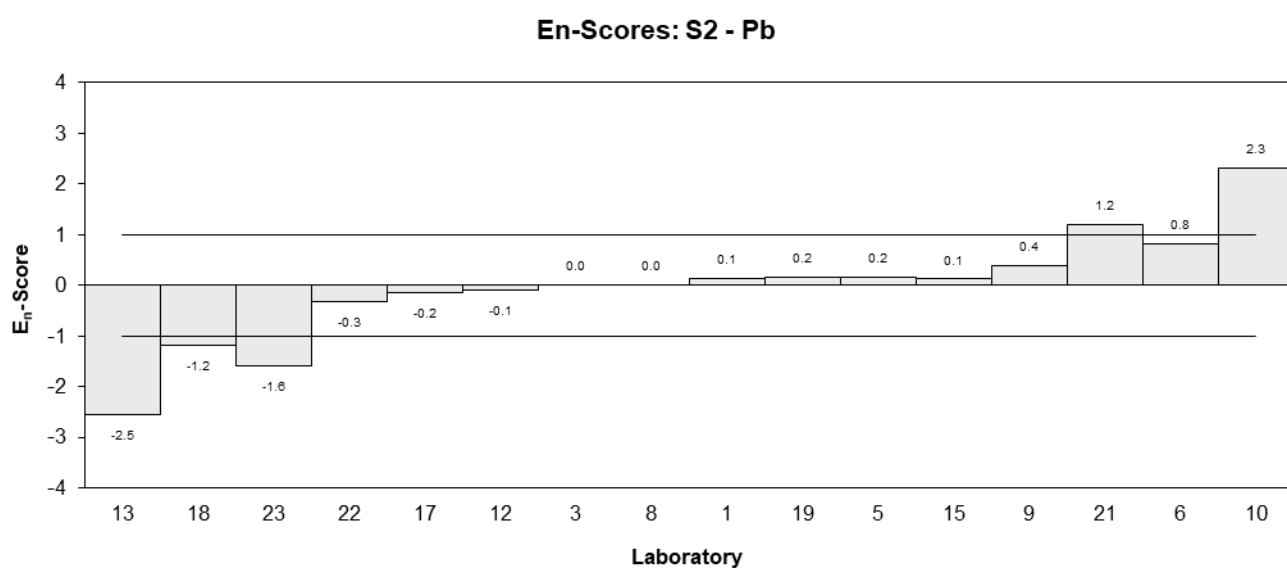
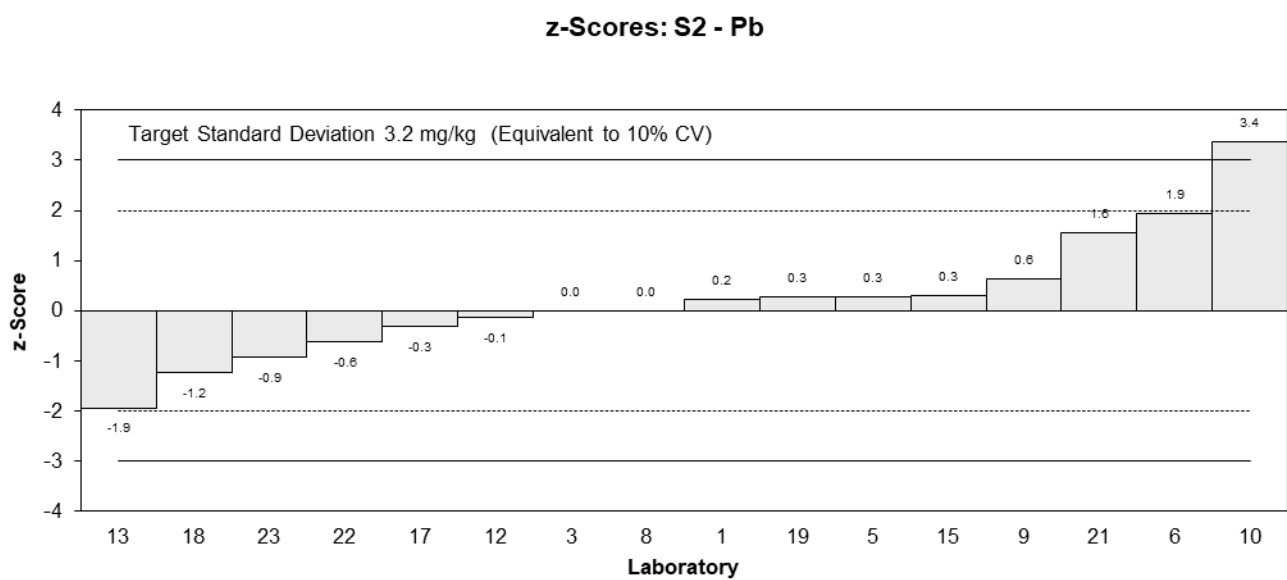
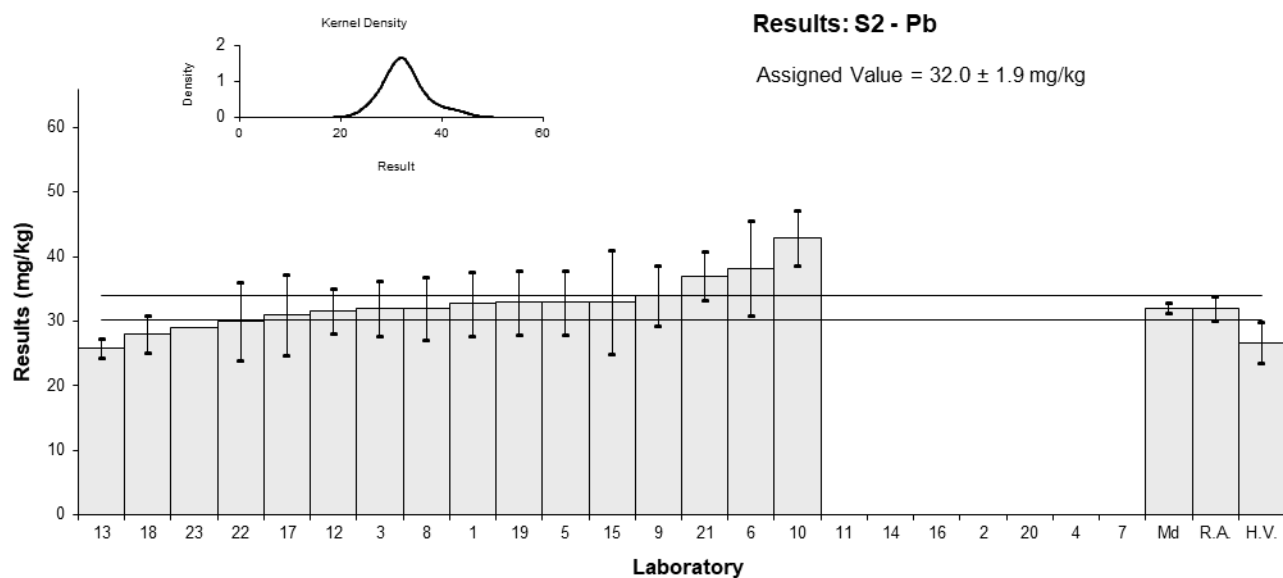


Figure 36

Table 48

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1.11	0.2	-0.30	-0.20
2	NT	NT		
3	< 2	NR		
4	NT	NT		
5	0.87	0.13	-1.31	-1.00
6	1	0.2	-0.76	-0.52
7	NT	NT		
8	<2	0.4		
9	<3	3		
10	10.3	1.03	38.64	8.54
11	NT	NT		
12	1.74	0.4	2.37	1.15
13	1	0.1	-0.76	-0.61
14	NT	NT		
15	<100	NR		
16	NT	NT		
17	1.3	0.26	0.51	0.31
18	1.37	0.137	0.81	0.61
19	< 20	14		
20	NT	NT		
21	<5	NR		
22	<2	0.4		
23	NT	NT		

Statistics*

Assigned Value	1.18	0.28
Spike	Not Spiked	
Homogeneity Value	1.25	0.15
Robust Average	1.18	0.28
Median	1.11	0.26
Mean	1.20	
N	7	
Max.	1.74	
Min.	0.87	
Robust SD	0.30	
Robust CV	25%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

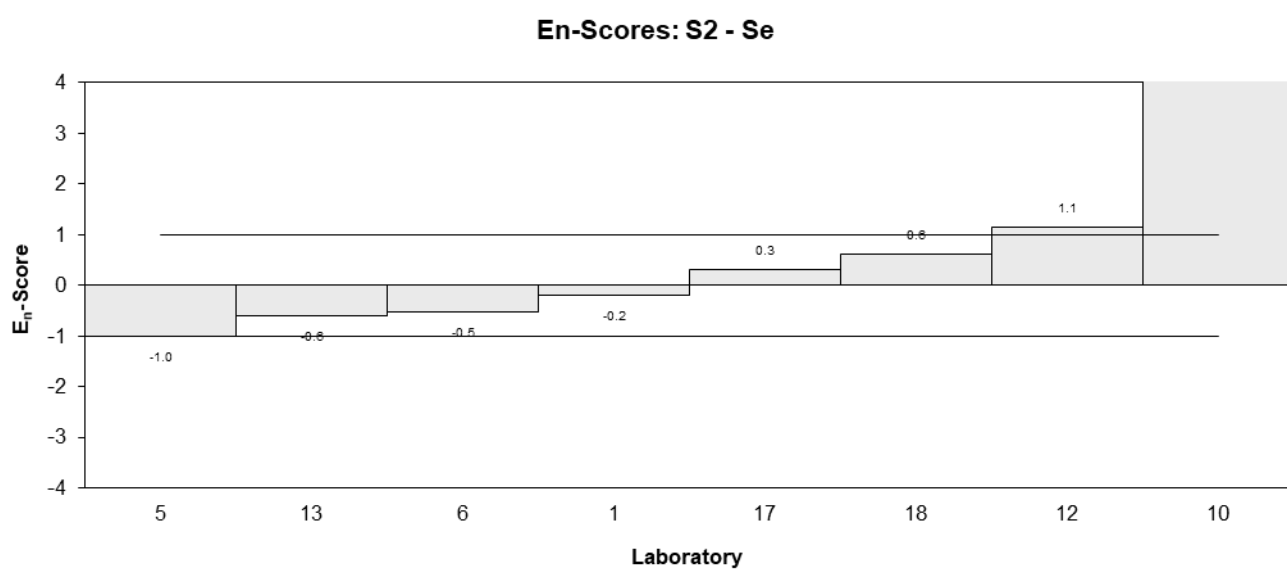
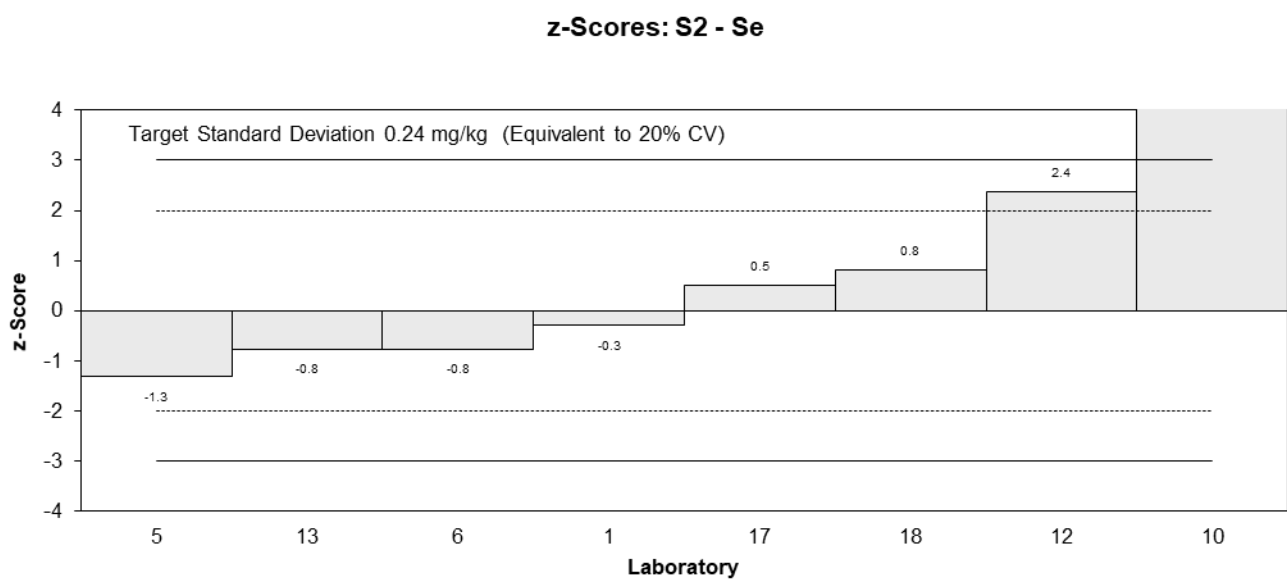
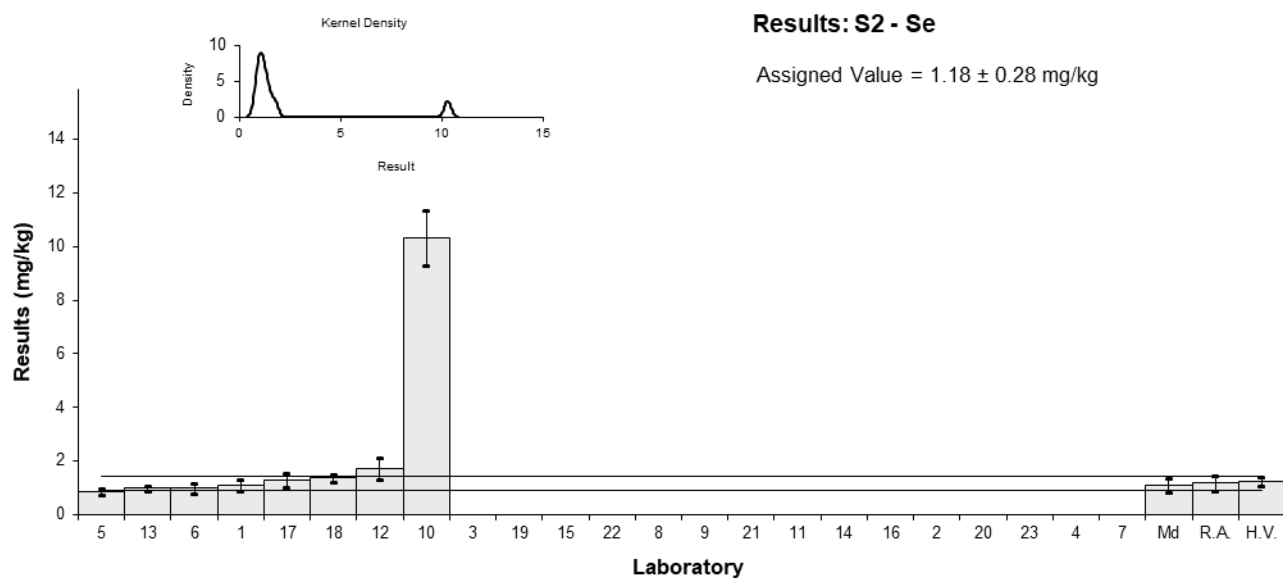


Figure 37

Table 49

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1.15	0.2	0.06	0.04
2	NT	NT		
3	< 10	NR		
4	NT	NT		
5	NT	NT		
6	1.4	0.4	1.52	0.60
7	NT	NT		
8	1.2	0.18	0.35	0.25
9	NT	NT		
10	1.11	0.111	-0.18	-0.15
11	NT	NT		
12	1.18	0.2	0.23	0.16
13	0.8	0.16	-1.99	-1.50
14	NT	NT		
15	NR	NR		
16	NT	NT		
17	1.2	0.24	0.35	0.21
18	0.971	0.09	-0.99	-0.92
19	1.122	0.095	-0.11	-0.10
20	NT	NT		
21	NT	NT		
22	<10	2		
23	NT	NT		

Statistics*

Assigned Value	1.14	0.16
Spike	Not Spiked	
Homogeneity Value	1.02	0.12
Robust Average	1.14	0.16
Median	1.17	0.05
Mean	1.13	
N	8	
Max.	1.4	
Min.	0.8	
Robust SD	0.18	
Robust CV	16%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.

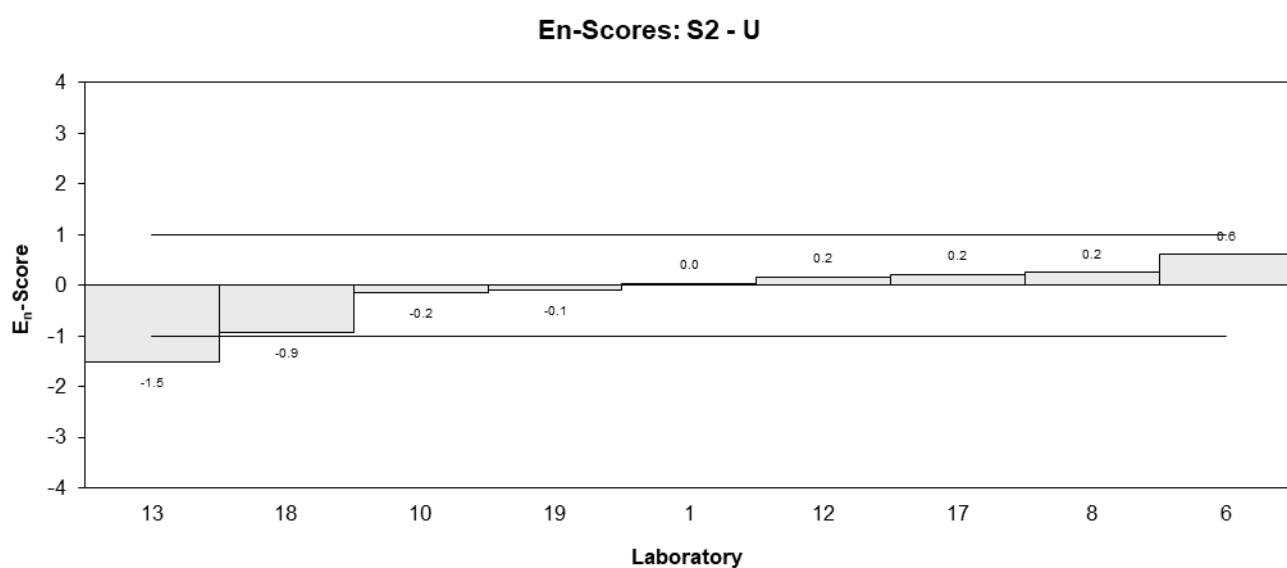
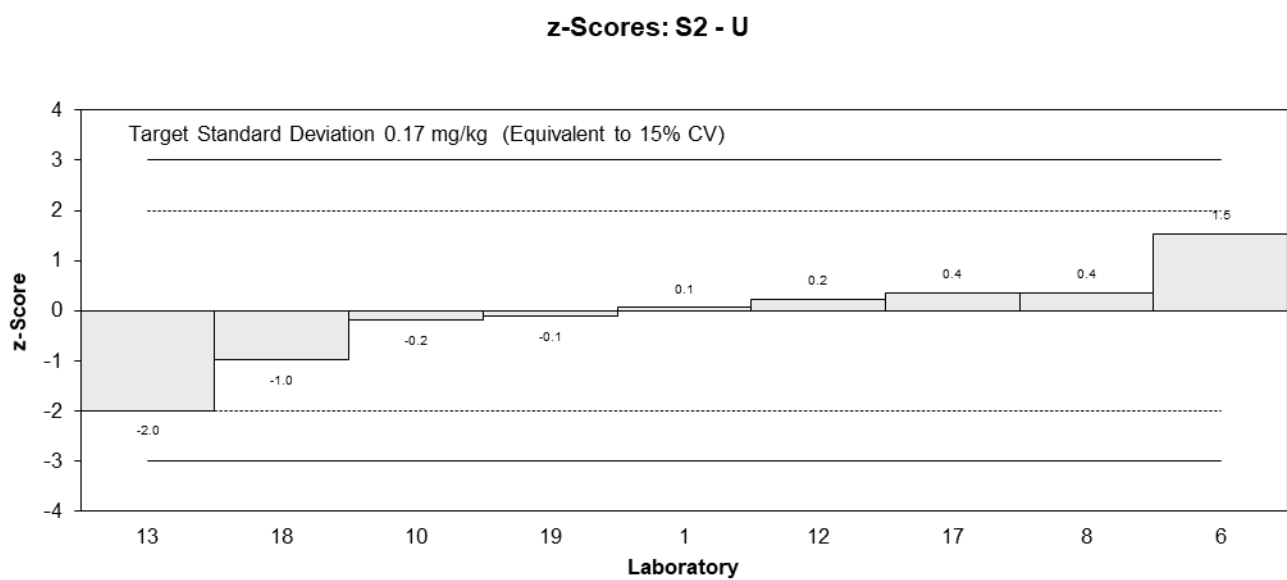
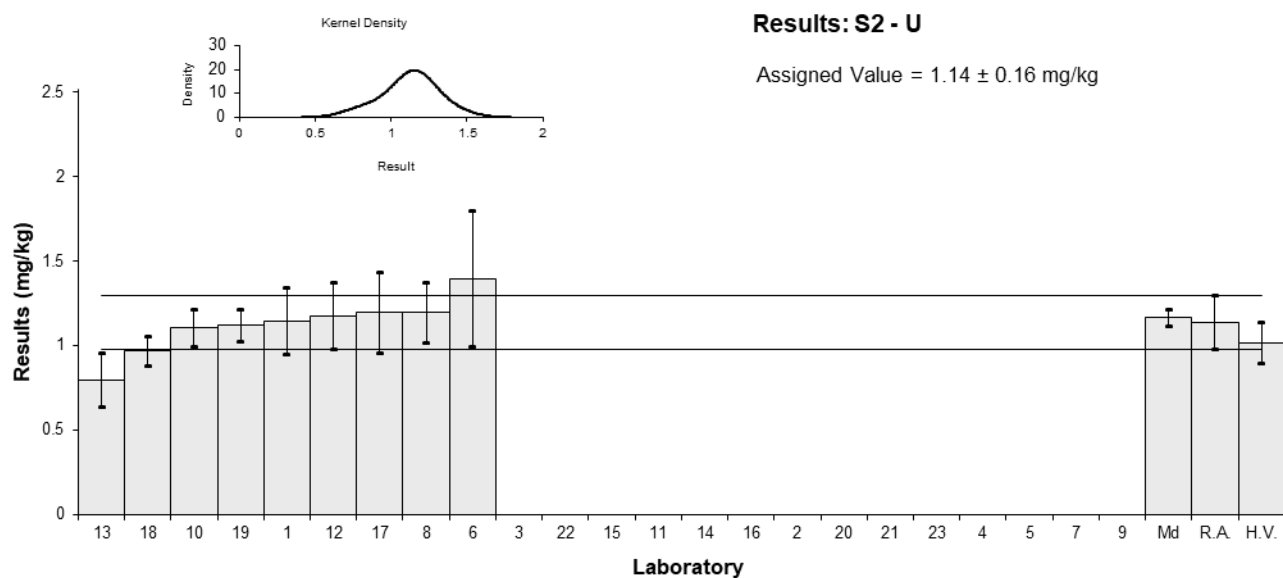


Figure 38

Table 50

Sample Details

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

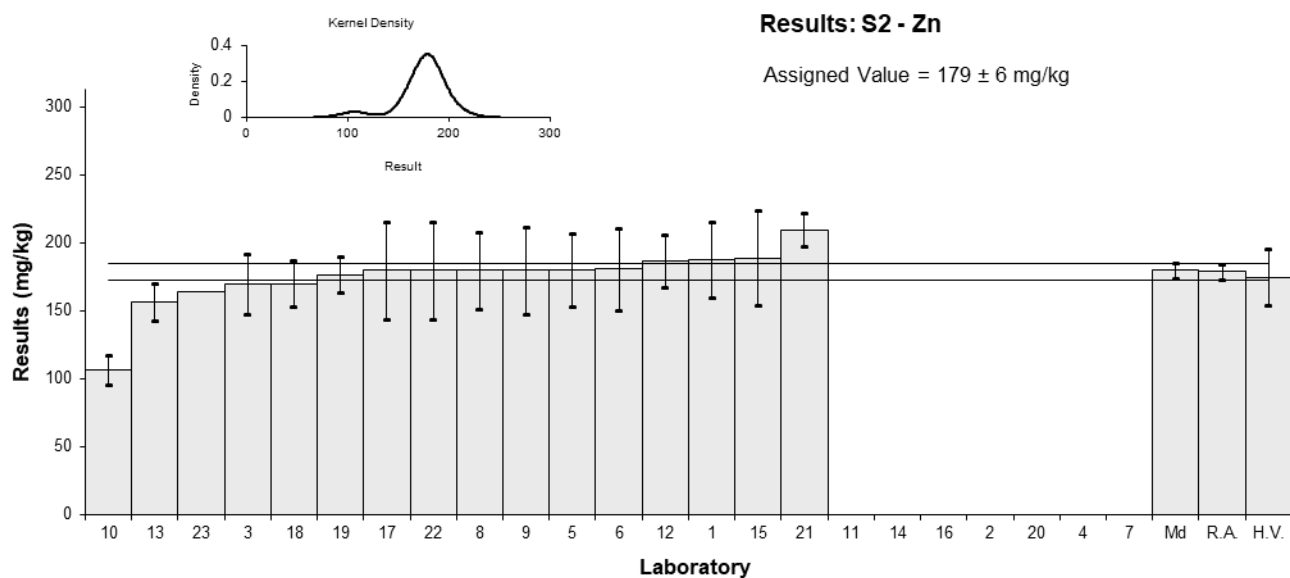
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	188	28	0.50	0.31
2	NT	NT		
3	170	22	-0.50	-0.39
4	NT	NT		
5	180.37	27.06	0.08	0.05
6	181	30	0.11	0.07
7	NT	NT		
8	180	28	0.06	0.03
9	180	32.4	0.06	0.03
10	107	10.7	-4.02	-5.87
11	NT	NT		
12	187	19	0.45	0.40
13	157	13.6	-1.23	-1.48
14	NT	NT		
15	189	35	0.56	0.28
16	NT	NT		
17	180	36	0.06	0.03
18	170.3	17.0	-0.49	-0.48
19	177	13	-0.11	-0.14
20	NT	NT		
21	210	12.6	1.73	2.22
22	180	36	0.06	0.03
23	164	NR	-0.84	-2.50

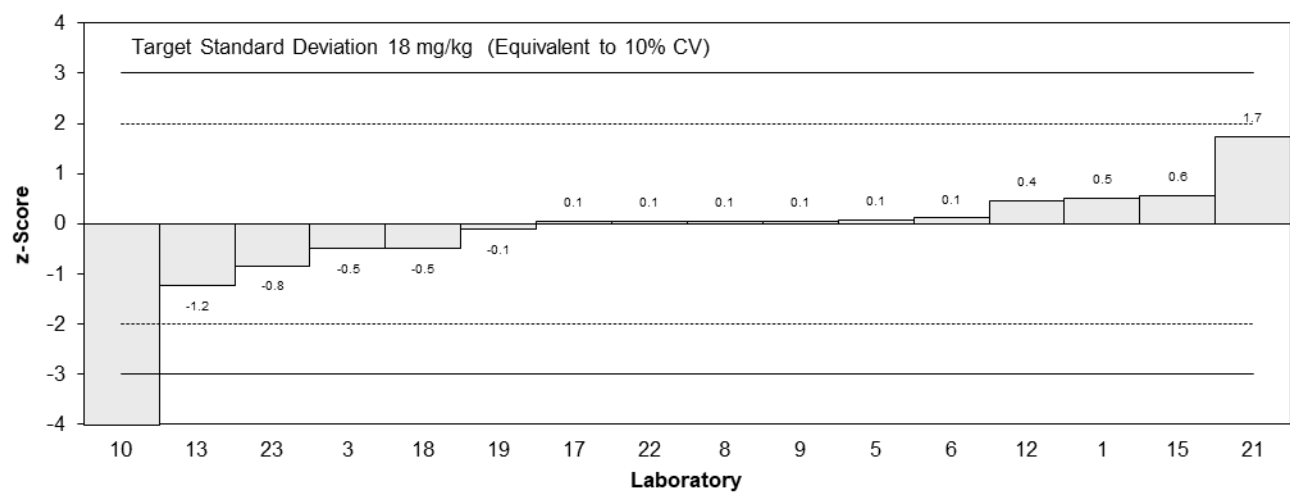
Statistics*

Assigned Value	179	6
Spike	Not Spiked	
Homogeneity Value	175	21
Robust Average	179	6
Median	180	6
Mean	180	
N	15	
Max.	210	
Min.	157	
Robust SD	10	
Robust CV	5.6%	

*Laboratory 10 was omitted from all statistical calculation as they mislabelled the samples.



z-Scores: S2 - Zn



En-Scores: S2 - Zn

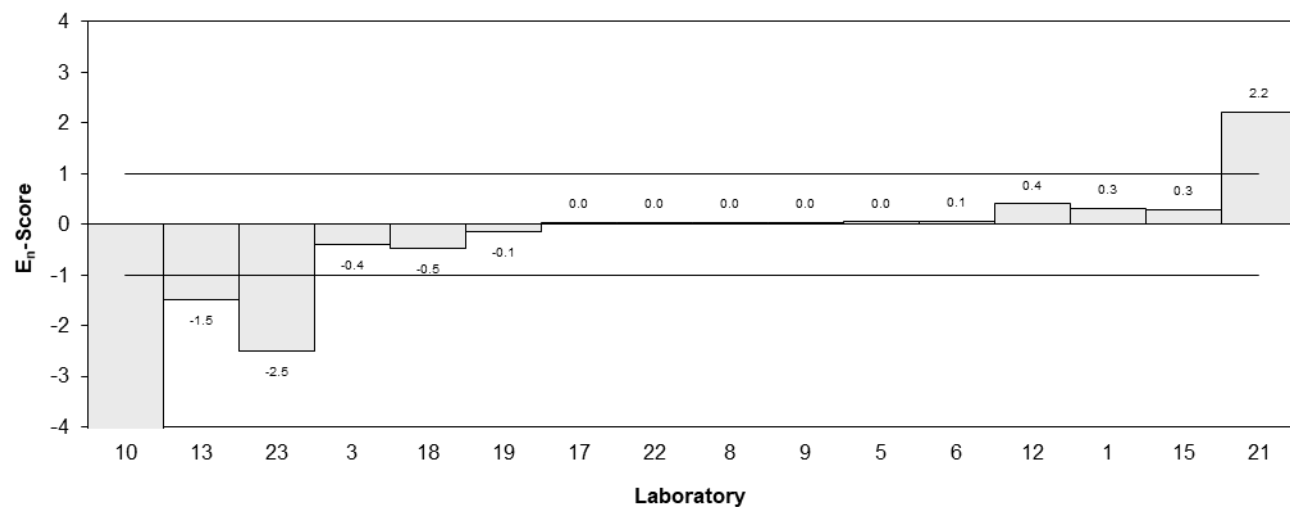


Figure 39

Table 51

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	494	74	0.33	0.21
2	401	60	-1.61	-1.19
3	NT	NT		
4	NT	NT		
5	498.10	74.72	0.42	0.26
6	470	9	-0.17	-0.31
7	NR	NR		
8	NT	NT		
9	480	61.44	0.04	0.03
10	NT	NT		
11	364	41.86	-2.38	-2.36
12	484	45	0.13	0.12
13	780	220	6.32	1.36
14	450	84	-0.59	-0.32
15	490	100	0.25	0.12
16	484	59	0.13	0.09
17	520	104	0.88	0.39
18	NT	NT		
19	NT	NT		
20	NR	NR		
21	510	61	0.67	0.49
22	NT	NT		
23	NT	NT		

Statistics

Assigned Value*	478	24
Spike	Not Spiked	
Homogeneity Value	485	58
Robust Average	482	27
Median	484	13
Mean	494	
N	13	
Max.	780	
Min.	364	
Robust SD	38	
Robust CV	8.0%	

*Robust Average excluding Laboratory 13.

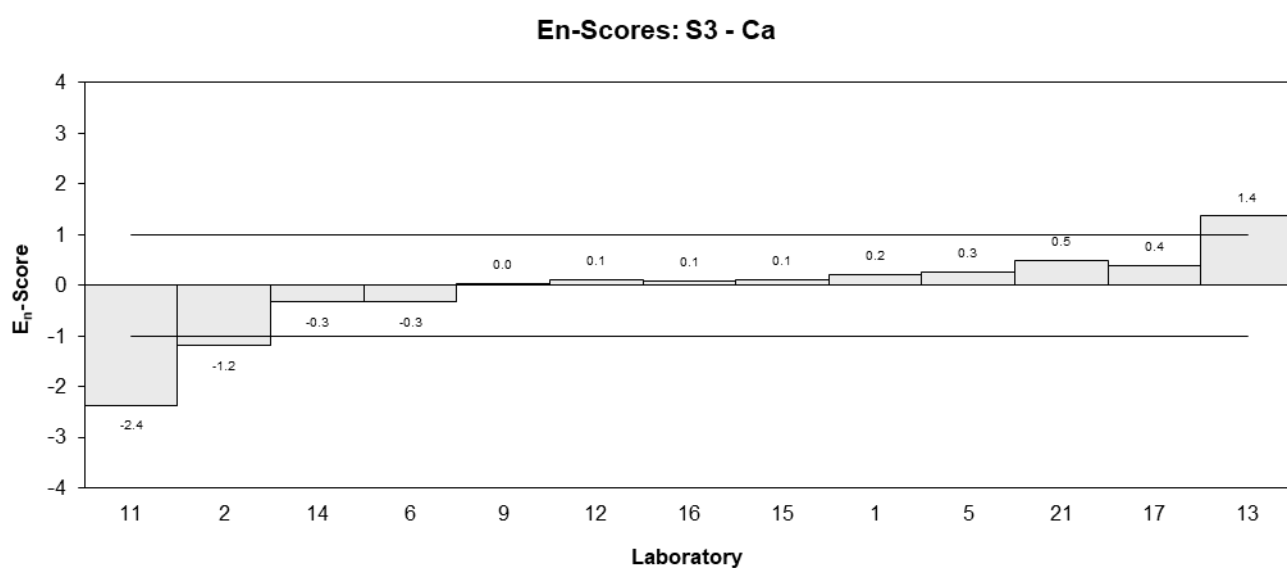
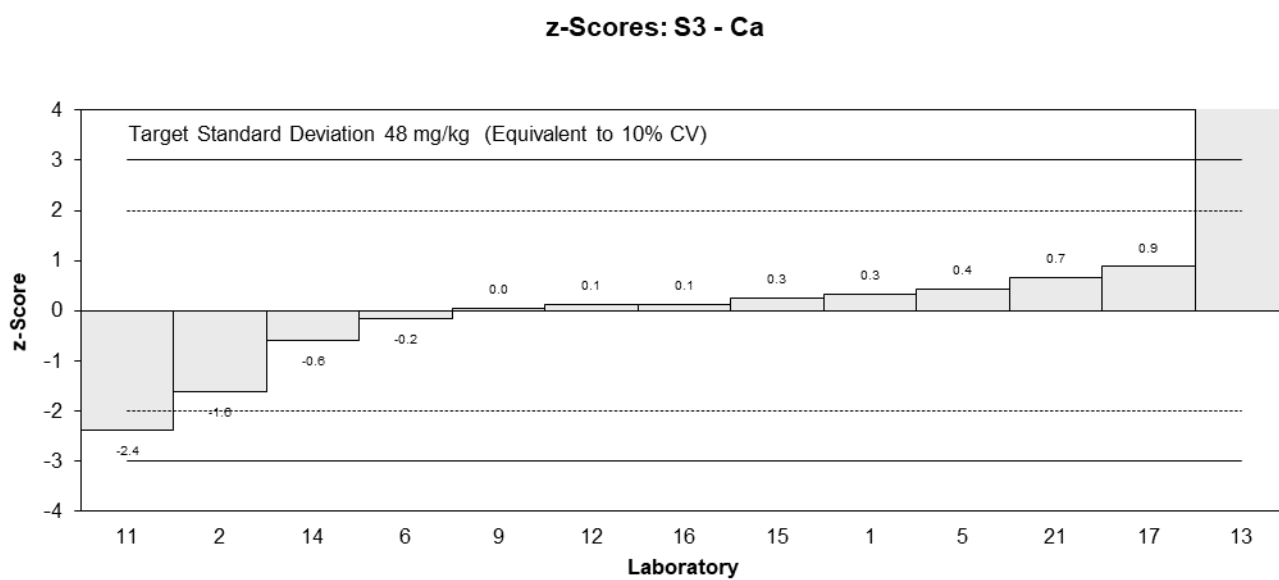
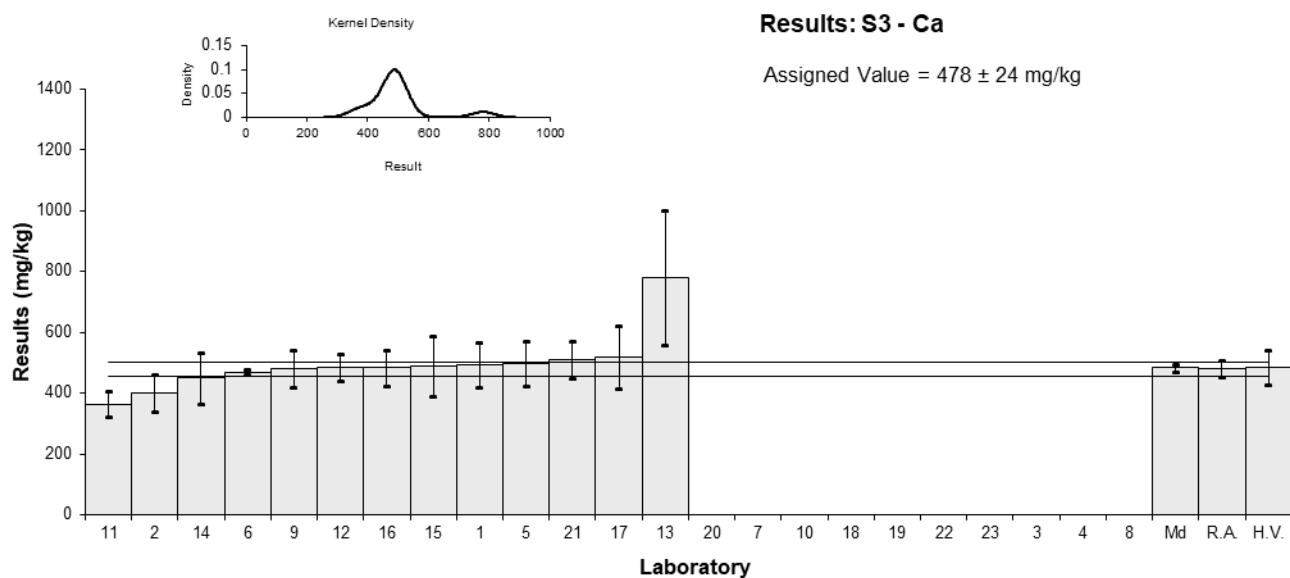


Figure 40

Table 52

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	32	13
3	NT	NT
4	NT	NT
5	NT	NT
6	<100	NR
7	NR	NR
8	NT	NT
9	NT	NT
10	NT	NT
11	38.4	3.23
12	31	3.0
13	NT	NT
14	NT	NT
15	NR	NR
16	NR	NR
17	NR	NR
18	NT	NT
19	NT	NT
20	NR	NR
21	NT	NT
22	NT	NT
23	NT	NT

Statistics

Assigned Value	Not Set	3.7
Spike	Not Spiked	
Median	32.0	
Mean	33.8	
N	3	
Max.	38.4	
Min.	31	
Robust SD	4.6	
Robust CV	13%	

Results: S3 - Colwell K

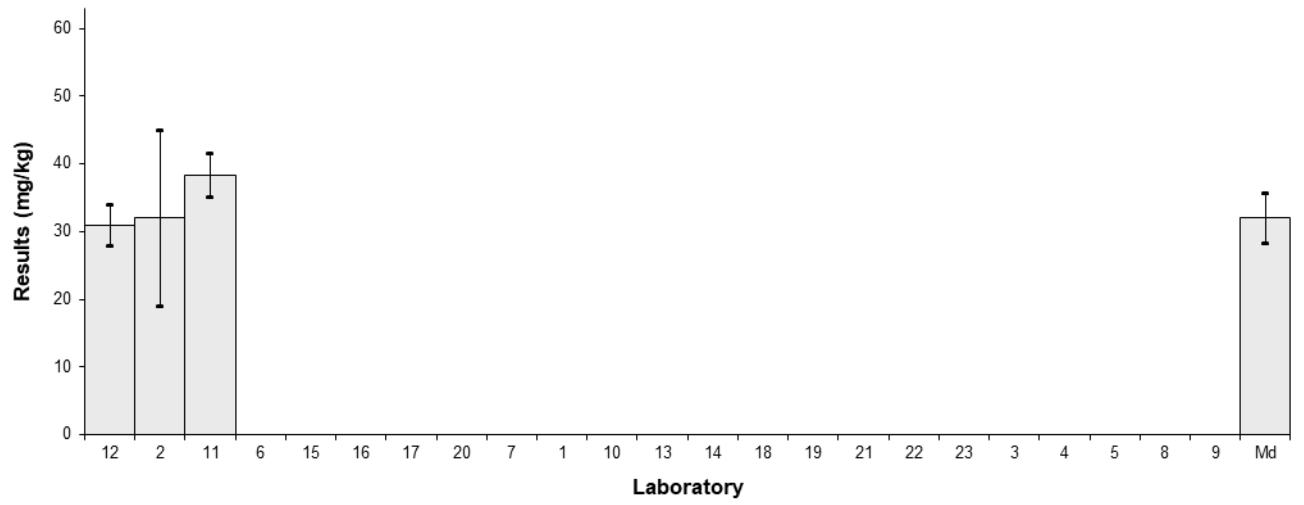


Figure 41

Table 53

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	64.1	6	0.42	0.37
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	64	15	0.41	0.16
7	NR	NR		
8	NT	NT		
9	57	17.1	-0.73	-0.26
10	NT	NT		
11	57.2	6.58	-0.70	-0.57
12	84	8.0	3.66	2.55
13	NT	NT		
14	NT	NT		
15	60	20	-0.24	-0.07
16	NR	NR		
17	61	12	-0.08	-0.04
18	NT	NT		
19	NT	NT		
20	60.9	6	-0.10	-0.09
21	NT	NT		
22	NT	NT		
23	NT	NT		

Statistics

Assigned Value	61.5	3.7
Spike	Not Spiked	
Homogeneity Value	56.0	6.7
Robust Average	61.5	3.7
Median	61.0	3.8
Mean	63.5	
N	8	
Max.	84	
Min.	57	
Robust SD	4.1	
Robust CV	6.7%	

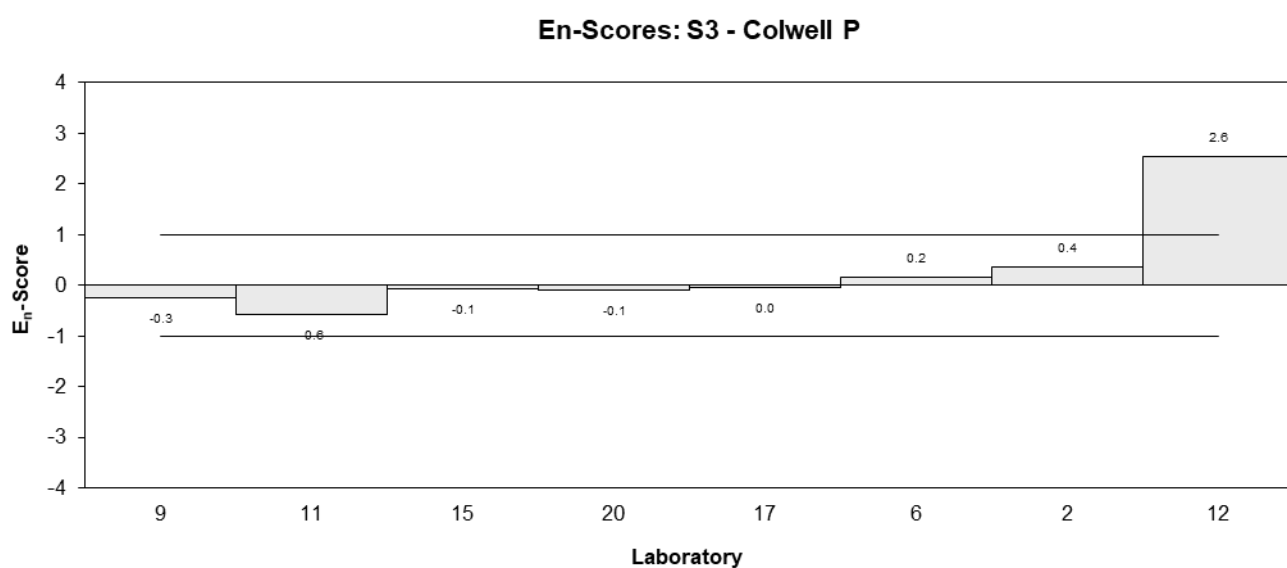
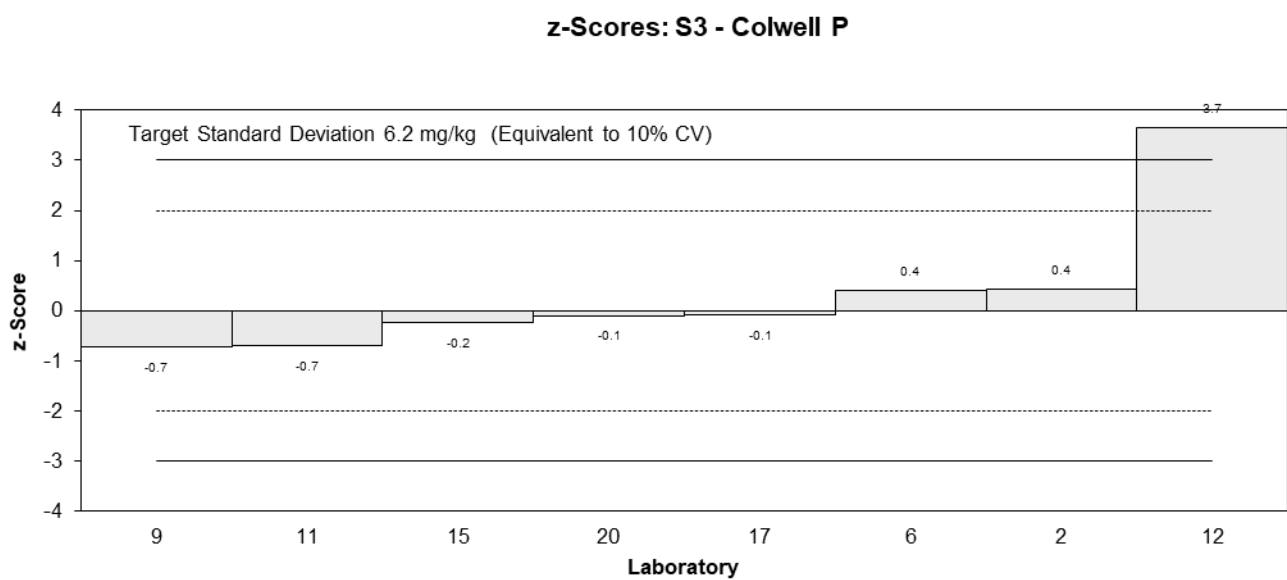
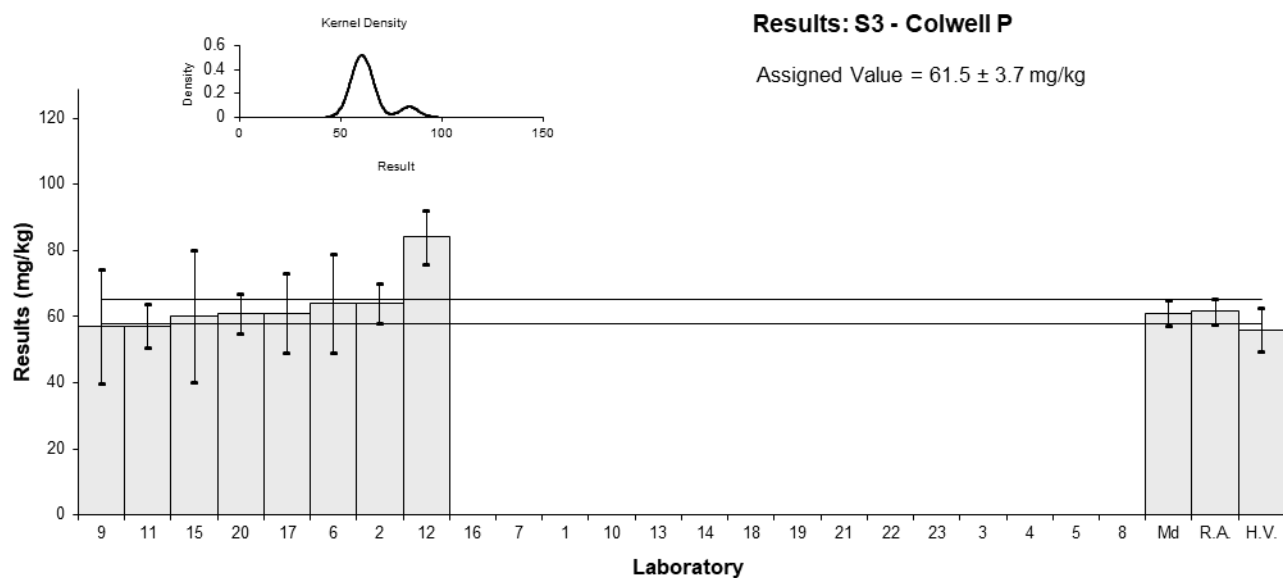


Figure 42

Table 54

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	305	45	0.41	0.26
2	266	25	-0.92	-0.96
3	NT	NT		
4	NT	NT		
5	291	29.1	-0.07	-0.06
6	298	7	0.17	0.34
7	282	14	-0.38	-0.58
8	NT	NT		
9	300	14.1	0.24	0.36
10	NT	NT		
11	295	24.78	0.07	0.07
12	273	30	-0.68	-0.61
13	320	22.6	0.92	1.04
14	320	55	0.92	0.48
15	294	15	0.03	0.05
16	NR	NR		
17	290	44	-0.10	-0.07
18	NT	NT		
19	NT	NT		
20	281	6	-0.41	-0.84
21	NT	NT		
22	NT	NT		
23	NT	NT		

Statistics

Assigned Value	293	13
Spike	Not Spiked	
Robust Average	293	13
Median	294	10
Mean	293	
N	13	
Max.	320	
Min.	266	
Robust SD	18	
Robust CV	6.2%	

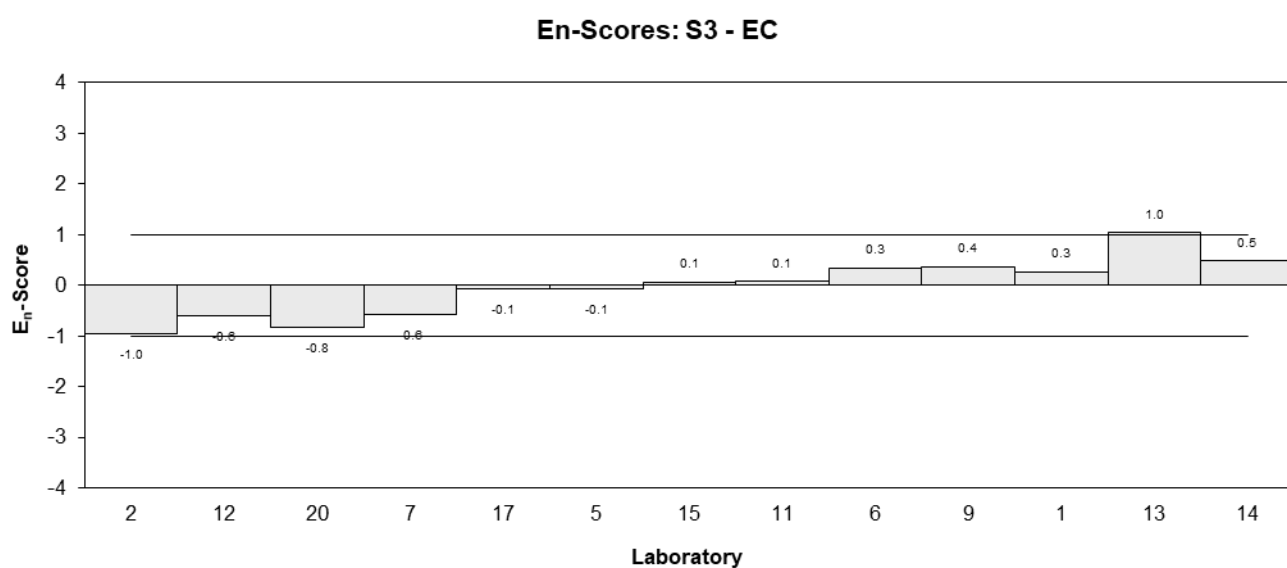
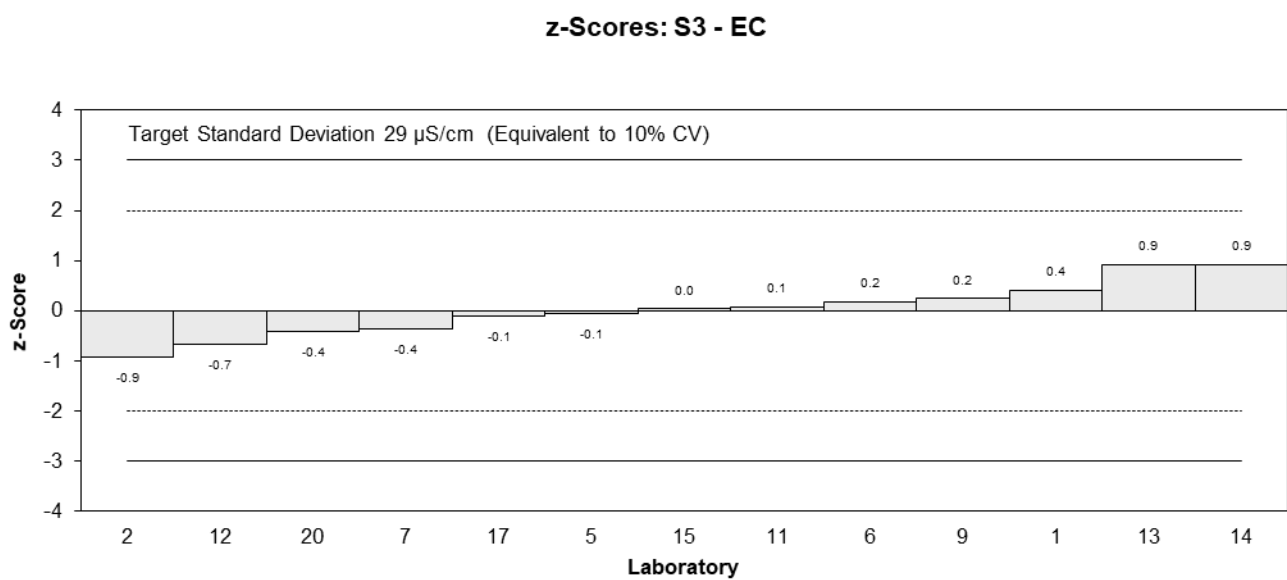
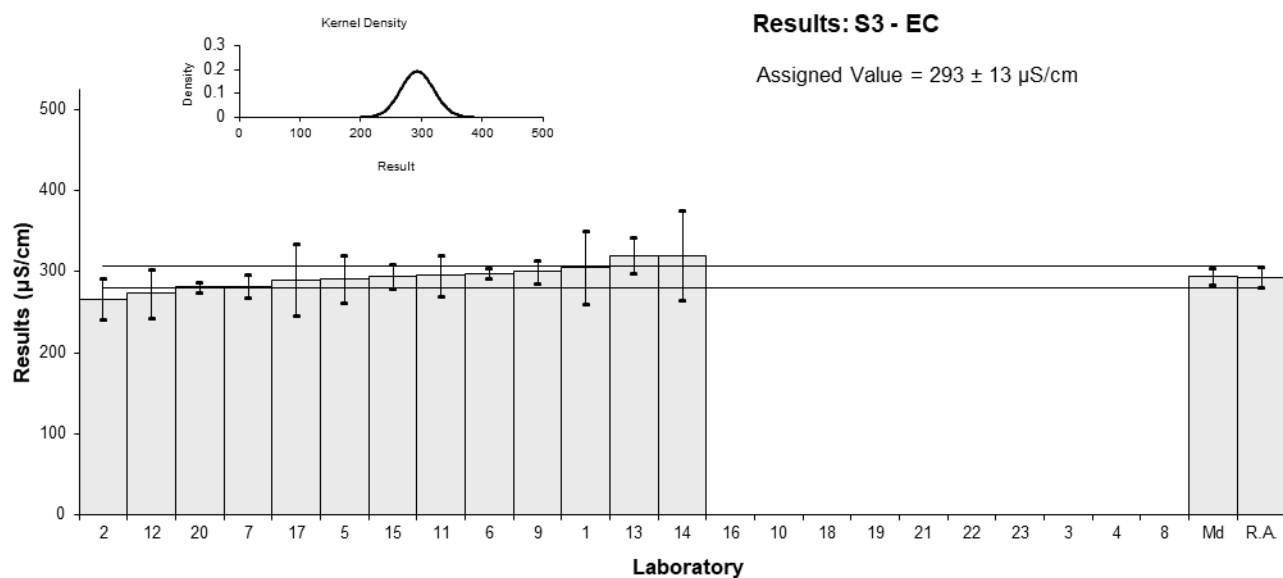


Figure 43

Table 55

Sample Details

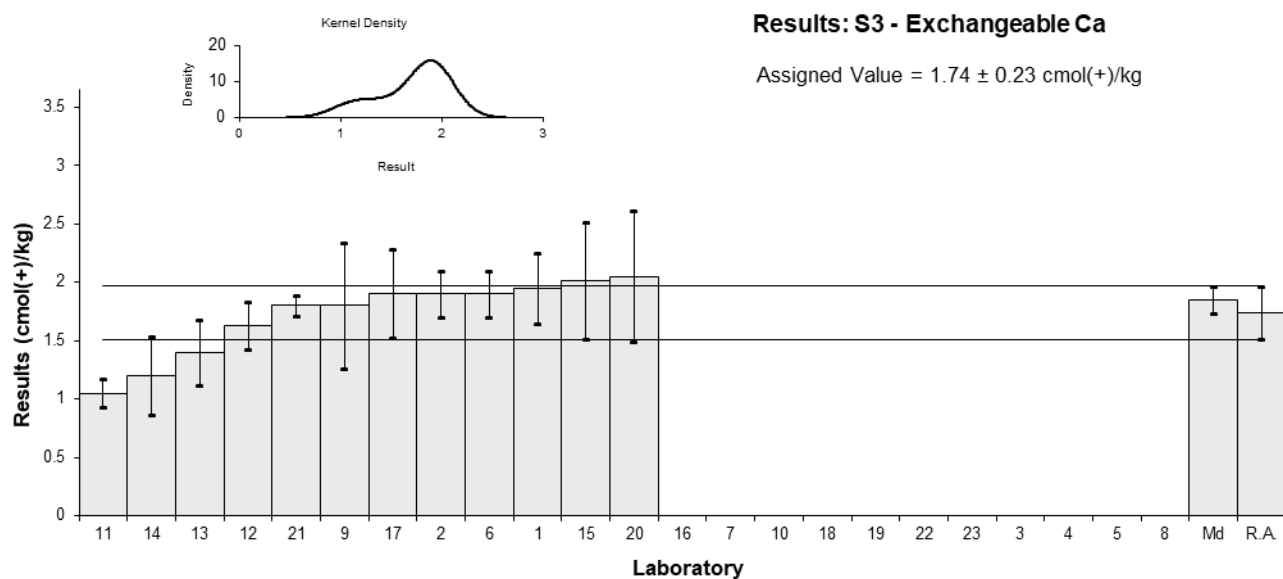
Sample No.	S3
Matrix.	Soil
Analyte.	Exchangeable Ca
Units	cmol(+)/kg

Participant Results

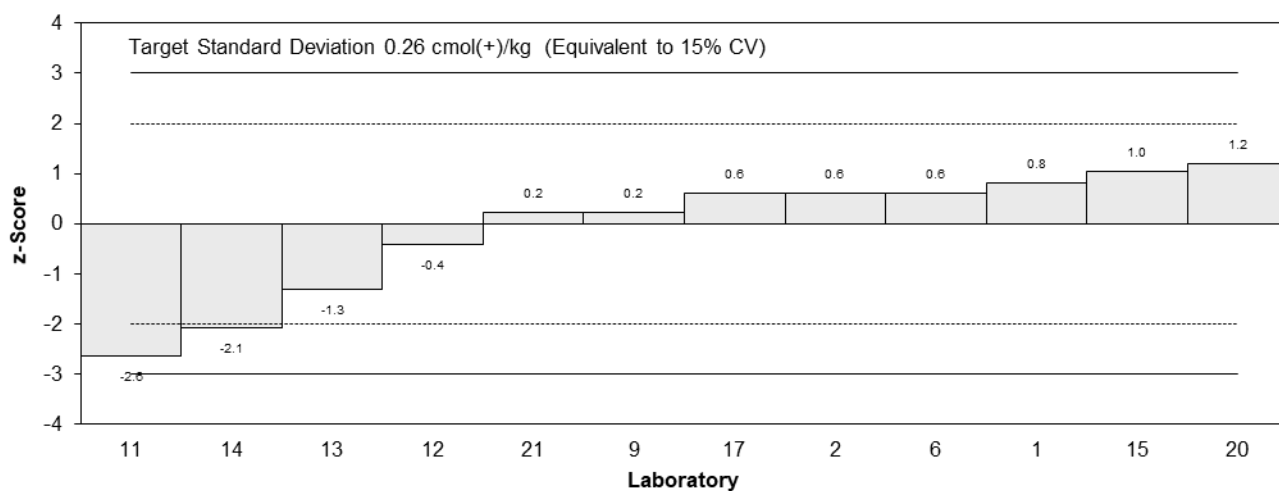
Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	1.95	0.3	0.80	0.56
2	1.90	0.20	0.61	0.52
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	1.9	0.2	0.61	0.52
7	NR	NR		
8	NT	NT		
9	1.8	0.54	0.23	0.10
10	NT	NT		
11	1.05	0.121	-2.64	-2.66
12	1.63	0.2	-0.42	-0.36
13	1.4	0.28	-1.30	-0.94
14	1.2	0.34	-2.07	-1.32
15	2.01	0.5	1.03	0.49
16	NR	NR		
17	1.9	0.38	0.61	0.36
18	NT	NT		
19	NT	NT		
20	2.05	0.56	1.19	0.51
21	1.8	0.09	0.23	0.24
22	NT	NT		
23	NT	NT		

Statistics

Assigned Value	1.74	0.23
Spike	Not Spiked	
Robust Average	1.74	0.23
Median	1.85	0.12
Mean	1.72	
N	12	
Max.	2.05	
Min.	1.05	
Robust SD	0.33	
Robust CV	19%	



z-Scores: S3 - Exchangeable Ca



En-Scores: S3 - Exchangeable Ca

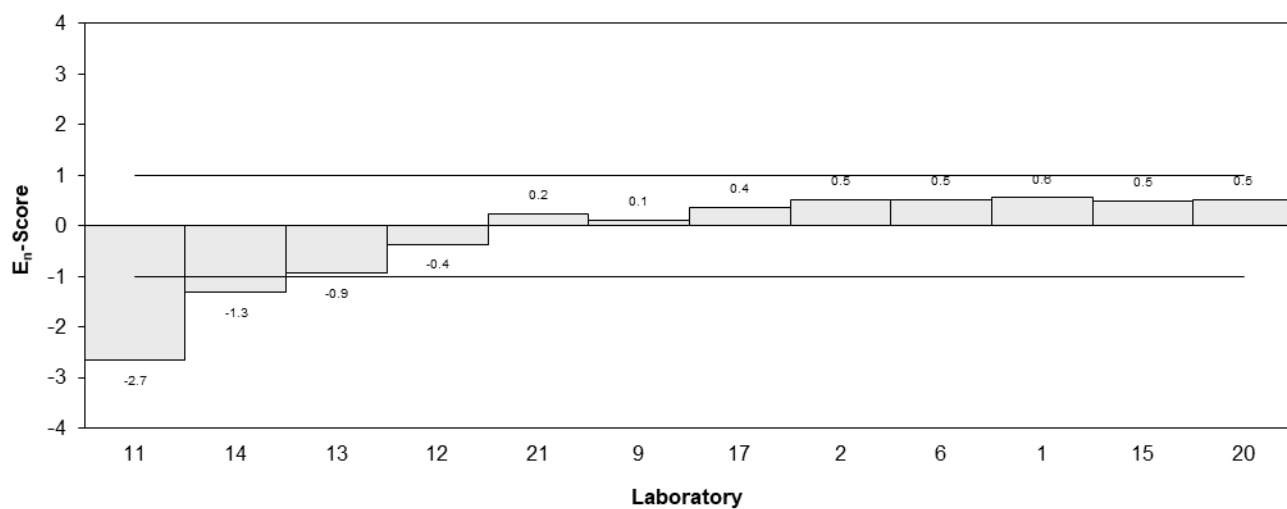


Figure 44

Table 56

Sample Details

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

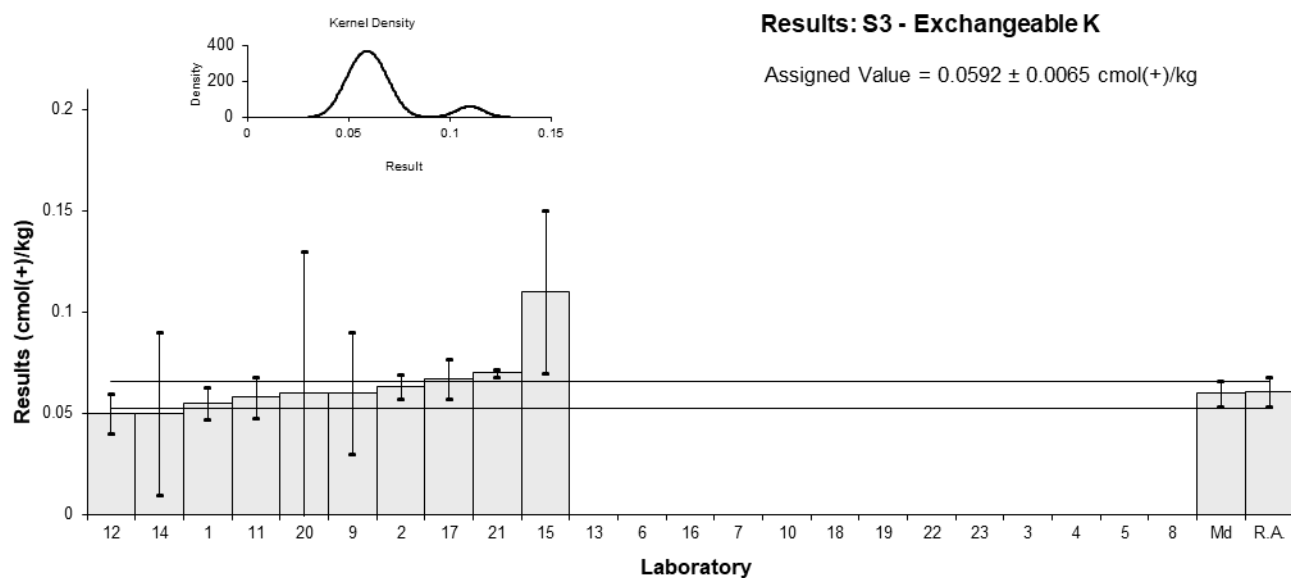
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.055	0.008	-0.47	-0.41
2	0.063	0.006	0.43	0.43
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	<0.1	NR		
7	NR	NR		
8	NT	NT		
9	0.06	0.03	0.09	0.03
10	NT	NT		
11	0.058	0.01	-0.14	-0.10
12	0.05	0.01	-1.04	-0.77
13	<0.1	0.02		
14	0.05	0.04	-1.04	-0.23
15	0.11	0.04	5.72	1.25
16	NR	NR		
17	0.067	0.01	0.88	0.65
18	NT	NT		
19	NT	NT		
20	0.06	0.07	0.09	0.01
21	0.07	0.002	1.22	1.59
22	NT	NT		
23	NT	NT		

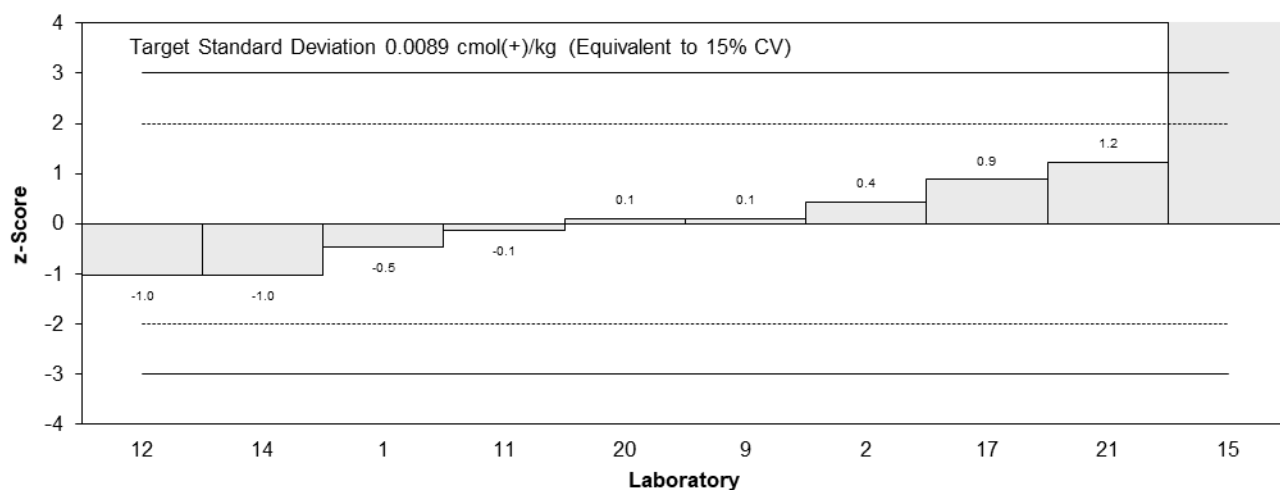
Statistics

Assigned Value*	0.0592	0.0065
Spike	Not Spiked	
Robust Average	0.0608	0.0073
Median	0.0600	0.0064
Mean	0.0643	
N	10	
Max.	0.11	
Min.	0.05	
Robust SD	0.0092	
Robust CV	15%	

*Robust Average excluding Laboratory 15.



z-Scores: S3 - Exchangeable K



En-Scores: S3 - Exchangeable K

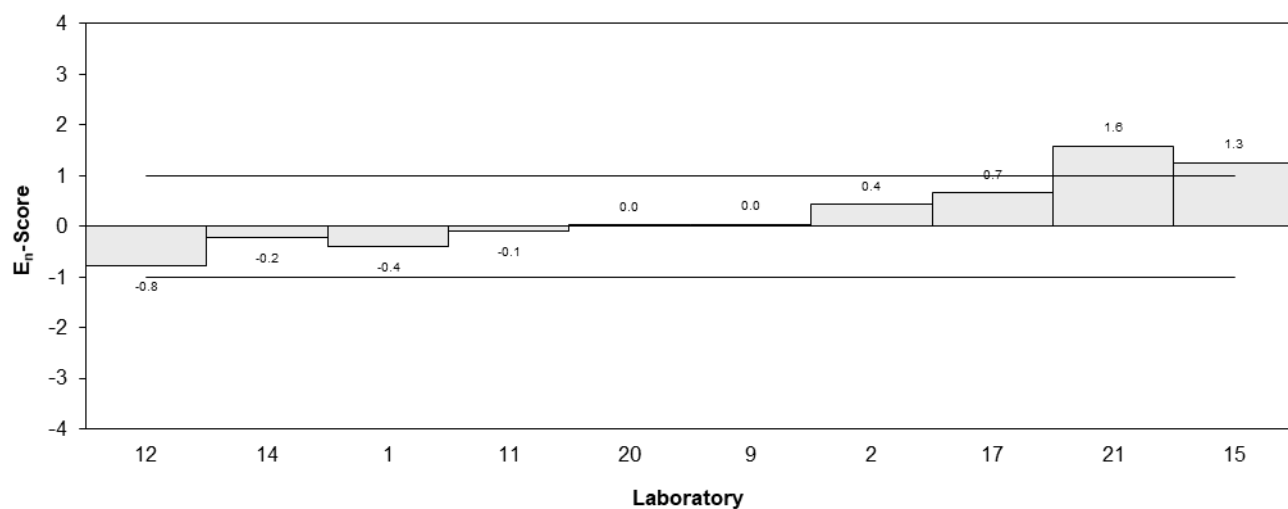


Figure 45

Table 57

Sample Details

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

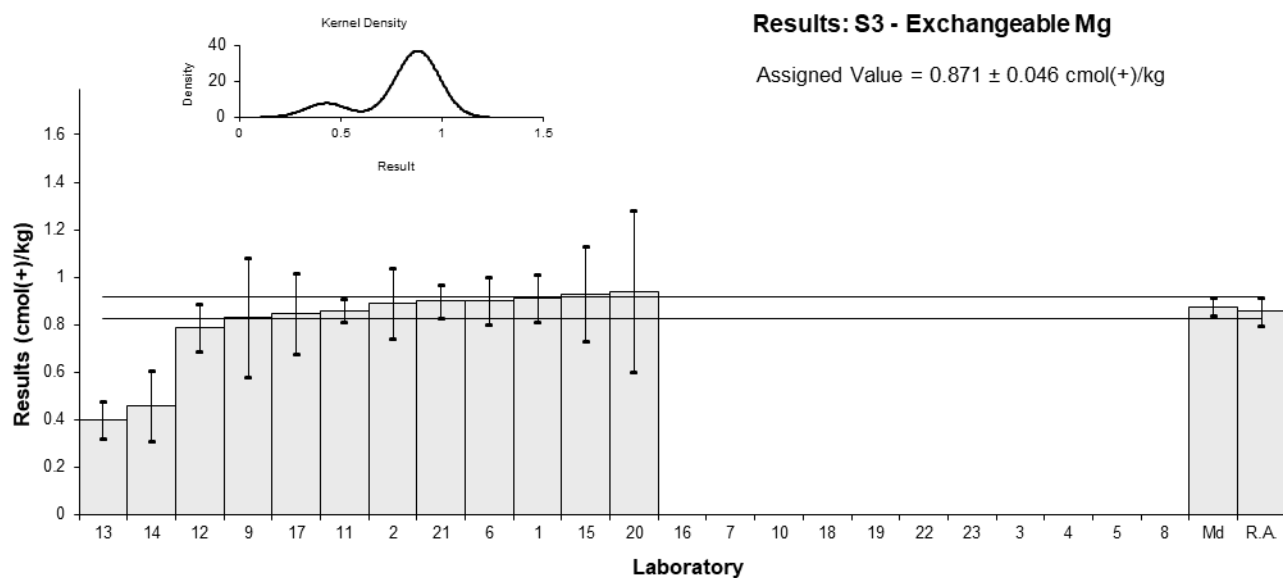
Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.91	0.1	0.30	0.35
2	0.89	0.15	0.15	0.12
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	0.9	0.1	0.22	0.26
7	NR	NR		
8	NT	NT		
9	0.83	0.25	-0.31	-0.16
10	NT	NT		
11	0.860	0.049	-0.08	-0.16
12	0.79	0.1	-0.62	-0.74
13	0.4	0.08	-3.61	-5.10
14	0.46	0.15	-3.15	-2.62
15	0.93	0.2	0.45	0.29
16	NR	NR		
17	0.85	0.17	-0.16	-0.12
18	NT	NT		
19	NT	NT		
20	0.94	0.34	0.53	0.20
21	0.9	0.07	0.22	0.35
22	NT	NT		
23	NT	NT		

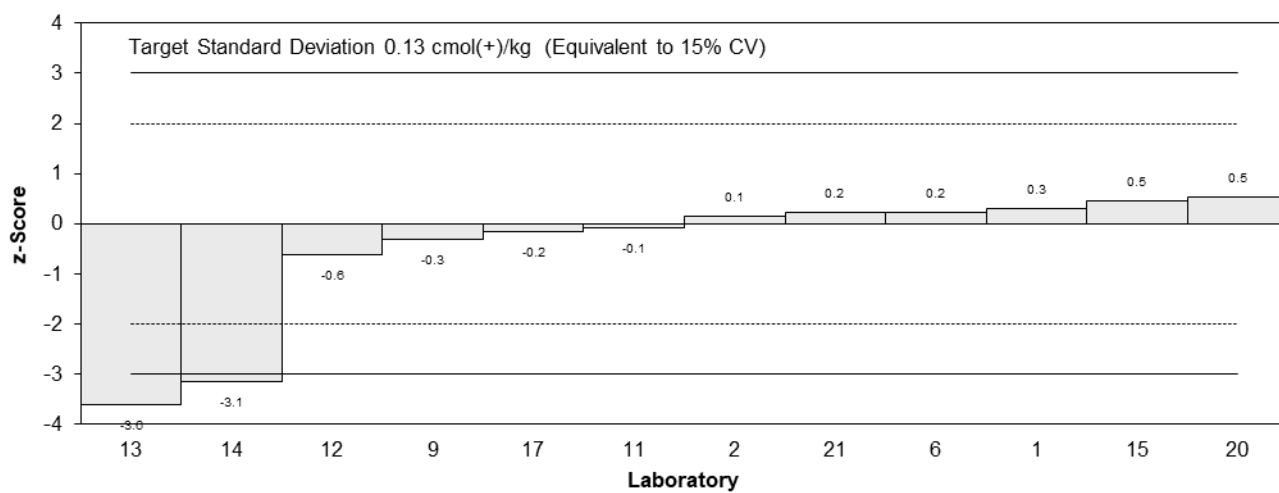
Statistics

Assigned Value*	0.871	0.046
Spike	Not Spiked	
Robust Average	0.856	0.057
Median	0.875	0.038
Mean	0.805	
N	12	
Max.	0.94	
Min.	0.4	
Robust SD	0.079	
Robust CV	9.2%	

*Robust Average excluding Laboratory 13.



z-Scores: S3 - Exchangeable Mg



En-Scores: S3 - Exchangeable Mg

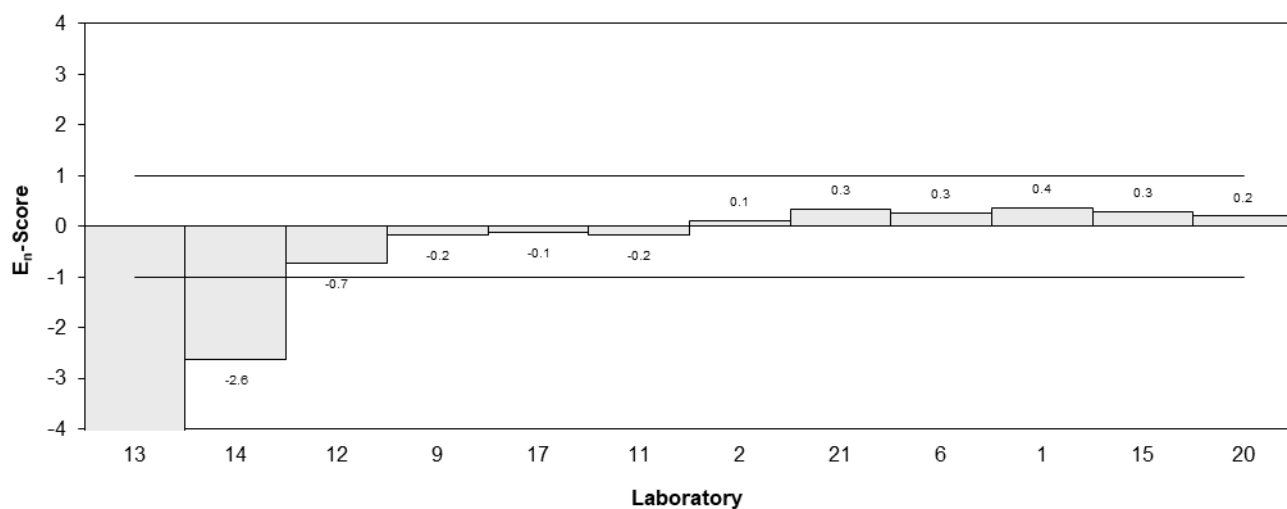


Figure 46

Table 58

Sample Details

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

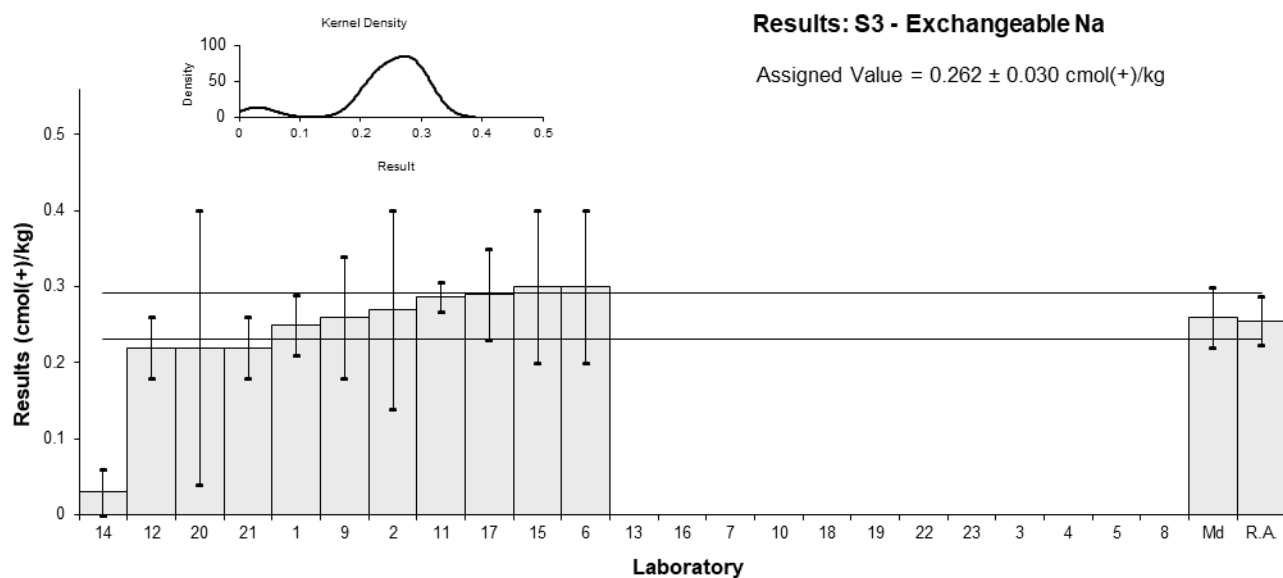
Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.25	0.04	-0.31	-0.24
2	0.27	0.13	0.20	0.06
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	0.3	0.1	0.97	0.36
7	NR	NR		
8	NT	NT		
9	0.26	0.08	-0.05	-0.02
10	NT	NT		
11	0.287	0.019	0.64	0.70
12	0.22	0.04	-1.07	-0.84
13	<0.1	0.02		
14	0.03	0.03	-5.90	-5.47
15	0.30	0.1	0.97	0.36
16	NR	NR		
17	0.29	0.06	0.71	0.42
18	NT	NT		
19	NT	NT		
20	0.22	0.18	-1.07	-0.23
21	0.22	0.04	-1.07	-0.84
22	NT	NT		
23	NT	NT		

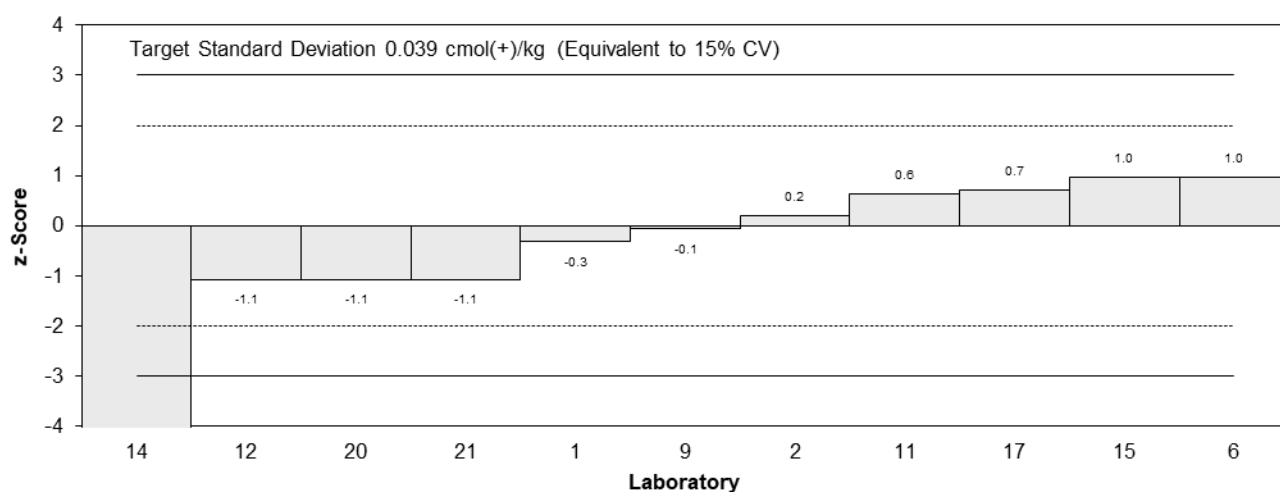
Statistics

Assigned Value*	0.262	0.030
Spike	Not Spiked	
Robust Average	0.255	0.032
Median	0.260	0.040
Mean	0.241	
N	11	
Max.	0.3	
Min.	0.03	
Robust SD	0.043	
Robust CV	17%	

*Robust Average excluding Laboratory 14.



z-Scores: S3 - Exchangeable Na



En-Scores: S3 - Exchangeable Na

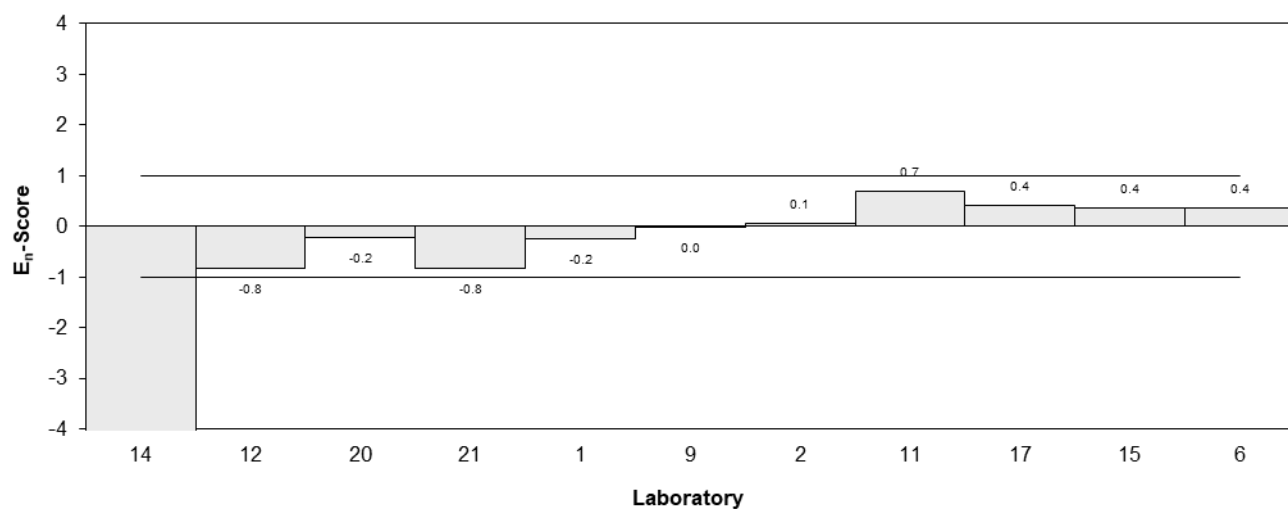


Figure 47

Table 59

Sample Details

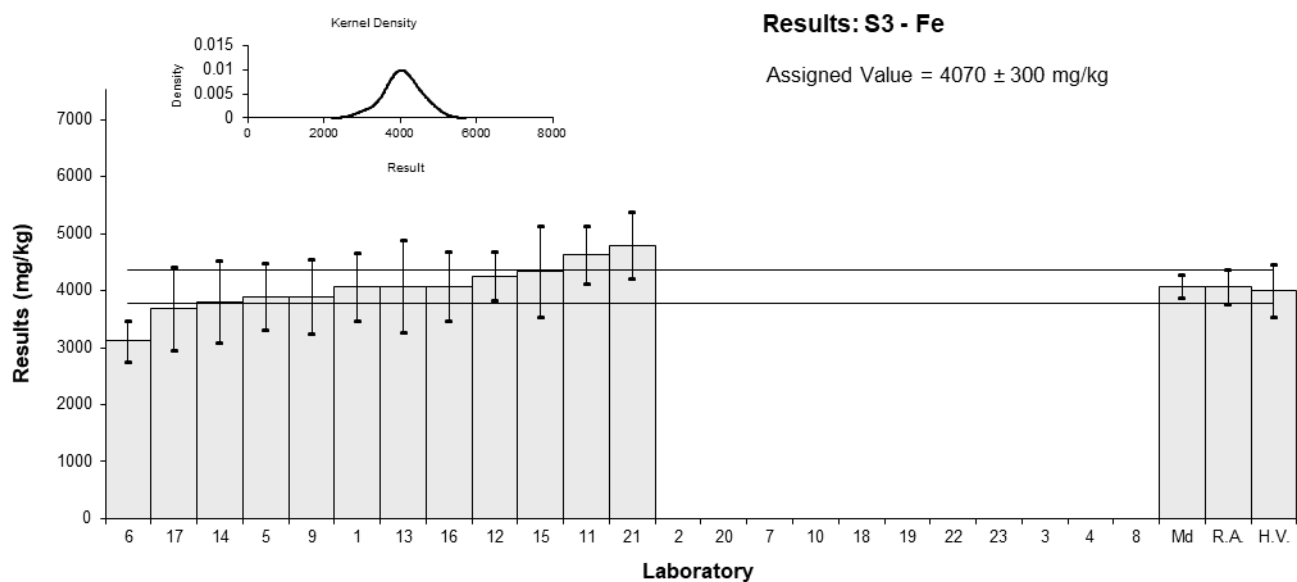
Sample No.	S3
Matrix.	Soil
Analyte.	Fe
Units	mg/kg

Participant Results

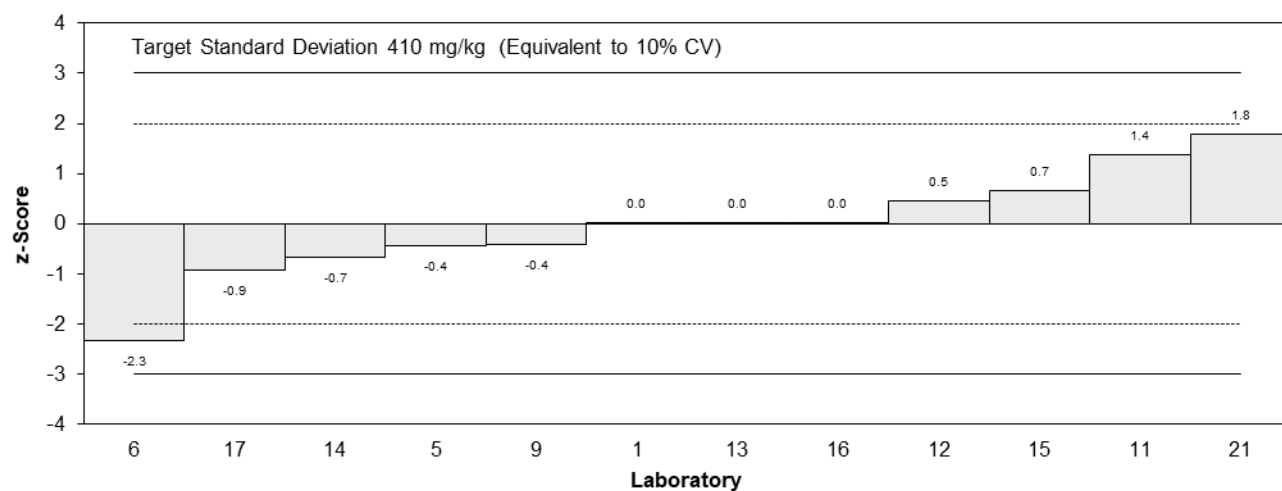
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	4075	600	0.01	0.01
2	NR	NR		
3	NT	NT		
4	NT	NT		
5	3892.36	583.85	-0.44	-0.27
6	3120	358	-2.33	-2.03
7	NR	NR		
8	NT	NT		
9	3900	643.5	-0.42	-0.24
10	NT	NT		
11	4630	509.3	1.38	0.95
12	4258	420	0.46	0.36
13	4080	816	0.02	0.01
14	3800	720	-0.66	-0.35
15	4340	800	0.66	0.32
16	4080	612	0.02	0.01
17	3690	730	-0.93	-0.48
18	NT	NT		
19	NT	NT		
20	NR	NR		
21	4800	576	1.79	1.12
22	NT	NT		
23	NT	NT		

Statistics

Assigned Value	4070	300
Spike	Not Spiked	
Homogeneity Value	4000	470
Robust Average	4070	300
Median	4080	210
Mean	4060	
N	12	
Max.	4800	
Min.	3120	
Robust SD	420	
Robust CV	10%	



z-Scores: S3 - Fe



En-Scores: S3 - Fe

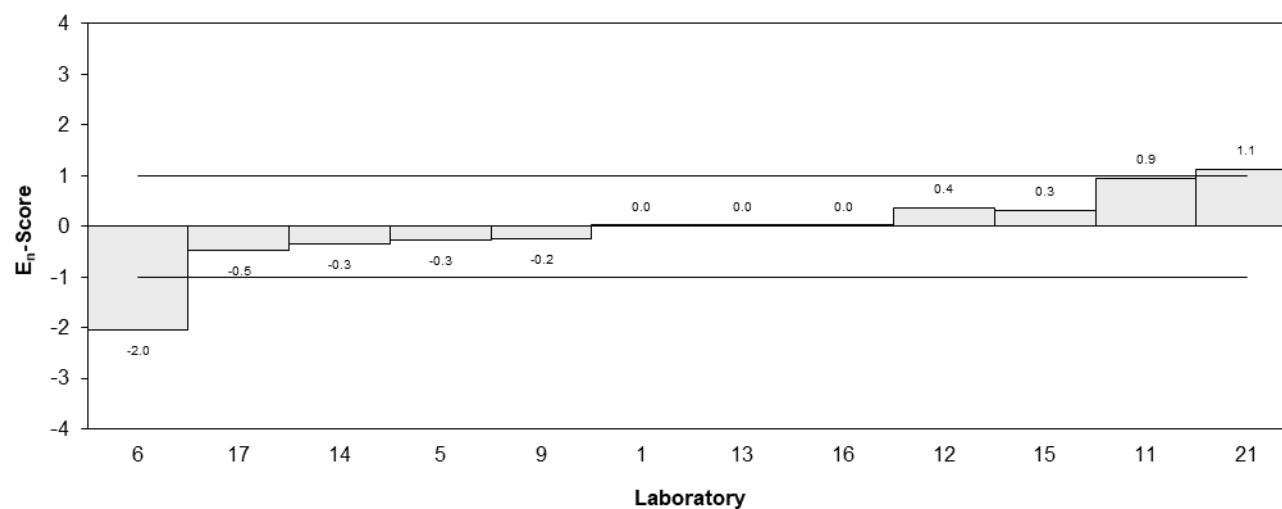


Figure 48

Table 60

Sample Details

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

Participant Results

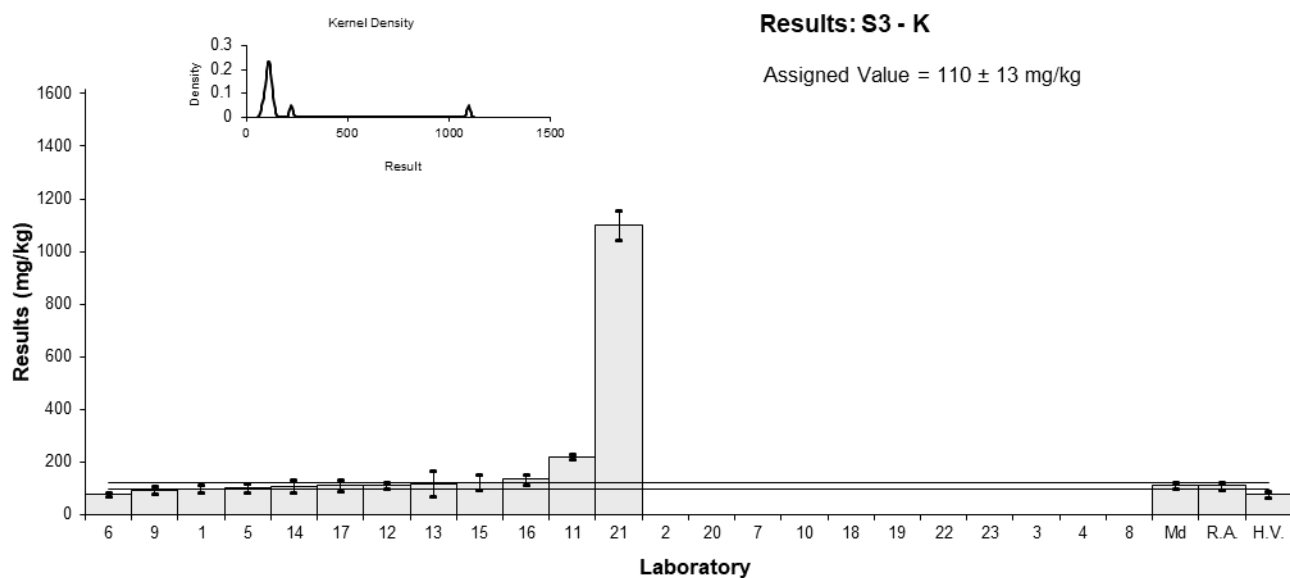
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	100	15	-0.91	-0.50
2	NR	NR		
3	NT	NT		
4	NT	NT		
5	102.40	15.36	-0.69	-0.38
6	80	8	-2.73	-1.97
7	NR	NR		
8	NT	NT		
9	94	14.288	-1.45	-0.83
10	NT	NT		
11	222	10.88	10.18	6.61
12	114	11	0.36	0.23
13	120	49.2	0.91	0.20
14	110	24	0.00	0.00
15	124	30	1.27	0.43
16	135	19	2.27	1.09
17	113	23	0.27	0.11
18	NT	NT		
19	NT	NT		
20	NR	NR		
21	1100	55	90.00	17.52
22	NT	NT		
23	NT	NT		

Statistics*

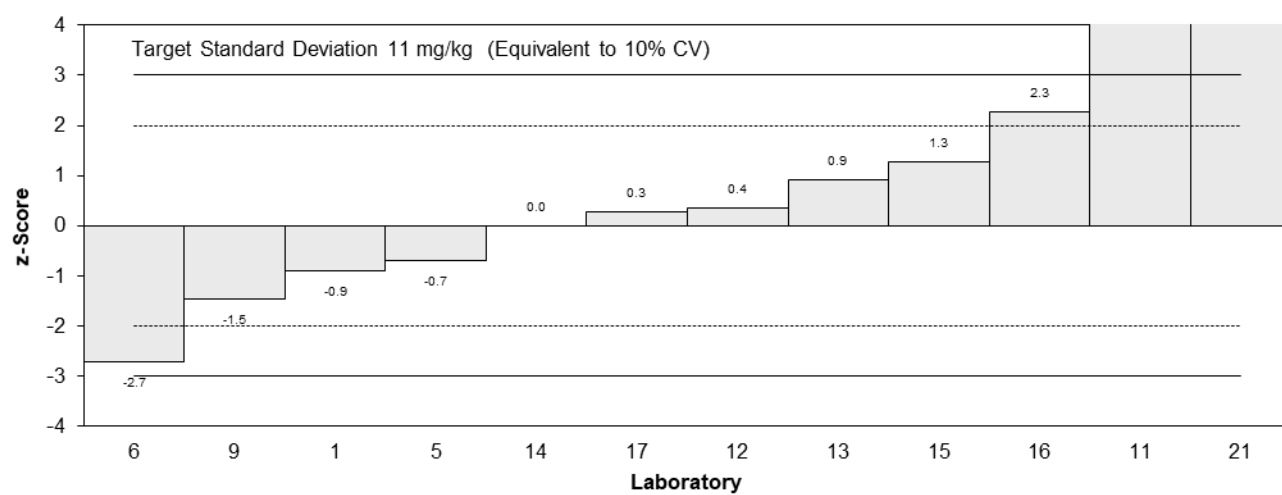
Assigned Value**	110	13
Spike	Not Spiked	
Homogeneity Value	80	12
Robust Average	112	15
Median	113	11
Mean	119	
N	11	
Max.	222	
Min.	80	
Robust SD	20	
Robust CV	18%	

*Laboratory 21 excluded from statistical calculation (extreme outlier).

**Robust Average excluding Laboratory 11.



z-Scores: S3 - K



En-Scores: S3 - K

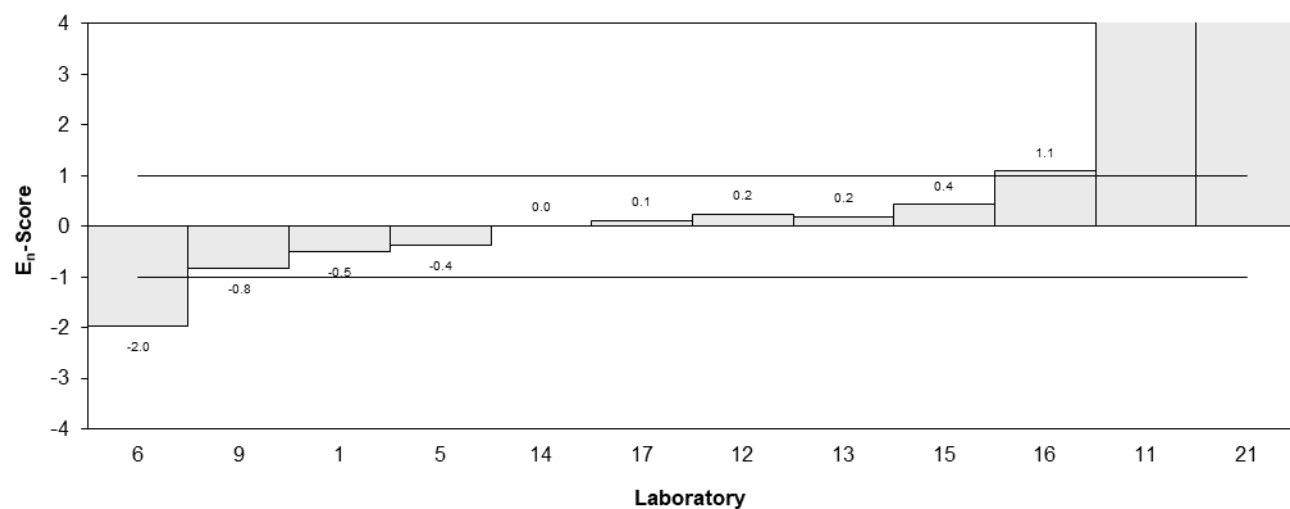


Figure 49

Table 61

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	202	30	-0.65	-0.53
2	NR	NR		
3	NT	NT		
4	NT	NT		
5	221.50	33.23	-0.07	-0.06
6	160	16	-1.90	-1.93
7	NR	NR		
8	NT	NT		
9	180	20.16	-1.31	-1.25
10	NT	NT		
11	281	16.02	1.70	1.72
12	226	22	0.06	0.05
13	230	77.7	0.18	0.07
14	230	42	0.18	0.12
15	233	90	0.27	0.10
16	232	33	0.24	0.18
17	200	40	-0.71	-0.49
18	NT	NT		
19	NT	NT		
20	NR	NR		
21	320	57	2.86	1.50
22	NT	NT		
23	NT	NT		

Statistics

Assigned Value	224	29
Spike	Not Spiked	
Robust Average	224	29
Median	228	15
Mean	226	
N	12	
Max.	320	
Min.	160	
Robust SD	40	
Robust CV	18%	

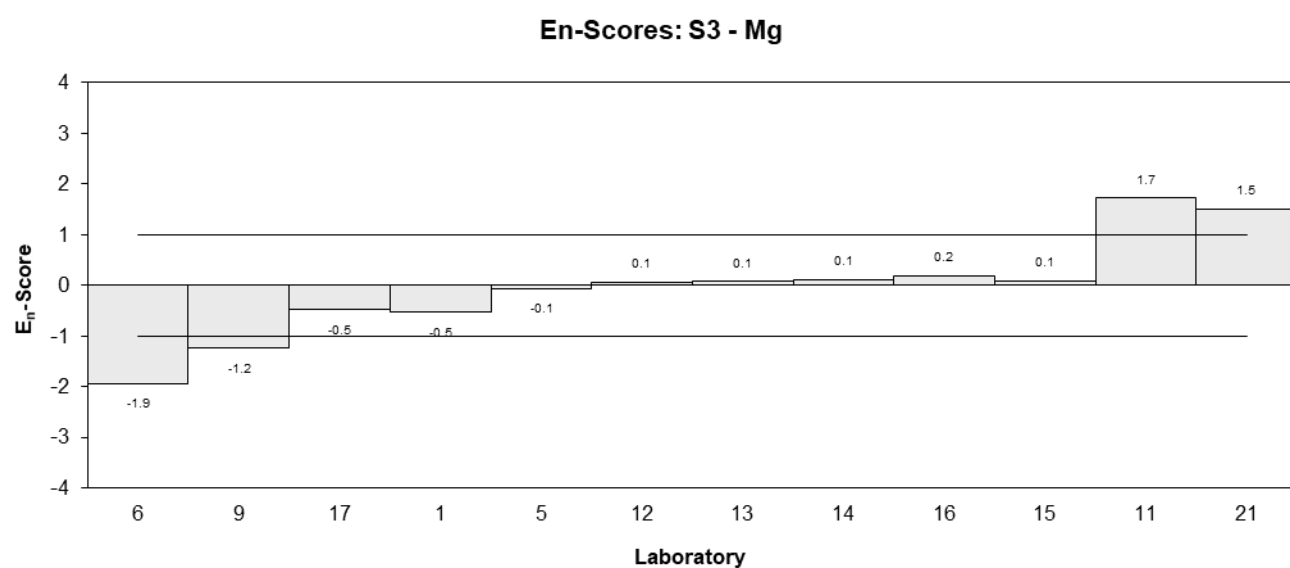
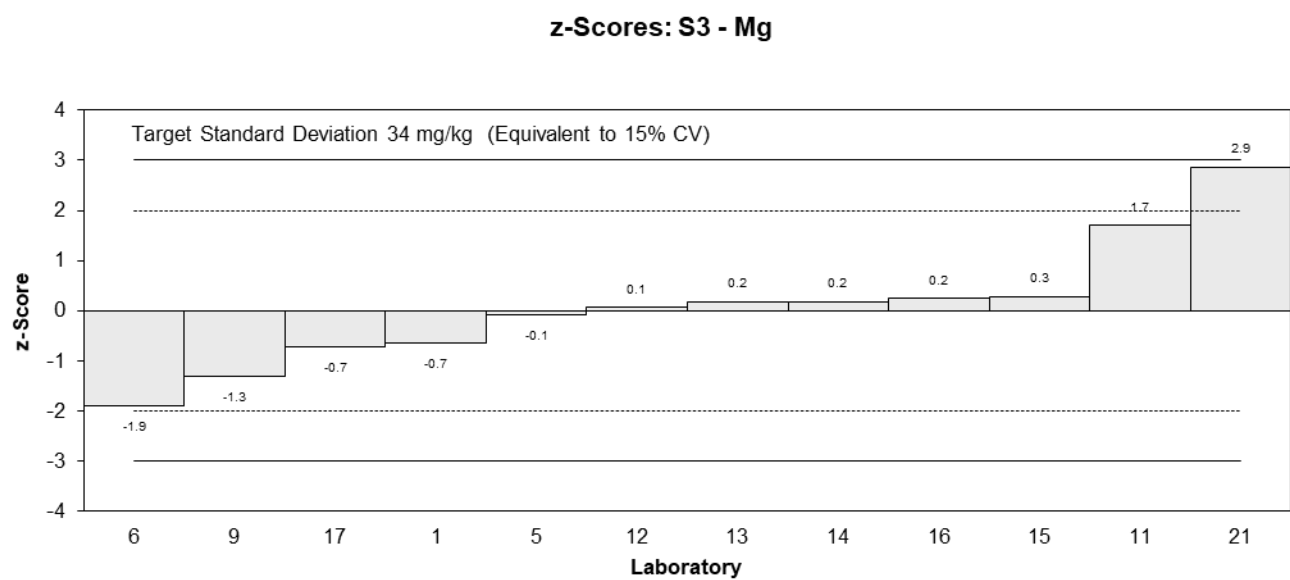
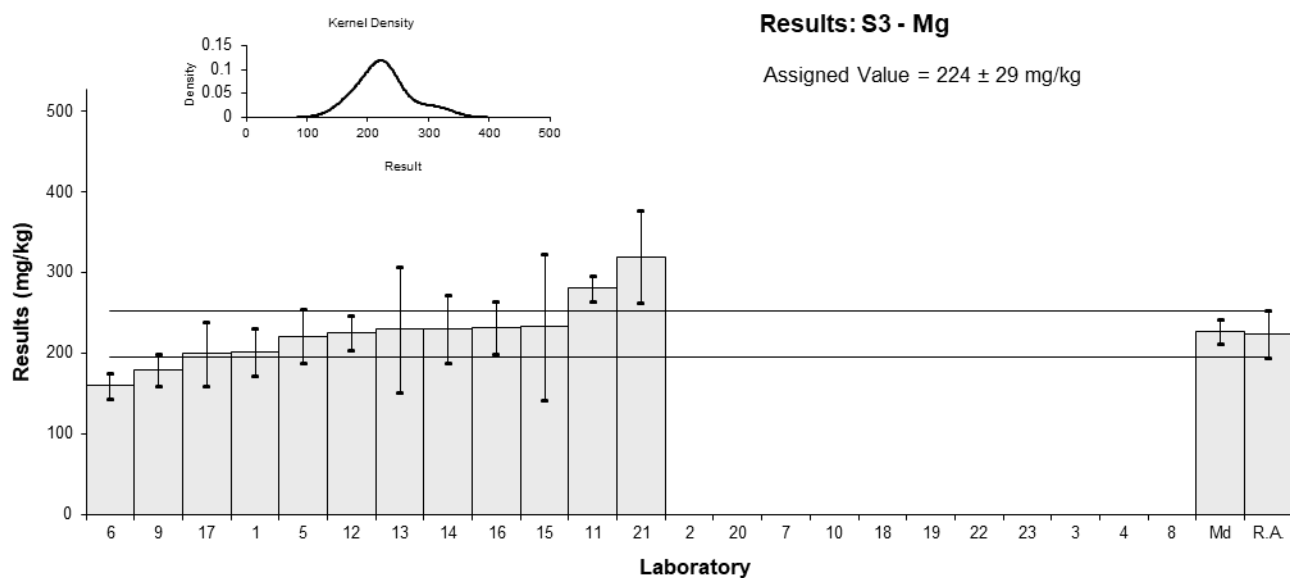


Figure 50

Table 62

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	95	14	1.10	0.56
2	95	9.6	1.10	0.71
3	NT	NT		
4	NT	NT		
5	83.90	12.59	-0.20	-0.11
6	80	8	-0.65	-0.46
7	NR	NR		
8	NT	NT		
9	65	15.6	-2.41	-1.14
10	NT	NT		
11	70.7	4.74	-1.74	-1.44
12	77	8.0	-1.00	-0.71
13	100	50.6	1.68	0.28
14	95	25	1.10	0.35
15	97.7	30	1.41	0.39
16	77.8	16.4	-0.91	-0.41
17	89	17	0.40	0.18
18	NT	NT		
19	NT	NT		
20	NR	NR		
21	390	31	35.56	9.41
22	NT	NT		
23	NT	NT		

Statistics*

Assigned Value	85.6	9.2
Spike	Not Spiked	
Homogeneity Value	82.4	9.9
Robust Average	85.6	9.2
Median	86.5	8.1
Mean	85.5	
N	12	
Max.	100	
Min.	65	
Robust SD	13	
Robust CV	15%	

*Laboratory 21 excluded from statistical calculation (extreme outlier).

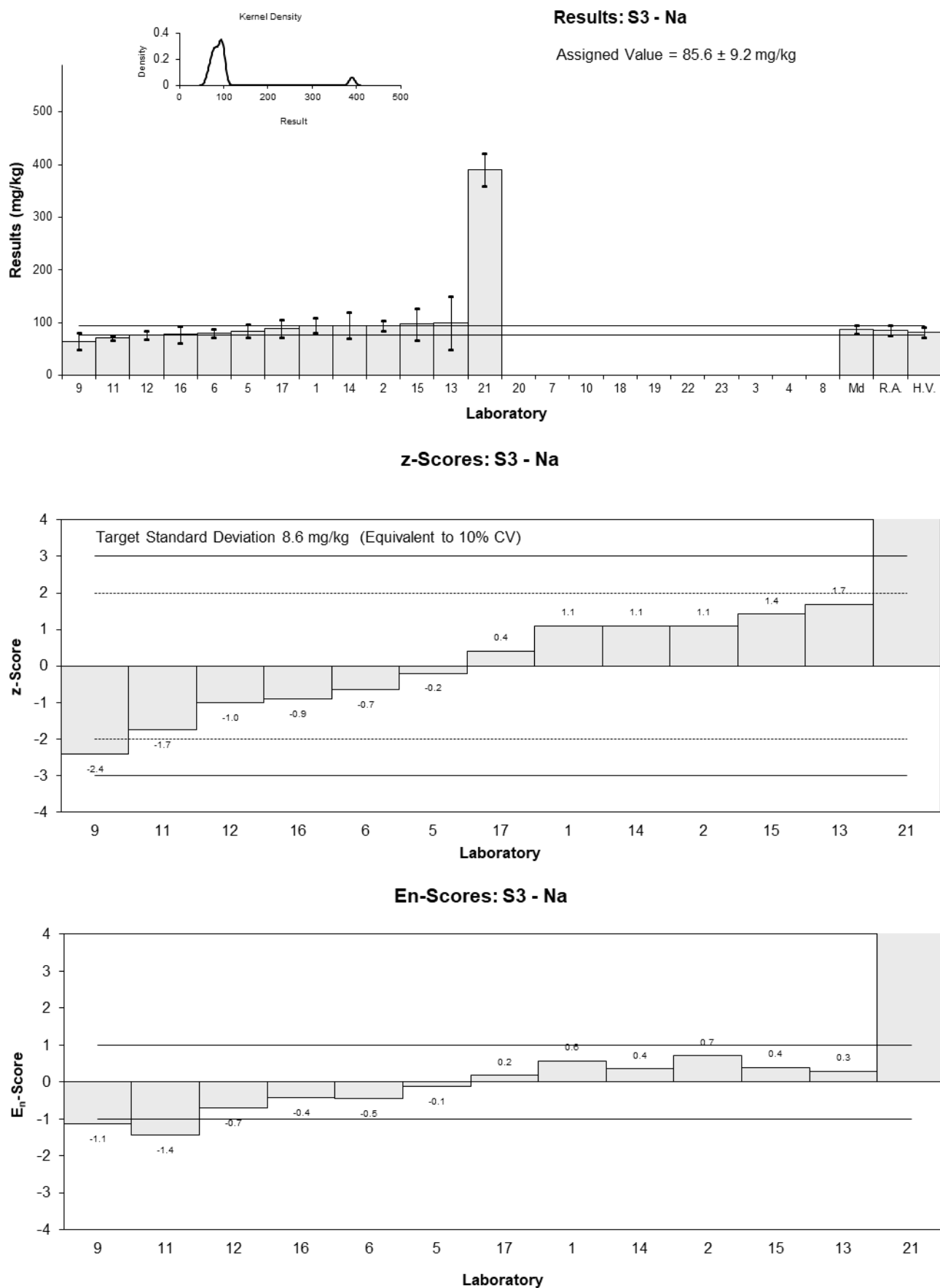


Figure 51

Table 63

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	173	26	0.18	0.10
2	152	15	-1.06	-0.82
3	NT	NT		
4	NT	NT		
5	171.65	25.75	0.10	0.05
6	150	14	-1.18	-0.94
7	NR	NR		
8	NT	NT		
9	170	36.55	0.00	0.00
10	NT	NT		
11	NT	NT		
12	208	21	2.24	1.44
13	230	46	3.53	1.23
14	175	29	0.29	0.15
15	156	30	-0.82	-0.41
16	173	16	0.18	0.13
17	147	29	-1.35	-0.69
18	NT	NT		
19	NT	NT		
20	NR	NR		
21	NT	NT		
22	NT	NT		
23	NT	NT		

Statistics

Assigned Value	170	16
Spike	Not Spiked	
Homogeneity Value	200	24
Robust Average	170	16
Median	172	16
Mean	173	
N	11	
Max.	230	
Min.	147	
Robust SD	22	
Robust CV	13%	

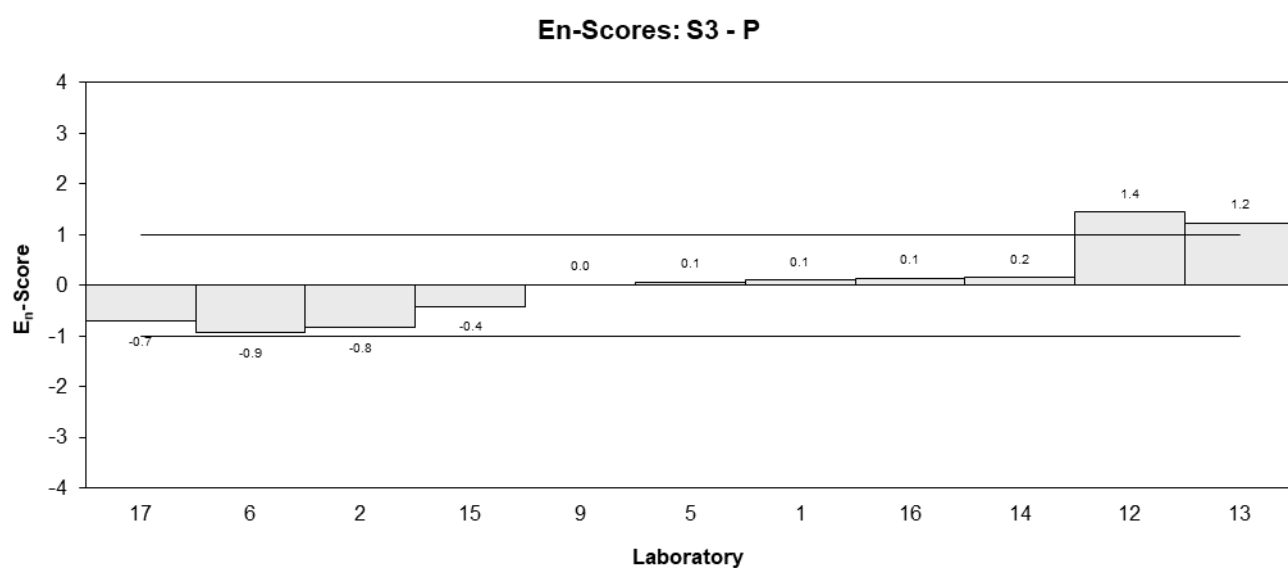
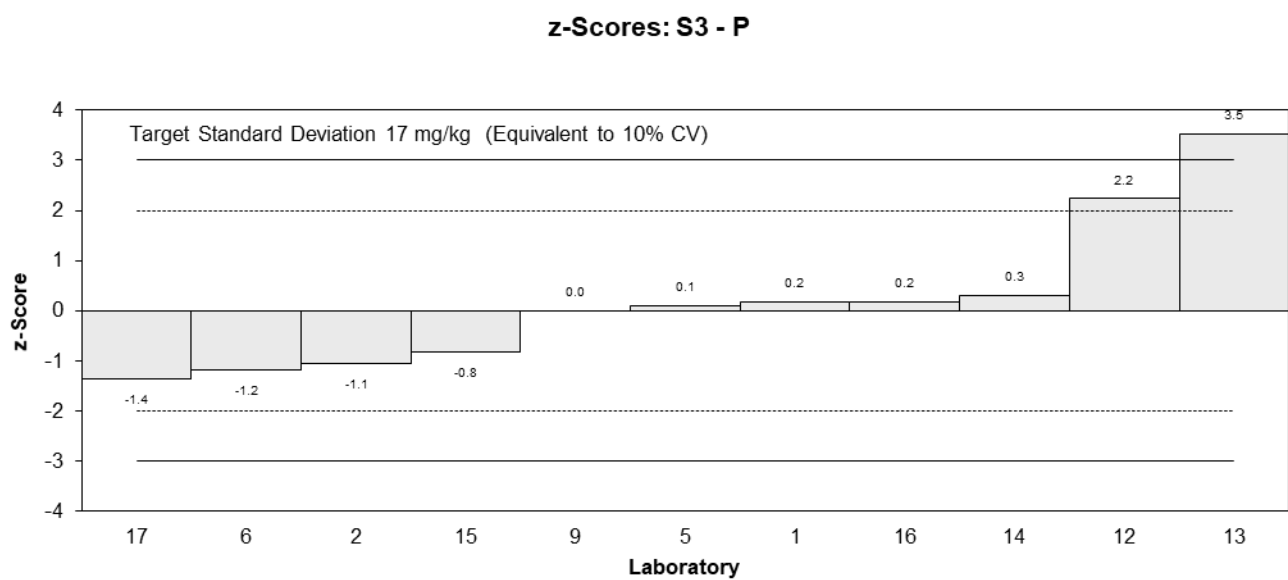
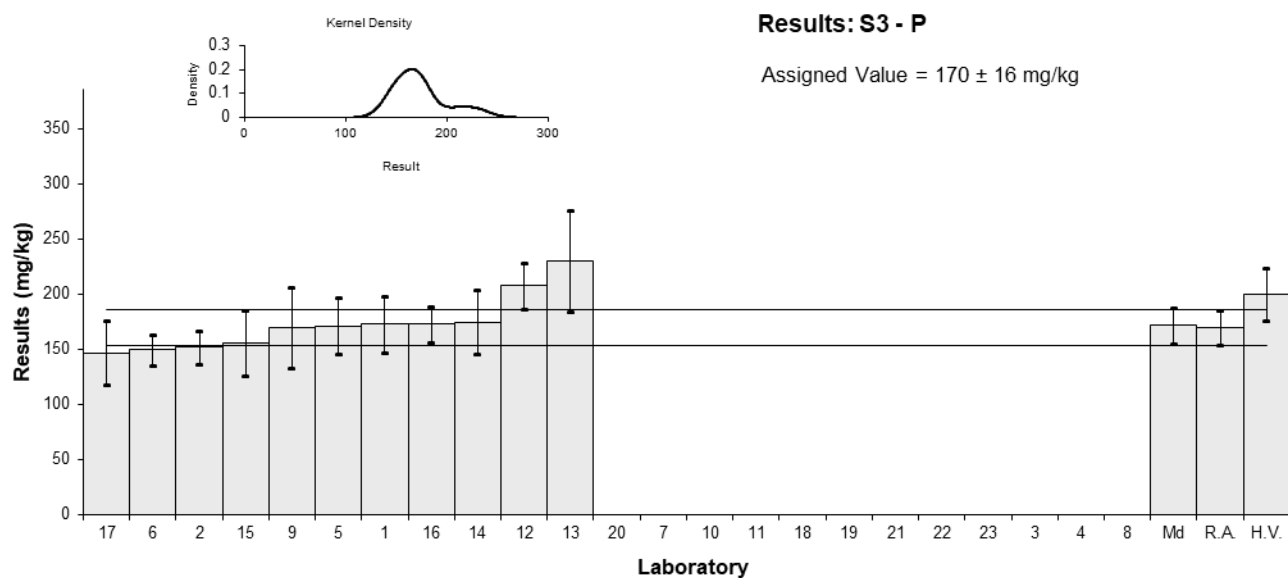


Figure 52

Table 64

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	41.3	4
3	NT	NT
4	NT	NT
5	NT	NT
6	NT	NT
7	NR	NR
8	NT	NT
9	NT	NT
10	NT	NT
11	49.2	5.66
12	39	4.0
13	NT	NT
14	NT	NT
15	47	20
16	NR	NR
17	NR	NR
18	NT	NT
19	NT	NT
20	40.8	NR
21	NT	NT
22	NT	NT
23	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Robust Average	43.5	5.6
Median	41.3	4.2
Mean	43.5	
N	5	
Max.	49.2	
Min.	39	
Robust SD	5.0	
Robust CV	11%	

Results: S3 - PBI

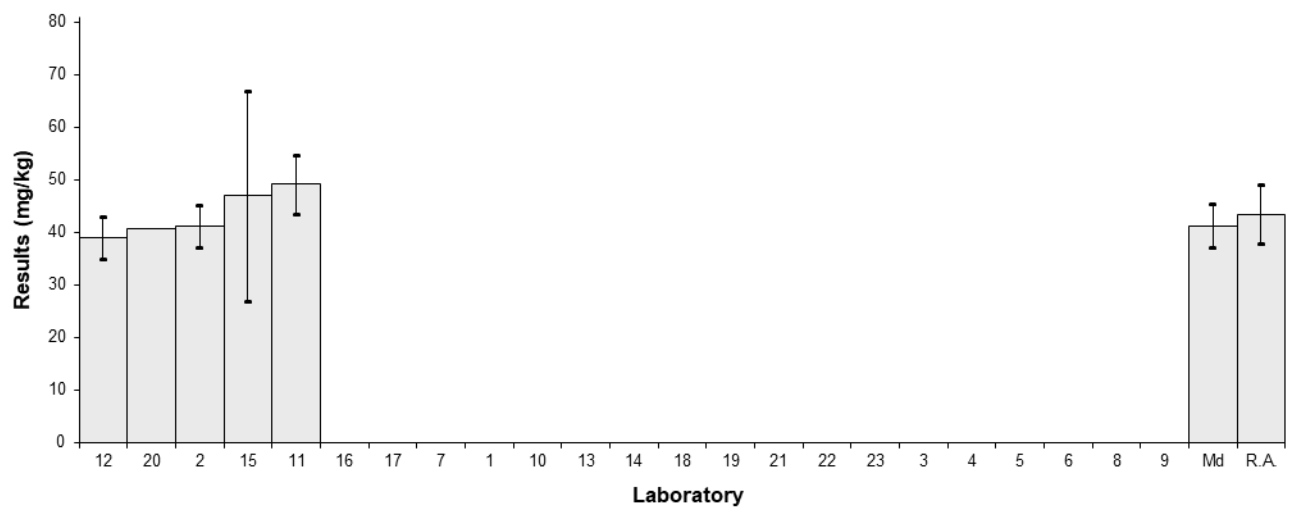


Figure 53

Table 65

Sample Details

Sample No.	S3
Matrix.	Soil
Analyte.	pH

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	4.32	0.1	-1.62	-1.21
2	4.40	0.11	-1.12	-0.82
3	NT	NT		
4	NT	NT		
5	4.75	0.48	1.06	0.33
6	4.3	0.1	-1.75	-1.30
7	NR	NR		
8	NT	NT		
9	4.4	0.2	-1.12	-0.65
10	NT	NT		
11	4.72	0.076	0.87	0.68
12	4.51	0.2	-0.44	-0.25
13	4.7	0.081	0.75	0.58
14	4.9	0.2	2.00	1.16
15	4.9	0.2	2.00	1.16
16	NR	NR		
17	4.3	0.2	-1.75	-1.01
18	NT	NT		
19	NT	NT		
20	4.76	0.1	1.12	0.84
21	NT	NT		
22	NT	NT		
23	NT	NT		

Statistics

Assigned Value	4.58	0.19
Spike	Not Spiked	
Robust Average	4.58	0.19
Median	4.61	0.19
Mean	4.58	
N	12	
Max.	4.9	
Min.	4.3	
Robust SD	0.26	
Robust CV	5.8%	

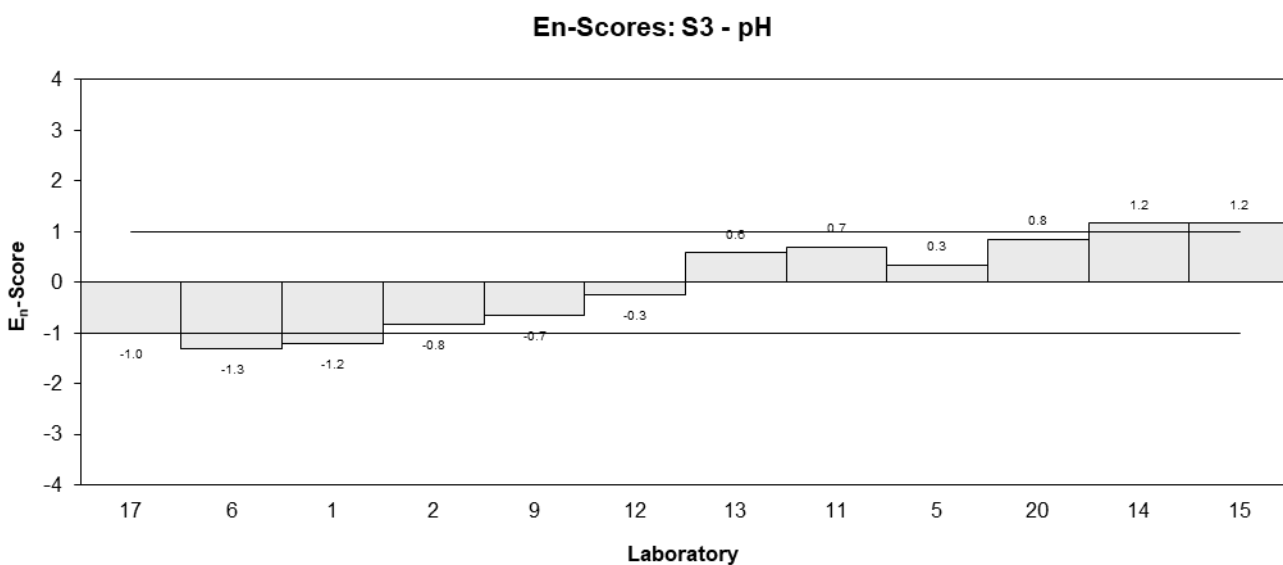
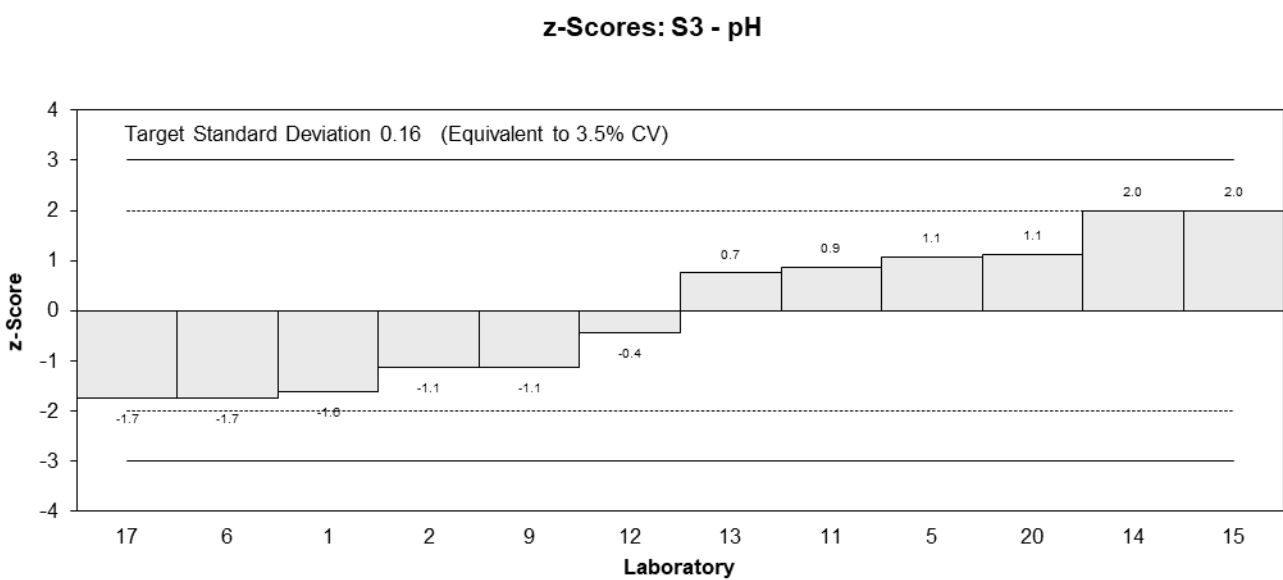
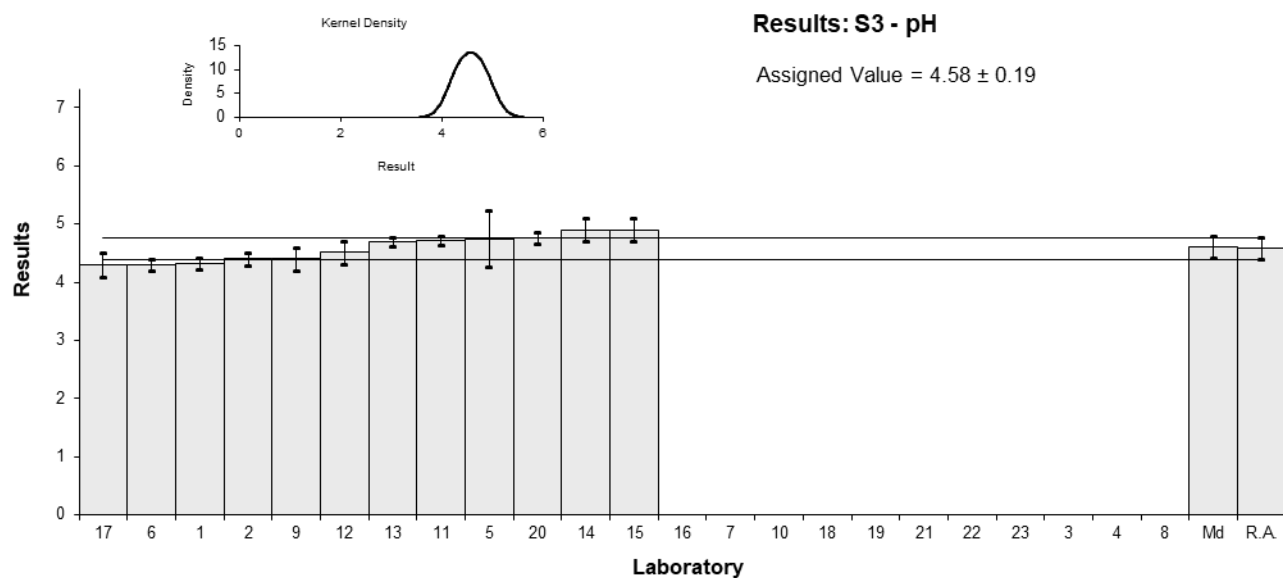


Figure 54

Table 66

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	221	33	1.16	0.60
2	193	29	-0.25	-0.14
3	NT	NT		
4	NT	NT		
5	200.50	30.08	0.13	0.07
6	180	16	-0.91	-0.72
7	NR	NR		
8	NT	NT		
9	200	39	0.10	0.05
10	NT	NT		
11	165	28.05	-1.67	-0.97
12	225	25	1.36	0.86
13	220	44	1.11	0.46
14	NT	NT		
15	206	30	0.40	0.23
16	208	32	0.51	0.27
17	160	32	-1.92	-1.02
18	NT	NT		
19	NT	NT		
20	NR	NR		
21	696	42	25.15	10.80
22	NT	NT		
23	NT	NT		

Statistics*

Assigned Value	198	19
Spike	Not Spiked	
Robust Average	198	19
Median	201	19
Mean	198	
N	11	
Max.	221	
Min.	160	
Robust SD	25	
Robust CV	12%	

*Laboratory 21 excluded from statistical calculation (extreme outlier).

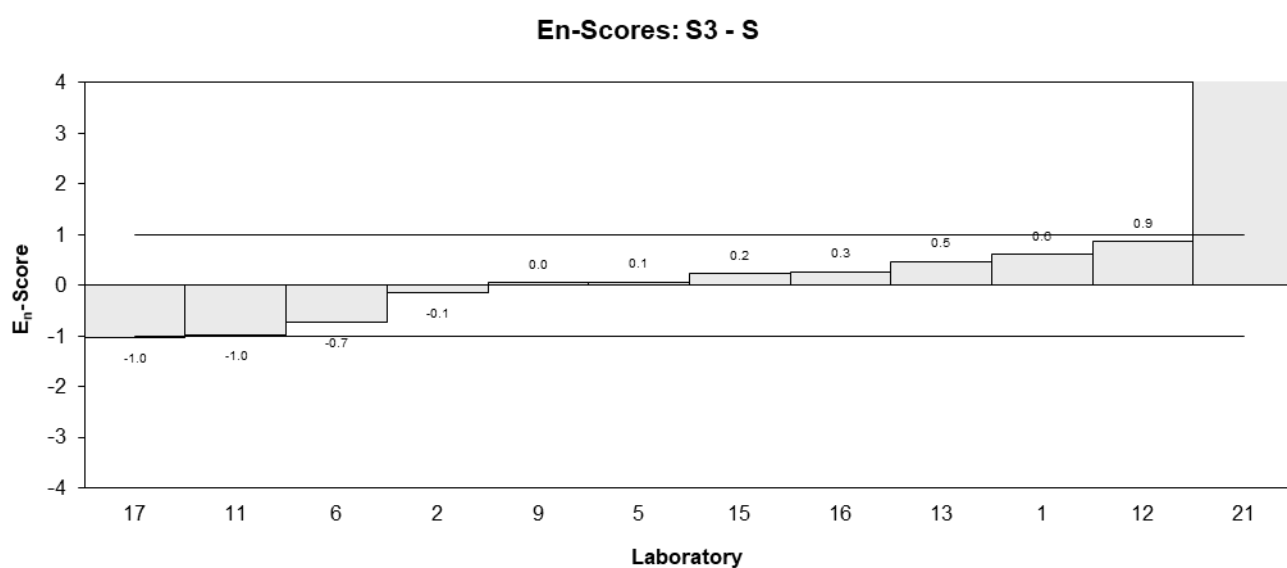
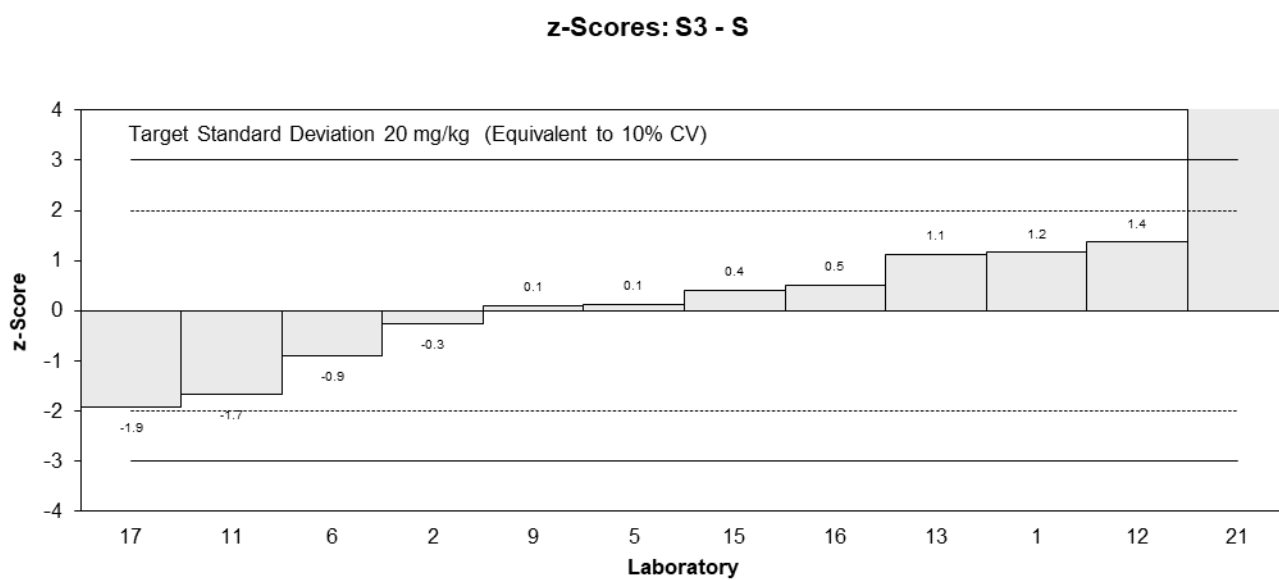
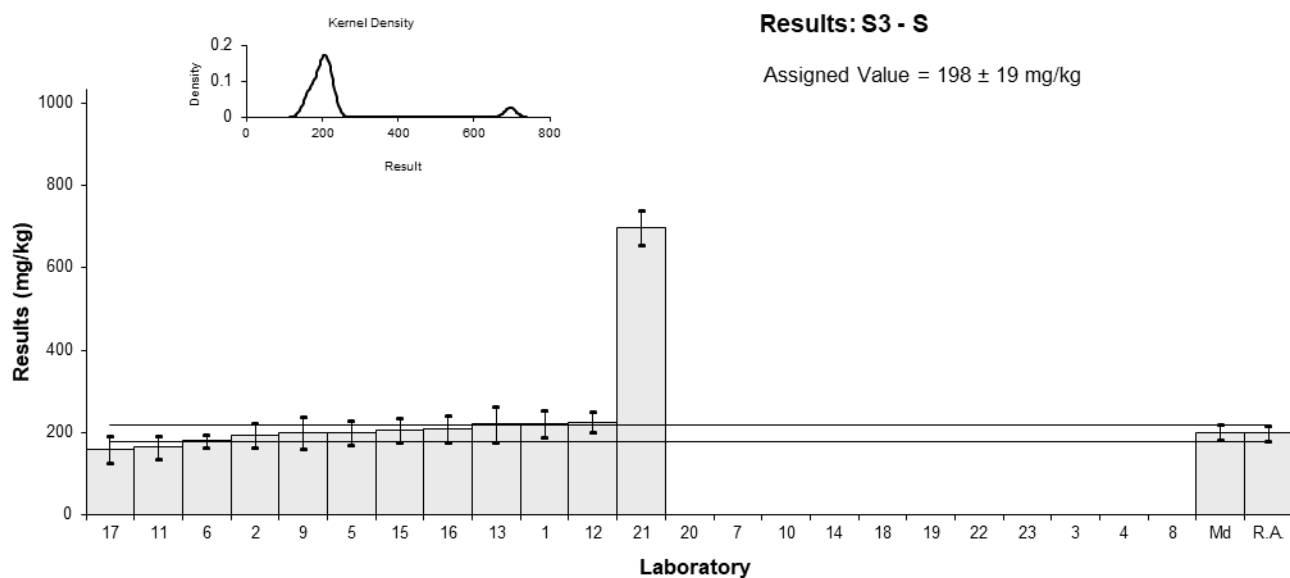


Figure 55

Table 67

Sample Details

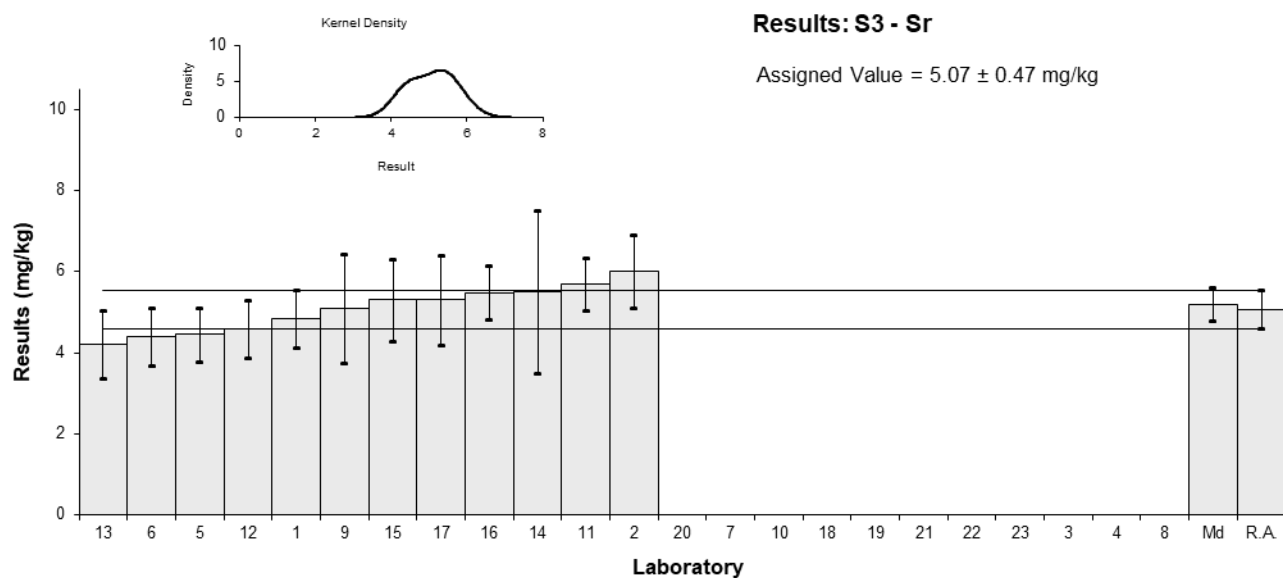
Sample No.	S3
Matrix.	Soil
Analyte.	Sr
Units	mg/kg

Participant Results

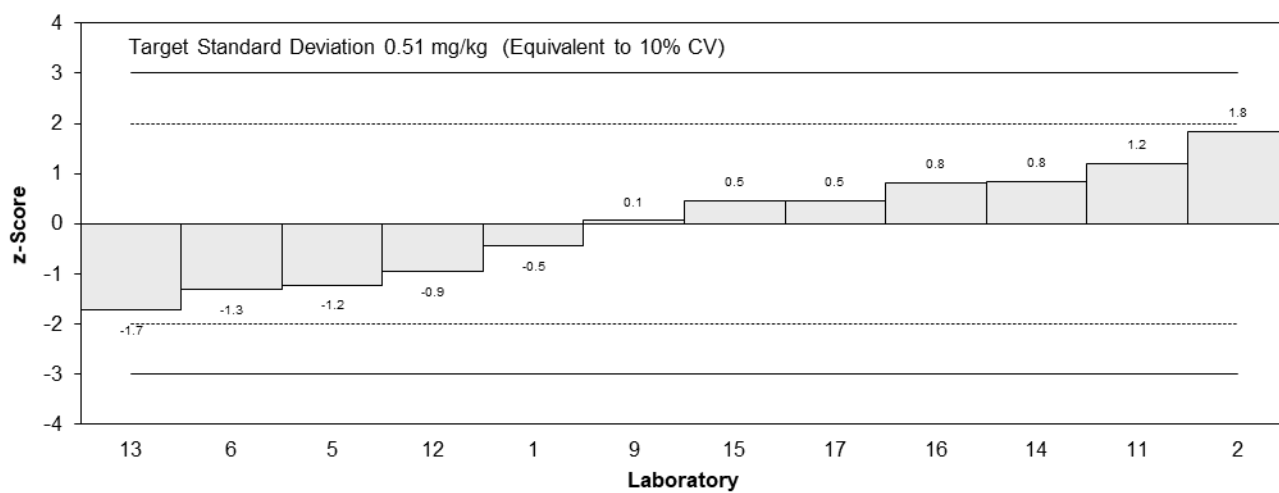
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	4.84	0.7	-0.45	-0.27
2	6	0.9	1.83	0.92
3	NT	NT		
4	NT	NT		
5	4.45	0.67	-1.22	-0.76
6	4.4	0.7	-1.32	-0.79
7	NR	NR		
8	NT	NT		
9	5.1	1.3362	0.06	0.02
10	NT	NT		
11	5.68	0.648	1.20	0.76
12	4.59	0.7	-0.95	-0.57
13	4.2	0.84	-1.72	-0.90
14	5.5	2.0	0.85	0.21
15	5.3	1	0.45	0.21
16	5.48	0.66	0.81	0.51
17	5.3	1.1	0.45	0.19
18	NT	NT		
19	NT	NT		
20	NR	NR		
21	NT	NT		
22	NT	NT		
23	NT	NT		

Statistics

Assigned Value	5.07	0.47
Spike	Not Spiked	
Robust Average	5.07	0.47
Median	5.20	0.40
Mean	5.07	
N	12	
Max.	6	
Min.	4.2	
Robust SD	0.65	
Robust CV	13%	



z-Scores: S3 - Sr



En-Scores: S3 - Sr

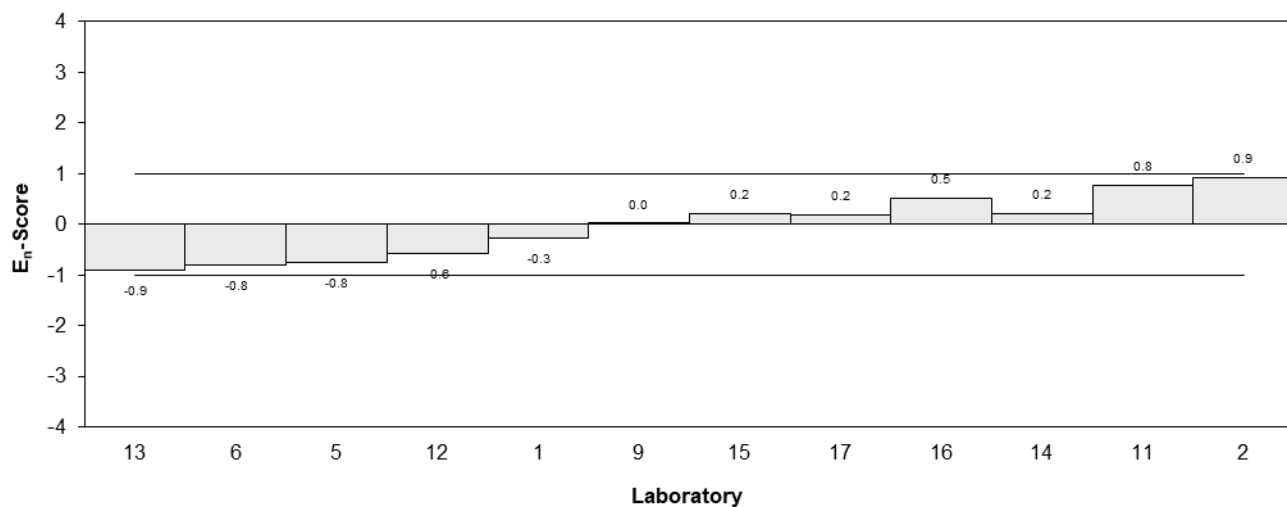


Figure 56

Table 68

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	9300	600	-0.68	-0.78
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	10000	1400	0.02	0.01
7	NR	NR		
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	NT	NT		
12	11400	1140	1.42	1.09
13	NT	NT		
14	10100	1210	0.12	0.09
15	10550	2000	0.57	0.27
16	9820	1180	-0.16	-0.12
17	9100	1820	-0.88	-0.46
18	NT	NT		
19	NT	NT		
20	9950	800	-0.03	-0.03
21	NT	NT		
22	NT	NT		
23	NT	NT		

Statistics

Assigned Value	9980	630
Spike	Not Spiked	
Robust Average	9980	630
Median	9980	450
Mean	10000	
N	8	
Max.	11400	
Min.	9100	
Robust SD	710	
Robust CV	7.1%	

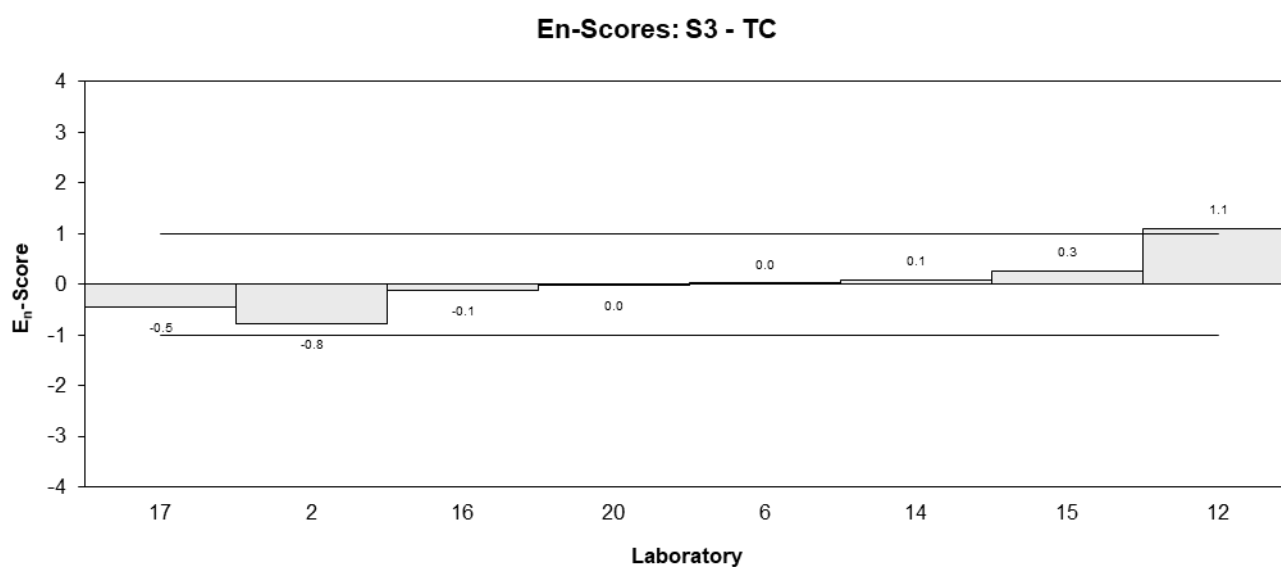
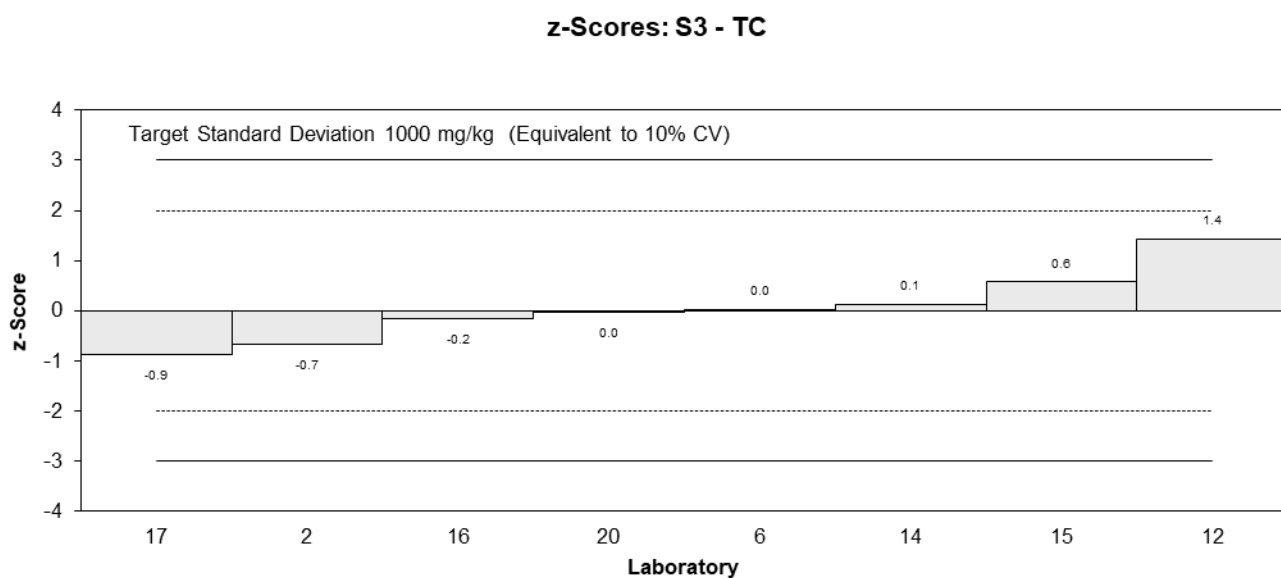
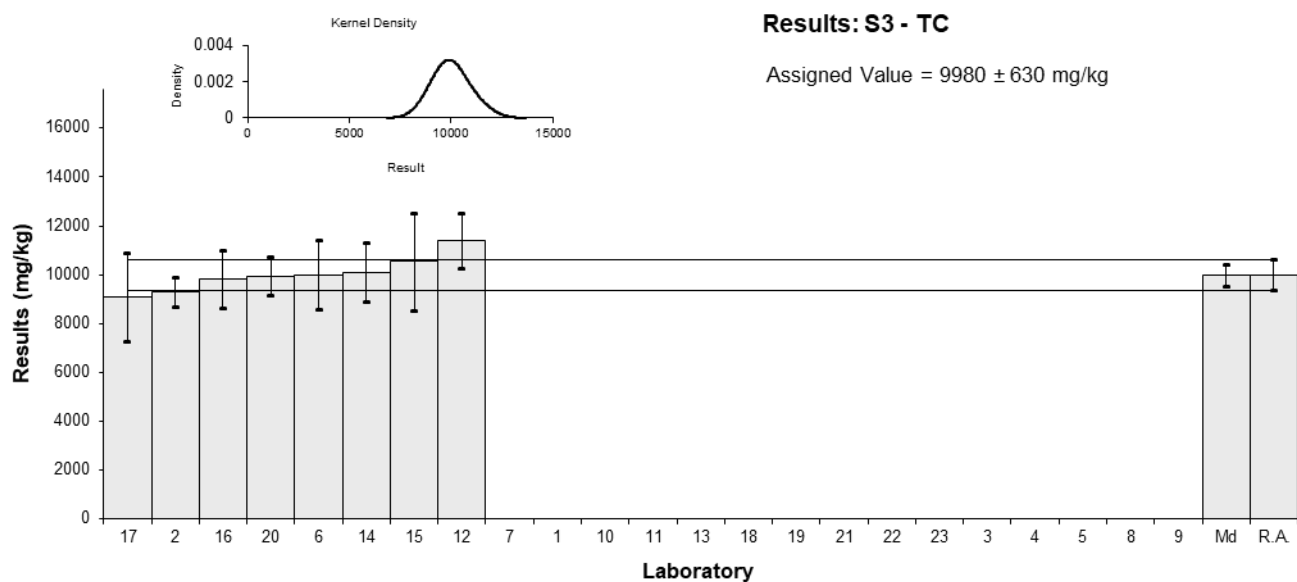


Figure 57

Table 69

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1037	155	2.03	1.03
2	750	80	-1.30	-1.07
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	NT	NT		
7	840	126	-0.26	-0.15
8	NT	NT		
9	920	133	0.67	0.39
10	NT	NT		
11	736	169	-1.46	-0.69
12	750	75	-1.30	-1.11
13	877.1	175	0.18	0.08
14	950	105	1.02	0.70
15	900	90	0.44	0.34
16	860	172	-0.02	-0.01
17	860	170	-0.02	-0.01
18	NT	NT		
19	NT	NT		
20	900	100	0.44	0.31
21	NT	NT		
22	NT	NT		
23	NT	NT		

Statistics

Assigned Value	862	68
Spike	Not Spiked	
Homogeneity Value	1000	130
Robust Average	862	68
Median	869	39
Mean	865	
N	12	
Max.	1037	
Min.	736	
Robust SD	94	
Robust CV	11%	

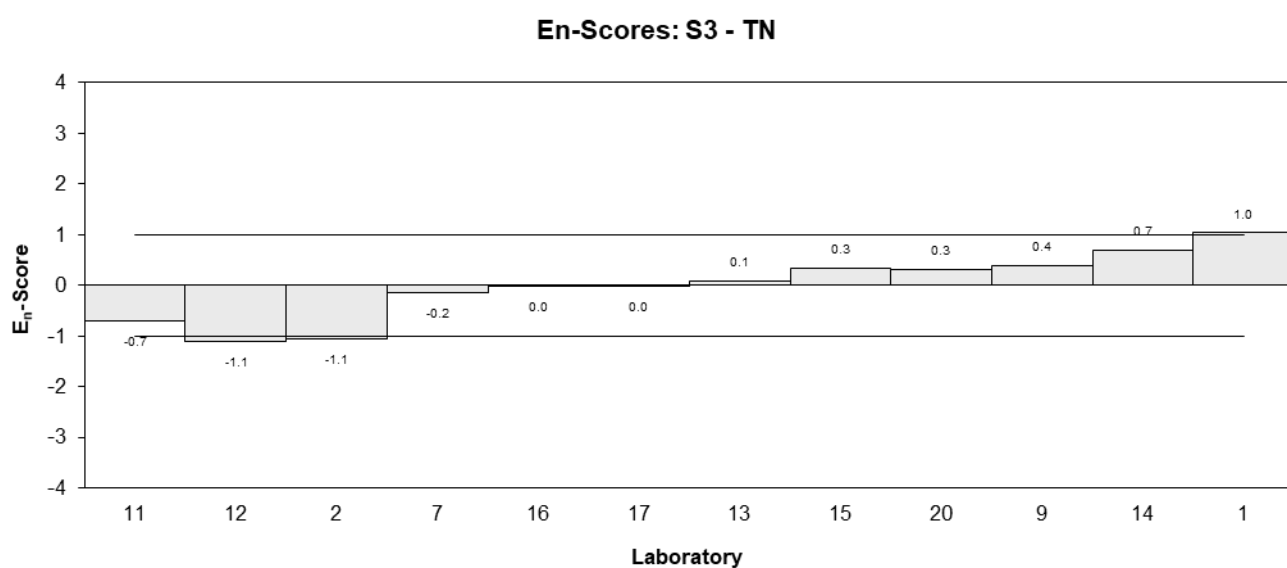
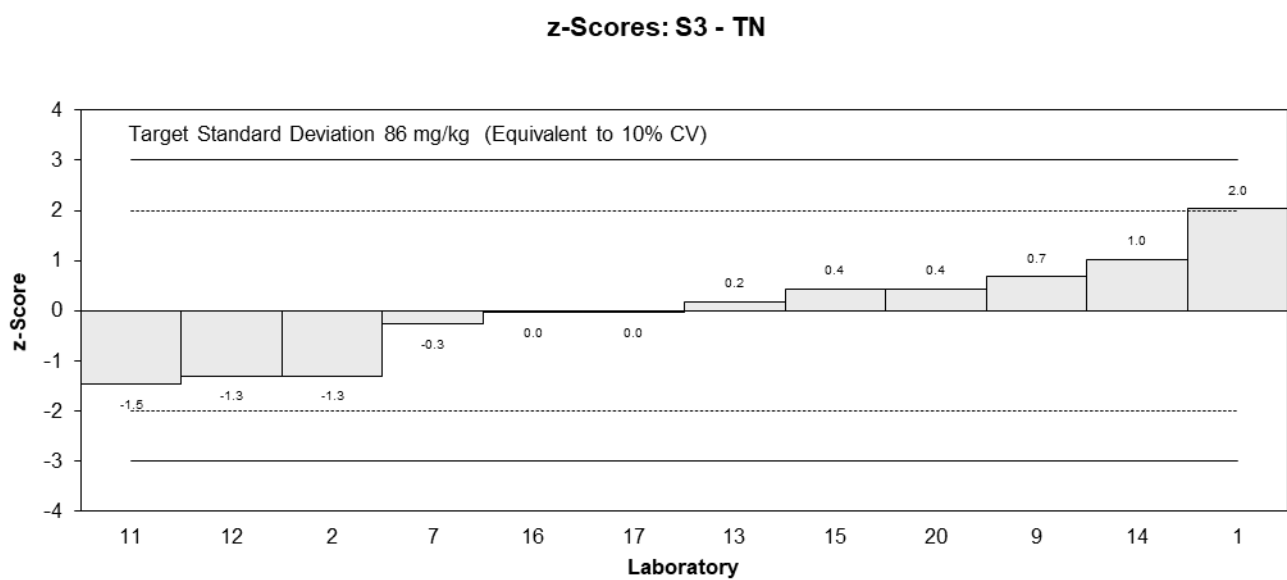
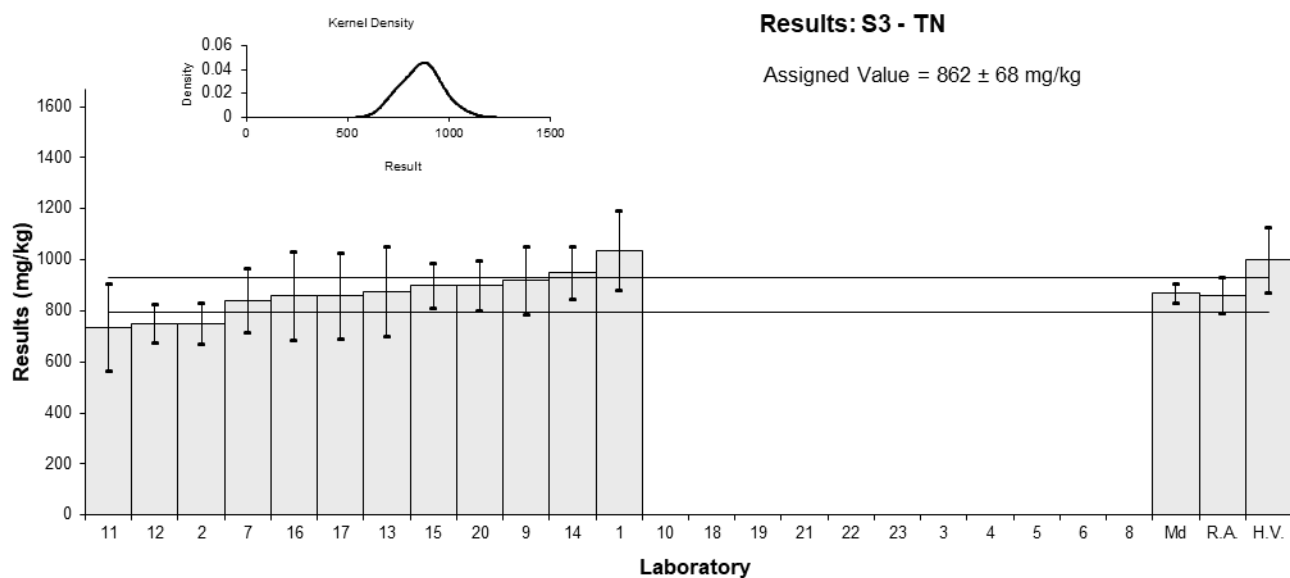


Figure 58

Table 70

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	10060	1500	0.44	0.25
2	9300	600	-0.35	-0.35
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	8500	1290	-1.18	-0.76
7	9000	2000	-0.66	-0.30
8	NT	NT		
9	12000	4252	2.45	0.55
10	NT	NT		
11	8240	898	-1.45	-1.19
12	10900	1090	1.31	0.95
13	1.3	0.26	-10.00	-12.68
14	9700	1190	0.06	0.04
15	10500	2000	0.89	0.40
16	9370	1130	-0.28	-0.20
17	8900	1780	-0.77	-0.38
18	NT	NT		
19	NT	NT		
20	9950	853	0.32	0.27
21	NT	NT		
22	NT	NT		
23	NT	NT		

Statistics*

Assigned Value	9640	760
Spike	Not Spiked	
Homogeneity Value	9200	1100
Robust Average	9640	760
Median	9540	550
Mean	9700	
N	12	
Max.	12000	
Min.	8240	
Robust SD	1100	
Robust CV	11%	

*Laboratory 13 excluded from statistical calculation (extreme outlier).

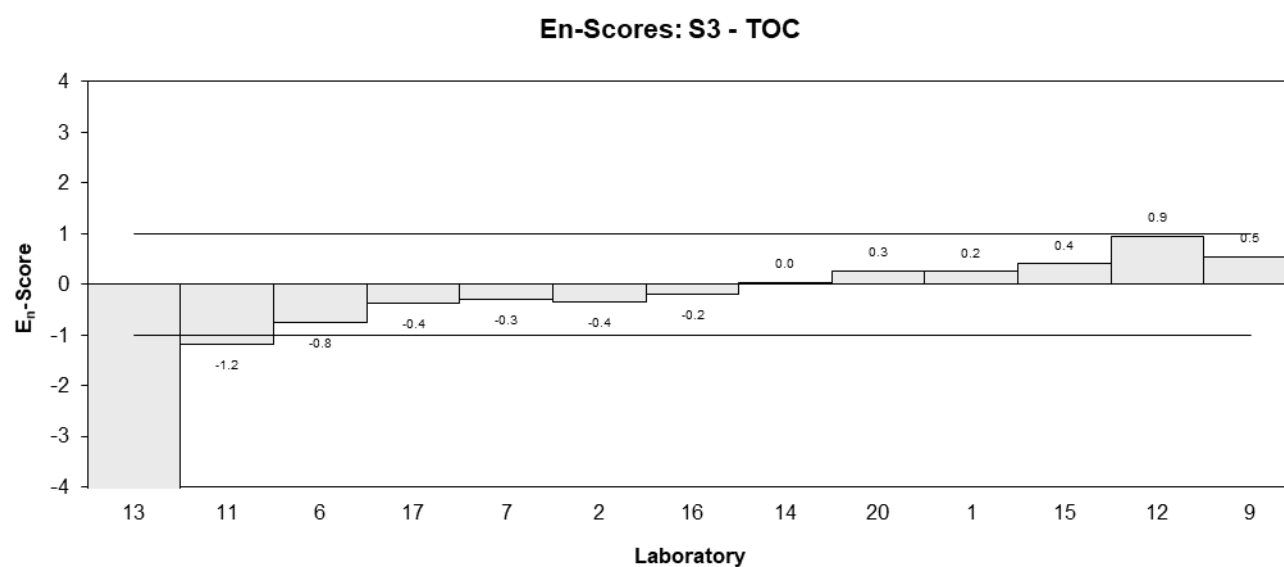
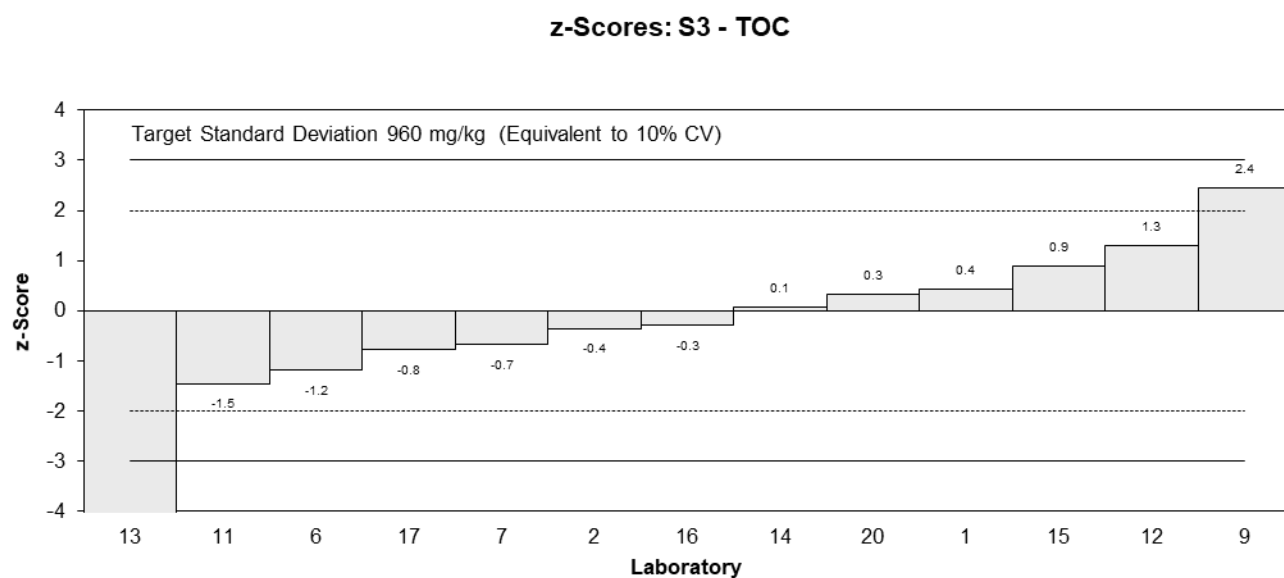
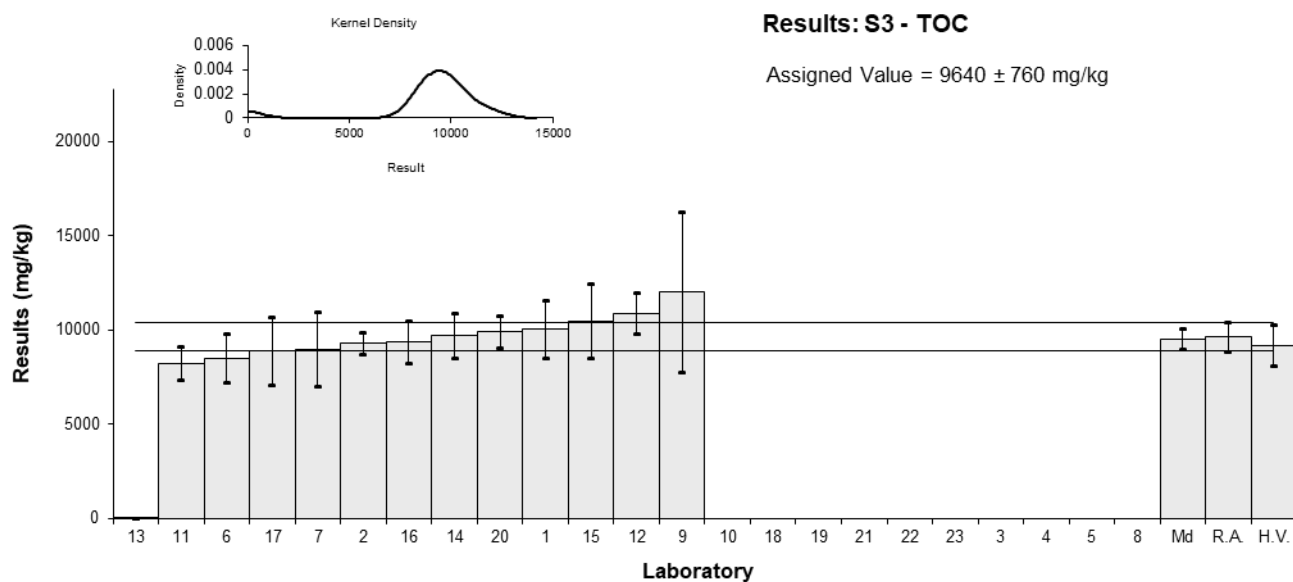


Figure 59

Table 71

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	198	30	0.43	0.30
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	NT	NT		
7	210	31	0.86	0.59
8	NT	NT		
9	220	20	1.22	1.04
10	NT	NT		
11	167	13.36	-0.68	-0.65
12	NR	NR		
13	193	38.6	0.25	0.15
14	NT	NT		
15	156	50	-1.08	-0.53
16	197	60	0.39	0.17
17	147	29	-1.40	-1.00
18	NT	NT		
19	NT	NT		
20	NR	NR		
21	NT	NT		
22	NT	NT		
23	NT	NT		

Statistics

Assigned Value	186	26
Spike	Not Spiked	
Homogeneity Value	145	20
Robust Average	186	26
Median	195	25
Mean	186	
N	8	
Max.	220	
Min.	147	
Robust SD	30	
Robust CV	16%	

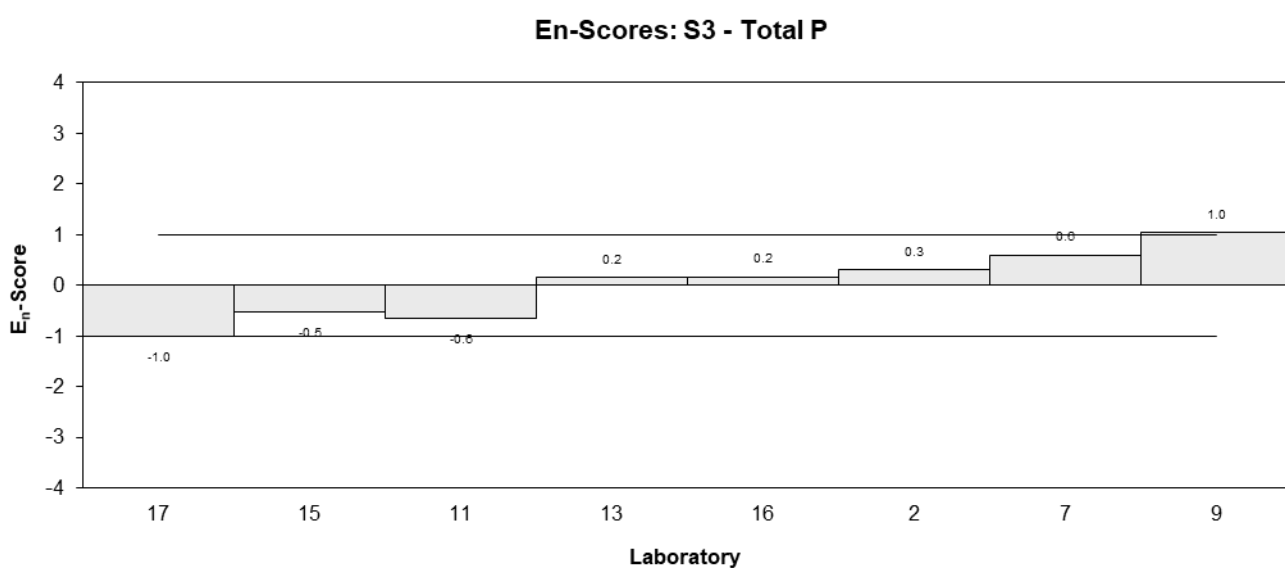
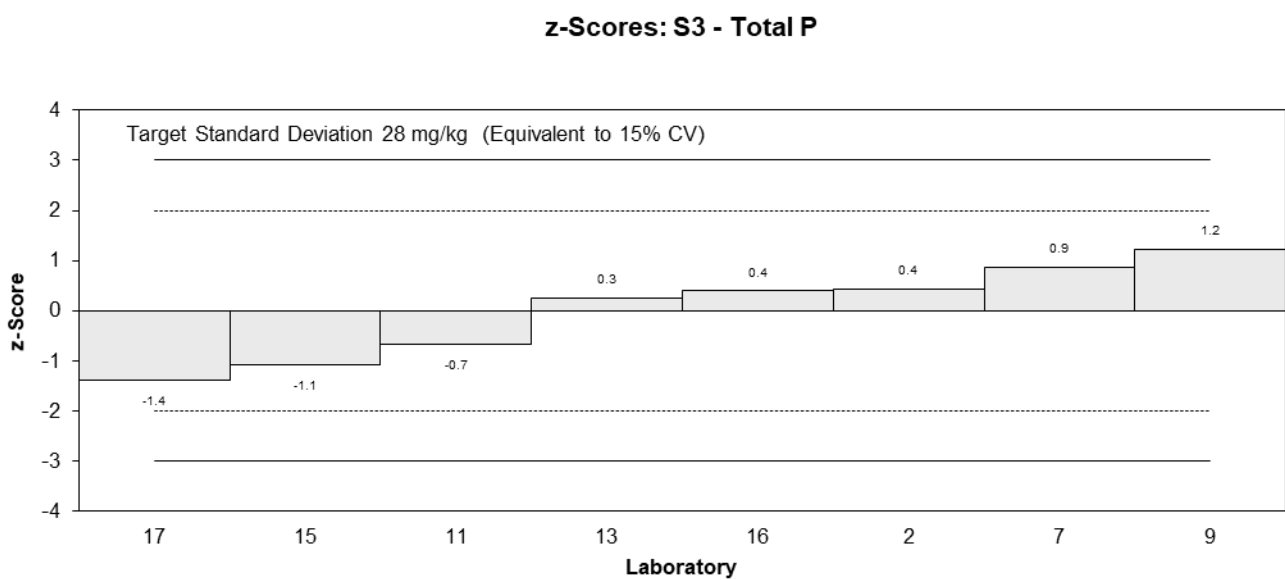
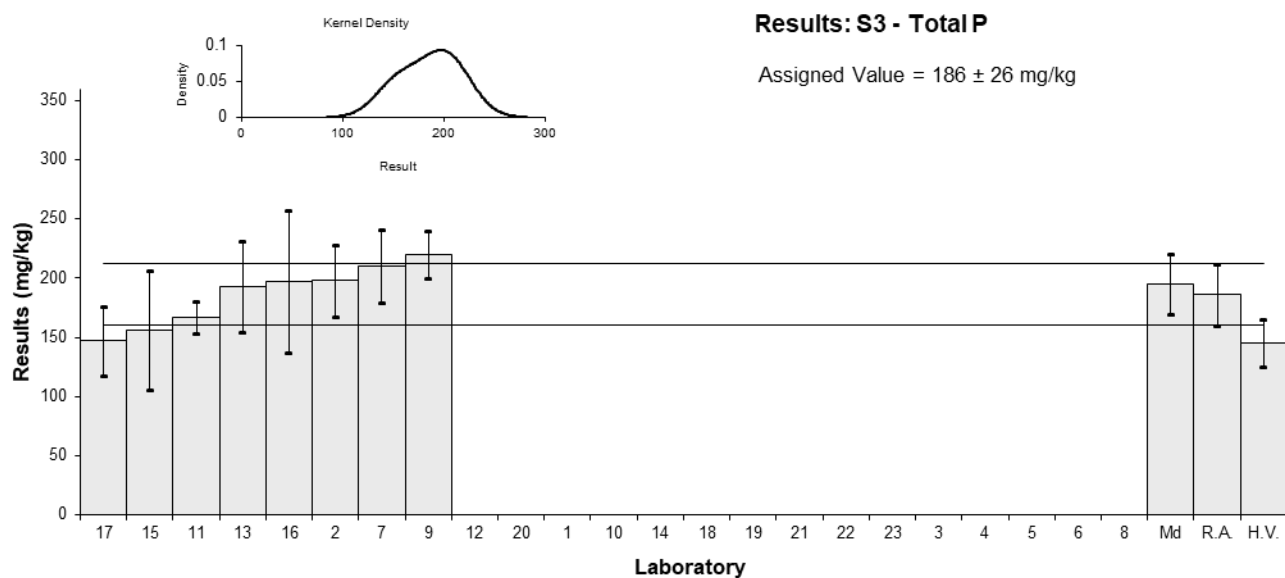


Figure 60

6 DISCUSSION OF RESULTS

6.1 Assigned Value

Sample S1 was dried sediment and **Sample S2** was wet sludge. Participants were asked to report the results for Sample S1 on as received basis and to correct the results for Sample S2 for moisture content.

Sample S3 was dried agricultural soil.

Assigned values for 51 tests in the study samples were the robust averages of participants' results. The robust averages 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 the assigned value.⁶ Appendix 2 sets out the calculation of the robust average of Fe in Sample S3 and its associated uncertainty. Laboratory 10 results were omitted from all statistical calculation as they mislabelled the samples.

No assigned value was set for B, Rb, Sb, Th in S1, Bi, Cs, Tl in S2 and Colwell K, extractable B and phosphorus buffer index in S3 because the reported results were either too variable or too few. However, participants may still compare their reported results for these elements with the robust average of participants' results and/or the homogeneity value. Descriptive statistics for these elements are presented in Section 5. No descriptive statistics were presented for Tl in S2 and extractable B in S3 because only one result was reported for these tests: 0.11 mg/kg for Tl and 0.57 mg/kg for extractable B.

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.

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded measurement uncertainty associated with their results. Of 782 numerical results, 749 were reported with an expanded measurement uncertainty, indicating that not all laboratories have addressed this requirement of ISO 17025.⁸ The participants used a wide variety of procedures to estimate the expanded measurement uncertainty. These are presented in Table 11.

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.⁹⁻¹⁶

Proficiency tests allow a check of participants' uncertainty estimates. Results and the expanded MU are presented in the bar charts for each analyte (Figure 2 to 60). In this study, the reported expanded measurement uncertainty has been over-estimated in some cases (e.g. Lab 2 for Be in Sample S1) or under-estimated (e.g. Lab 21 for Hg and Li in Sample S1). As a simple rule of thumb, when the uncertainty estimate is smaller than the uncertainty of the assigned 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 when preparing uncertainty budgets. According to General Accreditation Guidance-Estimating and Reporting MU of Chemical Test Results¹² and to NORDTEST TR 537,¹⁰ 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.^{10, 12} An example of estimating measurement uncertainty using proficiency testing data only is given in Appendix 3.

Laboratories 8, 9, 13, 19 and 22 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.⁹

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 $383.55 \pm 76.71 \text{ mg/kg}$, it is better to report $384 \pm 77 \text{ mg/kg}$ or instead of $84 \pm 16.1 \text{ mg/kg}$, it is better to report $84 \pm 16 \text{ mg/kg}$.⁹

6.3 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 61. 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 722 results for which E_n-scores were calculated, 564 (78%) 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.

6.4 z-Score

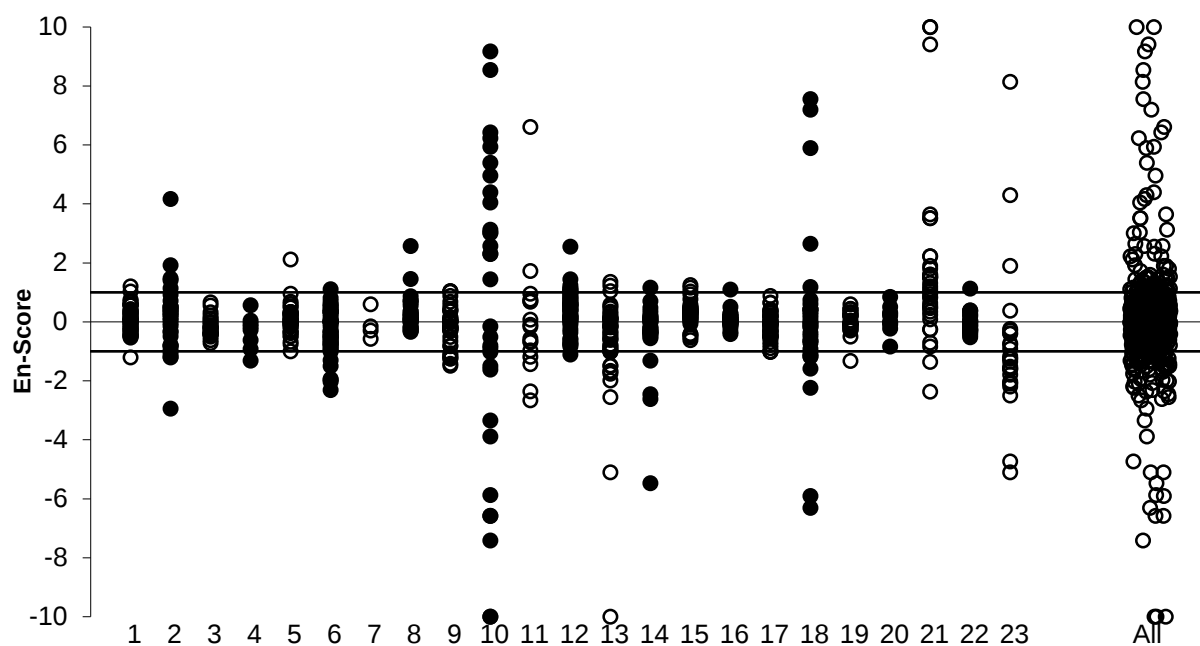
The z-score compares participant's deviation from the assigned value with the target standard deviation set for proficiency assessment.

The target standard deviation defines satisfactory performance in a proficiency test. Target standard deviations equivalent to 3.5%, 10%, 15% and 20% PCV were used to calculate z-scores. Unlike the standard deviation based on between laboratories CV, setting the target

standard deviation as a realistic, set value enables z-scores to be used as a fixed reference value point for assessment of laboratory performance, independent of group performance.

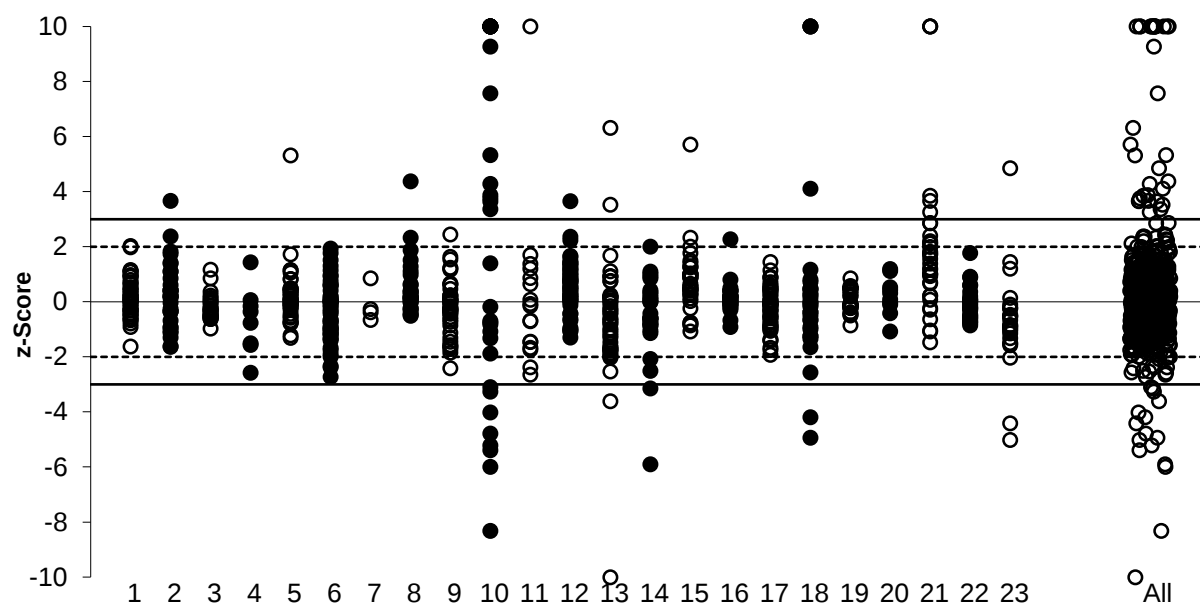
The between laboratory coefficient of variation predicted by the Thompson equation⁷ and the between laboratory coefficient of variation resulted in this study are presented for comparison in Table 72. The dispersal of participants' z-scores is presented in Figure 62 (by laboratory code) and in Figure 63 (by test). Of 722 results for which z-scores were calculated, 645 (89%) returned a satisfactory score of $|z| \leq 2.0$ and 25 (3%) were questionable of $2.0 < |z| < 3.0$. Participants with multiple z-scores larger than 2.0 or smaller than -2.0 should check for laboratory bias.

Laboratories 17 and 12 reported results for 47 of a total of 51 analytes for which z-scores were calculated. All results reported by Laboratory 17 returned satisfactory z-scores; Laboratory 17 also had the highest number of satisfactory E_n -scores, 46. Laboratory 12 returned satisfactory results for 44 results out of a total of 47 reported.



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

Figure 61 E_n -Score Dispersal by Laboratory



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

Figure 62 z-Score Dispersal by Laboratory

Table 72 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 PCV)
S1	As	6.26	6.9%	12%	10%
S1	B	Not Set	30%	NA	Not Set
S1	Ba	80.8	11%	8.3%	10%
S1	Be	0.603	12%	17%	15%
S1	Cd	1.97	7.4%	14%	10%
S1	Cr	80.1	9.9%	8.3%	10%
S1	Cu	68.1	6%	8.5%	10%
S1	Hg	0.642	16%	17%	15%
S1	Li	7.5	22%	12%	20%
S1	Mn	256	8.6%	6.9%	10%
S1	Ni	20.2	17%	10%	10%
S1	Pb	39.1	7.8%	9.2%	10%
S1	Rb	Not Set	50%	NA	Not Set
S1	Sb	Not Set	44%	NA	Not Set
S1	Se	8.08	11%	12%	15%
S1	Sn	1.44	11%	15%	10%
S1	Th	Not Set	66%	NA	Not Set
S1	V	37.2	9.2%	9.3%	10%
S1	Zn	93.1	7.2%	8.1%	10%
S2	Ag	6.19	8.6%	12%	10%
S2	Al	9600	22%	4%	20%
S2	As	3.58	13%	13%	10%
S2	Ba	129	11%	7.7%	10%
S2	Bi	Not Set	54%	NA	Not Set
S2	Cd	0.718	12%	17%	10%
S2	Co	8.36	7.3%	12%	10%
S2	Cr	32.8	9.7%	9.5%	10%
S2	Cs	Not Set	40%	NA	Not Set
S2	Cu	158	10%	7.5%	10%
S2	Hg	0.474	17%	18%	15%
S2	La	11.5	22%	11%	15%
S2	Mo	1.38	19%	15%	15%
S2	Ni	16.5	13%	10%	10%
S2	Pb	32.0	9.3%	9.5%	10%
S2	Se	1.18	25%	16%	20%
S2	U	1.14	16%	16%	15%
S2	Zn	179	5.6%	7.3%	10%
S2	Moisture content	43.0 %	5.1%	9.1%	10%
S3	Ca	478	7%	6.3%	10%
S3	EC	293 μ S/cm	6.2%	6.8%	10%
S3	Colwell P	61.5	6.7%	8.6%	10%
S3	Colwell K	Not Set	13%	NA	Not Set
S3	Exchangeable Ca^{2+}	1.74 cmol(+)/kg	19%	15%	15%
S3	Exchangeable Mg^{2+}	0.871 cmol(+)/kg	7%	16%	15%
S3	Exchangeable Na^+	0.262 cmol(+)/kg	14%	20%	15%
S3	Exchangeable K^+	0.0592 cmol(+)/kg	13%	22%	15%
S3	Fe	4070	10%	4.6%	10%
S3	K	110	16%	7.9%	10%
S3	Mg	224	18%	7.1%	15%
S3	Na	85.6	15%	8.2%	10%
S3	P	170	13%	7.4%	10%

Sample	Test	Assigned Value (mg/kg)	Between Laboratories CV*	Thompson/Horwitz CV	Target SD (as PCV)
S3	PBI+ColP	Not Set	11%	NA	Not Set
S3	Total P	186	16%	7.3%	15%
S3	pH	4.58	5.8%	13%	3.5%**
S3	S	198	12%	7.2%	10%
S3	Sr	5.07	13%	13%	10%
S3	TC	9980	7.1%	4%	10%
S3	TOC	9640	11%	4%	10%
S3	TN	862	11%	5.8%	10%

*Robust between Laboratories CV with outliers removed; N/A = Not Applicable, **As per APHA Method 4500H, requirements for precision and bias. NA = Not applicable

6.5 Participants' Results and Analytical Methods for Acid Extractable Elements

A summary of participants' results and performance is presented in Tables 73 to 75 and in Figures 62 and 63.

B and Sb in S1 were the most difficult elements to analyse. No agreement was found between the results reported by participants for these elements.

Arsenic in S1 and Zn and moisture content in S2 were the tests that presented the least analytical difficulty to participating laboratories with a between laboratory CV of less than 6%.

Subsampling of the wet sludge sample S2 has not presented difficulties to participating laboratories. The between-laboratory CVs in the wet sludge Sample S2 were comparable with the between-laboratory CVs of the dried sample S1.

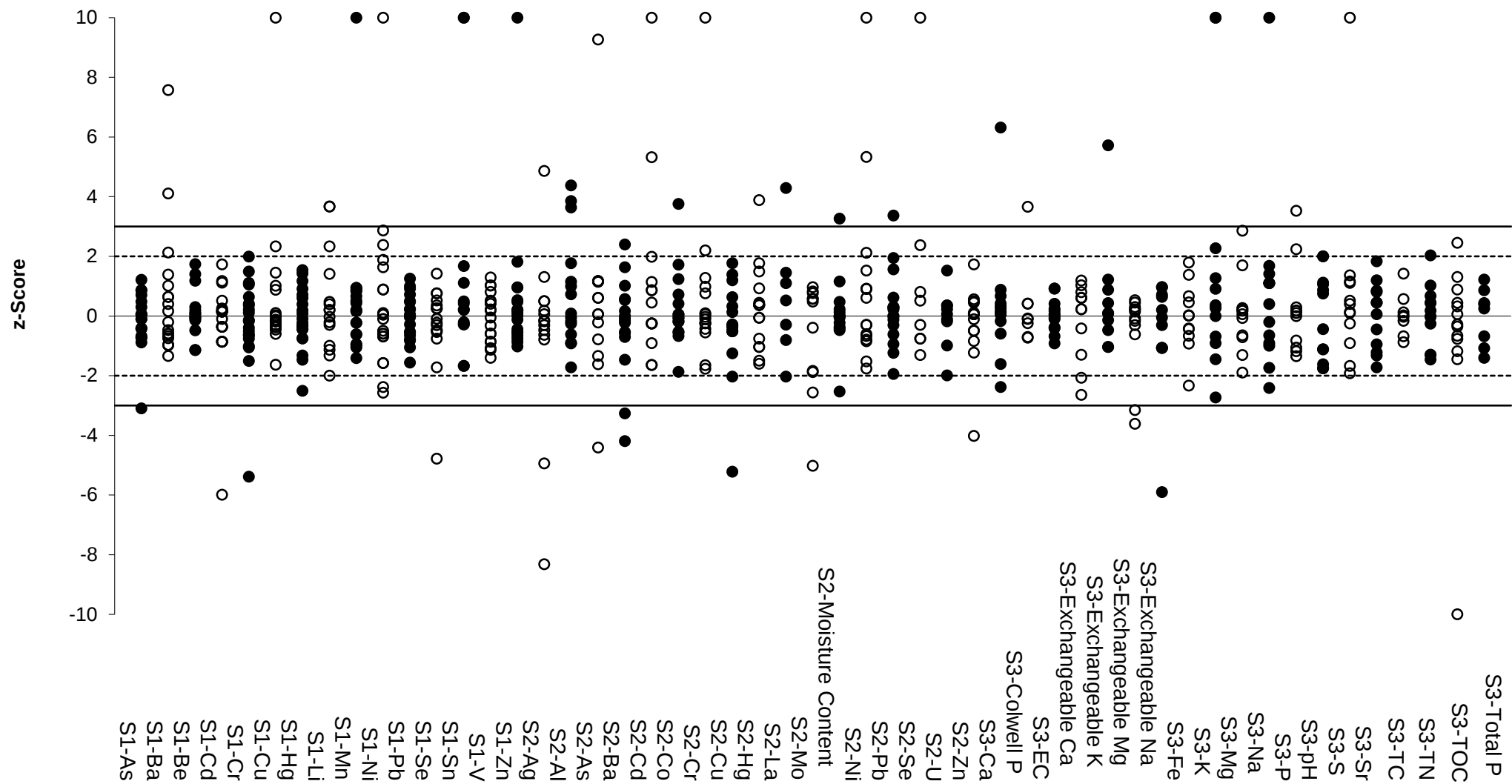
Participation in proficiency testing is intended to assess laboratories' compliance with ISO 17025 (including their ability to report results in the format requested by their client). Laboratory 11 reported S as sulphate while Laboratory 3 corrected results for moisture content in all three study samples.

Laboratory 10 mislabelled the samples. The results from this laboratory were not included in the analysis of the extraction methods and instrumental techniques employed by participants.

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

Extraction Methods

In previous studies conducted by NMI for trace elements in garden soil, compost, sediment or clay, relationships were found to exist between the results reported for Al, Cr, Ni, V and extraction regime employed. In the present study, the samples were dried sediment and wet sludge. Participants used various sample sizes (from 0.5 g to 2.5 g), digestion temperatures (from 85°C to 120°C) and digestion times (from 30 min to 240 min); no relationship was evident between extraction method employed and the results reported for targeted analytes including for the method dependent ones.



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

Figure 63 z-Score Dispersal by Element

Table 73 Summary of Participants' Results and Performance for Sample S1

Lab Code	As (mg/kg)	B (mg/kg)	Ba (mg/kg)	Be (mg/kg)	Cd (mg/kg)	Cr (mg/kg)	Cu (mg/kg)	Hg (mg/kg)	Li (mg/kg)	Mn (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Rb (mg/kg)	Sb (mg/kg)	Se (mg/kg)	Sn (mg/kg)	Th (mg/kg)	V (mg/kg)	Zn (mg/kg)
A.V.	6.26	Not Set	80.8	0.603	1.97	80.1	68.1	0.642	7.5	256	20.2	39.1	Not Set	Not Set	8.08	1.44	Not Set	37.2	93.1
H.V.	6.57	5.53	75.9	0.578	2.10	81.2	66.8	0.640	6.03	217	20.5	33.8	10.8	23.8	8.07	1.23	5.80	35.5	91
1	5.84	6.16	79.2	0.62	2.19	85.2	68.3	0.73	7.05	273	20.4	41.9	NT	35.8	7.43	1.47	NT	39.1	102
2	6.8	9.7	92	0.76	1.9	83	57	0.61	13	232	25	39	22	18	9.8	1.6	5.7	38	95
3	6.8	< 10	73	< 2	2.0	77	66	0.65	NR	240	19	39	NR	35	7.5	< 10	NR	36	89
4	6.3	NT	NT	NT	1.9	68	67	0.78	NT	NT	15	33	NT	NT	NT	NT	NT	NT	86
5	6.45	NT	74.79	0.61	2.31	81.75	68.09	0.68	NT	277.36	20.29	40.98	NT	34.28	7.16	1.51	NT	38.72	97.47
6	5.8	<50	70.0	0.5	1.9	71.7	67.3	0.6	4.5	231	15.4	42.7	7.6	11.9	9	1.2	1.2	32	83.6
7	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
8	6	<10	86	<2	2	89	66	0.66	11	260	24	39	NT	33	8.4	<10	NT	42	94
9	6	<5	86	0.6	1.9	75	67	0.79	NT	250	17	40	NT	8	6	<3	NT	34	88
10	4.32	3.73	142	0.73	0.79	36.9	170	0.57	5.52	524	18.8	35.9	18.3	1.32	2.29	17.8	NT	33.2	206
11	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
12	7.02	8.12	82.1	0.71	2.02	88.5	74.1	0.71	9.61	261	23.5	39.3	NT	33.4	8.71	1.68	3.34	40.2	102
13	6.3	<50	73	0.6	2	75	67	0.6	5.8	280	18.8	38	9.2	21	9	1.4	1.6	35	95
14	6.0	4.8	76	NT	1.8	74	68	0.40	NT	240	19	36	NT	22	8.5	1.5	NT	33	85
15	<25	<5	89	<1	1.8	92	84	0.57	8.2	278	22	44	NR	<100	<100	<50	NR	41	98
16	6.29	5.55	75.4	0.596	2.01	83.4	68.8	0.633	8.08	267	20.3	40.0	NR	37.0	7.97	1.41	NR	37.9	94.8
17	5.7	7.0	73	0.56	1.8	72	64	0.61	7.8	220	20	35	12	24	7.5	1.2	3.8	35	88
18	6.56	4.95	114	0.63	1.95	78.7	65.5	0.755	7.46	260	78.31	36.57	NT	NT	8.38	3.77	NT	39.0	93.2
19	6.2	< 20	84.0	0.59	2.07	79	66.8	0.62	7.19	269	19.2	41.0	12.8	21.5	< 20	1.47	NT	< 100	94.9
20	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
21	6.7	NT	98	0.61	2.2	96	75	0.5	13	280	26	43	NT	30	7.8	1.4	NT	41	110
22	5.8	<10	76	<2	1.8	81	65	0.7	7.8	250	22	37	NT	33	7.7	<10	NT	37	92
23	6.2	14	77	0.5	2.0	76	78	0.515	6.0	229	17	37	NT	6.5	NT	NT	NT	34	87

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

Table 74 Summary of Participants' Results and Performance for Sample S2

Lab Code	Ag (mg/kg)	Al (mg/kg)	As (mg/kg)	Ba (mg/kg)	Bi (mg/kg)	Cd (mg/kg)	Co (mg/kg)	Cr (mg/kg)	Cs (mg/kg)	Cu (mg/kg)	Hg (mg/kg)	La (mg/kg)	Mo (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Se (mg/kg)	Tl (mg/kg)	U (mg/kg)	Zn (mg/kg)	Moisture Content (%)
A.V.	6.19	9600	3.58	129	Not Set	0.718	8.36	32.8	Not Set	158	0.474	11.5	1.38	16.5	32.0	1.18	Not Set	1.14	179	43.0
H.V.	5.82	9200	3.99	122	0.750	0.783	7.58	33.2	0.84	162	0.467	12.5	1.37	17.0	26.7	1.25	0.11	1.02	175	43.3
1	6.08	9750	3.30	128	1.81	0.86	8.31	33.1	NT	153	0.54	13.4	1.58	15.5	32.7	1.11	<0.1	1.15	188	41.2
2	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
3	6.1	8400	4.0	126	<10	0.7	7.9	31	NR	150	0.50	NR	< 5	16	32	< 2	< 10	< 10	170	44
4	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
5	NT	9254.31	3.99	121.70	NT	1.10	7.92	32.45	NT	153.65	0.47	NT	1.55	15.19	32.9	0.87	NT	NT	180.37	48
6	7.0	7830	3.6	129	0.9	0.8	8.4	32.7	0.6	168	0.6	11.0	1.5	15.4	38.2	1	<0.1	1.4	181	43.0
7	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
8	5.9	18000	3.8	131	<10	0.7	8.7	36	NT	150	0.5	NT	<5	19	32	<2	<10	1.2	180	42
9	6	9100	4	150	NT	0.6	9.4	32	NT	160	0.36	NT	1	16	34	<3	NT	NT	180	45
10	1.04	16580	6.9	87	0.13	2.17	11.5	90.5	1.35	75.5	0.75	18.9	0.99	25.3	42.8	10.3	NT	1.11	107	NT
11	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
12	6.23	11520	3.99	136	0.68	0.78	8.96	35.3	NT	163	0.58	NT	1.54	17.5	31.6	1.74	0.11	1.18	187	42.9
13	6.3	6290	3	110	0.7	0.6	6.8	27	0.5	126	0.4	10.1	1.3	13.6	25.8	1	<0.1	0.8	157	32.1
14	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
15	NR	NR	<25	142	NR	<1	NR	37	NR	186	0.42	NR	NR	18	33	<100	NR	NR	189	43.5
16	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
17	6.5	11800	3.6	127	0.78	0.75	8.4	32	1.1	153	0.47	14	1.5	18	31	1.3	<0.5	1.2	180	42.8
18	3.13	11000	3.10	75	NT	0.653	7.80	27.4	NT	138.3	0.505	NT	0.851	71.4	28.02	1.37	NT	0.971	170.3	41.4
19	6.5	9500	3.5	136.4	0.82	0.78	8.3	31.4	0.82	152	0.50	12.4	1.48	15.1	32.9	< 20	< 0.2	1.122	177	41
20	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
21	5.8	17000	<5	160	NT	<1	9.8	40	NT	180	0.4	NT	<5	20	37	<5	<50	NT	210	57
22	5.7	13000	3.8	120	<10	0.7	8.2	33	NT	150	0.5	NT	<5	18	30	<2	<10	<10	180	43
23	9.2	7866	2.0	122	1.8	0.7	8.2	32	NT	177	0.367	8.0	0.34	14	29	NT	NT	NT	164	NR

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

Table 75 Summary of Participants' Results and Performance for Sample S3

Lab Code	Ca (mg/kg)	Extractable-B (mg/kg)	Colwell-P (mg/kg)	Colwell-K (mg/kg)	EC (μ S/cm)	Exchangeable-Ca (cmol+)/kg)	Exchangeable-Mg (cmol+)/kg)	Exchangeable-Na (cmol+)/kg)	Exchangeable-K (cmol+)/kg)	Fe (mg/kg)	K (mg/kg)
A.V.	478	Not Set	61.5	Not Set	293	1.74	0.871	0.262	0.0592	4070	110
H.V.	485	N/A	56.0	N/A	N/A	N/A	N/A	N/A	N/A	4000	80
1	494	NT	NT	NT	305	1.95	0.91	0.25	0.055	4075	100
2	401	NT	64.1	32	266	1.90	0.89	0.27	0.063	NR	NR
3	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
4	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
5	498.10	NT	NT	NT	291	NT	NT	NT	NT	3892.36	102.40
6	470	<0.2	64	<100	298	1.9	0.9	0.3	<0.1	3120	80
7	NR	NR	NR	NR	282	NR	NR	NR	NR	NR	NR
8	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
9	480	<5	57	NT	300	1.8	0.83	0.26	0.06	3900	94
10	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
11	364	NT	57.2	38.4	295	1.05	0.860	0.287	0.058	4630	222
12	484	0.57	84	31	273	1.63	0.79	0.22	0.05	4258	114
13	780	NT	NT	NT	320	1.4	0.4	<0.1	<0.1	4080	120
14	450	NT	NT	NT	320	1.2	0.46	0.03	0.05	3800	110
15	490	<1	60	NR	294	2.01	0.93	0.30	0.11	4340	124
16	484	NR	NR	NR	NR	NR	NR	NR	NR	4080	135
17	520	NR	61	NR	290	1.9	0.85	0.29	0.067	3690	113
18	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
19	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
20	NR	NR	60.9	NR	281	2.05	0.94	0.22	0.06	NR	NR
21	510	NT	NT	NT	NT	1.8	0.9	0.22	0.07	4800	1100
22	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
23	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT

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

Table 75 Summary of Participants' Results and Performance for Sample S3 (Continued)

Lab Code	Mg (mg/kg)	Na (mg/kg)	P (mg/kg)	Total P (mg/kg)	PBI _{1-ColP} (mg/kg)	pH	S (mg/kg)	Sr (mg/kg)	TC (mg/kg)	TOC (mg/kg)	TN (mg/kg)
A.V.	224	85.6	170	186	Not Set	4.58	198	5.07	9980	9640	862
H.V.	N/A	82.4	200	145	N/A	N/A	N/A	N/A	N/A	9200	1000
1	202	95	173	NT	NT	4.32	221	4.84	NT	10060	1037
2	NR	95	152	198	41.3	4.40	193	6	9300	9300	750
3	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
4	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
5	221.50	83.90	171.65	NT	NT	4.75	200.50	4.45	NT	NT	NT
6	160	80	150	NT	NT	4.3	180	4.4	10000	8500	NT
7	NR	NR	NR	210	NR	NR	NR	NR	NR	9000	840
8	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
9	180	65	170	220	NT	4.4	200	5.1	NT	12000	920
10	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
11	281	70.7	NT	167	49.2	4.72	165	5.68	NT	8240	736
12	226	77	208	NR	39	4.51	225	4.59	11400	10900	750
13	230	100	230	193	NT	4.7	220	4.2	NT	1.3	877.1
14	230	95	175	NT	NT	4.9	NT	5.5	10100	9700	950
15	233	97.7	156	156	47	4.9	206	5.3	10550	10500	900
16	232	77.8	173	197	NR	NR	208	5.48	9820	9370	860
17	200	89	147	147	NR	4.3	160	5.3	9100	8900	860
18	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
19	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
20	NR	NR	NR	NR	40.8	4.76	NR	NR	9950	9950	900
21	320	390	NT	NT	NT	NT	696	NT	NT	NT	NT
22	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
23	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT

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

Individual Element Commentary

Aluminium Calculation error may explain the high unsatisfactory results reported by Laboratories 8 and 21. The results they have reported were higher than the assigned value by a factor of approximately 2.

Plots of results for Al versus instrumental technique used are presented in Figure 64.

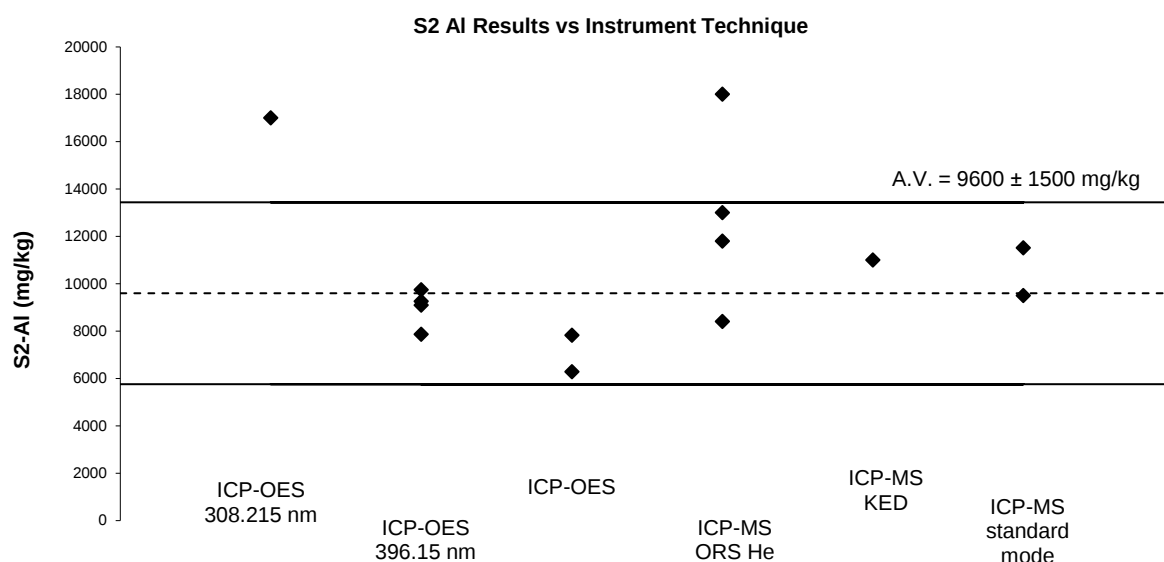


Figure 64 Al Results vs. Instrumental Technique

Antimony in the soil sample S1 was one of the most difficult elements to analyse. Participants used a wide variety of digestion methods; no agreement was found between the results they produced. Antimony is an element whose recovery strongly depends on the acids employed for digestion. It is known that in nitric acid only, Sb is transformed in a mixture of insoluble oxides (Sb_2O_3 , Sb_2O_5 , $\text{Sb}_4\text{O}_4(\text{OH})_2(\text{NO}_3)_2$) but when hydrochloric acid is also involved it changes into chloro-complexes (SbCl_6^-). In aqueous solution, sufficient hydrogen ion concentration must be maintained in order to prevent SbCl_6^- hydrolysis.¹⁷⁻¹⁰ Laboratories should consider increasing their estimates of uncertainty for Sb measurements in soil.

There was no evident relationship between participants' results for Sb and the instrumental technique they used (Figure 65).

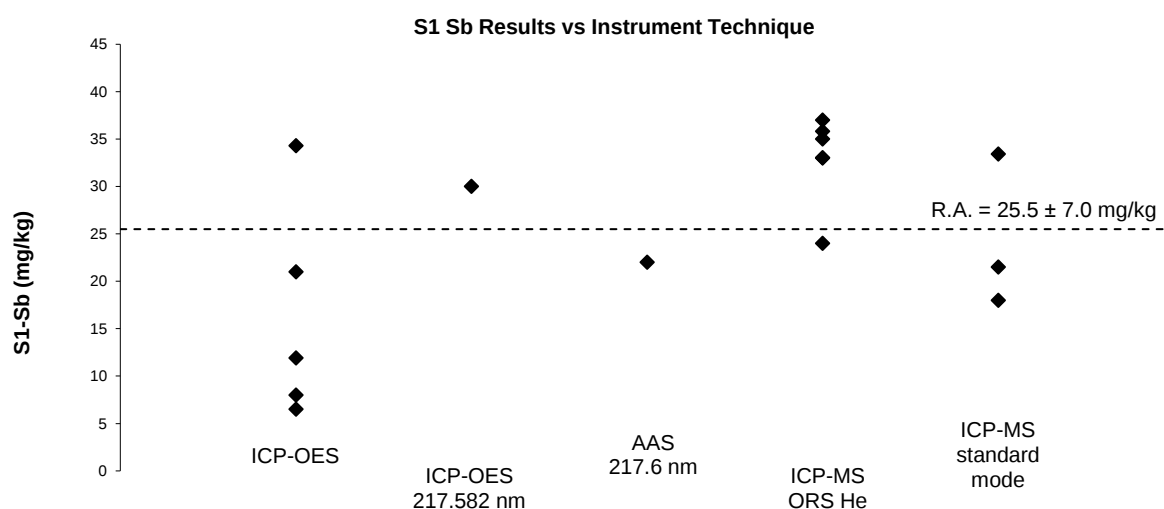


Figure 65 Sb Performance in S1 vs. Instrumental Technique

Arsenic measurements in the two study samples did not present difficulty to participating laboratories, as all reported results returned satisfactory results except for one. For ICP-OES, As sensitivity is low at 193 nm, better use 188 nm (Figure 66).

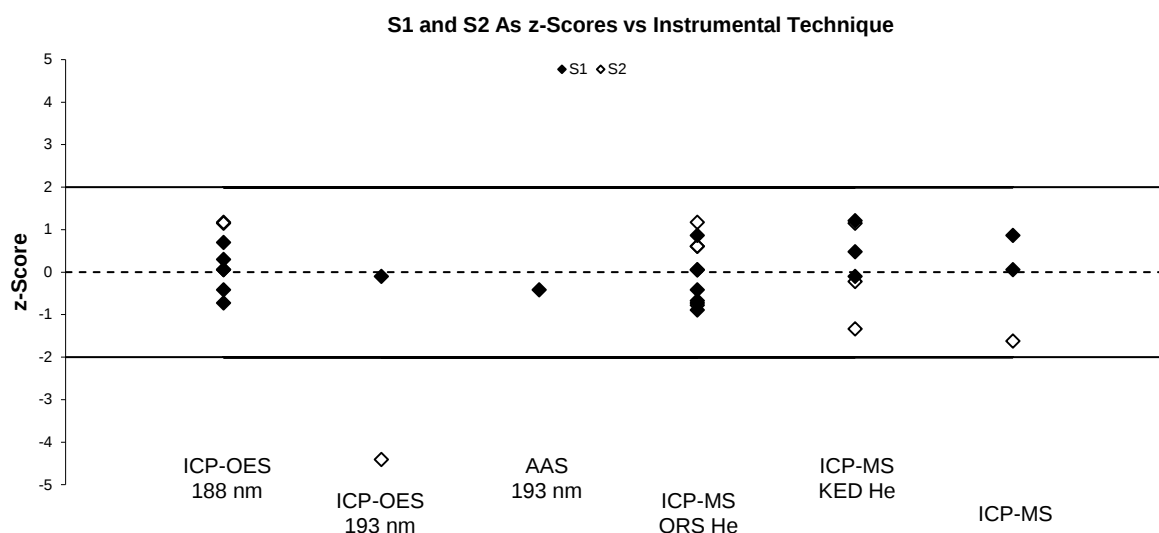


Figure 66 As Performance in S1 and S2 vs. Instrumental Technique

Boron level in Sample S1 was low, which may have presented difficulty to some laboratories. The between-laboratory CV was high (30%).

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 67.

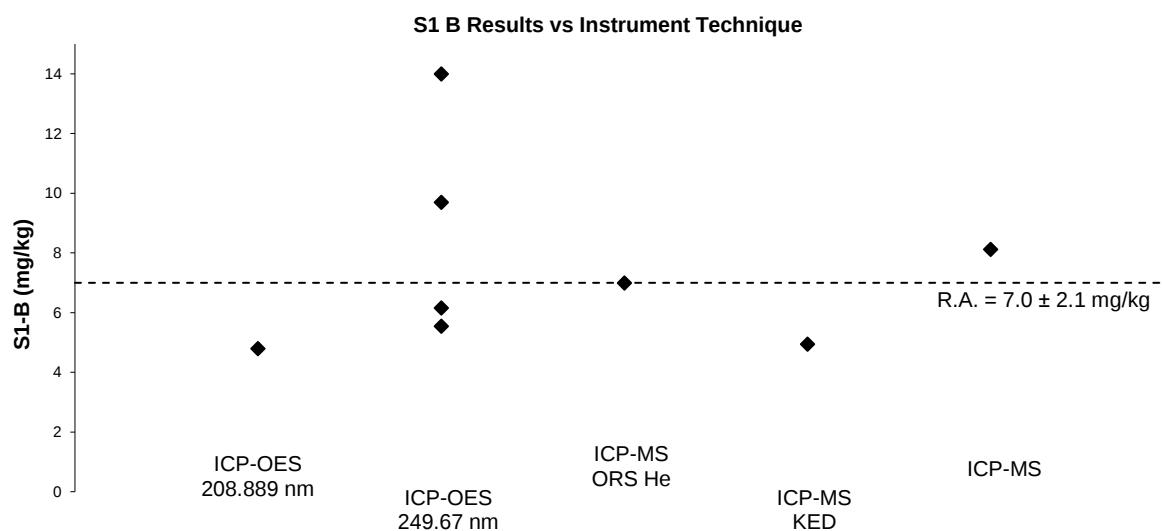


Figure 67 B Results in S1 vs. Instrumental Technique

Bismuth mass fraction in S2 was 1.1 mg/kg and ICP-OES may not be the best choice for Bi measurements at this levels (Figure 68).

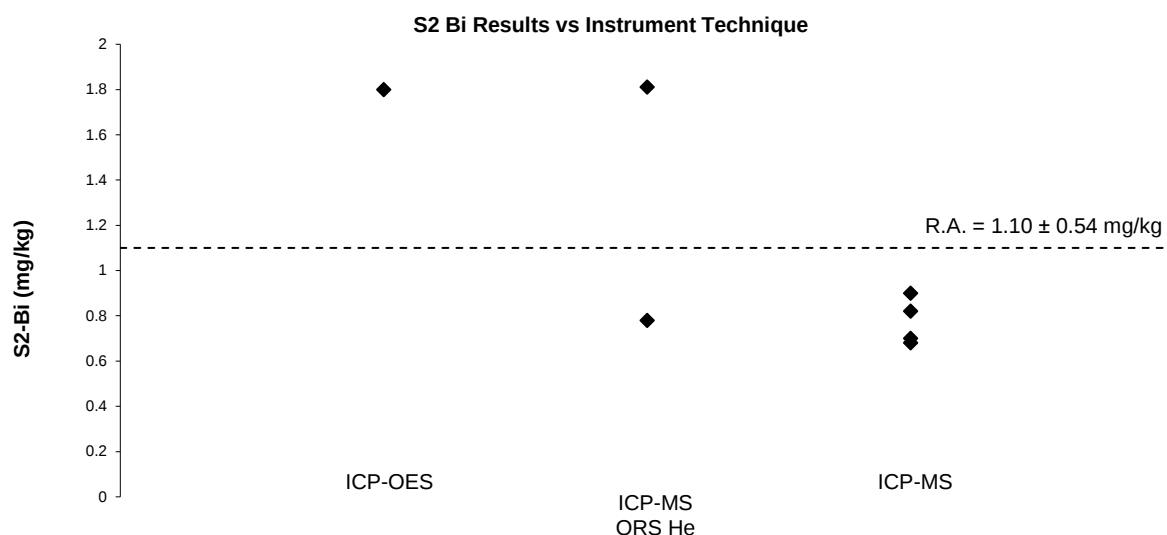


Figure 68 Bi Results in S2 vs. Instrumental Technique

Lithium The between-laboratory CV for this element was larger than predicted by Thomson and Harwitz (22%). ICP-MS has low sensitivity for light elements due to space-charge effects. An internal standard with similar behaviour may overcome this problem.

Mercury With one exception, all results reported for Hg in S1 and S2 returned satisfactory results. ICP-MS in standard mode and CVAAS were the preferred techniques (Figure 69).

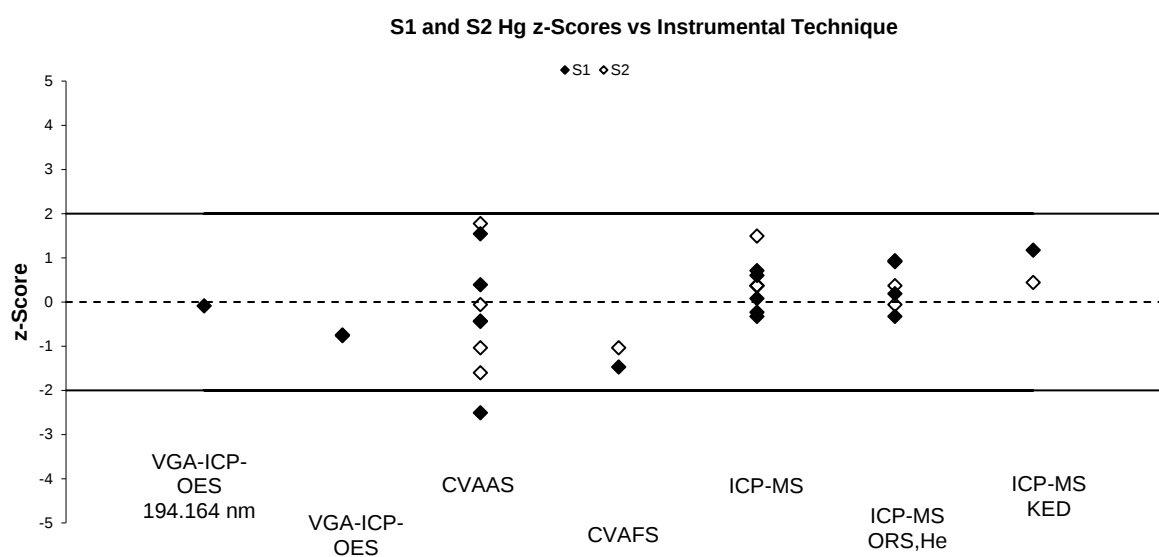


Figure 69 Hg Results in S1 and S2 vs. Instrumental Technique

Selenium level in S2 was low (1.18 mg/kg) and challenged participants' instrumental techniques, returning a between-laboratory CV of 25%. When ICP-MS with collision/reaction cell cannot be used, ICP-MS and isotope ^{82}Se may not be the best choice for Se measurements. Consider using ^{77}Se or ^{78}Se as these isotopes have less interferences.

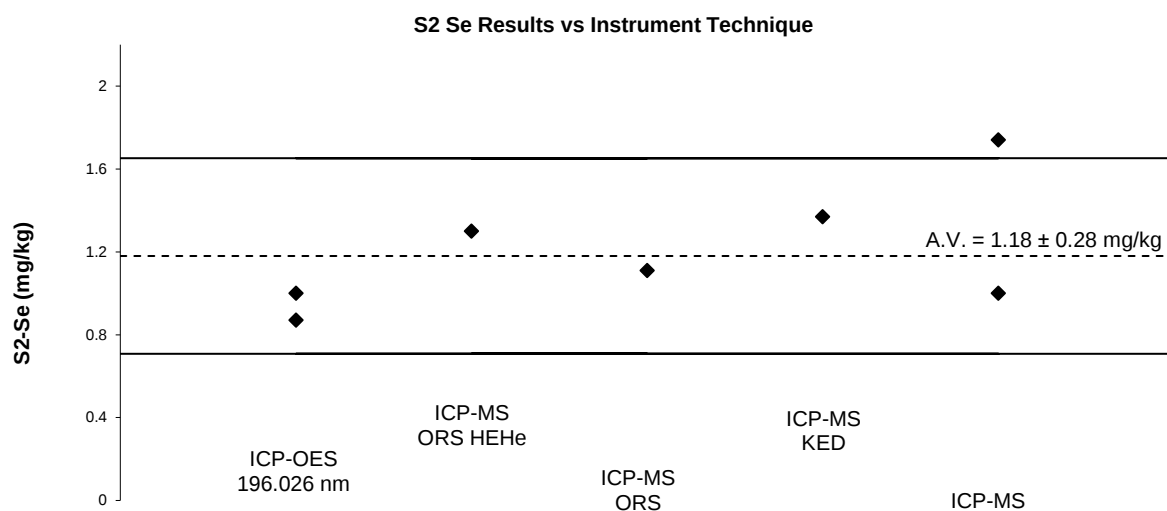


Figure 70 S2-Se Results vs. Instrumental Technique

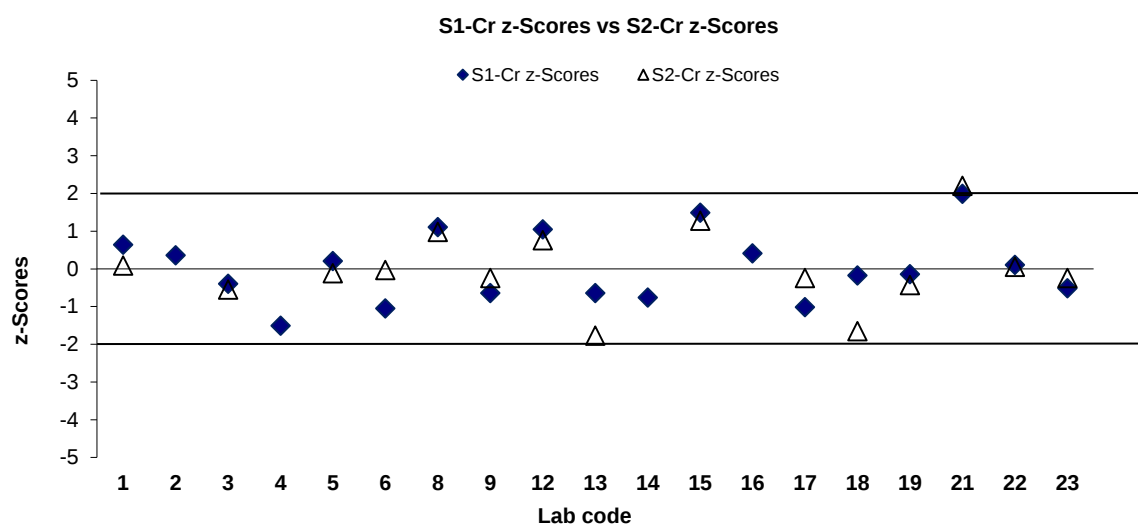
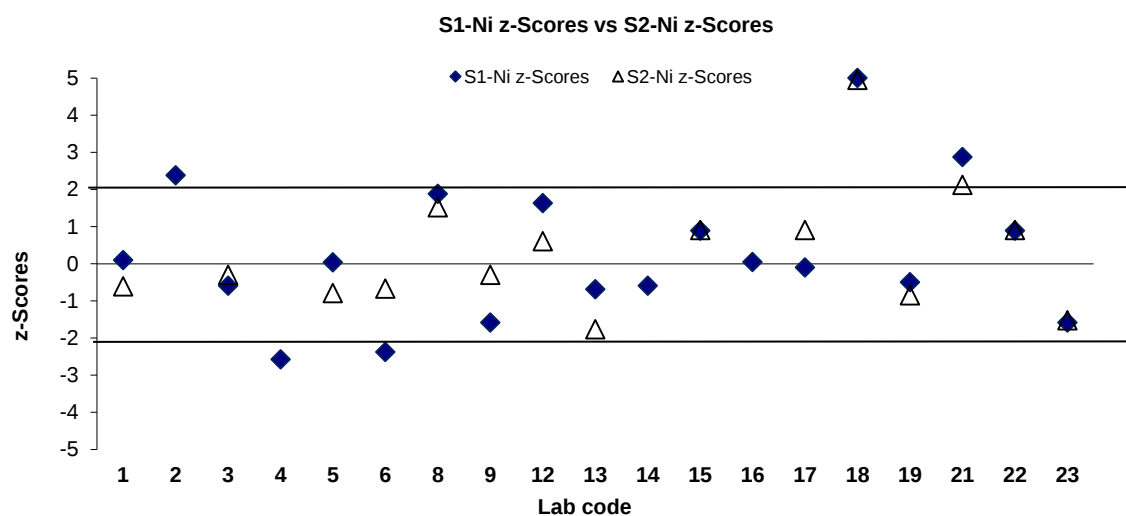


Figure 71 Comparison of Participants' Performance for Cr in S1 and S2



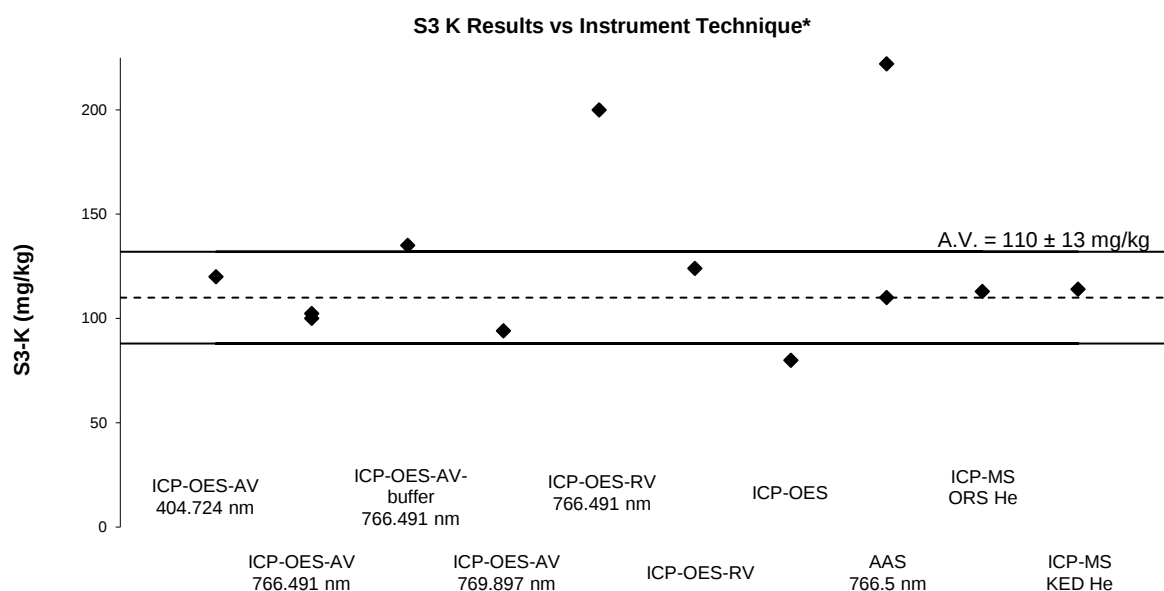
*z-Scores > 5 have been plotted as 5.

Figure 72 Comparison of Participants' Performance for Ni in S1 and S2

Chromium and Nickel are highly dependent on extraction regime. Plots of participants' performance for these elements in S1 and S2 are presented in Figures 71 and 72.

Laboratories whose z-scores for both elements lie on the same side of the centre line may need to monitor their procedure as this can be an indication of method bias.

Potassium, Phosphorus and Sodium Plots of K, P and Na results versus instrumental technique are presented in Figures 73 to 75. ICP-OES was the preferred analytical technique.



*Result > 200 mg/kg has been plotted as 200 mg/kg.

Figure 73 K Results vs. Instrumental Technique

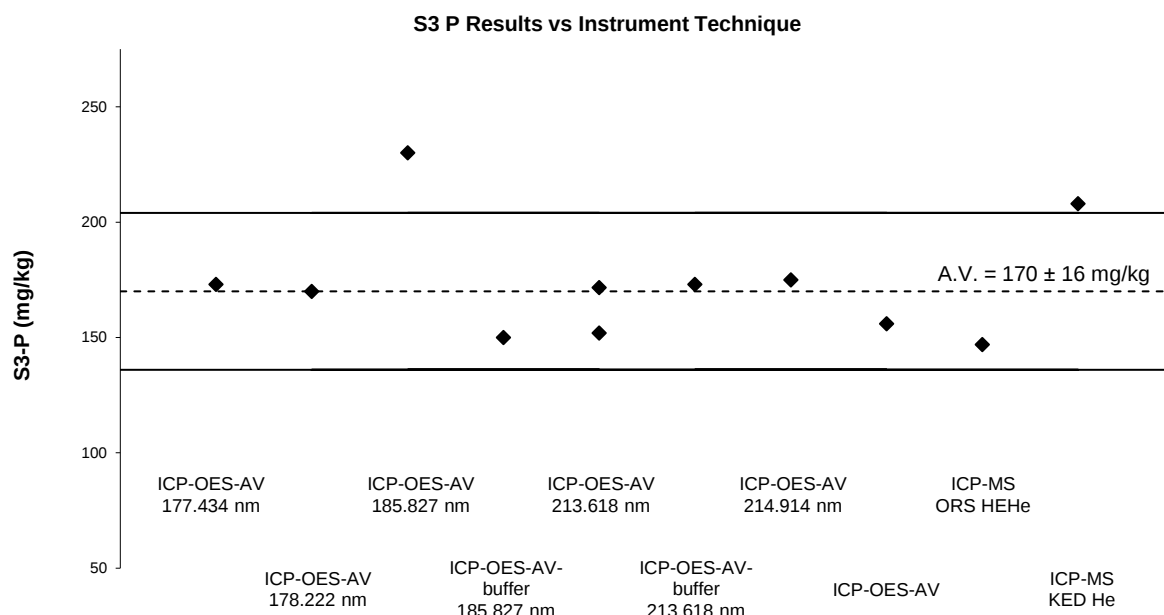
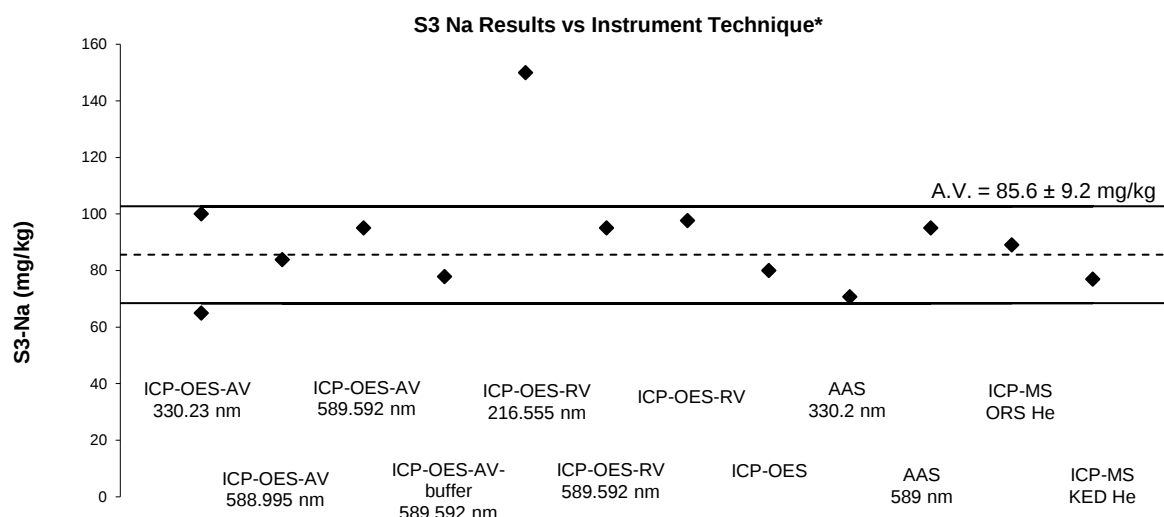


Figure 74 P Results vs. Instrumental Technique



*Result > 150 mg/kg has been plotted as 150 mg/kg.

Figure 75 Na Results vs. Instrumental Technique

Moisture content Measurement of moisture content in the test sample S2 did not present significant difficulty to participating laboratories, the between laboratory CV was low, 5.1%.

Laboratory 21 might have reported result for solid content and not for moisture content.

6.6 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 Method 15A1 as defined by Rayment, G.E. and David, J. L in “Soil Chemical Methods- Australasia”.²⁷

The method descriptions provided by participants are presented in Table 10. With two exceptions, all participants used a ratio sample mass/extraction solution of 1 to 20 and shook the sample for 1-2 min. Laboratory 1 used a ratio of 1:10 for sample mass/extraction solution and Laboratory 14 used a ratio of 1:25. Plots of participants' results versus the analytical methods used for the exchangeable bases measurement are presented in Figures 76 to 78.

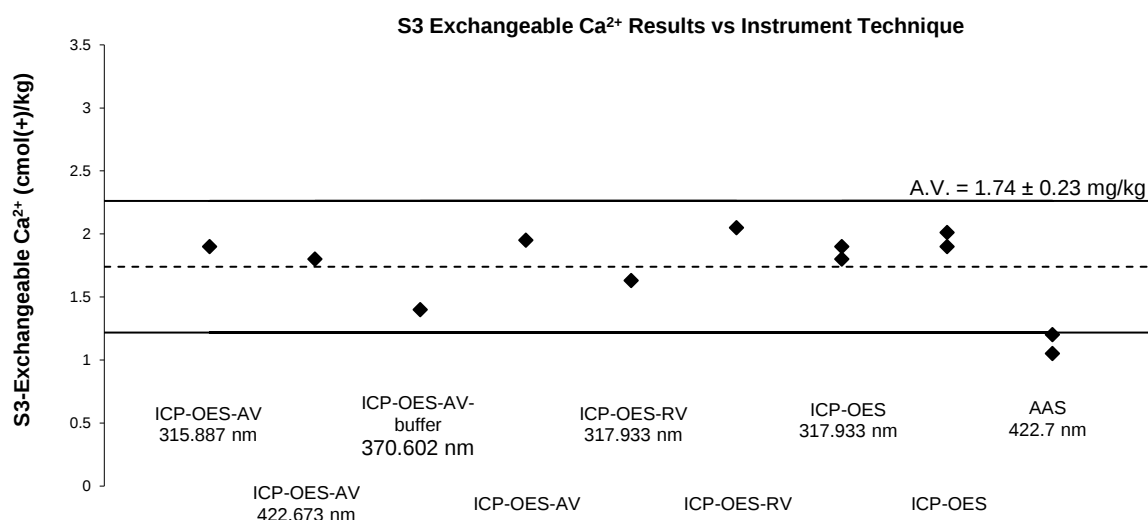


Figure 76 Exchangeable Ca²⁺ Results vs. Analytical Methods

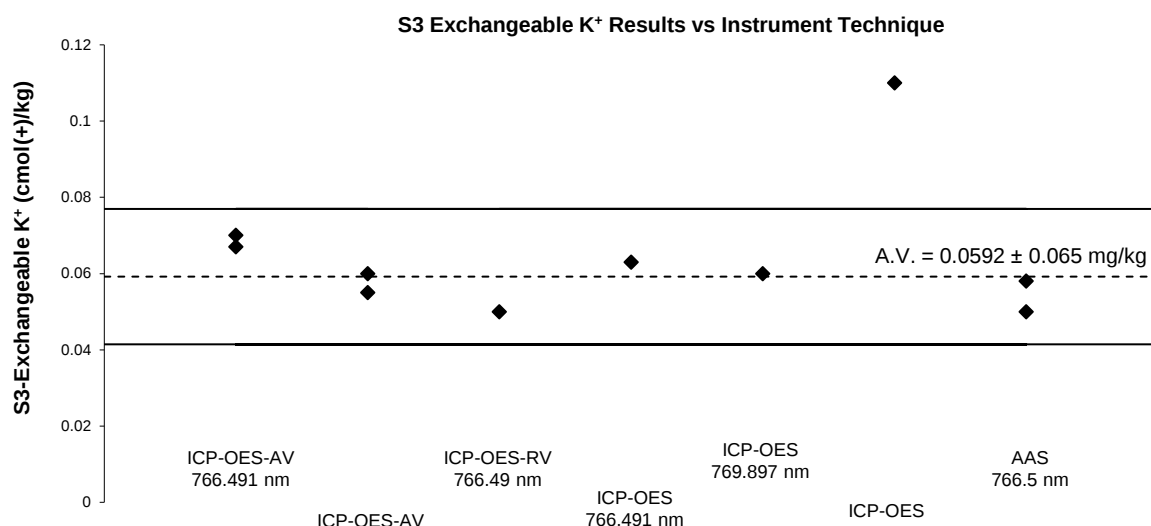


Figure 77 Exchangeable K⁺ Results vs. Analytical Methods

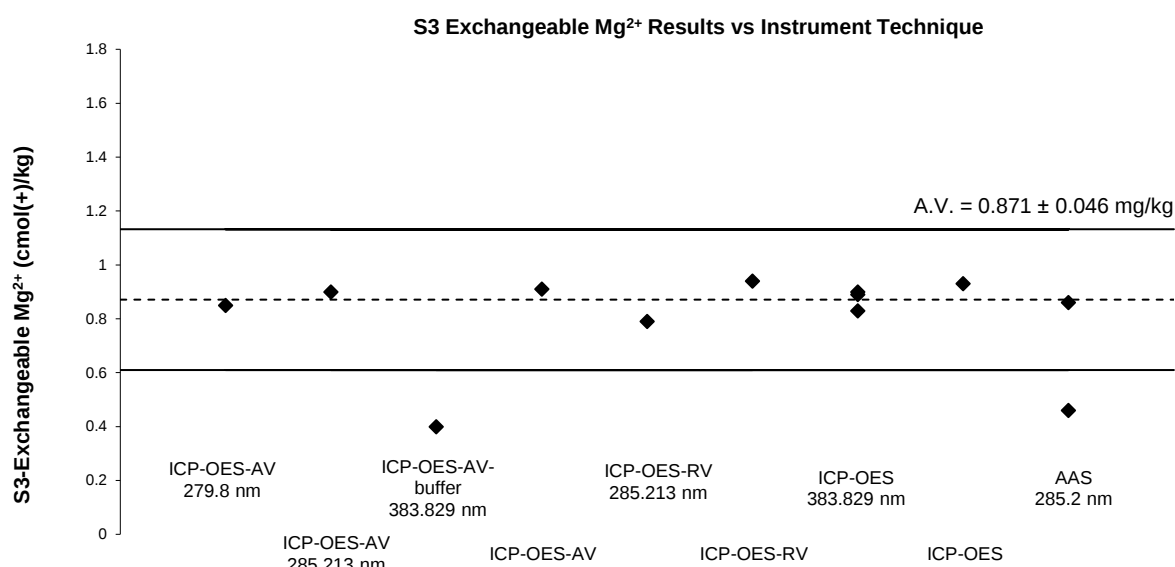


Figure 78 Exchangeable Mg²⁺ Results vs. Analytical Methods

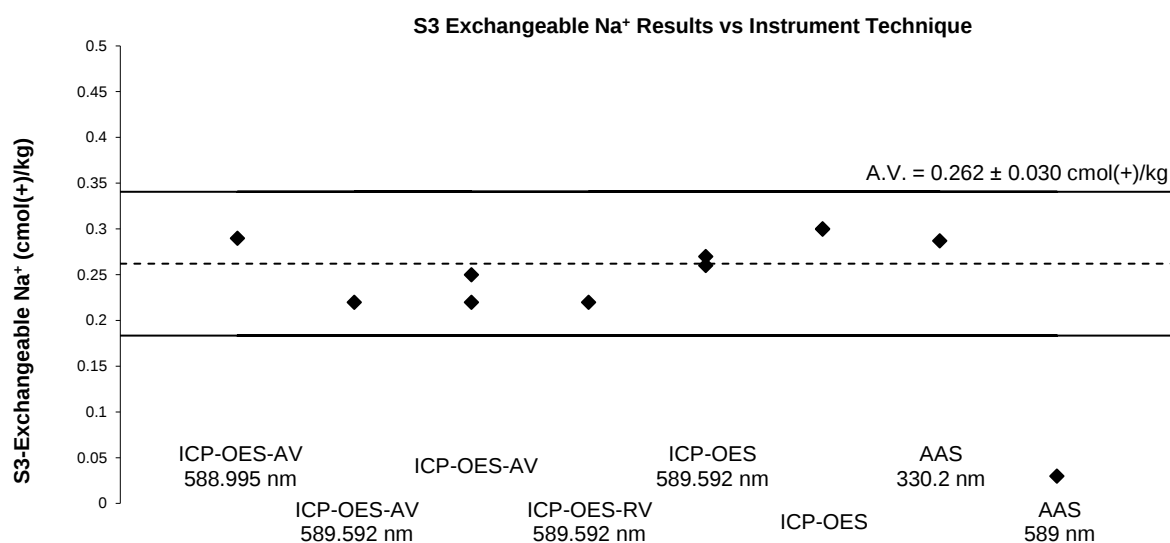


Figure 79 Exchangeable Na⁺ Results vs. Analytical Methods

6.7 Participants' Results and Analytical Methods for Colwell P and Colwell K

The participating laboratories were asked to follow the preparation procedure described in Method 9B1 as defined by Rayment, G.E. and David, J. L in “Soil Chemical Methods- Australasia”.²⁷ All but one participant shook the sample for 16 hours and used a ratio of 1 :100 sample mass/extraction solution (Table 5). Laboratory 20 shook the sample for 17 hours and used a 1:10 ratio.

Colwell K Three participants extracted K in S3 using 0.5 M NaHCO₃ and reported results for this test. One laboratory used ICP-OES to measure Colwell K, one used AAS and one used ICP-MS. The three results were in a relative agreement with each other, centred on 33.8 mg/kg (Figure 80).

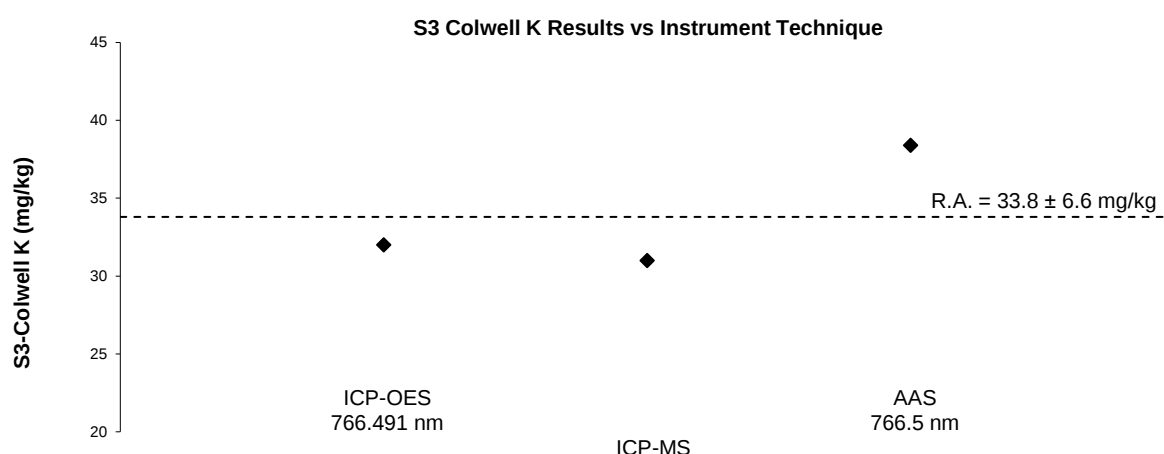


Figure 80 Colwell K Results vs. Instrumental Technique

Colwell P Eight results were reported for Colwell P in S3 and seven were compatible with each other and with the assigned value of 61.5 mg/kg. Plots of participants' results versus the instrumental technique used are presented in Figure 81.

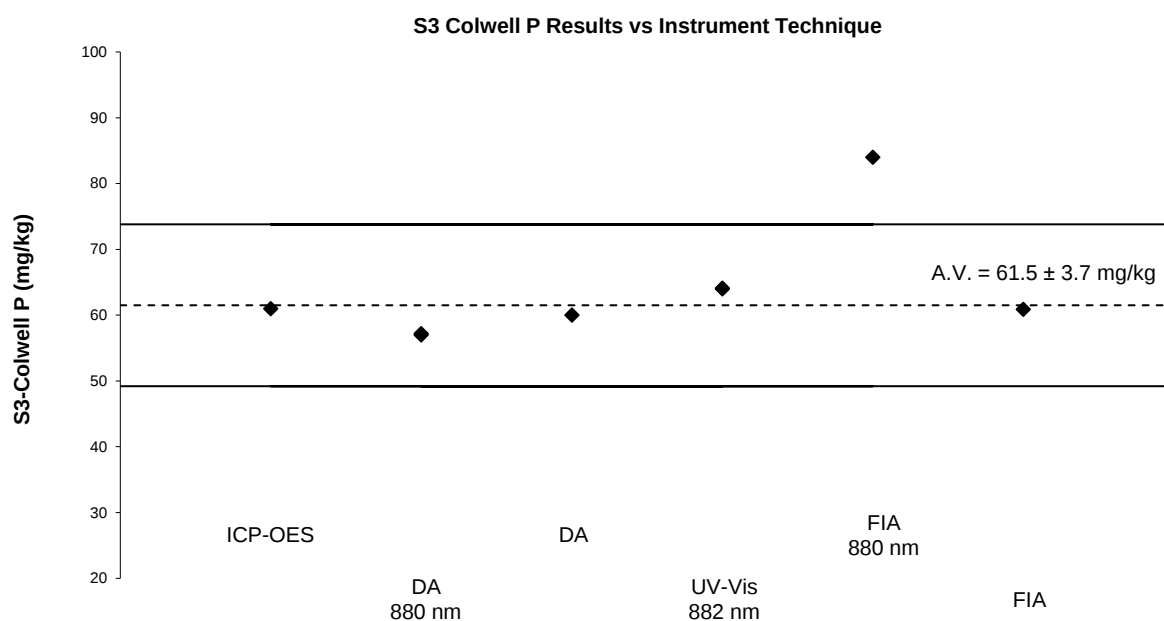


Figure 81 Colwell P Results vs. Instrumental Technique

6.8 Participants' Results and Analytical Methods for Phosphorus Buffer Index-PBI+ColP

P Buffer Index-PBI+ColP gives an indication of soil ability to fix P and make it unavailable to plant uptake. Five laboratories reported results for this test. The results were in relatively good agreement with each other (CV11%), centred on the value of 43.5 mg/kg (Figure 82).

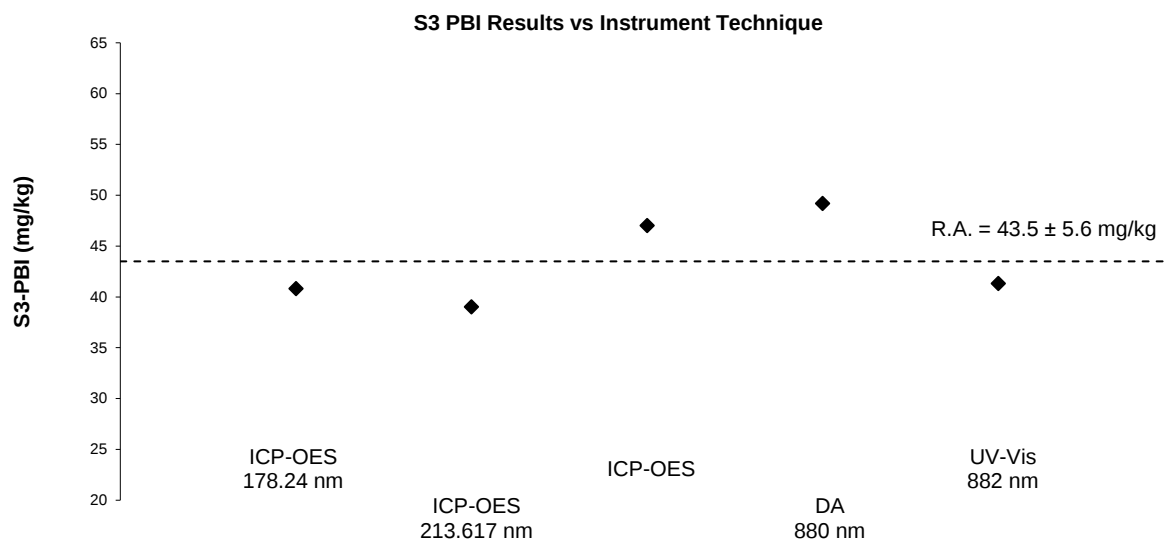


Figure 82 PBI Results vs. Instrumental Technique

6.9 Participants' Results and Analytical Methods for Total P

Total P assigned value was 186 mg/kg. Eight participants reported results for total P and all performed satisfactorily (Figure 83).

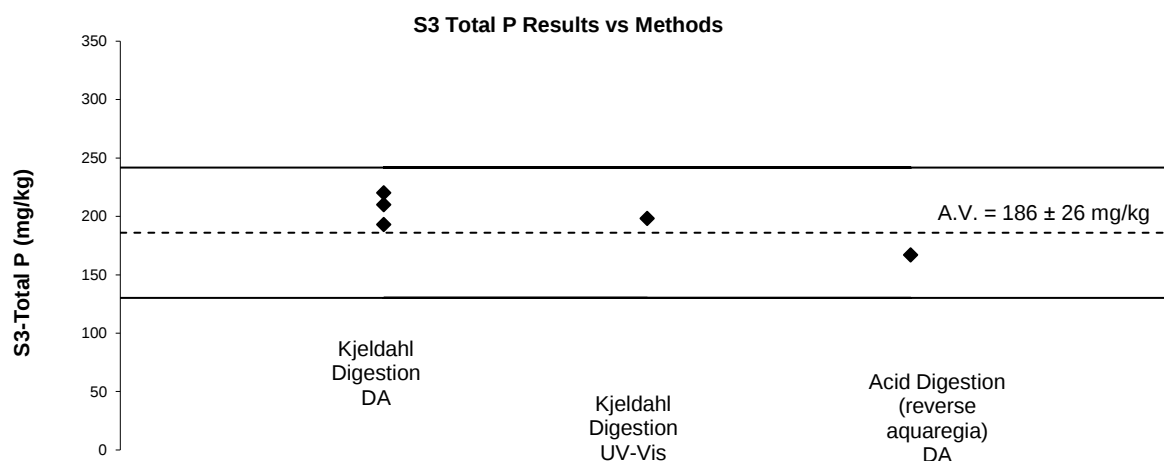


Figure 83 TP Results vs. Analytical Method

Andrew, is this possible? See above in red

6.10 Participants' Results and Analytical Methods for Total Nitrogen

No significant difference was found between TN results from combustion and those results calculated from TKN and NO_x. The method descriptions provided by participants are presented in Table 9. A plot of participants' results versus analytical method and measurement technique used for TN analysis in S3 is presented in Figure 84.

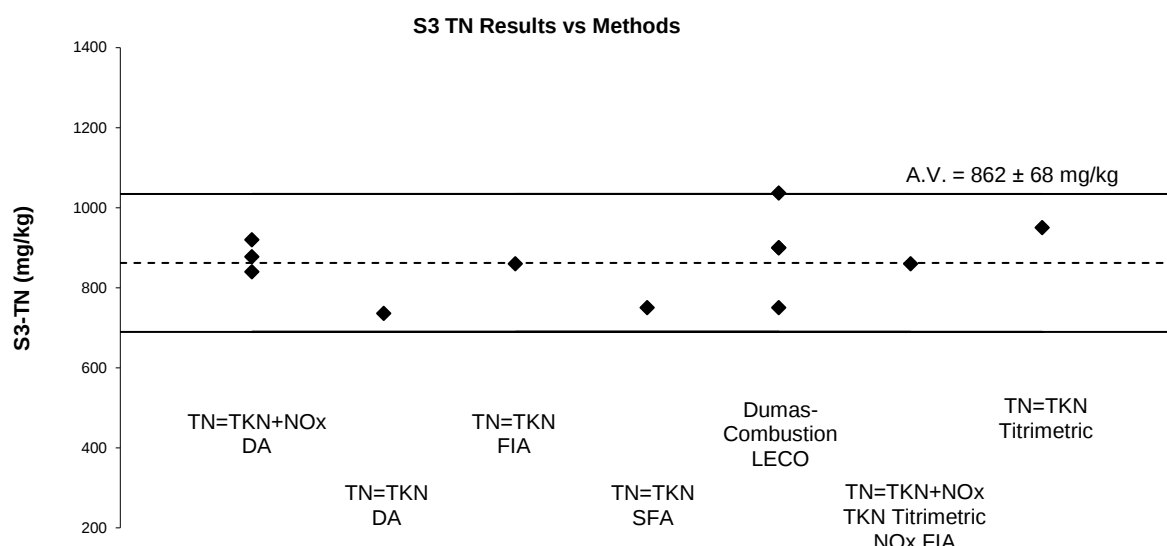


Figure 84 TN Results vs. Analytical Method

6.11 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 assigned value was 99800 mg/kg. All reported results returned satisfactory z-scores.

Total Organic Carbon assigned value was 30100 mg/kg.

Total organic carbon (TOC) measurements should involve the 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 a soil sample, all the NPOC reported results in sample S3 have been considered as TOC.^{21 to 24}

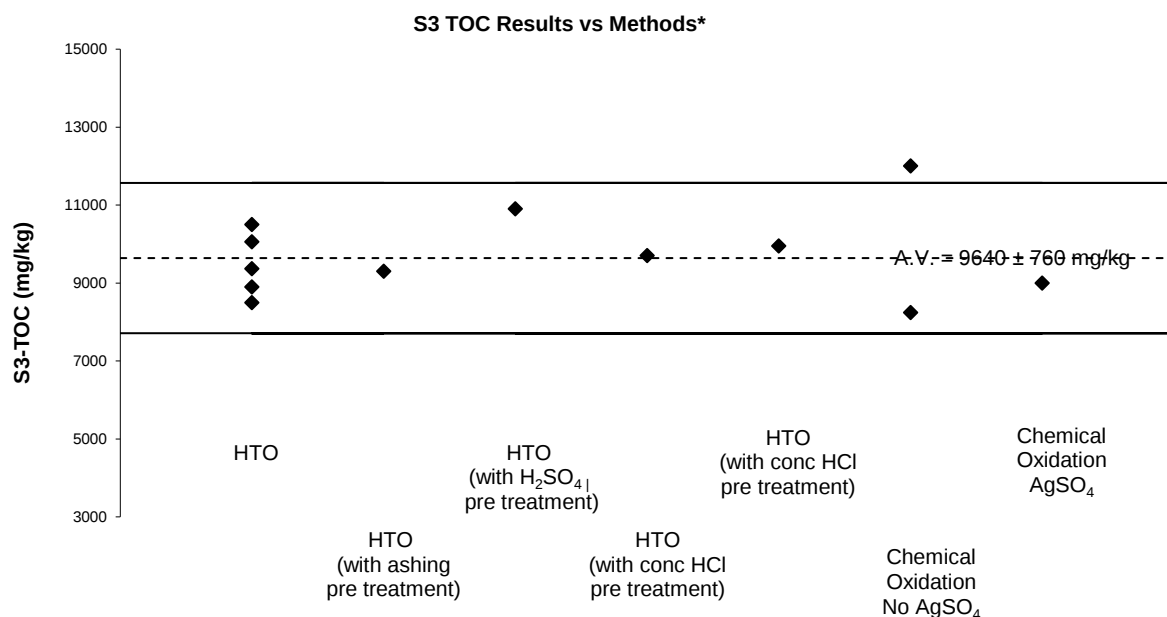
Nine participants used a high temperature oxidation method and three used a chemical oxidation method based on the "Walkley-Black" method.

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. One participant used ashing for TOC removal and 3 used acid pre-treatment (concentrated HCl, 4M HCl or sulphuric acid).

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 the sample to be taken for analysis and the right type and concentration of acid to be used can help avoid these problems.^{24, 25}

Comparison studies on the efficiency of TOC methods found that the most appropriate method for soil TOC analyses is the automated dry combustion technique after pre-testing and pre-treatment for IC removal.^{24, 25, 26}

Laboratory 13 reported their result for TOC in the wrong units. Their result was removed from statistics calculation as extreme outlier.



*HTO- High Temperature Oxidation.

Figure 85 TOC Result vs. Analytical Method

6.12 Comparison with Previous NMI Proficiency Tests of Metals in Soil

AQA 21-01 is the twenty-eight NMI proficiency test of metals in soil.

Participants' performance in measurement of metals in soil over the last ten years is presented in Figure 87. Despite different matrices, analytes and analyte concentrations, on average participants' performance remained consistent.

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

6.13 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 76).

Table 76 Control Samples Used by Participants

Lab. Code	Description of Control Samples
2	PACS3
3	AGAL-10 and AGAL-12
4	RM – Previous AQA PT scheme samples
5	CRM
6	CRM
7	CRM
8	AGAL 10 & AGAL 12

10	RM
11	Spiked Sample
12	RM – AGAL 12
13	CRM
14	CRM – AGAL-10/In-House SRM's
15	RM
16	AQA, Inhouse
17	RM – agal 10, agal 12
18	RM - NMI
19	CRM – Agal-10 Hawkesbury River Sediment
20	CRM - ASPAC 6052 and ASPAC 7098-C1
21	CRM – NMI AGAL-12
22	Spiked Sample
23	CRM - NMI PT Studies

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 procedures²⁷*

Surplus test samples from this study are available from NMI.

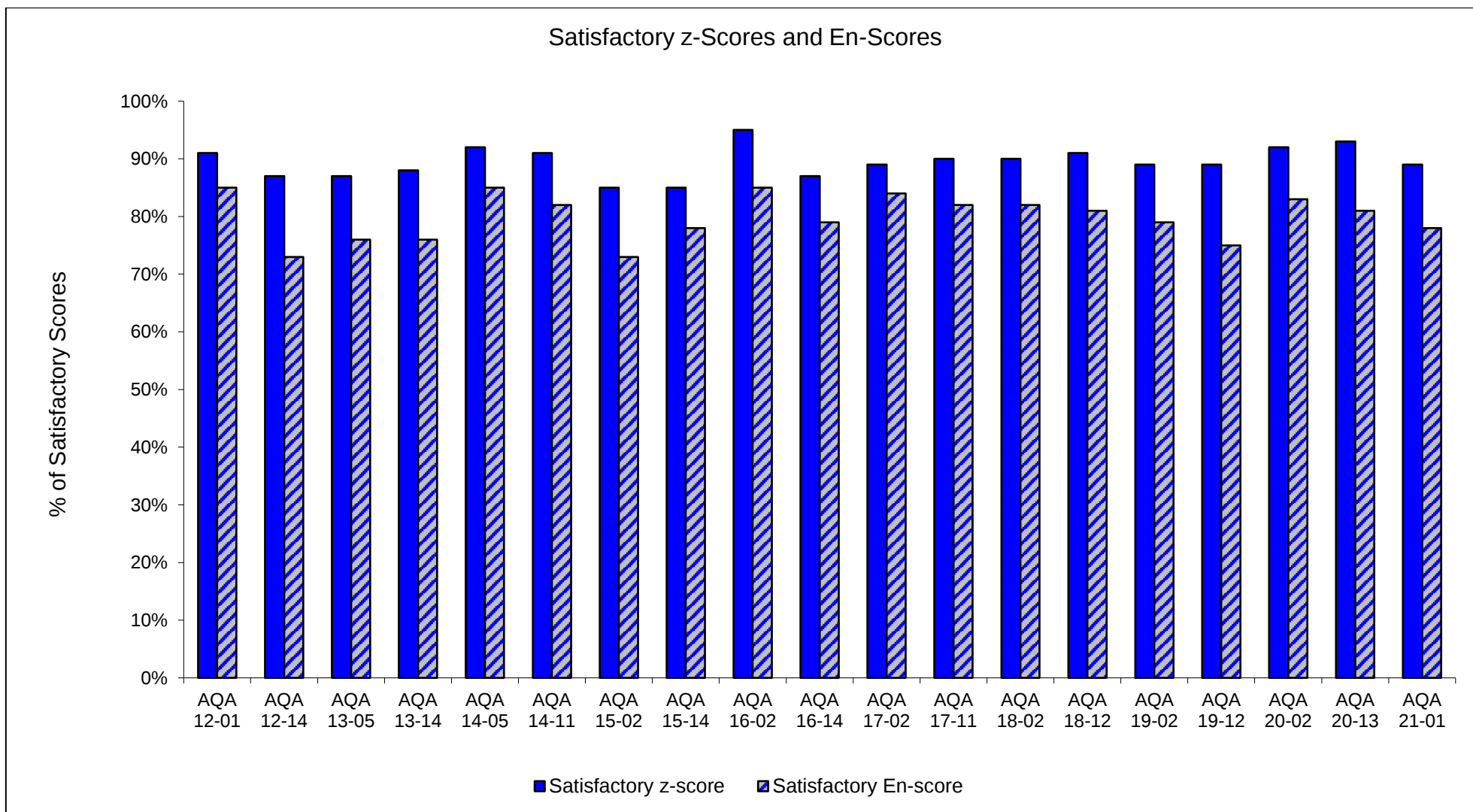


Figure 86 Participants' Performance over Time (2012-2021)

7 REFERENCES

- [1] ISO/IEC 17043:2010, *Conformity assessment – General requirements for proficiency testing*.
- [2] NMI 2016, *NMI Chemical Proficiency Testing Study Protocol*, viewed 20 February 2020, <<http://www.industry.gov.au>>.
- [3] NMI 2016, *NMI Chemical Proficiency Testing Statistical Manual*, viewed 20 February 2020, <<http://www.industry.gov.au>>.
- [4] Thompson, M, Ellison, S & Wood, R 2006, 'The international harmonized protocol for proficiency testing of (chemical) analytical laboratories', *Pure Appl. Chem*, vol 78, pp 145-196.
- [5] National Environmental Protection Council, *NEPM Schedule B (1) Guidelines on the Investigation Levels for Soil and Groundwater*, viewed 20 February 2020, <https://www.legislation.gov.au/Details/F2013C00288/Html/Volume_2>.
- [6] ISO 13528:2015(E), *Statistical methods for use in proficiency testing by interlaboratory comparisons*.
- [7] Thompson, M, Ellison 2000, 'Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing', *Analyst*, vol 125, pp 385-386.
- [8] ISO/IEC 17025:2018, *General requirements for the competence of testing and calibration laboratories*.
- [9] Eurachem/CITAC Guide, *Quantifying uncertainty in analytical measurement* 3rd edition, viewed 20 February 2020, <<http://www.eurachem.org>>.
- [10] Bertil, M, Naykki, T, Hovind, H & Krysell, M 2012, *Nordtest Report Handbook for Calculation of Measurement Uncertainty in Environmental Laboratories* TR 537 3rd Edition Nordest Tekniikantie, Finland, Esopo,.
- [11] Hibbert, B 2007, *Quality Assurance for the Analytical Chemistry Laboratory*, Oxford University Press.
- [12] NATA 2018, *General Accreditation, Guidance, Estimating and Reporting Measurement Uncertainty of Chemical Test Results*
- [13] ISO (2008), *Guide to the Expression of Uncertainty in Measurement (GUM)*, Geneva, Switzerland.
- [14] Eurolab 2002, Technical Report No 1/2002 - *Measurement Uncertainty in Testing*.
- [15] NMI, *Estimating Measurement Uncertainty for Chemists* – viewed 4 February 2020, <www.industry.gov.au/client-services/training-and-assessment>.
- [16] ASTM E2554-13, *Standard Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques*.
- [17] Roje, V.,(2010) *Multi-elemental analysis of marine sediment reference material MESS-3: one-step microwave digestion and determination by high resolution inductively coupled plasma-mass spectrometry (HR-ICP-MS)*, *Chemical papers* 64 (4) 409-414
- [18] Cotton, F. A., Wilkinson, G., (1998) *Advanced Inorganic Chemistry*, (4th ed, p394-401). NY, USA

- [19] Wiley Lee, J.D., (1996) *Concise Inorganic Chemistry* (p 510), London, UK Chapman & Hill
- [20] Bailar, J.C., et. al (1973) *Comprehensive Inorganic Chemistry*, (1st ed. p558-680) Pergamon Press Ltd., Headington Hill Hall, Oxford
- [21] Rayment, G & Lyons D 2011, *Soil Chemical Methods – Australasia*, CSIRO Publishing, Collingwood VIC Australia
- [22] Bisutti, I. & Hilke, I. 2004, 'Determination of total organic carbon – an overview of current methods', *Trends in Analytical Chemistry*, 23, 716-726.
- [23] Schumacher, B.A. 2002, United States Environmental Protection Agency Environmental Sciences Division National Exposure Research Laboratory, *Methods for the determination of total organic carbon (TOC) in soils and sediments*, NCEA-C-1282, EMASC-001.
- [24] USEPA 2002, NCEA-C-1282, EMASC-001-*Methods for the determination of total organic carbon (TOC) in soils and sediments*.
- [25] Soon, K & Abboud, S 1991, 'A comparison of some methods for soil organic carbon determination', *Communication in Soil Science and Plant Analysis*, vol 22, pp 943-954.
- [26] Leong, S & Taner, A 1999, 'Comparison of Methods for Determination of Organic Carbon in Marine Sediment', *Marine Pollution Bulletin*, vol 38, pp 875-876.
- [27] JCGM 200:2008, *International vocabulary of metrology – Basic and general concepts and associated terms (VIM)*, 3rd edition.

APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING

Sample Preparation

Sample S1 was a sediment material fortified for 8 elements, dried, ground and passed through a 212 µm sieve prior to being divided into portions of approximately 25 g each.

Sample S2 was a wet sludge sample prepared from a soil material previous used in a PT study. The soil material was accurately weighed and wet with a known amount of water. The moist soil was mixed and divided into portions of 100 g each.

Sample S3 was an unfortified, dried, agricultural soil material. It was ground and sieved through a 350 µm sieve, further mixed and divided into portions of approximately 75 g each.

Sample Analysis and Homogeneity Testing

The same procedure was followed for the preparation of Samples S1, S2 and S3 as in previous NMI PT studies. Therefore only a partial homogeneity test was conducted for elements of interest. Three bottles were analysed in duplicate and the average of the results was reported as the homogeneity value. Measurements were made under repeatability conditions in random order.

Sample Analysis for Acid Extractable Elements

Measurements for acid extractable elements involved solubilisation of metals and metal complexes using a mixture of nitric acid and hydrochloric acid. Metals were then measured using ICP-MS.

Test portions of approximately 0.5 g for the dried sediment sample and 1.5 g for the moist soil sample were 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 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 77.

Table 77 Instrumental Technique used for Acid Extractable Elements

Analyte	Instrument	Internal Standard	Reaction/ Collision Cell (if applicable)	Cell Mode/Gas (if applicable)	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Ion (m/z)/ Wavelength (nm)
Ag	ICP-MS	Rh	NA	He	NA	800	107 m/z
Al	ICP-MS	Rh	NA	NA	NA	800	27 m/z
As	ICP-MS	Rh	ORS	He	800	800	75 m/z
B	ICP-MS	Rh	NA	NA	800	NA	11 m/z
Ba	ICP-MS	Rh	ORS	He	800	800	137 m/z
Be	ICP-MS	Rh	NA	NA	800	NA	9 m/z
Bi	ICP-MS	Ir	NA	He	NA	800	209 m/z
Ca	ICP-MS	Rh	ORS	He	800	NA	43 m/z
Cd	ICP-MS	Rh	NA	He	800	800	111 m/z
Co	ICP-MS	Rh	ORS	He	NA	800	59 m/z
Cr	ICP-MS	Rh	ORS	He	800	800	52 m/z
Cu	ICP-MS	Rh	ORS	He	800	800	63 m/z
Fe	ICP-MS	Rh	NA	He	800	NA	56 m/z

Hg	ICP-MS	Rh	NA	He	800	800	201 m/z
K	ICP-MS	Rh	ORS	He	800	NA	39 m/z
La	ICP-MS	Rh	ORS	He	NA	800	139 m/z
Li	ICP-MS	Rh	ORS	He	800	NA	7 m/z
Mn	ICP-MS	Rh	ORS	He	800	NA	55 m/z
Mo	ICP-MS	Rh	ORS	He	NA	800	95 m/z
Na	ICP-MS	Rh	ORS	He	800	NA	23 m/z
Ni	ICP-MS	Rh	ORS	He	800	800	60 m/z
P	ICP-MS	Rh	ORS	HEHe	800	NA	31 m/z
Pb	ICP-MS	Ir	NA	He	800	800	Average of 206, 207 m/z
Rb	ICP-MS	Rh	ORS	He	800	NA	85 m/z
Sb	ICP-MS	Rh	ORS	He	800	NA	121 m/z
Se	ICP-MS	Rh	ORS	HEHe	800	800	78 m/z
Sn	ICP-MS	Rh	NA	He	800	NA	118 m/z
Th	ICP-MS	Rh	ORS	He	800	NA	232 m/z
Tl	ICP-MS	Rh	ORS	He	NA	800	205 m/z
U	ICP-MS	Ir	NA	He	NA	800	238 m/z
V	ICP-MS	Rh	ORS	He	800	NA	51 m/z
Zn	ICP-MS	Rh	ORS	He	800	800	64 m/z

Sample Analysis for Total Nitrogen

Total Nitrogen in Sample S3 was measured as the sum of TKN +NO_x.

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.

For NO_x measurements a test portion of 10 g was weighed into a 100 mL polypropylene container. The container was then filled with 95 mL Milli-Q water. The suspension was shaken, at room temperature for 1 h, centrifuged, and filtered through 0.45 µm filter. NO₃⁻-N was further measured by cadmium reduction to NO₂⁻-N followed by NO_x (the reduced NO₂⁻-N plus original NO₂⁻-N) measurements by FIA.

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:

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

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 78.

Table 78 Uncertainty of Assigned Value for Fe in Sample S3

No. results (p)	12
Robust Average	4070 mg/kg
$S_{rob\ av}$	415 mg/kg
$u_{rob\ av}$	150 mg/kg
k	2
$U_{rob\ av}$	300 mg/kg

The assigned value for **Fe** in Sample S3 is **4070 ± 300 mg/kg**

z-Score and E_n-score

For each participant's result z-score and E_n-score are calculated according to Equation 2 and Equation 3 respectively (see page 15).

A worked example is set out below in Table 79.

Table 79 z-Score and E_n-score for Fe result reported by Laboratory 1 in S3

Fe Result mg/kg	Assigned Value mg/kg	Set Target Standard Deviation	z-Score	E _n -Score
4075±600	4070±300	10% as PCV or 0.10x4070= =407 mg/kg	$z = \frac{(4075 - 4070)}{407}$ $z = 0.012$	$En = \frac{(4075 - 4070)}{\sqrt{600^2 + 300^2}}$ $En = 0.007$

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.^{10, 12} An example is given. Between 2009 and 2021 NMI carried out 23 proficiency tests of metals in soil. These studies involved analyses of acid-extractable elements at low and high levels in dried soil, moist soil, biosoil, sediment and sludge. **Laboratory X** submitted results for As in all of these PTs. All reported results returned satisfactory z-scores. This data can usefully be separated into two ranges of results 0.5 to 10 mg/kg and 10 to 100 mg/kg.

Table 80 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	S1 - Biosoil	4.091	3.64	16	11
	S2 - Soil	4.29	4.57	15	12
AQA 11-01	S1 - Biosoil	3.54	3.57	19.7	18
AQA 13-05	S1 - Soil	9.22	9.21	14	22
AQA 14-11	S1 - Sediment	7.91	7.37	11.8	21
AQA 15-02	S1 - Moist Sludge	8.29	7.02	13	22
	S2 - Moist Sludge	7.42	7.02	11.3	17
AQA 15-14	S1 - Sediment	10	9.95	6.7	17
	S2 - Soil	4.53	4.47	6.4	14
AQA 16-02	S2 - Clay	2.67	2.11	14	20
AQA 16-14	S1 - Soil	6.03	5.61	20	17
AQA 17-02	S2 - Soil	3.71	3.76	10	13
AQA 18-02	S1 - Compost	2.22	2.73	11	17
AQA 19-02	S1 - Soil	2.83	2.65	11	24
AQA 19-12	S1 - Soil	2.32	2.12	16	16
AQA 20-13	S1 - Biosoil	2.85	3.29	11	17
AQA 21-01	S1 - Sediment	7.02	6.26	6.9	18
AQA 21-01	S2 - Moist Sludge	3.99	3.58	12.6	13
Average				12.1**	

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

Table 81 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	S1 - Soil	16.6	14.4	8.5	19
AQA 11-12	S1 - Moist Sludge	25	21.6	15	13
AQA 12-01	S1 - Sediment	18.4	17.3	8.1	21
AQA 12-14	S2 - Soil	16.6	14.8	11	20
AQA 13-14	S1 - Sandy Soil	16.6	15.1	10.4	21
AQA 14-05	S1 - Soil	13.2	12.3	7.8	25
AQA 17-11	S1 - Sediment	18.1	17.4	11	22
AQA 18-12	S2 - Soil	10.4	9.6	8	20
AQA 19-12	S2 - Sediment	21	19.9	9	19
AQA 20-02	S1 - Soil	18.8	21.6	8.8	23
AQA 20-02	S2 - Moist Soil	16.5	17.8	6.7	24
Average				9.5**	

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

Taking the average of the robust CV over these PT samples for each concentration range gives estimates of the relative standard uncertainty of 12% and 10% respectively. Using a coverage factor of two gives relative expanded uncertainties of 25% and 20% respectively, at a level of confidence of approximately 95%.

Table 82 sets out the expanded uncertainty for results of the measurement of As in soil, biosoil, clay, sediment, sludge, sandy soil, moist soil, compost and agricultural soil over the ranges 0.5 to 10 mg/kg and 10 to 100 mg/kg.

Table 82 Uncertainty of As Results Estimated Using PT Data.

Results mg/kg	Uncertainty mg/kg
1.00	0.24
5.0	1.2
20	4
75	15

The estimates of 25% and 20% relative passes the test of being reasonable, and the analysis of the 29 different PT samples over twelve years can be assumed to include all the relevant uncertainty components (different matrices, operators, reagents, calibrators etc.), and so complies with ISO 17025:2018.

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 Fluorescence Spectrometry
DA	Discreet Analyser
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-equation	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
Max	Maximum value in a set of results
Md	Median
Min	Minimum value in a set of results
NMI	National Measurement Institute (of Australia)
NR	Not Reported
NT	Not Tested
ORS	Octopole Reaction System
PCV	Performance Coefficient of Variation
RM	Reference Material
Robust CV	Robust Coefficient of Variation
Robust SD	Robust Standard Deviation
S.V.	Spiked value 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 83 Instrument Conditions Ag

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Ge 72	ORS		N/A		107 m/z
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-MS	Rh 103	ORS	He	500	500	107
4	NA	NA	NA	NA	NA	NA	NA
5					NA		
6	ICP-OES	Ir, Rh & Sc	NA	NA	N/A	50	328.069nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Rh	ORS	He	NA	500	107
9	ICP-OES-AV	Lu			NA	83	328.068
10	ICP-MS	Rh	NA		NA	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Rh	NA	NA	NA	625	109
13	ICP-MS/MS	Sc, Rh, Ir		He	NA		
14	NA	NA	NA	NA	NA	NA	NA
15					NA		
16	ICP-MS	103	ORS	He	100	NA	107
17	ICP-MS				NA		
18	ICP-MS	Y 89	KED		NA	10	107
19	ICP-MS	Rh	NA	standard mode	NA	2000	109
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				NA	100	328.068
22					NA		
23					NA		

Table 84 Instrument Conditions A1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Y377	NA		N/A		396.152 nm
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-MS	Sc 45	ORS	He	500	500	27
4	NA	NA	NA	NA	NA	NA	NA
5	ICP-OES-AV	Lu 261.541			NA	200	396.152
6	ICP-OES	Eu & Cs	NA	NA	N/A	50	236.707, 308.215, 396.15nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Sc	ORS	He	NA	2500	27
9	ICP-OES-AV	Lu			NA	166	396.152
10	ICP-MS	Sc	NA		NA	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	NA	NA	NA	625	27
13	ICP-OES-AV	Eu, Cs			NA		206.834
14	NA	NA	NA	NA	NA	NA	NA
15					NA		
16	ICP-MS	45	ORS	He	100	NA	27
17	ICP-MS				NA		
18	ICP-MS	Sc-2 45	KED		NA	1000	27
19	ICP-MS	Sc	NA	standard mode	NA	2000	27
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				NA	100	308.215
22					NA		
23					NA		

Table 85 Instrument Conditions As

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Ge 72	ORS				75m/z
2						NA	
3	ICP-MS	Rh 103	ORS	He	500	500	75
4	ICP-OES-AV	Y	NA	NA	18	NA	188.979
5	ICP-OES-AV	Lu 261.541			200	200	188.98
6	ICP-OES	Ir, Rh & Sc	NA	NA	50	50	188.89nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Rh	ORS	He	500	500	75
9	ICP-OES-AV	Lu			83	83	188.98
10	ICP-MS	Rh	UC	He	250	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Rh	KED	He	625	625	75
13	ICP-MS						
14	AAS	None	NA	NA	20	NA	193.7
15					250		
16	ICP-MS	72	ORS	He	100	NA	75
17	ICP-MS						
18	ICP-MS	Ge-1 72	KED		10	10	75
19	ICP-MS	Rh	KED	He	1000	1000	75
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				100	100	188.98
22							
23							

Table 86 Instrument Conditions B

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Te214	NA			N/A	249.678nm
2						NA	
3	ICP-MS	Sc 45	ORS	He	500	500	11
4						NA	
5						NA	
6	ICP-OES	Eu & Cs	NA	NA	50	N/A	249.773nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Sc	ORS	He	500	NA	11
9	ICP-OES-AV	Lu			83	NA	182.577
10	ICP-MS	Sc	NA		250	NA	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	NA	NA	625	NA	10
13	ICP-OES-AV					NA	249.773
14	ICP-OES-AV	None	NA	NA	20	NA	208.889
15					250	NA	
16	ICP-OES-AV-buffer	Lu			100	NA	249.678
17	ICP-MS					NA	
18	ICP-MS	Y 89	KED		10	NA	11
19	ICP-MS	Sc	KED	He	1000	NA	11
20	NA	NA	NA	NA	NA	NA	NA
21						NA	
22						NA	
23						NA	

Table 87 Instrument Conditions Ba

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-RV	Y371	NA		N/A		493.408 nm
2						NA	
3	ICP-MS	Rh 103	ORS	He	500	500	135
4						NA	
5	ICP-OES-AV	Lu 261.541			200	200	413.064
6	ICP-OES	Ir, Rh & Sc	NA	NA	N/A	50	585.369nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Sc	ORS	He	500		135
9	ICP-OES-AV	Lu			83	83	493.408
10	ICP-MS	In	NA		250	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Rh	NA	NA	625	625	138
13	ICP-MS						
14	AAS	None	NA	NA	20	NA	553.6
15					250		
16	ICP-MS	103	ORS	He	100	NA	138
17	ICP-MS						
18	ICP-MS	In-1 115	KED				138
19	ICP-MS	Tb	NA	standard mode	2000	2000	137
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				100	100	455.403
22							
23							

Table 88 Instrument Conditions Be

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Ge 72	ORS			N/A	9m/z
2						NA	
3	ICP-MS	Sc 45	ORS	No Gas	500	500	9
4						NA	
5	ICP-OES-AV	Lu 261.541			200	NA	313.107
6	ICP-OES	Ir, Rh & Sc	NA	NA	N/A	50	313.042nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Sc[No Gas]	ORS	He	500	NA	9
9	ICP-OES-AV	Lu			83	NA	313.042
10	ICP-MS	Sc	NA		250	NA	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	NA	NA	625	NA	9
13	ICP-MS					NA	
14	NA	NA	NA	NA	NA	NA	NA
15					250	NA	
16	ICP-OES-AV-buffer	Lu			100	NA	313.107
17	ICPMS					NA	
18	ICP-MS	Sc-2 45	KED			NA	9
19	ICP-MS	Sc	NA	standard mode	2000	NA	9
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV					NA	
22						NA	
23						NA	

Table 89 Instrument Conditions Bi

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Rh 103	ORS		N/A		209m/z
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-MS	Lu 175	ORS	He	500	500	209
4	NA	NA	NA	NA	NA	NA	NA
5					NA		
6	ICP-MS	Ir, Rh & Sc	NA	NA	N/A	50	209 m/z
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Lu	ORS	He	NA	500	209
9	ICP-OES-AV	Lu			NA	NT	NT
10	ICP-MS	Ir	NA		NA	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Ir	NA	NA	NA	625	209
13	ICP-MS				NA		
14	NA	NA	NA	NA	NA	NA	NA
15					NA		
16	ICP-MS	193	ORS	He	100	NA	209
17	ICPMS				NA		
18	NT	NA			NA		NA
19	ICP-MS	Tb	NA	standard mode	NA	2000	209
20	NA	NA	NA	NA	NA	NA	NA
21					NA		
22					NA		
23					NA		

Table 90 Instrument Conditions Ca

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Y377	NA			N/A	317.933 nm
2						NA	
3	NA	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA	NA
5	ICP-OES-AV	Lu 261.541			200	NA	315.887
6	ICP-OES	Eu & Cs	NA	NA	N/A	50	315.887, 370.602nm
7	NA	NA				NA	NA
8	NA	NA	NA	NA	NA	500	NA
9	ICP-OES-AV	Lu			83	NA	317.933
10	NA	NA	NA	NA	NA	NA	NA
11	AAS					NA	
12	ICP-MS	Sc	KED	He	625	NA	44
13	ICP-OES-AV					NA	315.885
14	AAS	NA	NA	NA	50	NA	422.7
15					250	NA	
16	ICP-OES-AV-buffer	Lu			NA	100	430.253
17	ICPMS					NA	
18	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20						NA	
21	ICP-OES-RV				100	NA	318.887
22	NA	NA	NA	NA	NA	NA	NA
23	NA	NA	NA	NA	NA	NA	NA

Table 91 Instrument Conditions Cd

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Rh 103	ORS				111m/z
2						NA	
3	ICP-MS	Rh 103	ORS	He	500	500	111/114
4	ICP-OES-AV	Y	NA	NA	18	NA	228.802
5	ICP-OES-AV	Lu 261.541			200	200	214.439
6	ICP-OES	Ir, Rh & Sc	NA	NA	50	50	226.502nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Rh	ORS	He	500		111
9	ICP-OES-AV	Lu			83	83	214.439
10	ICP-MS	Rh	NA		250	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Rh	NA	NA	625	625	111
13	ICP-MS						
14	AAS	None	NA	NA	20	NA	228.8
15					250		
16	ICP-MS	103	ORS	He	100	NA	114
17	ICPMS						
18	ICP-MS	In-1 115	KED				111
19	ICP-MS	Rh	KED	He	1000	1000	111
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				100	100	214.439
22							
23							

Table 92 Instrument Conditions Co

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Te214	NA		N/A		228.615 nm
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-MS	Sc 45	ORS	He	500	500	59
4	NA	NA	NA	NA	NA	NA	NA
5	ICP-OES-AV	Lu 261.541			NA	200	228.615
6	ICP-OES	Ir, Rh & Sc	NA	NA	N/A	50	228.616nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Sc	ORS	He	NA	500	59
9	ICP-OES-AV	Lu			NA	83	230.786
10	ICP-MS	Rh	UC	He	NA	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Ge	KED	He	NA	625	59
13	ICP-MS				NA		
14	NA	NA	NA	NA	NA	NA	NA
15					NA		
16	ICP-MS	103	ORS	He	100	NA	59
17	ICPMS				NA		
18	ICP-MS	Sc 45	KED		NA		59
19	ICP-MS	Ga	KED	He	NA	2000	59
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				NA	100	228.615
22					NA		
23					NA		

Table 93 Instrument Conditions Cr

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Te214	NA				267.716 nm
2						NA	
3	ICP-MS	Sc 45	ORS	He	500	500	52
4	ICP-OES-AV	Y	NA	NA	18	NA	267.716
5	ICP-OES-AV	Lu 261.541			200	200	267.716
6	ICP-OES	Ir, Rh & Sc	NA	NA	N/A	50	267.716nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Sc	ORS	He	500	500	63
9	ICP-OES-AV	Lu			83	83	267.716
10	ICP-MS	Sc	UC	He	250	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	KED	He	625	625	52
13	ICP-OES-AV						267.716
14	AAS	None	NA	NA	20	NA	357.9
15					250		
16	ICP-MS	103	ORS	He	100	NA	52
17	ICPMS						
18	ICP-MS	Sc 45	KED				52
19	ICP-MS	Sc	KED	He	1000	1000	52
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				100	100	367.716
22							
23							

Table 94 Instrument Conditions Cs

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1					NA		
2	NA	NA	NA	NA	NA	NA	NA
3	NA	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA	NA
5					NA		
6	ICP-MS	Ir, Rh & Sc	NA	NA	N/A	50	133 m/z
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Lu			NA	NT	NT
10	ICP-MS	Rh	NA	He	NA	250	
11	NA	NA	NA	NA	NA	NA	NA
12					NA		
13	ICP-MS				NA		
14	NA	NA	NA	NA	NA	NA	NA
15					NA		
16						NA	
17	ICPMS				NA		
18	NT	NA			NA		NA
19	ICP-MS	Tb	NA	standard mode	NA	2000	133
20	NA	NA	NA	NA	NA	NA	NA
21					NA		
22					NA		
23					NA		

Table 95 Instrument Conditions Cu

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Y377	NA				327.395 nm
2						NA	
3	ICP-MS	Sc 45	ORS	He	500	500	63
4	ICP-OES-AV	Y	NA	NA	18	NA	324.752
5	ICP-OES-AV	Lu 261.541			200	200	324.754
6	ICP-OES	Ir, Rh & Sc	NA	NA	50	50	327.395nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Sc	ORS	He	500	500	52
9	ICP-OES-AV	Lu			83	83	327.395
10	ICP-MS	Ga	UC	He	250	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Ge	KED	He	625	625	63
13	ICP-OES-AV						327.397
14	AAS	None	NA	NA	20	NA	324.7
15					250		
16	ICP-MS	103	ORS	He	100	NA	65
17	ICPMS						
18	ICP-MS	Sc 45	KED				63
19	ICP-MS	Ga	KED	He	1000	1000	63
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				100	100	324.754
22							
23							

Table 96 Instrument Conditions Fe

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Te214	NA			N/A	259.940nm
2						NA	
3	NA	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA	NA
5	ICP-OES-AV	Lu 261.541			200	NA	240.489
6	ICP-OES	Eu & Cs	NA	NA	N/A	50	238.204, 258.588, 259.940nm
7	NA	NA				NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Lu			83	NA	238.204
10	NA	NA	NA	NA	NA	NA	NA
11	ICP-OES					NA	
12	ICP-MS	Sc	KED	He	625	NA	56
13	ICP-OES-AV					NA	238.204
14	AAS	None	NA	NA	50	NA	248.3
15					250	NA	
16	ICP-OES-AV-buffer	Lu			NA	500	239.563
17	ICPMS					NA	
18	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20						NA	
21	ICP-OES-RV				100	NA	234.35
22	NA	NA	NA	NA	NA	NA	NA
23	NA	NA	NA	NA	NA	NA	NA

Table 97 Instrument Conditions Hg

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Ir 193	ORS				202m/z
2						NA	
3	ICP-MS	Lu 175	NA	He	500	500	201/202
4	Hydride	NA	NA	NA	110	NA	253.7
5	CVAAS						253.7
6	FIMS-AAS	NA	NA	NA	50	N/A	253.7nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Lu	ORS	He	500	500	202
9	CVAAS				83	83	253.7
10	ICP-MS	Ir	NA		250	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Ir	NA	NA	625	625	201
13	FIMS						
14	AAS	None	NA	NA	25	NA	253.6
15					250		
16	VGA-ICP-OES				100	NA	194.164
17	ICPMS						
18	ICP-MS	Ir 193	KED				202
19	ICP-MS	Tb	NA	standard mode	1000	1000	201
20	NA	NA	NA	NA	NA	NA	NA
21	CVAFS						
22							
23							

Table 98 Instrument Conditions K

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Y377	NA			N/A	766.491nm
2						NA	
3	NA	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA	NA
5	ICP-OES-AV	Lu 261.541			200	NA	766.491
6	ICP-OES	Eu & Cs	NA	NA	N/A	50	404.721nm, 766.491nm
7	NA	NA				NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Lu			83	NA	769.897
10	NA	NA	NA	NA	NA	NA	NA
11	AAS					NA	
12	ICP-MS	Sc	KED	He	625	NA	39
13	ICP-OES-AV					NA	404.724
14	AAS	None	NA	NA	50	NA	766.5
15					250	NA	
16	ICP-OES-AV-buffer	Lu			NA	100	766.491
17	ICPMS					NA	
18	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20						NA	
21	ICP-OES-RV				100	NA	766.491
22	NA	NA	NA	NA	NA	NA	NA
23	NA	NA	NA	NA	NA	NA	NA

Table 99 Instrument Conditions La

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS				NA		
2	NA	NA	NA	NA	NA	NA	NA
3	NA	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA	NA
5					NA		
6	ICP-MS	Ir, Rh & Sc	NA	NA	N/A	50	139 m/z
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Lu			NA	NT	NT
10	ICP-MS	In	NA		NA	250	
11	NA	NA	NA	NA	NA	NA	NA
12					NA		
13	ICP-MS				NA		
14	NA	NA	NA	NA	NA	NA	NA
15					NA		
16						NA	
17	ICPMS				NA		
18	NT	NA			NA		NA
19	ICP-MS	Tb	NA	standard mode	NA	2000	139
20	NA	NA	NA	NA	NA	NA	NA
21					NA		
22					NA		
23					NA		

Table 100 Instrument Conditions Li

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Ge 72	ORS		N/A		7 m/z
2						NA	
3	NA	NA	NA	NA	NA	NA	NA
4						NA	
5						NA	
6	ICP-MS	Ir, Rh & Sc	NA	NA	N/A	50	7 m/z
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Sc[No Gas]	ORS	He	500	NA	7
9	ICP-OES-AV	Lu			NA	NA	NT
10	ICP-MS	Sc	NA		250	NA	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	NA	NA	625	NA	7
13	ICP-MS					NA	
14	NA	None	NA	NA	NA	NA	NA
15					250	NA	
16	ICP-OES-AV-buffer	Lu			100	NA	670.783
17	ICPMS					NA	
18	ICP-MS	Sc - 45	KED			NA	7
19	ICP-MS	Sc	NA	standard mode	2000	NA	7
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				100	NA	670.783
22						NA	
23						NA	

Table 101 Instrument Conditions Mg

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-RV	Y377	NA			N/A	280.27 nm
2						NA	
3	NA	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA	NA
5	ICP-OES-AV	Lu 261.541			200	NA	280.27
6	ICP-OES	Eu & Cs	NA	NA	N/A	50	383.829nm
7	NA	NA				NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Lu			83	NA	383.829
10	NA	NA	NA	NA	NA	NA	NA
11	AAS					NA	
12	ICP-MS	Sc	KED	He	625	NA	25
13	ICP-OES-AV					NA	383.83
14	AAS	None	NA	NA	50	NA	285.2
15					250	NA	
16	ICP-OES-AV-buffer	Lu			NA	100	279.078
17	ICPMS					NA	
18	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20						NA	
21	ICP-OES-RV				100	NA	383.23
22	NA	NA	NA	NA	NA	NA	NA
23	NA	NA	NA	NA	NA	NA	NA

Table 102 Instrument Conditions Mn

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Te214	NA			N/A	191.446 nm
2						NA	
3	ICP-MS	Sc 45	ORS	He	500	500	55
4						NA	
5	ICP-OES-AV	Lu 261.541			200	NA	257.61
6	ICP-OES	Ir, Rh & Sc	NA	NA	N/A	50	261.021nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Sc	ORS	He	500	NA	55
9	ICP-OES-AV	Lu			83	NA	260.568
10	ICP-MS	Rh	UC	He	250	NA	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	KED	He	625	NA	55
13	ICP-OES-AV					NA	261.021
14	AAS	None	NA	NA	20	NA	279.5
15					250	NA	
16	ICP-MS	103	ORS	He	100	NA	55
17	ICPMS					NA	
18	ICP-MS	Sc 45	KED		10	NA	55
19	ICP-MS	Sc	KED	He	2000	NA	55
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				100	NA	257.61
22						NA	
23						NA	

Table 103 Instrument Conditions Mo

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Te214	NA			N/A	204.598 nm
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-MS	Rh 103	ORS	He	500	500	95/98
4	NA	NA	NA	NA	NA	NA	NA
5	ICP-OES-AV	Lu 261.541			NA	200	202.032
6	ICP-OES	Ir, Rh & Sc	NA	NA	N/A	50	202.032nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Rh	ORS	He	NA	500	95
9	ICP-OES-AV	Lu			NA	83	202.032
10	ICP-MS	Rh	UC	He	NA	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Rh	NA	NA	NA	625	95
13	ICP-MS				NA		
14	NA	NA	NA	NA	NA	NA	NA
15					NA		
16	ICP-MS	103	ORS	He	100	NA	98
17	ICPMS				NA		
18	ICP-MS	Y 89	KED		NA		98
19	ICP-MS	Rh	NA	standard mode	NA	2000	98
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				NA	100	202.032
22					NA		
23					NA		

Table 104 Instrument Conditions Na

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-RV	Y377	NA			N/A	589.592 nm
2						NA	
3	NA	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA	NA
5	ICP-OES-AV	Lu 261.541			200	NA	588.995
6	ICP-OES	Eu & Cs	NA	NA	N/A	50	330.237, 589.592nm
7	NA	NA				NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Lu			83	NA	330.237
10	NA	N/A	NA	NA	NA	NA	NA
11	AAS					NA	
12	ICP-MS	Sc	KED	He	625	NA	23
13	ICP-OES-AV					NA	330.235
14	AAS	None	NA	NA	50	NA	589.0
15					250	NA	
16	ICP-OES-AV-buffer	Lu			NA	100	589.592
17	ICPMS					NA	
18	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20						NA	
21	ICP-OES-RV					NA	216.555
22	NA	NA	NA	NA	NA	NA	NA
23	NA	NA	NA	NA	NA	NA	NA

Table 105 Instrument Conditions Ni

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Te214	NA				216.555 nm
2						NA	
3	ICP-MS	Sc 45	ORS	He	500	500	60
4	ICP-OES-AV	Y	NA	NA	18	NA	231.604
5	ICP-OES-AV	Lu 261.541			200	200	231.604
6	ICP-OES	Ir, Rh & Sc	NA	NA	50	N/A	231.604nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Sc	ORS	He	500		60
9	ICP-OES-AV	Lu			83	83	231.604
10	ICP-MS	Rh	UC	He	250	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Ge	KED	He	625	625	60
13	ICP-MS						
14	AAS	None	NA	NA	20	NA	232.0
15					250		
16	ICP-MS	103	ORS	He	100	NA	60
17	ICPMS						
18	ICP-MS	Sc 45	KED				58
19	ICP-MS	Ga	KED	He	1000	1000	60
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV						
22							
23							

Table 106 Instrument Conditions P

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Te214	NA			N/A	177.434 nm
2						NA	
3	NA	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA	NA
5	ICP-OES-AV	Lu 261.541			200	NA	213.618
6	ICP-OES	Eu & Cs	NA	NA	N/A	50	185.827nm
7	NA	NA				NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Lu			83	NA	178.222
10	NA	N/A	NA	NA	NA	NA	NA
11						NA	
12	ICP-MS	Sc	KED	He	625	NA	31
13	ICP-OES-AV					NA	185.827
14	ICP-OES-AV	None	NA	NA	50	NA	214.914
15					250	NA	
16	ICP-OES-AV-buffer	Lu			NA	100	213.618
17	ICPMS					NA	
18	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20						NA	
21						NA	
22	NA	NA	NA	NA	NA	NA	NA
23	NA	NA	NA	NA	NA	NA	NA

Table 107 Instrument Conditions Pb

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Te214	NA				220.353 nm
2						NA	
3	ICP-MS	Lu 175	ORS	He	500	500	208
4	ICP-OES-AV	Y	NA	NA	18	NA	220.353
5	ICP-OES-AV	Lu 261.541			200	200	220.353
6	ICP-OES	Ir, Rh & Sc	NA	NA	50	N/A	220.353nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Lu	ORS	He	500	500	208
9	ICP-OES-AV	Lu			83	83	220.353
10	ICP-MS	Ir	NA		250	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Ir	NA	NA	625	625	206+207+208
13	ICP-MS						
14	AAS	None	NA	NA	20	NA	283.3
15					250		
16	ICP-MS	103	ORS	He	100	NA	208
17	ICP-MS						
18	ICP-MS	NA	KED				208
19	ICP-MS	Tb	KED	He	1000	1000	206+207+208
20	NA	NA	NA	NA	NA	NA	NA
21							
22							
23							

Table 108 Instrument Conditions Rb

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						N/A	
2						NA	
3	NA	NA	NA	NA	NA	NA	NA
4						NA	
5						NA	
6	ICP-MS	Ir, Rh & Sc	NA	NA	50	N/A	85 m/z
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Lu			NT	NA	NT
10	ICP-MS	Rh	NA		250	NA	
11	NA	NA	NA	NA	NA	NA	NA
12						NA	
13	ICP-MS					NA	
14	NA	None	NA	NA	NA	NA	NA
15						NA	
16						NA	
17	ICP-MS					NA	
18	NT	NA				NA	NA
19	ICP-MS	Rh	NA	standard mode	2000	NA	85
20	NA	NA	NA	NA	NA	NA	NA
21						NA	
22						NA	
23						NA	

Table 109 Instrument Conditions S

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Te214	NA			N/A	180.669 nm
2						NA	
3	NA	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA	NA
5	ICP-OES-AV	Lu 261.541			200	NA	180.669
6	ICP-OES	Eu & Cs	NA	NA	N/A	50	178.165,181.972nm
7	NA	NA				NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Lu			83	NA	181.972
10	NA	NA	NA	NA	NA	NA	NA
11	ICP-OES					NA	
12	ICP-OES-AV	Y	NA	NA	62.5	NA	181.975
13	ICP-OES-AV					NA	178.165
14	NA	None	NA	NA	NA	NA	NA
15					250	NA	
16	ICP-OES-AV-buffer	Lu			NA	100	181.972
17	ICP-OES-AV	Y			800	NA	
18	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20						NA	
21						NA	
22	NA	NA	NA	NA	NA	NA	NA
23	NA	NA	NA	NA	NA	NA	NA

Table 110 Instrument Conditions Sb

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Rh 103	ORS			N/A	122m/z
2						NA	
3	ICP-MS	Rh 103	ORS	He	500	500	121/123
4						NA	
5	ICP-OES-AV	Lu 261.541			200	NA	206.834
6	ICP-OES	Ir, Rh & Sc	NA	NA	50	N/A	206.834nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Rh	ORS	He	500	NA	123
9	ICP-OES-AV	Lu			83	NA	206.834
10	ICP-MS	Rh	NA		250	NA	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Rh	NA	NA	625	NA	121
13	ICP-OES-AV					NA	206.834
14	AAS	None	NA	NA	20	NA	217.6
15						NA	
16	ICP-MS	103	ORS	He	100	NA	121
17	ICPMS					NA	
18	NT	NA				NA	NA
19	ICP-MS	Rh	NA	standard mode	2000	NA	121
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				100	NA	217.582
22						NA	
23						NA	

Table 111 Instrument Conditions Se

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Rh 103	ORS				78m/z
2						NA	
3	ICP-MS	Sc 45	ORS	He/H2	500	500	78
4						NA	
5	ICP-OES-AV	Lu 261.541			200	200	196.026
6	ICP-OES	Ir, Rh & Sc	NA	NA	50	50	196.026nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Rh	ORS	H2	500	500	78
9	ICP-OES-AV	Lu			83	83	196.026
10	ICP-MS	Rh	UC	He	250	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Rh	NA	NA	625	625	82
13	ICP-MS						
14	AAS	None	NA	NA	20	NA	196.0
15					250		
16	ICP-MS	72	ORS	He	100	NA	78
17	ICPMS						
18	ICP-MS	Ge-1 72	KED			10	82
19	ICP-MS	Te	NA	standard mode	2000	2000	82
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				100	100	196.026
22							
23							

Table 112 Instrument Conditions Sn

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Rh 103	ORS			N/A	118m/z
2						NA	
3	ICP-MS	Rh 103	ORS	He	500	500	118
4						NA	
5	ICP-OES-AV	Lu 261.541			200	NA	189.925
6	ICP-OES	Ir, Rh & Sc	NA	NA	N/A	50	189.926nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Rh	ORS	He	500	NA	118
9	ICP-OES-AV	Lu			83	NA	189.925
10	ICP-MS	Rh	NA		250	NA	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Rh	NA	NA	625	NA	118
13	ICP-MS					NA	
14	AAS	None	NA	NA	20	NA	235.5
15					250	NA	
16	ICP-MS	103	ORS	He	100	NA	118
17	ICPMS					NA	
18	ICP-MS	In-1 115	KED			NA	118
19	ICP-MS	Rh	NA	standard mode	2000	NA	120
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				100	NA	189.925
22						NA	
23						NA	

Table 113 Instrument Conditions Sr

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Y371	NA			N/A	407.771 nm
2						NA	
3	NA	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA	NA
5	ICP-OES-AV	Lu 261.541			200	NA	460.733
6	ICP-OES	Ir, Rh & Sc	NA	NA	N/A	50	430.545nm
7	NA	NA				NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Lu			83	NA	407.771
10	NA	N/A	NA	NA	NA	NA	NA
11	ICP-OES					NA	
12	ICP-MS	Rh	NA	NA	625	NA	88
13	ICP-MS					NA	
14	ICP-OES-AV	None	NA	NA	20	NA	407.771
15						NA	
16	ICP-OES-AV-buffer	Lu			NA	100	407.771
17	ICPMS					NA	
18	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20						NA	
21						NA	
22	NA	NA	NA	NA	NA	NA	NA
23	NA	NA	NA	NA	NA	NA	NA

Table 114 Instrument Conditions Th

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						NA	
2						NA	
3	NA	NA	NA	NA	NA	NA	NA
4						NA	
5						NA	
6	ICP-MS	Ir, Rh & Sc	NA	NA	50	N/A	232 m/z
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Lu			NT	NA	NT
10	NA	N/A	NA	NA	NA	NA	NA
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Ir	NA	NA	625	NA	232
13	ICP-MS					NA	
14	NA	None	NA	NA	NA	NA	NA
15						NA	
16						NA	
17	ICPMS					NA	
18	NT	NA				NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20	NA	NA	NA	NA	NA	NA	NA
21						NA	
22						NA	
23						NA	

Table 115 Instrument Conditions T1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Rh 103	ORS		N/A		205 m/z
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-MS	Lu 175	ORS	He	500	500	205
4	NA	NA	NA	NA	NA	NA	NA
5					NA		
6	ICP-MS	Ir, Rh & Sc	NA	NA	N/A	50	203 m/z
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Lu	ORS	He	NA	500	205
9					NA	NT	NT
10	ICP-MS	Ir	NA		NA	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Ir	NA	NA	NA	625	205
13	ICP-MS				NA		
14	NA	NA	NA	NA	NA	NA	NA
15					NA		
16	ICP-MS	103	ORS	He	100	NA	205
17	ICPMS				NA		
18	NT	NA			NA		NA
19	ICP-MS	Tb	NA	standard mode	NA	2000	205
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				NA	100	190.794
22					NA		
23					NA		

Table 116 Instrument Conditions U

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Ir 193	ORS		N/A		238m/z
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-MS	Lu 175	ORS	He	500	500	238
4	NA	NA	NA	NA	NA	NA	NA
5					NA		
6	ICP-MS	Ir, Rh & Sc	NA	NA	N/A	50	238 m/z
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Lu	ORS	He	NA	500	238
9					NA	NT	NT
10	ICP-MS	Ir	NA		NA	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Ir	NA	NA	NA	625	238
13	ICP-MS				NA		
14	NA	NA	NA	NA	NA	NA	NA
15					NA		
16	ICP-MS	103	ORS	He	100	NA	238
17	ICP-MS				NA		
18	ICP-MS	Ir 193	KED		NA		238
19	ICP-MS	Tb	NA	standard mode	NA	2000	238
20	NA	NA	NA	NA	NA	NA	NA
21					NA		
22					NA		
23					NA		

Table 117 Instrument Conditions V

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Y371	NA				310.229 nm
2						NA	
3	ICP-MS	Sc 45	ORS	He	500	500	51
4						NA	
5	ICP-OES-AV	Lu 261.541			200	NA	292.401
6	ICP-OES	Ir, Rh & Sc	NA	NA	50	N/A	311.837nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Sc	ORS	He	500	NA	51
9	ICP-OES-AV	Lu			83	NA	292.401
10	ICP-MS	Sc	UC	He	250	NA	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	KED	He	625	NA	51
13	ICP-OES-AV					NA	311.839
14	AAS	None	NA	NA	20	NA	318.5
15					250	NA	
16	ICP-MS	103	ORS	He	100	NA	51
17	ICP-MS					NA	
18	ICP-MS	Sc 45	KED			NA	51
19	ICP-MS	Sc	KED	He	2000	NA	51
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				100	NA	292.401
22						NA	
23						NA	

Table 118 Instrument Conditions Zn

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Te214	NA				202.548 nm
2						NA	
3	ICP-MS	Sc 45	ORS	He	500	500	66
4	ICP-OES-AV	Y	NA	NA	18	NA	213.857
5	ICP-OES-AV	Lu 261.541			200	200	206.2
6	ICP-OES	Ir, Rh & Sc	NA	NA	50	N/A	206.2, 334.502nm
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-MS	Sc	ORS	He	500	500	66
9	ICP-OES-AV	Lu			83	83	213.857
10	ICP-MS	Rh	UC	He	250	250	
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Ge	KED	He	625	625	66
13	ICP-OES-AV						206.202
14	AAS	None	NA	NA	20	NA	213.9
15					250		
16	ICP-MS	103	ORS	He	100	NA	66
17	ICP-MS						
18	ICP-MS	Sc 45	KED				66
19	ICP-MS	Ga	KED	He	1000	1000	66
20	NA	NA	NA	NA	NA	NA	NA
21	ICP-OES-RV				100	100	206.2
22							
23							

Table 119 Instrument Conditions Exchangeable Ca²⁺

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm) /Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Y377				
2						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5						
6	ICP-OES	Eu & Cs	NA	NA	500	315.887, 370.602nm
7						
8	NA	NA	NA	NA	NA	NA
9	ICP-OES	Lu			20	317.933
10	NA	NA	NA	NA	NA	NA
11	AAS	NA	NA	NA	neat	422.7
12	ICP-OES-RV	Y	NA		20	317.933
13	ICP-OES-AV	Eu, Cs				
14	AAS	None	NA	NA	25	422.7
15						
16						
17	ICP-OES-AV					
18	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA
20	ICP-OES-RV					
21	ICP-OES-AV				20	422.673
22	NA	NA	NA	NA	NA	NA
23	NA	NA	NA	NA	NA	NA

Table 120 Instrument Conditions Exchangeable Mg²⁺

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Y377				
2						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5						
6	ICP-OES	Eu & Cs	NA	NA	500	383.829nm
7						
8	NA	NA	NA	NA	NA	NA
9	ICP-OES	Lu			20	383.829
10	NA	NA	NA	NA	NA	NA
11	AAS	NA	NA	NA	neat	285.2
12	ICP-OES-RV	Y	NA		20	285.213
13	ICP-OES-AV	Eu, Cs				
14	AAS	None	NA	NA	25	285.2
15						
16						
17	ICP-OES-AV					
18	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA
20	ICP-OES-RV					
21	ICP-OES-AV				20	285.213
22	NA	NA	NA	NA	NA	NA
23	NA	NA	NA	NA	NA	NA

Table 121 Instrument Conditions Exchangeable Na⁺

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Y377				
2						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5						
6	ICP-OES	Eu & Cs	NA	NA	500	330.237, 589.592nm
7						
8	NA	NA	NA	NA	NA	NA
9	ICP-OES	Lu			20	589.592
10	NA	NA	NA	NA	NA	NA
11	AAS	NA	NA	NA	neat	330.2
12	ICP-OES-RV	Y	NA		20	589.592
13	ICP-OES-AV	Eu, Cs				
14	AAS	None	NA	NA	25	589.0
15						
16						
17	ICP-OES-AV					
18	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA
20	ICP-OES-AV					
21	ICP-OES-AV				20	589.592
22	NA	NA	NA	NA	NA	NA
23	NA	NA	NA	NA	NA	NA

Table 122 Instrument Conditions Exchangeable K⁺

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Y377				
2						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5						
6	ICP-OES	Eu & Cs	NA	NA	500	404.721nm, 766.491nm
7						
8	NA	NA	NA	NA	NA	NA
9	ICP-OES	Lu			20	769.897
10	NA	NA	NA	NA	NA	NA
11	AAS	NA	NA	NA	neat	766.5
12	ICP-OES-RV	Y	NA		20	766.49
13	ICP-OES-AV	Eu, Cs				
14	AAS	None	NA	NA	25	766.5
15						
16						
17	ICP-OES-AV					
18	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA
20	ICP-OES-AV					
21	ICP-OES-AV				20	766.491
22	NA	NA	NA	NA	NA	NA
23	NA	NA	NA	NA	NA	NA

END OF REPORT