



Australian Government
Department of Industry,
Science and Resources

National
Measurement
Institute

Proficiency Test Final Report AQA 23-02 Metals, Nutrients and Exchangeable Bases in Soil

June 2023

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ACKNOWLEDGMENTS

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

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

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

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SUMMARY

This report presents the results of the proficiency test AQA 23-02 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 K, Colwell P, EC, pH of 1:5 soil / 0.01 M $CaCl_2$ extract, exchangeable bases (Ca^{2+} , K^+ , Mg^{2+} , Na^+) - 1M NH_4Cl extract and moisture content was also included in the program.

The sample set consisted of one dried soil 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. The results reported by Laboratories 8, 16, 17, 19 and 21 in Sample S2, were consistently either lower or higher than the robust average and spiked value by around the same factor for almost all analytes, an indication of laboratory bias. To avoid unfair scoring, these results were excluded from robust average calculations to avoid bias in calculation of the assigned value; they were also excluded from the calculation of all summary statistics.

25 laboratories enrolled and 24 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 763 z-scores, 662 (87%) returned a satisfactory score of $|z| \leq 2.0$.

Of 769 E_n -scores, 578 (76%) were satisfactory with $|E_n| \leq 1.0$.

No laboratory reported results for all 54 tests for which a z-score was calculated.

Laboratory 1 returned the highest number of satisfactory z-scores (51 out of 52 reported) and the highest number of satisfactory E_n -scores (50 out of 52 reported).

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

All participants, but one, used both HNO_3 and HCl as extraction reagents and most used a digestion temperature of $90^\circ C$ to $100^\circ C$.

Most participating laboratories were challenged by the process of subsampling a representative test portion from the sludge sample S2 and reporting results for this sample corrected for moisture content.

- 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 791 numerical results, 773 (98%) were reported with an expanded measurement uncertainty. The magnitude of these expanded uncertainties was within the range 0.06% to 155% of the reported value. An example of estimating measurement uncertainty using only proficiency testing data is given in Appendix 3.

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

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

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;
- controlled drug assay; and
- folic acid in flour.

AQA 23-02 is the 32nd 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 determination of inorganic analytes in soil;
- 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 60 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

25 laboratories participated and 24 submitted results.

The timetable of the study was:

Invitations issued: 6 February 2023
Samples dispatched: 6 March 2023
Results due: 14 April 2023
Interim report issued: 19 April 2023
Preliminary report issued 27 April 2023

2.3 Test Material Specification

Three samples were provided for analysis:

Sample S1 was 30 g of dried soil.

Sample S2 was 100 g of wet sludge.

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 B, Li, Rb, Sb, and V in S1; Al and Ba in S2; and calcium chloride-extractable B, Colwell K, Colwell P, electrical conductivity, exchangeable Ca, K, Mg and Na, PBI_{+ColP}, pH, S and total P in S3. The results of 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 and sediment 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 6 March 2023.

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 moist 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 determination of calcium chloride – extractable B, exchangeable bases (Ca²⁺, Mg²⁺, Na⁺, K⁺) - 1M NH₄Cl extract and of P buffer index (with Colwell

P)- PBI_{+ColP}, participants are asked to use the methods defined by Rayment, G.E. and David, J. L in “Soil Chemical Methods-Australasia”.²

- These samples are an attempt to mime the real samples encountered by a laboratory in its routine activities. Please use appropriate Good Laboratory Practice when handling them.
- 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 basis (corrected for moisture content) and in units of mg/kg.
- For S3 report results on as received basis in units of cmol(+)/kg for exchangeable bases (Ca²⁺, Mg²⁺, Na⁺, K⁺) - 1M NH₄Cl extract. Except for pH and EC, for all the other tests, report results on as received basis in units of mg/kg. EC results are to be reported in units of μS/cm.

SAMPLE S1		SAMPLE S2		SAMPLE S3	
Test acid extractable	Approximate Conc. Range (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	0.5-25	Ag	0.5-25	Ca (acid extractable)	>1000
B	2.5-125	As	0.5-25	Calcium chloride –extractable B ¹	Not Available
Be	0.25-15	Al	>1000	Colwell P	>10
Cd	0.25-25	Ba	10-250	Colwell K	>200
Cr	2.5-125	Bi	0.25-20	EC	>500μS/cm
Cu	10-250	Cd	0.25-20	Exchangeable Ca-1MNH ₄ Cl extract ²	>5 cmol(+)/kg
Hg	0.15-10	Co	5-250	Exchangeable Mg-1MNH ₄ Cl extract ²	>1 cmol(+)/kg
Li	0.5-25	Cu	10-250	Exchangeable Na-1MNH ₄ Cl extract ²	>0.5 cmol(+)/kg
Mn	20-800	Cs	0.25-20	Exchangeable K-1MNH ₄ Cl extract ²	>0.5cmol(+)/kg
Mo	2.5-125	Hg	0.25-20	Fe (acid extractable)	>1000
Ni	2.5-125	La	1-50	K (acid extractable)	>1000
Pb	2.5-125	Mo	1-50	Mg (acid extractable)	>1000
Rb	0.5-25	Ni	2.5-125	Na (acid extractable)	>100
Sb	1-50	Pb	2.5-125	P (acid extractable)	>100
Se	1-50	Se	0.5-25	P (total)	Not Available
Sn	1-50	Sr	1-50	P buffer index (with Colwell P)- PBI _{+ColP} ³	Not Available
Tl	0.25-25	Th	0.25-20	pH of 1:5soil/ 0.01M CaCl ₂ extract	Not Available
V	2.5-125	U	0.5-25	S (acid extractable)	Not Available
Zn	10-250	Zn	10-250	Total Carbon	300-12000
		Moisture	20-80% (g/100g)	Total Organic Carbon	300-12000
				Total Nitrogen	30-1200

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.
- Return the completed results sheet by e-mail (proficiency@measurement.gov.au), by 31 March 2023.

The due date for results was extended to 14 April 2023 due to delays in sample delivery to some of our overseas participants.

2.9 Interim and Preliminary Reports

An Interim Report was emailed to participants on 19 April 2023.

A Preliminary Report was issued on 27 April 2023. This report included: a summary of the results reported by laboratories, assigned values, performance coefficient of variations, z-scores and En-scores for each analyte tested by participants.

No data from the preliminary report has been changed in the present Final Report.

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. H ₂ O ₂ (mL)	Other (mL)
1*		1	100	120	3	3			
3	USEPA Method 6010c, USEPA Methods 7471B, 7470A, 7471B	2.5	90-98	90	3	3			
5		0.5	105	120	2	1			
6	USEPA 3050B	0.5	95	120	3	3			
7	US EPA METHOD 3010	2	90-95	60	4	12			4 (H ₂ O)
8	US EPA 6010C	1	98	150	5	5			
9	In house	2	95	180			10	5	
10	EPA Method 200.8 (Environmental Express HotBlock Digestion System)	S1 & S3: 0.25 S2: 5	85	240	5	5			
11	USEPA 3050B								
13		1	95	120	7.5	2.5			
14	US EPA 3050B	0.5	95	120	7.5	5		1.5	
16*	USEPA Method 3050								
17	SCP Science DigiPrep apparatus digestion block	1	90	240	4	1			
18	Soil Chemical Methods - Australasia (Rayment and Lyons) - Method 17B1	S1: 0.504g S2: 0.562g	95	120	18.75	6.25			
19	In House, US EPA 6020B	2	90-95	60	4	12			
20	USEPA Method 6010c, USEPA Methods 7471B, 7470A, 7471B	2.5	90-90	90	3	3			
21	US-EPA Method 200.2	1	95	50	2	2			10 (H ₂ O)
22	Acid Digestion of sediment, sludges and soil-USEPA 3050	1	95	90	3	3			
23	In House S6 - referencing APHA 3125	0.4	120	60	2.5	7.5			
24	US EPA 3051A	0.5	175	15	9	3			
25*	In house reverse aqua regia digestion	1	110	60	3.5	1.5			

*See Additional Information for Methodology in Table 2

Table 2 Additional Information for Acid Extractable Elements

Lab Code	Additional Information
1	Made to 20mL after one-hour, final hour with additional vertexing and final vol 40mL
16	Instrument for Hg: Cetac Hg Analyser
25	Cell Gas: Standard mode (No gas)

Table 3 Methodology for Total Carbon

Lab. Code	Method Reference	Total Carbon Test Method	Total Carbon Measurement Technique	Additional Information
1		High Temperature Oxidation	High temp combustion and infrared detection	
2	6B2b (TC) 6B3 (TOC)	High Temperature Oxidation		
3		High Temperature Oxidation		
6	Inhouse	High Temperature Oxidation	Infrared Gas Analysis	
8		High Temperature Oxidation		
11		High Temperature Oxidation		
15	AS 1289.4.1.1	High Temperature Oxidation		
16	Based on Rayment & Higginson 6B1.	Colourimetrically by Discrete Analyser at 600 nm.	Colourimetrically by Discrete Analyser at 600 nm.	
18	Soil Chemical Methods - Australasia (Rayment & Lyons) - Method 6A1			Mercuric sulfate is added to remove chloride interference
20	AS1289.4.1.1 2019 (Walkley Black method)			
22	Combustion	High Temperature Oxidation	LECO	
23	In house S4a	High Temperature Oxidation	LECO	
24	6B2 and 6B3	High Temperature Oxidation	Dumas Combustion	

Table 4 Methodology for Total Organic Carbon

Lab. Code	Method Reference	Total Organic Carbon Test Method	Total Organic Carbon Measurement Technique	Additional Information
1		High Temperature Oxidation	High temp combustion and infrared detection	
2	6B2b (TC) 6B3 (TOC)	High Temperature Oxidation		Sample was Fizz test with 4 M HCl and no Fizzing observed. Therefore no acid treatment was carried out for TOC
3		Chemical Oxidation		
6	Inhouse	High Temperature Oxidation	Infrared Gas Analysis	
8		High Temperature Oxidation		
11		High Temperature Oxidation		
15	AS 1289.4.1.1	Chemical Oxidation Ag ₂ SO ₄ added	Walkely & Black Method	
16	Based on Rayment & Higginson 6B1.	Chemical Oxidation	Discrete Analyser	The sample is digested in Dichromate / Sulfuric Acid to oxidise the organic carbon.
18	Soil Chemical Methods - Australasia (Rayment & Lyons) - Method 6A1	Chemical Oxidation	UV Vis at 600nm	Mercuric sulfate is added to remove chloride interference
20	AS1289.4.1.1 2019 (Walkley Black method)	Chemical Oxidation Ag ₂ SO ₄ added	Titration	
22	Combustion	High Temperature Oxidation	LECO	
23	In house S4a	High Temperature Oxidation	LECO	Sample digested with sulfurous acid prior to analysis on LECO
24	6B2 and 6B3	High Temperature Oxidation	Dumas Combustion	

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)
1		0.5	50	16		1000		DA 880 nm
2	9B2	1	100	17				FIA 880 nm
3		1	100	16	200	200	ICP-OES- AV-buffer 766.491 nm	ICP-OES- AV-buffer 213.618 nm
5		1	100	16			N/A	FIA
8	Method 9B1: George E. Rayment and David J. Lyons		120	16				
13	QWI-EN/EK080	1	100	16		1/1000		DA 880 nm
16	Based on Rayment & Higginson 9B1.	1	100	16	NT		NT	DA 880 nm
18	Soil Chemical Methods- Australasia - Rayment & Lyons - method 9B (Colwell P) and 18A1 (Colwell K)	Colwell K 1.005g Colwell P 1.003g	100	16	497.5	498.5	AAS 766.5 nm	DA 880 nm
23	Colwell P 9B2, Colwell K 18A1	0.4	40	16	3280	328	ICPMS 31 nm	FIA 880 nm
24	9B2	0.5	50	16	1000	100	ICP-OES-RV 766.49 nm	Continuous Flow Analyzer 880 nm

Table 6 Methodology for P Buffer Index – PBI_{+CoIP}

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)
2	9I2a	7	70	17	FIA	100	880
8	9I2				ICP-OES	100	213.617
18	9I2	6.999	70	16	DA	1000	880
23	9I2b	2	20	16	ICP-OES	10	213.617
24	9I2	4	40	17	Continuous Flow Analyzer	10	880

*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 Kjeldahl digestion and FIA
15	Total P by Kjeldahl digestion and DA
16	Total P by kjeldahl digestion and DA
18	In-house method; Soil digested as per Acid extractable metals (reverse aquaregia as above) then digest analysed on DA.
24	US EPA 3051A

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)
3	12C	10	20	10	ICP-OES-AV buffer	2	208.956
8	12C	2	20	10			249.68
22	12C	10	20	10	ICP-OES	2	208.956
23	12C2	10	20	10	ICP-OES	2	208.889
24	12C	5	10	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		Digestion Distillation TN=TKN+NO _x	Titrimetric method	Manual Analysis
2	7A5	Combustion Dumas	Dumas –High temperature combustion	LECO
3		Digestion TN=TKN+NO _x	Colorimetric – salicylate method	DA
6	Inhouse	Digestion TN=TKN	Colorimetric – phenate method	FIA
8		Combustion	Dumas –High temperature combustion	LECO
11		Digestion TN=TKN	Colorimetric – salicylate method	SFA
15	APHA 4500-Norg. A & D	Digestion	Colorimetric – salicylate method	DA
16	ASTM D2216-98	Digestion TN=TKN+NO _x	Colorimetric – salicylate method	DA
18	In-house method based on APHA 23rd Edition 4500 -Norg B	Digestion Distillation TN=TKN	Colorimetric – salicylate method	DA
20	USEPA m7c & APHA 4500-D	Digestion TN=TKN+NO _x		
22	Lyons, G.E.R.a.D.J,2011 Total soil N-Dumas high temperature combustion	Combustion	Dumas –High temperature combustion	LECO
23	In house S4a - Dumas combustion	Combustion	Dumas –High temperature combustion	LECO
24	7A5	Combustion	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)
1	15A1	2	2	1M NH ₄ Cl	40
2	15A1	2	1	1M NH ₄ Cl	40
3	15A1	2.5	2	1M NH ₄ Cl	50
5	15A1	5	1	1M NH ₄ Cl	100
8	15A1	2	1	1M NH ₄ Cl	10
13	QWI-EN.ED003 and testing via QWI-EN/WD004	5	1	1M NH ₄ Cl	50
15	15A1	2.5	1		
16	Method is referenced to Rayment and Lyons, 2011, sections 15D3 and 15N1.	5	1	1M NH ₄ Cl	100
18	15A1	5.03	1	1M NH ₄ Cl	100
20	Rayment and Lyons 15B1	2	1		
22	15A1	2.5	1	1M NH ₄ Cl	50
23	15A1	1	1	1M NH ₄ Cl	20
24	15A1	2.5	1	1M NH ₄ Cl	50

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 (Tables 11 and 12).

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 - CRM Duplicate Analysis	CRM Variation in Sample Moisture Content Recoveries of SS	Nordtest Report TR537
2	Top Down - reproducibility (standard deviation) from PT studies used directly	Control Samples - RM Duplicate Analysis Instrument Calibration	CRM	NMI Uncertainty Course
3	Top Down - precision and estimates of the method and laboratory bias	Control Samples - SS		Eurachem/CITAC Guide
5			CRM	
6	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples Duplicate Analysis		
7*	See 'Additional Information' section below	Control samples - CRM Instrument Calibration	CRM	See 'Additional Information' section below
8	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM	CRM Laboratory Bias from PT Studies	Eurachem/CITAC Guide
9	Top Down - precision and estimates of the method and laboratory bias	Control Samples - RM	CRM	Eurolab Technical Report No1/2007
10	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples - SS Duplicate Analysis Instrument Calibration	Laboratory Bias from PT Studies Recoveries of SS	ISO/GUM
11	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM Duplicate Analysis	CRM	NMI Uncertainty Course
12	Top Down - precision and estimates of the method and laboratory bias	Standard deviation from PT studies only		Eurachem/CITAC Guide
		Control Samples Duplicate Analysis	CRM Instrument Calibration Recoveries of SS	
13	Top Down - precision and estimates of the method and laboratory bias	Control Samples - SS Duplicate Analysis Instrument Calibration	Instrument Calibration Recoveries of SS	Eurachem/CITAC Guide
14	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM Duplicate Analysis	CRM Variation in Sample Moisture Content Recoveries of SS	John Eames
15	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM	CRM Recoveries of SS	Eurachem/CITAC Guide
16	Top Down - precision and estimates of the method and laboratory bias	Control Samples - SS Duplicate Analysis Instrument Calibration	Instrument Calibration Laboratory Bias from PT Studies Recoveries of SS	
17	Top Down - precision and estimates of the method and laboratory bias	Duplicate Analysis	CRM	NMI Proficiency results
18	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)

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation ^a		Guide Document for Estimating MU
		Precision	Method Bias	
19	Top Down - precision and estimates of the method and laboratory bias	Control Samples - SS	Recoveries of SS	ISO/GUM
20	Top Down - precision and estimates of the method and laboratory bias	Control Samples - SS	Recoveries of SS	
21	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
22	Top Down - precision and estimates of the method and laboratory bias	Control Samples	Recoveries of SS	ISO/GUM
23	Top Down - precision and estimates of the method and laboratory bias	Control Samples - RM Duplicate Analysis	Instrument Calibration Standard Purity	Nordtest Report TR537
24	Standard deviation of replicate analyses multiplied by 2 or 3	Standard deviation from PT studies only		NMI Uncertainty Course
		Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration	
25	Top Down - precision and estimates of the method and laboratory bias	Duplicate Analysis Instrument Calibration	CRM Laboratory Bias from PT Studies Recoveries of SS	Eurachem/CITAC Guide

*Additional information in Table 12. ^aRM = Reference Material, CRM = Certified Reference Material, SS =Spiked samples.

Table 12 Additional Information for Basis of Uncertainty Estimate

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

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.

There were no comments given in this study.

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed Tables 13 to 72 with resultant summary statistics: robust average, median, mean, number of numeric results, 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 61. An example chart with interpretation guide is shown in Figure 1.

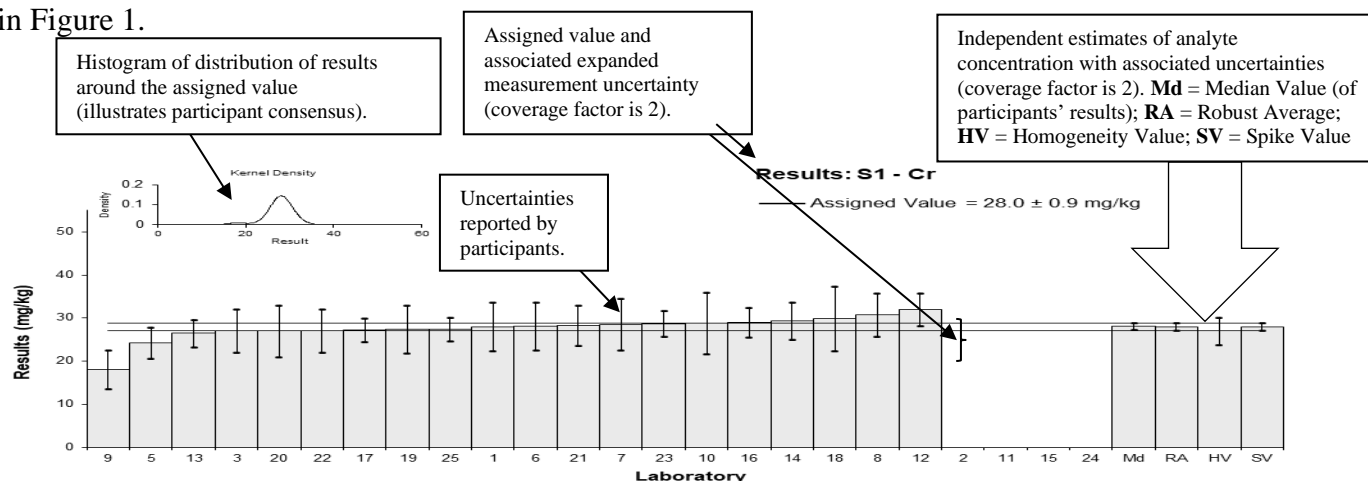


Figure 1 Guide to Presentation of Results

4.2 Outliers and Extreme Outliers

Outliers were results less than 50% and greater than 150% of the robust average and were removed before assigned value calculation. Gross errors were obvious blunders, such as those with incorrect units, decimal errors, or results from a different PT samples and were removed for calculation of summary statistics.^{3,4}

The results reported by Laboratories 8, 16, 17, 19 and 21 in Sample S2, were consistently either lower or higher than the robust average and spiked value by around the same factor for almost all analytes, an indication of laboratory bias. To avoid unfair scoring, these results were excluded from robust average calculations to avoid bias in calculation of the assigned value; they were also excluded from the calculation of all summary statistics.

4.3 Assigned Value

The assigned value is defined as: ‘the value attributed to a particular property of a proficiency test item.’¹ In this PT study, the property is the mass fraction of analyte. Assigned values were the robust average of participants’ results, outliers removed; the expanded uncertainties were estimated from the associated robust standard deviations.^{4,6}

4.4 Robust Average and Robust Between-Laboratory Coefficient of Variation

The robust averages and associated expanded measurement uncertainties were calculated using the procedure described in ‘Statistical methods for use in proficiency testing by interlaboratory comparisons, ISO13528.’⁶ 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 ISO13528.⁶

4.5 Target Standard Deviation for Proficiency Assessment

The target standard deviation for proficiency assessment (σ) is the product of the assigned value (X) and the performance coefficient of variation (PCV). 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 the Thompson Horwitz equation.⁷

4.6 z-Score

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

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

Where:

- z is z-score;
- χ is participant's result;
- X is the assigned value;
- σ is the target standard deviation.

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

- $|z| \leq 2.0$ is satisfactory.
- $2.0 < |z| < 3.0$ is questionable;
- $|z| \geq 3.0$ 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 participant's result;
- X is the assigned value;
- U_χ is the expanded uncertainty of the participant's result;
- U_X is the expanded uncertainty of the assigned value.

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

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

4.8 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025 must establish and demonstrate the traceability and measurement uncertainty associated with their test results.⁸ 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	Soil
Analyte	As
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	4.2	0.84	0.13	0.09
2	NT	NT		
3	4.4	4	0.45	0.07
5	3.837	0.692	-0.46	-0.40
6	4.4	0.9	0.45	0.31
7	4.19	0.84	0.11	0.08
8	<15	NR		
9	3.2	0.90	-1.49	-1.01
10	4.34	1.09	0.36	0.20
11	NT	NT		
12	<5	0.03		
13	<5	NR		
14	4.2	1.0	0.13	0.08
15	NT	NT		
16	4	0.532	-0.19	-0.22
17	3.85	0.38	-0.44	-0.65
18	<5	NR		
19	4.00	0.801	-0.19	-0.15
20	4.2	4	0.13	0.02
21*	6.9	1.7	4.50	1.63
22	4	4	-0.19	-0.03
23	4.41	0.8	0.47	0.36
24	NT	NT		
25	4.0724	0.41	-0.08	-0.11

* Outlier, see Section 4.2

Statistics

Assigned Value	4.12	0.16
Spike Value	3.90	0.24
Homogeneity Value	3.90	0.47
Robust Average	4.15	0.17
Median	4.20	0.18
Mean	4.26	
N	16	
Max	6.9	
Min	3.2	
Robust SD	0.26	
Robust CV	6.4%	

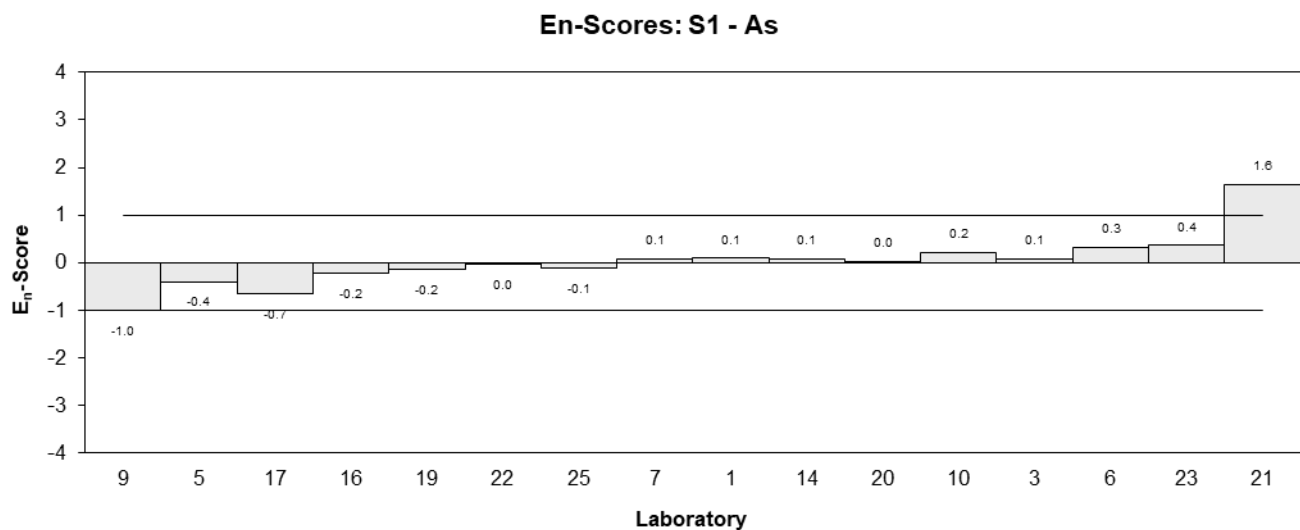
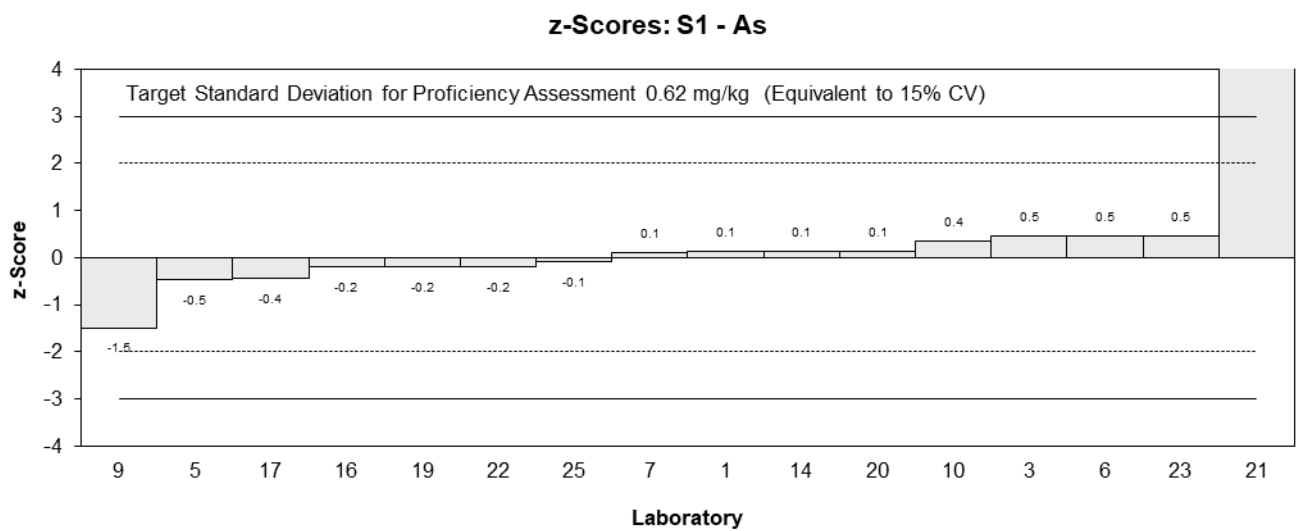
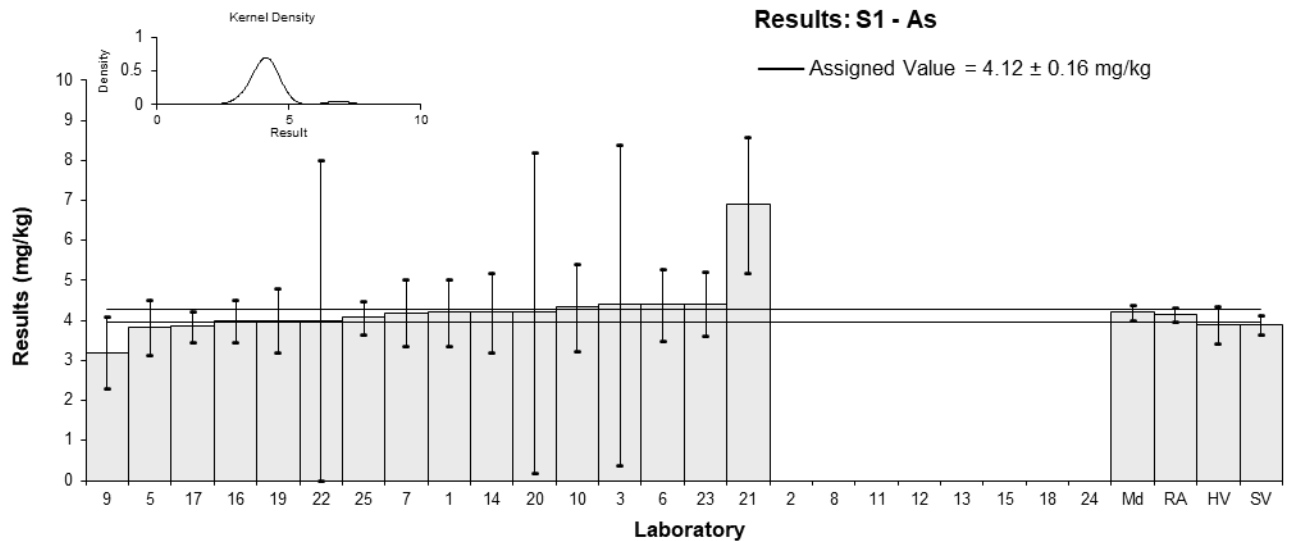


Figure 2

Table 14

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	6.1	1.2	0.98	0.44
2	NT	NT		
3	3.1	3	-1.96	-0.56
5	<20	NR		
6	NR	NR		
7	< 10	NR		
8	<5	NR		
9	NT	NT		
10	5.91	1.48	0.79	0.34
11	NT	NT		
12	5.2	0.312	0.10	0.05
13	<10	NR		
14	<10	NR		
15	NT	NT		
16	<5	NR		
17*	2.01	0.20	-3.03	-1.62
18*	28.0	7.0	22.45	3.16
19	<10	NR		
20	3.5	2	-1.57	-0.58
21	<20	3.6		
22	5	3	-0.10	-0.03
23	6.71	1.2	1.58	0.72
24	NT	NT		
25	NR	NR		

* Outlier, see Section 4.2

Statistics

Assigned Value	5.1	1.9
Spike Value	5.32	0.36
Robust Average	5.1	1.9
Median	5.2	1.9
Mean	7.3	
N	9	
Max	28	
Min	2.01	
Robust SD	2.3	
Robust CV	44%	

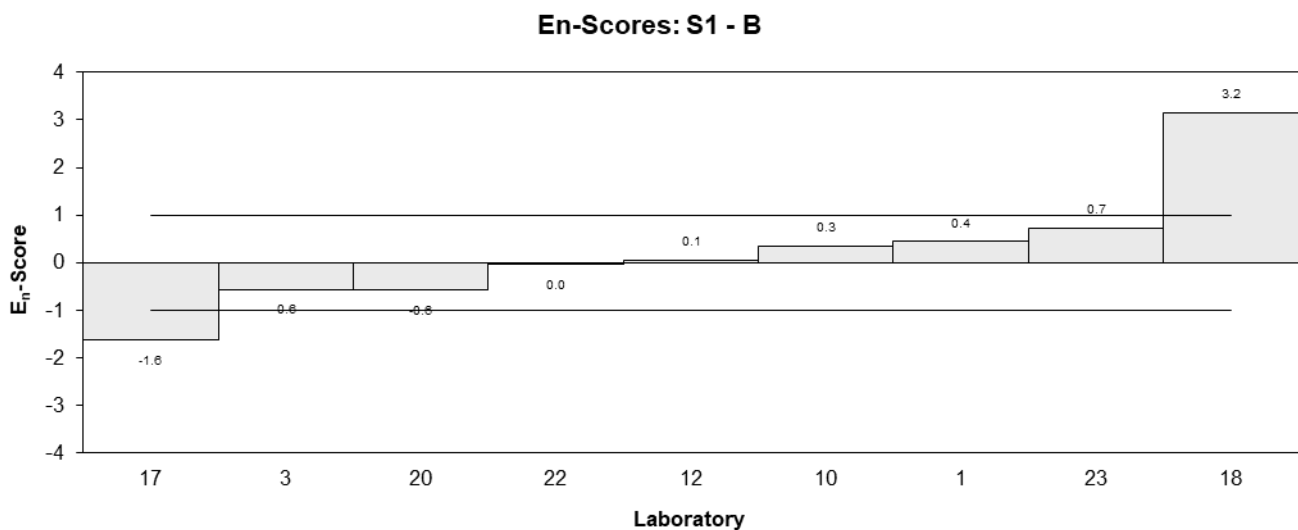
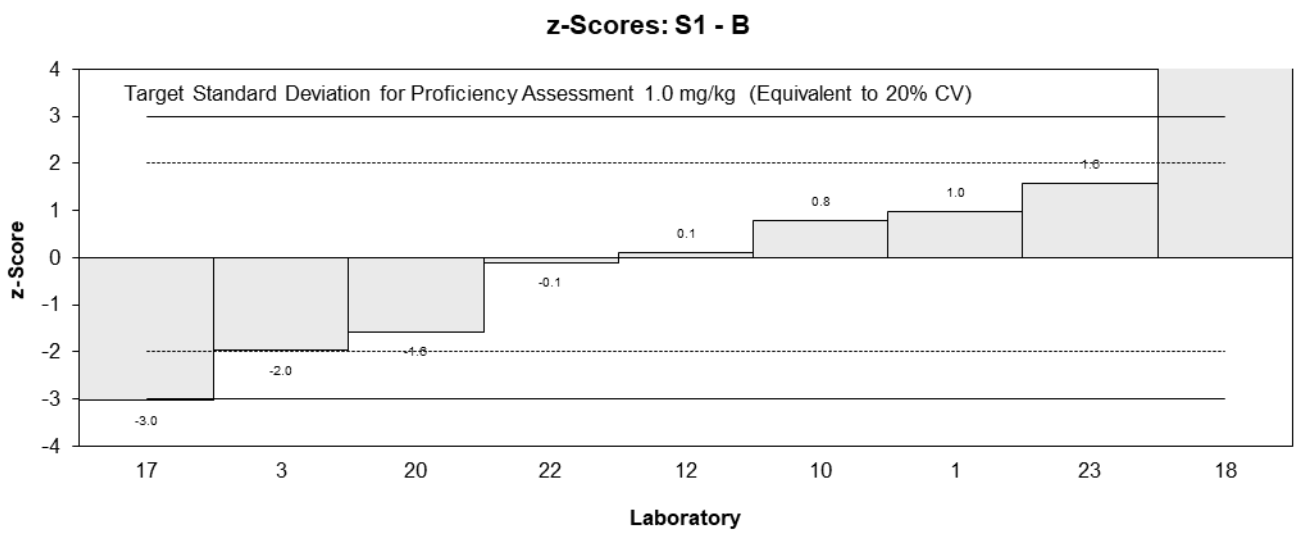
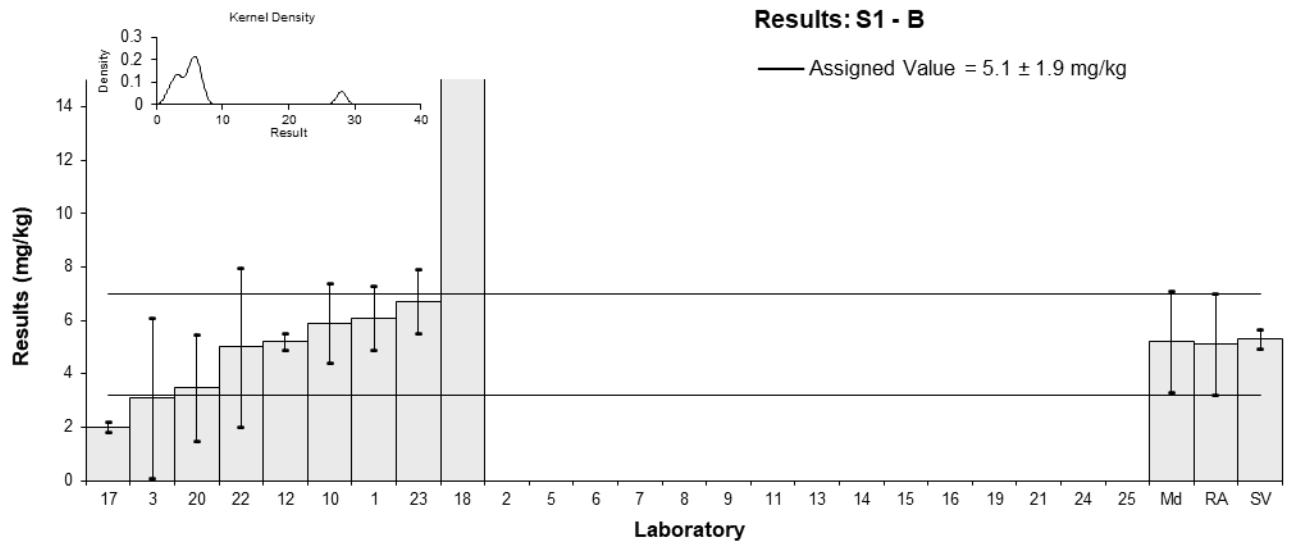


Figure 3

Table 15

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	0.75	0.15	0.28	0.20
2	NT	NT		
3	<1	NR		
5	0.709	0.192	-0.10	-0.06
6	0.72	0.16	0.00	0.00
7	< 2	NR		
8	<1	NR		
9	NT	NT		
10	0.529	0.135	-1.77	-1.37
11	NT	NT		
12	0.75	0.045	0.28	0.53
13	<5	NR		
14	<1	NR		
15	NT	NT		
16	0.7	0.091	-0.19	-0.21
17	0.71	0.07	-0.09	-0.13
18	<5	NR		
19	<2	NR		
20	<1	1		
21	0.7	0.18	-0.19	-0.11
22	<1	1		
23	0.84	0.1	1.11	1.14
24	NT	NT		
25	NR	NR		

Statistics

Assigned Value	0.720	0.034
Spike Value	0.720	0.050
Homogeneity Value	0.698	0.084
Robust Average	0.720	0.034
Median	0.710	0.012
Mean	0.712	
N	9	
Max	0.84	
Min	0.529	
Robust SD	0.04	
Robust CV	5.6%	

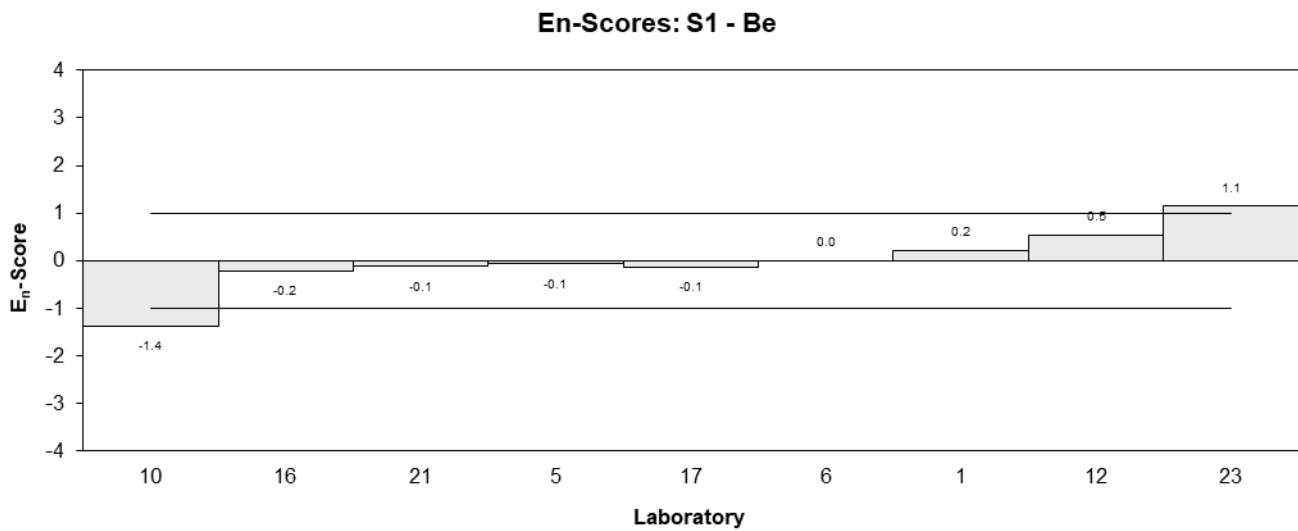
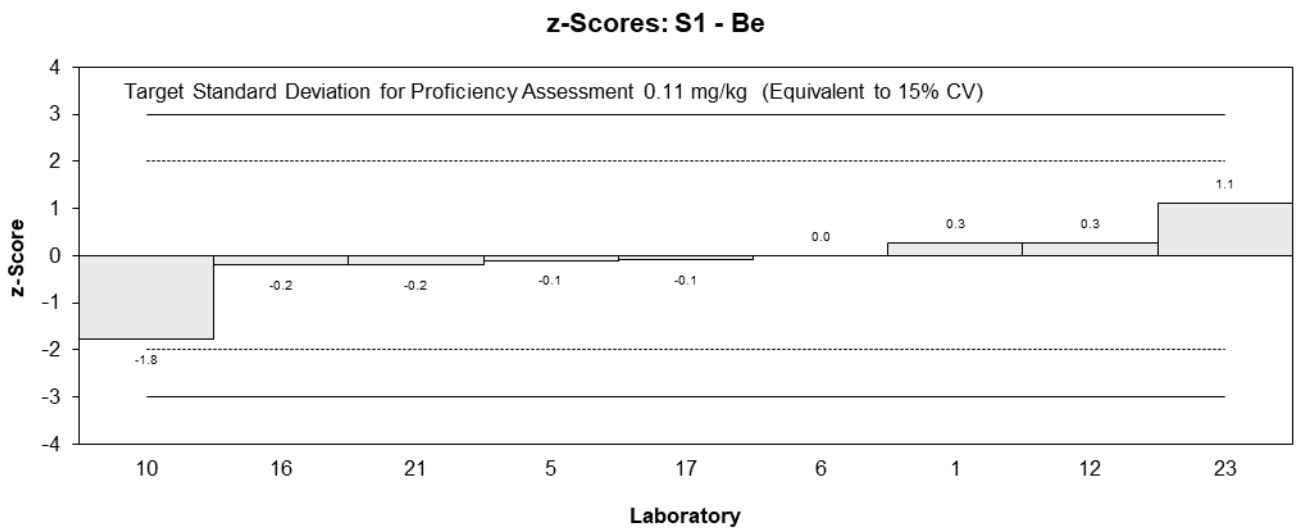
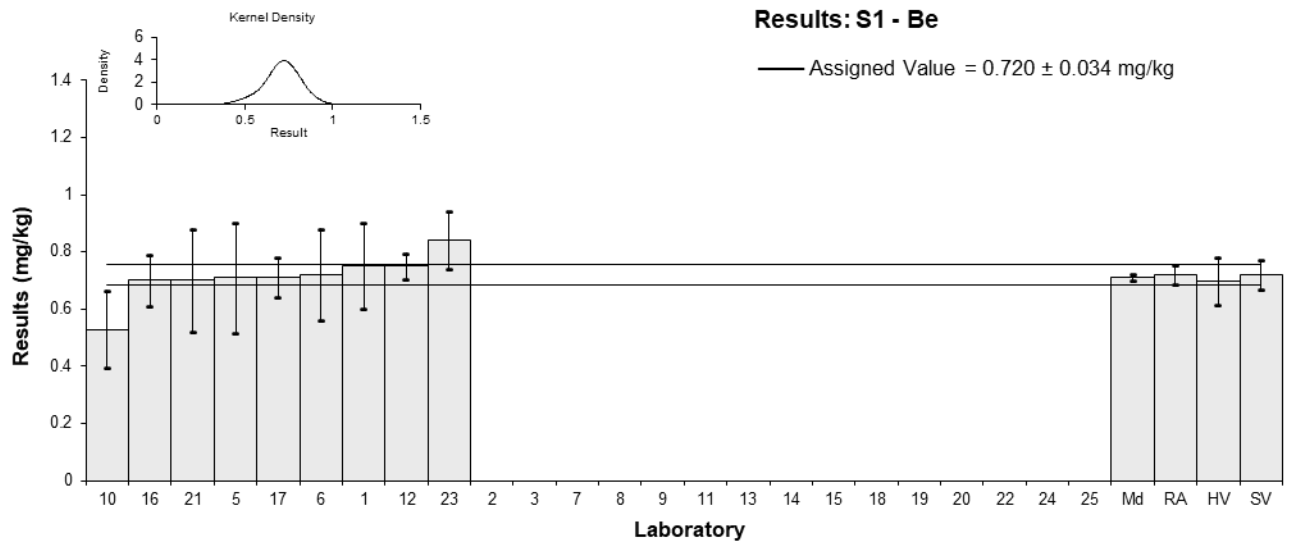


Figure 4

Table 16

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	0.76	0.15	0.51	0.33
2	NT	NT		
3	0.62	0.4	-0.81	-0.21
5	0.888	0.188	1.72	0.92
6	0.73	0.16	0.23	0.14
7	0.69	0.13	-0.15	-0.11
8	<1	NR		
9	0.51	0.10	-1.85	-1.68
10	0.651	0.163	-0.52	-0.32
11	NT	NT		
12	<1	0.04		
13	0.76550	0.092	0.56	0.54
14	<1	NR		
15	NT	NT		
16	0.6	0.0882	-1.00	-0.99
17	<1	NR		
18	<1	NR		
19	0.695	0.139	-0.10	-0.07
20	0.64	0.2	-0.62	-0.32
21	0.7	0.12	-0.06	-0.04
22	0.7	0.3	-0.06	-0.02
23	0.80	0.1	0.89	0.81
24	NT	NT		
25	0.82235	0.08	1.10	1.16

Statistics

Assigned Value	0.706	0.060
Spike Value	0.730	0.015
Homogeneity Value	0.75	0.11
Robust Average	0.706	0.060
Median	0.700	0.057
Mean	0.705	
N	15	
Max	0.888	
Min	0.51	
Robust SD	0.094	
Robust CV	13%	

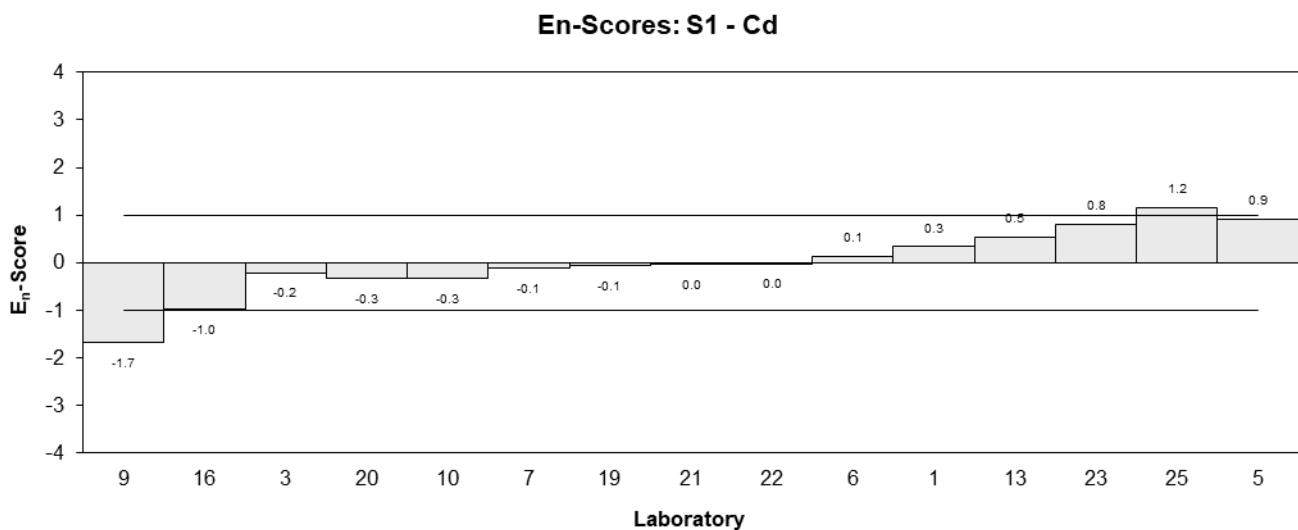
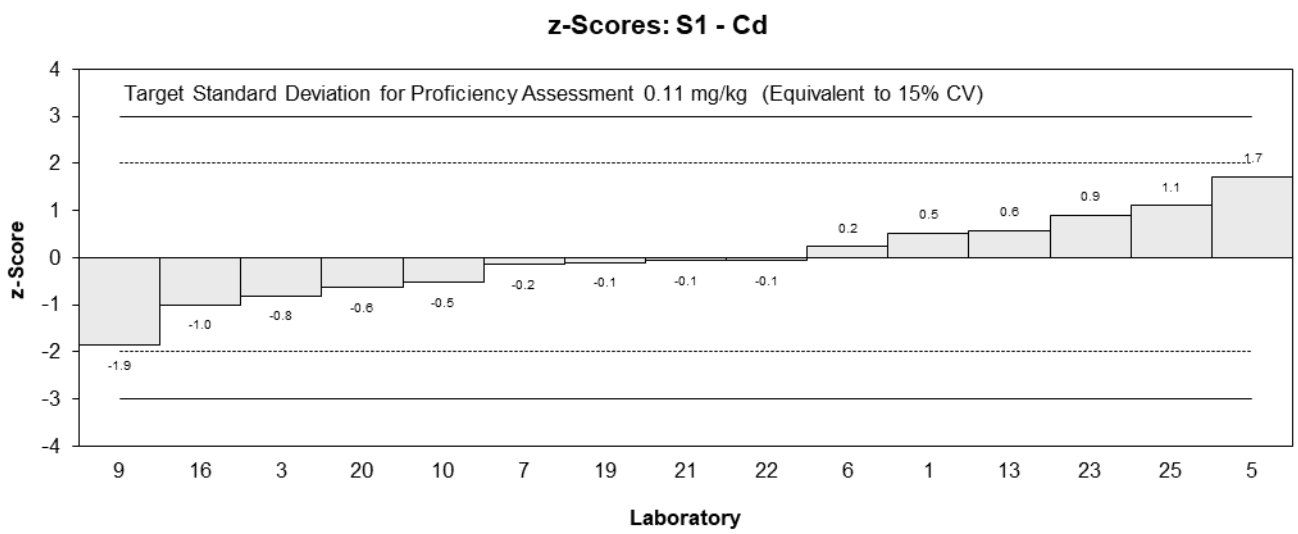
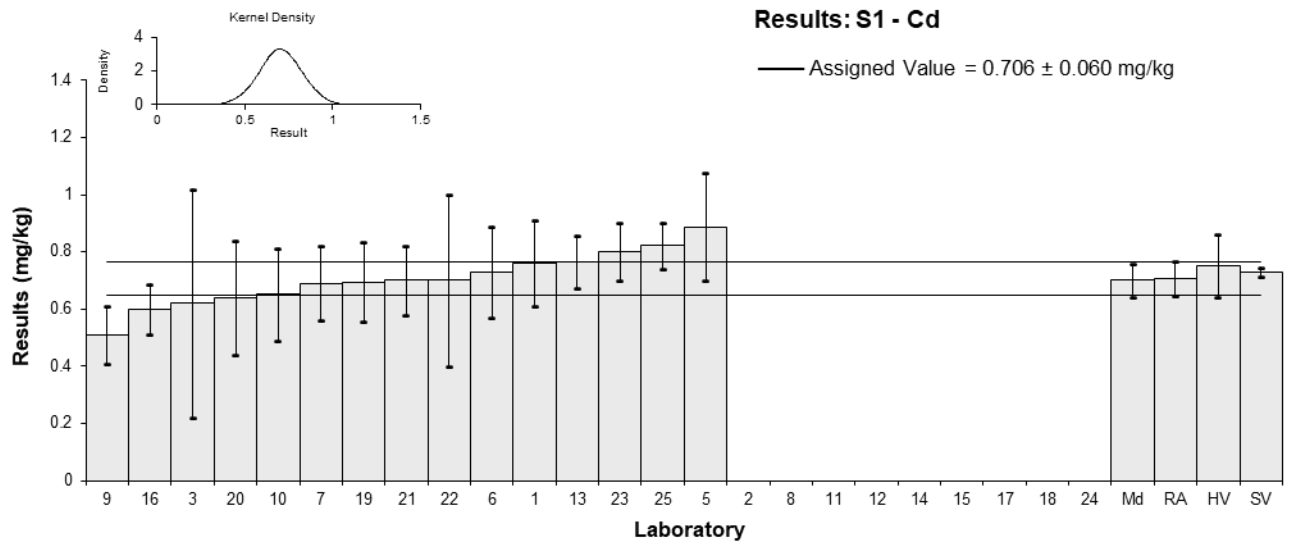


Figure 5

Table 17

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	28	5.6	0.00	0.00
2	NT	NT		
3	27	5	-0.36	-0.20
5	24.211	3.66	-1.35	-1.01
6	28.1	5.6	0.04	0.02
7	28.50	6.01	0.18	0.08
8	30.7	5	0.96	0.53
9	18	4.5	-3.57	-2.18
10	28.8	7.20	0.29	0.11
11	NT	NT		
12	32	3.84	1.43	1.01
13	26.44563	3.2	-0.56	-0.47
14	29.4	4.3	0.50	0.32
15	NT	NT		
16	29	3.451	0.36	0.28
17	27.2	2.7	-0.29	-0.28
18	29.9	7.48	0.68	0.25
19	27.4	5.49	-0.21	-0.11
20	27	6	-0.36	-0.16
21	28.3	4.6	0.11	0.06
22	27	5	-0.36	-0.20
23	28.7	3.0	0.25	0.22
24	NT	NT		
25	27.42554	2.74	-0.21	-0.20

Statistics

Assigned Value	28.0	0.9
Spike Value	28.0	0.9
Homogeneity Value	27.0	3.2
Robust Average	28.0	0.9
Median	28.1	0.8
Mean	27.7	
N	20	
Max	32	
Min	18	
Robust SD	1.6	
Robust CV	5.7%	

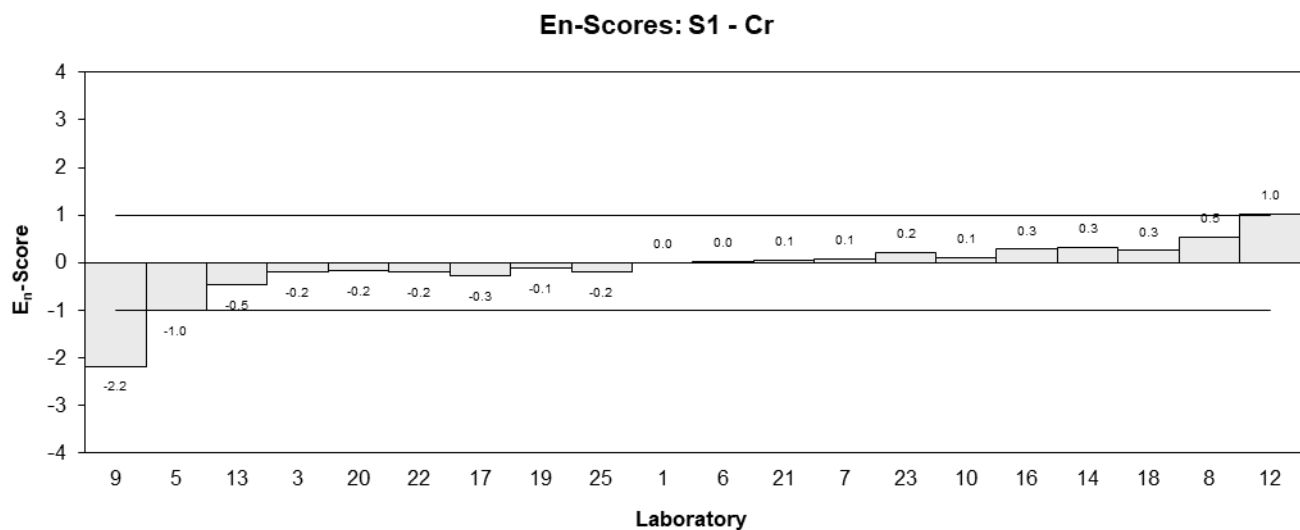
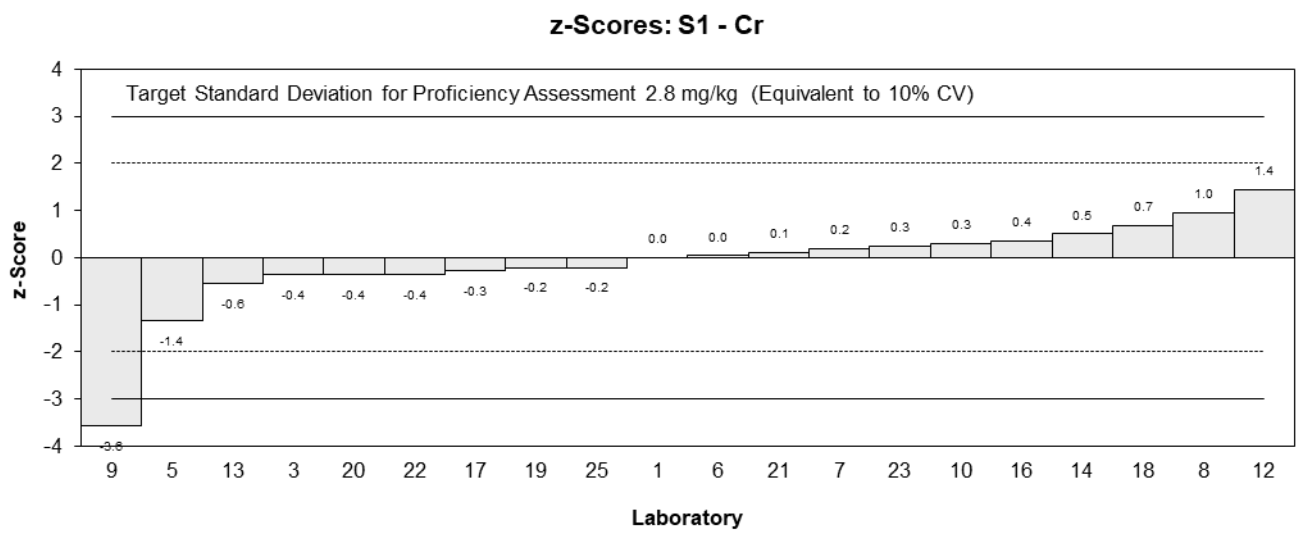
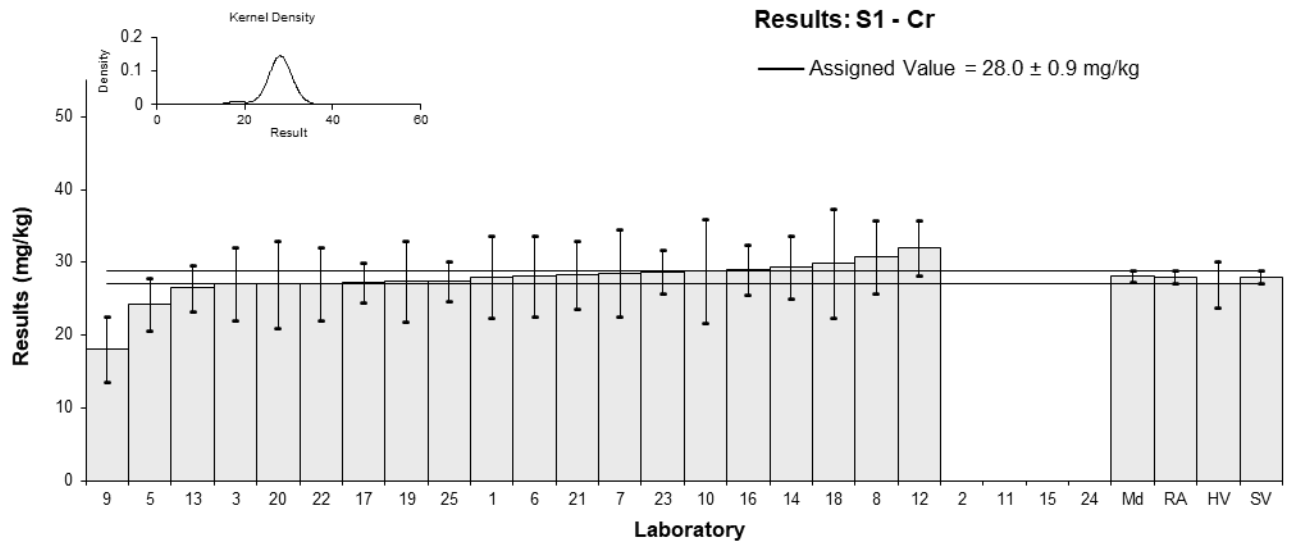


Figure 6

Table 18

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	29	5.8	0.47	0.22
2	NT	NT		
3	26	5	-0.61	-0.33
5	27.700	3.92	0.00	0.00
6	28.0	5.9	0.11	0.05
7	28.41	6.12	0.26	0.11
8	29.2	5	0.54	0.29
9	25	6.3	-0.97	-0.42
10	30.2	7.55	0.90	0.33
11	NT	NT		
12	32	1.28	1.55	2.55
13	24.77887	3.7	-1.05	-0.76
14	28.8	4.6	0.40	0.23
15	NT	NT		
16	26	3.25	-0.61	-0.50
17	28.2	2.8	0.18	0.17
18	26.2	6.55	-0.54	-0.23
19	26.1	5.23	-0.58	-0.30
20	28	6	0.11	0.05
21	29.3	4.2	0.58	0.37
22	26	5	-0.61	-0.33
23	29.1	3.0	0.51	0.44
24	NT	NT		
25	27.53416	2.75	-0.06	-0.06

Statistics

Assigned Value	27.7	1.1
Spike Value	29.6	0.6
Homogeneity Value	28.0	3.4
Robust Average	27.7	1.1
Median	28.0	1.0
Mean	27.8	
N	20	
Max	32	
Min	24.77887	
Robust SD	1.9	
Robust CV	6.9%	

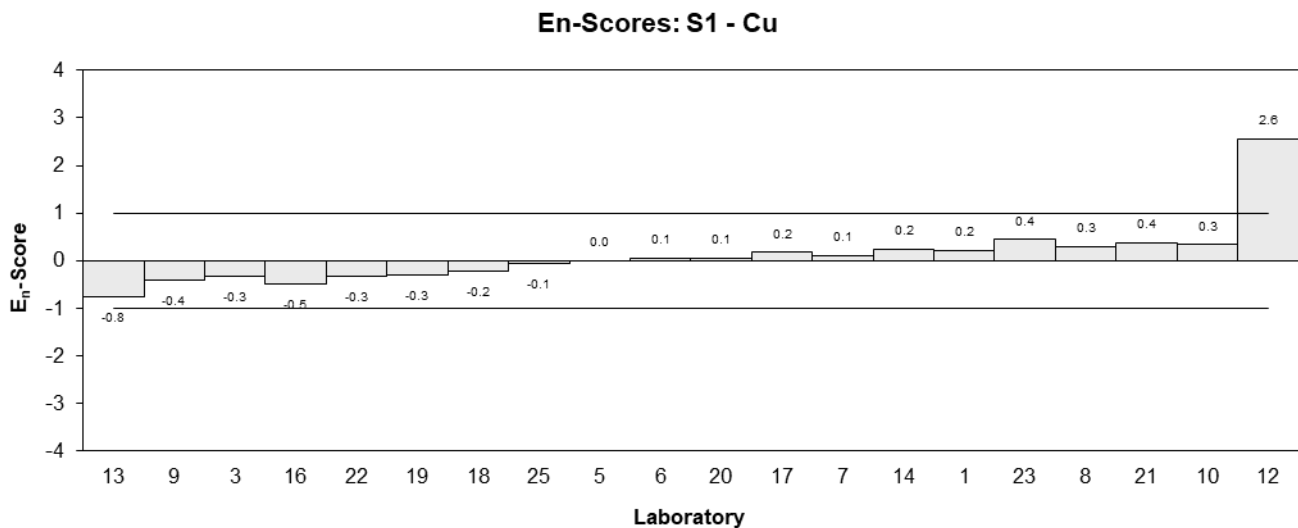
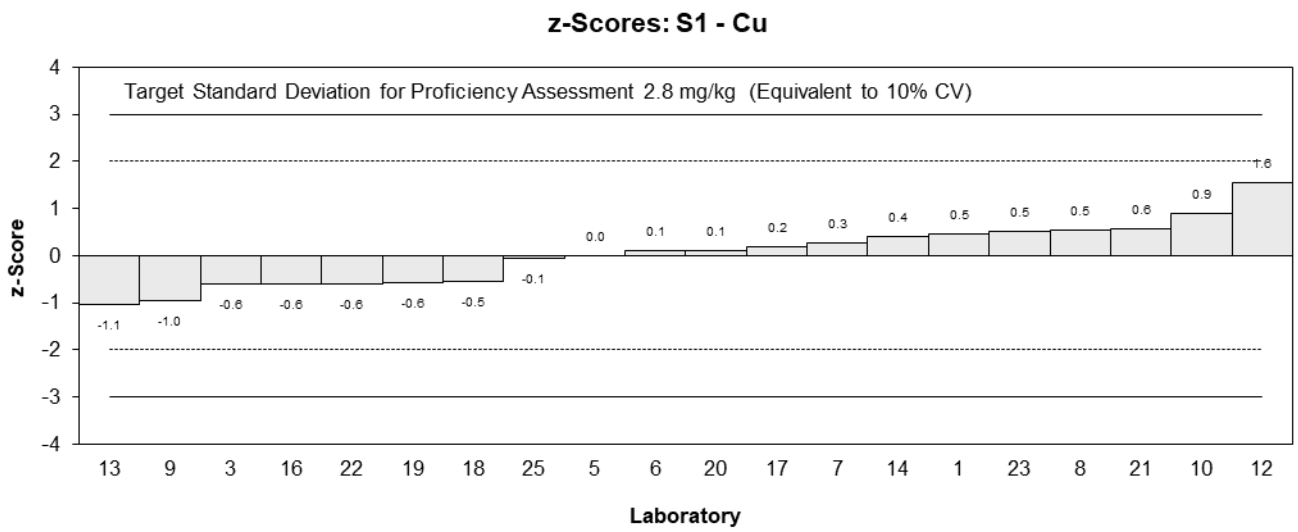
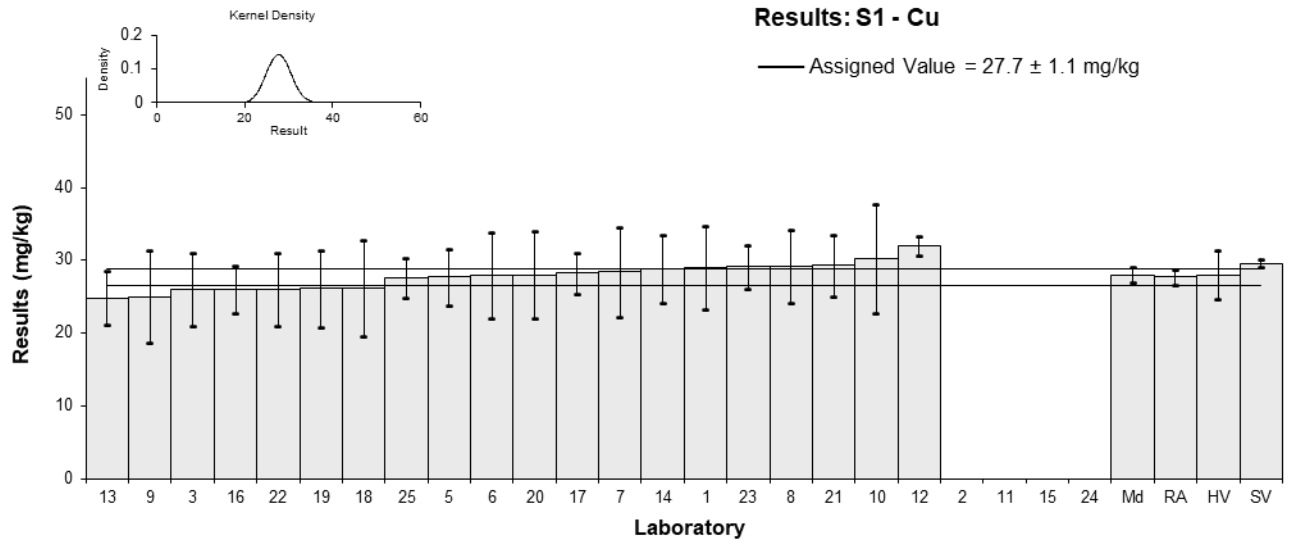


Figure 7

Table 19

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	0.36	0.072	-0.02	-0.01
2	NT	NT		
3	0.38	0.2	0.35	0.09
5	0.352	0.0921	-0.17	-0.09
6	0.31	0.05	-0.94	-0.93
7	0.37	0.07	0.17	0.12
8	0.45	0.1	1.64	0.87
9	0.37	0.09	0.17	0.10
10	0.329	0.0823	-0.59	-0.37
11	NT	NT		
12	0.4	0.04	0.72	0.85
13	0.33635	0.06	-0.46	-0.38
14	0.31	0.04	-0.94	-1.11
15	NT	NT		
16	0.37	0.06216	0.17	0.14
17	<1	NR		
18	0.338	0.058	-0.42	-0.37
19	0.314	0.0785	-0.87	-0.57
20	0.35	0.1	-0.20	-0.11
21	0.338	0.084	-0.42	-0.26
22	0.4	0.2	0.72	0.19
23	0.39	0.06	0.54	0.45
24	NT	NT		
25	0.41947	0.04	1.08	1.27

Statistics

Assigned Value	0.361	0.023
Spike Value	0.725	0.015
Homogeneity Value	0.333	0.040
Robust Average	0.361	0.023
Median	0.360	0.020
Mean	0.362	
N	19	
Max	0.45	
Min	0.31	
Robust SD	0.039	
Robust CV	11%	

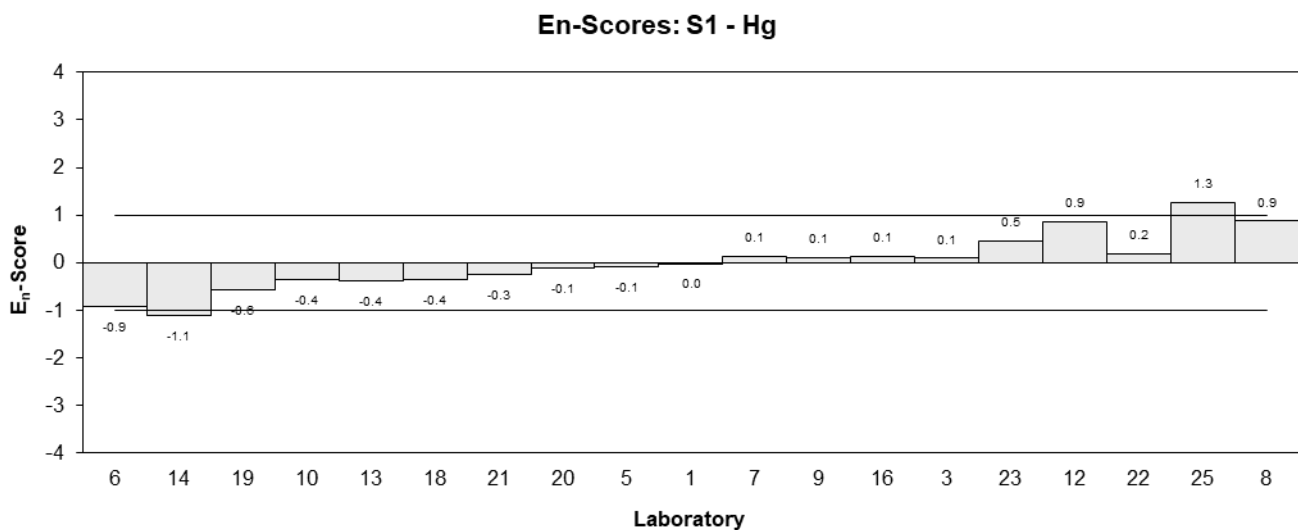
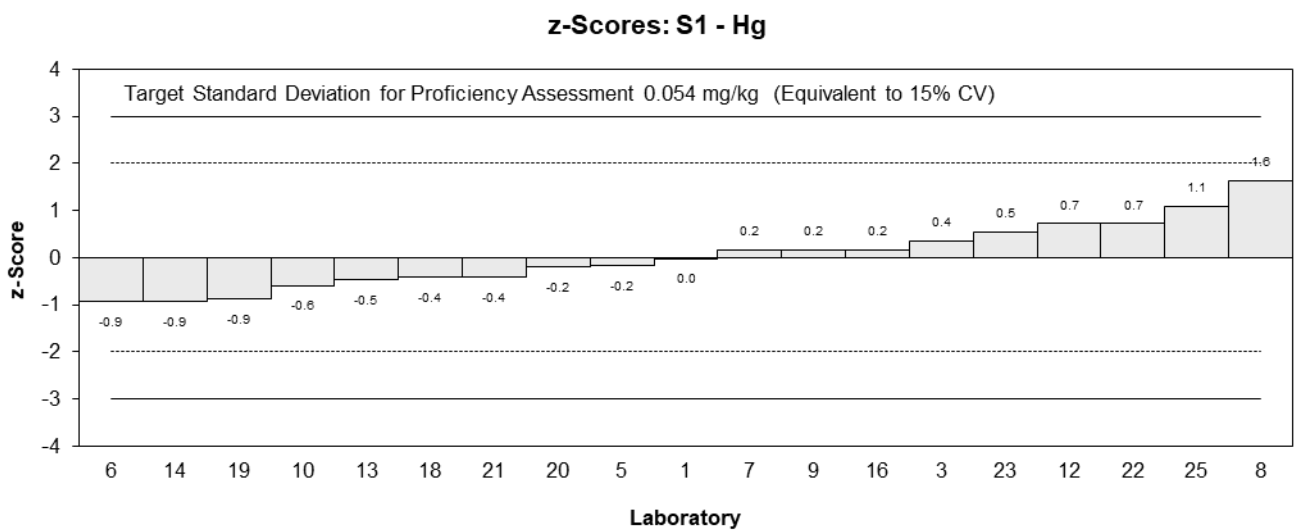
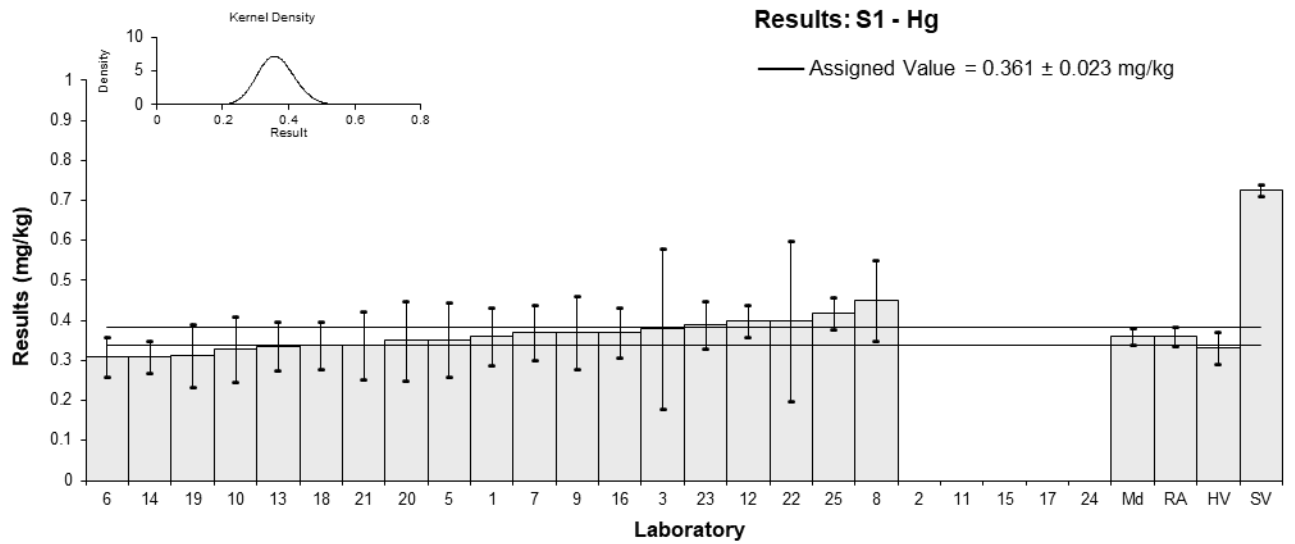


Figure 8

Table 20

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	3.3	0.66	0.36	0.24
2	NT	NT		
3	3.4	2	0.58	0.13
5	<20	NR		
6	NR	NR		
7	< 5	NR		
8	3.9	1.5	1.64	0.51
9	NT	NT		
10*	1.47	0.368	-3.54	-3.83
11	NT	NT		
12*	4.75	0.285	3.45	4.42
13	<10	NR		
14	3.15	1.0	0.04	0.02
15	NT	NT		
16	NT	NT		
17	3.06	0.31	-0.15	-0.18
18	NT	NT		
19	<5	NR		
20	3	0.9	-0.28	-0.14
21	2.41	0.4	-1.53	-1.56
22	3	2	-0.28	-0.06
23	3.26	0.5	0.28	0.24
24	NT	NT		
25	2.90415	0.29	-0.48	-0.61

* Outlier, see Section 4.2

Statistics

Assigned Value	3.13	0.23
Spike Value	Not Spiked	
Robust Average	3.14	0.41
Median	3.11	0.21
Mean	3.13	
N	12	
Max	4.75	
Min	1.47	
Robust SD	0.57	
Robust CV	18%	

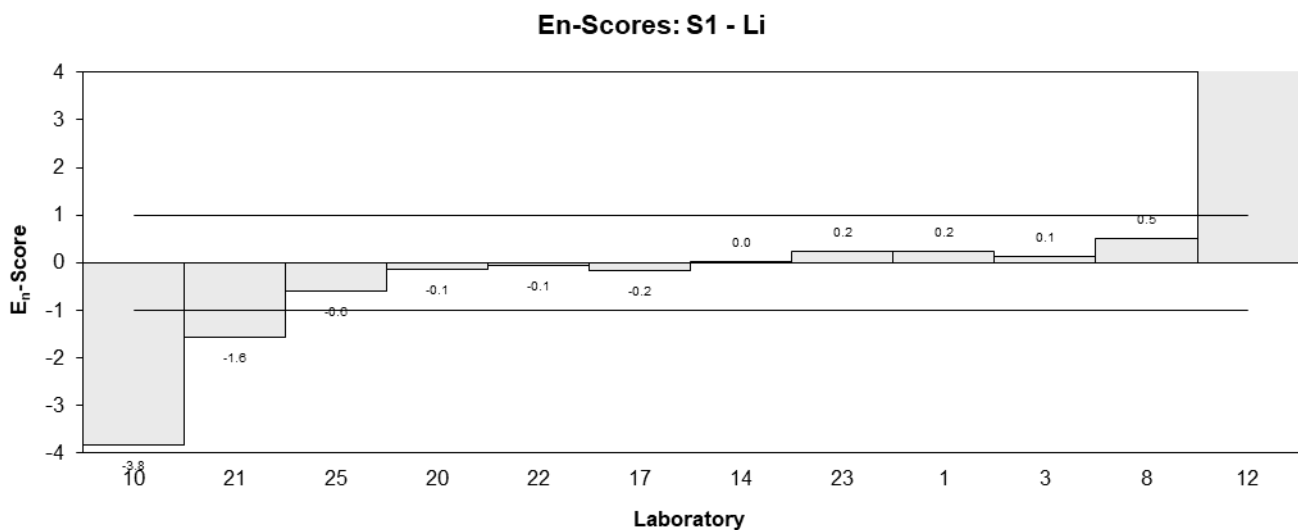
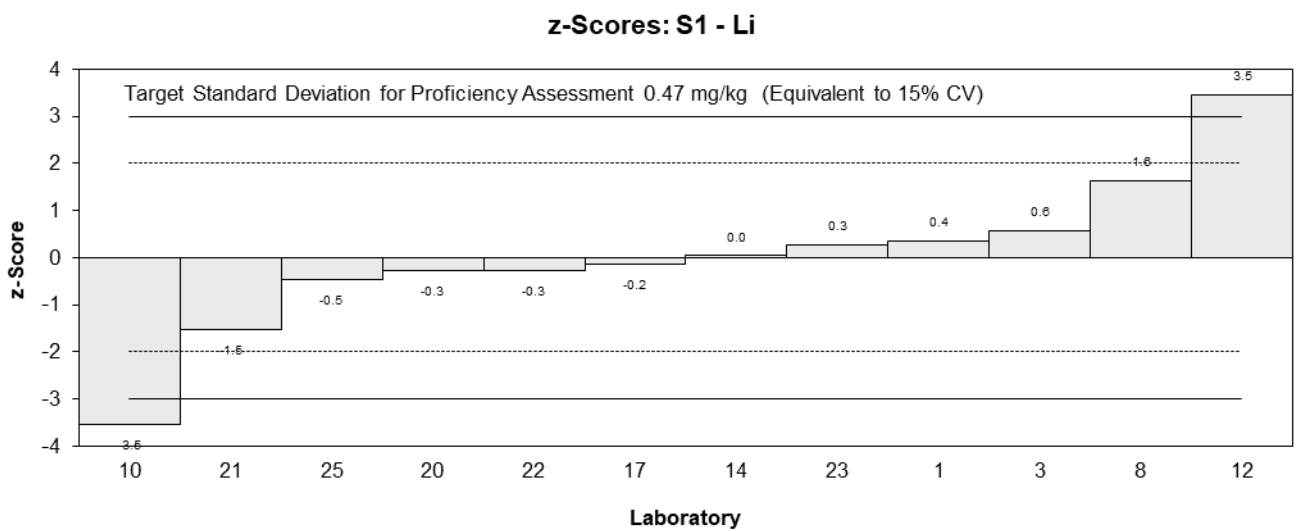
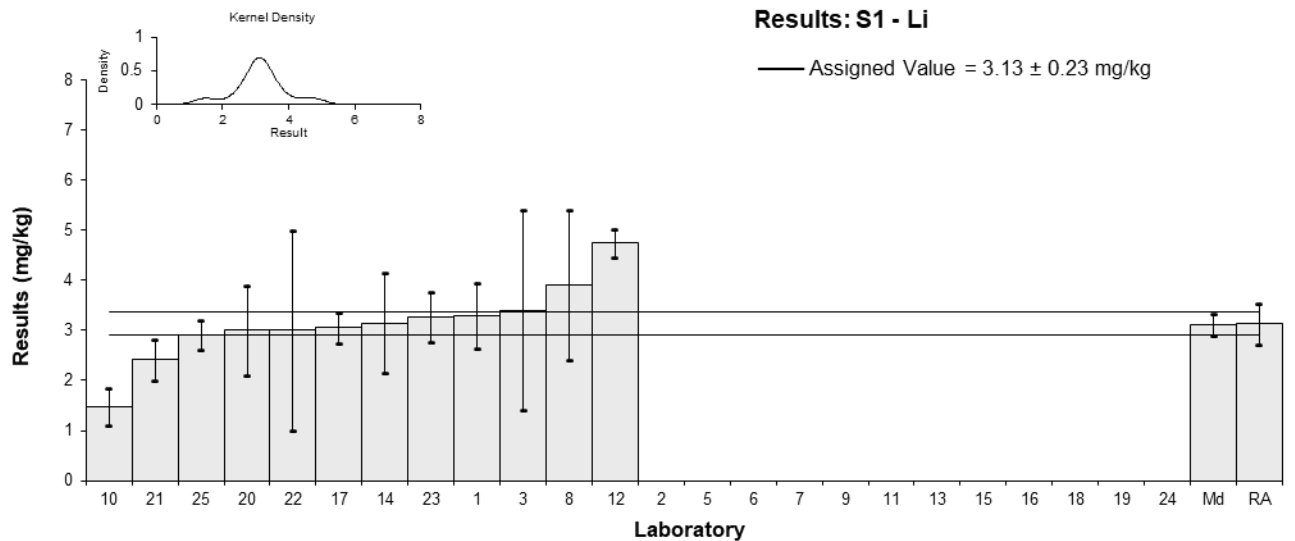


Figure 9

Table 21

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	370	74	0.39	0.19
2	NT	NT		
3	340	60	-0.45	-0.26
5	375.100	39.8	0.54	0.45
6	332	67	-0.67	-0.35
7	364.05	86.00	0.23	0.09
8	413	60	1.60	0.93
9	NT	NT		
10	345	86.3	-0.31	-0.13
11	NT	NT		
12	380	7.6	0.67	1.51
13	348.02197	38.28	-0.22	-0.20
14	368	25	0.34	0.42
15	NT	NT		
16	370	41.44	0.39	0.32
17	334	33.4	-0.62	-0.61
18	353	88.3	-0.08	-0.03
19	328	65.6	-0.79	-0.42
20	300	60	-1.57	-0.91
21	355	36	-0.03	-0.03
22	340	50	-0.45	-0.31
23	365	35	0.25	0.24
24	NT	NT		
25	394.97768	3.95	1.09	2.68

Statistics

Assigned Value	356	14
Spike Value	Not Spiked	
Homogeneity Value	349	42
Robust Average	356	14
Median	355	13
Mean	357	
N	19	
Max	413	
Min	300	
Robust SD	24	
Robust CV	6.7%	

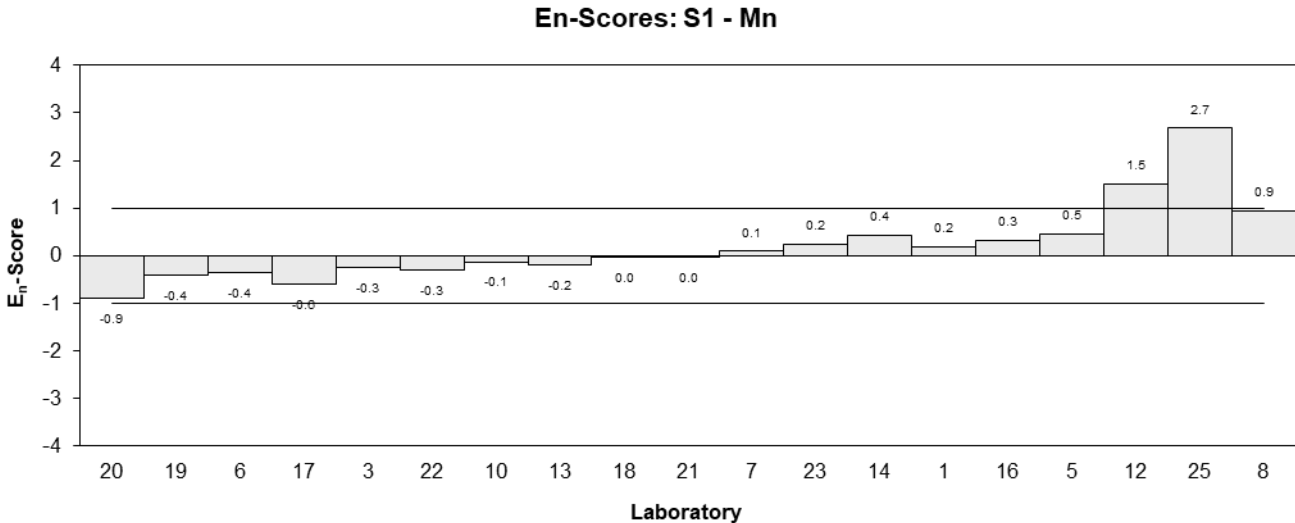
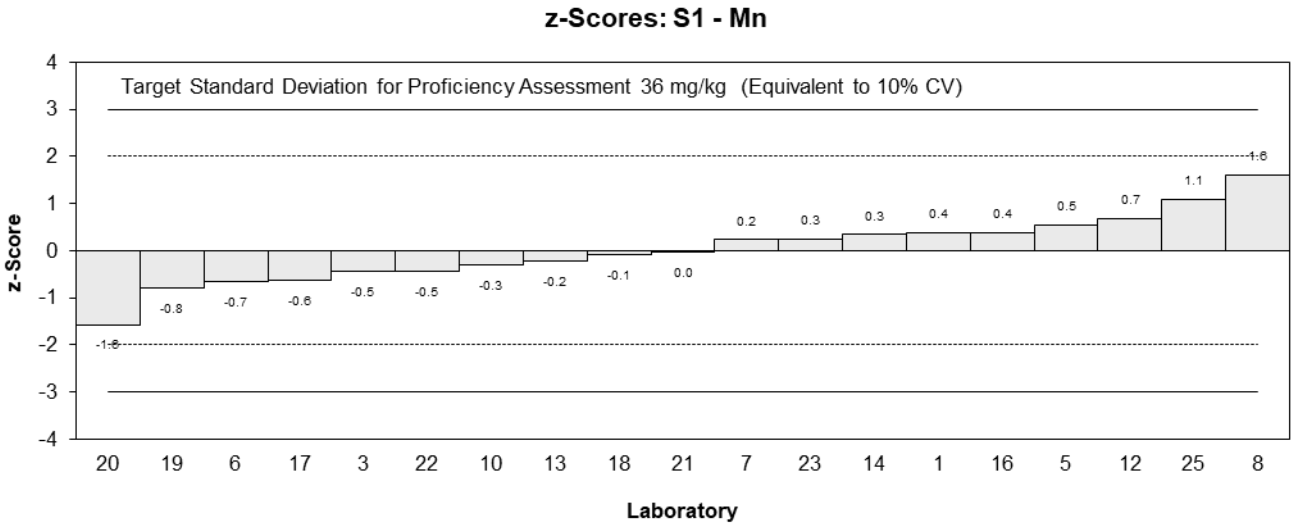
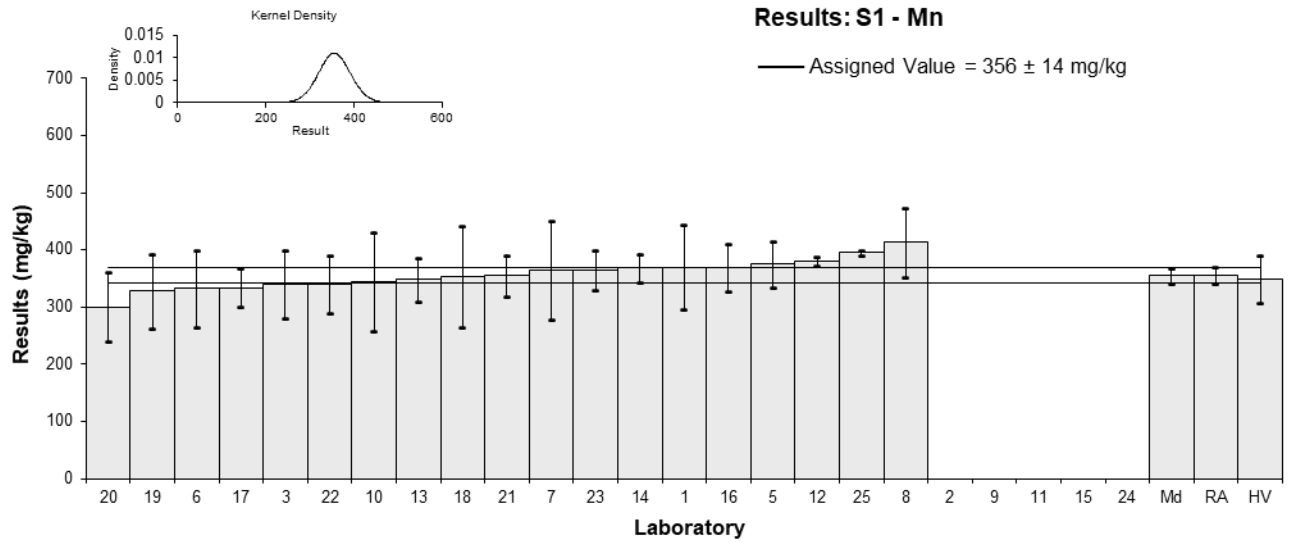


Figure 10

Table 22

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	13	2.6	-0.30	-0.15
2	NT	NT		
3	14	3	0.45	0.20
5	12.482	2.82	-0.69	-0.32
6	12.5	2.8	-0.67	-0.31
7	14.91	2.90	1.13	0.51
8	12.6	4	-0.60	-0.20
9	NT	NT		
10	13.4	3.35	0.00	0.00
11	NT	NT		
12	14	0.98	0.45	0.52
13	12.74716	1.66	-0.49	-0.37
14	13.8	2.5	0.30	0.16
15	NT	NT		
16	13	1.677	-0.30	-0.22
17	13.7	1.4	0.22	0.20
18	16.2	4.1	2.09	0.68
19	13.5	2.70	0.07	0.04
20	11	2	-1.79	-1.15
21	12.6	2.3	-0.60	-0.34
22	13	4	-0.30	-0.10
23	14.3	1.5	0.67	0.56
24	NT	NT		
25	14.60021	1.46	0.90	0.76

Statistics

Assigned Value	13.4	0.6
Spike Value	15.0	1.5
Homogeneity Value	12.0	1.4
Robust Average	13.4	0.6
Median	13.4	0.6
Mean	13.4	
N	19	
Max	16.2	
Min	11	
Robust SD	0.99	
Robust CV	7.4%	

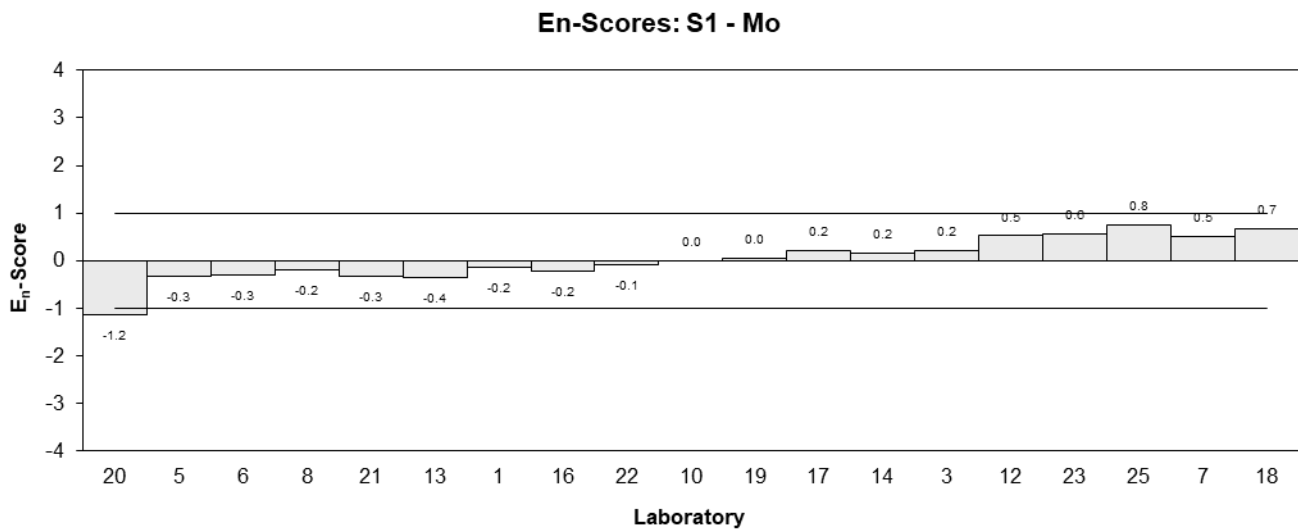
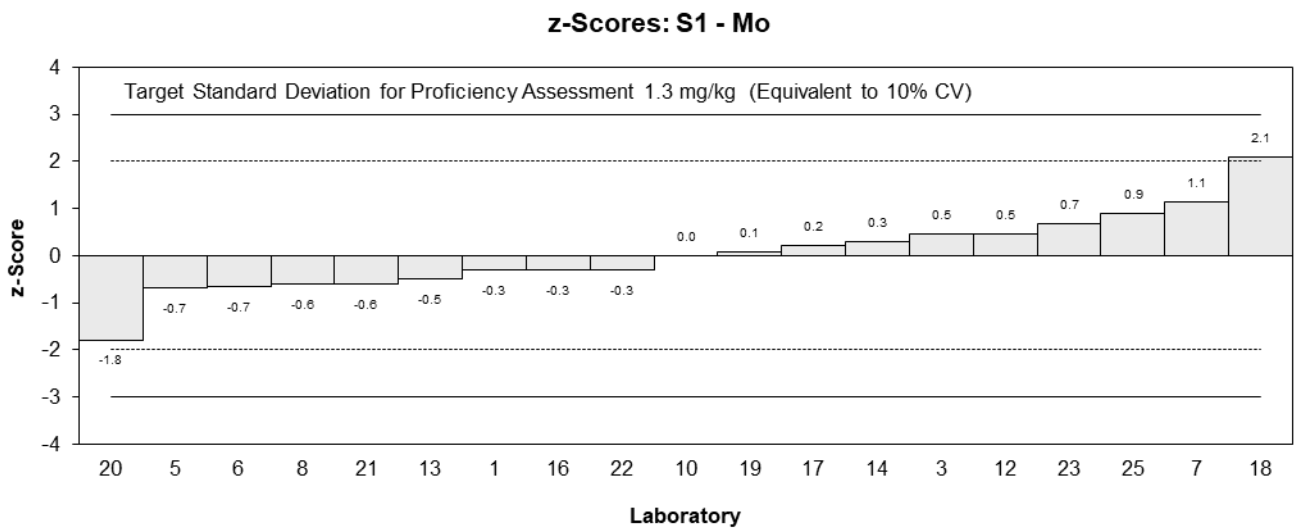
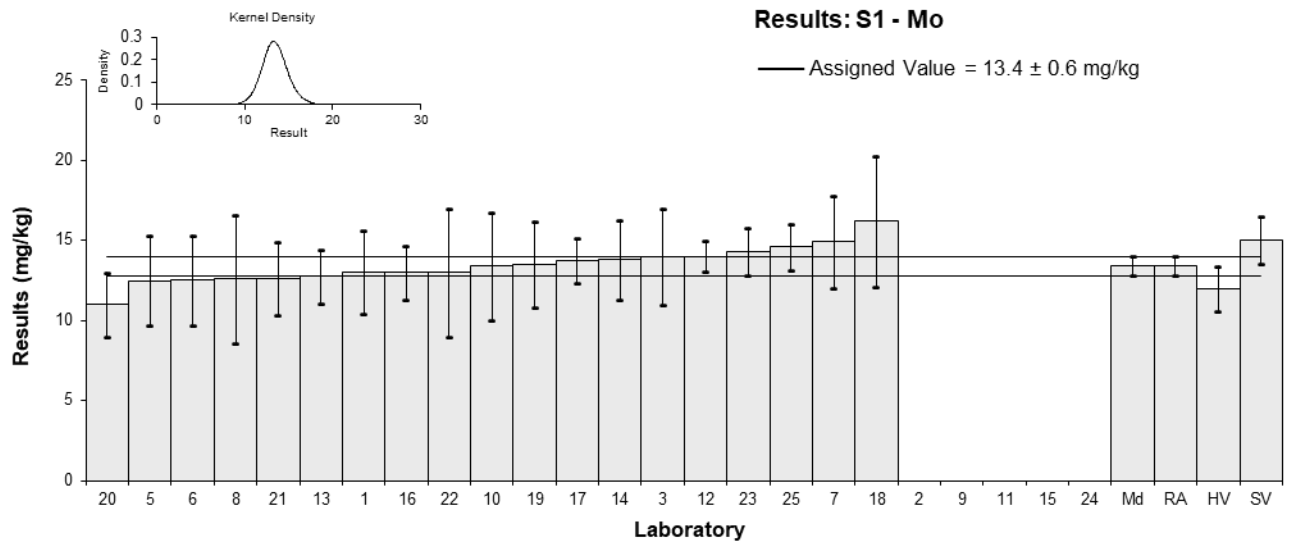


Figure 11

Table 23

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	19	3.8	0.67	0.31
2	NT	NT		
3	18	4	0.11	0.05
5	17.521	2.92	-0.16	-0.09
6	17.5	3.7	-0.17	-0.08
7	19.26	3.89	0.82	0.37
8	17.8	7	0.00	0.00
9	12	3.7	-3.26	-1.55
10	17.4	4.35	-0.22	-0.09
11	NT	NT		
12	22	1.54	2.36	2.54
13	17.48060	1.92	-0.18	-0.16
14	19.2	3.3	0.79	0.42
15	NT	NT		
16	19	2.052	0.67	0.56
17	16.6	1.7	-0.67	-0.67
18	17.9	4.48	0.06	0.02
19	17.4	3.49	-0.22	-0.11
20	16	4	-1.01	-0.45
21	17.1	2.6	-0.39	-0.26
22	18	4	0.11	0.05
23	18.6	2.0	0.45	0.38
24	NT	NT		
25	17.31943	1.73	-0.27	-0.26

Statistics

Assigned Value	17.8	0.6
Spike Value	18.1	0.6
Homogeneity Value	18.3	2.2
Robust Average	17.8	0.6
Median	17.7	0.4
Mean	17.8	
N	20	
Max	22	
Min	12	
Robust SD	1.1	
Robust CV	6.3%	

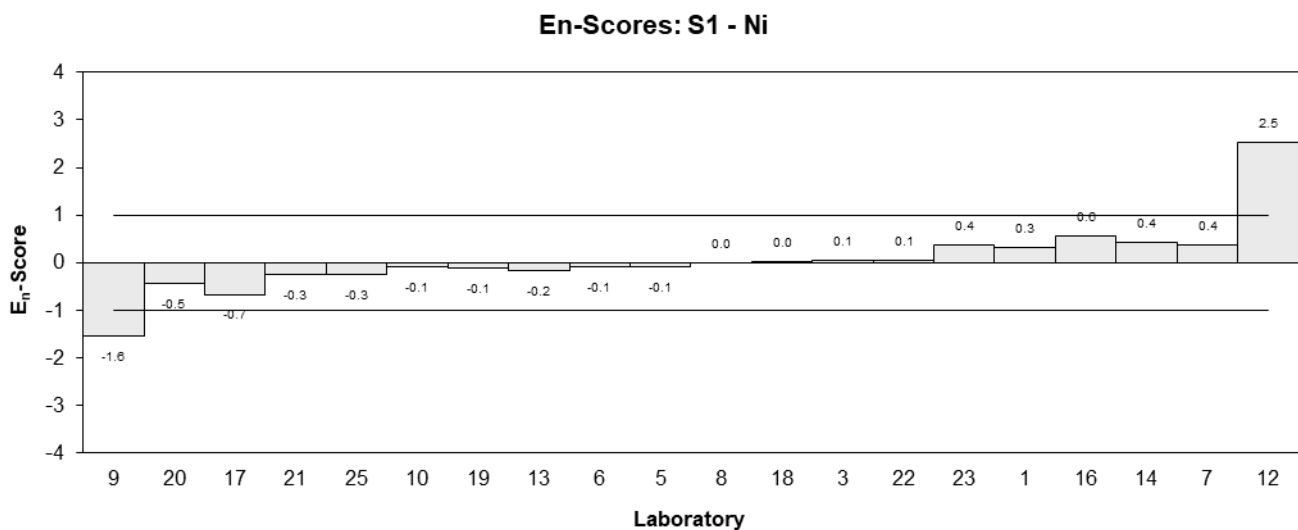
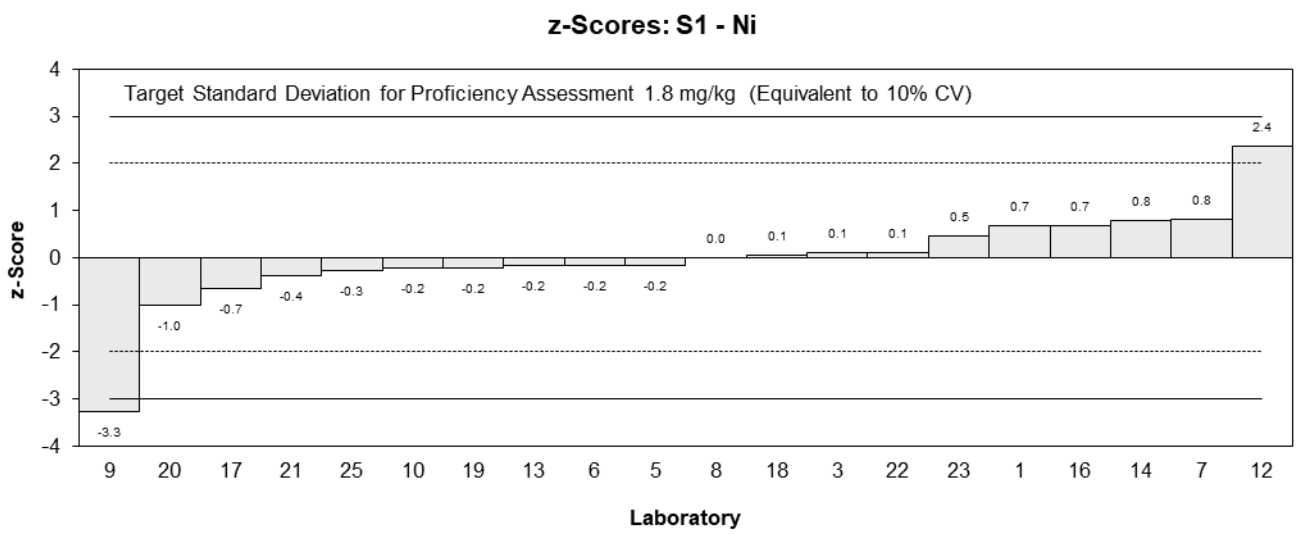
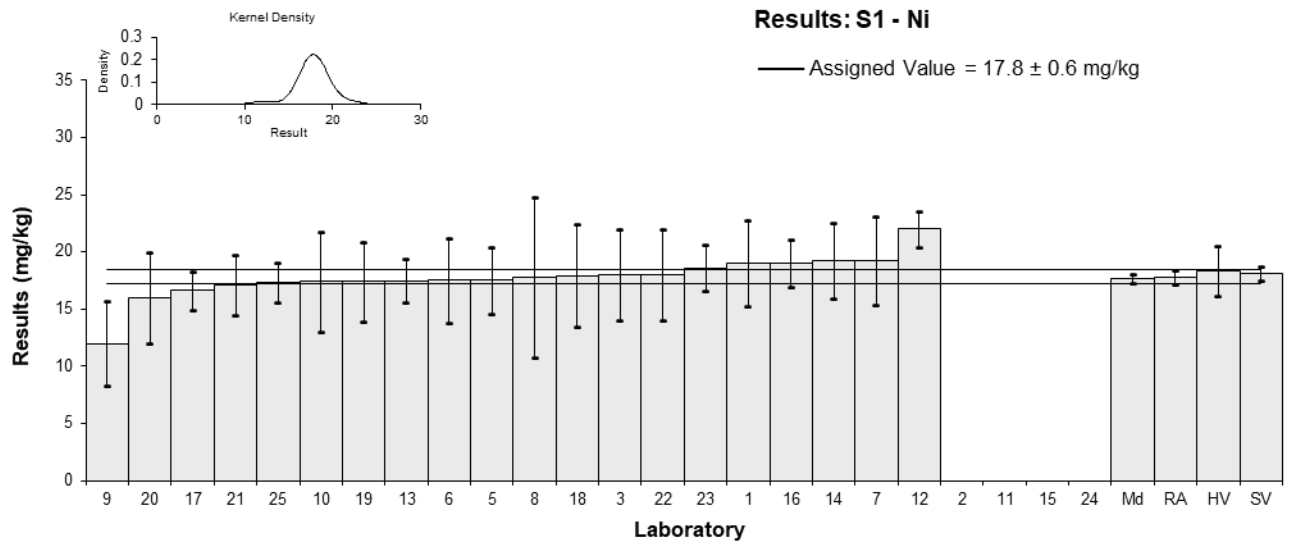


Figure 12

Table 24

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	21	4.2	-0.09	-0.05
2	NT	NT		
3	22	4	0.38	0.19
5	21.418	3.92	0.10	0.05
6	21.4	4.3	0.09	0.05
7	21.77	4.66	0.27	0.12
8	22.1	5	0.42	0.18
9	16	3.0	-2.45	-1.63
10	24.4	6.10	1.51	0.52
11	NT	NT		
12	23	1.15	0.85	1.13
13	21.00991	2.73	-0.09	-0.06
14	21.8	3.8	0.28	0.15
15	NT	NT		
16	22	2.992	0.38	0.25
17	16.3	1.6	-2.31	-2.52
18	18.5	4.63	-1.27	-0.57
19	20.5	4.10	-0.33	-0.16
20	18	5	-1.51	-0.63
21	21.4	3.3	0.09	0.06
22	21	4	-0.09	-0.05
23	23.5	2.5	1.08	0.84
24	NT	NT		
25	22.93368	2.29	0.82	0.68

Statistics

Assigned Value	21.2	1.1
Spike Value	22.6	0.5
Homogeneity Value	21.1	2.5
Robust Average	21.2	1.1
Median	21.4	0.5
Mean	21.0	
N	20	
Max	24.4	
Min	16	
Robust SD	1.9	
Robust CV	9.2%	

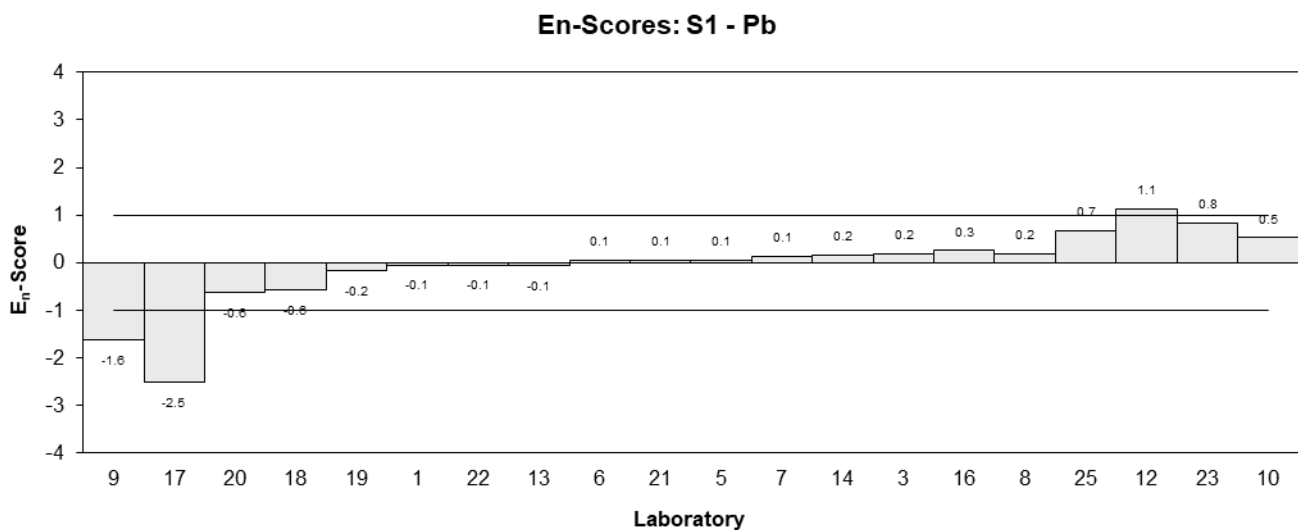
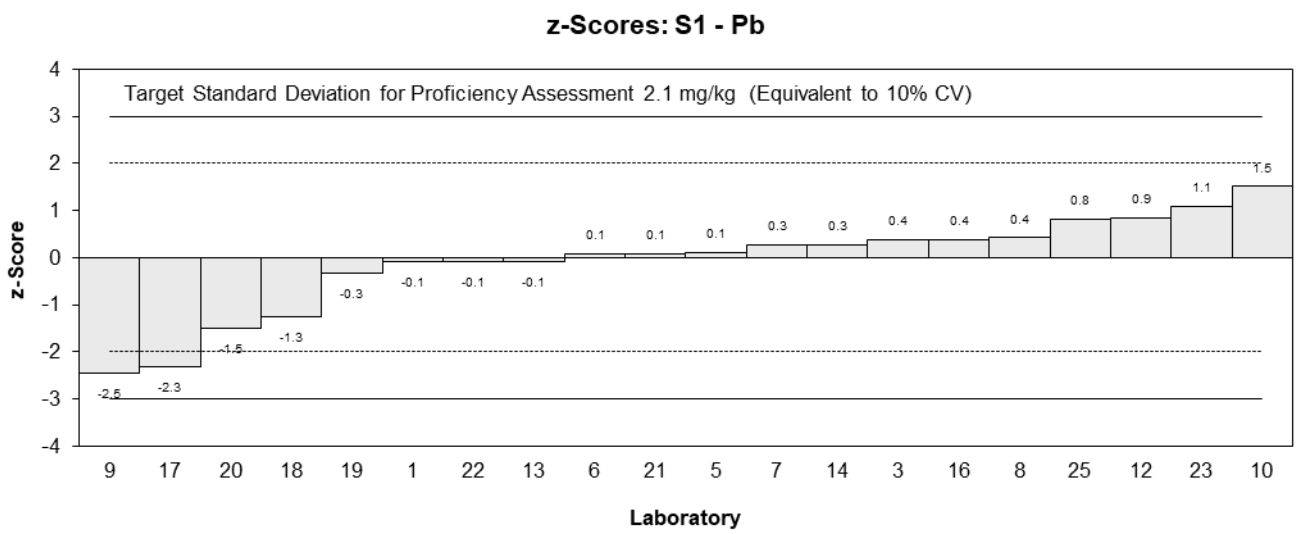
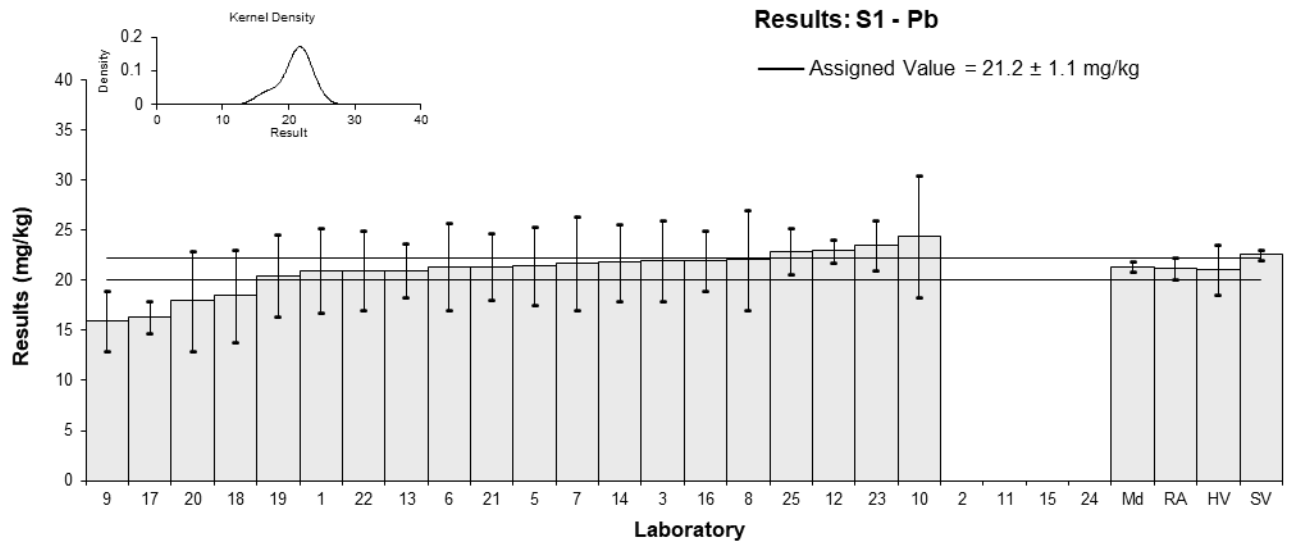


Figure 13

Table 25

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	7.6	1.5
2	NT	NT
3	7.8	2
5	NT	NT
6	NR	NR
7	NT	NT
8	NT	NT
9	NT	NT
10	NT	NT
11	NT	NT
12	NT	NT
13	NT	NT
14	NT	NT
15	NT	NT
16	NT	NT
17	NT	NT
18	NT	NT
19	NT	NT
20	5	1
21	5.82	0.64
22	6	1
23	NT	NT
24	NT	NT
25	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Median	6.0	1.7
Mean	6.4	
N	5	
Max	7.8	
Min	5	

Results: S1 - Rb

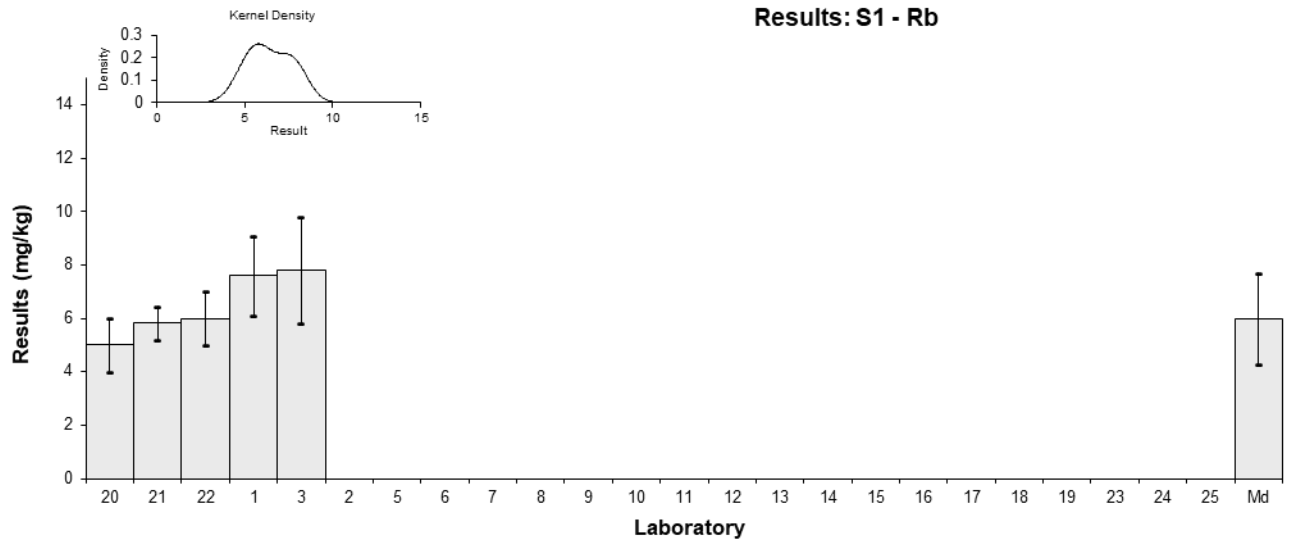


Figure 14

Table 26

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	4.3	0.86	-0.93	-0.88
2	NT	NT		
3	<7	NR		
5	<5	NR		
6	5.7	2.3	0.07	0.04
7	< 10	NR		
8	NT	NT		
9	NT	NT		
10	5.39	1.35	-0.15	-0.12
11	NT	NT		
12	4.5	0.135	-0.79	-0.91
13	<5	NR		
14	NT	NT		
15	NT	NT		
16	<3	NR		
17	NT	NT		
18	7.9	1.98	1.64	0.99
19	<10	NR		
20	<7	7		
21	4.2	0.8	-1.00	-0.97
22	<7	7		
23	6.44	0.9	0.60	0.56
24	NT	NT		
25	6.4295	0.64	0.59	0.61

Statistics

Assigned Value	5.6	1.2
Spike Value	8.00	0.40
Robust Average	5.6	1.2
Median	5.5	1.3
Mean	5.61	
N	8	
Max	7.9	
Min	4.2	
Robust SD	1.4	
Robust CV	25%	

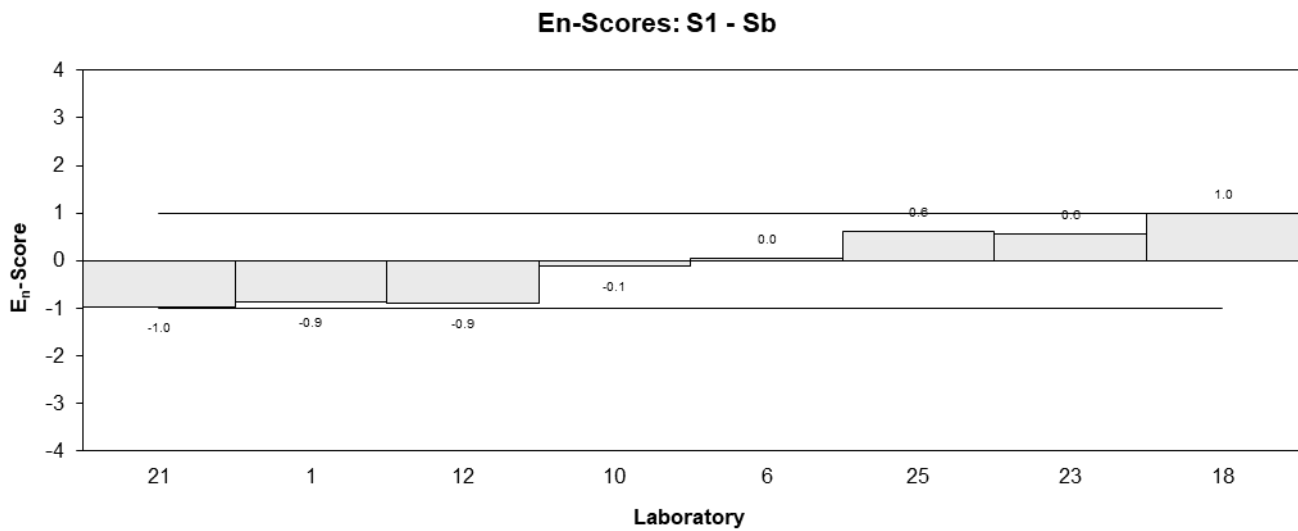
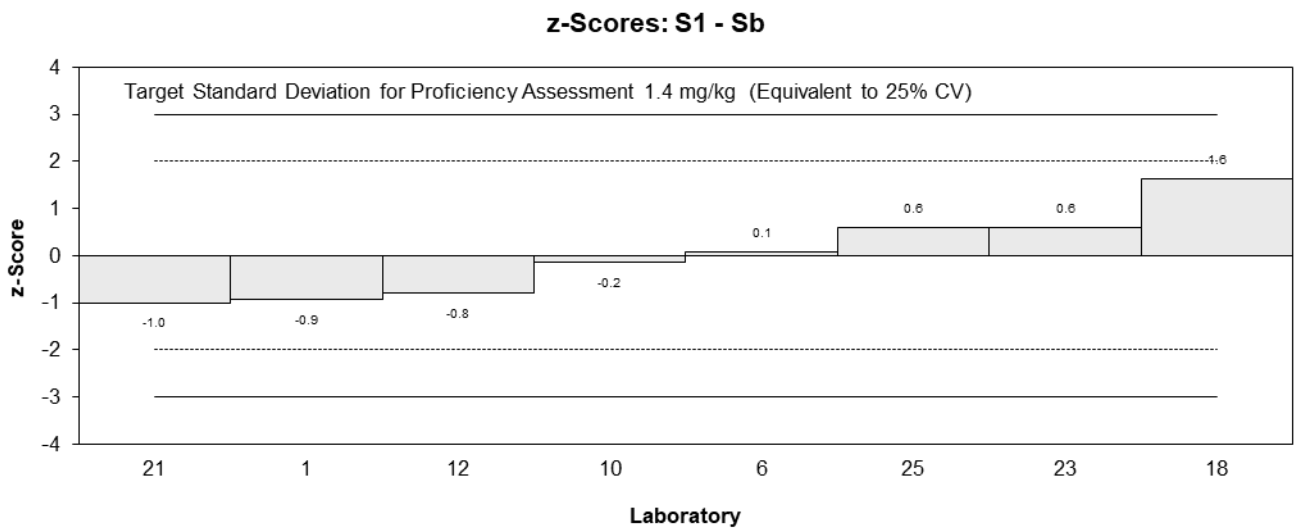
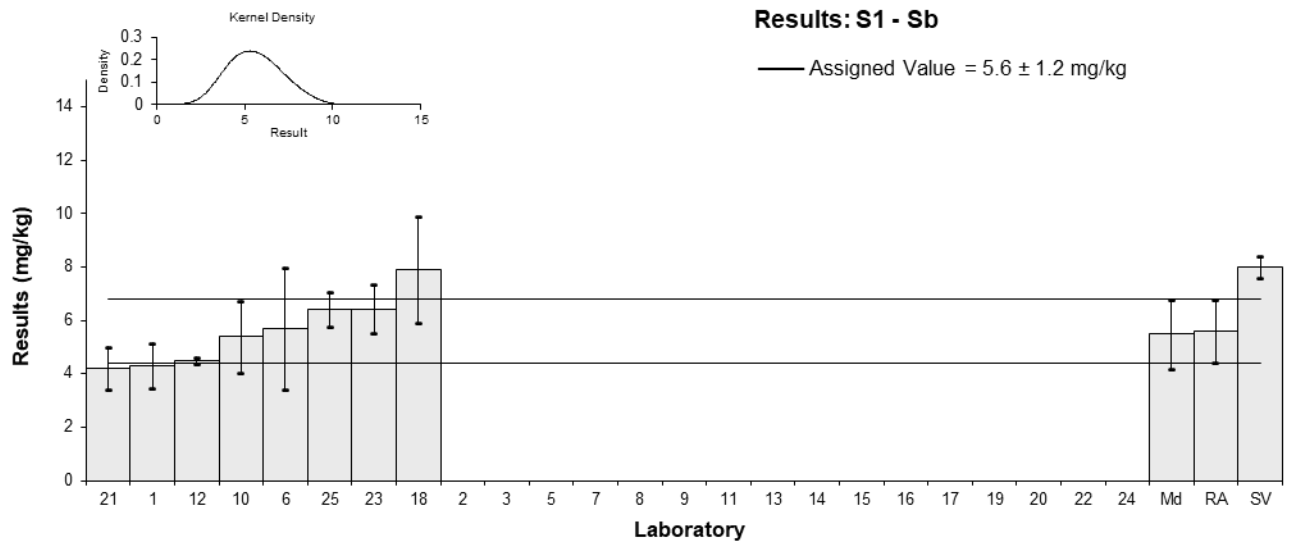


Figure 15

Table 27

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	2.6	0.52	0.18	0.14
2	NT	NT		
3	<2	NR		
5	3.955	0.932	2.88	1.45
6	2.6	0.9	0.18	0.09
7	2.47	0.46	-0.08	-0.07
8	NT	NT		
9	NT	NT		
10	2.35	0.588	-0.32	-0.23
11	NT	NT		
12	<5	0.15		
13	3.35684	0.57	1.69	1.26
14	<4	NR		
15	NT	NT		
16*	4	0.964	2.97	1.45
17	1.86	0.19	-1.29	-1.60
18	<5	NR		
19	2.49	0.498	-0.04	-0.03
20	<2	2		
21	<20	14		
22	2	2	-1.02	-0.25
23	2.45	0.4	-0.12	-0.11
24	NT	NT		
25	2.33416	0.23	-0.35	-0.41

* Outlier, see Section 4.2

Statistics

Assigned Value	2.51	0.36
Spike Value	2.71	0.06
Homogeneity Value	2.40	0.29
Robust Average	2.65	0.49
Median	2.48	0.15
Mean	2.71	
N	12	
Max	4	
Min	1.86	
Robust SD	0.68	
Robust CV	25%	

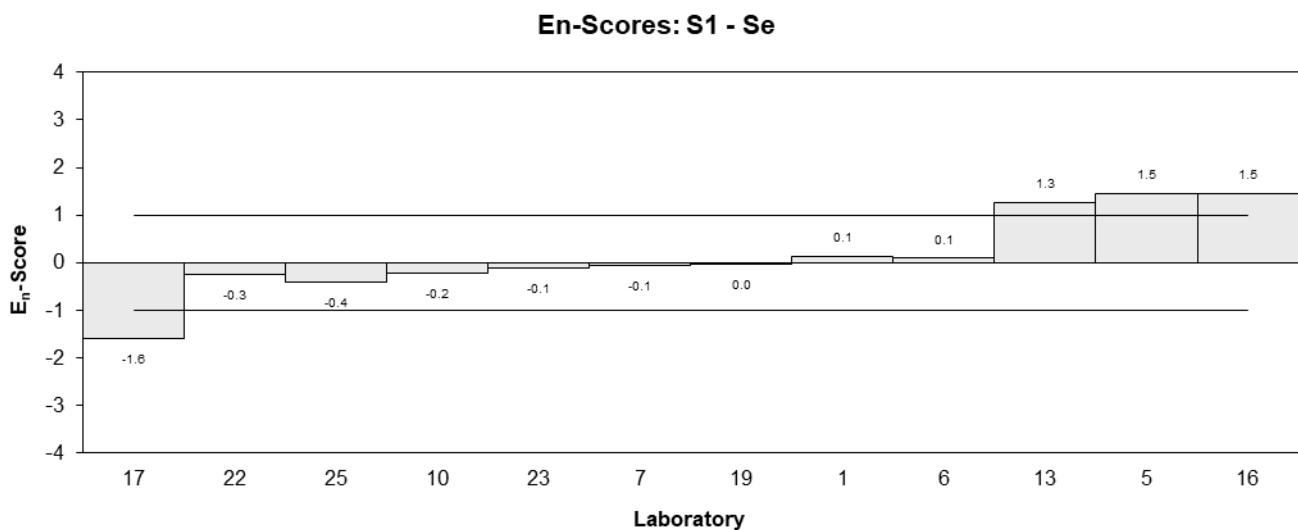
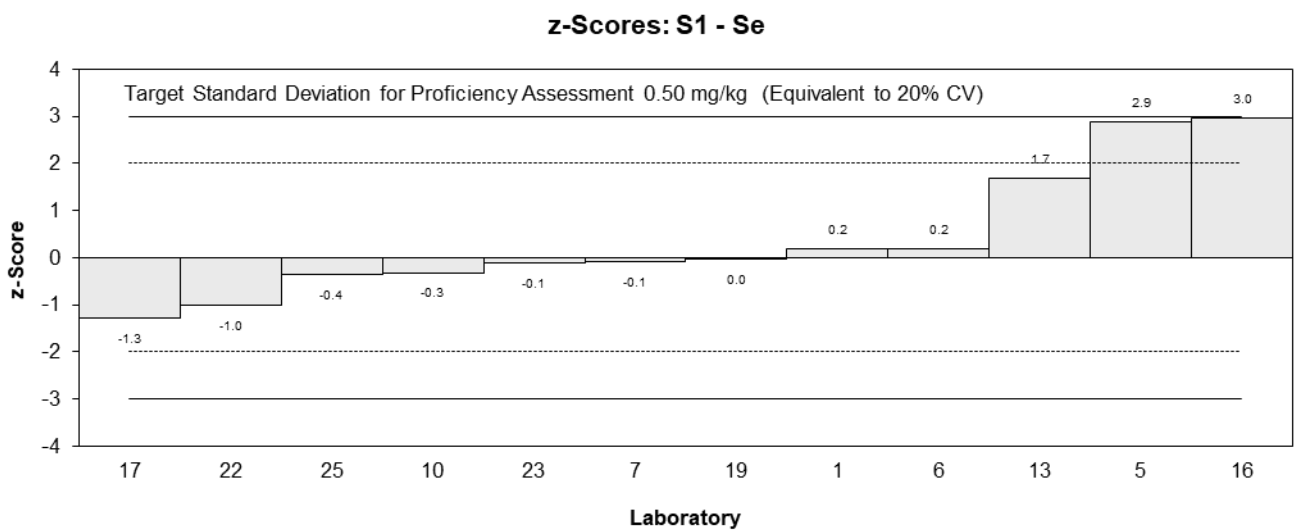
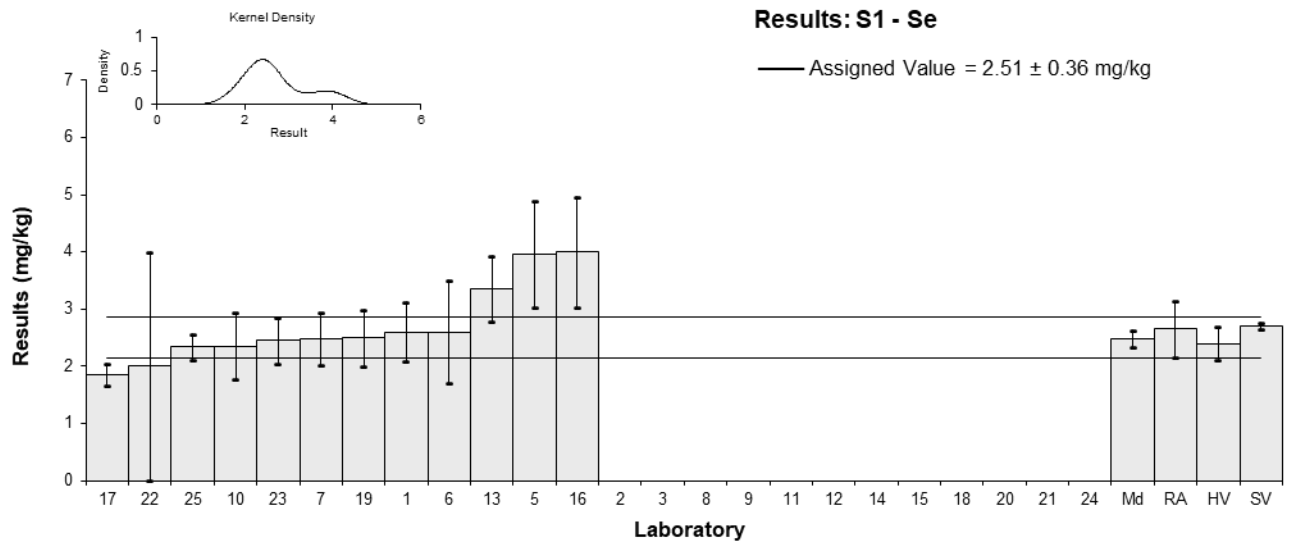


Figure 16

Table 28

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	13	2.6	0.44	0.29
2	NT	NT		
3	14	3	0.98	0.57
5	10.460	2.82	-0.95	-0.59
6	11.9	2.6	-0.16	-0.11
7	13.64	2.52	0.79	0.54
8	17.6	NR	2.95	6.00
9	NT	NT		
10	11.6	2.90	-0.33	-0.20
11	NT	NT		
12	13	0.26	0.44	0.85
13	11.60597	1.50	-0.32	-0.34
14	11.6	2.0	-0.33	-0.27
15	NT	NT		
16	10	1.73	-1.20	-1.13
17	14.3	1.4	1.15	1.26
18	9.9	2.48	-1.26	-0.87
19	12.4	2.49	0.11	0.08
20	11	3	-0.66	-0.38
21	12	2.5	-0.11	-0.08
22	12	4	-0.11	-0.05
23	NT	NT		
24	NT	NT		
25	12.3728	1.24	0.09	0.11

Statistics

Assigned Value	12.2	0.9
Spike Value	12.0	0.2
Homogeneity Value	11.8	1.4
Robust Average	12.2	0.9
Median	12.0	0.9
Mean	12.4	
N	18	
Max	17.6	
Min	9.9	
Robust SD	1.6	
Robust CV	13%	

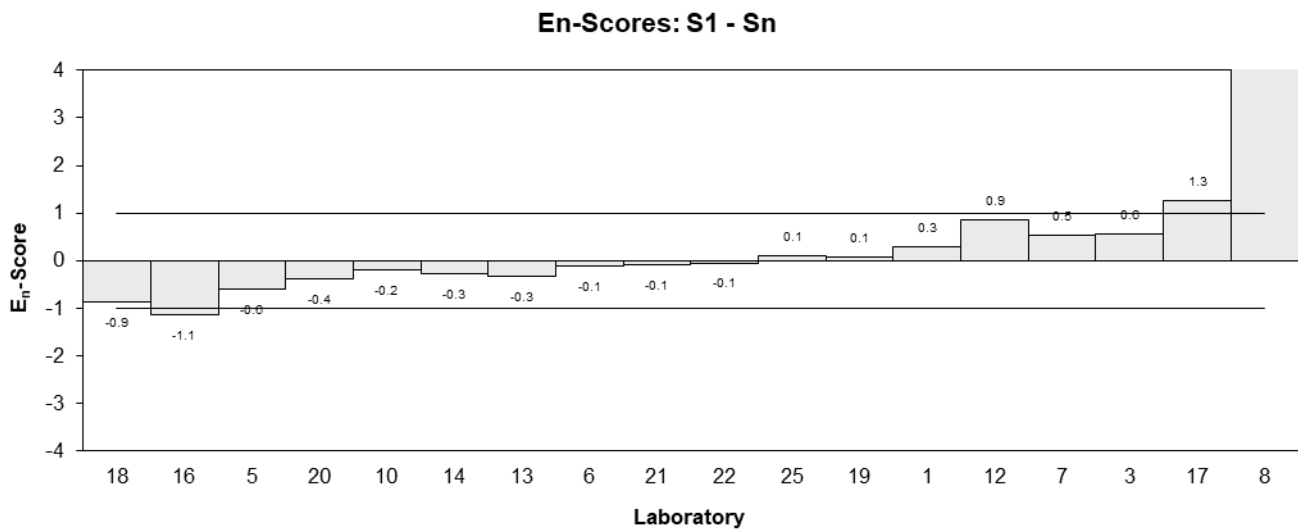
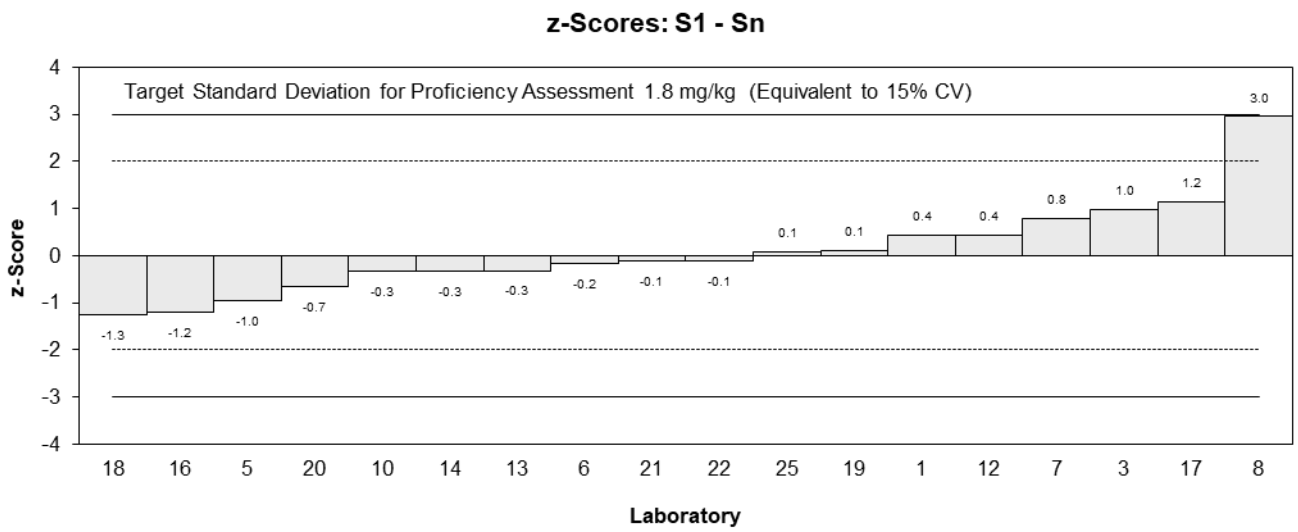
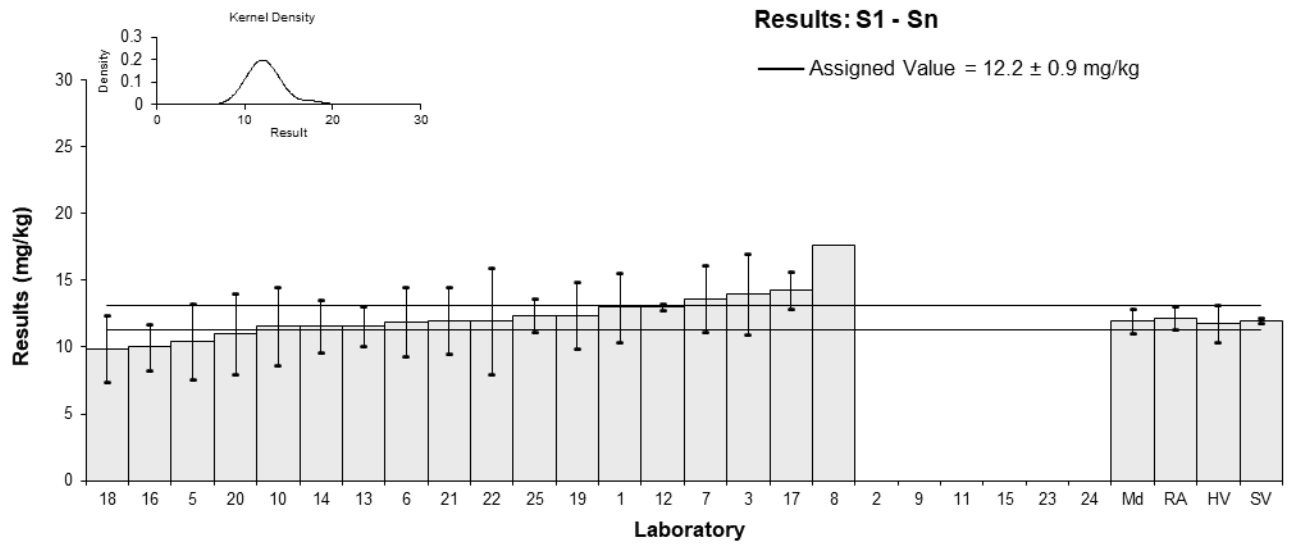


Figure 17

Table 29

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	1.9	0.38	0.70	0.41
2	NT	NT		
3	<2	NR		
5	1.560	0.392	-0.62	-0.36
6	1.7	0.5	-0.08	-0.04
7	< 10	NR		
8	<50	NR		
9	NT	NT		
10	1.46	0.365	-1.01	-0.61
11	NT	NT		
12	<5	0.3		
13	<5	NR		
14	<2	NR		
15	NT	NT		
16	NT	NT		
17	NT	NT		
18	<5	NR		
19	<10	NR		
20	<2	2		
21	1.7	0.25	-0.08	-0.06
22	<2	2		
23	2.06	0.3	1.32	0.91
24	NT	NT		
25	1.65296	0.17	-0.26	-0.24

Statistics

Assigned Value	1.72	0.22
Spike Value	2.00	0.04
Homogeneity Value	1.80	0.22
Robust Average	1.72	0.22
Median	1.70	0.20
Mean	1.72	
N	7	
Max	2.06	
Min	1.46	
Robust SD	0.23	
Robust CV	13%	

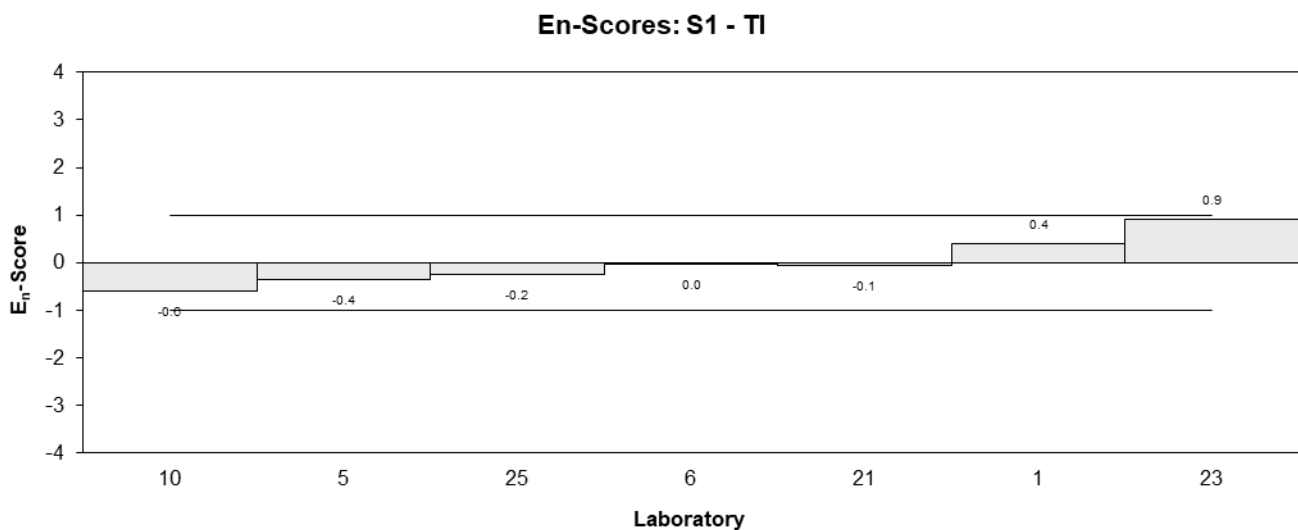
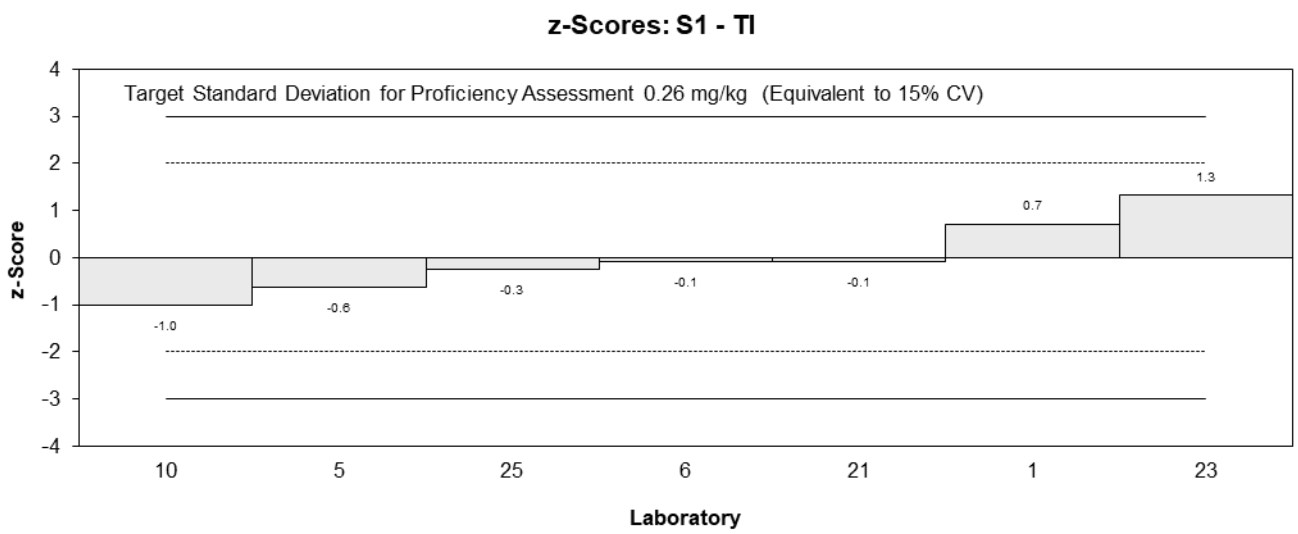
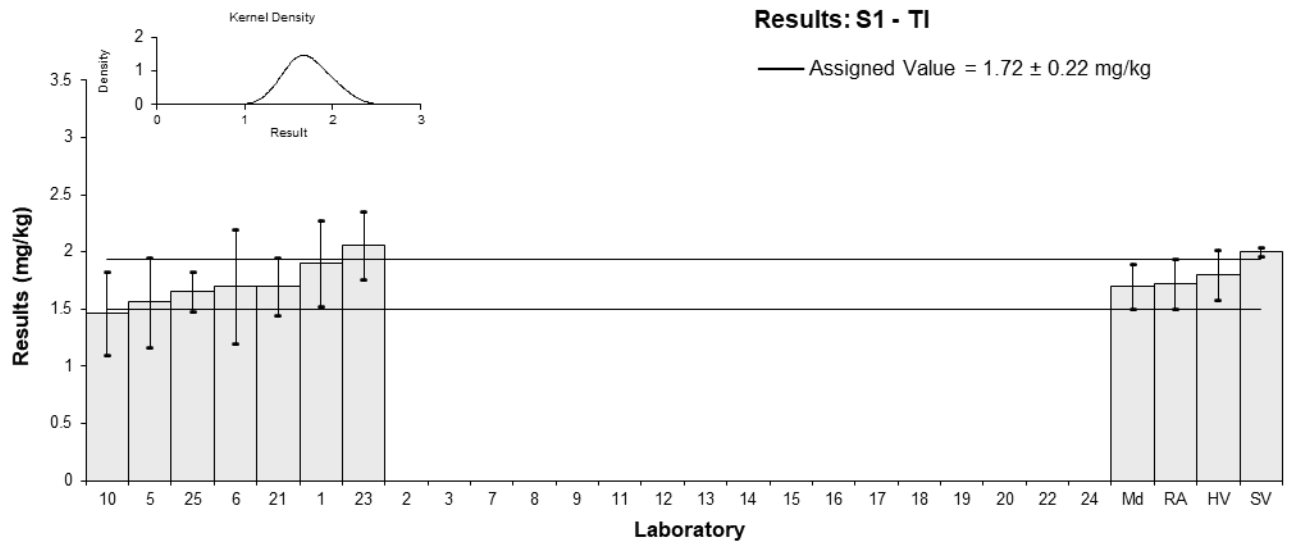


Figure 18

Table 30

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	20	4.0	-0.15	-0.07
2	NT	NT		
3	21	5	0.34	0.14
5	19.713	2.92	-0.29	-0.19
6	19.8	4.0	-0.25	-0.12
7	21.23	4.78	0.46	0.19
8	22.3	6	0.99	0.33
9	NT	NT		
10	21.2	5.30	0.44	0.17
11	NT	NT		
12	24	1.44	1.82	2.18
13	19.83762	1.98	-0.23	-0.21
14	22.5	6.8	1.08	0.32
15	NT	NT		
16	19	1.9	-0.64	-0.62
17	19.6	1.9	-0.34	-0.33
18	18.2	4.55	-1.03	-0.45
19	20.6	3.10	0.15	0.09
20	18	5	-1.13	-0.45
21	<100	67		
22	20	5	-0.15	-0.06
23	20.5	2.5	0.10	0.08
24	NT	NT		
25	19.05914	1.91	-0.61	-0.59

Statistics

Assigned Value	20.3	0.9
Spike Value	Not Spiked	
Robust Average	20.3	0.9
Median	20.0	0.8
Mean	20.4	
N	18	
Max	24	
Min	18	
Robust SD	1.5	
Robust CV	7.5%	

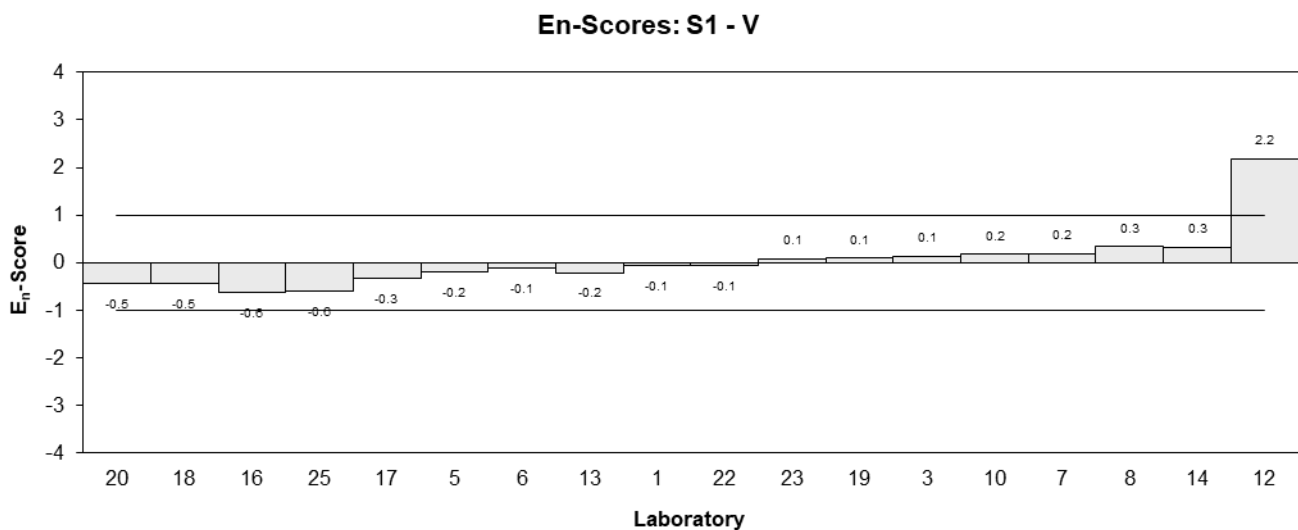
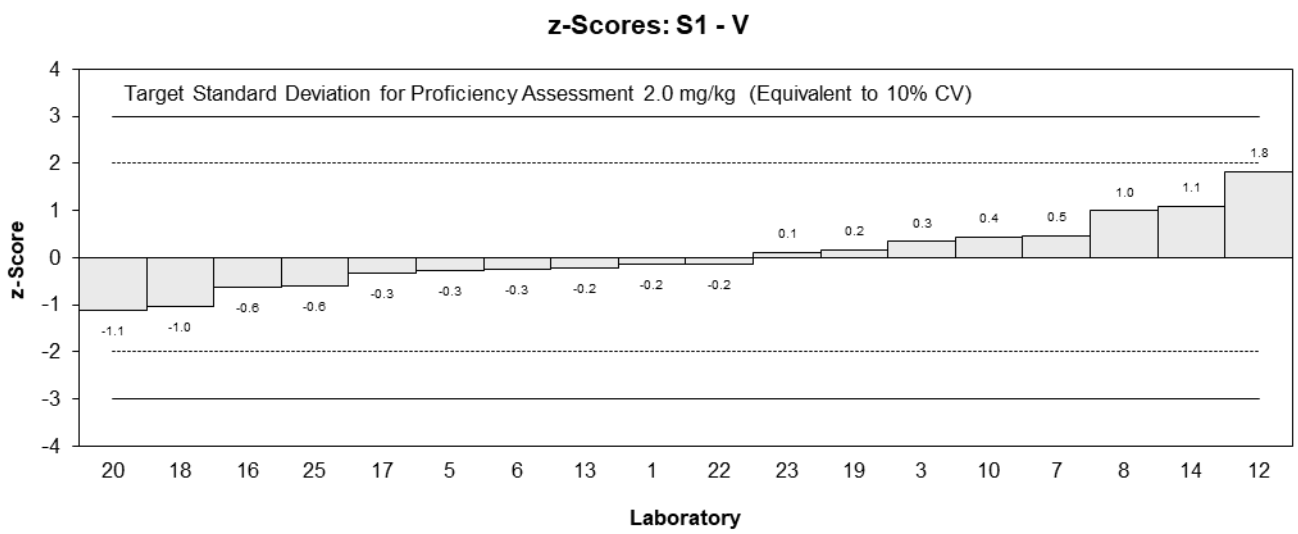
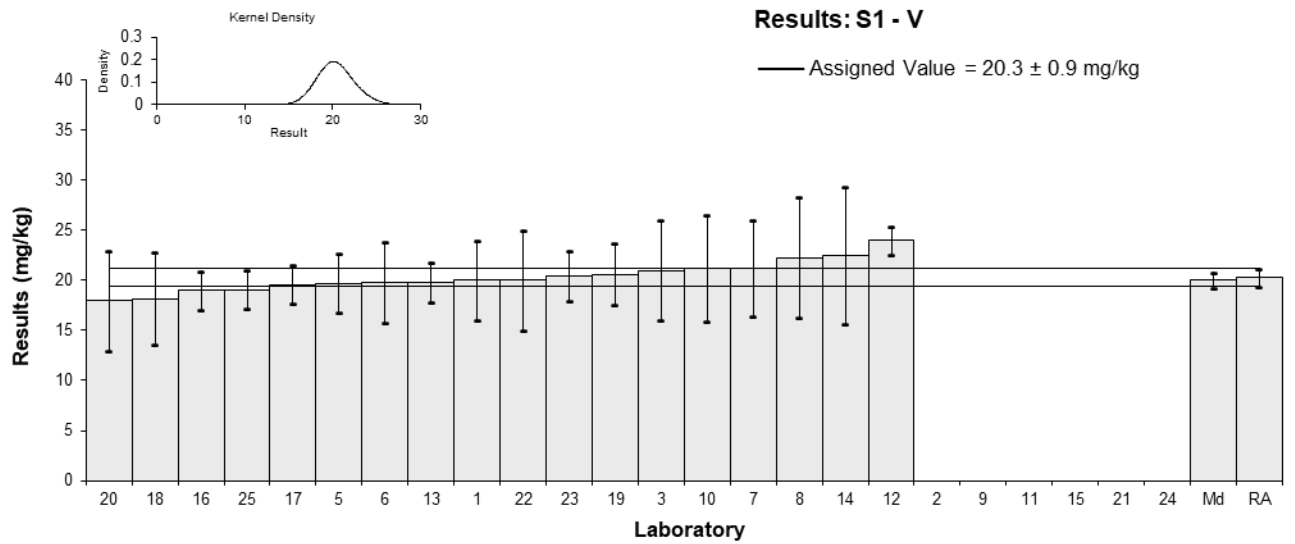


Figure 19

Table 31

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	66	13	0.17	0.08
2	NT	NT		
3	65	15	0.02	0.01
5	68.610	9.54	0.57	0.38
6	61.4	12.9	-0.54	-0.27
7	66.08	15.29	0.18	0.08
8	69.1	14	0.65	0.30
9	55	11	-1.53	-0.89
10	65.0	16.3	0.02	0.01
11	NT	NT		
12	72	2.88	1.09	2.06
13	63.13952	6.95	-0.27	-0.24
14	69.5	8.2	0.71	0.55
15	NT	NT		
16	66	11.88	0.17	0.09
17	65.1	6.5	0.03	0.03
18	60.7	15.2	-0.65	-0.27
19	62.7	12.5	-0.34	-0.17
20	64	15	-0.14	-0.06
21	64	5.3	-0.14	-0.16
22	61	20	-0.60	-0.19
23	67.7	7.0	0.43	0.39
24	NT	NT		
25	63.13113	6.31	-0.27	-0.27

Statistics

Assigned Value	64.9	1.9
Spike Value	65.0	1.6
Homogeneity Value	64.2	7.7
Robust Average	64.9	1.9
Median	65.0	1.7
Mean	64.8	
N	20	
Max	72	
Min	55	
Robust SD	3.4	
Robust CV	5.3%	

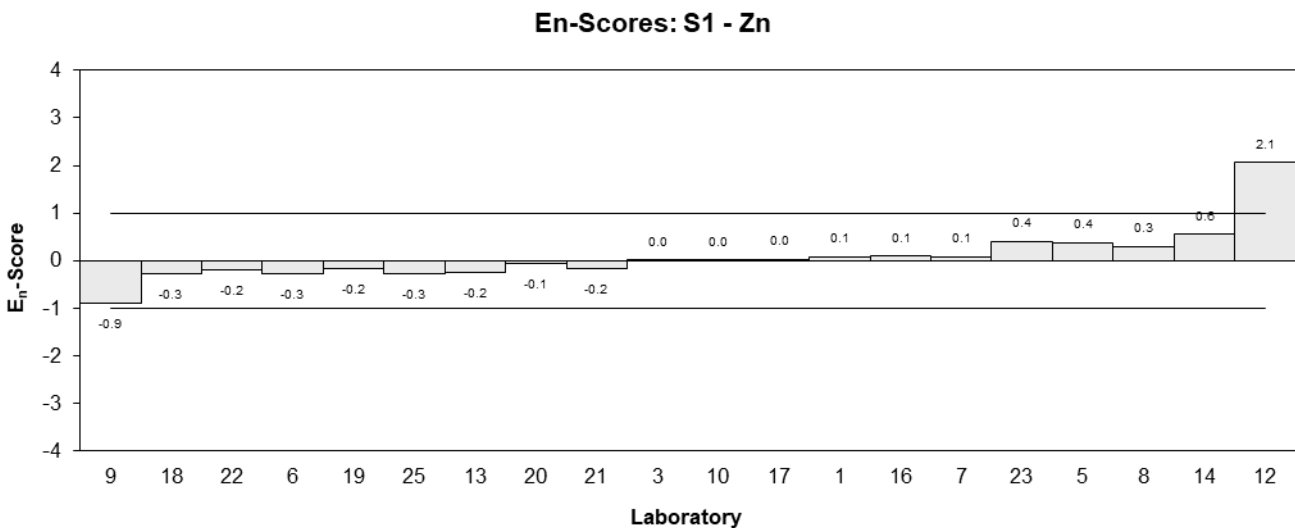
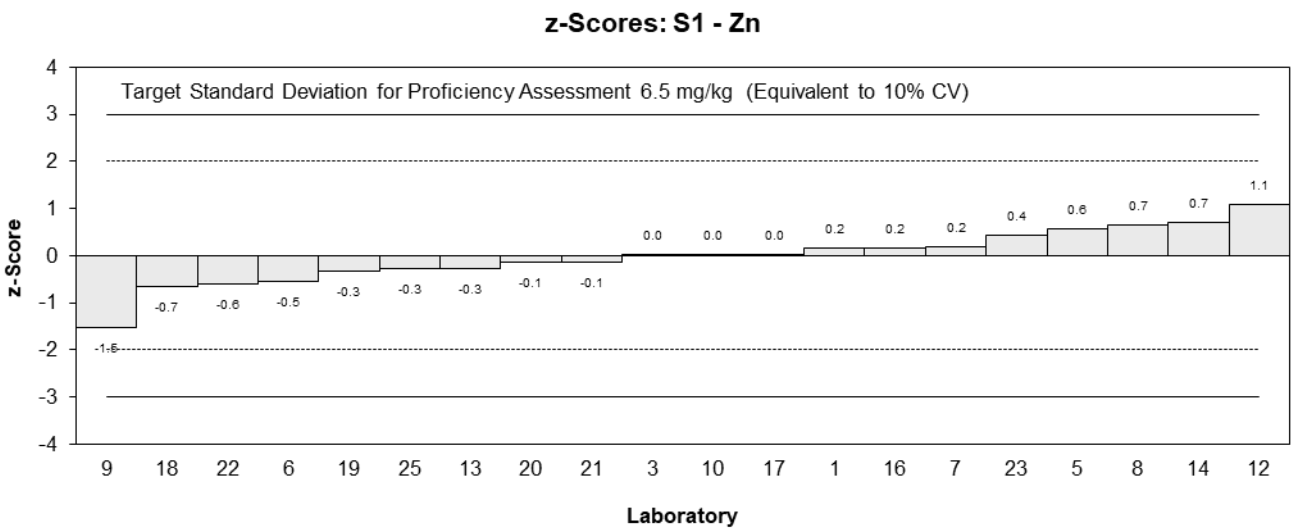
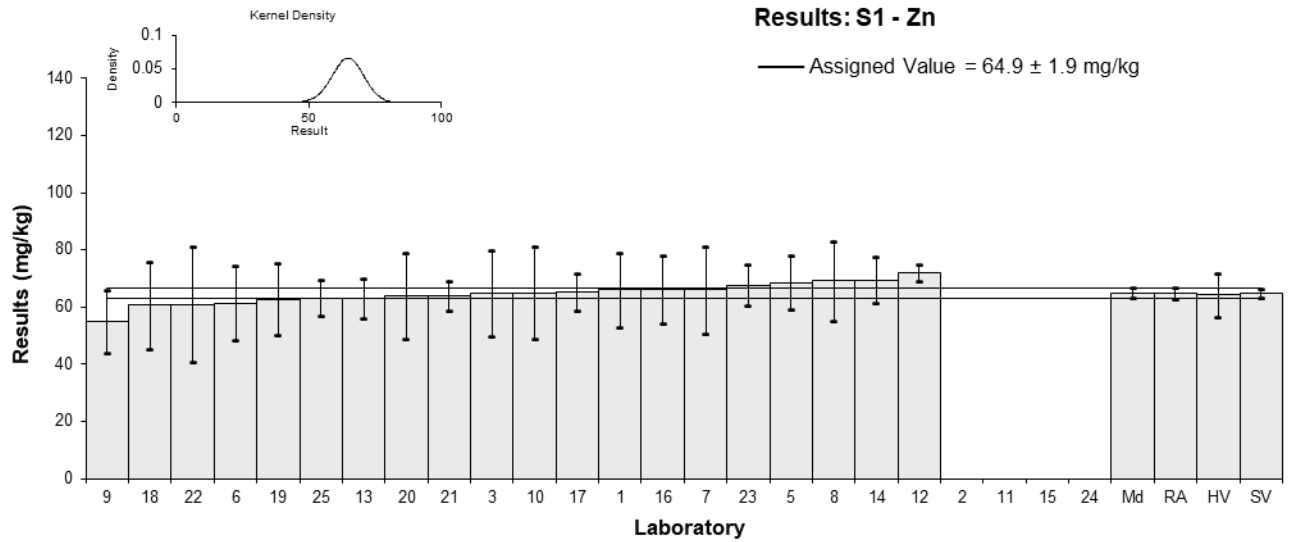


Figure 20

Table 32

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	NR	NR		
2	NT	NT		
3	7	4	1.25	0.33
5	NR	NR		
6	NT	NT		
7	7.68	1.53	1.86	1.04
8**	14.2	7	7.68	1.21
9	NT	NT		
10	5.36	1.34	-0.21	-0.13
11	NT	NT		
12	5.5	0.33	-0.09	-0.07
13	<5	NR		
14	NR	NR		
15	NT	NT		
16**	2	0.362	-3.21	-2.67
17**	2.15	0.21	-3.08	-2.62
18	NT	NT		
19**	7.40	1.48	1.61	0.91
20	5.1	2	-0.45	-0.21
21**	3.6	0.84	-1.79	-1.29
22	5	2	-0.54	-0.25
23	5.59	0.6	-0.01	-0.01
24	NT	NT		
25	3.03491	0.30	-2.29	-1.92

** Gross Error, see Section 4.2

Statistics

Assigned Value	5.6	1.3
Spike Value	5.98	0.12
Homogeneity Value	5.26	0.63
Robust Average	5.6	1.3
Median	5.43	0.50
Mean	5.53	
N	8	
Max	7.68	
Min	3.03491	
Robust SD	1.5	
Robust CV	27%	

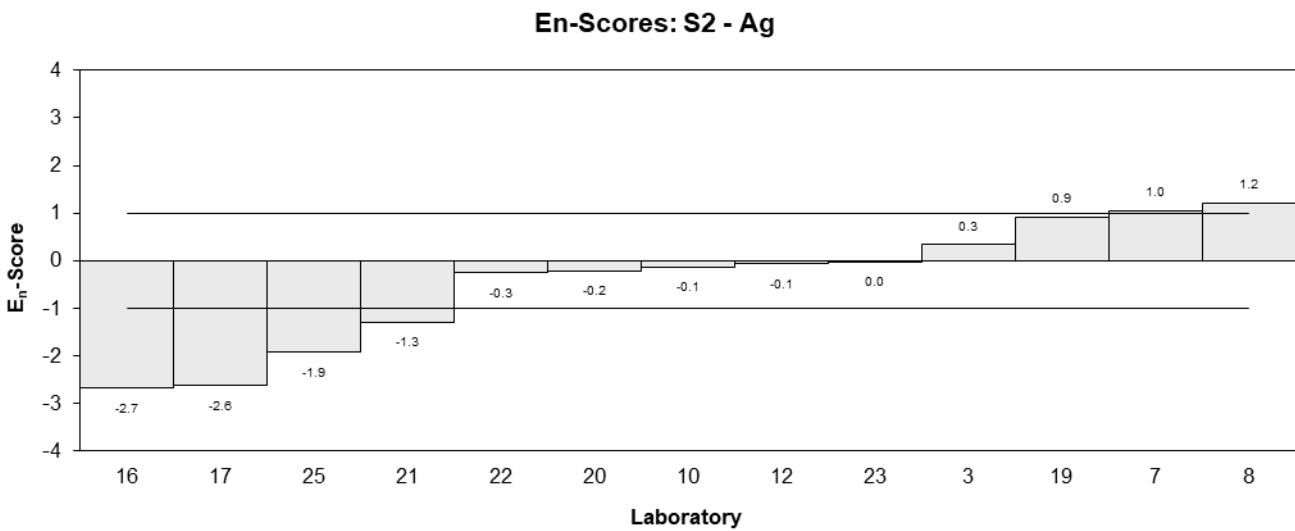
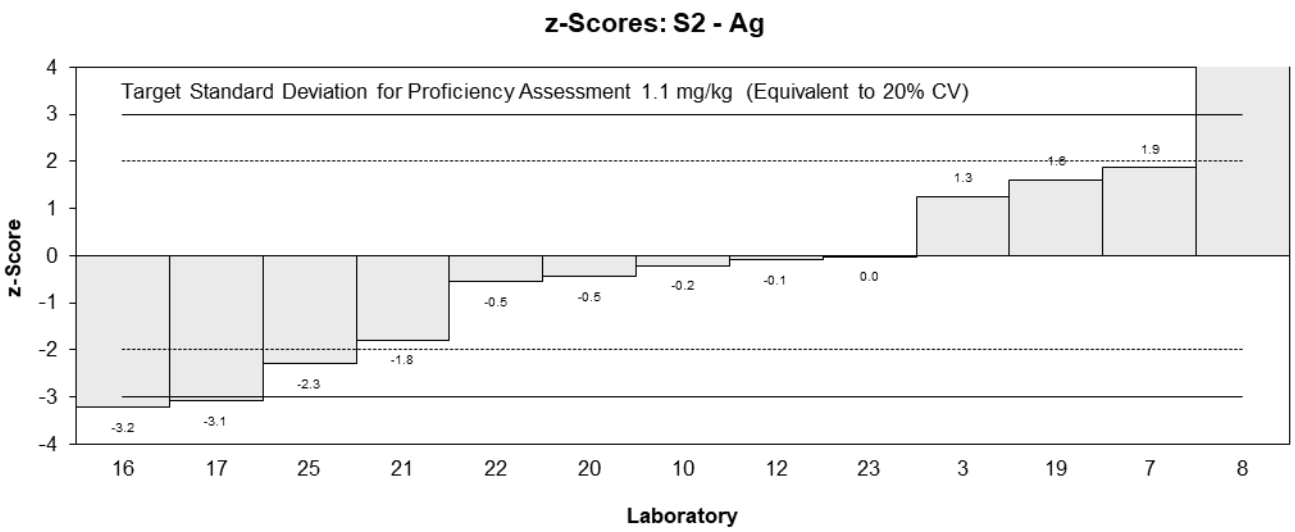
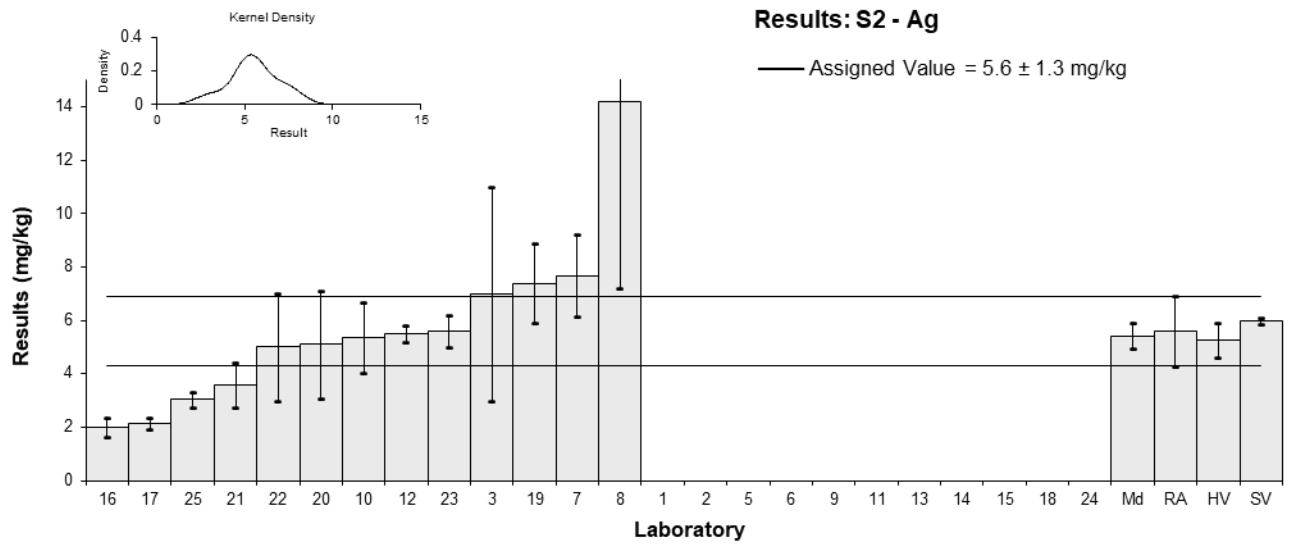


Figure 21

Table 33

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	8960	1790	1.05	0.74
2	NT	NT		
3	8300	3000	0.61	0.28
5	NR	NR		
6	NT	NT		
7	8300.33	1756.42	0.61	0.43
8**	20088	3000	8.57	3.97
9	NT	NT		
10	6849	685	-0.37	-0.43
11	NT	NT		
12	9000	450	1.08	1.35
13	6875.50311	1100	-0.35	-0.34
14	NR	NR		
15	NT	NT		
16**	2700	456.3	-3.18	-3.95
17**	4960	500	-1.65	-2.02
18	NT	NT		
19**	12700	2540	3.58	1.91
20	6100	2000	-0.88	-0.57
21**	4240	510	-2.14	-2.61
22	7100	2000	-0.20	-0.13
23	6888	700	-0.35	-0.39
24	NT	NT		
25	4504.86744	450	-1.96	-2.44

** Gross Error, see Section 4.2

Statistics

Assigned Value	7400	1100
Spike Value	Not Spiked	
Robust Average	7400	1100
Median	7000	1300
Mean	7290	
N	10	
Max	9000	
Min	4504.86744	
Robust SD	1400	
Robust CV	19%	

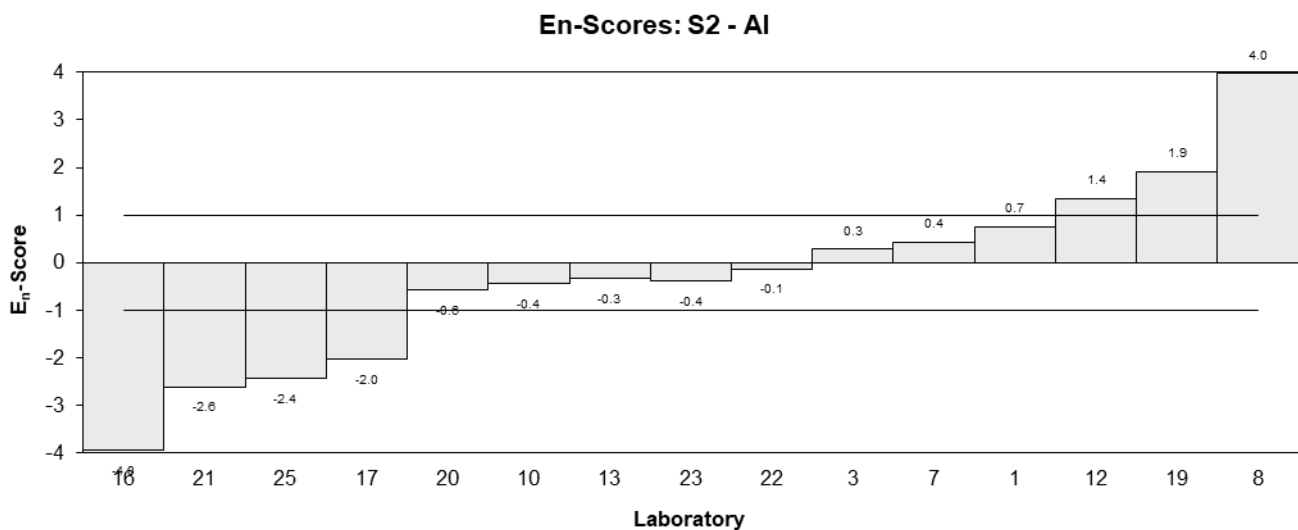
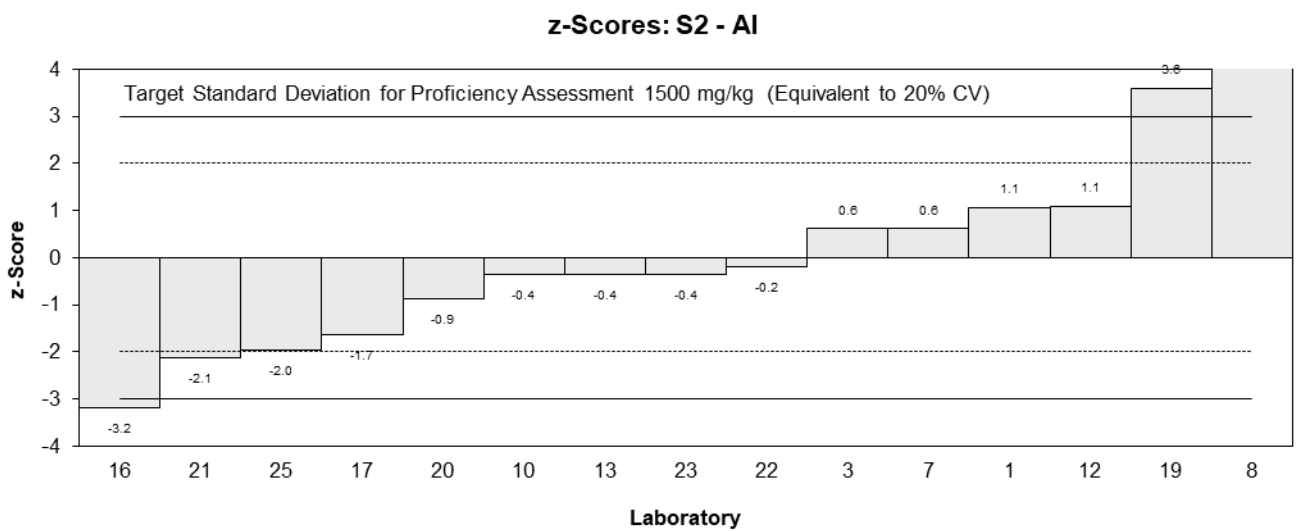
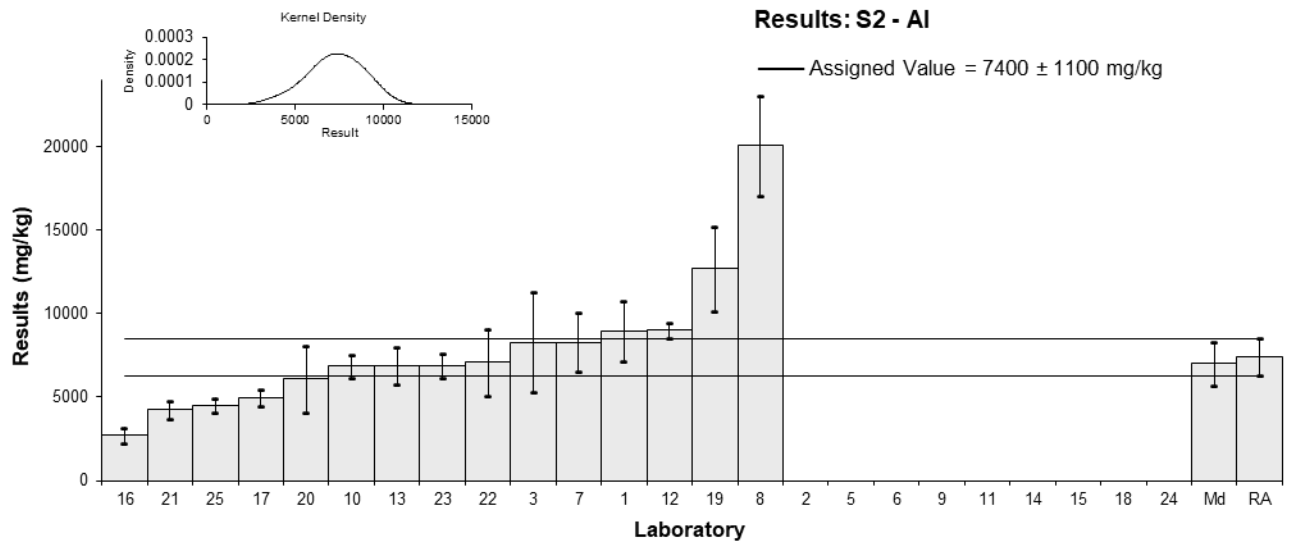


Figure 22

Table 34

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	6.1	1.2	1.35	0.83
2	NT	NT		
3	5	5	0.21	0.04
5	NR	NR		
6	NT	NT		
7	6.16	1.27	1.42	0.84
8	<15	NR		
9	NT	NT		
10	4.31	1.08	-0.51	-0.33
11	NT	NT		
12	<5	0.03		
13	<5	NR		
14	NR	NR		
15	NT	NT		
16**	2	0.266	-2.92	-2.71
17**	2.40	0.24	-2.50	-2.33
18	NT	NT		
19**	7.07	1.41	2.36	1.31
20	4.2	2	-0.62	-0.27
21**	3.5	1.5	-1.35	-0.72
22	5	4	0.21	0.05
23	4.43	0.8	-0.39	-0.29
24	NT	NT		
25	3.07584	0.31	-1.80	-1.65

** Gross Error, see Section 4.2

Statistics

Assigned Value	4.8	1.0
Spike Value	4.00	0.24
Homogeneity Value	3.78	0.45
Robust Average	4.8	1.0
Median	4.72	0.60
Mean	4.78	
N	8	
Max	6.16	
Min	3.07584	
Robust SD	1.2	
Robust CV	24%	

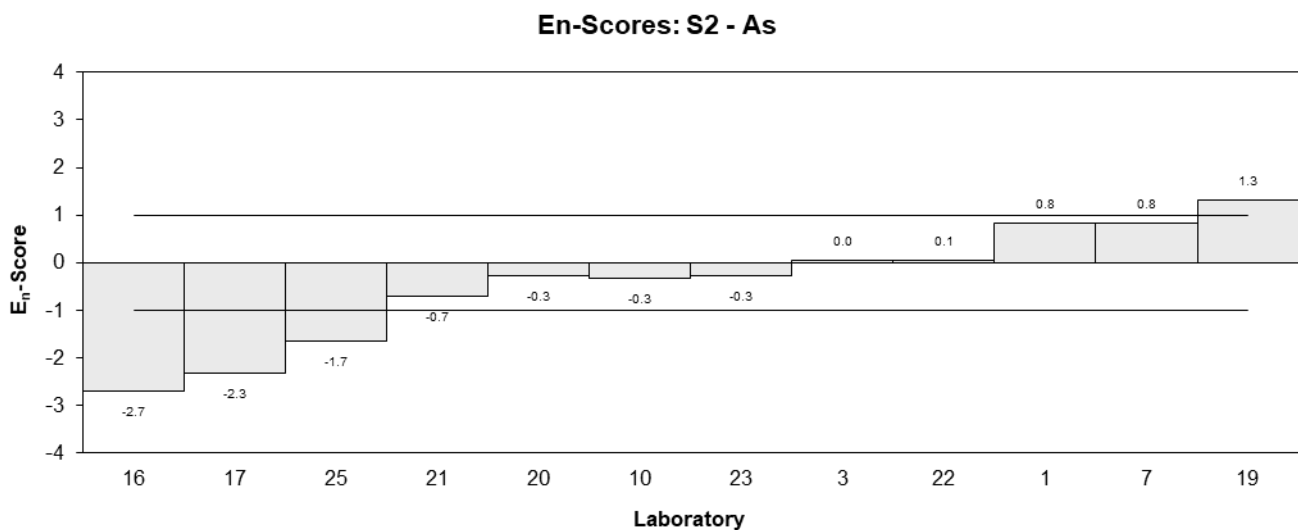
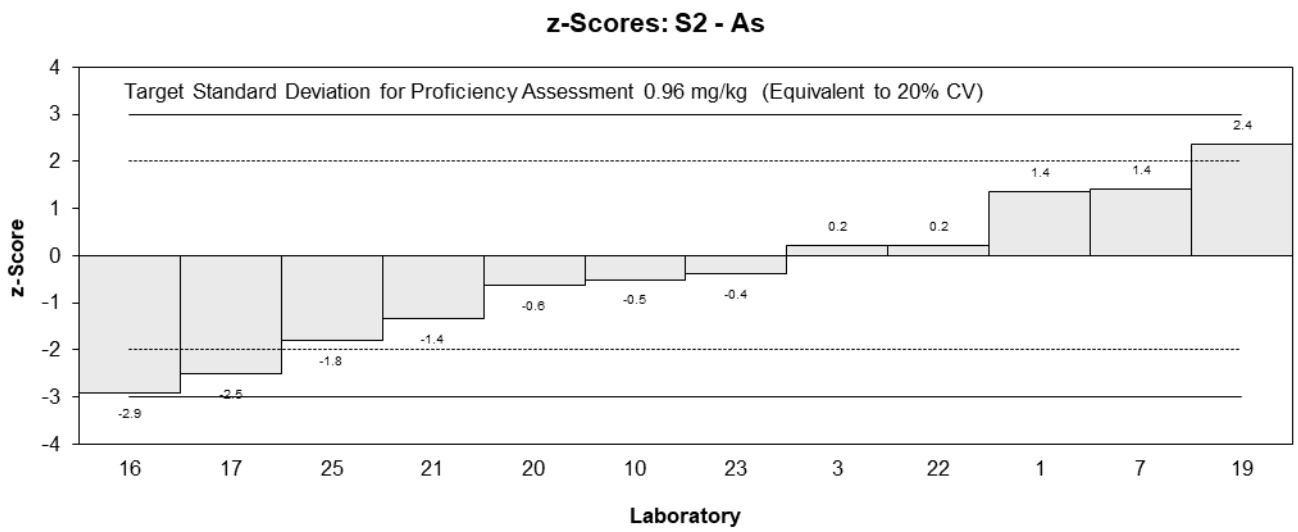
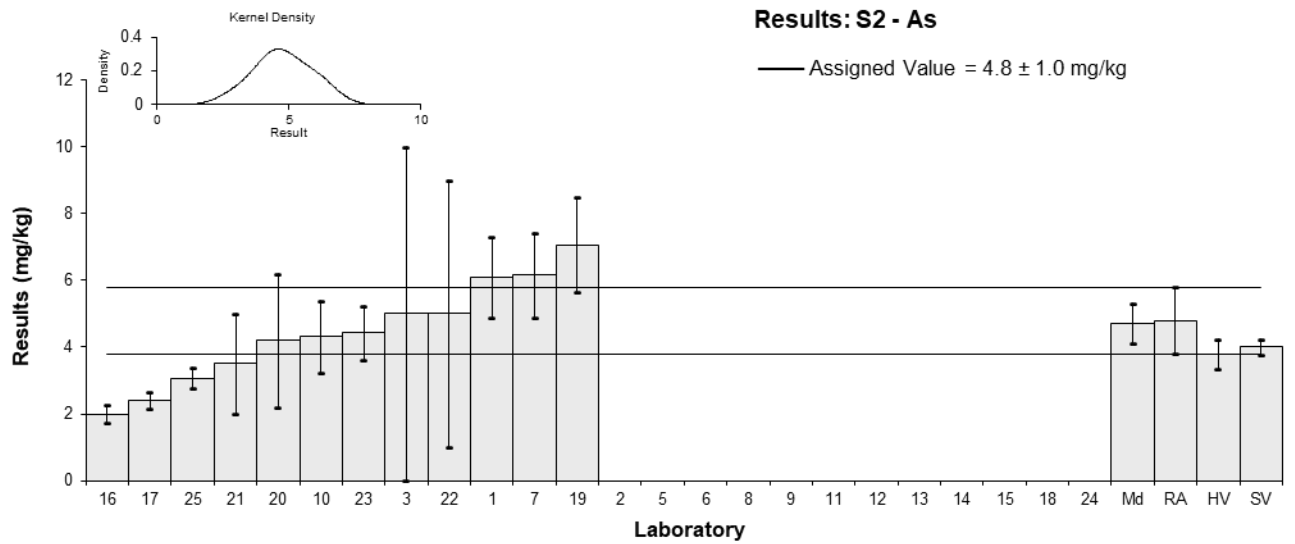


Figure 23

Table 35

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	65	13	0.51	0.39
2	NT	NT		
3	66	20	0.59	0.33
5	NR	NR		
6	NT	NT		
7	71.28	14.46	1.04	0.75
8**	165	40	8.98	2.60
9	NT	NT		
10	49.5	12.4	-0.81	-0.65
11	NT	NT		
12	63	3.15	0.34	0.47
13	54.34968	7.6	-0.39	-0.42
14	NR	NR		
15	NT	NT		
16**	23	2.829	-3.05	-4.29
17**	31.5	3.1	-2.33	-3.24
18	NT	NT		
19**	94.8	19.0	3.03	1.74
20	51	14	-0.68	-0.50
21**	29.7	1.9	-2.48	-3.61
22	57	20	-0.17	-0.09
23	67.5	7.0	0.72	0.81
24	NT	NT		
25	37.39776	3.74	-1.83	-2.47

** Gross Error, see Section 4.2

Statistics

Assigned Value	59.0	7.9
Spike Value	Not Spiked	
Robust Average	59.0	7.9
Median	60.0	7.9
Mean	58.2	
N	10	
Max	71.28	
Min	37.39776	
Robust SD	10	
Robust CV	17%	

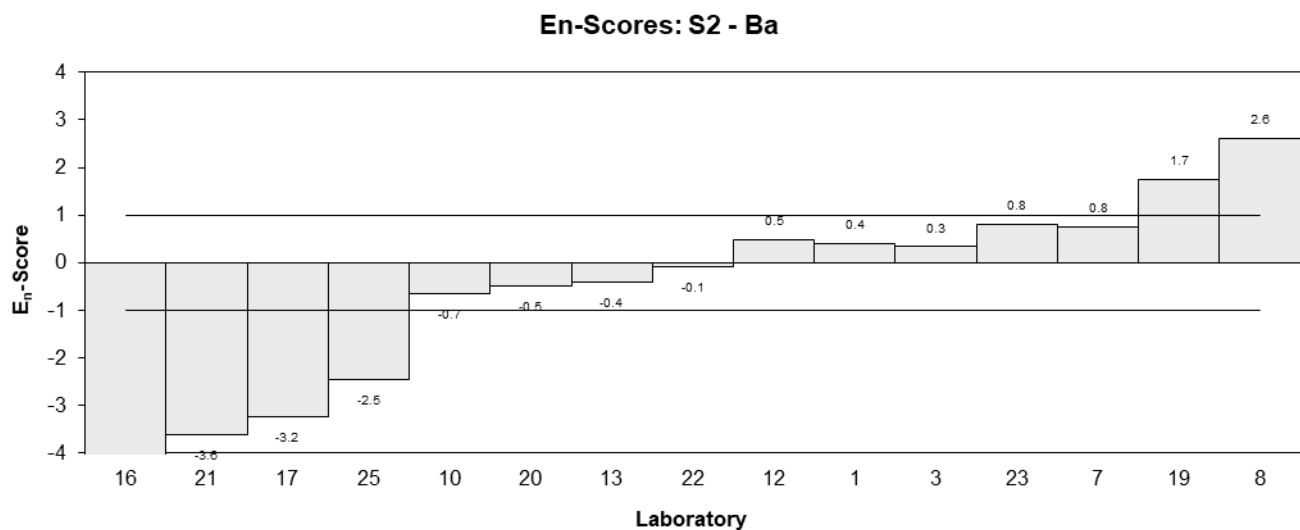
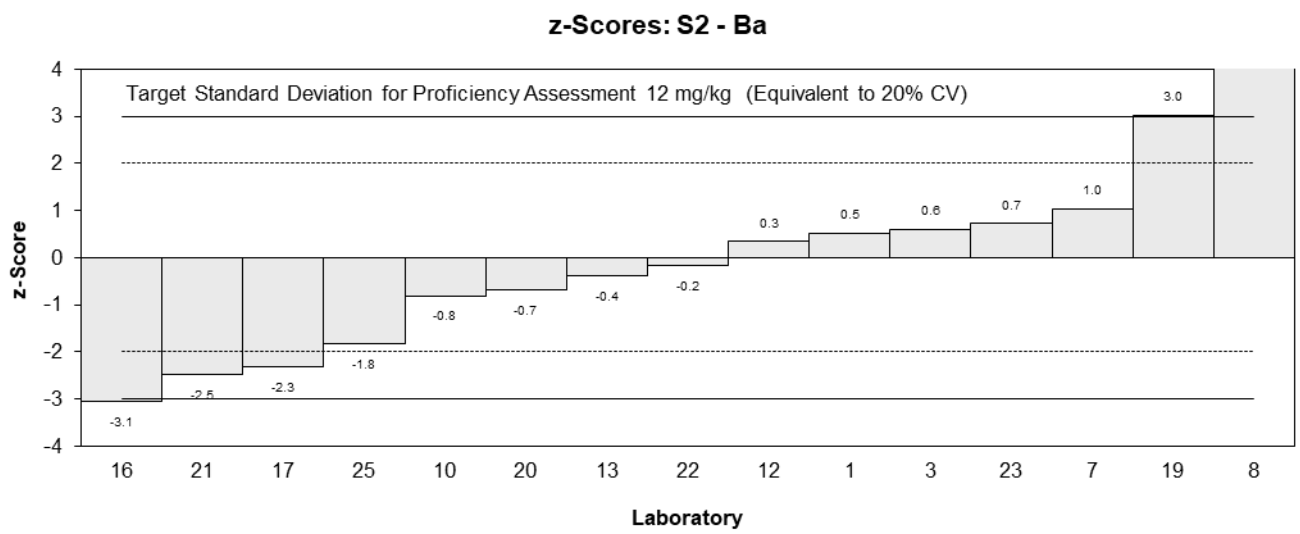
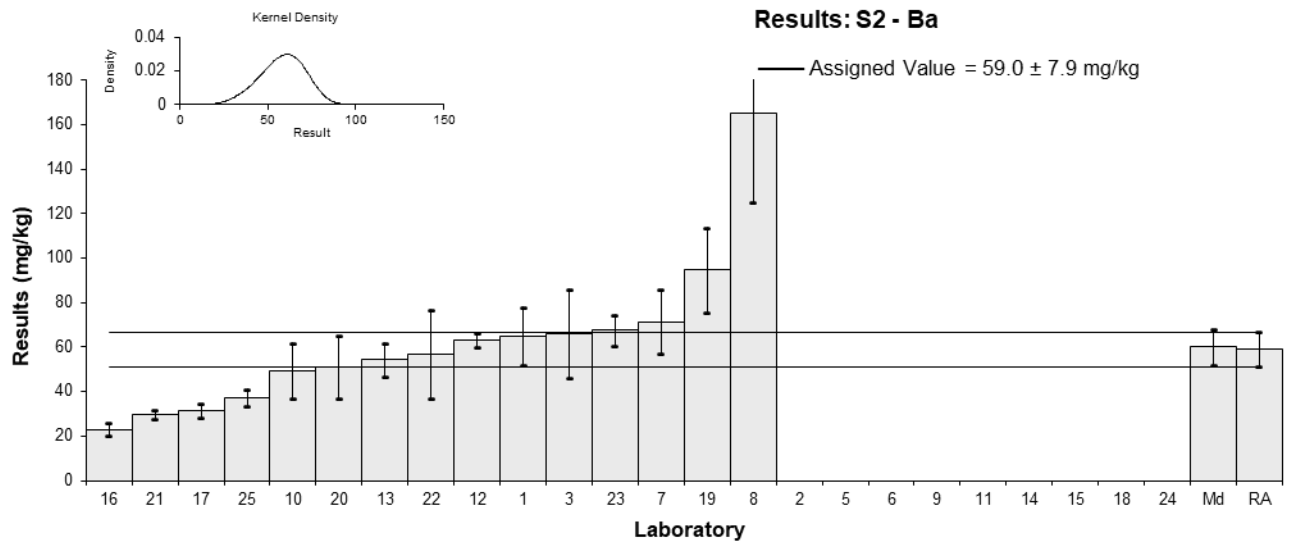


Figure 24

Table 36

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	NR	NR
2	NT	NT
3	<1	NR
5	NR	NR
6	NT	NT
7	< 10	NR
8	NT	NT
9	NT	NT
10	NT	NT
11	NT	NT
12	NT	NT
13	NT	NT
14	NR	NR
15	NT	NT
16	NT	NT
17	NT	NT
18	NT	NT
19	<10	NR
20	<1	1
21**	0.56	0.28
22	1	1
23	0.80	0.1
24	NT	NT
25	0.51027	0.05

** Gross Error, see Section 4.2

Statistics

Assigned Value	Not Set	
Spike Value	0.760	0.015
Homogeneity Value	0.742	0.089
Median	0.80	0.43
Mean	0.77	
N	3	
Max	1	
Min	0.51027	

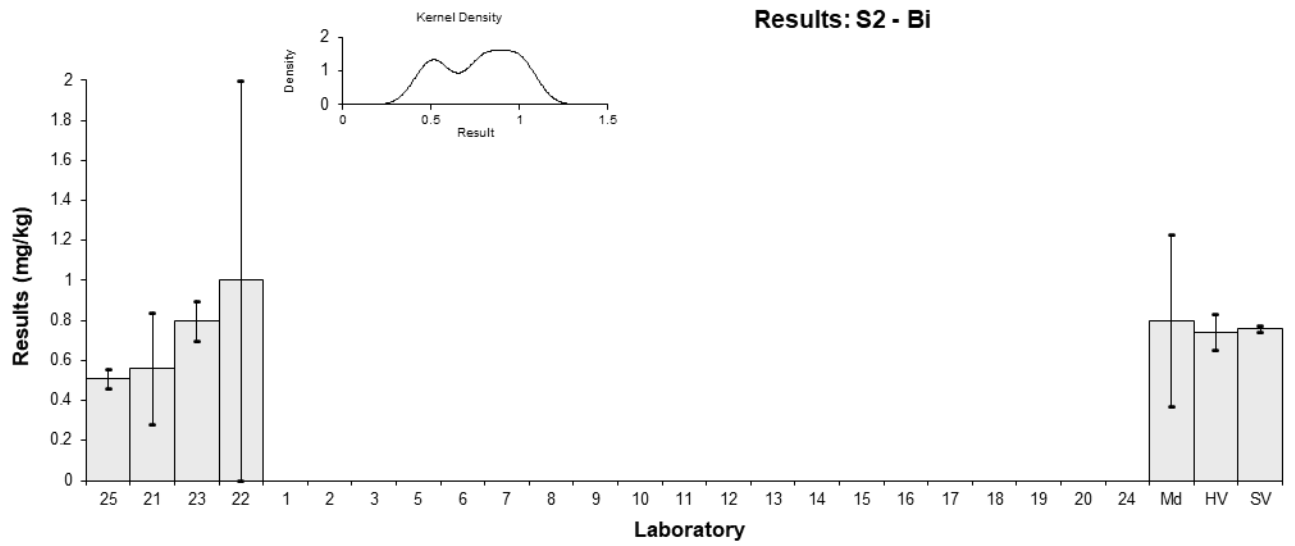


Figure 25

Table 37

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	0.85	0.17	1.34	0.82
2	NT	NT		
3	0.74	0.5	0.52	0.13
5	NR	NR		
6	NT	NT		
7*	1.11	0.23	3.28	1.63
8	<1	NR		
9	NT	NT		
10	0.618	0.155	-0.39	-0.25
11	NT	NT		
12	<1	0.04		
13	0.50237	0.06	-1.25	-1.10
14	NR	NR		
15	NT	NT		
16	<0.3	NR		
17	<1	NR		
18	NT	NT		
19**	1.13	0.266	3.43	1.53
20	0.55	0.2	-0.90	-0.49
21**	0.362	0.083	-2.30	-1.89
22	0.7	0.4	0.22	0.07
23	0.87	0.1	1.49	1.16
24	NT	NT		
25	0.52071	0.05	-1.11	-1.00

* Outlier, ** Gross Error, see Section 4.2

Statistics

Assigned Value	0.67	0.14
Spike Value	0.730	0.015
Homogeneity Value	0.736	0.088
Robust Average	0.71	0.17
Median	0.70	0.19
Mean	0.72	
N	9	
Max	1.11	
Min	0.50237	
Robust SD	0.2	
Robust CV	28%	

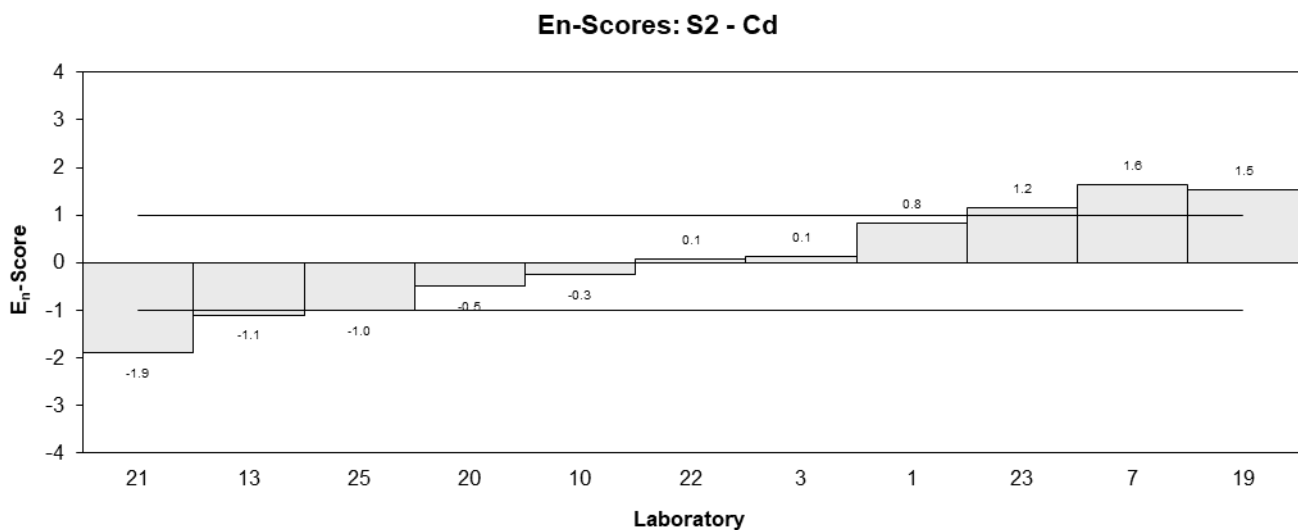
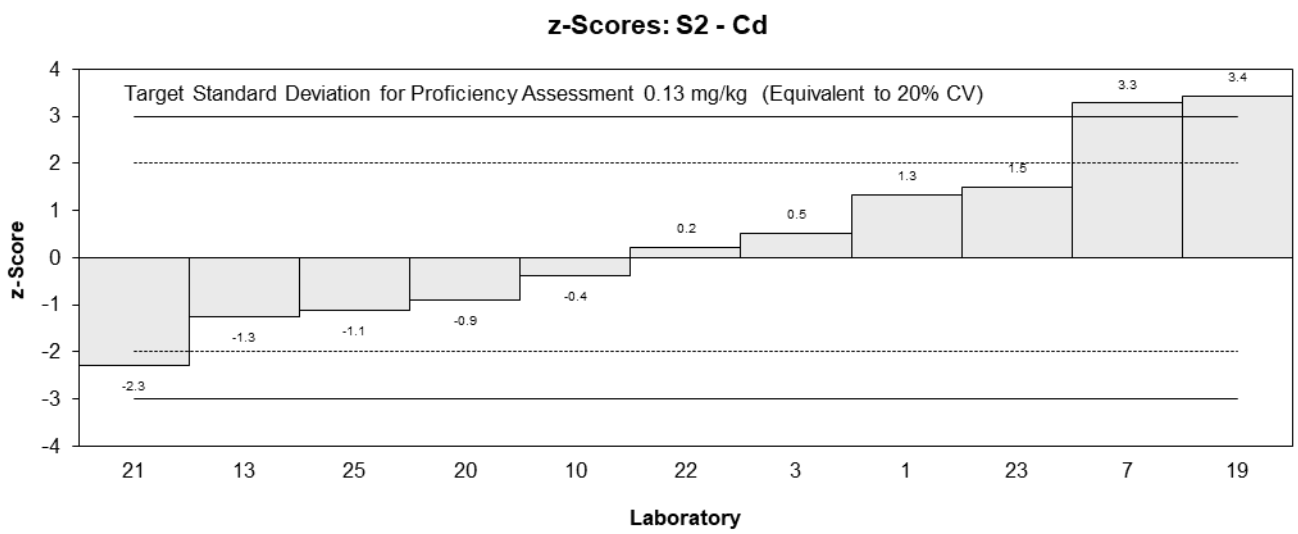
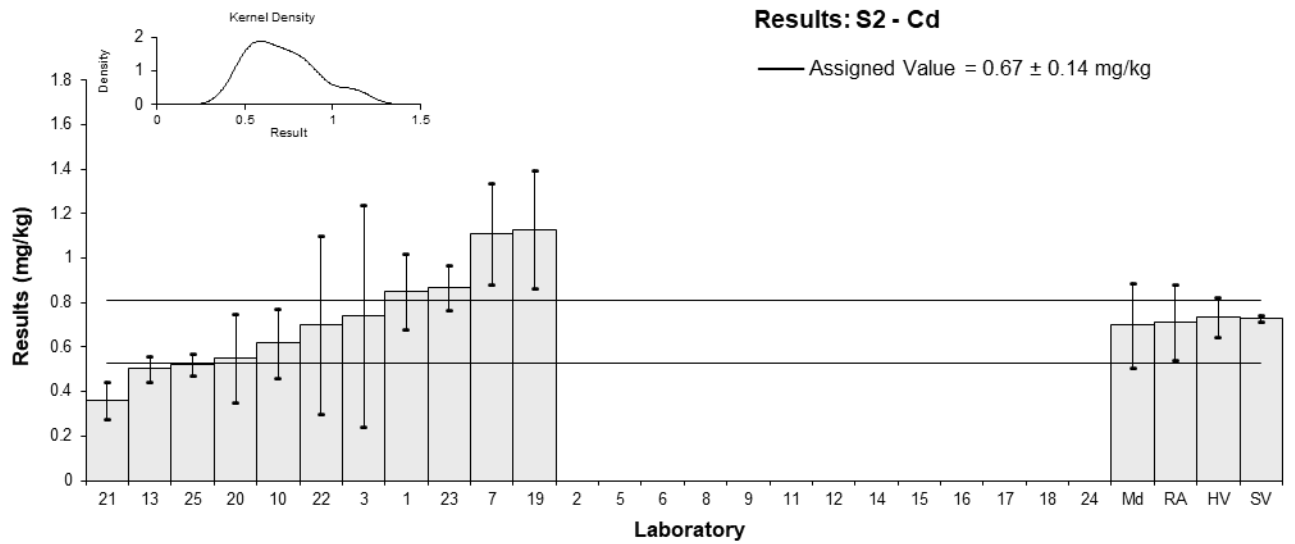


Figure 26

Table 38

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	11	2.2	1.25	0.76
2	NT	NT		
3	10	5	0.68	0.22
5	NR	NR		
6	NT	NT		
7	12.17	2.56	1.91	1.06
8**	24.7	5	9.03	2.97
9	NT	NT		
10	7.63	1.91	-0.66	-0.43
11	NT	NT		
12	9.1	0.364	0.17	0.16
13	5.24081	0.63	-2.02	-1.78
14	NR	NR		
15	NT	NT		
16**	4.8	0.5664	-2.27	-2.02
17**	5.9	0.59	-1.65	-1.46
18	NT	NT		
19**	13.4	2.67	2.61	1.40
20	9.3	2	0.28	0.18
21**	4.97	0.75	-2.18	-1.87
22	9	3	0.11	0.06
23	8.98	1.0	0.10	0.08
24	NT	NT		
25	5.90303	0.59	-1.65	-1.46

** Gross Error, see Section 4.2

Statistics

Assigned Value	8.8	1.9
Spike Value	8.70	0.84
Homogeneity Value	8.26	0.99
Robust Average	8.8	1.9
Median	9.1	1.4
Mean	8.8	
N	10	
Max	12.17	
Min	5.24081	
Robust SD	2.4	
Robust CV	27%	

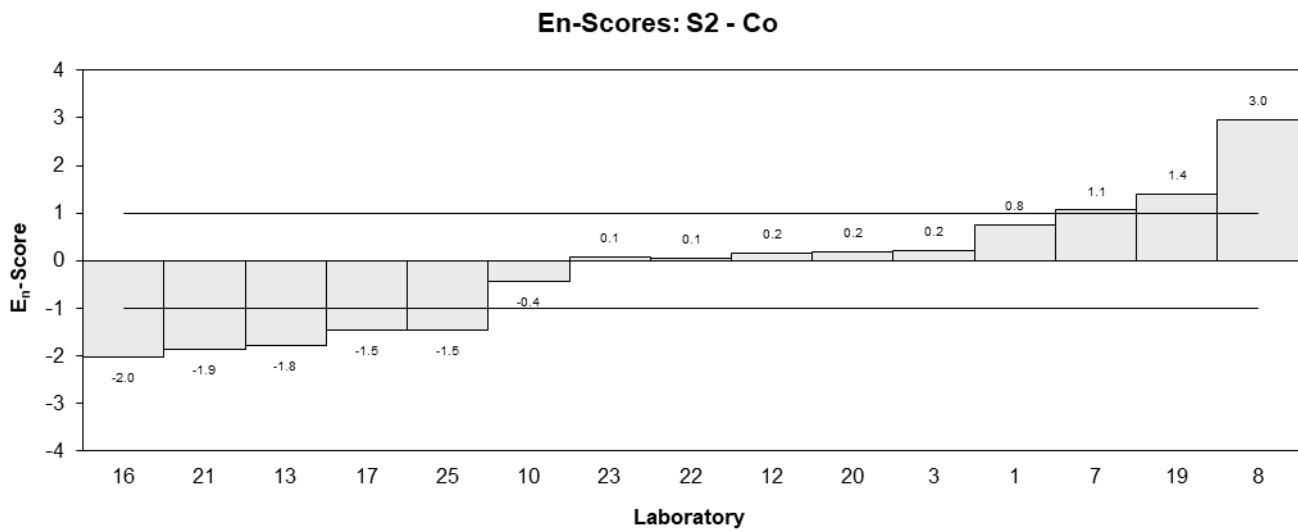
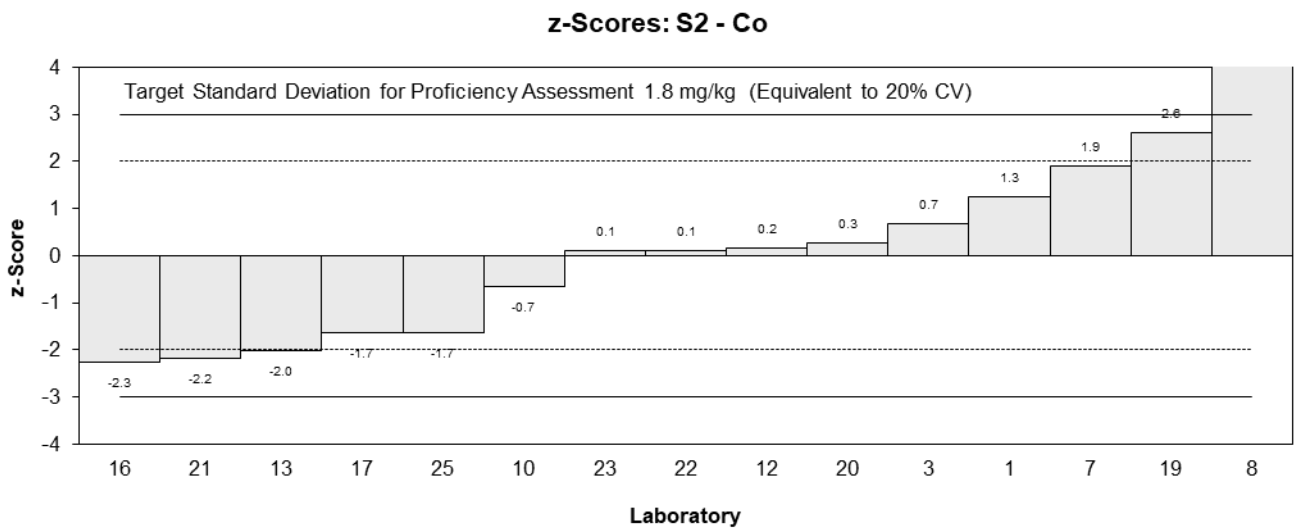
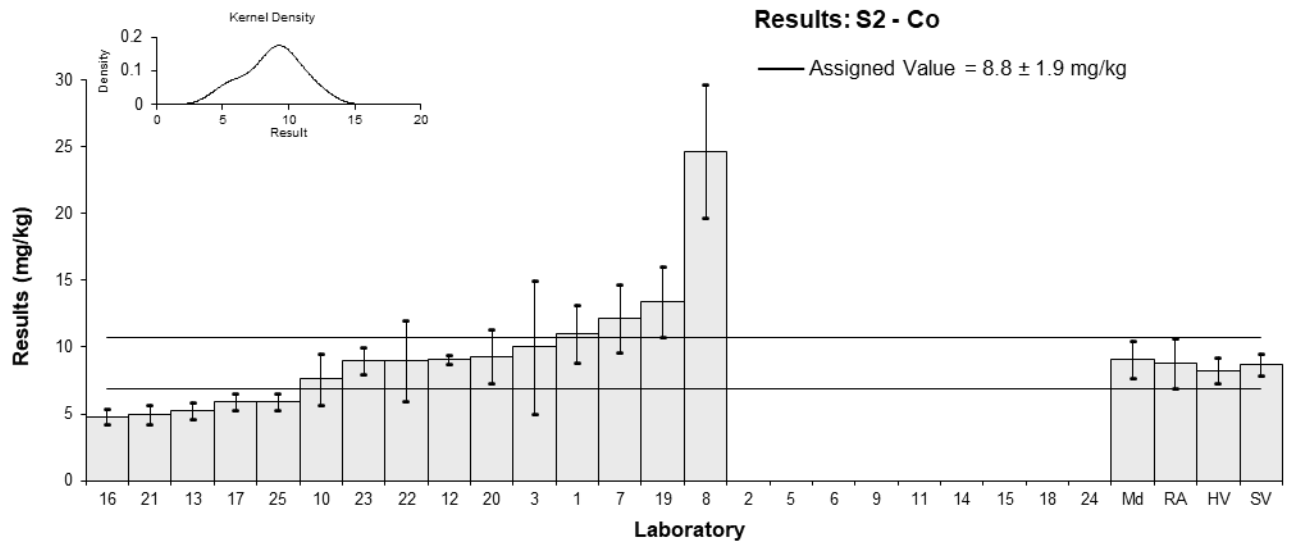


Figure 27

Table 39

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	NR	NR
2	NT	NT
3	<1	0.4
5	NR	NR
6	NT	NT
7	NT	NT
8	NT	NT
9	NT	NT
10	NT	NT
11	NT	NT
12	NT	NT
13	NT	NT
14	NR	NR
15	NT	NT
16	NT	NT
17	NT	NT
18	NT	NT
19	NT	NT
20	1	0.3
21**	0.84	0.16
22	2	1
23	NT	NT
24	NT	NT
25	NR	NR

** Gross Error, see Section 4.2

Statistics

Assigned Value	Not Set	
Spike Value	1.13	0.06
Homogeneity Value	1.16	0.14
Mean	1.5	
N	2	
Max	2	
Min	1	

Results: S2 - Cs

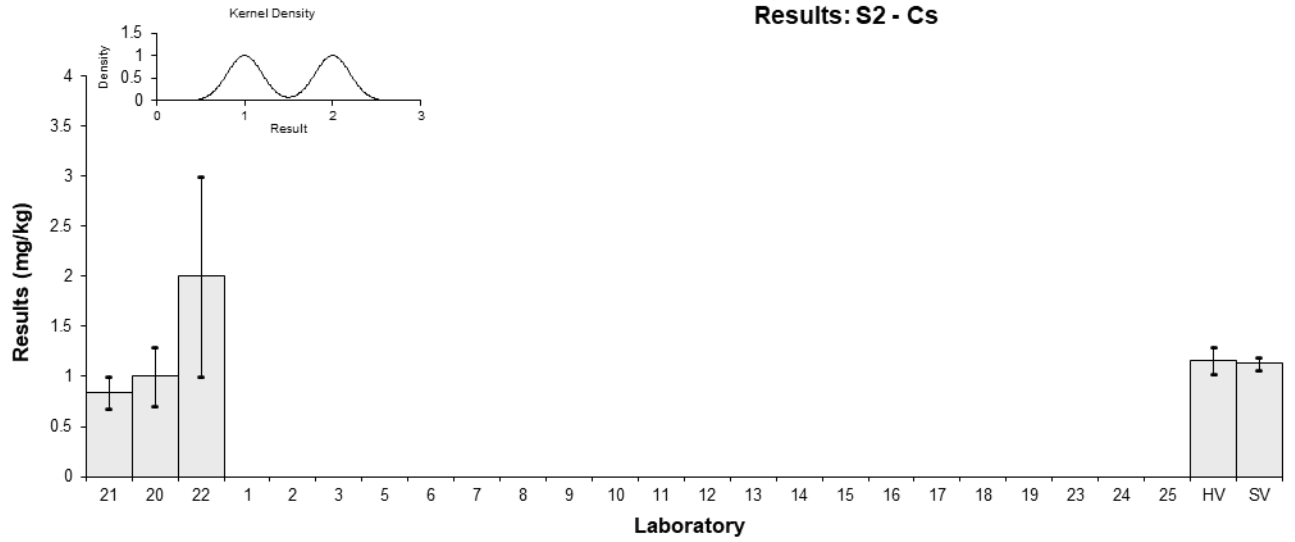


Figure 28

Table 40

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	50	10	1.13	0.71
2	NT	NT		
3	46	15	0.64	0.31
5	NR	NR		
6	NT	NT		
7	53.59	11.93	1.57	0.89
8**	110.9	20	8.59	3.25
9	NT	NT		
10	38.1	9.53	-0.33	-0.22
11	NT	NT		
12	44	1.76	0.39	0.39
13	25.95603	3.89	-1.82	-1.65
14	NR	NR		
15	NT	NT		
16**	17	2.125	-2.92	-2.84
17**	25.4	2.5	-1.89	-1.82
18	NT	NT		
19**	56.0	11.2	1.86	1.10
20	38	11	-0.34	-0.20
21**	26.5	3.9	-1.75	-1.59
22	45	10	0.51	0.33
23	40.4	4.0	-0.05	-0.04
24	NT	NT		
25	26.69469	2.67	-1.73	-1.65

** Gross Error, see Section 4.2

Statistics

Assigned Value	40.8	8.1
Spike Value	42.4	0.9
Homogeneity Value	36.4	5.5
Robust Average	40.8	8.1
Median	42.2	4.9
Mean	40.8	
N	10	
Max	53.59	
Min	25.95603	
Robust SD	10	
Robust CV	25%	

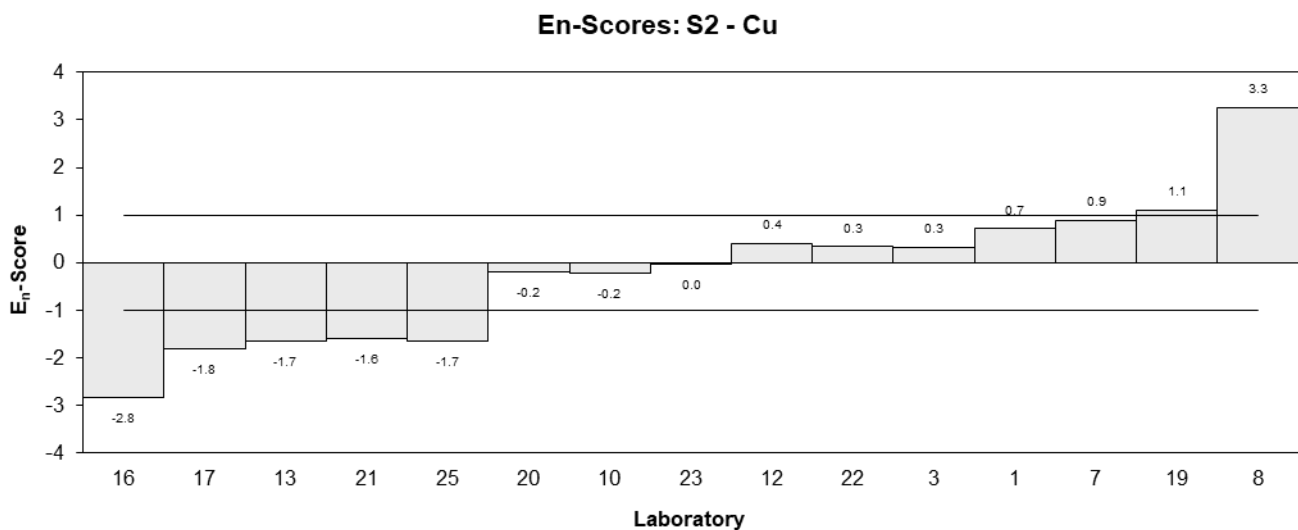
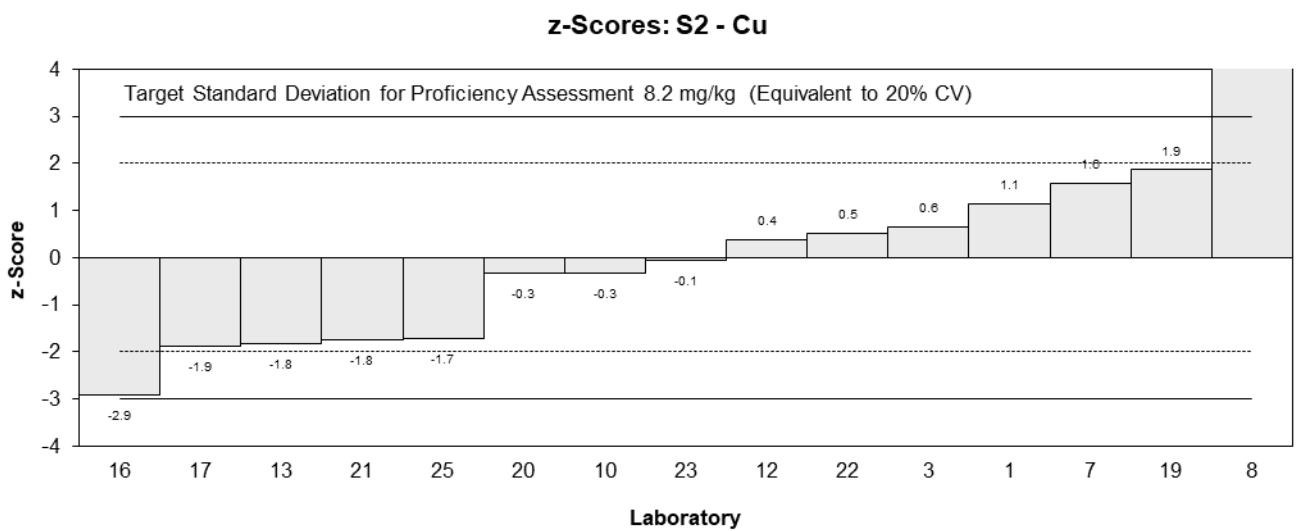
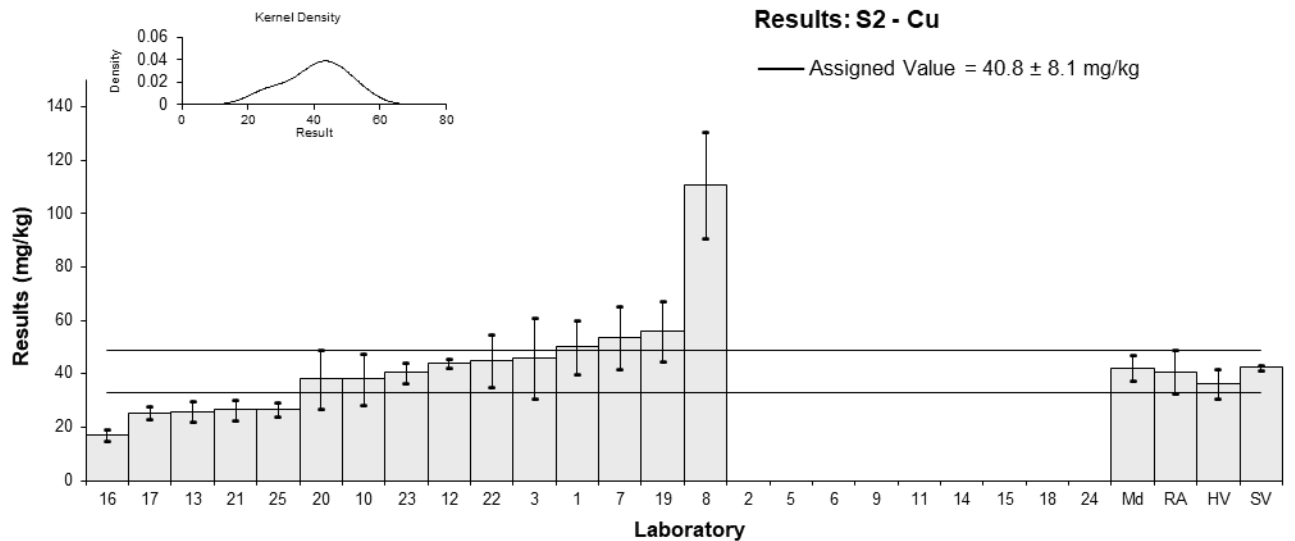


Figure 29

Table 41

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	0.72	0.14	0.90	0.56
2	NT	NT		
3	0.75	0.4	1.15	0.33
5	NR	NR		
6	NT	NT		
7	0.91	0.19	2.46	1.27
8**	2.8	1	17.95	2.17
9	NT	NT		
10	0.548	0.137	-0.51	-0.32
11	NT	NT		
12	0.5	0.05	-0.90	-0.74
13	0.30399	0.06	-2.51	-2.01
14	NR	NR		
15	NT	NT		
16**	0.25	0.042	-2.95	-2.46
17	<1	NR		
18	NT	NT		
19**	0.89	0.223	2.30	1.06
20	0.65	0.2	0.33	0.16
21**	0.241	0.075	-3.02	-2.32
22	0.7	0.4	0.74	0.21
23	0.51	0.06	-0.82	-0.66
24	NT	NT		
25	0.47762	0.05	-1.09	-0.89

** Gross Error, see Section 4.2

Statistics

Assigned Value	0.61	0.14
Spike Value	0.866	0.018
Homogeneity Value	0.588	0.071
Robust Average	0.61	0.14
Median	0.60	0.13
Mean	0.61	
N	10	
Max	0.91	
Min	0.30399	
Robust SD	0.18	
Robust CV	30%	

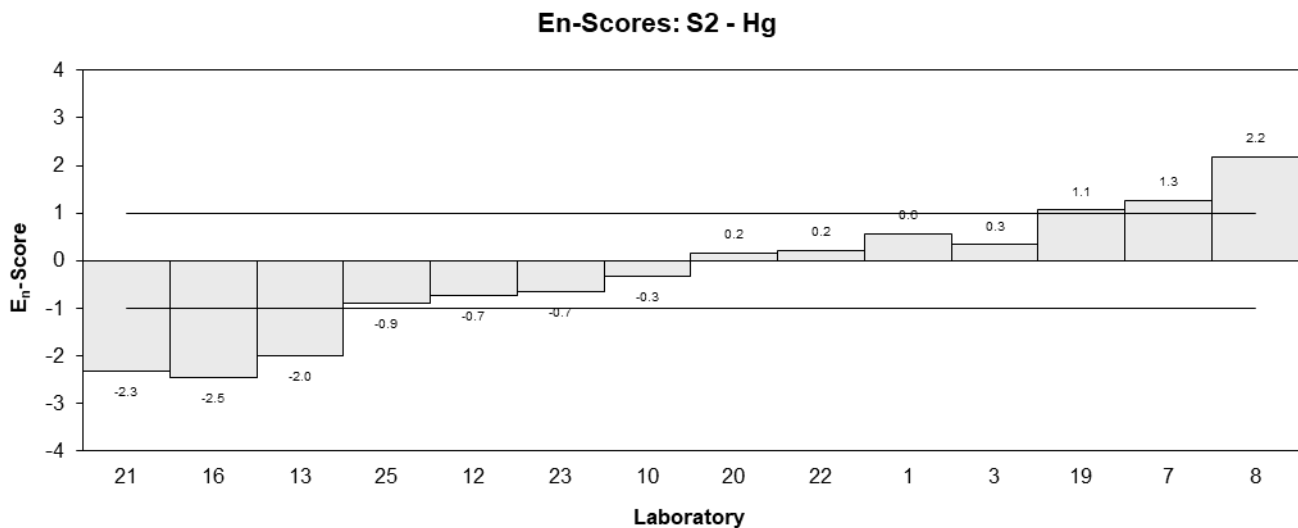
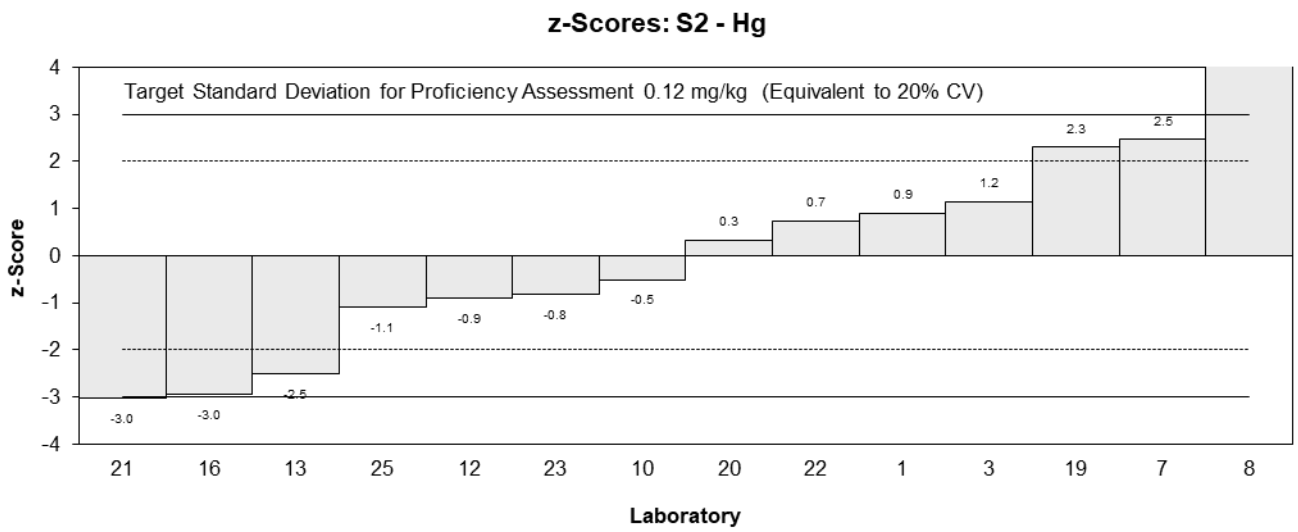
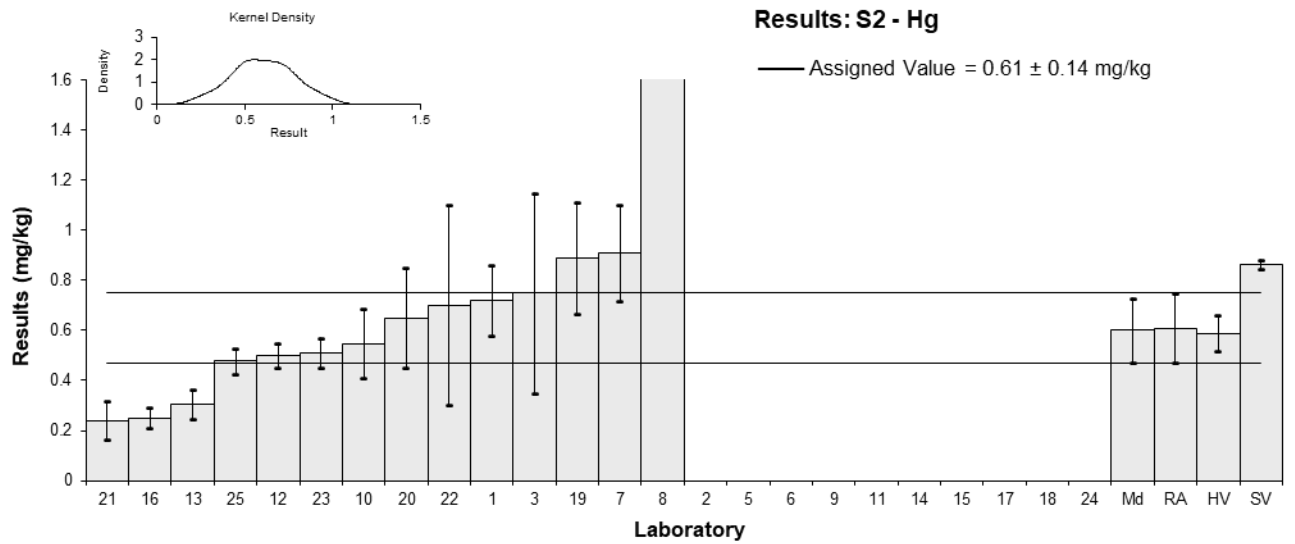


Figure 30

Table 42

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	14	2.8	1.19	0.76
2	NT	NT		
3	13	4	0.75	0.37
5	NR	NR		
6	NT	NT		
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	9.09	2.27	-0.98	-0.70
11	NT	NT		
12	NT	NT		
13	11.92480	2.4	0.28	0.19
14	NR	NR		
15	NT	NT		
16	NT	NT		
17	NT	NT		
18	NT	NT		
19	NT	NT		
20	10	3	-0.58	-0.35
21**	5.37	0.45	-2.62	-2.64
22	10	3	-0.58	-0.35
23	NT	NT		
24	NT	NT		
25	NT	NT		

** Gross Error, see Section 4.2

Statistics

Assigned Value	11.3	2.2
Spike Value	11.0	0.8
Homogeneity Value	10.1	1.2
Robust Average	11.3	2.2
Median	11.0	2.1
Mean	11.3	
N	6	
Max	14	
Min	9.09	
Robust SD	2.2	
Robust CV	19%	

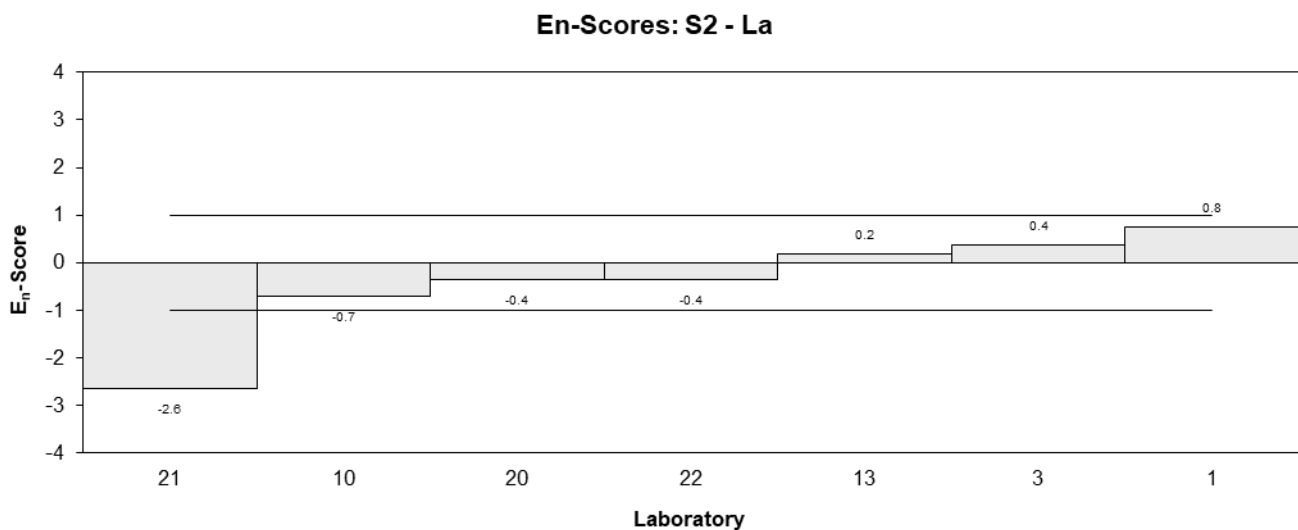
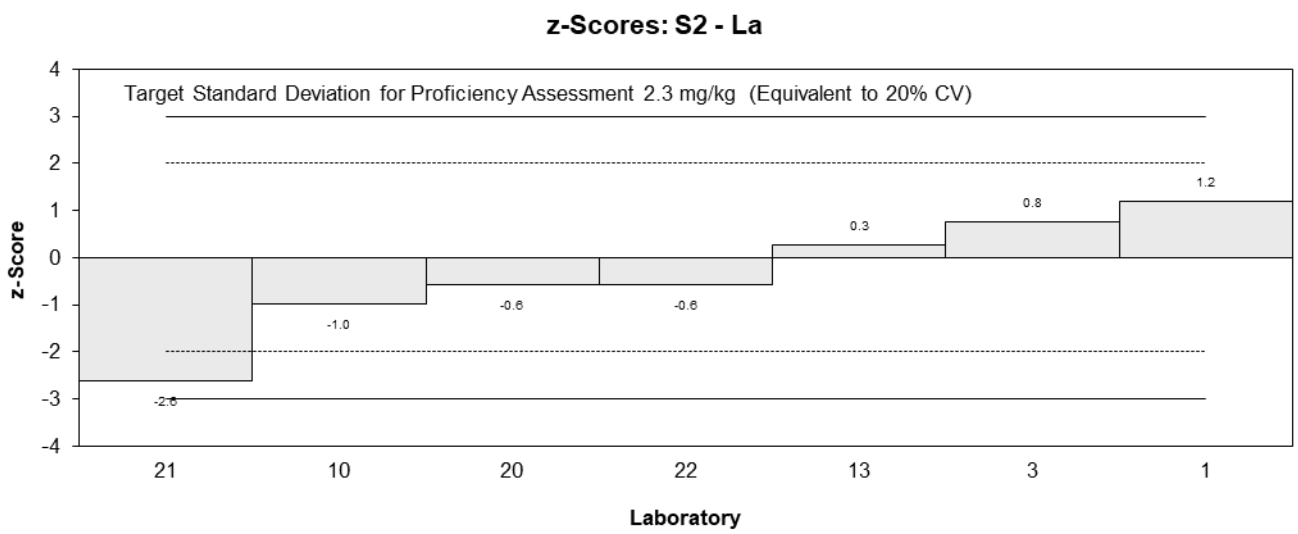
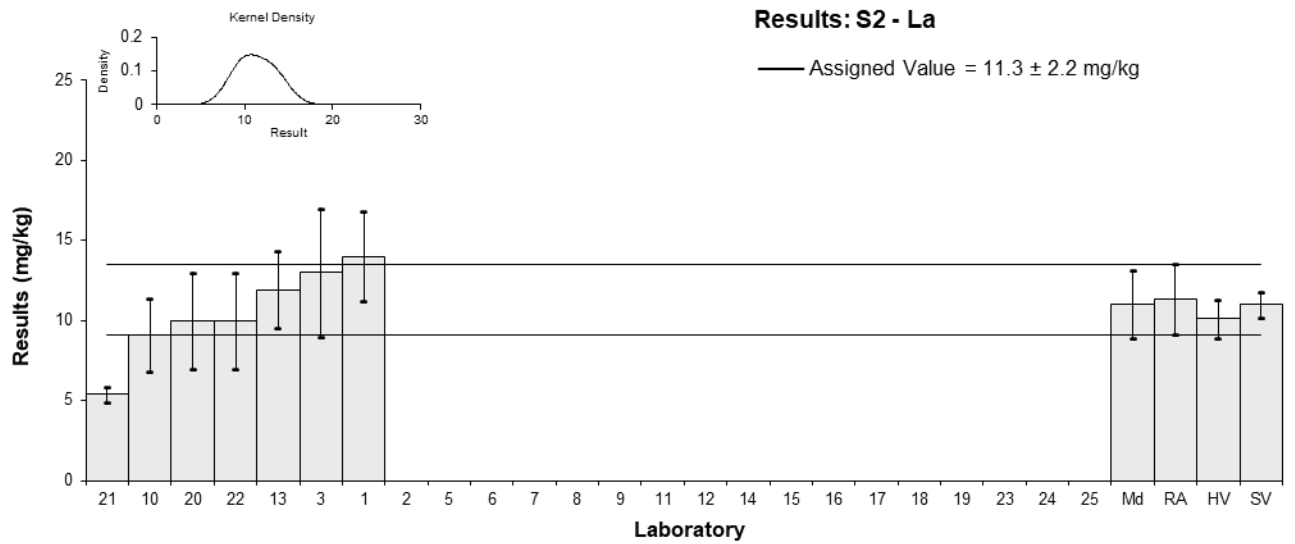


Figure 31

Table 43

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	28	5.6	0.76	0.55
2	NT	NT		
3	26	8	0.35	0.19
5	NR	NR		
6	NT	NT		
7	35.68	7.2	2.34	1.40
8**	61.2	12	7.59	2.93
9	NT	NT		
10	23.6	5.90	-0.14	-0.10
11	NT	NT		
12	25	1.75	0.14	0.17
13	19.47358	2.53	-0.99	-1.06
14	NR	NR		
15	NT	NT		
16**	11	1.419	-2.74	-3.28
17**	13.2	1.3	-2.28	-2.76
18	NT	NT		
19**	35.9	7.18	2.39	1.43
20	21	4	-0.68	-0.60
21**	16.1	3	-1.69	-1.69
22	27	8	0.56	0.30
23	24.1	2.5	-0.04	-0.04
24	NT	NT		
25	17.31786	1.73	-1.44	-1.67

** Gross Error, see Section 4.2

Statistics

Assigned Value	24.3	3.8
Spike Value	24.9	0.5
Homogeneity Value	21.6	2.6
Robust Average	24.3	3.8
Median	24.6	3.5
Mean	24.7	
N	10	
Max	35.68	
Min	17.31786	
Robust SD	4.8	
Robust CV	20%	

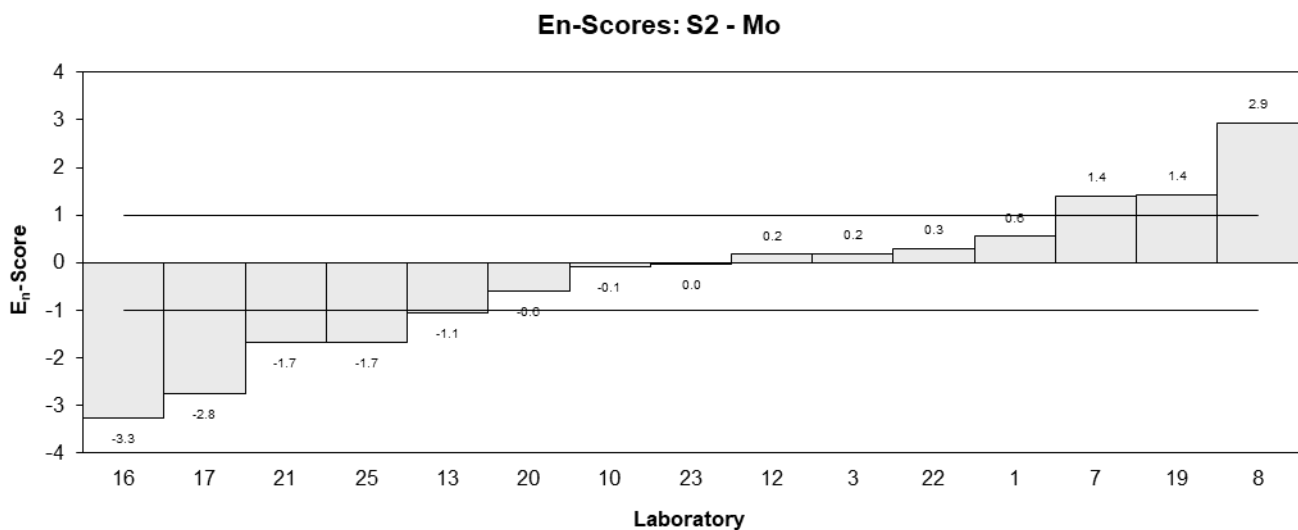
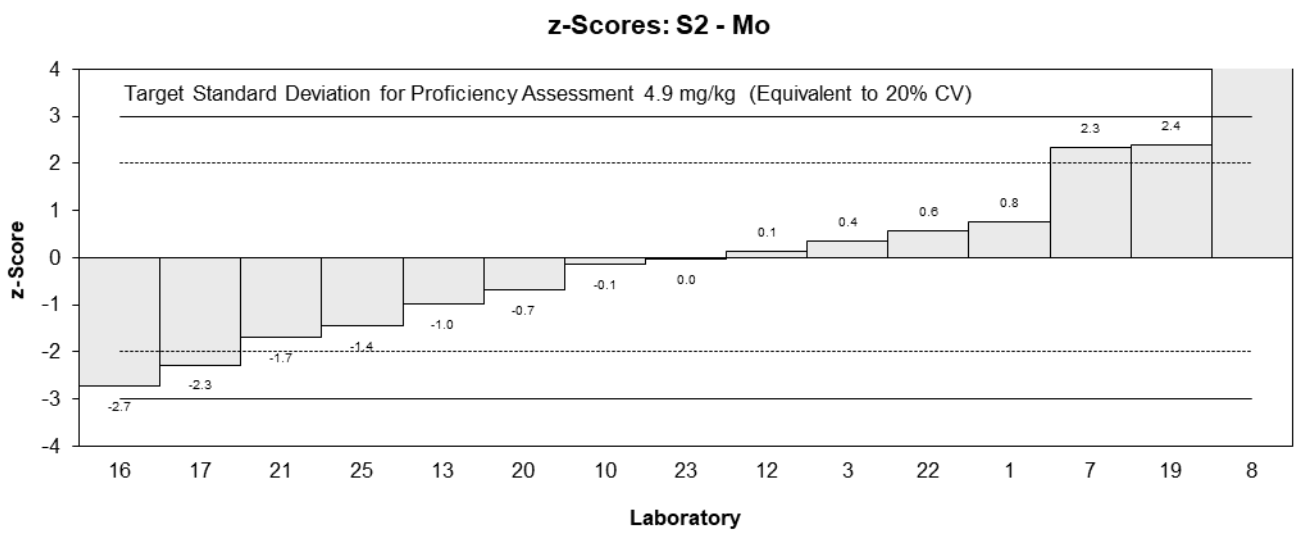
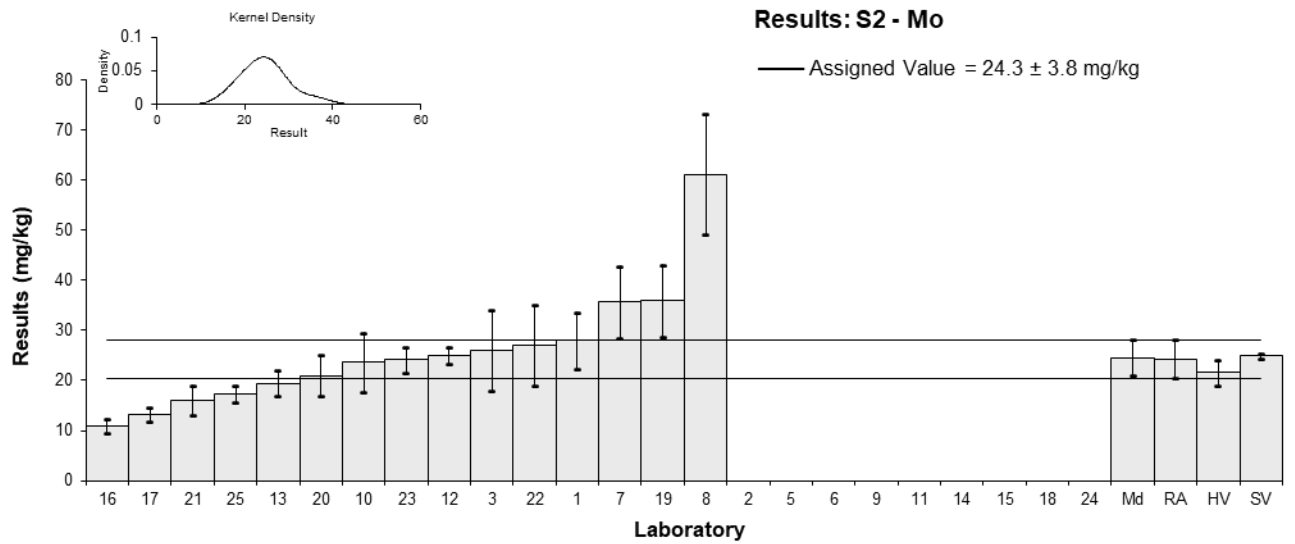


Figure 32

Table 44

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	39	7.8	1.09	0.67
2	NT	NT		
3	35	10	0.47	0.25
5	NR	NR		
6	NT	NT		
7	44.47	9.51	1.95	1.06
8**	96.6	20	10.09	3.05
9	NT	NT		
10	30.7	7.67	-0.20	-0.13
11	NT	NT		
12	37	2.59	0.78	0.67
13	18.65128	2.05	-2.09	-1.83
14	NR	NR		
15	NT	NT		
16**	14	1.512	-2.81	-2.51
17**	20.2	2.0	-1.84	-1.62
18	NT	NT		
19**	47.1	9.42	2.36	1.29
20	28	6	-0.62	-0.43
21**	17.3	2.6	-2.30	-1.97
22	33	10	0.16	0.08
23	33.1	3.5	0.17	0.14
24	NT	NT		
25	21.12977	2.11	-1.70	-1.49

** Gross Error, see Section 4.2

Statistics

Assigned Value	32.0	7.0
Spike Value	34.9	0.9
Homogeneity Value	31.0	3.7
Robust Average	32.0	7.0
Median	33.1	5.3
Mean	32.0	
N	10	
Max	44.47	
Min	18.65128	
Robust SD	8.9	
Robust CV	28%	

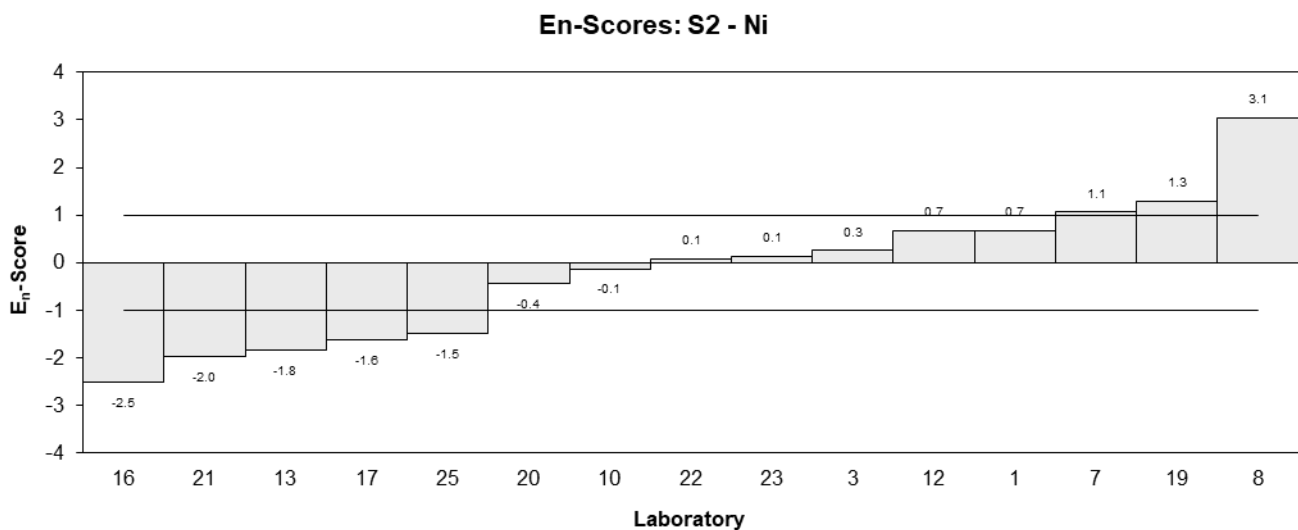
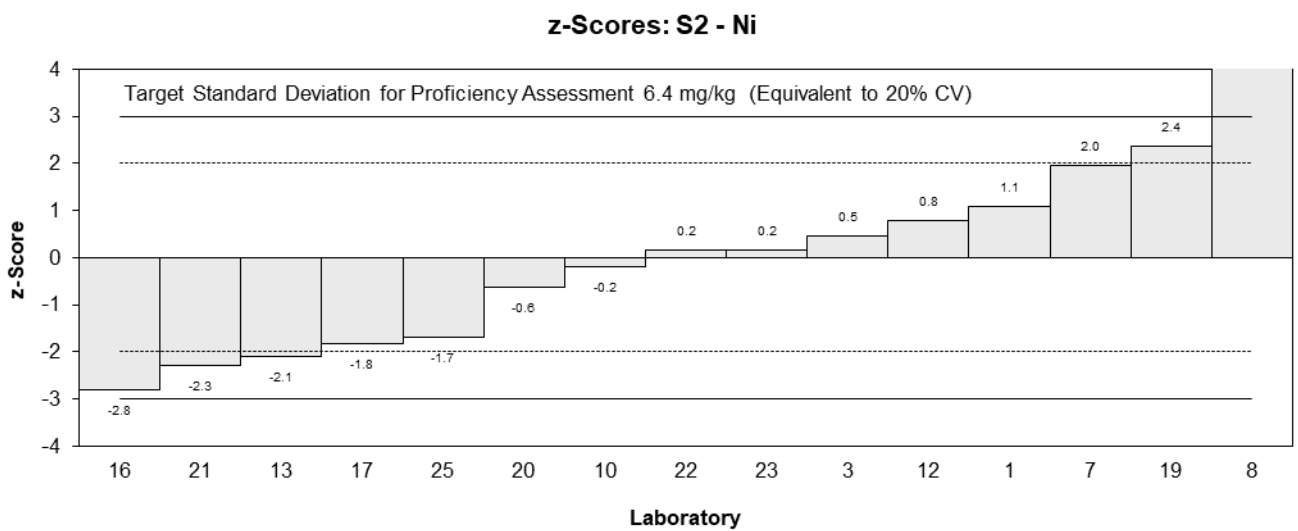
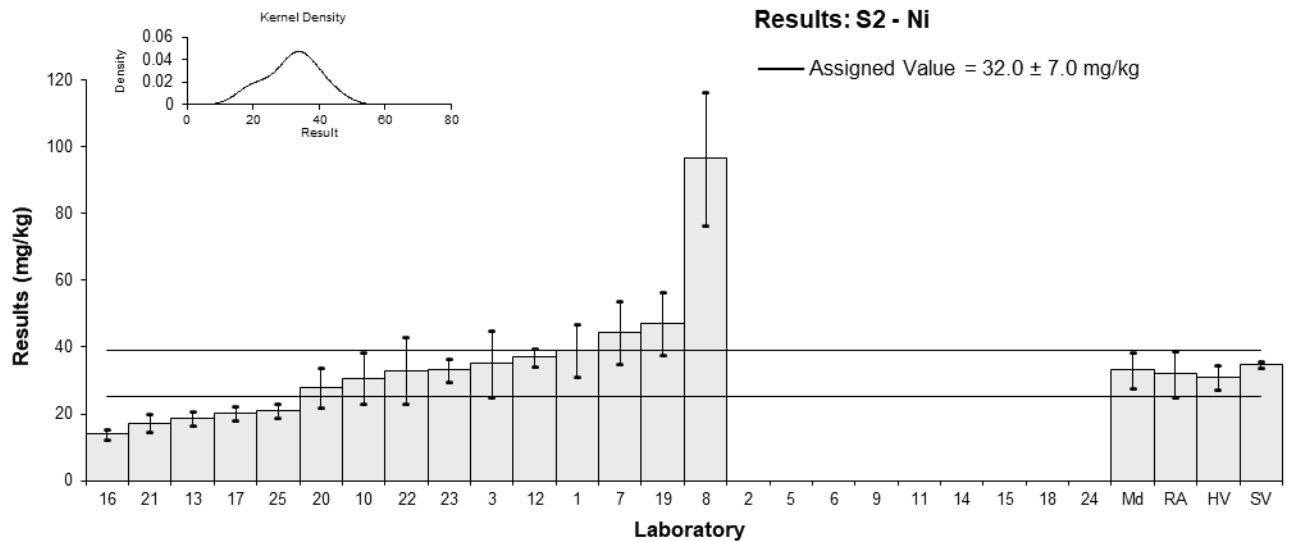


Figure 33

Table 45

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	55	10	1.35	0.95
2	NT	NT		
3	46	12	0.31	0.19
5	NR	NR		
6	NT	NT		
7	57.55	12.67	1.65	0.98
8**	119.3	30	8.78	2.47
9	NT	NT		
10	43.0	10.8	-0.03	-0.02
11	NT	NT		
12	45	2.25	0.20	0.23
13	37.66088	4.895	-0.65	-0.65
14	NR	NR		
15	NT	NT		
16**	20	2.72	-2.69	-3.06
17**	22.3	2.2	-2.42	-2.83
18	NT	NT		
19**	61.4	12.3	2.09	1.27
20	40	7	-0.38	-0.33
21**	29.3	4.4	-1.62	-1.68
22	34	10	-1.07	-0.76
23	42.9	4.5	-0.05	-0.05
24	NT	NT		
25	32.88306	3.29	-1.20	-1.33

** Gross Error, see Section 4.2

Statistics

Assigned Value	43.3	7.1
Spike Value	42.9	0.9
Homogeneity Value	39.3	4.7
Robust Average	43.3	7.1
Median	43.0	4.9
Mean	43.4	
N	10	
Max	57.55	
Min	32.88306	
Robust SD	9	
Robust CV	21%	

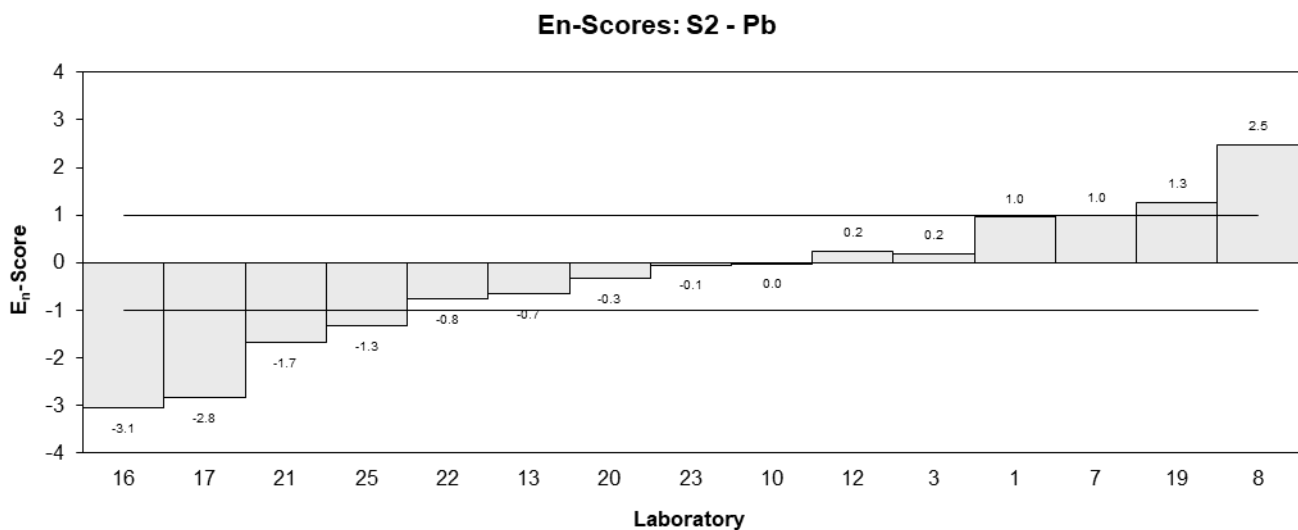
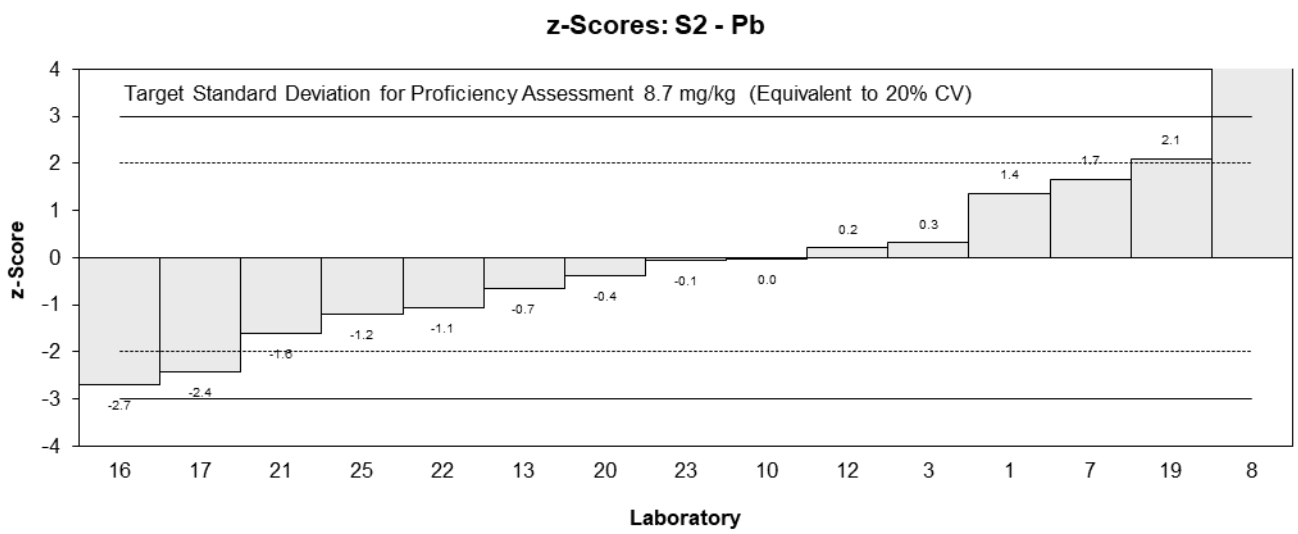
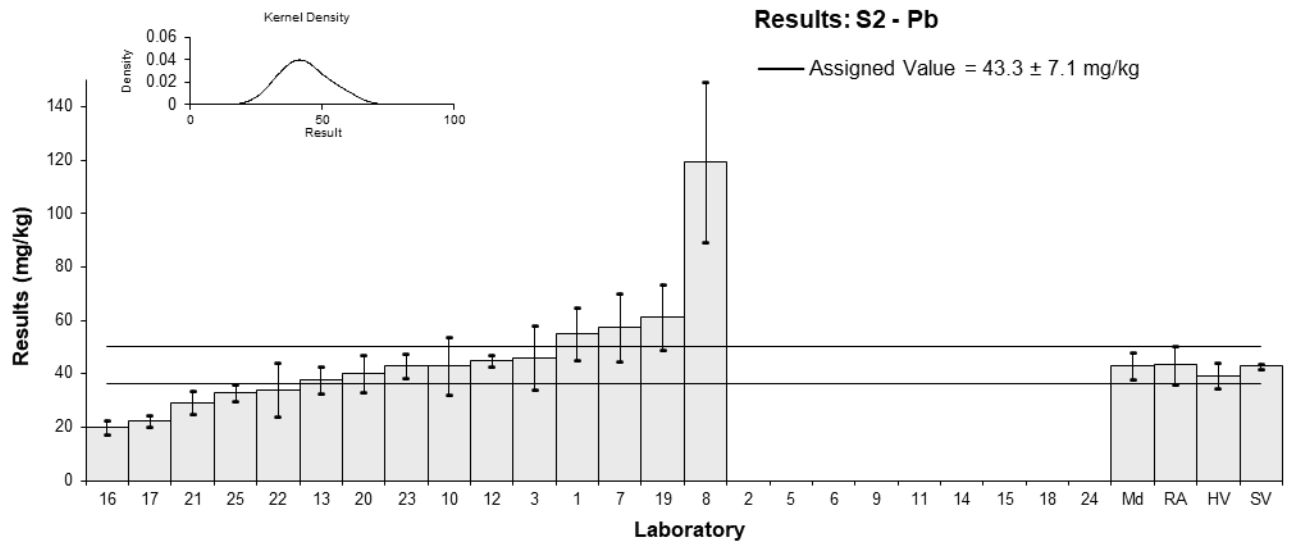


Figure 34

Table 46

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	4.2	0.84	0.93	0.57
2	NT	NT		
3	2.9	3	-0.90	-0.21
5	NR	NR		
6	NT	NT		
7	4.69	0.95	1.62	0.93
8	NT	NT		
9	NT	NT		
10	2.89	0.723	-0.92	-0.60
11	NT	NT		
12	<5	0.15		
13	3.68343	0.63	0.20	0.14
14	NR	NR		
15	NT	NT		
16**	3	0.723	-0.76	-0.50
17**	1.84	0.18	-2.40	-2.07
18	NT	NT		
19**	5.83	1.17	3.23	1.62
20	<2	2		
21	<20	14		
22	4	3	0.65	0.15
23	3.67	0.4	0.18	0.15
24	NT	NT		
25	2.25367	0.23	-1.82	-1.55

** Gross Error, see Section 4.2

Statistics

Assigned Value	3.54	0.80
Spike Value	3.49	0.07
Homogeneity Value	2.98	0.36
Robust Average	3.54	0.80
Median	3.68	0.85
Mean	3.54	
N	8	
Max	4.69	
Min	2.25367	
Robust SD	0.91	
Robust CV	26%	

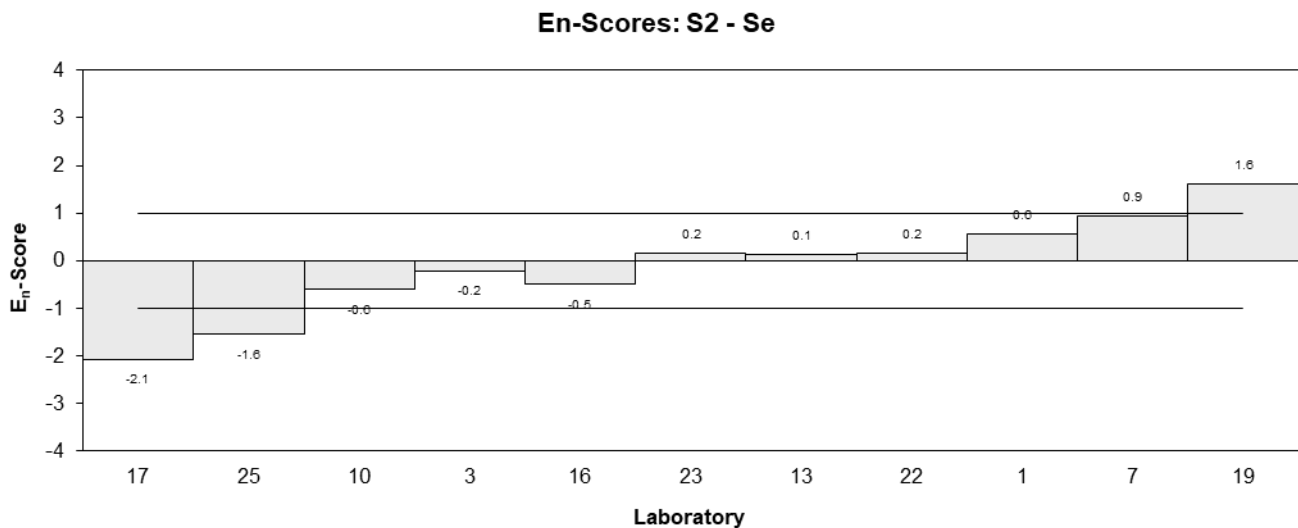
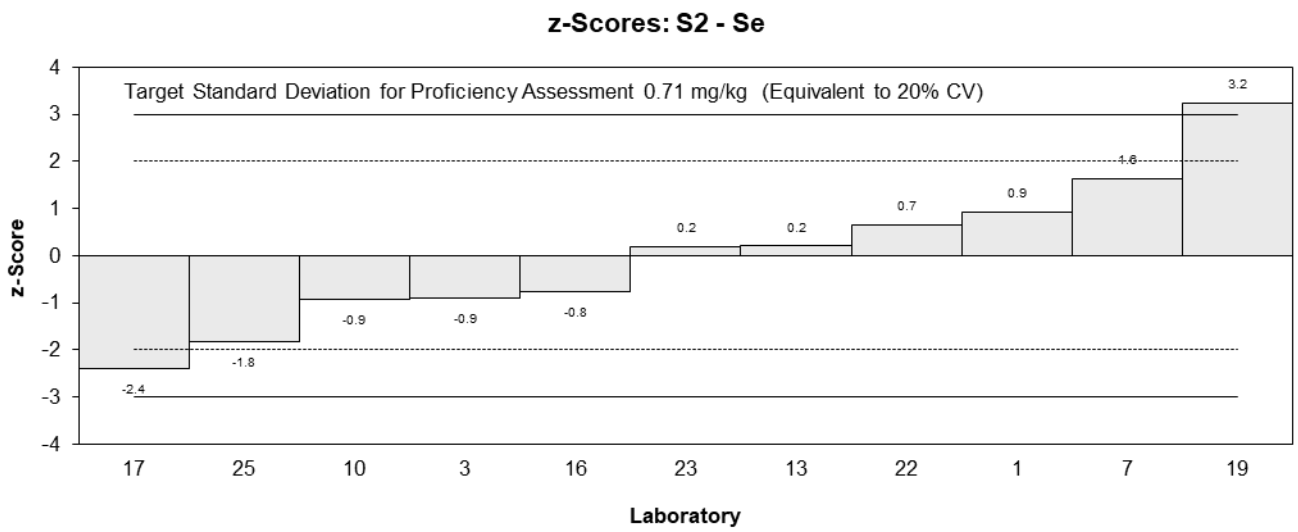
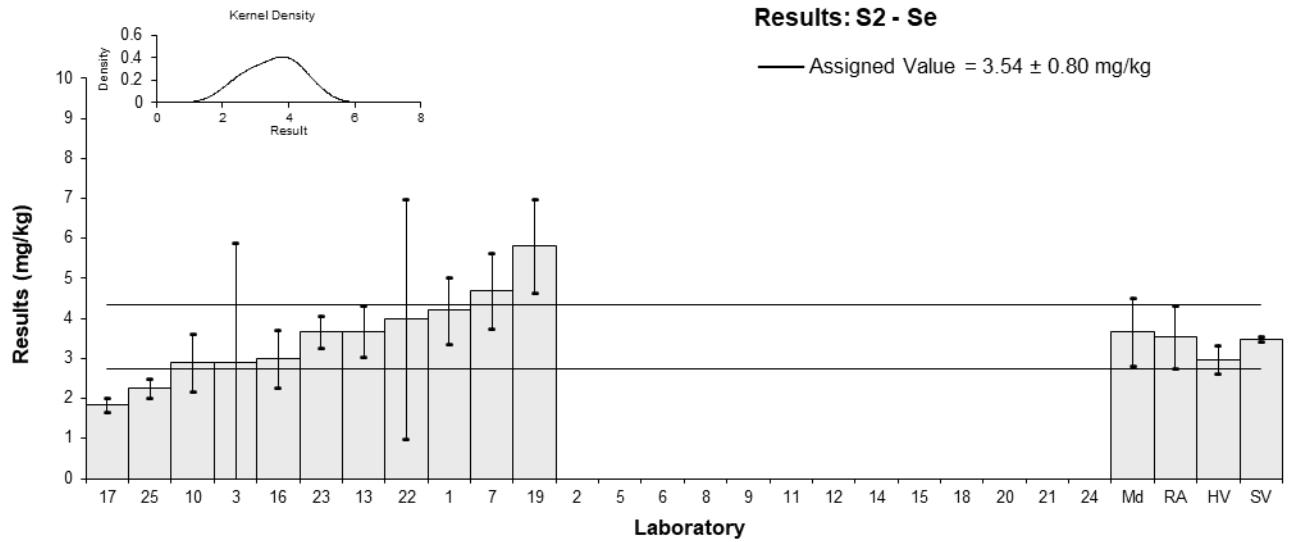


Figure 35

Table 47

Sample Details

Sample No.	S2
Matrix	Sludge
Analyte	Sr
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	13	2.6	1.44	0.82
2	NT	NT		
3	12	5	0.94	0.34
5	NR	NR		
6	NT	NT		
7	13.19	2.80	1.53	0.84
8**	31.5	10	10.59	2.08
9	NT	NT		
10	6.95	1.74	-1.56	-1.06
11	NT	NT		
12	13	0.78	1.44	1.15
13	8.38332	1.2	-0.85	-0.64
14	NR	NR		
15	NT	NT		
16**	3.3	0.8646	-3.37	-2.67
17**	6.27	0.63	-1.90	-1.54
18	NT	NT		
19**	23.1	4.63	6.44	2.49
20	8.6	3	-0.74	-0.39
21**	4.52	0.8	-2.76	-2.21
22	10	3	-0.05	-0.03
23	10.0	1.2	-0.05	-0.04
24	NT	NT		
25	5.28664	0.53	-2.38	-1.96

** Gross Error, see Section 4.2

Statistics

Assigned Value	10.1	2.4
Spike Value	Not Spiked	
Homogeneity Value	11.2	1.3
Robust Average	10.1	2.4
Median	10.0	2.9
Mean	10.0	
N	10	
Max	13.19	
Min	5.28664	
Robust SD	3.1	
Robust CV	31%	

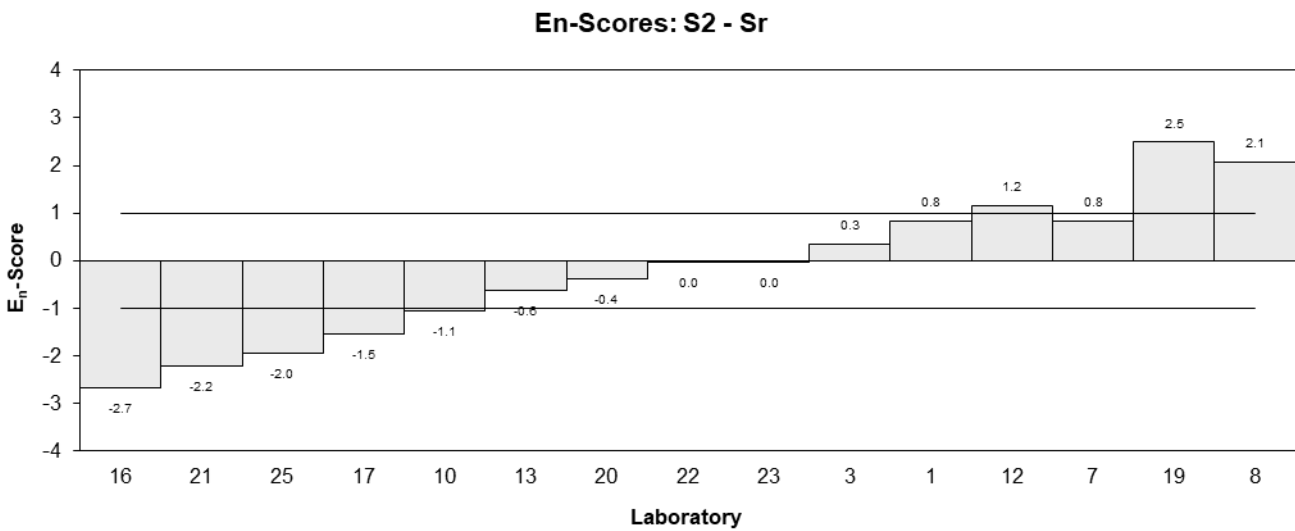
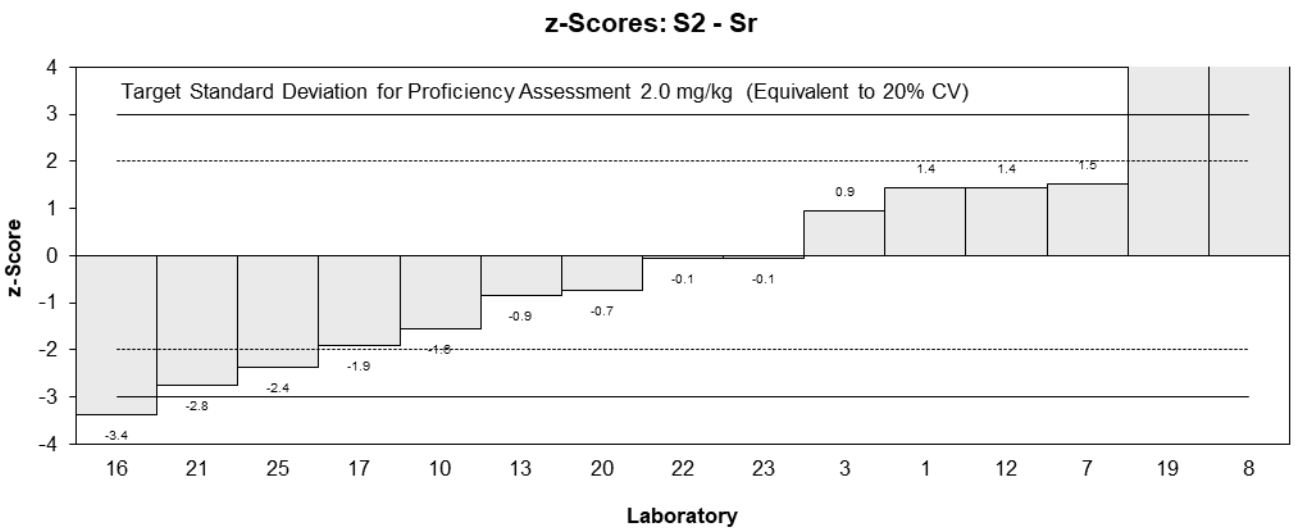
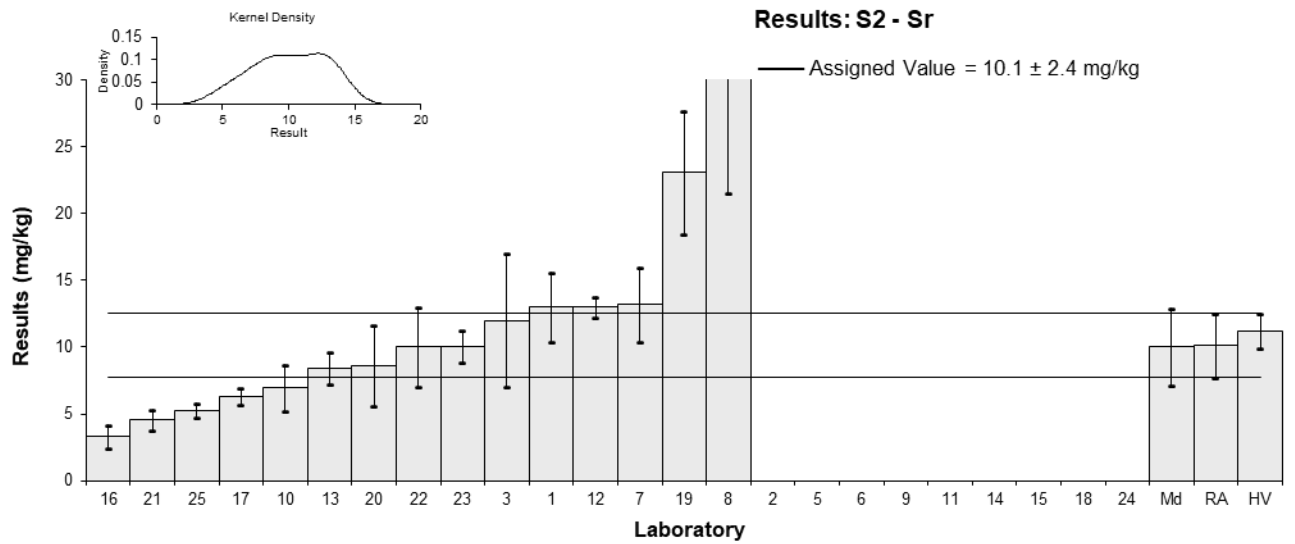


Figure 36

Table 48

Sample Details

Sample No.	S2
Matrix	Sludge
Analyte	Th
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	4.0	0.80	2.25	1.28
2	NT	NT		
3	2.8	1	0.07	0.04
5	NR	NR		
6	NT	NT		
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	2.29	0.575	-0.85	-0.60
11	NT	NT		
12	NT	NT		
13	<5	NR		
14	NR	NR		
15	NT	NT		
16	NT	NT		
17	NT	NT		
18	NT	NT		
19	NT	NT		
20	2.5	0.7	-0.47	-0.29
21	NT	NT		
22	3	1	0.43	0.21
23	2.42	0.5	-0.62	-0.46
24	NT	NT		
25	NT	NT		

Statistics

Assigned Value	2.76	0.54
Spike Value	2.70	0.20
Homogeneity Value	2.78	0.33
Robust Average	2.76	0.54
Median	2.65	0.44
Mean	2.84	
N	6	
Max	4	
Min	2.29	
Robust SD	0.53	
Robust CV	19%	

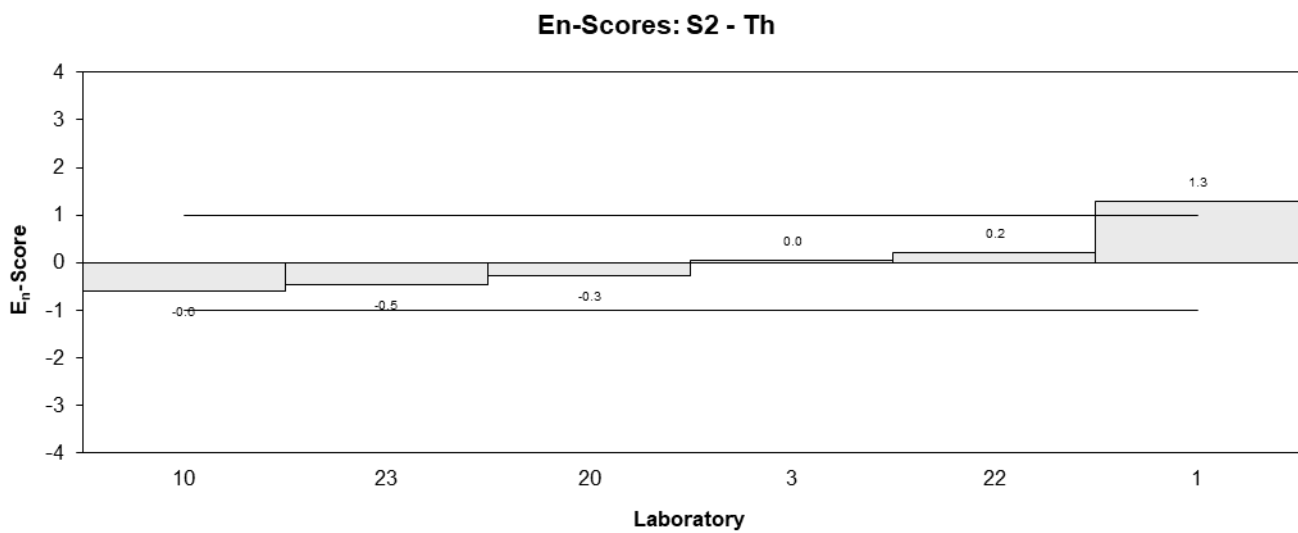
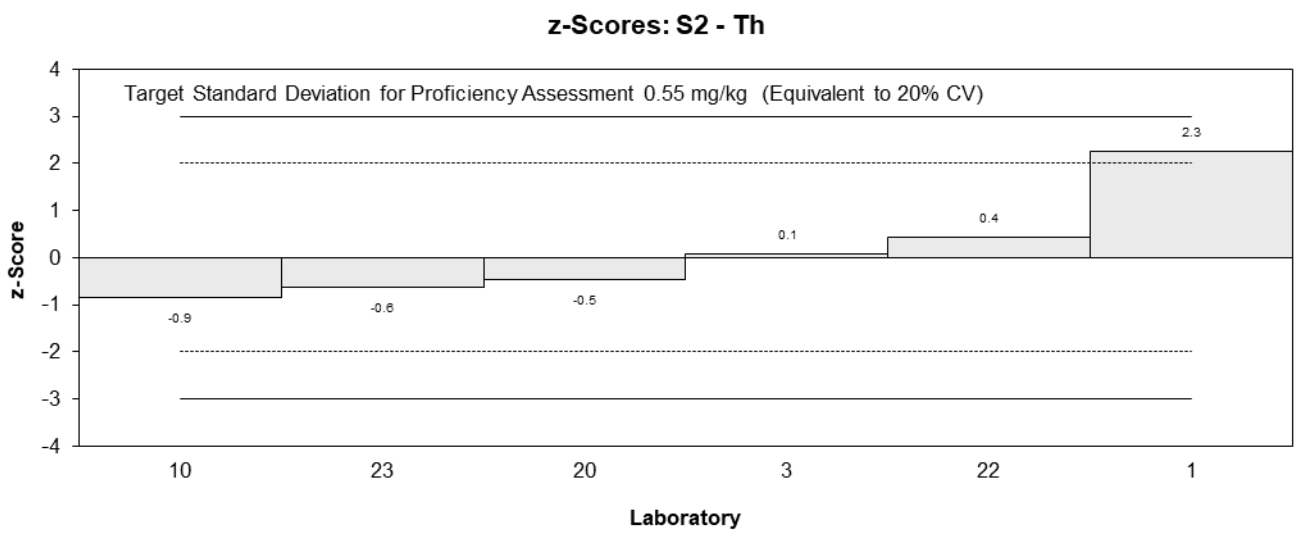
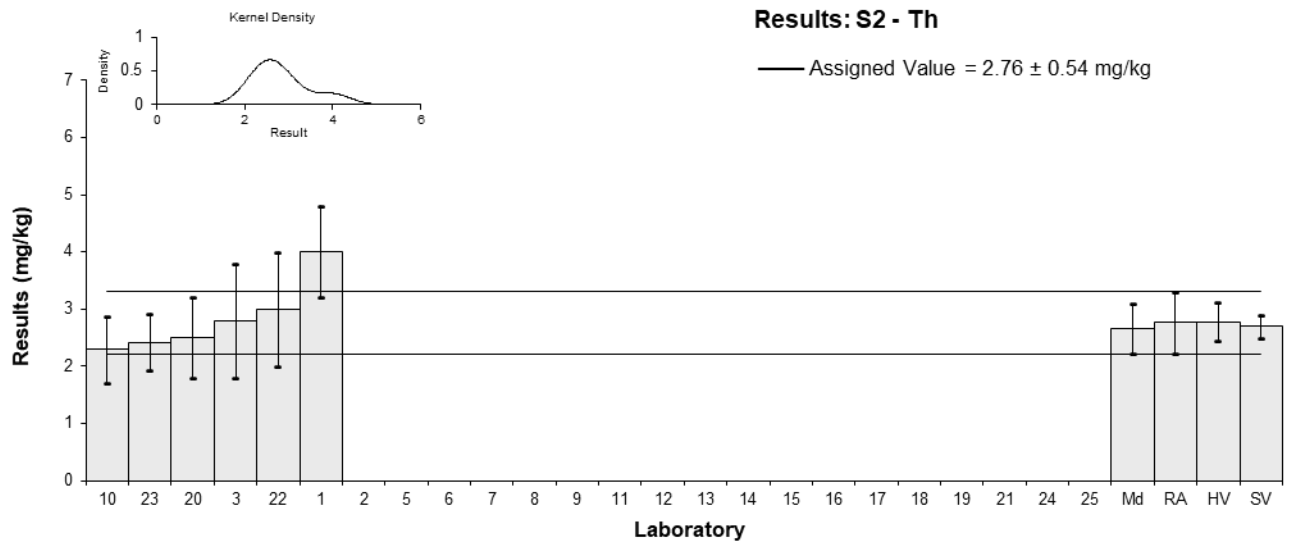


Figure 37

Table 49

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	3.5	0.70	0.45	0.27
2	NT	NT		
3	4	2	1.23	0.37
5	NR	NR		
6	NT	NT		
7	< 10	NR		
8	NT	NT		
9	NT	NT		
10	2.87	0.718	-0.53	-0.31
11	NT	NT		
12	NT	NT		
13	<5	NR		
14	NR	NR		
15	NT	NT		
16	NT	NT		
17**	1.92	0.19	-2.01	-1.55
18	NT	NT		
19	<10	NR		
20	2.8	0.7	-0.64	-0.38
21**	1.91	0.14	-2.02	-1.58
22	4.1	1	1.39	0.69
23	3.30	0.5	0.14	0.09
24	NT	NT		
25	1.8296	0.18	-2.15	-1.66

** Gross Error, see Section 4.2

Statistics

Assigned Value	3.21	0.81
Spike Value	3.00	0.06
Homogeneity Value	2.78	0.33
Robust Average	3.21	0.81
Median	3.30	0.70
Mean	3.20	
N	7	
Max	4.1	
Min	1.8296	
Robust SD	0.86	
Robust CV	27%	

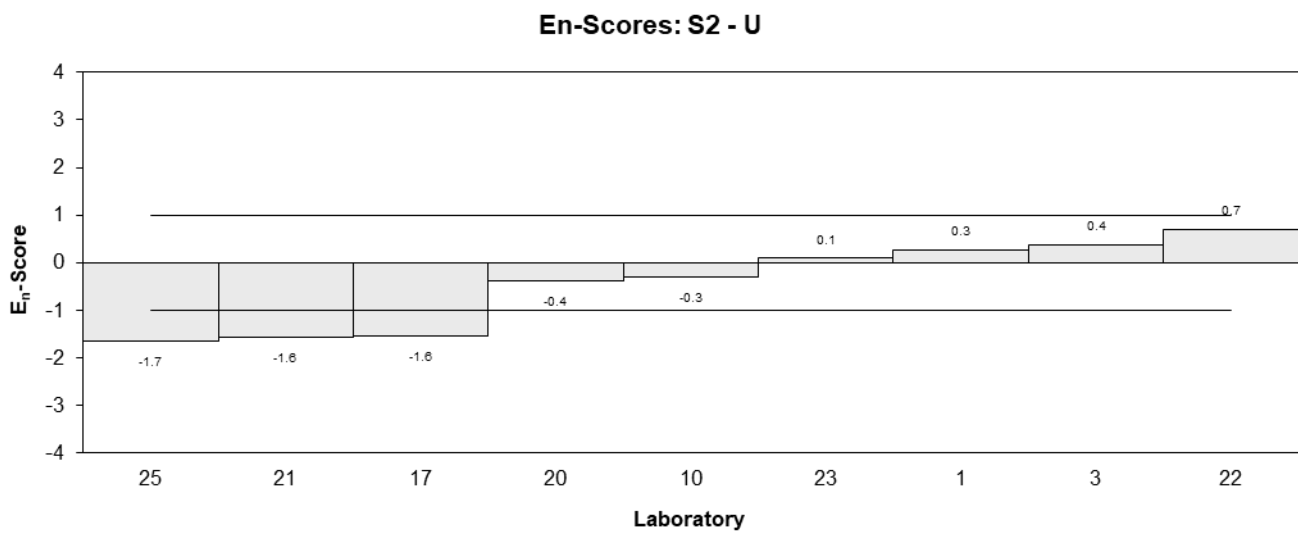
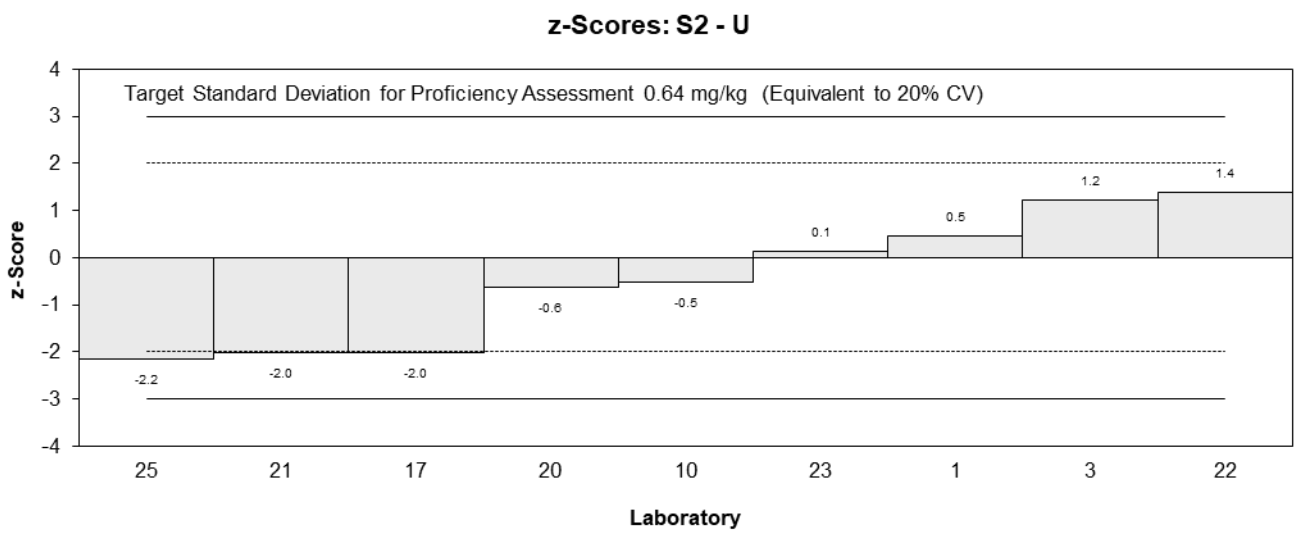
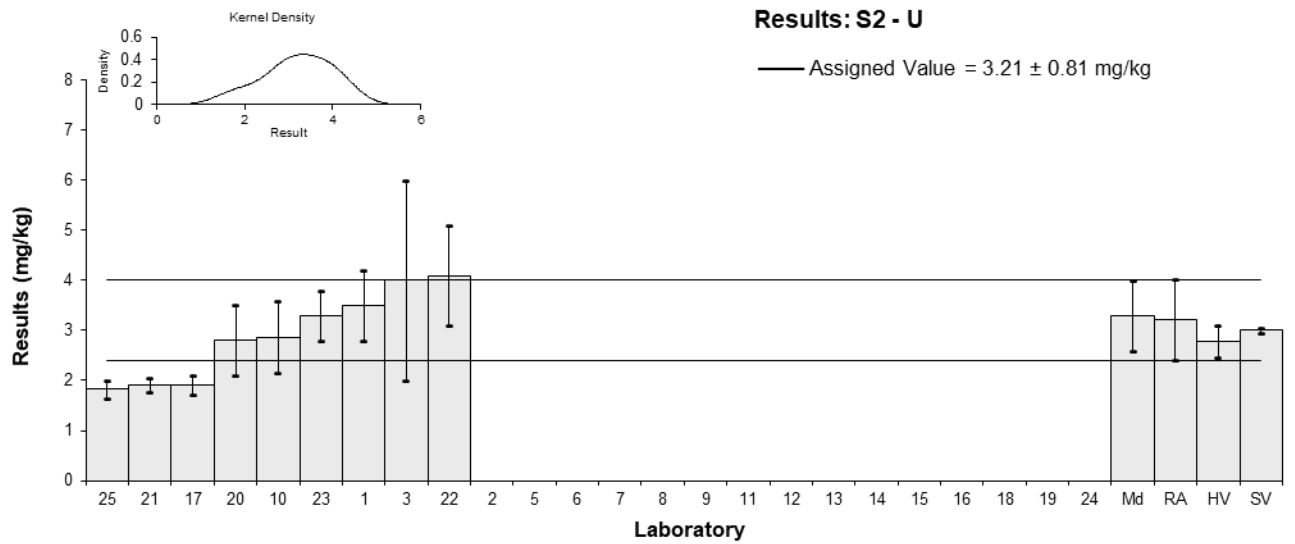


Figure 38

Table 50

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	78	16	1.29	0.82
2	NT	NT		
3	65	20	0.24	0.13
5	NR	NR		
6	NT	NT		
7	84.39	17.73	1.81	1.07
8**	180.5	35	9.56	3.23
9	NT	NT		
10	55.5	13.9	-0.52	-0.37
11	NT	NT		
12	66	2.64	0.32	0.35
13	50.34103	5.54	-0.94	-0.95
14	NR	NR		
15	NT	NT		
16**	27	4.86	-2.82	-2.91
17**	38.6	3.9	-1.89	-2.00
18	NT	NT		
19**	92.5	18.5	2.46	1.42
20	58	10	-0.32	-0.27
21**	36.3	3.7	-2.07	-2.21
22	63	20	0.08	0.04
23	61.0	6.0	-0.08	-0.08
24	NT	NT		
25	41.00652	4.1	-1.69	-1.79

** Gross Error, see Section 4.2

Statistics

Assigned Value	62	11
Spike Value	55.2	1.5
Homogeneity Value	56.4	6.8
Robust Average	62	11
Median	62.0	6.2
Mean	62.2	
N	10	
Max	84.39	
Min	41.00652	
Robust SD	14	
Robust CV	22%	

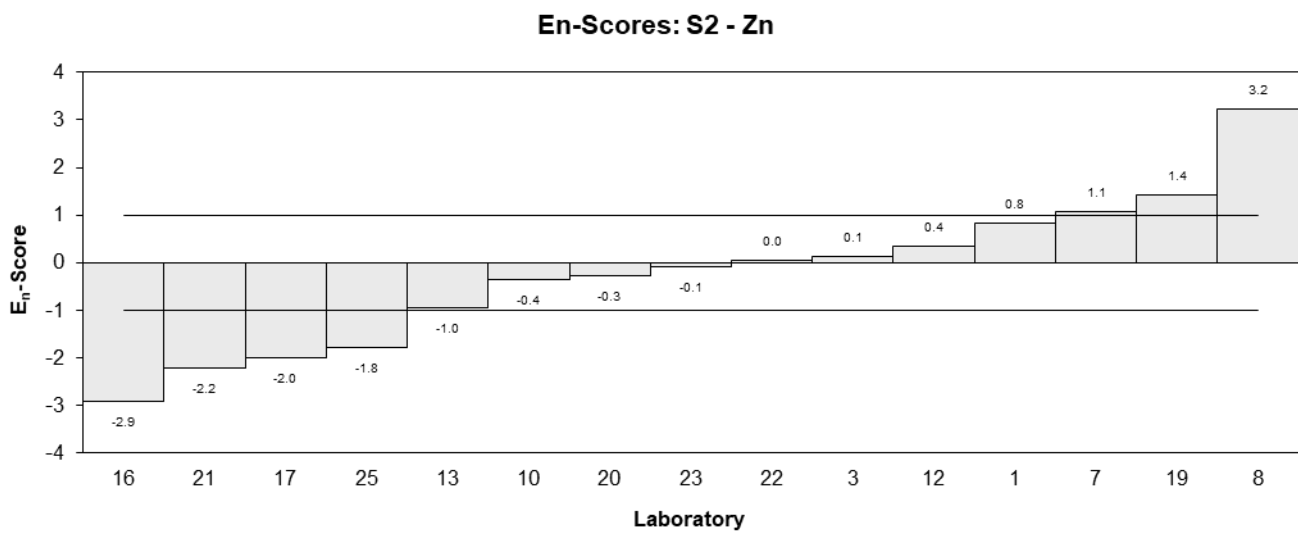
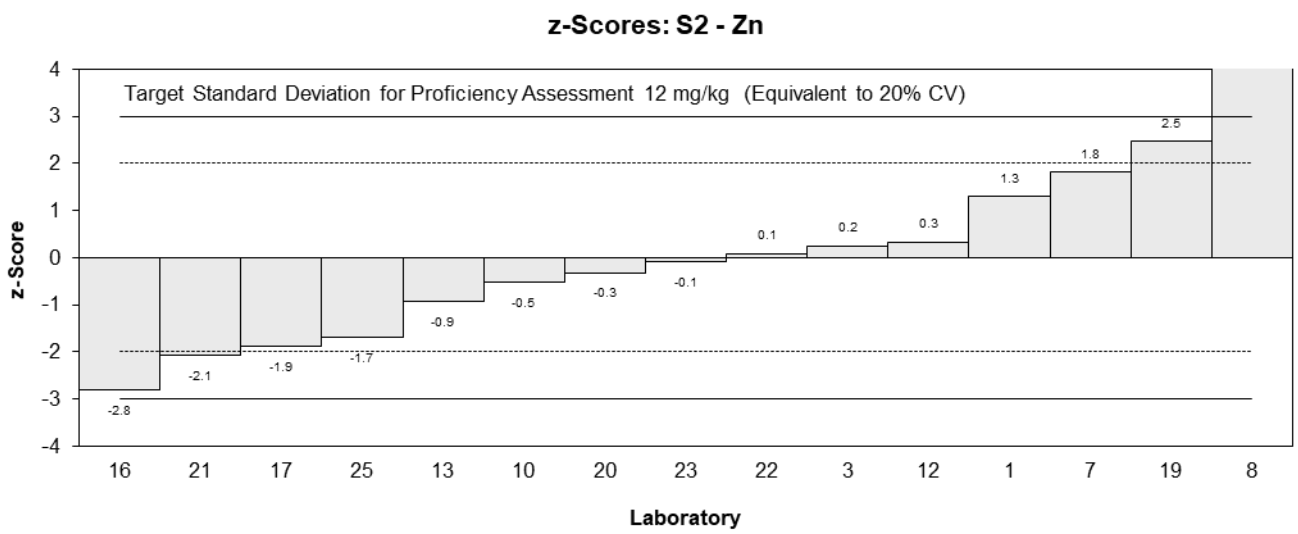
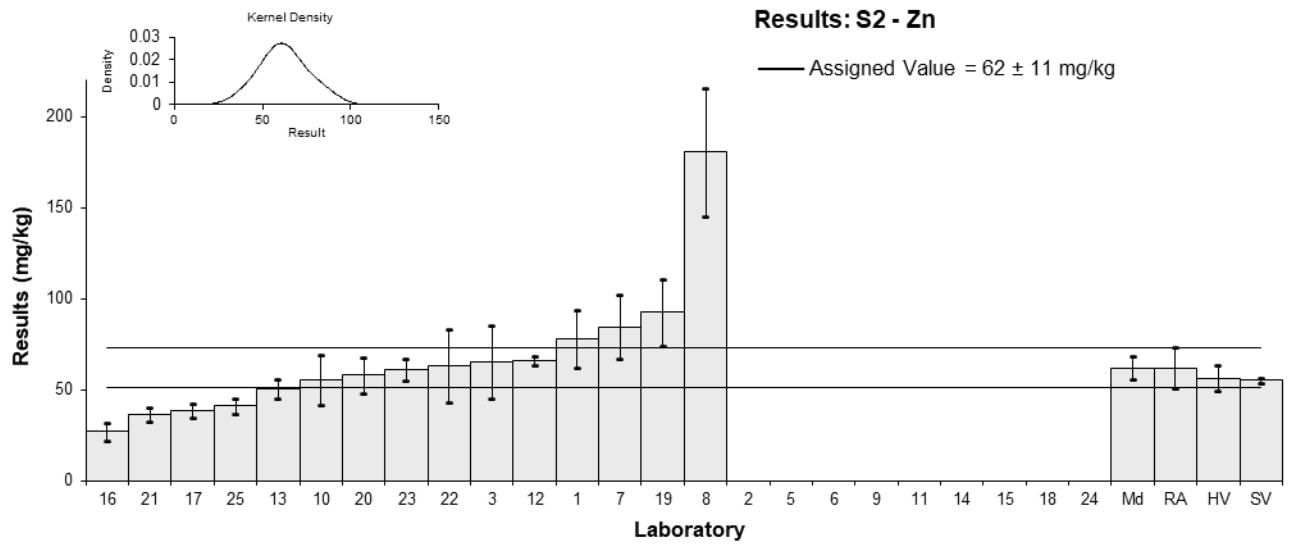


Figure 39

Table 51

Sample Details

Sample No.	S2
Matrix	Sludge
Analyte	Moisture Content
Unit	%

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	35.1	7.0	-1.17	-1.14
2	NT	NT		
3	49	10	0.35	0.27
5	43	NR	-0.31	-0.44
6	NT	NT		
7	60.91	14.84	1.65	0.94
8**	78.9	10	3.61	2.80
9	NT	NT		
10	44.3	11.1	-0.16	-0.12
11	NT	NT		
12	45.7	NR	-0.01	-0.02
13	NR	NR		
14	NR	NR		
15	NT	NT		
16**	95	1.5105	5.37	7.59
17**	45	5	-0.09	-0.10
18	NT	NT		
19**	60.3	12.1	1.58	1.06
20	50	10	0.46	0.36
21**	24	NR	-2.38	-3.46
22	50	5	0.46	0.52
23	38.2	2.0	-0.83	-1.15
24	NT	NT		
25	NR	NR		

** Gross Error, see Section 4.2

Statistics

Assigned Value	45.8	6.3
Spike Value	43.0	0.9
Homogeneity Value	37.2	3.7
Robust Average	45.8	6.3
Median	45.7	5.3
Mean	46.2	
N	9	
Max	60.91	
Min	35.1	
Robust SD	7.6	
Robust CV	17%	

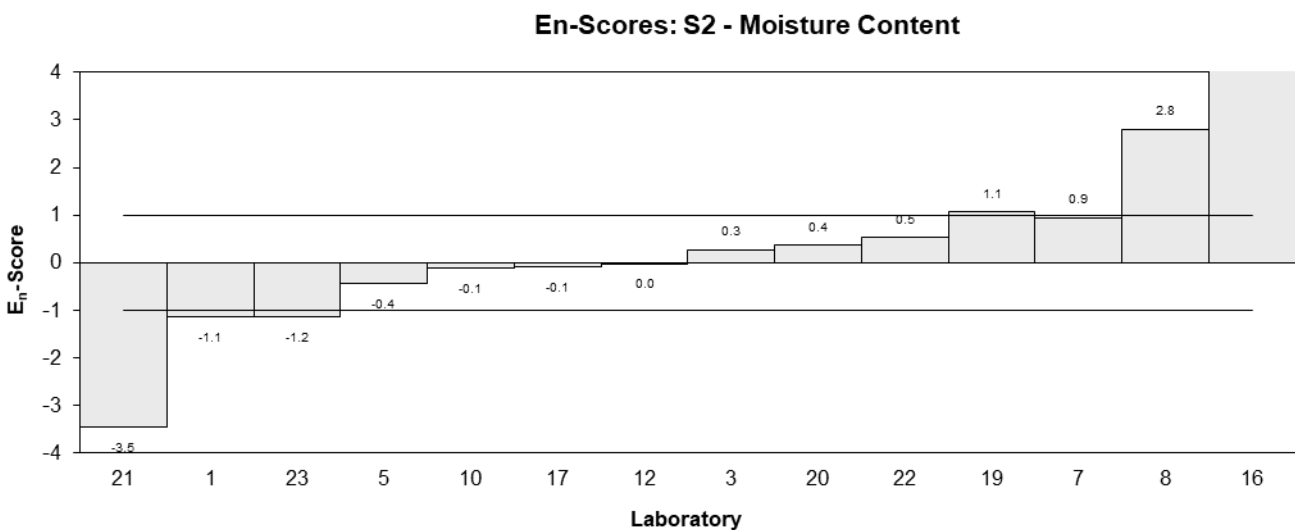
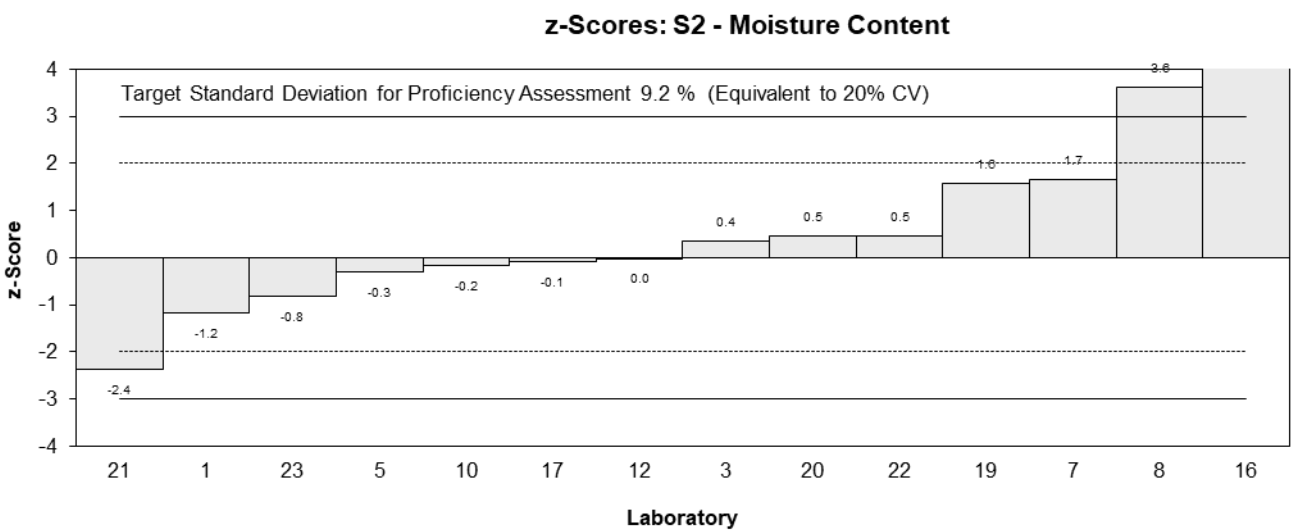
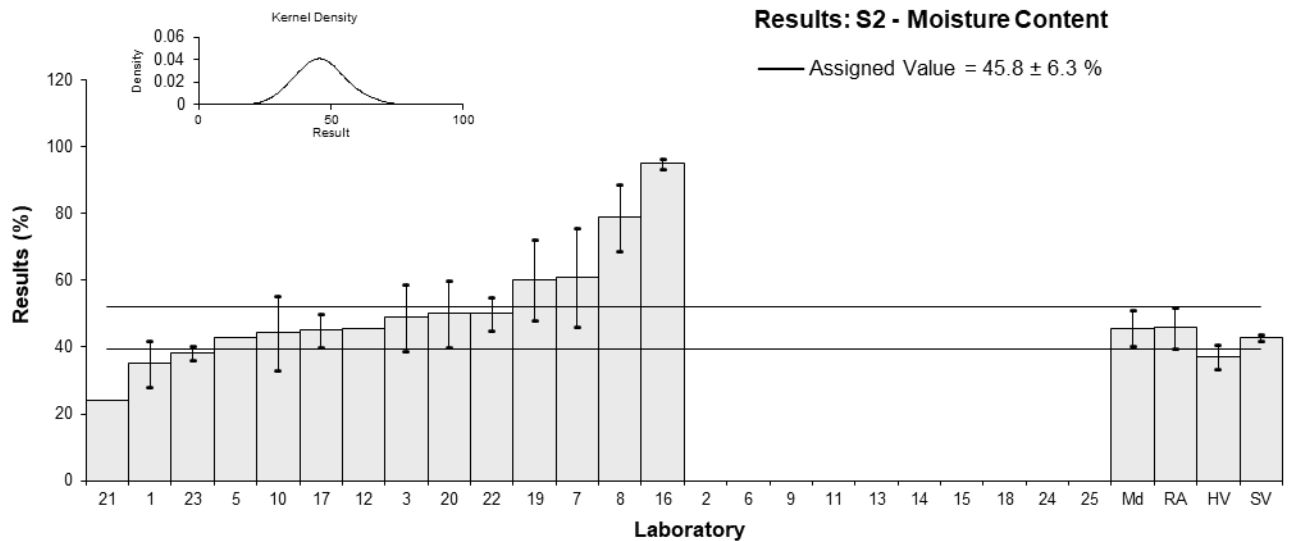


Figure 40

Table 52

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	5330	1066	0.23	0.11
2	NR	NR		
3	4700	1000	-0.98	-0.49
5	5654	592	0.85	0.68
6	5120	620	-0.17	-0.13
7	NT	NT		
8	5850	1000	1.23	0.62
9	NT	NT		
10	5203	520	-0.01	-0.01
11	4300	260	-1.75	-2.38
12	5700	456	0.94	0.92
13	5246.975	1049	0.07	0.03
14	5450	450	0.46	0.45
15	5097	927	-0.22	-0.12
16	5200	665.6	-0.02	-0.01
17	NT	NT		
18	4750	1188	-0.88	-0.38
19	NT	NT		
20	5600	2000	0.75	0.19
21	NT	NT		
22	5100	1000	-0.21	-0.11
23	5544	550	0.64	0.54
24	4430.2	443.02	-1.50	-1.49
25	NT	NT		

Statistics

Assigned Value	5210	280
Spike Value	Not Spiked	
Homogeneity Value	5110	610
Robust Average	5210	280
Median	5200	310
Mean	5190	
N	17	
Max	5850	
Min	4300	
Robust SD	460	
Robust CV	8.8%	

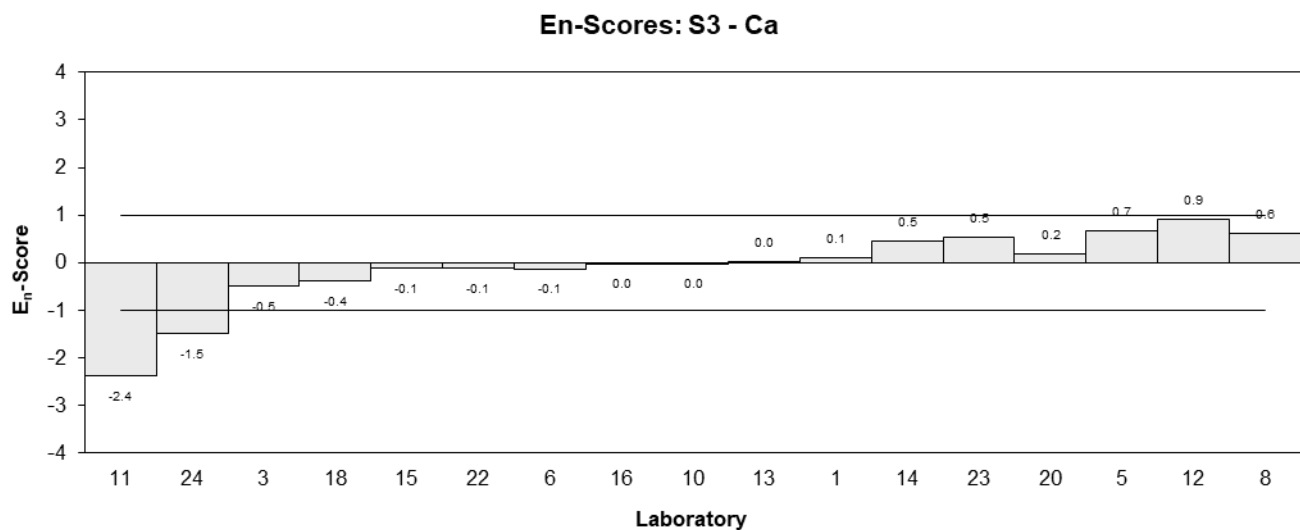
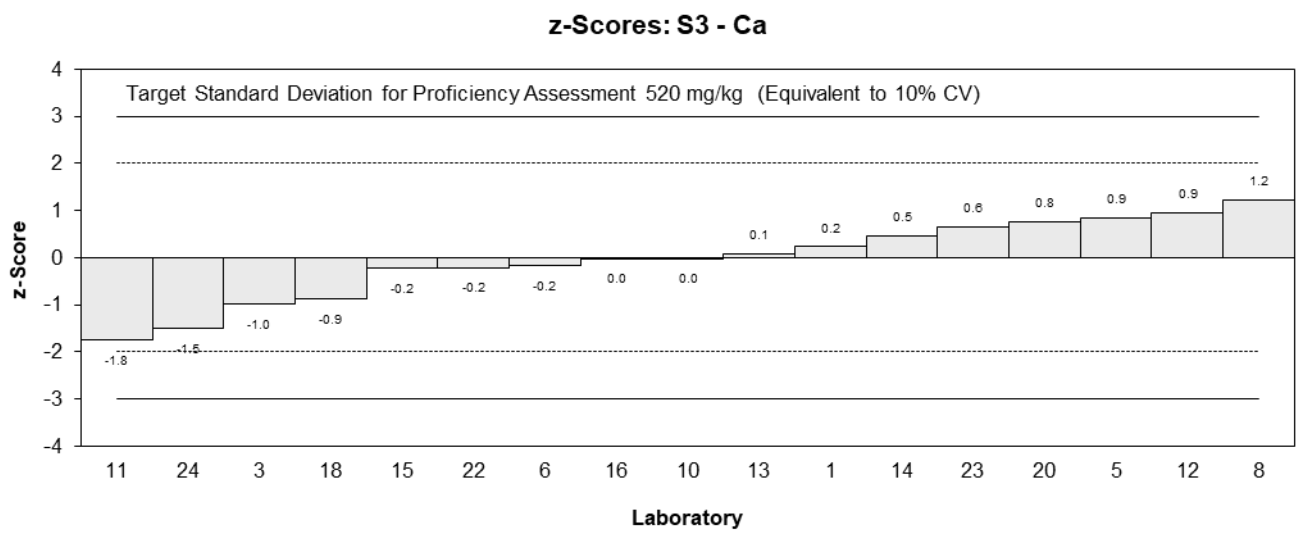
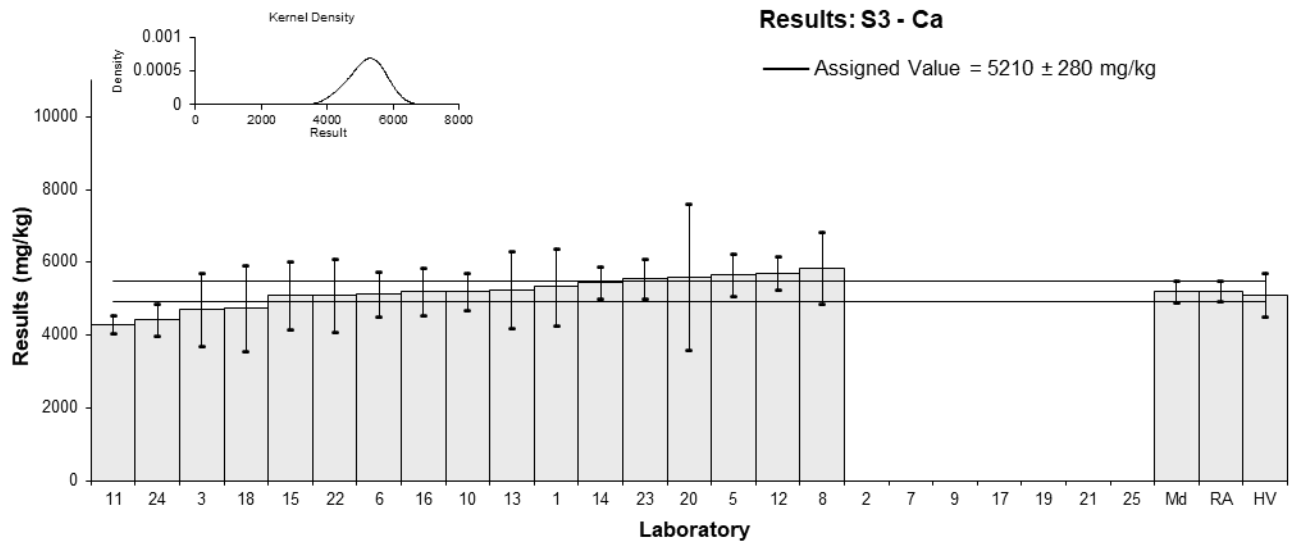


Figure 41

Table 53

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	26800	5360	-0.31	-0.22
2	NR	NR		
3	28000	5000	-0.02	-0.02
5	23840	3940	-1.01	-0.90
6	31000	5000	0.69	0.51
7	NT	NT		
8	31400	6000	0.78	0.50
9	NT	NT		
10	34800	348	1.59	2.55
11	NR	NR		
12	34000	2040	1.40	1.79
13	22781.69514	3417	-1.26	-1.24
14	26800	1300	-0.31	-0.45
15	27703	3630	-0.09	-0.09
16	20000	3300	-1.92	-1.93
17	NT	NT		
18	29000	7250	0.21	0.12
19	NT	NT		
20	29000	4000	0.21	0.19
21	NT	NT		
22	30000	5000	0.45	0.34
23	28976	2900	0.21	0.22
24	24723.1	2472.31	-0.80	-0.94
25	NT	NT		

Statistics

Assigned Value	28100	2600
Spike Value	Not Spiked	
Homogeneity Value	27900	3300
Robust Average	28100	2600
Median	28500	1900
Mean	28100	
N	16	
Max	34800	
Min	20000	
Robust SD	4100	
Robust CV	15%	

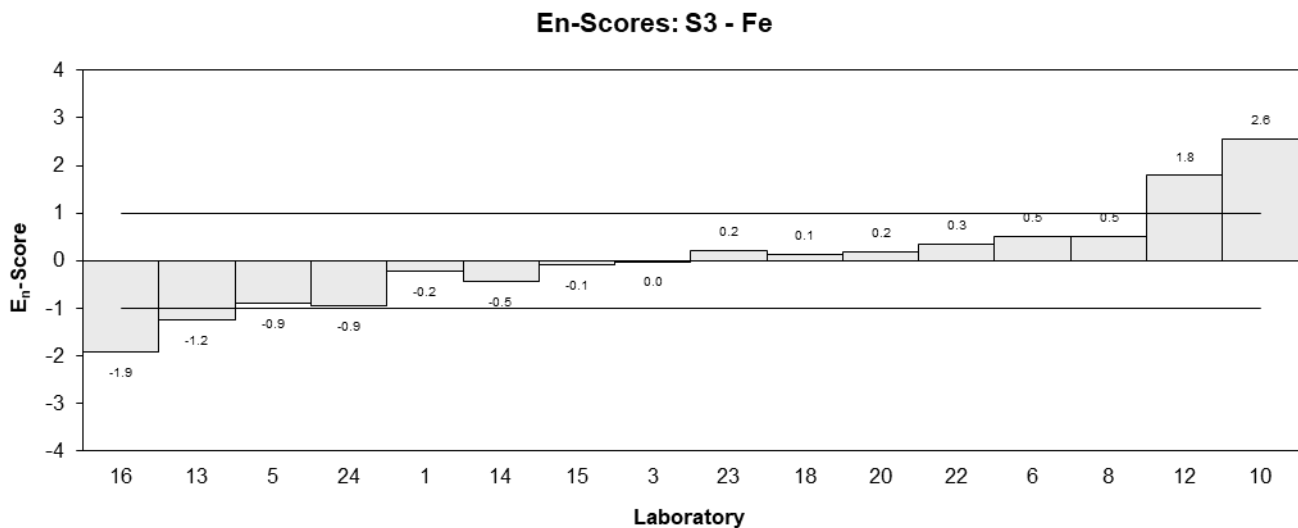
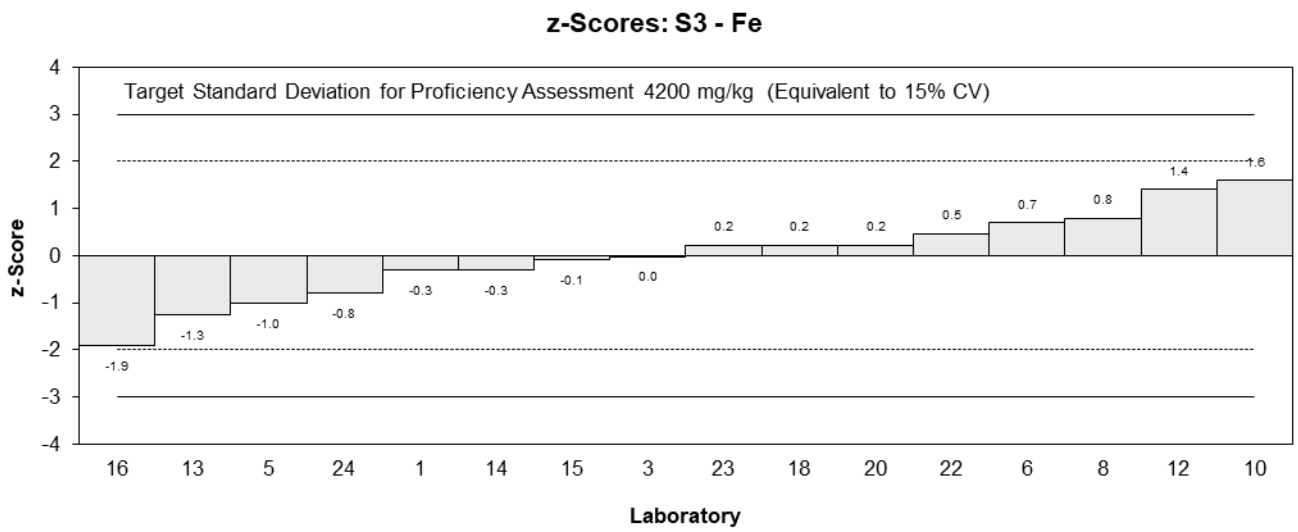
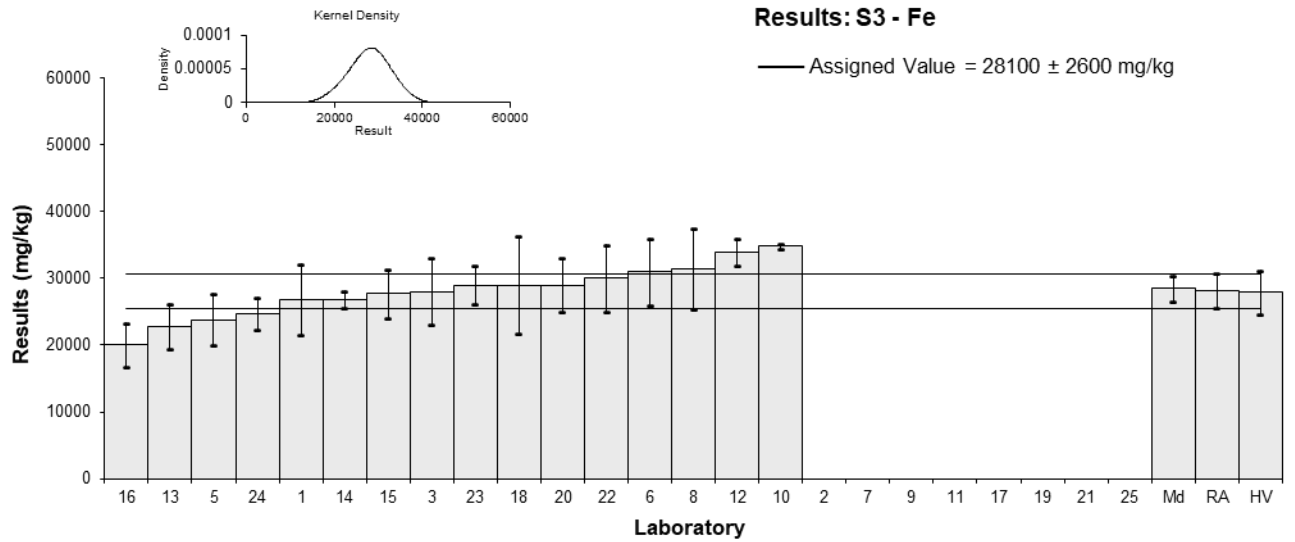


Figure 42

Table 54

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	4980	996	-0.26	-0.18
2	NR	NR		
3	4900	1000	-0.36	-0.24
5	4640	596	-0.69	-0.66
6	5300	1400	0.15	0.08
7	NT	NT		
8	5245	1000	0.08	0.06
9	NT	NT		
10	5270	527	0.12	0.12
11	NR	NR		
12	7000	560	2.34	2.30
13	6458.926	1098	1.65	1.04
14	4760	540	-0.54	-0.54
15	4734	637	-0.57	-0.53
16	4200	638.4	-1.26	-1.15
17	NT	NT		
18	5220	1305	0.05	0.03
19	NT	NT		
20	4100	1000	-1.39	-0.94
21	NT	NT		
22	4300	1000	-1.13	-0.77
23	5750	575	0.73	0.71
24	6570.6	657.06	1.79	1.61
25	NT	NT		

Statistics

Assigned Value	5180	560
Spike Value	Not Spiked	
Homogeneity Value	5220	630
Robust Average	5180	560
Median	5100	380
Mean	5210	
N	16	
Max	7000	
Min	4100	
Robust SD	890	
Robust CV	17%	

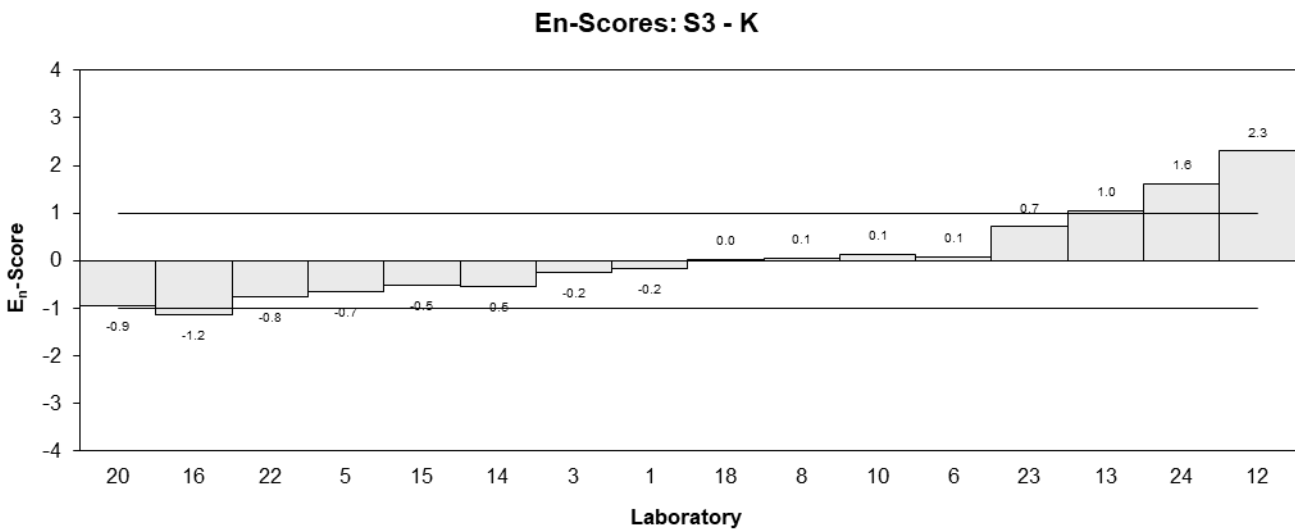
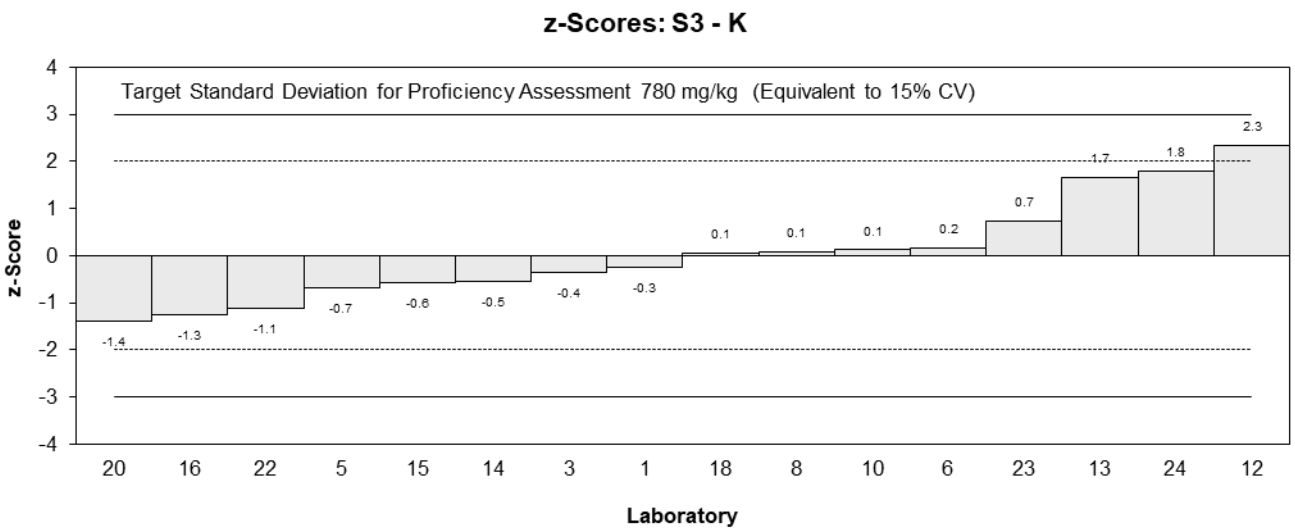
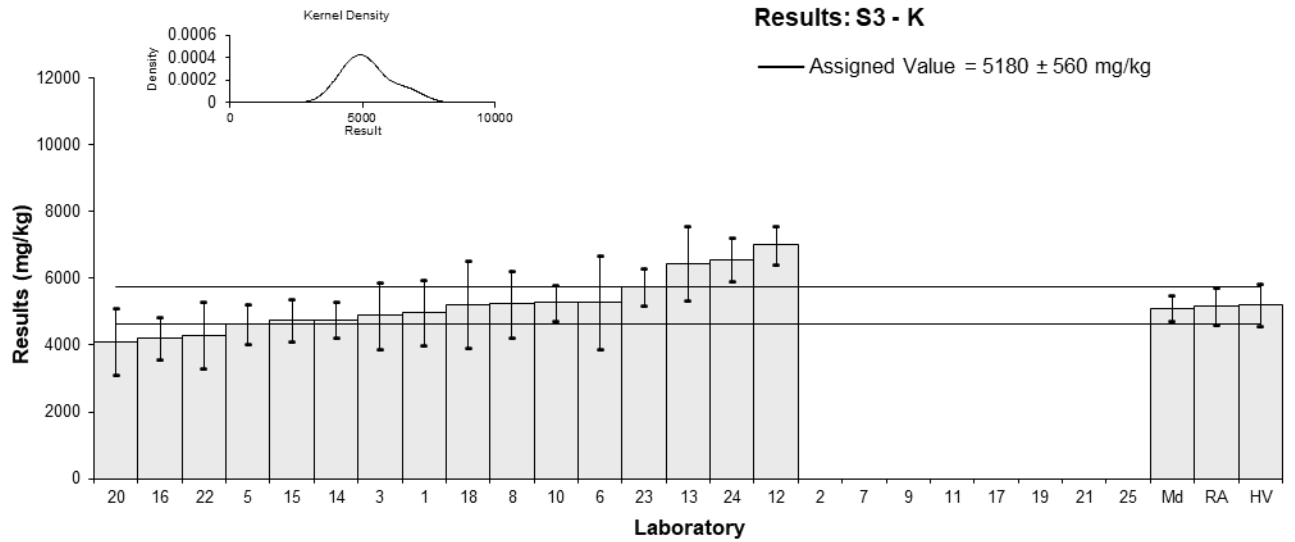


Figure 43

Table 55

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	4710	942	-0.56	-0.28
2	NR	NR		
3	5000	1000	0.02	0.01
5	4746	452	-0.49	-0.44
6	4800	700	-0.38	-0.25
7	NT	NT		
8	5610	1000	1.24	0.59
9	NT	NT		
10	5080	508	0.18	0.15
11	4900	340	-0.18	-0.19
12	6000	360	2.02	2.10
13	5528.802	1106	1.08	0.47
14	4900	1440	-0.18	-0.06
15	4887	815	-0.21	-0.12
16	4200	751.8	-1.58	-0.97
17	NT	NT		
18	5220	1305	0.46	0.17
19	NT	NT		
20	4100	1000	-1.78	-0.85
21	NT	NT		
22	4900	1000	-0.18	-0.09
23	5696	570	1.41	1.08
24	4640.0	464	-0.70	-0.62
25	NT	NT		

Statistics

Assigned Value	4990	320
Spike Value	Not Spiked	
Homogeneity Value	4960	600
Robust Average	4990	320
Median	4900	170
Mean	5000	
N	17	
Max	6000	
Min	4100	
Robust SD	520	
Robust CV	11%	

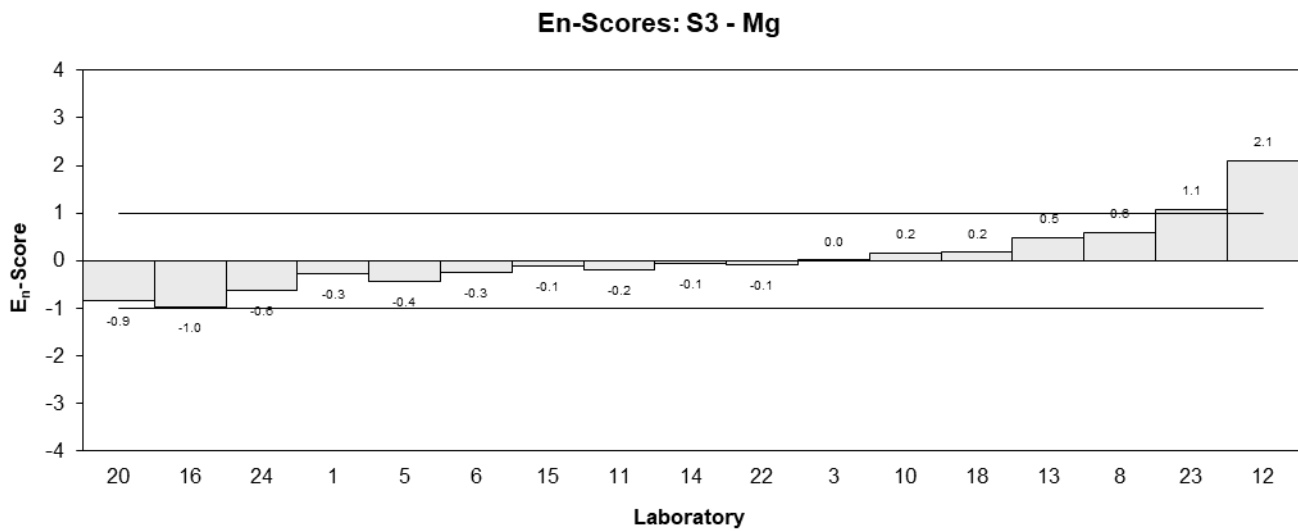
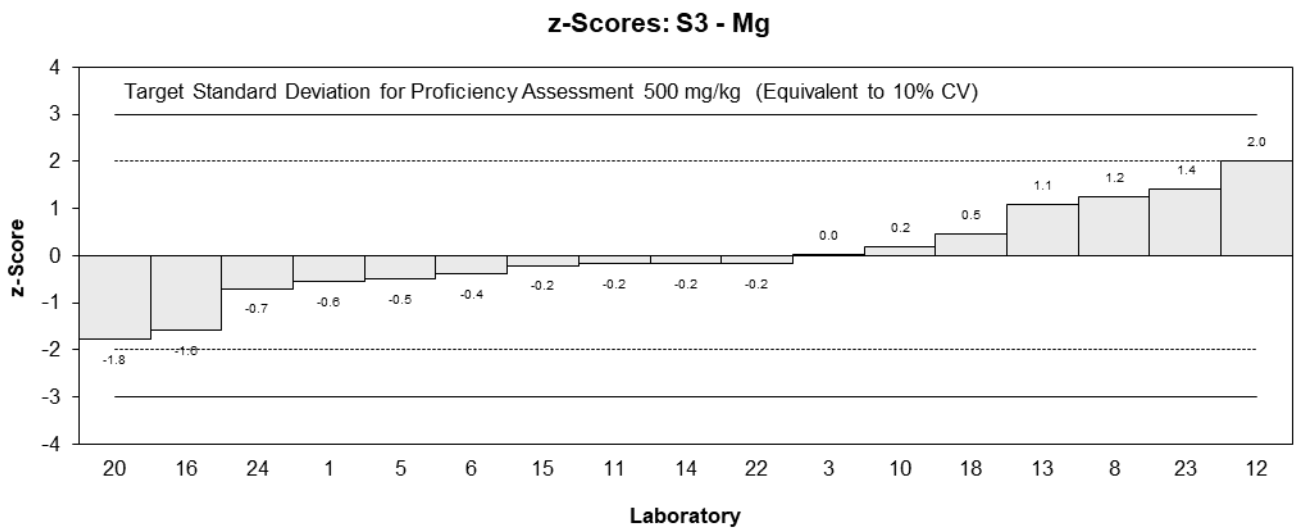
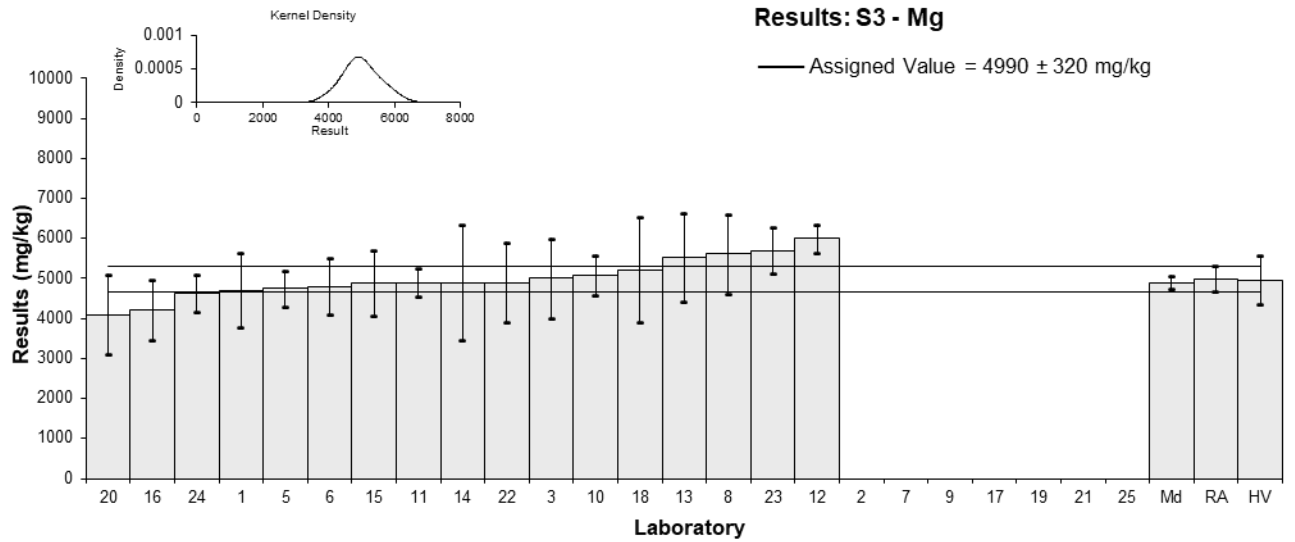


Figure 44

Table 56

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	410	82	-1.24	-0.65
2	NR	NR		
3	520	100	1.11	0.49
5	397	59	-1.52	-1.03
6	454	95	-0.30	-0.14
7	NT	NT		
8	447.5	45	-0.44	-0.36
9	NT	NT		
10	384	38.4	-1.79	-1.62
11	540	87	1.54	0.77
12	640	32	3.68	3.63
13	487.385	107	0.41	0.17
14	456	75	-0.26	-0.14
15	452.9	78.0	-0.32	-0.18
16	460	110.4	-0.17	-0.07
17	NT	NT		
18	403	101	-1.39	-0.61
19	NT	NT		
20	520	100	1.11	0.49
21	NT	NT		
22	510	100	0.90	0.40
23	481	50	0.28	0.21
24	478.2	47.82	0.22	0.17
25	NT	NT		

Statistics

Assigned Value	468	35
Spike Value	Not Spiked	
Homogeneity Value	433	52
Robust Average	468	35
Median	460	45
Mean	473	
N	17	
Max	640	
Min	384	
Robust SD	58	
Robust CV	12%	

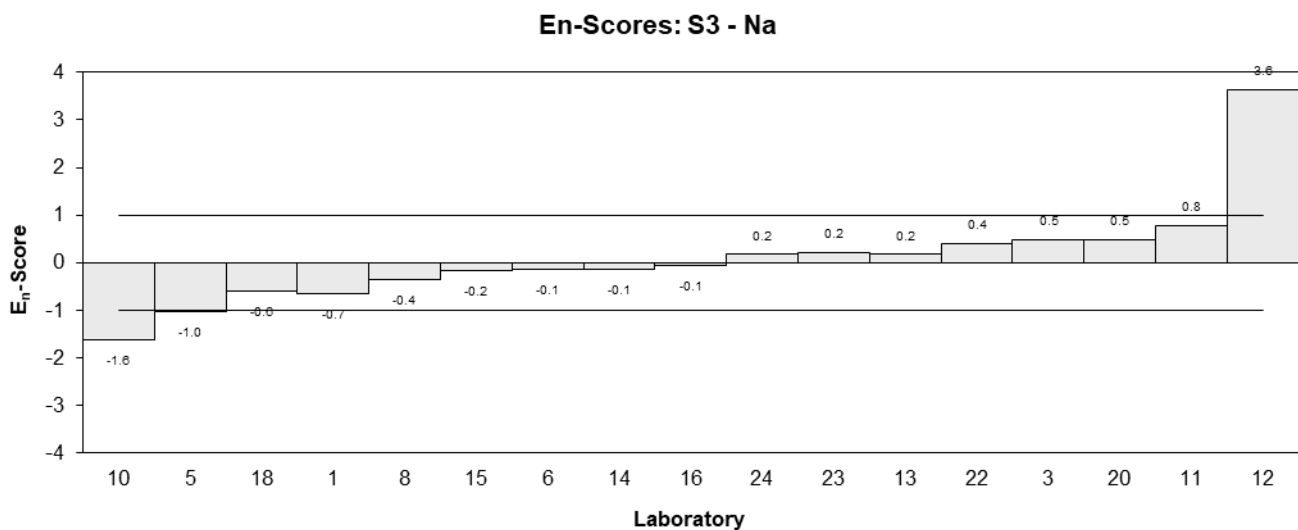
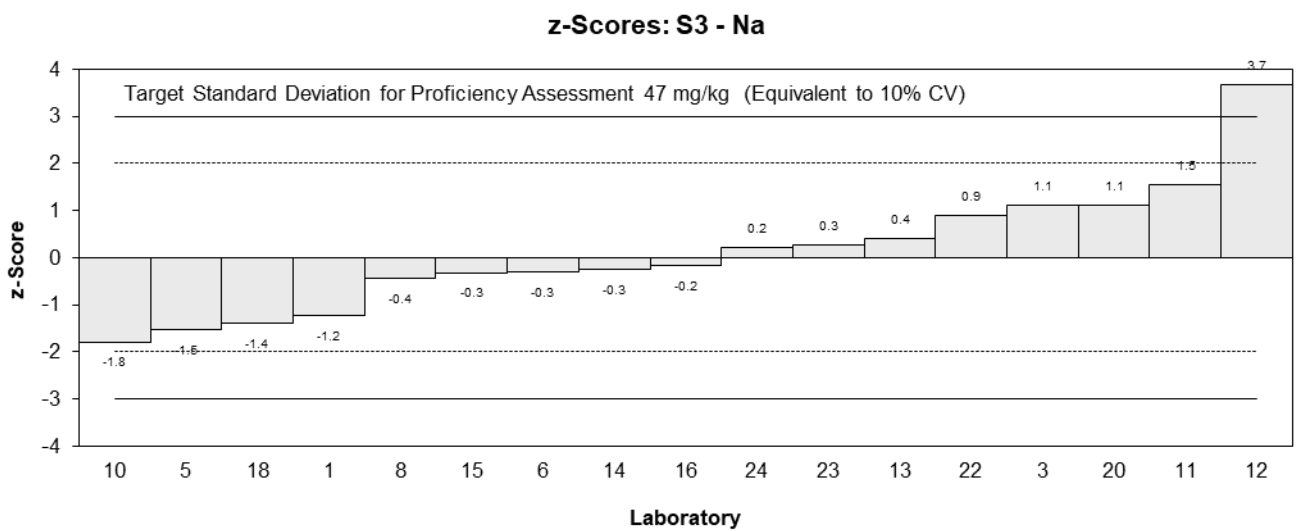
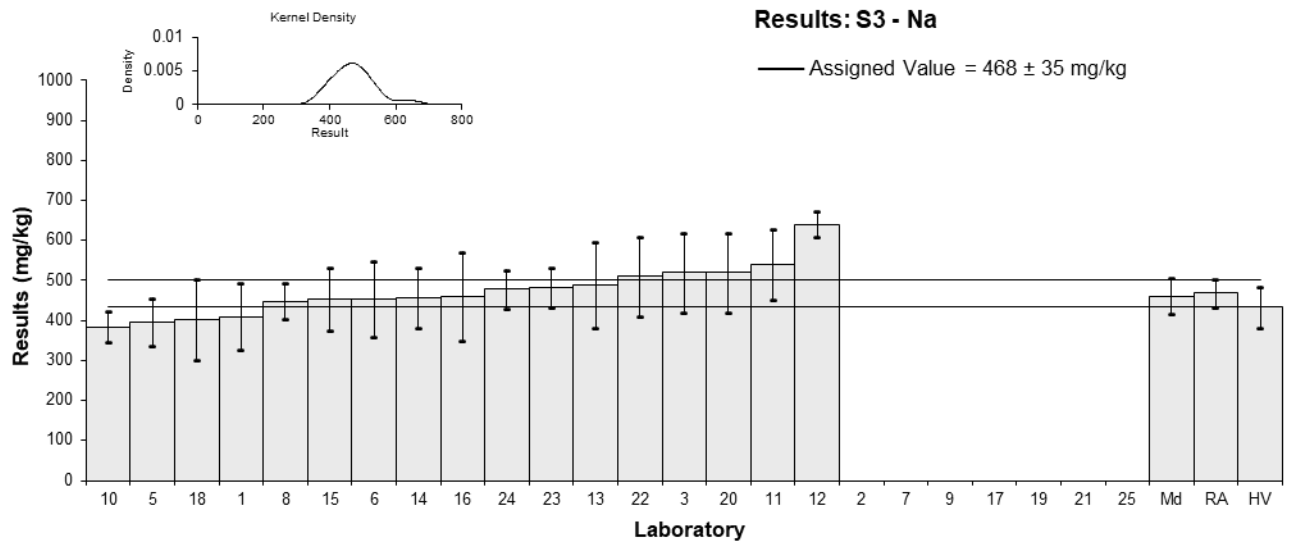


Figure 45

Table 57

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	150	30	-0.91	-0.44
2	NR	NR		
3	160	30	-0.30	-0.15
5	NR	NR		
6	142	26	-1.39	-0.75
7	NT	NT		
8	204	10	2.36	2.07
9	NT	NT		
10	161	161	-0.24	-0.02
11	170	22	0.30	0.18
12	NT	NT		
13	179.615	28.73	0.89	0.44
14	181	33	0.97	0.44
15	157.4	19.3	-0.46	-0.30
16	130	27.95	-2.12	-1.09
17	NT	NT		
18	NT	NT		
19	NT	NT		
20	140	50	-1.52	-0.48
21	NT	NT		
22	160	50	-0.30	-0.10
23	172	20	0.42	0.27
24	203.2	20.32	2.32	1.48
25	NT	NT		

Statistics

Assigned Value	165	16
Spike Value	Not Spiked	
Homogeneity Value	156	19
Robust Average	165	16
Median	161	15
Mean	165	
N	14	
Max	204	
Min	130	
Robust SD	24	
Robust CV	15%	

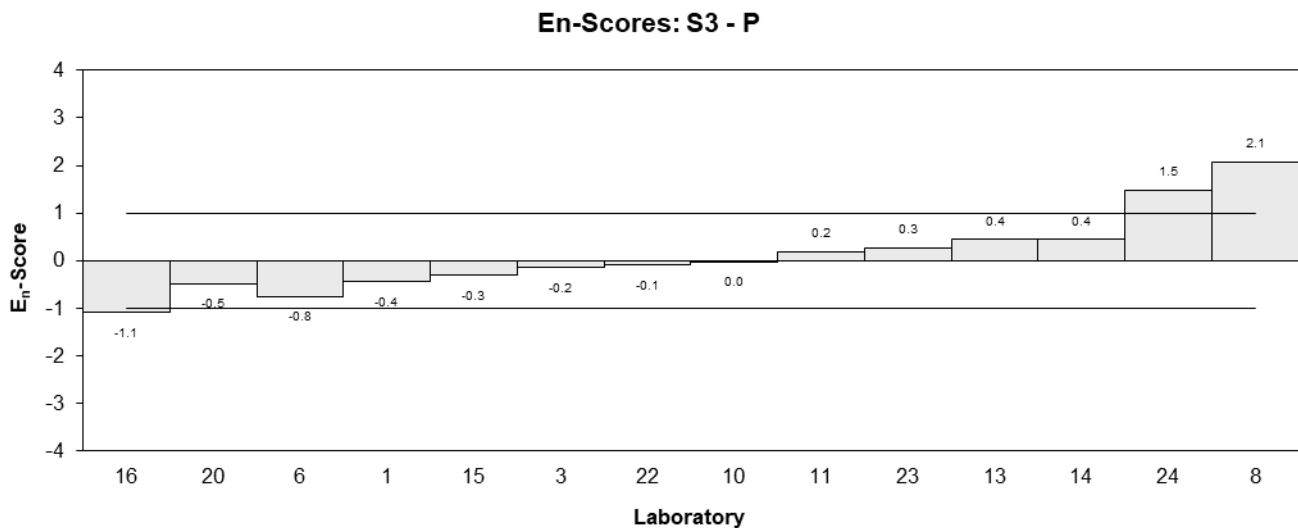
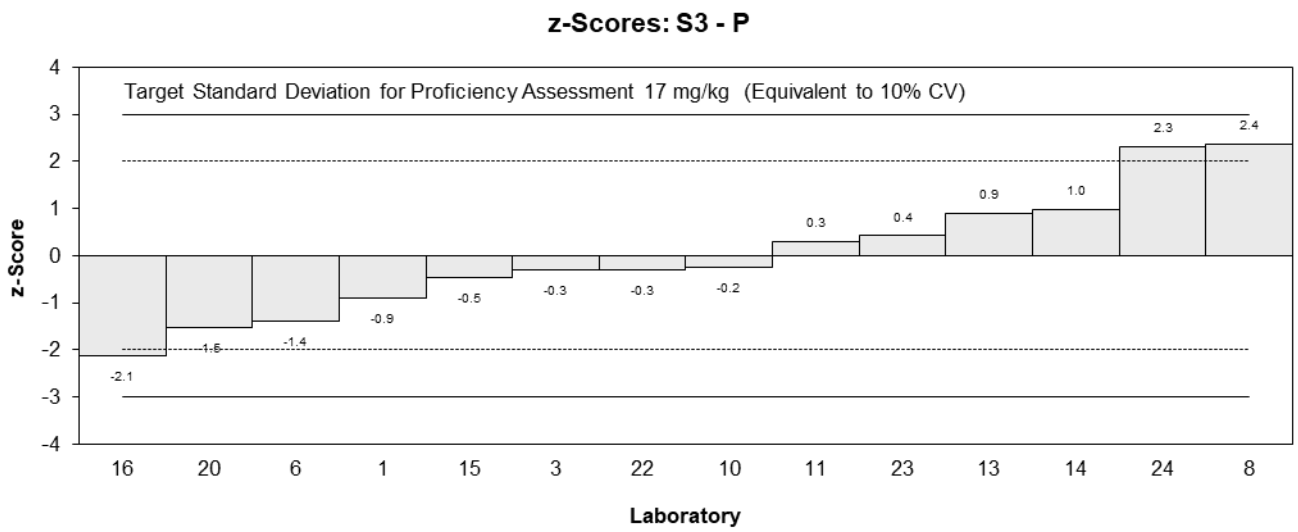
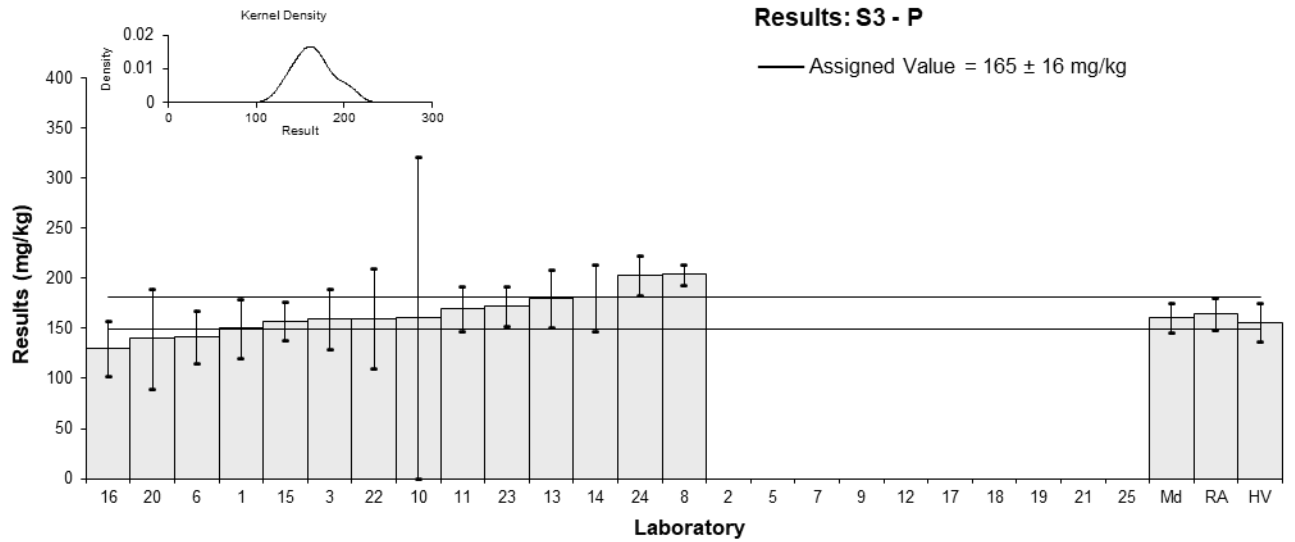


Figure 46

Table 58

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	87	17	0.60	0.26
2	NR	NR		
3	83	20	0.11	0.04
5	NR	NR		
6	77	16	-0.62	-0.29
7	NT	NT		
8	50	10	-3.91	-2.58
9	NT	NT		
10	83.5	20.9	0.17	0.06
11	91	9	1.08	0.76
12	<1	NR		
13	80.932	14.6	-0.14	-0.07
14	91	5	1.08	1.00
15	78.73	6.76	-0.41	-0.34
16	68	13.26	-1.72	-0.93
17	NT	NT		
18	106	26.5	2.91	0.87
19	NT	NT		
20	81	20	-0.13	-0.05
21	NT	NT		
22	NR	NR		
23	<100	NR		
24*	126.6	12.66	5.42	3.03
25	NT	NT		

* Outlier, see Section 4.2

Statistics

Assigned Value	82.1	7.4
Spike Value	Not Spiked	
Robust Average	84.1	9.2
Median	83.0	6.2
Mean	85	
N	13	
Max	126.6	
Min	50	
Robust SD	13	
Robust CV	16%	

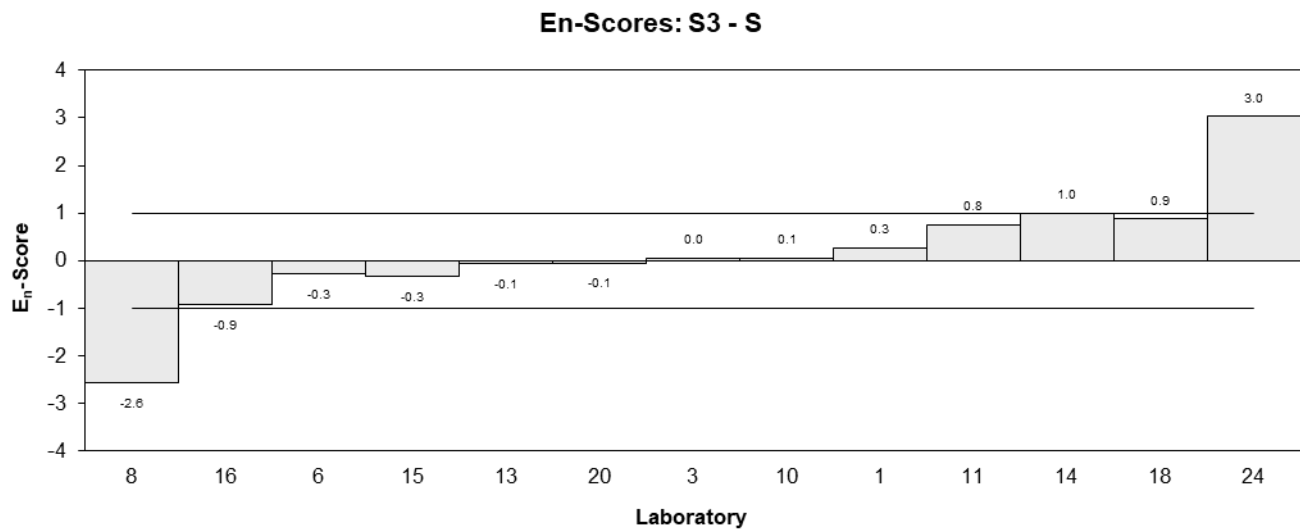
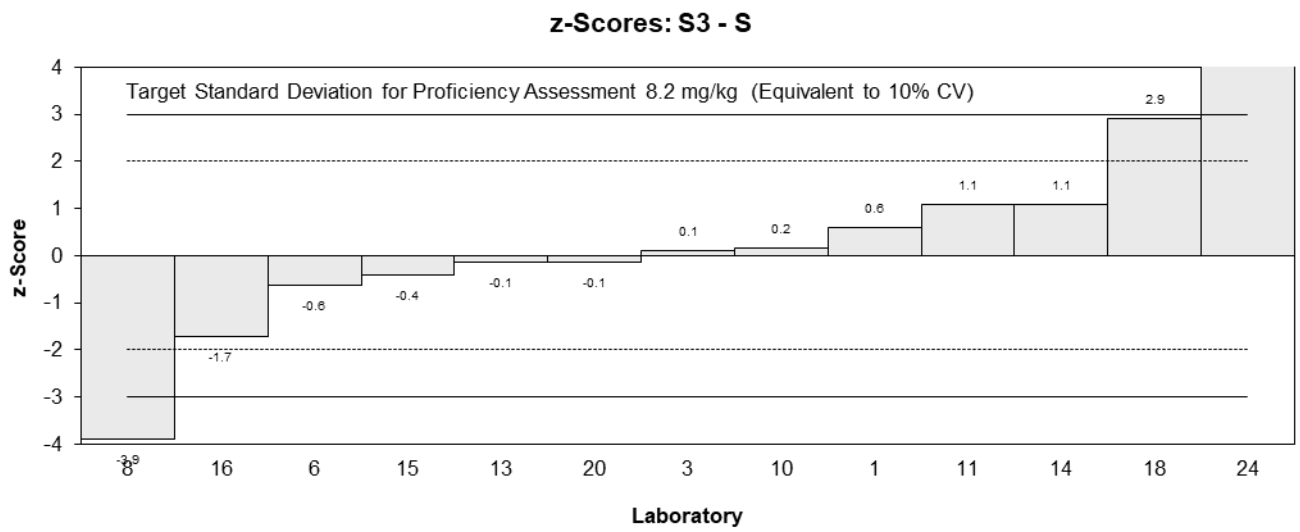
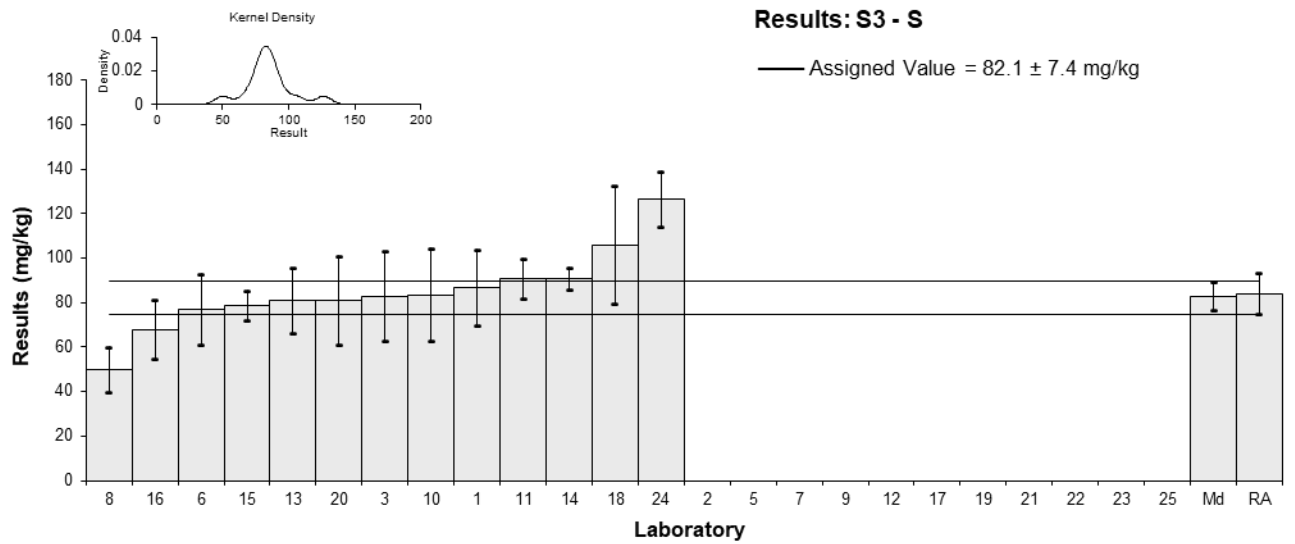


Figure 47

Table 59

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	NR	NR
2	NR	NR
3	700	200
5	NR	NR
6	NR	NR
7	NT	NT
8	NT	NT
9	NT	NT
10	NT	NT
11	750	150
12	NT	NT
13	NR	NR
14	NT	NT
15	NT	NT
16	NT	NT
17	NT	NT
18	680	102
19	NT	NT
20	NT	NT
21	NT	NT
22	NR	NR
23	2309	230
24	238.5	23.85
25	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Median	700	83
Mean	940	
N	5	
Max	2309	
Min	238.5	

Results: S3 - Colwell K

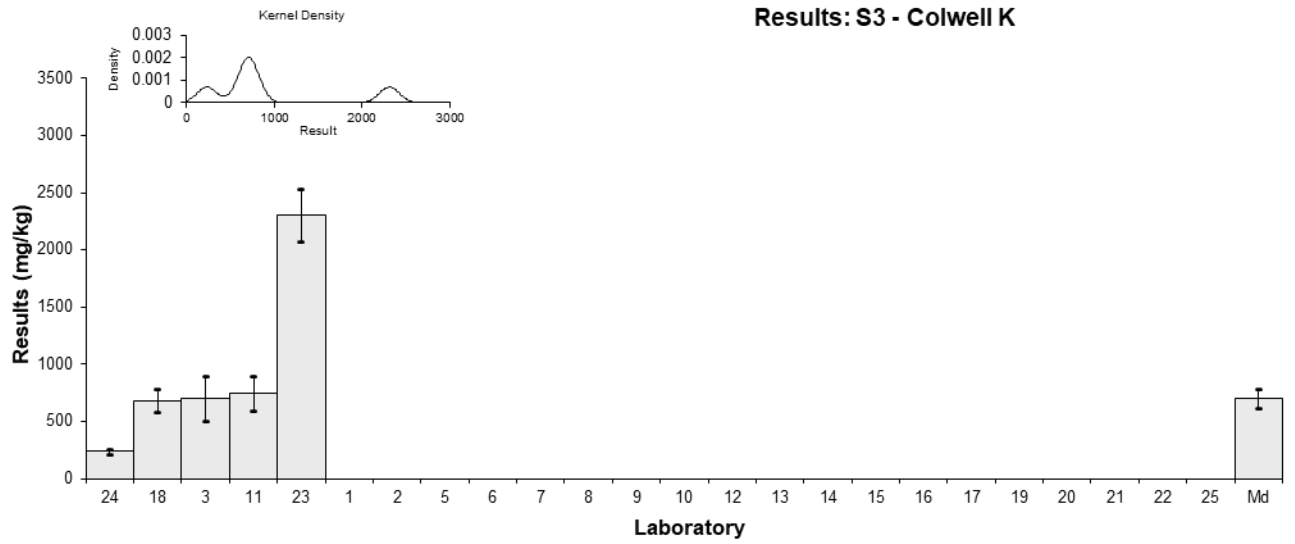


Figure 48

Table 60

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	12	2.4	-1.30	-0.87
2	20.5	6.0	1.33	0.59
3	21	6	1.48	0.66
5	13	NR	-0.99	-0.76
6	NR	NR		
7	NT	NT		
8	13	4	-0.99	-0.55
9	NT	NT		
10	NT	NT		
11	20	4	1.17	0.66
12	NT	NT		
13	<50	NR		
14	NT	NT		
15	NT	NT		
16*	6	0.5198	-3.15	-2.41
17	NT	NT		
18	11.6	1.74	-1.42	-1.01
19	NT	NT		
20	NT	NT		
21	NT	NT		
22	NR	NR		
23	22	2.5	1.79	1.19
24	13.0	1.3	-0.99	-0.73
25	NT	NT		

* Outlier, see Section 4.2

Statistics

Assigned Value	16.2	4.2
Spike Value	Not Spiked	
Robust Average	15.2	4.7
Median	13.0	4.9
Mean	15.2	
N	10	
Max	22	
Min	6	
Robust SD	5.9	
Robust CV	39%	

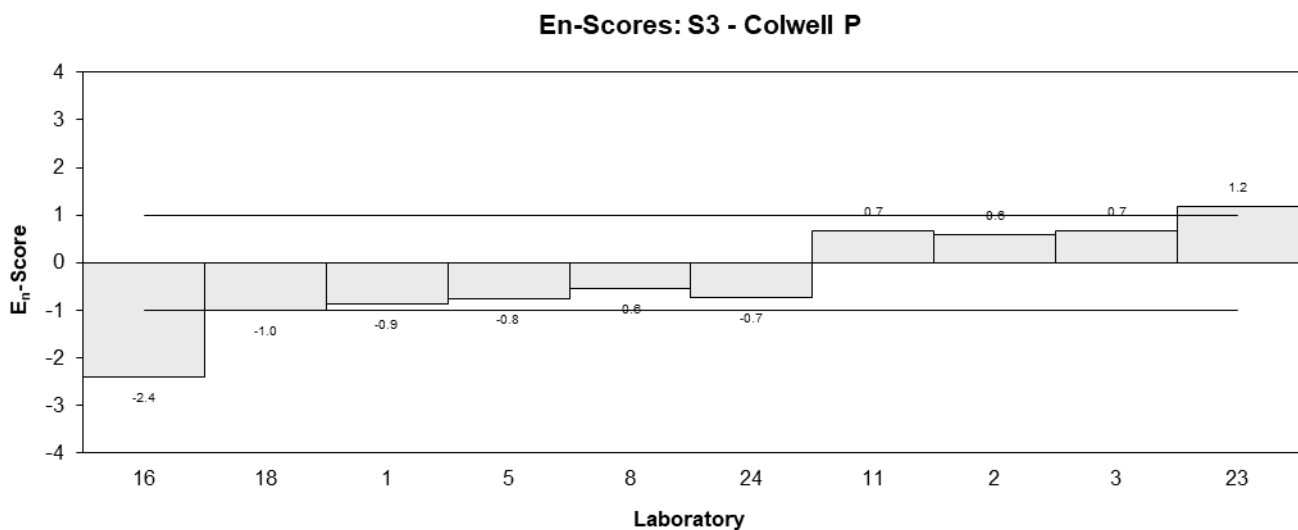
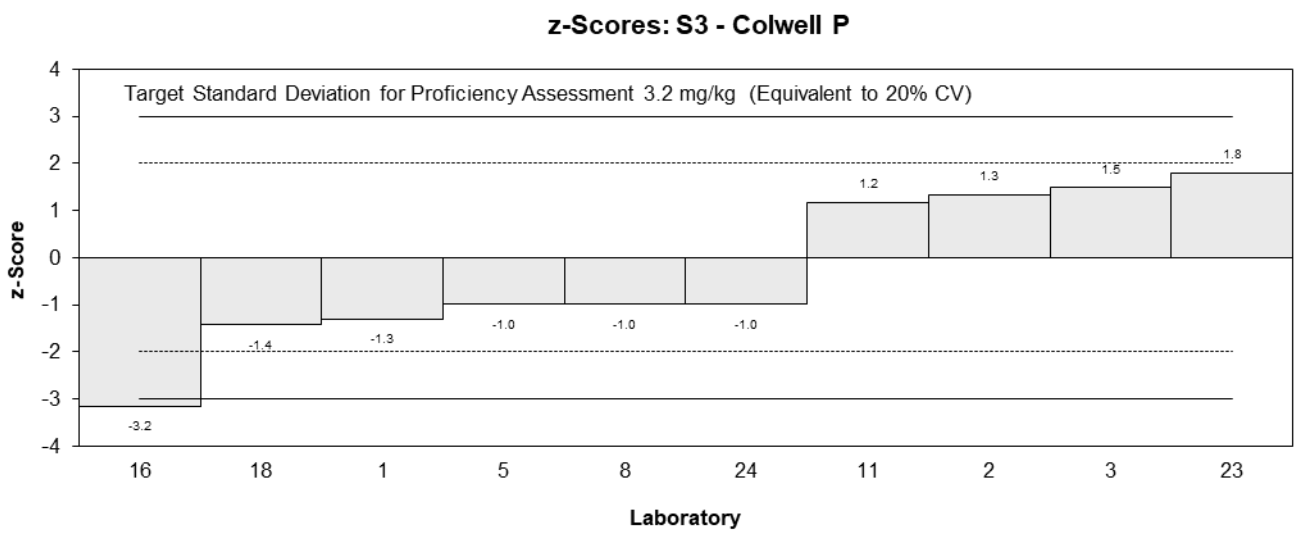
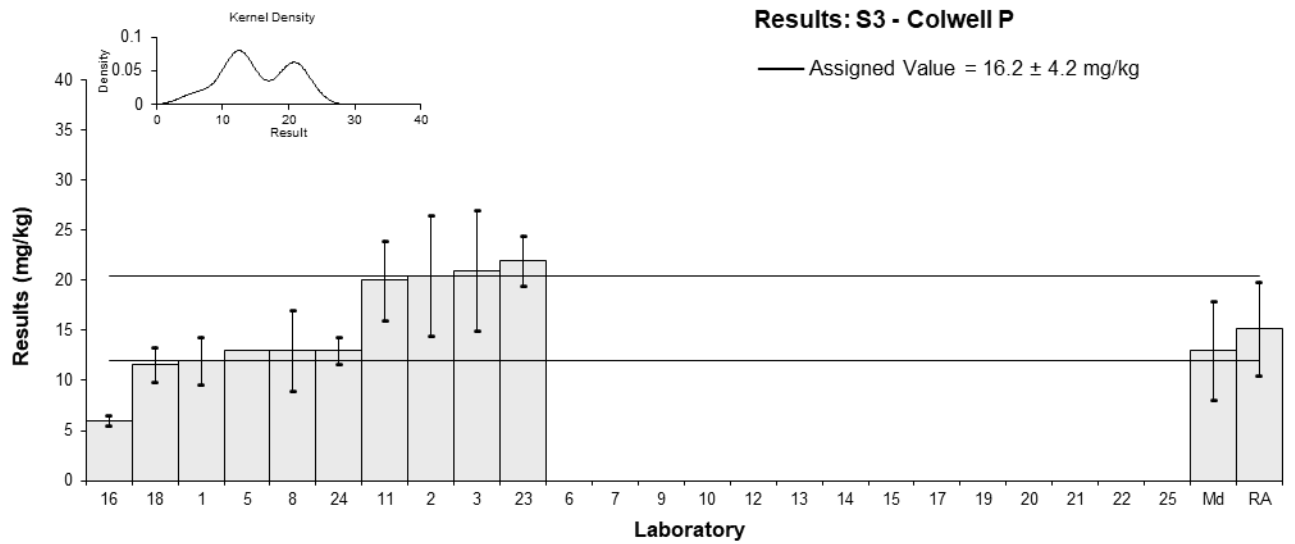


Figure 49

Table 61

Sample Details

Sample No.	S3
Matrix	Soil
Analyte	EC
Unit	$\mu\text{S/cm}$

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	240	24	0.30	0.27
2	241	NR	0.34	0.89
3	275	50	1.80	0.83
5	245	8.13	0.52	0.99
6	NR	NR		
7	NT	NT		
8	224	NR	-0.39	-1.00
9	NT	NT		
10	171	42.9	-2.66	-1.41
11	218	22	-0.64	-0.63
12	NT	NT		
13	230	12	-0.13	-0.20
14	230	14	-0.13	-0.18
15	305.3	6.6	3.10	6.48
16	230	14.72	-0.13	-0.17
17	NT	NT		
18	227	33.5	-0.26	-0.17
19	NT	NT		
20	250	50	0.73	0.33
21	NT	NT		
22	240	50	0.30	0.14
23	225	22	-0.34	-0.34
24	220.6	3.394112549695	-0.53	-1.29
25	NT	NT		

Statistics

Assigned Value	233	9
Spike Value	Not Spiked	
Robust Average	233	9
Median	230	9
Mean	236	
N	16	
Max	305.3	
Min	171	
Robust SD	15	
Robust CV	6.3%	

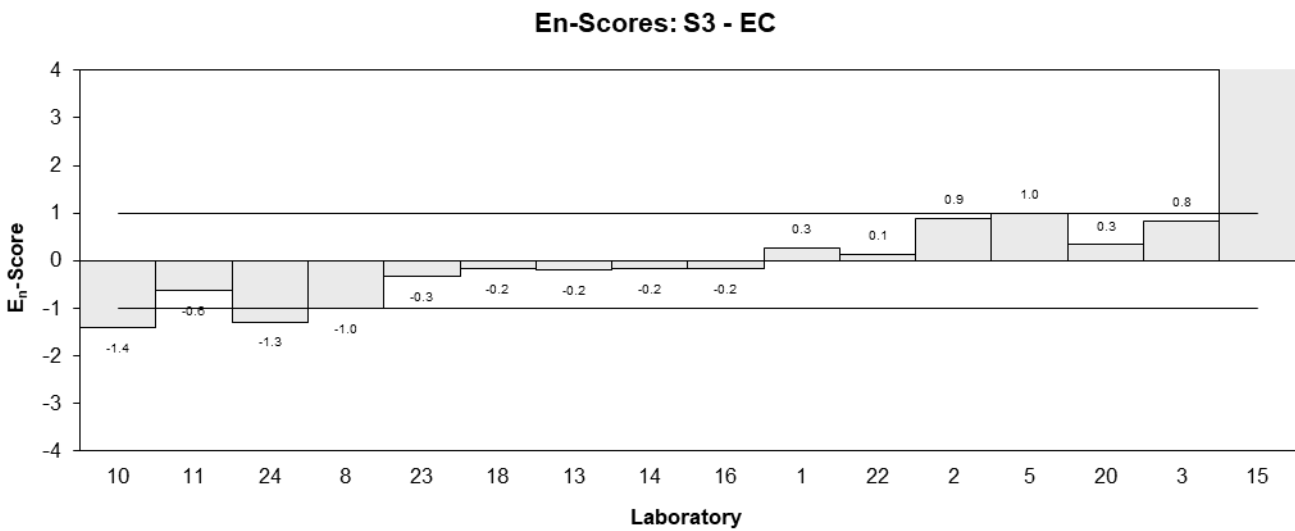
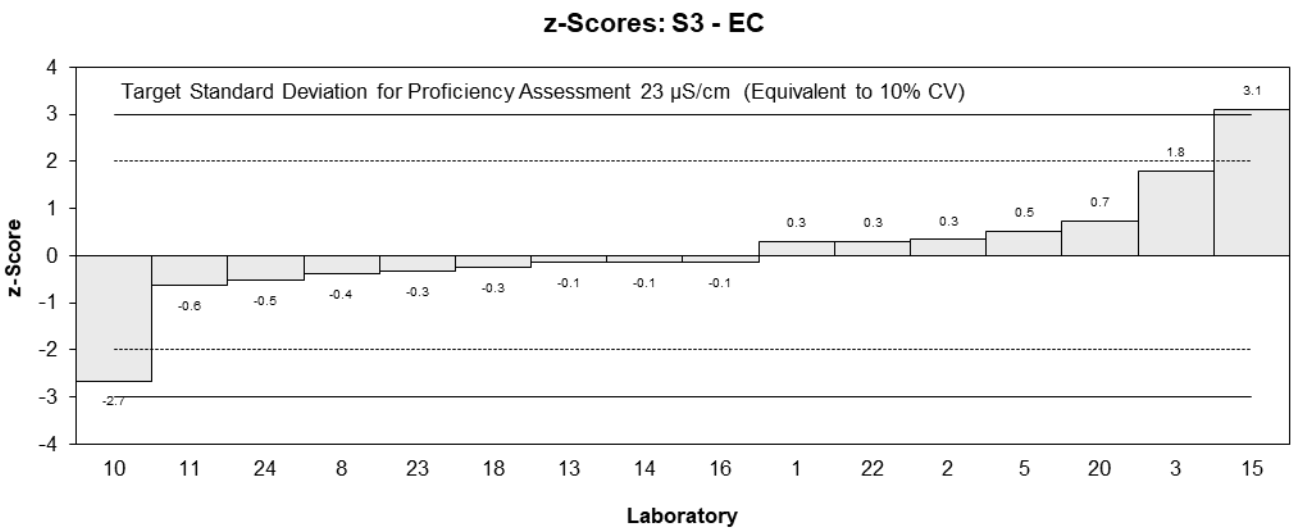
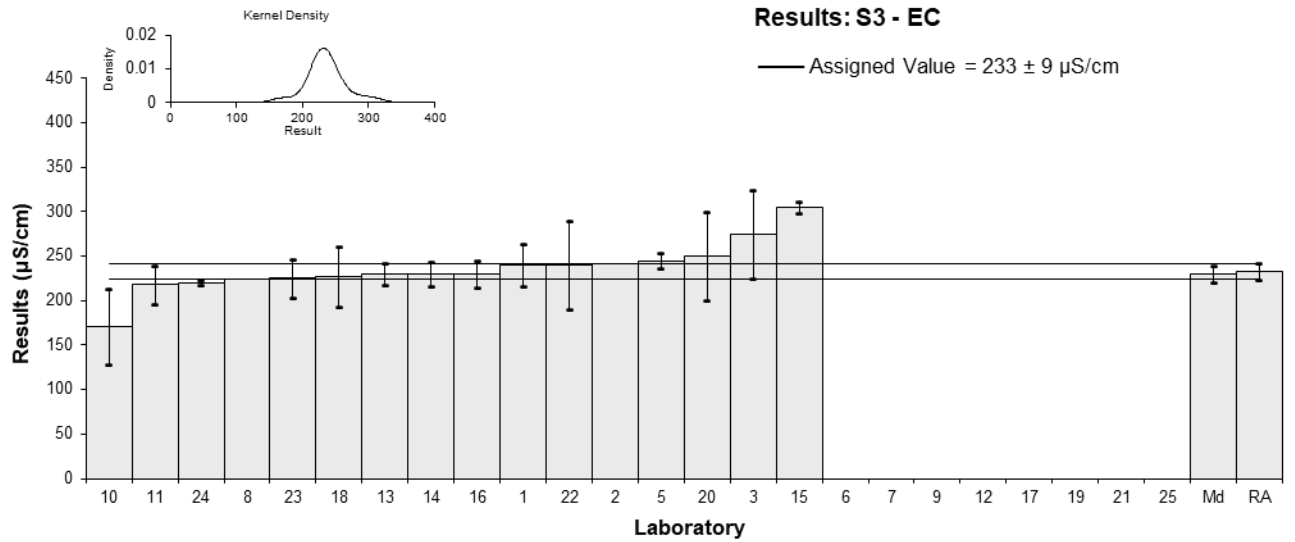


Figure 50

Table 62

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	22	4.4	-0.09	-0.04
2	23.5	0.56	0.59	1.05
3	20	5	-0.99	-0.43
5	NR	NR		
6	NR	NR		
7	NT	NT		
8	19.85	NR	-1.06	-2.14
9	NT	NT		
10	NT	NT		
11	25	3	1.26	0.88
12	22	NR	-0.09	-0.18
13	15	3	-3.24	-2.25
14	NT	NT		
15	22.638	2.554	0.20	0.16
16	23	1.687	0.36	0.40
17	NT	NT		
18	21.9	5.48	-0.14	-0.05
19	NT	NT		
20	23	9	0.36	0.09
21	NT	NT		
22	23	6	0.36	0.13
23	22.2	2.5	0.00	0.00
24	23.2	22.06	0.45	0.05
25	NT	NT		

Statistics

Assigned Value	22.2	1.1
Spike Value	Not Spiked	
Robust Average	22.2	1.1
Median	22.4	0.6
Mean	21.9	
N	14	
Max	25	
Min	15	
Robust SD	1.7	
Robust CV	7.4%	

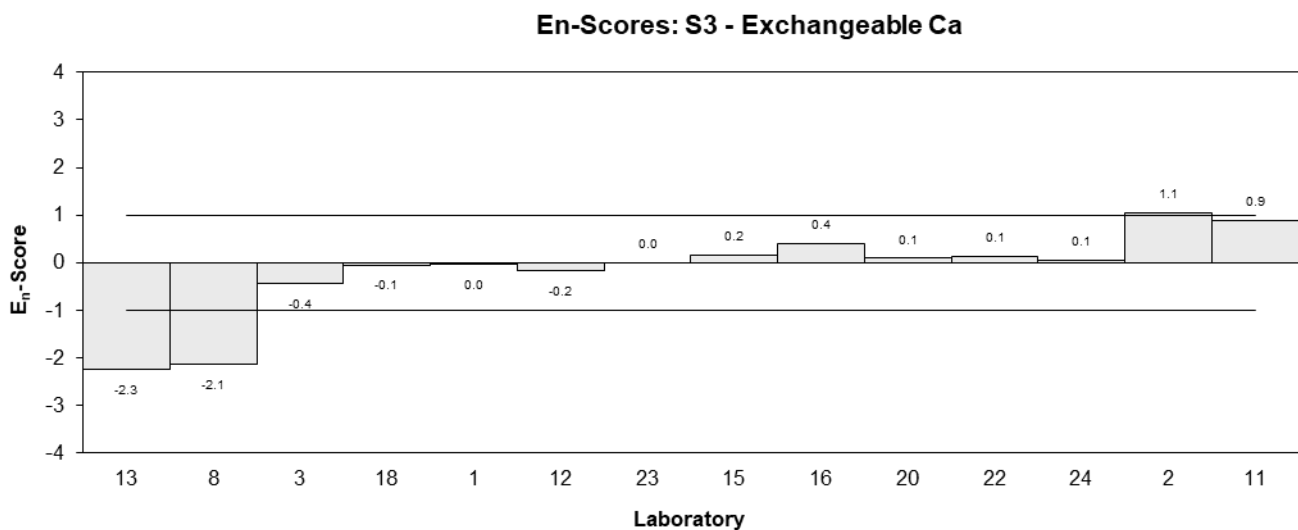
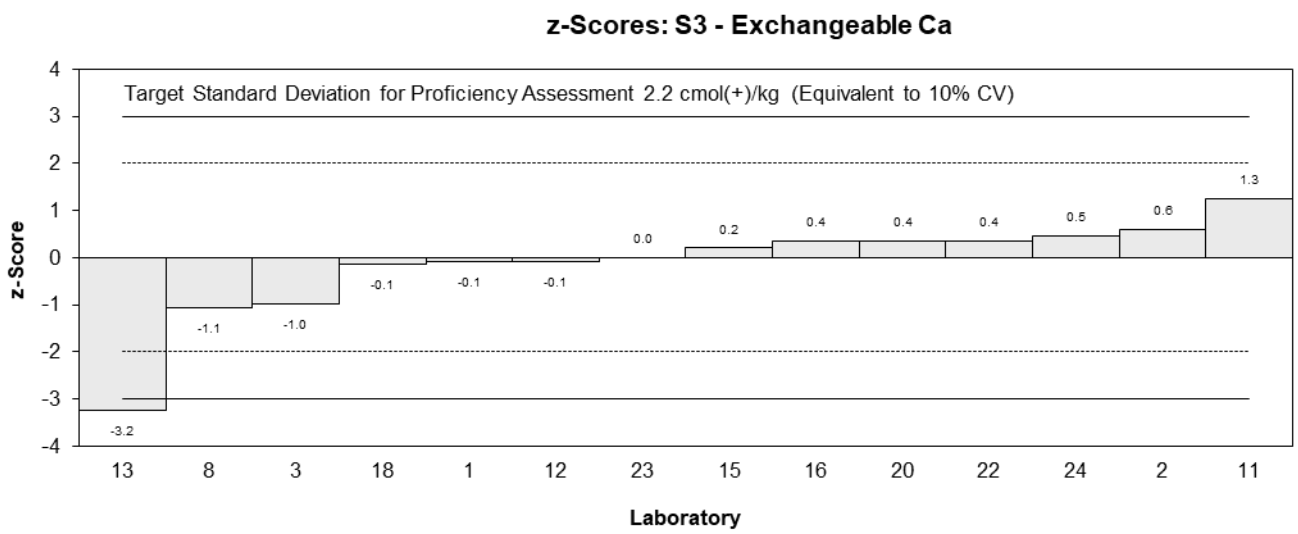
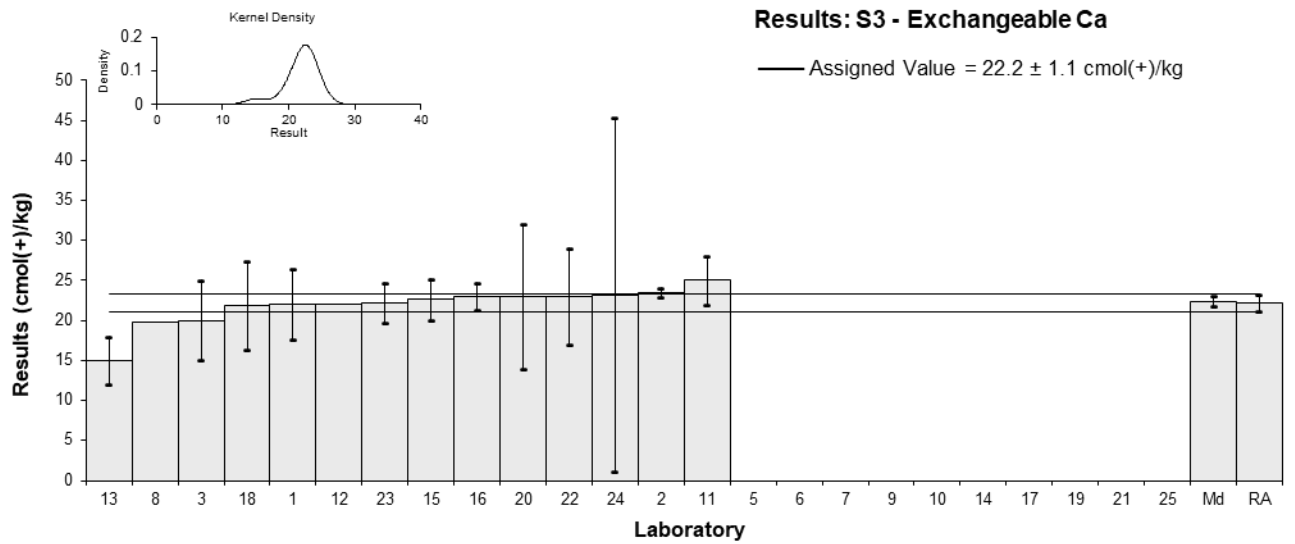


Figure 51

Table 63

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	2.1	0.42	-0.06	-0.04
2	1.9	0.07	-0.69	-1.04
3	2.2	1	0.25	0.08
5	NR	NR		
6	NR	NR		
7	NT	NT		
8	1.95	NR	-0.53	-0.85
9	NT	NT		
10	NT	NT		
11	2.2	0.4	0.25	0.18
12	2.5	NR	1.19	1.90
13	2.0	0.4	-0.38	-0.27
14	NT	NT		
15	2.022	0.261	-0.31	-0.30
16	2.0	0.1439	-0.38	-0.49
17	NT	NT		
18	1.99	0.498	-0.41	-0.24
19	NT	NT		
20	3	1	2.77	0.86
21	NT	NT		
22	3	1	2.77	0.86
23	2.01	0.2	-0.35	-0.39
24	1.5	2.32	-1.95	-0.27
25	NT	NT		

Statistics

Assigned Value	2.12	0.20
Spike Value	Not Spiked	
Robust Average	2.12	0.20
Median	2.02	0.10
Mean	2.17	
N	14	
Max	3	
Min	1.5	
Robust SD	0.3	
Robust CV	14%	

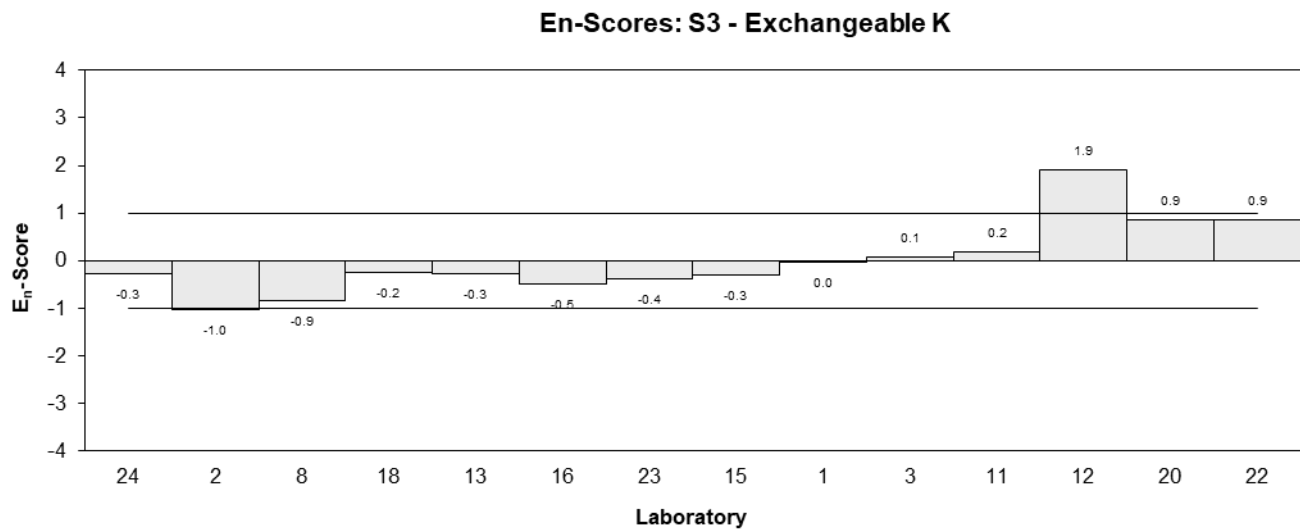
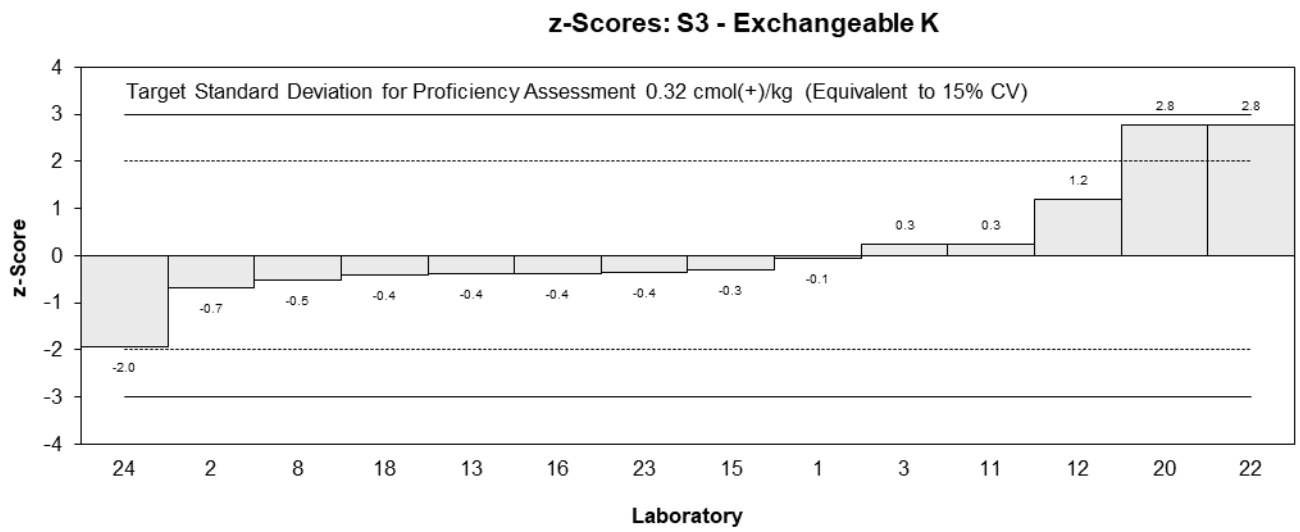
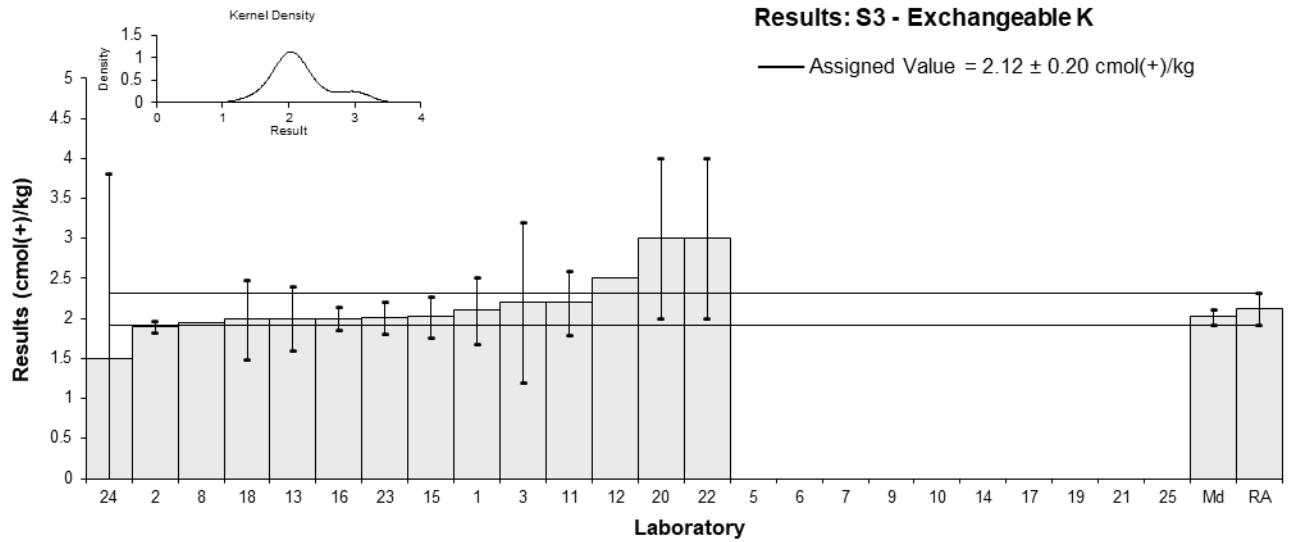


Figure 52

Table 64

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	11	2.2	0.48	0.23
2	10.5	0.34	0.00	0.00
3	11	3	0.48	0.17
5	NR	NR		
6	NR	NR		
7	NT	NT		
8	10.5	NR	0.00	0.00
9	NT	NT		
10	NT	NT		
11	11	1.5	0.48	0.33
12	10	NR	-0.48	-1.67
13	9.1	1.82	-1.33	-0.76
14	NT	NT		
15	10.372	1.581	-0.12	-0.08
16	10	0.2937	-0.48	-1.19
17	NT	NT		
18	10.3	2.58	-0.19	-0.08
19	NT	NT		
20	11	3	0.48	0.17
21	NT	NT		
22	11	3	0.48	0.17
23	10.1	1.0	-0.38	-0.38
24	10.5	0.15	0.00	0.00
25	NT	NT		

Statistics

Assigned Value	10.5	0.3
Spike Value	Not Spiked	
Robust Average	10.5	0.3
Median	10.5	0.5
Mean	10.5	
N	14	
Max	11	
Min	9.1	
Robust SD	0.5	
Robust CV	4.8%	

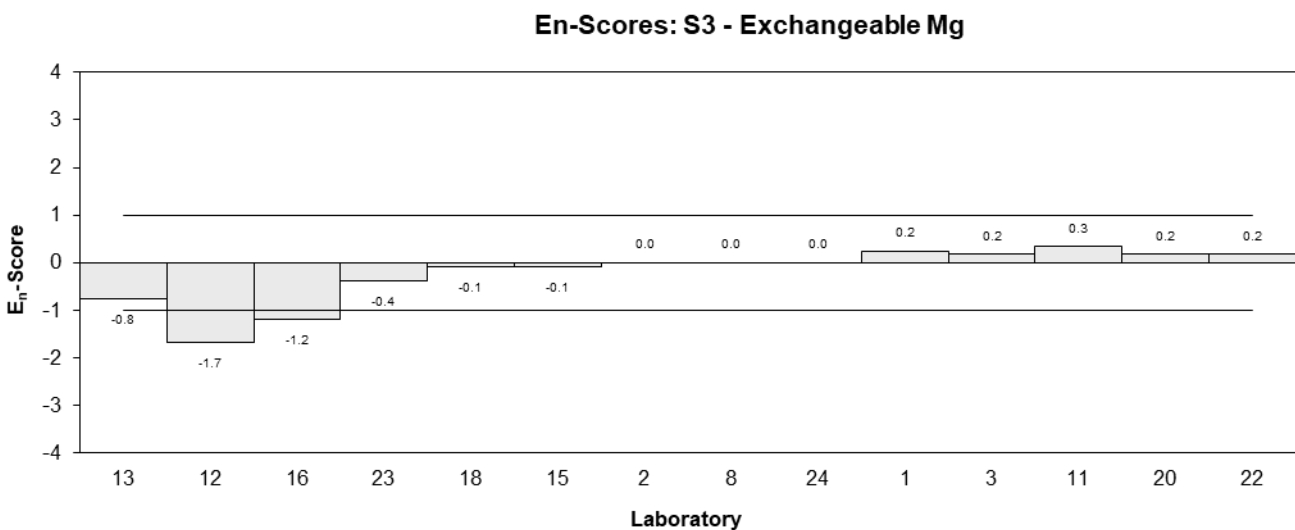
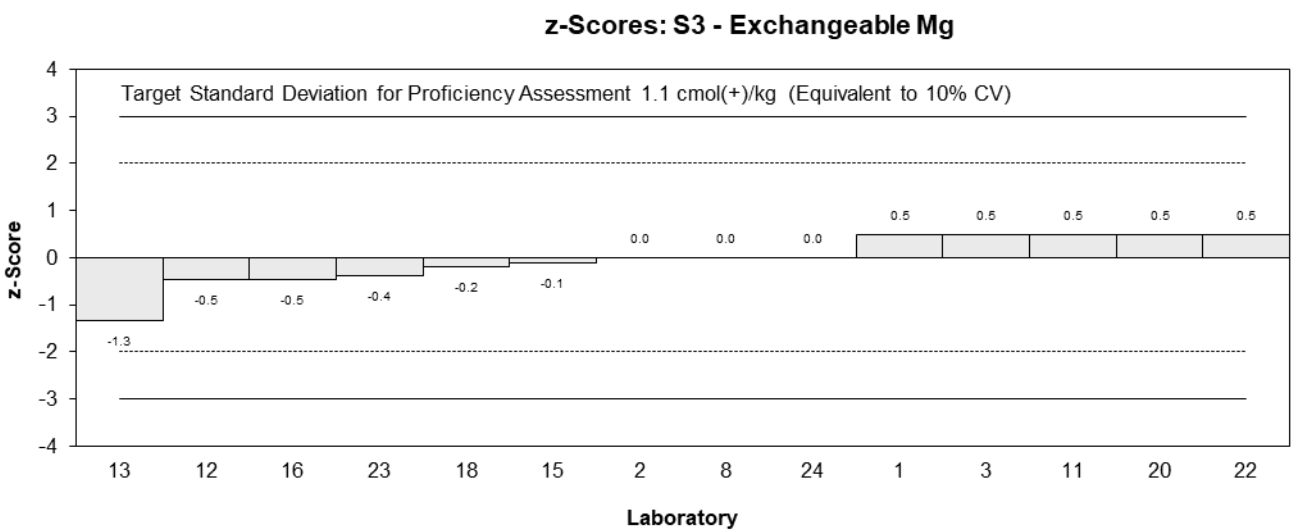
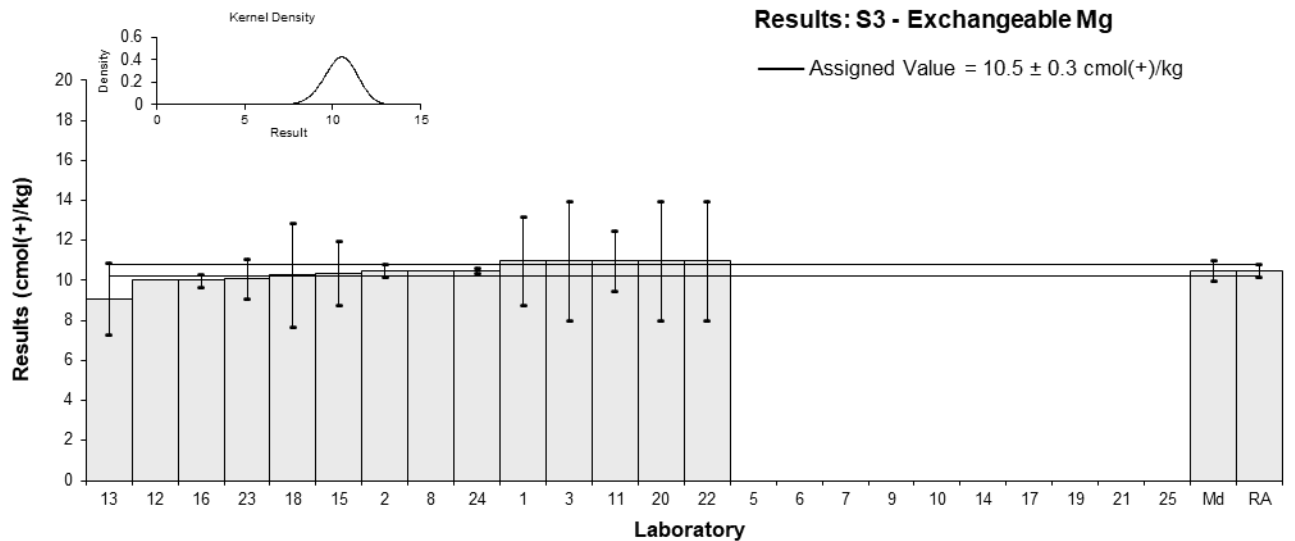


Figure 53

Table 65

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	1.3	0.26	-0.10	-0.07
2	1.35	0.18	0.15	0.13
3	1.4	1	0.40	0.08
5	NR	NR		
6	NR	NR		
7	NT	NT		
8	1.25	NR	-0.35	-0.50
9	NT	NT		
10	NT	NT		
11	1.6	0.2	1.41	1.15
12	1.8	NR	2.42	3.43
13	1.2	0.24	-0.61	-0.43
14	NT	NT		
15	1.366	0.165	0.23	0.21
16	1.4	0.0436	0.40	0.55
17	NT	NT		
18	1.32	0.33	0.00	0.00
19	NT	NT		
20	1.5	0.7	0.91	0.25
21	NT	NT		
22	1	1	-1.62	-0.32
23	0.96	0.1	-1.82	-2.09
24	1.2	1.05	-0.61	-0.11
25	NT	NT		

Statistics

Assigned Value	1.32	0.14
Spike Value	Not Spiked	
Robust Average	1.32	0.14
Median	1.34	0.11
Mean	1.33	
N	14	
Max	1.8	
Min	0.96	
Robust SD	0.22	
Robust CV	16%	

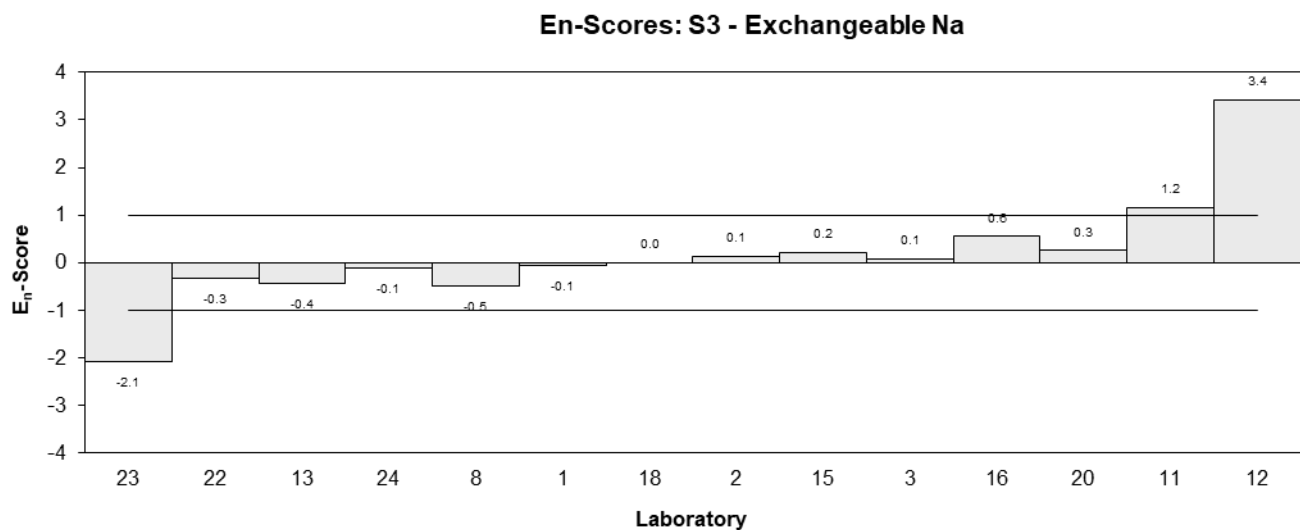
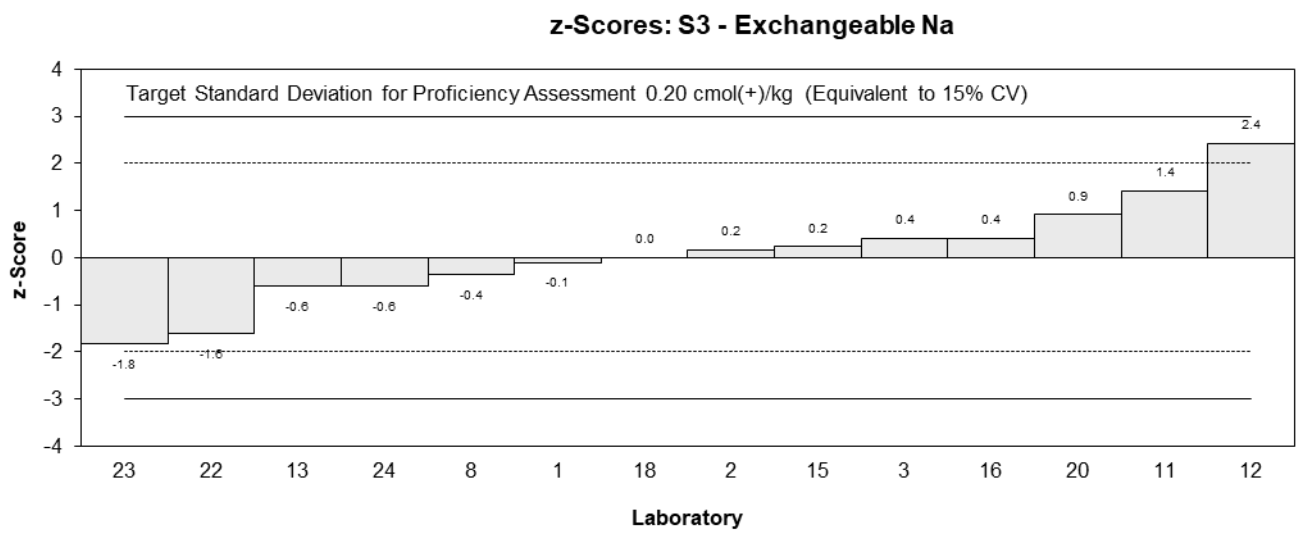
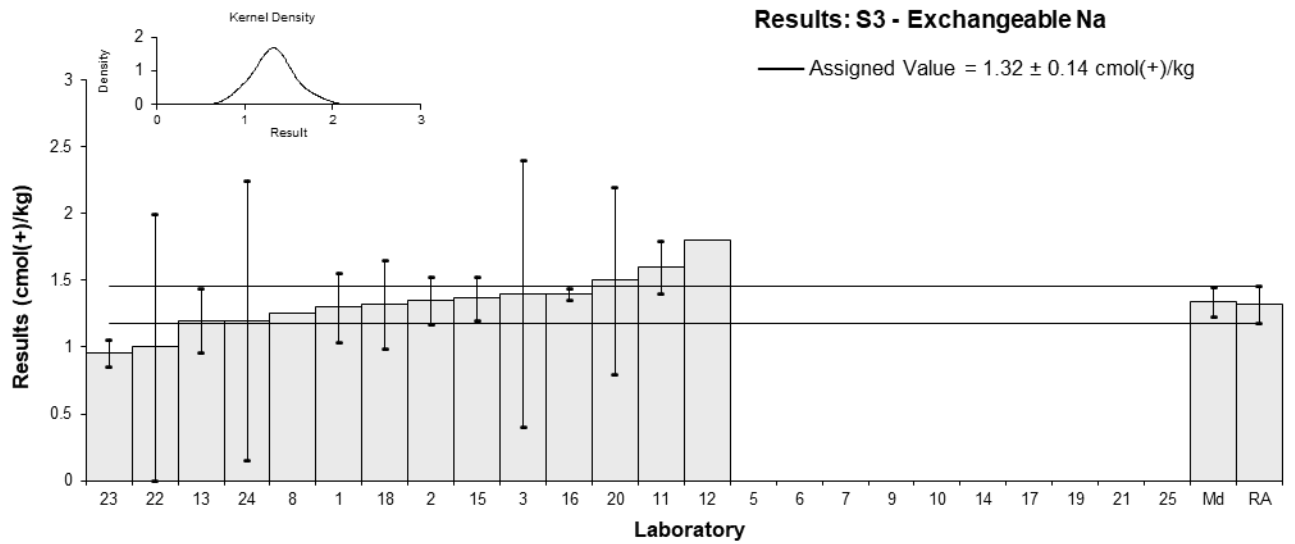


Figure 54

Table 66

Sample Details

Sample No.	S3
Matrix	Soil
Analyte	Extractable B
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty
1	NR	NR
2	NR	NR
3	1.4	1
5	NR	NR
6	NR	NR
7	NT	NT
8	3.35	NR
9	NT	NT
10	NT	NT
11	NT	NT
12	NT	NT
13	NR	NR
14	NT	NT
15	NT	NT
16	NT	NT
17	NT	NT
18	NT	NT
19	NT	NT
20	NT	NT
21	NT	NT
22	2.3	1
23	0.95	0.1
24	1.51	0.12
25	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Median	1.51	0.93
Mean	1.90	
N	5	
Max	3.35	
Min	0.95	

Results: S3 - Extractable B

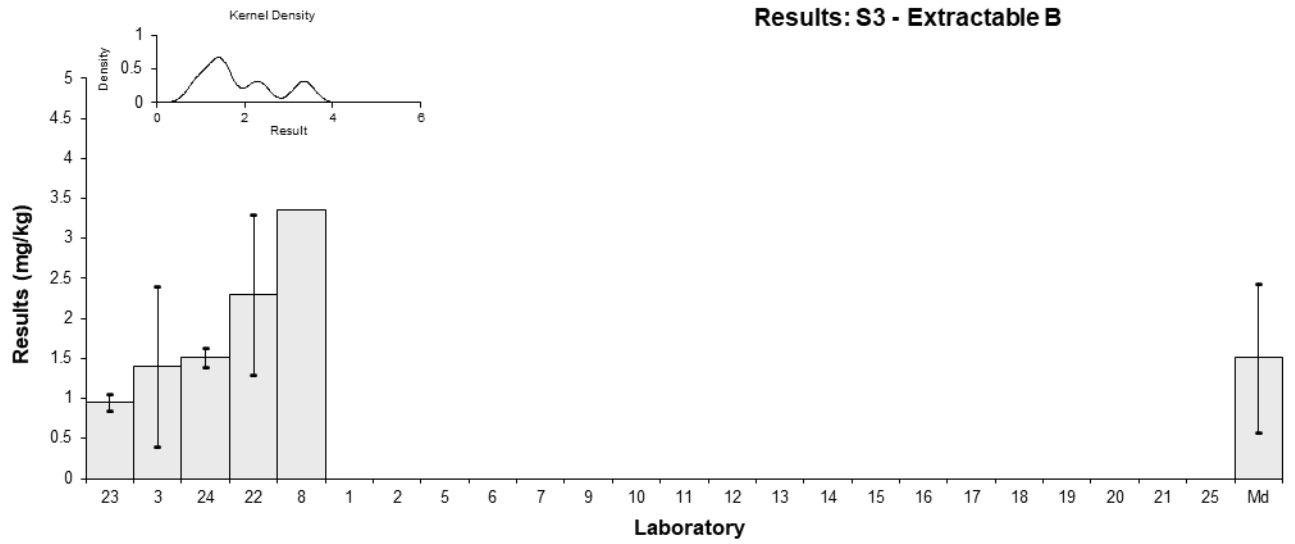


Figure 55

Table 67

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	NR	NR
2	185.0	NR
3	NR	NR
5	NR	NR
6	NR	NR
7	NT	NT
8	174	30
9	NT	NT
10	NT	NT
11	153	15
12	NT	NT
13	NR	NR
14	NT	NT
15	NT	NT
16	NT	NT
17	NT	NT
18	147	22.1
19	NT	NT
20	NT	NT
21	NT	NT
22	NR	NR
23	163	16
24	248.6	0.151
25	NT	NT

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Robust Average	173	29
Median	169	24
Mean	178	
N	6	
Max	248.6	
Min	147	
Robust SD	28	
Robust CV	16%	

Results: S3 - PBI

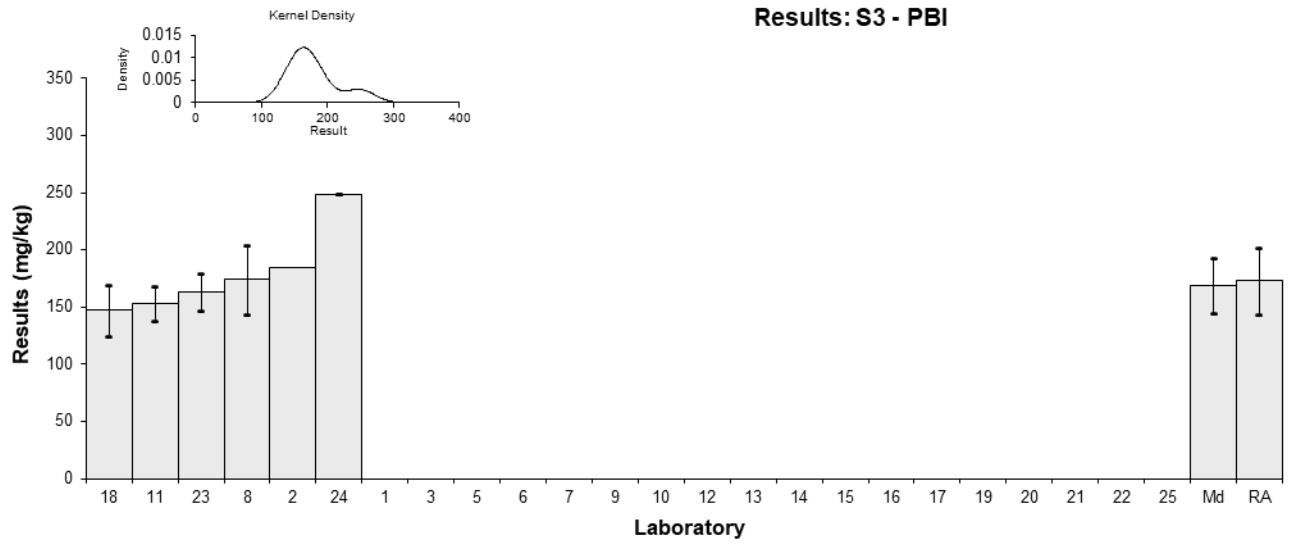


Figure 56

Table 68

Sample Details

Sample No.	S3
Matrix	Soil
Analyte	pH

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	7.6	0.1	-0.91	-0.81
2	8.37	0.08	1.89	1.73
3	7.5	0.2	-1.27	-0.99
5	7.6	0.41	-0.91	-0.50
6	NR	NR		
7	NT	NT		
8	8.41	NR	2.04	1.93
9	NT	NT		
10	8.28	2.07	1.57	0.21
11	7.7	0.2	-0.55	-0.43
12	NT	NT		
13	7.6	0.3	-0.91	-0.60
14	7.7	0.2	-0.55	-0.43
15	7.28	0.10	-2.07	-1.86
16	8.3	0.2	1.64	1.28
17	NT	NT		
18	8.35	0.3	1.82	1.20
19	NT	NT		
20	7.4	0.2	-1.64	-1.28
21	NT	NT		
22	7.4	0.2	-1.64	-1.28
23	8.35	0.2	1.82	1.42
24	7.815	0.014142135623	-0.13	-0.12
25	NT	NT		

Statistics

Assigned Value	7.85	0.29
Spike Value	Not Spiked	
Robust Average	7.85	0.29
Median	7.70	0.28
Mean	7.85	
N	16	
Max	8.41	
Min	7.28	
Robust SD	0.47	
Robust CV	6%	

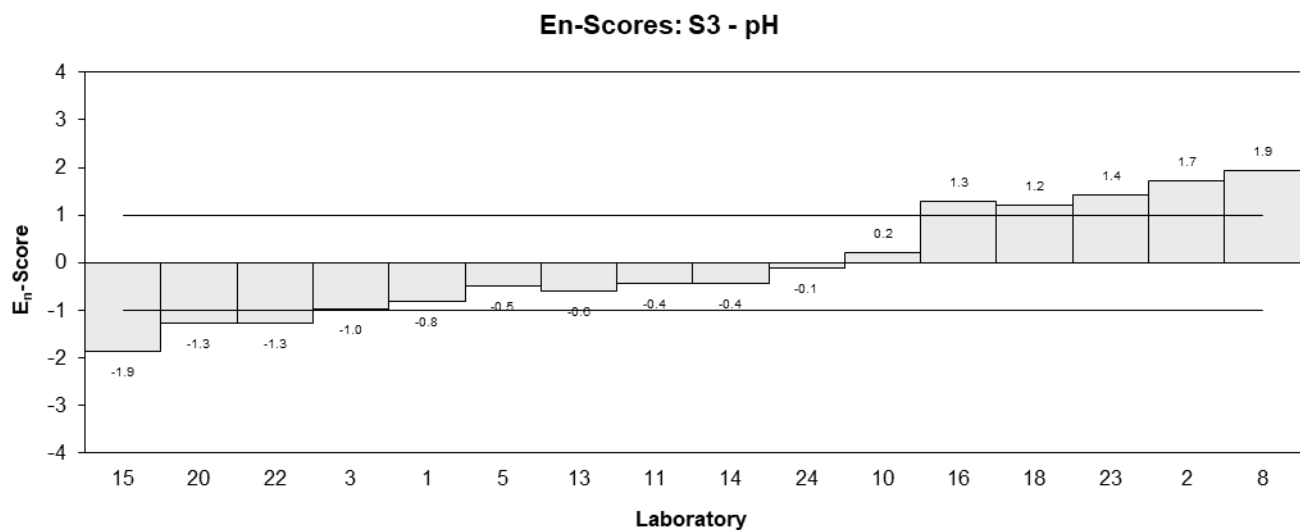
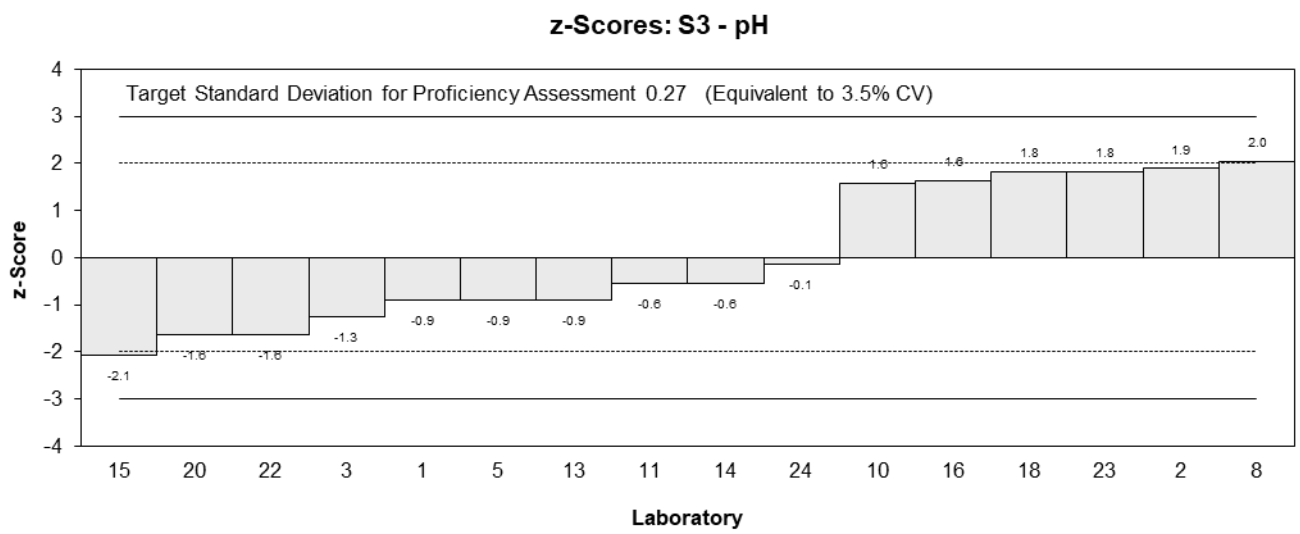
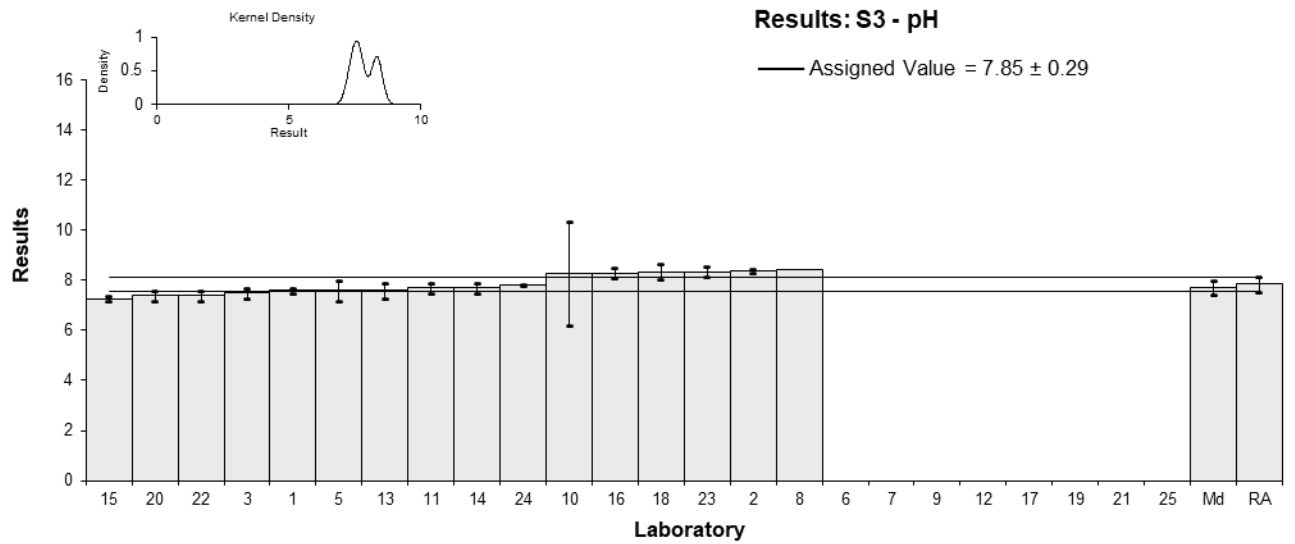


Figure 57

Table 69

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	7000	1050	-0.51	-0.33
2	7200	800	-0.24	-0.19
3	6900	1500	-0.65	-0.30
5	NR	NR		
6	8100	1100	0.98	0.60
7	NT	NT		
8	7600	70	0.30	0.44
9	NT	NT		
10	NT	NT		
11	7020	510	-0.49	-0.50
12	NT	NT		
13	NR	NR		
14	NT	NT		
15	NT	NT		
16	NT	NT		
17	NT	NT		
18	NT	NT		
19	NT	NT		
20	NT	NT		
21	NT	NT		
22	6800	2000	-0.79	-0.28
23	8500	850	1.52	1.14
24	7479.591836734	174.1018216608	0.13	0.19
	69	13		
25	NT	NT		

Statistics

Assigned Value	7380	500
Spike Value	Not Spiked	
Homogeneity Value	7100	1100
Robust Average	7380	500
Median	7200	370
Mean	7400	
N	9	
Max	8500	
Min	6800	
Robust SD	600	
Robust CV	8.2%	

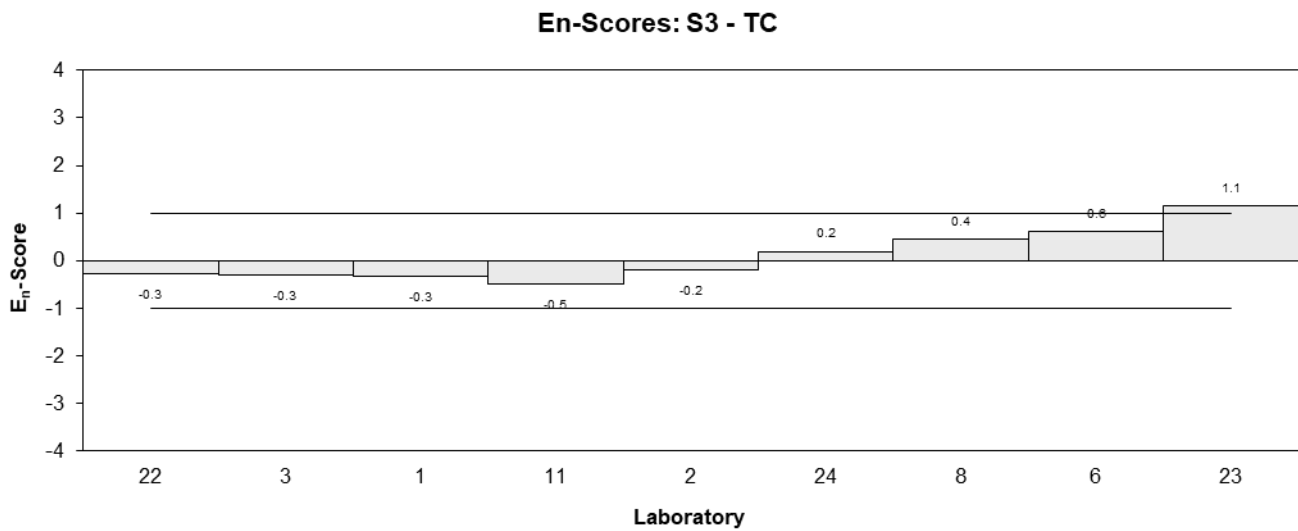
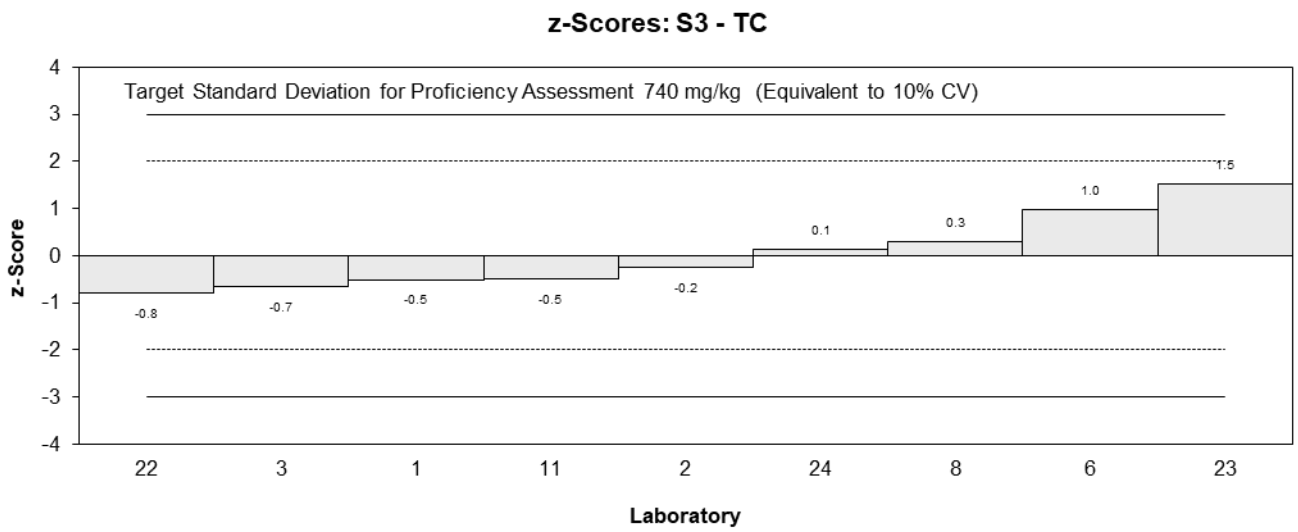
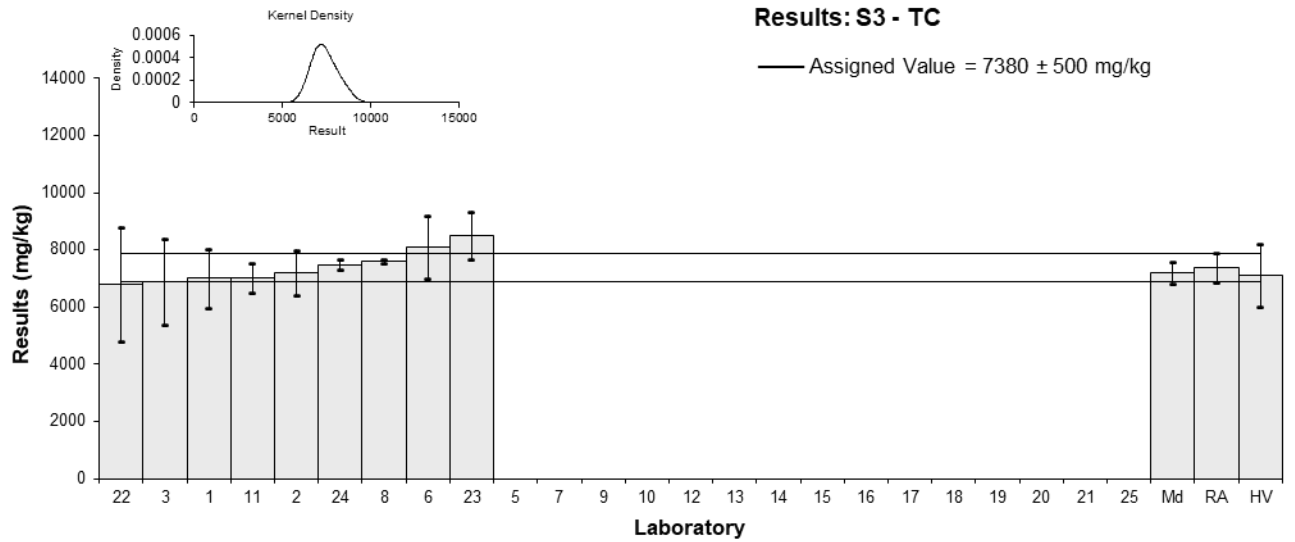


Figure 58

Table 70

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	680	100	0.03	0.02
2	600	100	-1.15	-0.67
3	660	200	-0.27	-0.09
5	NR	NR		
6	750	150	1.06	0.45
7	NT	NT		
8	800	80	1.80	1.22
9	NT	NT		
10	NT	NT		
11	630	70	-0.71	-0.52
12	NT	NT		
13	NR	NR		
14	NT	NT		
15	670.3	48.8	-0.11	-0.10
16	730	105.85	0.77	0.43
17	NT	NT		
18	532	122	-2.15	-1.07
19	NT	NT		
20	670	200	-0.12	-0.04
21	NT	NT		
22	620	200	-0.86	-0.28
23	900	90	3.27	2.05
24	647.1428571428 57	72.76838664721 17	-0.46	-0.33
25	NT	NT		

Statistics

Assigned Value	678	60
Spike Value	Not Spiked	
Homogeneity Value	660	99
Robust Average	678	60
Median	670	51
Mean	684	
N	13	
Max	900	
Min	532	
Robust SD	86	
Robust CV	13%	

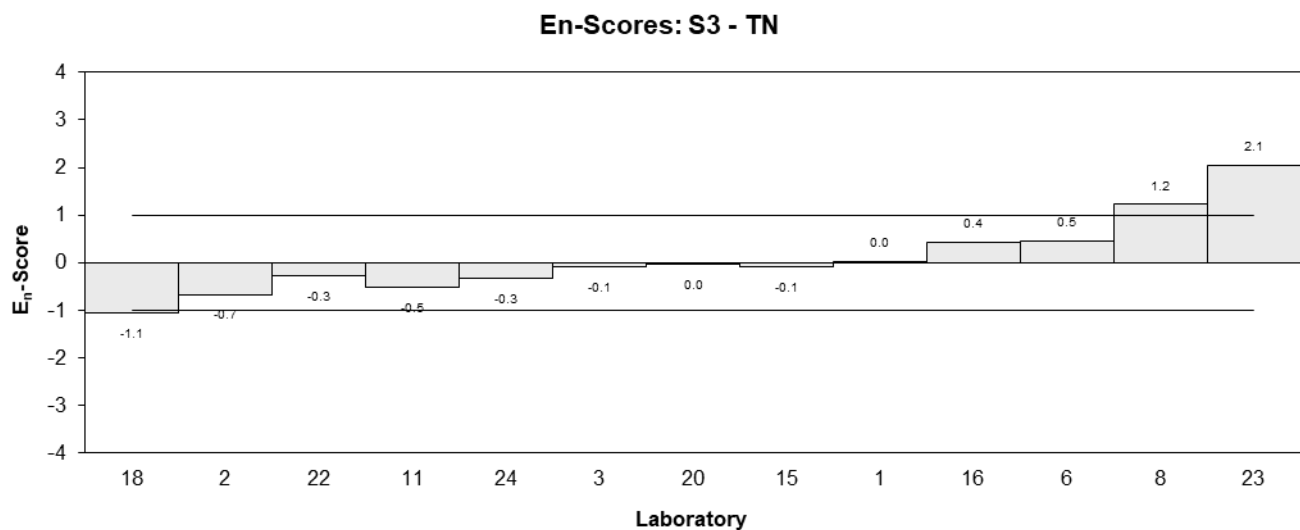
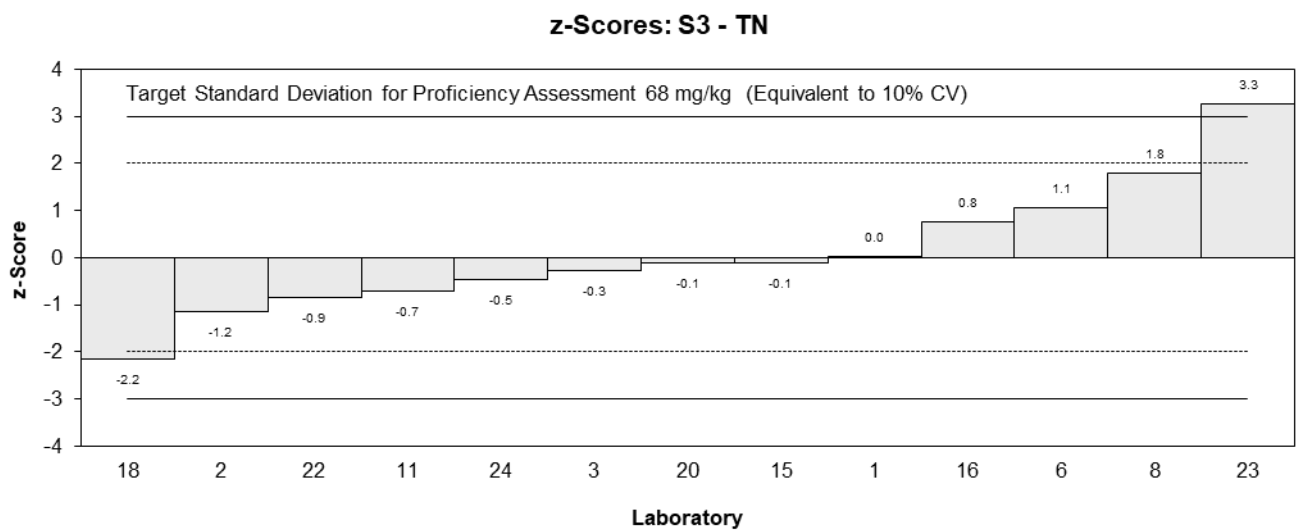
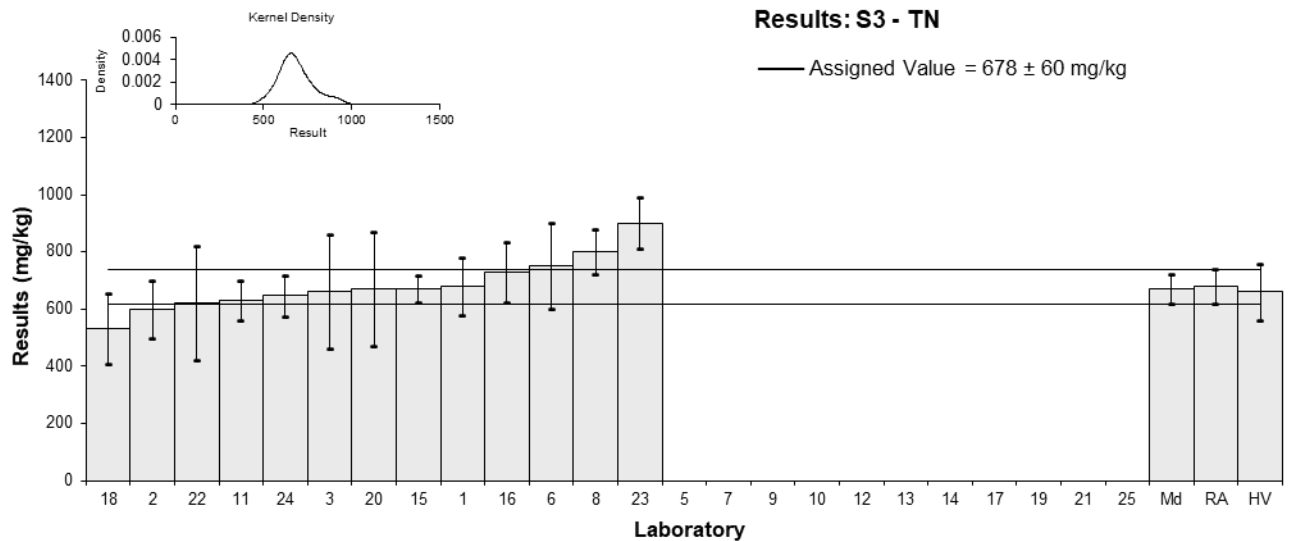


Figure 59

Table 71

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	6100	920	-0.87	-0.58
2	7200	853	0.78	0.56
3	5800	1500	-1.32	-0.57
5	NR	NR		
6	7100	1000	0.63	0.39
7	NT	NT		
8	6900	140	0.33	0.54
9	NT	NT		
10	NT	NT		
11	6700	670	0.03	0.03
12	NT	NT		
13	NR	NR		
14	NT	NT		
15	7080	1297	0.60	0.30
16**	0.68	0.06868	-10.00	-17.58
17	NT	NT		
18	6110	917	-0.85	-0.57
19	NT	NT		
20	6900	2000	0.33	0.11
21	NT	NT		
22	6700	2000	0.03	0.01
23*	15000	1500	12.46	5.38
24	6714.285714285 71	101.0152544552 21	0.05	0.09
25	NT	NT		

* Outlier, ** Gross Error, see Section 4.2

Statistics

Assigned Value	6680	380
Spike Value	Not Spiked	
Homogeneity Value	6900	1000
Robust Average	6750	410
Median	6810	300
Mean	7400	
N	12	
Max	15000	
Min	5800	
Robust SD	570	
Robust CV	8.4%	

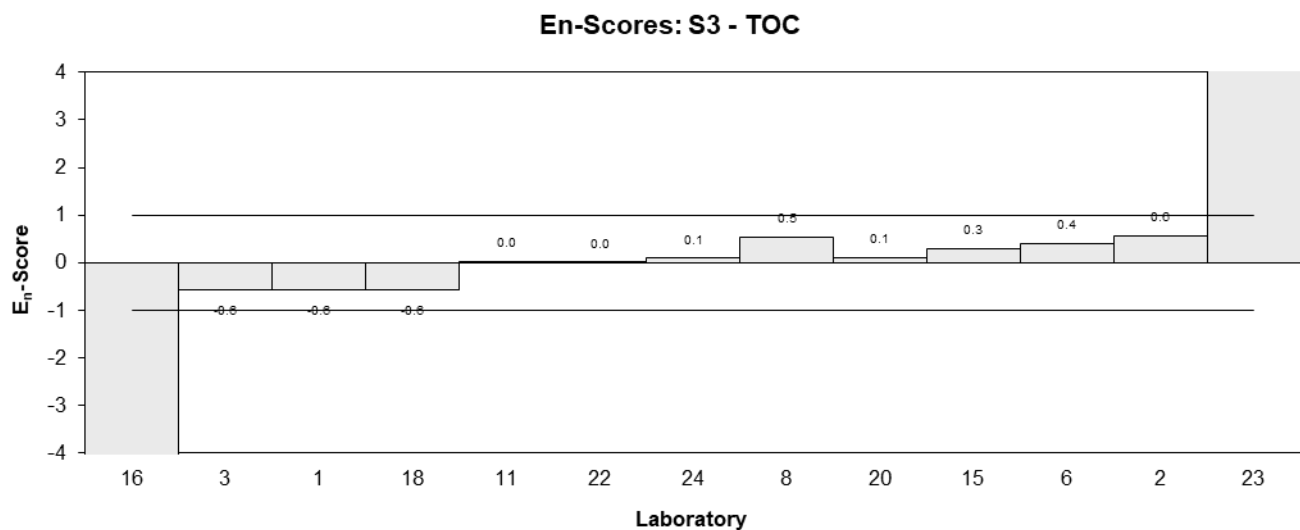
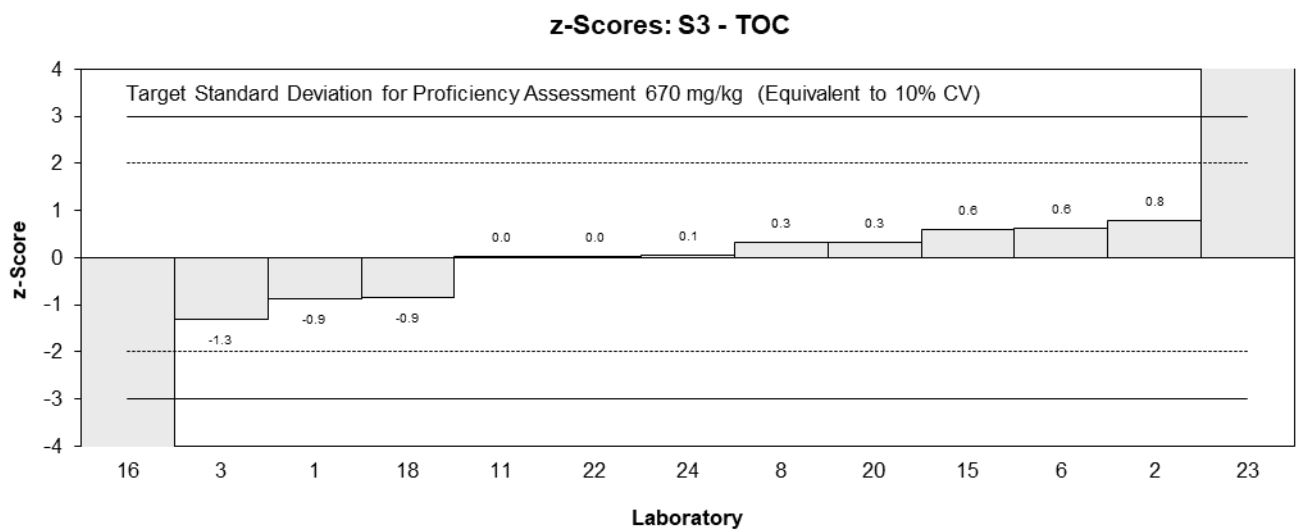
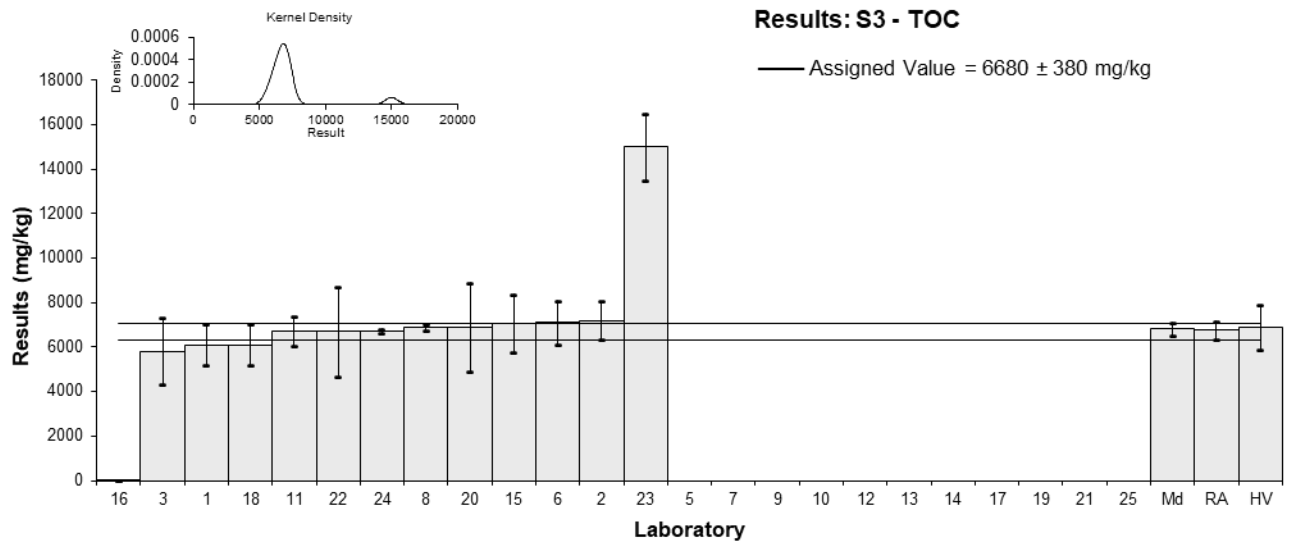


Figure 60

Table 72

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NR	NR		
2	NR	NR		
3	NR	NR		
5	NR	NR		
6	216	60	0.74	0.41
7	NT	NT		
8	204	40	0.43	0.30
9	NT	NT		
10	NT	NT		
11	180	26	-0.21	-0.19
12	NT	NT		
13	NR	NR		
14	NT	NT		
15	159.3	37.3	-0.76	-0.57
16	220	40.26	0.85	0.61
17	NT	NT		
18	136	20.4	-1.38	-1.31
19	NT	NT		
20	NT	NT		
21	NT	NT		
22	NR	NR		
23	NR	NR		
24	203.2	20.3	0.40	0.38
25	NT	NT		

Statistics

Assigned Value	188	34
Spike Value	Not Spiked	
Robust Average	188	34
Median	203	24
Mean	188	
N	7	
Max	220	
Min	136	
Robust SD	36	
Robust CV	19%	

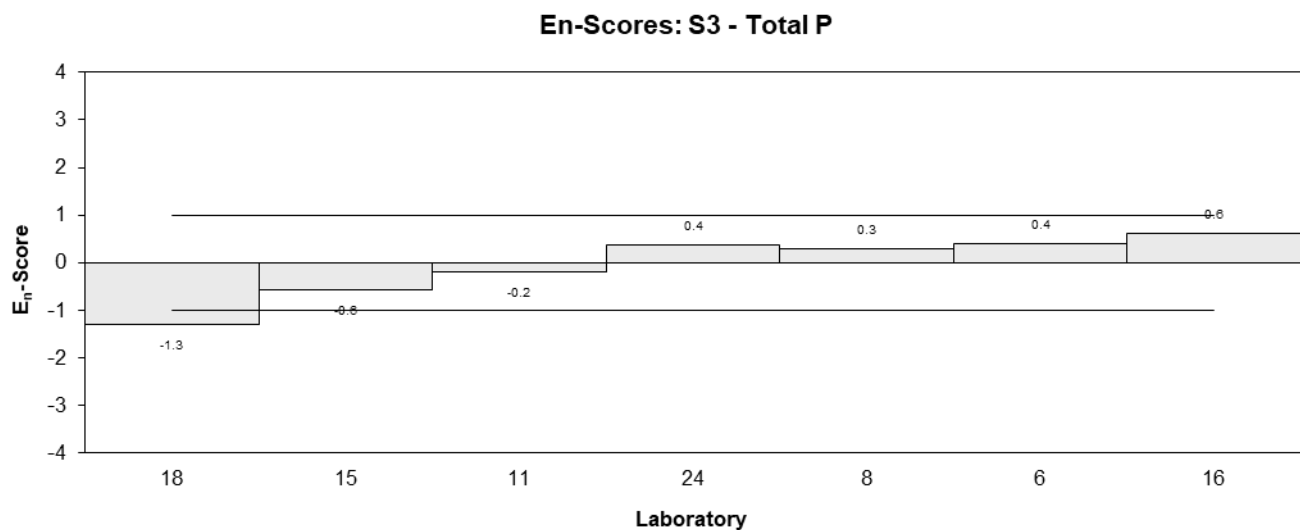
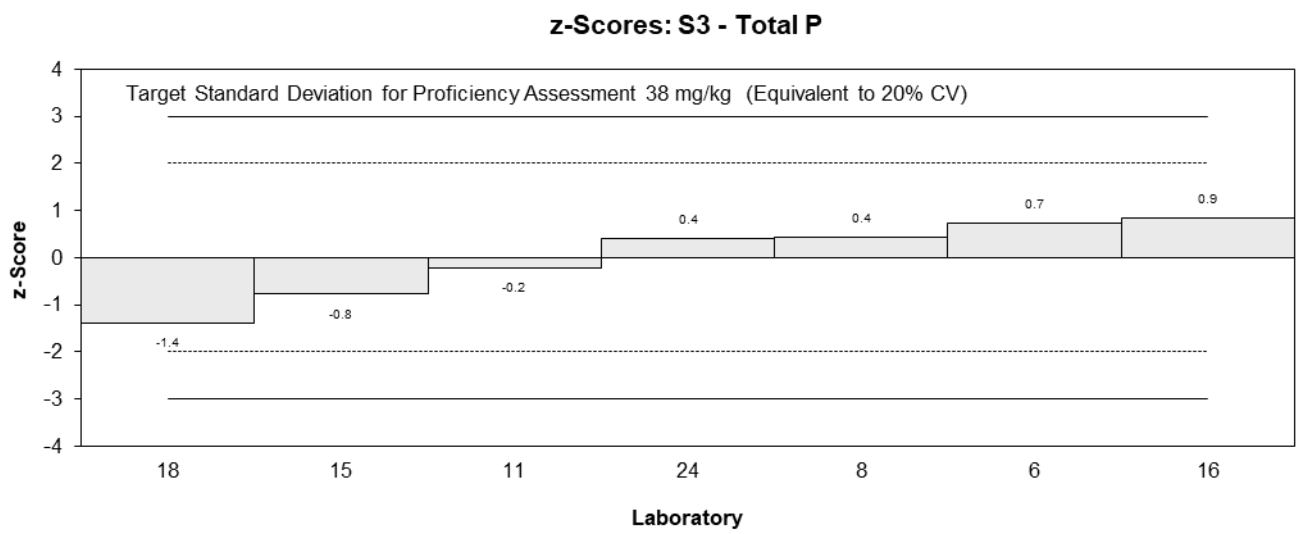
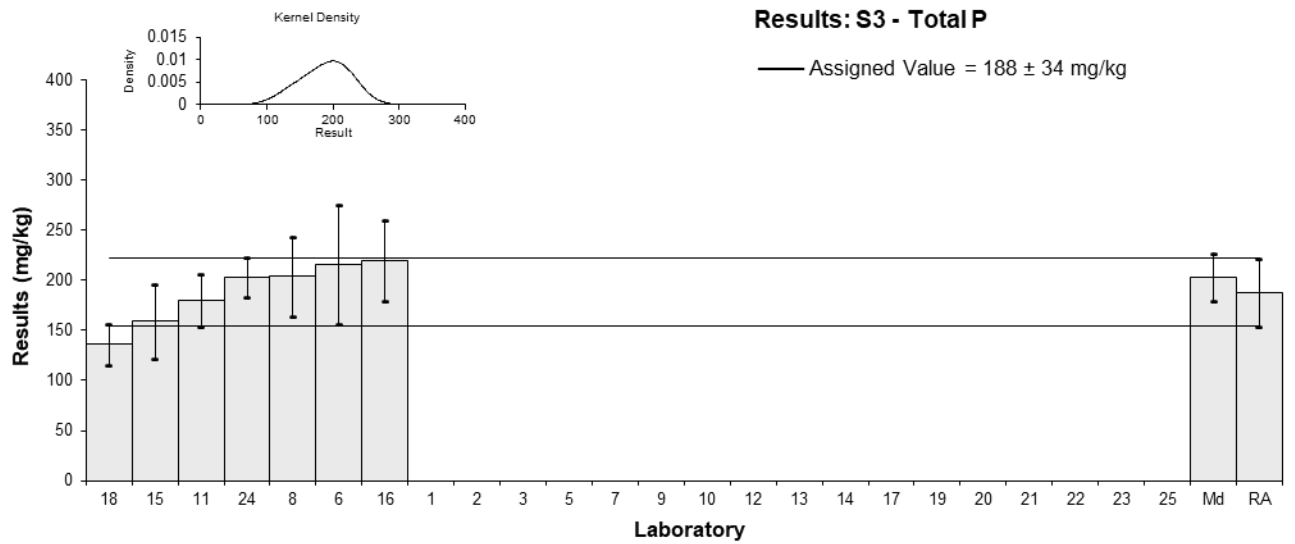


Figure 61

6 DISCUSSION OF RESULTS

6.1 Assigned Value

Sample S1 was dried fortified soil. Participants were asked to report results for Sample S1 on an as-received basis.

Sample S2— was fortified soil material to which a known amount of water was added. Participants were asked to use their normal method but to report results corrected for moisture content.

Sample S3 was dried agricultural soil.

Assigned values for 54 tests in the study samples were calculated as the robust averages of participants' results. The robust averages and their associated expanded uncertainties were calculated using the procedures described in ISO 13528.⁶ Outliers and results not included in robust average calculations were removed before the calculation of the assigned value (see Section 4.2 for additional information).^{3,4} Appendix 2 sets out the calculation of the robust average of As in Sample S1 and its associated uncertainty.

No assigned value was set for Rb in S1, Bi and Cs 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.

Losses during the drying process may explain the discrepancies between the spike value and the assigned value for Hg in S1 and S2. Similar differences have also been observed in previous studies.

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 791 numerical results, 773 (98%) were reported with an expanded measurement uncertainty. The magnitude of these expanded uncertainties was within the range 0.06% to 155% of the reported value. The participants used a wide variety of procedures to estimate the expanded measurement uncertainty. These are presented in Tables 11 and 12.

Approaches to estimating measurement uncertainty include standard deviation of replicate analysis, Horwitz formula, long term reproducibility, professional judgement, bottom-up approach, top-down approach using precision and estimates of method and laboratory bias, and top down approach using only the reproducibility from inter-laboratory comparison studies.^{9–15}

Participation in proficiency testing programs allows participants to check how reasonable their estimates of uncertainty are. Results and the expanded MU are presented in the bar charts for each analyte (Figure 2 to 61). As a simple rule of thumb, when the uncertainty estimate is smaller than 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. For example, 19 laboratories reported results for Mn in S1. The uncertainty of the assigned value estimated from the robust standard deviation of the 19 laboratories' results is 14 mg/kg (see equation 4, Appendix 2). Laboratory 25 might have under-estimated its expanded measurement uncertainties reported for Mn in S1 (3.95 mg/kg) as an uncertainty estimated from one measurement cannot be smaller than the uncertainty estimated from 19

measurements. Alternatively, estimates of uncertainties for As in S1 larger than 1.4 mg/kg (the uncertainty of the assigned value, 0.16 mg/kg plus the allowable variation from the assigned value, the target standard deviation of 0.62 mg/kg, multiplied by 2, the coverage factor for a confidence interval of 95%), should also be viewed as suspect. For example, the expanded measurement uncertainties reported by laboratories 3, 20 and 23 for As in S1 of 4 mg/kg, might have been over-estimated.

Laboratories 3 and 22 should review their procedure for estimating measurement uncertainty as many of their estimated uncertainties were over-estimated.

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 An example of estimating measurement uncertainty using proficiency testing data only is given in Appendix 3.

Laboratories 3, 12, 20, 21 and 22 attached estimates of the expanded measurement uncertainty to results reported as less than their limit of detection. An estimate of uncertainty expressed as a value cannot be attached to a result expressed as a range.⁹

Laboratories 3, 10, 22 and 24 reported an estimate of expanded uncertainty for some measurement results equal or larger than the results themselves.

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 2495.52 ± 374.33 mg/kg, it is better to report 2500 ± 370 mg/kg or instead of 9910 ± 1486.50 mg/kg, it is better to report 9910 ± 1500 mg/kg.⁹

Laboratory 24 reported results and uncertainties with up to 15 significant figures. Although all significant figures were used in the assessment of results (z and En- score calculation), the last 2 to 4 digits were omitted for some of these results presented in Tables and Figures Chapter 5.

6.3 z-Score

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

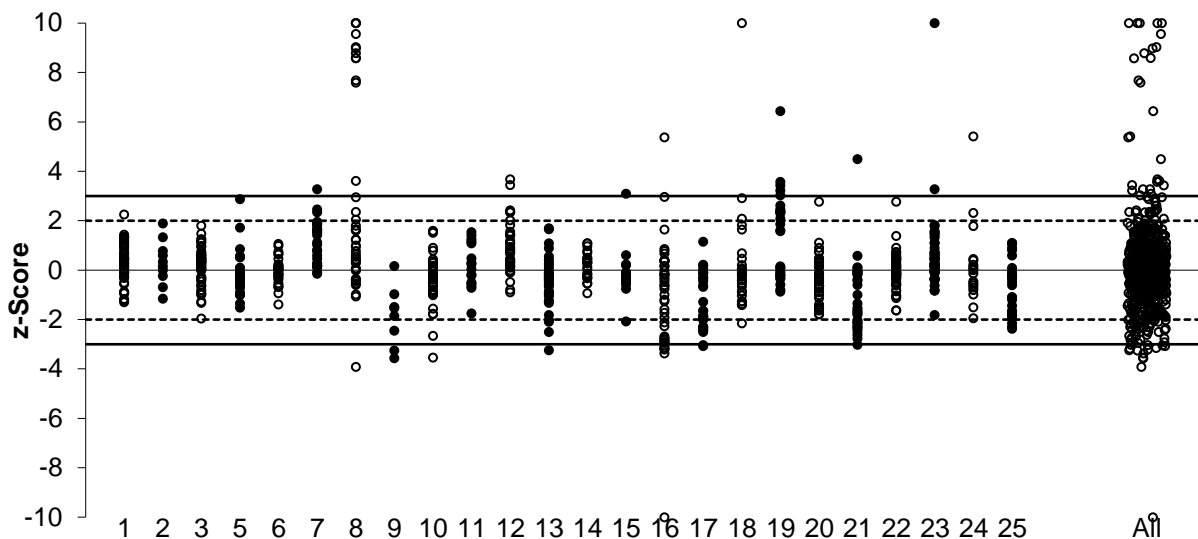
The target standard deviation defines satisfactory performance in a proficiency test. Target standard deviations equivalent to 3.5% to 25% PCV were used to calculate z-scores. Unlike the standard deviation based on between laboratories CV, setting the target standard deviation as a realistic, set value 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 73. The dispersal of participants' z-scores is presented in Figure 62 (by laboratory code) and in Figure 64 (by test). Of 763 results for which z-scores were calculated, 662 (87%) returned a satisfactory score of $|z| \leq 2.0$ and 60 (8%) 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.

Summary of participants' performance is presented in Figure 65. No laboratory reported results for all 54 tests for which a z-score was calculated.

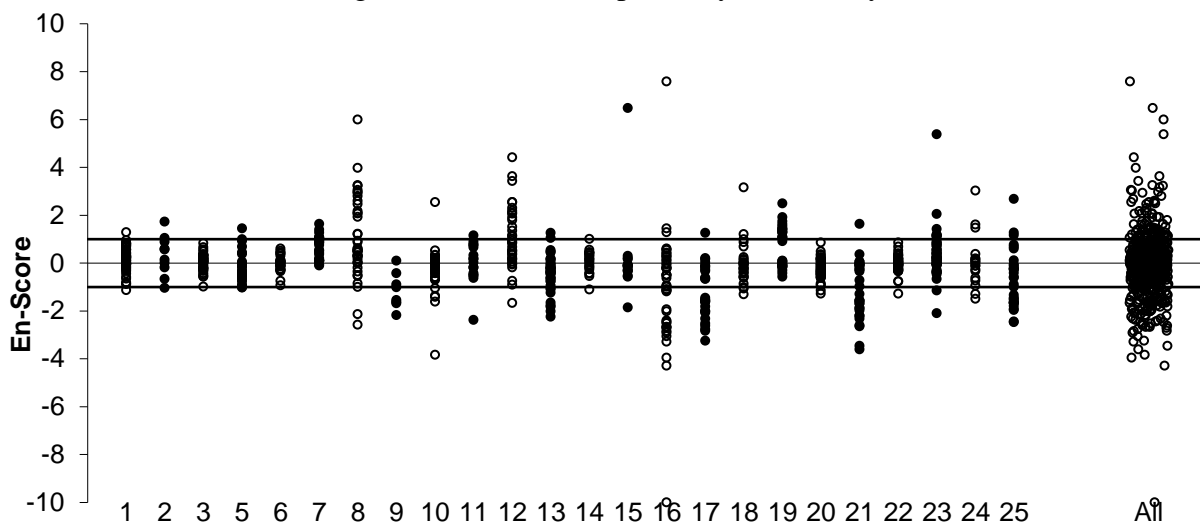
Laboratory 1 returned the highest number of satisfactory z-scores (51 out of 52 reported). Laboratory 1 also had the highest number of satisfactory En-scores at 50.

Laboratory 3 returned satisfactory results for 49 results out of a total of 49 reported.



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

Figure 62 z-Score Dispersal by Laboratory



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

Figure 63 E_n -Score Dispersal by Laboratory

6.4 E_n -score

E_n -score can be interpreted in conjunction with z-scores. The E_n -score indicates how closely a result agrees with the assigned value considering 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 63. 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.

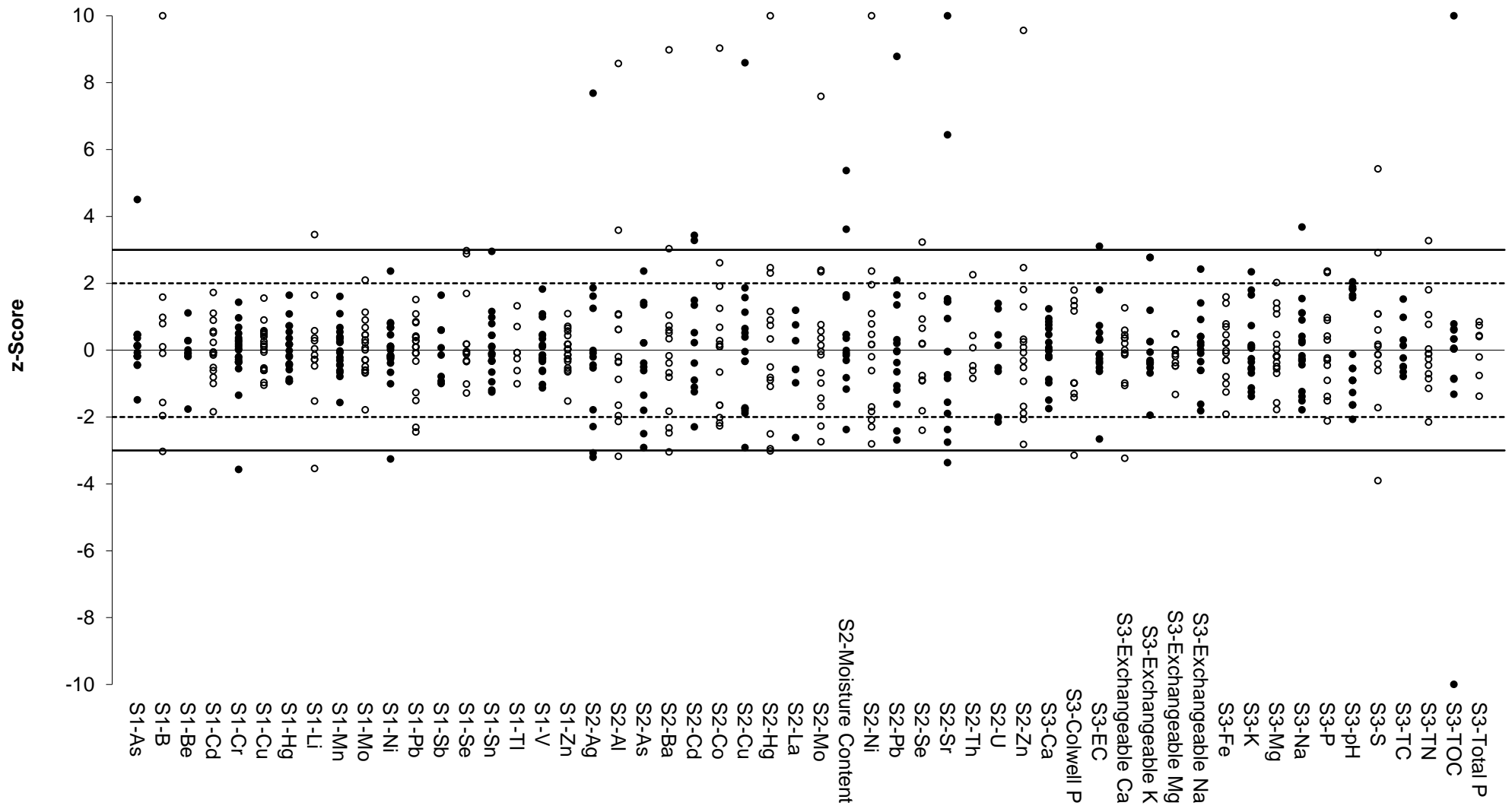
Of 763 results for which E_n -scores were calculated, 578 (76%) returned a satisfactory score of $|E_n| \leq 1$ indicating agreement of the participants' results with the assigned values within their respective expanded measurement uncertainties.

Table 73 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	4.12	5.9%	13%	15%
S1	B	5.1	30%	13%	20%
S1	Be	0.720	5.6%	17%	15%

Sample	Test	Assigned Value (mg/kg)	Between Laboratories CV*	Thompson/Horwitz CV	Target SD (as PCV)
S1	Cd	0.706	13%	17%	15%
S1	Cr	28.0	5.7%	9.7%	10%
S1	Cu	27.7	6.9%	9.7%	10%
S1	Hg	0.361	11%	19%	15%
S1	Li	3.13	9.2%	13%	15%
S1	Mn	356	6.7%	6.6%	10%
S1	Mo	13.4	7.4%	11%	10%
S1	Ni	17.8	6.3%	10%	10%
S1	Pb	21.2	9.2%	10%	10%
S1	Sb	5.6	25%	12%	25%
S1	Se	2.51	19%	14%	20%
S1	Sn	12.2	13%	11%	15%
S1	Tl	1.72	13%	15%	15%
S1	V	20.3	7.5%	10%	10%
S1	Zn	64.9	5.3%	8.5%	10%
S2	Ag	5.6	27%	12%	20%
S2	Al	7400	19%	4.2%	20%
S2	As	4.8	24%	13%	20%
S2	Ba	59.0	17%	8.7%	20%
S2	Cd	0.67	25%	17%	20%
S2	Co	8.8	27%	12%	20%
S2	Cu	40.8	25%	9.2%	20%
S2	Hg	0.61	30%	17%	20%
S2	La	11.3	19%	11%	20%
S2	Mo	24.3	20%	9.9%	20%
S2	Ni	32.0	28%	9.5%	20%
S2	Pb	43.3	21%	9.1%	20%
S2	Se	3.54	26%	13%	20%
S2	Sr	10.1	31%	11%	20%
S2	Th	2.76	19%	14%	20%
S2	U	3.21	27%	13%	20%
S2	Zn	62	22%	8.6%	20%
S2	Moisture Content	45.8%	17%	1.5%	20%
S3	Ca	5210	8.8%	4.4%	10%
S3	Fe	28100	15%	3.4%	15%
S3	K	5180	17%	4.4%	15%
S3	Mg	4990	11%	4.4%	10%
S3	Na	468	12%	6.3%	10%
S3	P	165	15%	7.4%	10%
S3	S	82.1	12%	8.2%	10%
S3	Colwell P	16.2	31%	11%	20%
S3	EC	233 µS/cm	6.3%	7%	10%
S3	Exchangeable Ca	22.2 cmol(+)/kg	7.4%	10%	10%
S3	Exchangeable K	2.12 cmol(+)/kg	14%	14%	15%
S3	Exchangeable Mg	10.5 cmol(+)/kg	4.8%	11%	10%
S3	Exchangeable Na	1.32 cmol(+)/kg	16%	15%	15%
S3	Extractable B	Not Set	NA	NA	Not Set
S3	PBI	Not Set	16%	NA	Not Set
S3	pH	7.85	6%	12%	3.5%
S3	TC	7380	8.2%	4.2%	10%
S3	TN	678	13%	6%	10%
S3	TOC	6680	7.5%	4.3%	10%
S3	Total P	188	19%	7.3% _s	20%

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



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

Figure 64: z-Score Dispersal by Element

Summary of Participant's Performance in AQA 23-02 Samples S1, S2 and S3

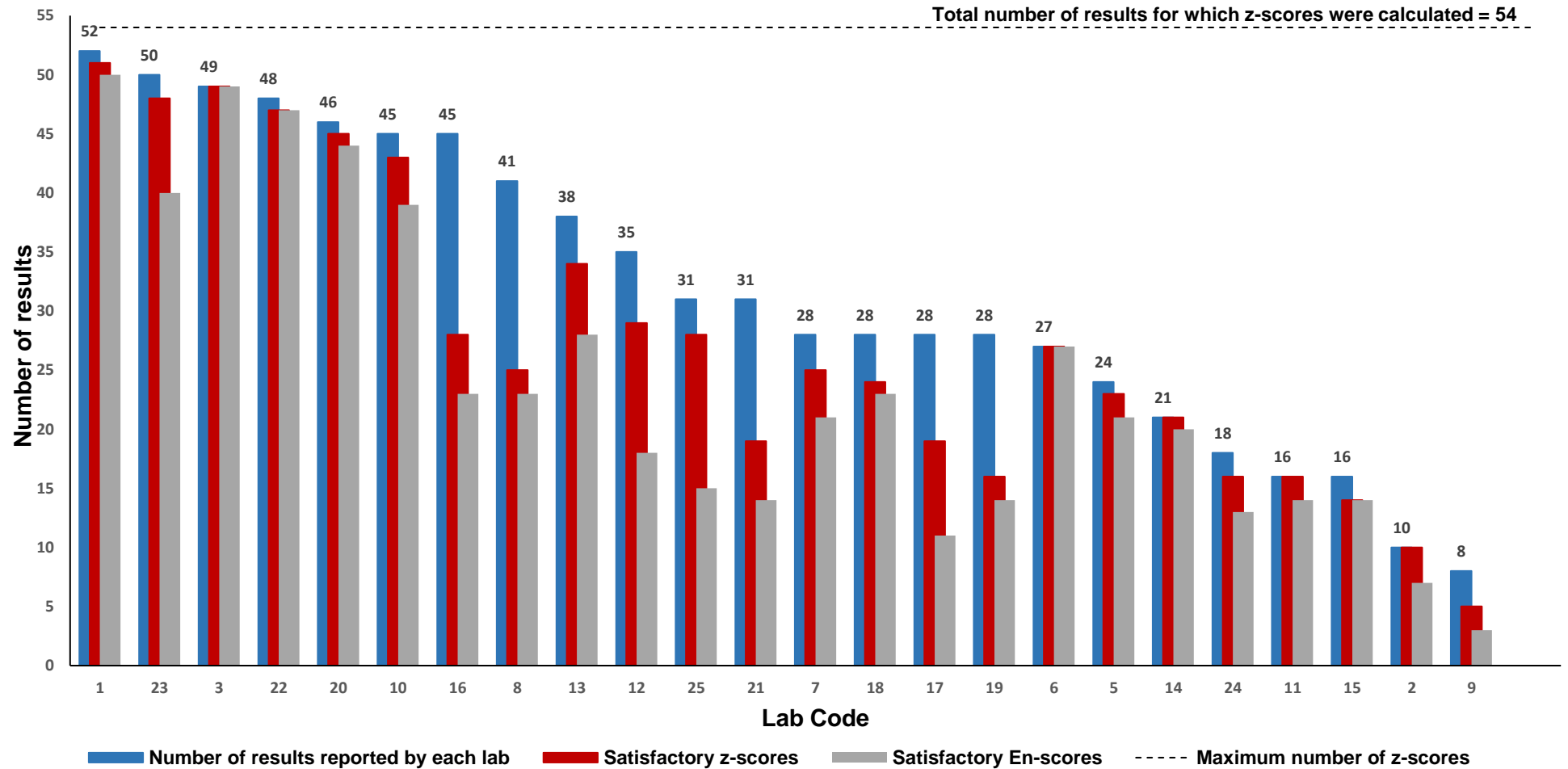


Figure 65: Summary of Participants Performance in AQA 23-02

Table 74 Summary of Participants' Results and Performance for Sample S1

Lab Code	As (mg/kg)	B (mg/kg)	Be (mg/kg)	Cd (mg/kg)	Cr (mg/kg)	Cu (mg/kg)	Hg (mg/kg)	Li (mg/kg)	Mn (mg/kg)	Mo (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Rb (mg/kg)	Sb (mg/kg)	Se (mg/kg)	Sn (mg/kg)	Tl (mg/kg)	V (mg/kg)	Zn (mg/kg)	
A.V.	4.12	5.1	0.720	0.706	28.0	27.7	0.361	3.13	356	13.4	17.8	21.2	Not Set	5.6	2.51	12.2	1.72	20.3	64.9	
H.V.	3.90	NA	0.698	0.75	27.0	28.0	0.333	NA	349	12.0	18.3	21.1	NA	NA	2.40	11.8	1.80	NA	64.2	
1	4.2	6.1	0.75	0.76	28	29	0.36	3.3	370	13	19	21	7.6	4.3	2.6	13	1.9	20	66	
2	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
3	4.4	3.1	<1	0.62	27	26	0.38	3.4	340	14	18	22	7.8	<7	<2	14	<2	21	65	
5	3.837	<20	0.709	0.888	24.211	27.700	0.352	<20	375.100	12.482	17.521	21.418	NT	<5	3.955	10.460	1.560	19.713	68.610	
6	4.4	NR	0.72	0.73	28.1	28.0	0.31	NR	332	12.5	17.5	21.4	NR	5.7	2.6	11.9	1.7	19.8	61.4	
7	4.19	<10	<2	0.69	28.50	28.41	0.37	<5	364.05	14.91	19.26	21.77	NT	<10	2.47	13.64	<10	21.23	66.08	
8	<15	<5	<1	<1	30.7	29.2	0.45	3.9	413	12.6	17.8	22.1	NT	NT	NT	17.6	<50	22.3	69.1	
9	3.2	NT	NT	0.51	18	25	0.37	NT	NT	NT	12	16	NT	NT	NT	NT	NT	NT	55	
10	4.34	5.91	0.529	0.651	28.8	30.2	0.329	1.47	345	13.4	17.4	24.4	NT	5.39	2.35	11.6	1.46	21.2	65.0	
11	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
12	<5	5.2	0.75	<1	32	32	0.4	4.75	380	14	22	23	NT	4.5	<5	13	<5	24	72	
13	<5	<10	<5	0.76550	26.4456 3	24.7788 7	0.33635	<10	348.021 97	12.7471 6	17.4806 0	21.0099 1	NT	<5	3.35684	11.6059 7	<5	19.8376 2	63.1395 2	
14	4.2	<10	<1	<1	29.4	28.8	0.31	3.15	368	13.8	19.2	21.8	NT	NT	<4	11.6	<2	22.5	69.5	
15	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
16	4	<5	0.7	0.6	29	26	0.37	NT	370	13	19	22	NT	<3	4	10	NT	19	66	
17	3.85	2.01	0.71	<1	27.2	28.2	<1	3.06	334	13.7	16.6	16.3	NT	NT	1.86	14.3	NT	19.6	65.1	
18	<5	28.0	<5	<1	29.9	26.2	0.338	NT	353	16.2	17.9	18.5	NT	7.9	<5	9.9	<5	18.2	60.7	
19	4.00	<10	<2	0.695	27.4	26.1	0.314	<5	328	13.5	17.4	20.5	NT	<10	2.49	12.4	<10	20.6	62.7	
20	4.2	3.5	<1	0.64	27	28	0.35	3	300	11	16	18	5	<7	<2	11	<2	18	64	
21	6.9	<20	0.7	0.7	28.3	29.3	0.338	2.41	355	12.6	17.1	21.4	5.82	4.2	<20	12	1.7	<100	64	
22	4	5	<1	0.7	27	26	0.4	3	340	13	18	21	6	<7	2	12	<2	20	61	
23	4.41	6.71	0.84	0.80	28.7	29.1	0.39	3.26	365	14.3	18.6	23.5	NT	6.44	2.45	NT	2.06	20.5	67.7	
24	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
25	4.0724	NR	NR	0.82235	27.4255 4	27.5341 6	0.41947	2.90415	394.977 68	14.6002 1	17.3194 3	22.9336 8	NT	6.4295	2.33416	12.3728	1.65296	19.0591 4	63.1311 3	

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

Table 75 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)	Cs (mg/kg)	Cu (mg/kg)	Hg (mg/kg)	La (mg/kg)	Mo (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Se (mg/kg)	Sr (mg/kg)	Th (mg/kg)	U (mg/kg)	Zn (mg/kg)	Moisture Content (%)
A.V.	5.6	7400	4.8	59.0	Not Set	0.67	8.8	Not Set	40.8	0.61	11.3	24.3	32.0	43.3	3.54	10.1	2.76	3.21	62	45.8
H.V.	5.26	NA	3.78	NA	0.742	0.736	8.26	1.16	36.4	0.588	10.1	21.6	31.0	39.3	2.98	11.2	2.78	2.78	56.4	37.2
1	NR	8960	6.1	65	NR	0.85	11	NR	50	0.72	14	28	39	55	4.2	13	4.0	3.5	78	35.1
2	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
3	7	8300	5	66	<1	0.74	10	<1	46	0.75	13	26	35	46	2.9	12	2.8	4	65	49
5	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	43
6	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
7	7.68	8300.33	6.16	71.28	< 10	1.11	12.17	NT	53.59	0.91	NT	35.68	44.47	57.55	4.69	13.19	NT	< 10	84.39	60.91
8	14.2	20088	<15	165	NT	<1	24.7	NT	110.9	2.8	NT	61.2	96.6	119.3	NT	31.5	NT	NT	180.5	78.9
9	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
10	5.36	6849	4.31	49.5	NT	0.618	7.63	NT	38.1	0.548	9.09	23.6	30.7	43.0	2.89	6.95	2.29	2.87	55.5	44.3
11	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
12	5.5	9000	<5	63	NT	<1	9.1	NT	44	0.5	NT	25	37	45	<5	13	NT	NT	66	45.7
13	<5	6875.50 3	<5	54.3496 8	NT	0.50237	5.24081	NT	25.9560 3	0.30399	11.9248 0	19.4735 8	18.6512 8	37.6608 8	3.68343	8.38332	<5	<5	50.341 03	NR
14	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
15	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
16	2	2700	2	23	NT	<0.3	4.8	NT	17	0.25	NT	11	14	20	3	3.3	NT	NT	27	95
17	2.15	4960	2.40	31.5	NT	<1	5.9	NT	25.4	<1	NT	13.2	20.2	22.3	1.84	6.27	NT	1.92	38.6	45
18	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
19	7.40	12700	7.07	94.8	<10	1.13	13.4	NT	56.0	0.89	NT	35.9	47.1	61.4	5.83	23.1	NT	<10	92.5	60.3
20	5.1	6100	4.2	51	<1	0.55	9.3	1	38	0.65	10	21	28	40	<2	8.6	2.5	2.8	58	50
21	3.6	4240	3.5	29.7	0.56	0.362	4.97	0.84	26.5	0.241	5.37	16.1	17.3	29.3	<20	4.52	NT	1.91	36.3	24
22	5	7100	5	57	1	0.7	9	2	45	0.7	10	27	33	34	4	10	3	4.1	63	50
23	5.59	6888	4.43	67.5	0.80	0.87	8.98	NT	40.4	0.51	NT	24.1	33.1	42.9	3.67	10.0	2.42	3.30	61.0	38.2
24	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
25	3.03491	4504.86 7	3.07584	37.3977 6	0.51027	0.52071	5.90303	NR	26.6946 9	0.47762	NT	17.3178 6	21.1297 7	32.8830 6	2.25367	5.28664	NT	1.8296	41.006 52	NR

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

Table 76 Summary of Participants' Results and Performance for Sample S3

Lab Code	Ca (mg/kg)	Fe (mg/kg)	K (mg/kg)	Mg (mg/kg)	Na (mg/kg)	P (mg/kg)	S (mg/kg)	Colwell K (mg/kg)	Colwell P (mg/kg)	EC (µS/cm)
A.V.	5210	28100	5180	4990	468	165	82.1	Not Set	16.2	233
H.V.	5110	27900	5220	4960	433	156	NA	NA	NA	NA
1	5330	26800	4980	4710	410	150	87	NR	12	240
2	NR	NR	NR	NR	NR	NR	NR	NR	20.5	241
3	4700	28000	4900	5000	520	160	83	700	21	275
5	5654	23840	4640	4746	397	NR	NR	NR	13	245
6	5120	31000	5300	4800	454	142	77	NR	NR	NR
7	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
8	5850	31400	5245	5610	447.5	204	50	NT	13	224
9	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
10	5203	34800	5270	5080	384	161	83.5	NT	NT	171
11	4300	NR	NR	4900	540	170	91	750	20	218
12	5700	34000	7000	6000	640	NT	<1	NT	NT	NT
13	5246.975	22781.69541	6458.926	5528.802	487.385	179.615	80.932	NR	<50	230
14	5450	26800	4760	4900	456	181	91	NT	NT	230
15	5097	27703	4734	4887	452.9	157.4	78.73	NT	NT	305.3
16	5200	20000	4200	4200	460	130	68	NT	6	230
17	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
18	4750	29000	5220	5220	403	NT	106	680	11.6	227
19	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
20	5600	29000	4100	4100	520	140	81	NT	NT	250
21	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
22	5100	30000	4300	4900	510	160	NR	NR	NR	240
23	5544	28976	5750	5696	481	172	<100	2309	22	225
24	4430.2	24723.1	6570.6	4640.0	478.2	203.2	126.6	238.5	13.0	220.6
25	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, NA = Not Available

Table 76 Summary of Participants' Results and Performance for Sample S3 (continued)

Lab Code	Exchangeable Ca (cmol(+)/kg)	Exchangeable K (cmol(+)/kg)	Exchangeable Mg (cmol(+)/kg)	Exchangeable Na (cmol(+)/kg)	Extractable B (mg/kg)	PBI+CoIP (mg/kg)	pH	TC (mg/kg)	TN (mg/kg)	TOC (mg/kg)	Total P (mg/kg)
A.V.	22.2	2.12	10.5	1.32	Not Set	Not Set	7.85	7380	678	6680	188
H.V.	NA	NA	NA	NA	NA	NA	NA	7100	660	6900	NA
1	22	2.1	11	1.3	NR	NR	7.6	7000	680	6100	NR
2	23.5	1.9	10.5	1.35	NR	185.0	8.37	7200	600	7200	NR
3	20	2.2	11	1.4	1.4	NR	7.5	6900	660	5800	NR
5	NR	NR	NR	NR	NR	NR	7.6	NR	NR	NR	NR
6	NR	NR	NR	NR	NR	NR	NR	8100	750	7100	216
7	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
8	19.85	1.95	10.5	1.25	3.35	174	8.41	7600	800	6900	204
9	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
10	NT	NT	NT	NT	NT	NT	8.28	NT	NT	NT	NT
11	25	2.2	11	1.6	NT	153	7.7	7020	630	6700	180
12	22	2.5	10	1.8	NT	NT	NT	NT	NT	NT	NT
13	15	2.0	9.1	1.2	NR	NR	7.6	NR	NR	NR	NR
14	NT	NT	NT	NT	NT	NT	7.7	NT	NT	NT	NT
15	22.638	2.022	10.372	1.366	NT	NT	7.28	NT	670.3	7080	159.3
16	23	2.0	10	1.4	NT	NT	8.3	NT	730	0.68	220
17	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
18	21.9	1.99	10.3	1.32	NT	147	8.35	NT	532	6110	136
19	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
20	23	3	11	1.5	NT	NT	7.4	NT	670	6900	NT
21	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
22	23	3	11	1	2.3	NR	7.4	6800	620	6700	NR
23	22.2	2.01	10.1	0.96	0.95	163	8.35	8500	900	15000	NR
24	23.2	1.5	10.5	1.2	1.51	248.6	7.815	7479.59183673 4	647.142857142 8	6714.28571428 5	203.2
25	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, NA = Not Available

6.5 Participants' Results and Analytical Methods for Acid Extractable Elements

Sample S1 was dried soil while Sample S2 was wet sludge which required a good preparation procedure to subsample a representative test portion. An appropriate calculation/reporting procedure was also required for this sample as participants were asked to report results corrected for moisture content.

A summary of participants' results and performance is presented in Tables 74 to 76 and in Figures 62 and 64. The results reported for most elements in the Sample S2 were significantly more variable, relative to Sample S1.

Most participants were challenged by the process of subsampling a representative test portion from the sludge sample S2 and reporting results for this sample corrected for moisture content.

Sb followed by Se were the elements which presented most analytical difficulty to participants in Sample S1, with between-laboratory CVs of 25% and 19% respectively.

Measurement of Ca, Fe, K, Mg, Na, P and S also presented difficulty to participating laboratories. The between-laboratory CV for these tests was almost double that predicted by Thomson and Horwitz.

Cr and Zn in S1 were the tests that presented the least analytical difficulty to participating laboratories, with a between-laboratory CV under 6%.

The results reported by laboratories 8,16,17,18,19 and 21 in Sample S2 were consistently either lower or higher than the robust average and spike value by around the same factor for almost all analytes (Figure 66 and Table 75). The results reported by these laboratories in S2 were not included in the analysis of the extraction methods and instrumental techniques employed by participants.

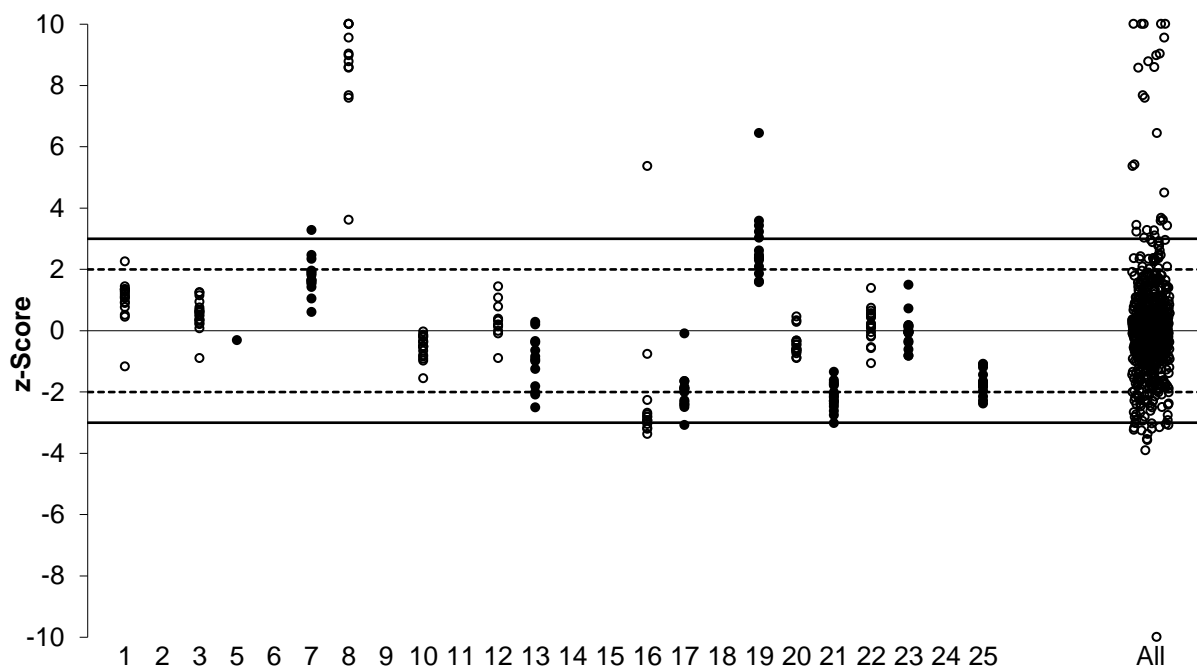


Figure 66 S2 z-Score Dispersal by Laboratory

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

Moisture Content and Reporting Results on a Dry Weight Basis (Corrected for Moisture Content).

Laboratories 16, 17 and 21 may have reported results on as received basis. Their results were lower than the assigned value by approximately 50%.

Laboratory 19 may have reported solid content (fraction dry mass) rather than the moisture content (loss on evaporation) and corrected results for solid content, as their results were higher than the assigned value by approximately same factor 1.5 (60% divided by 40%). Laboratory 8 may also have corrected for solid content in their results, as their results were approximately 3 times higher than the assigned values (they reported a result of 78.9% for moisture content).

The mass fraction of elements in the sample is calculated by multiplying the concentration of analyte in the final solution multiplied by the dilution factor and further divided by the amount of sample taken for analyses. If the sample is analysed as received, then to correct for moisture content the result is divided by the percentage of solid content in the sample multiplied by 100. If analysis was conducted upon an already dried sample, then correction for moisture content is no longer required.

Extraction Methods

The request was for acid extractable elements; NMI PT studies of metals in soil focus on ‘pseudo-total’ analyses of elements in soil rather than on true total metal content because when an assessment of the anthropogenic impact of the metal content in a soil sample is made, aggressive digestion regimes (HF, high digestion temperature) can lead to misleading conclusions – since metals can be extracted from the fraction naturally present in the soil matrix.^{5, 16-19} While an aggressive digestion regime can produce high, misleading results, weak digestion regimes (low digestion temperature, reduced digestion time, diluted acids and/or a low ratio of acid to sample size) may extract just a fraction of the contaminants from the soil.

In the present study, the samples were dried soil and moist soil. Participants used various sample sizes, digestion temperatures and digestion times.

All participants but one used both HNO₃ and HCl as extraction agents and most used a digestion temperature of 90°C to 100°C.

One laboratory used dilute HNO₃ and hydrogen peroxide and digested their samples at 95°C for 180 min. The digestion regime used might have extracted only a fraction of acid extractable elements from the soil Sample S1 as all the results reported by this laboratory were lower than the assigned value except for Hg.

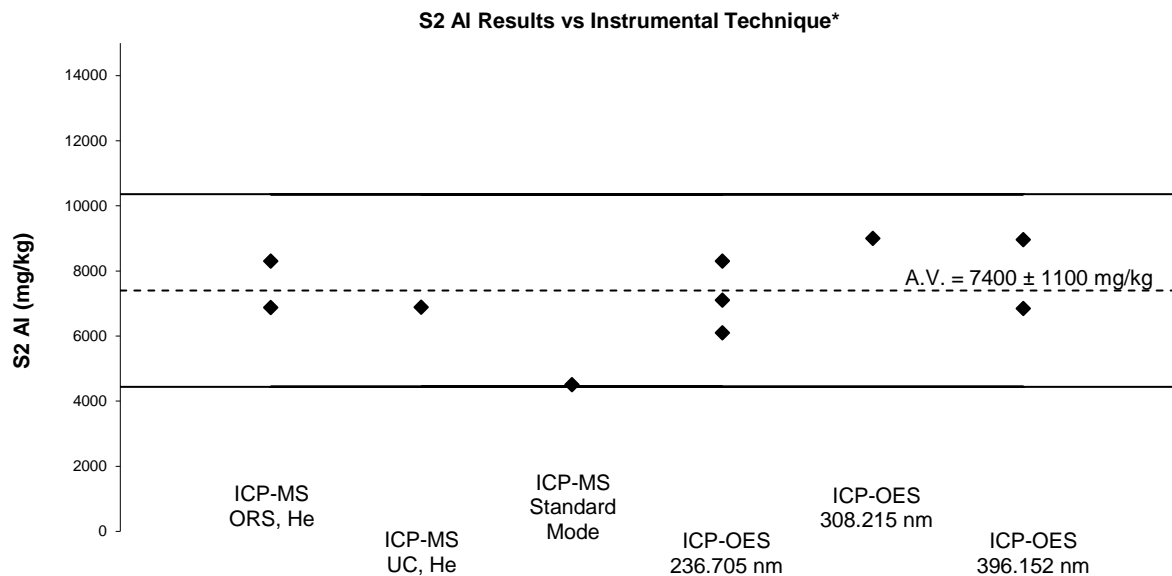
One laboratory used a digestion temperature of 85°C for 240 min and a large ratio of acid to sample size of 10 mL to 0.25 g of dried soil. All results reported by this laboratory for acid extractable elements returned satisfactory z-scores but one. Caution should be exercised when a small sample size is taken for analysis as this may not be representative of the whole sample.

Laboratory 24 reported conducting their extraction procedure at 175°C for 15 min. They used a sample size of 0.5 g and 9 mL of HNO₃ and 3 mL of HCl. Both of their unsatisfactory results were higher than the assigned value.

Laboratory 1 conducted a staggered digestion: “*Made to 20mL after one-hour, final hour with additional vertexing and final vol 40mL.*” All results reported by them returned satisfactory z-score except for one.

Individual Element Commentary

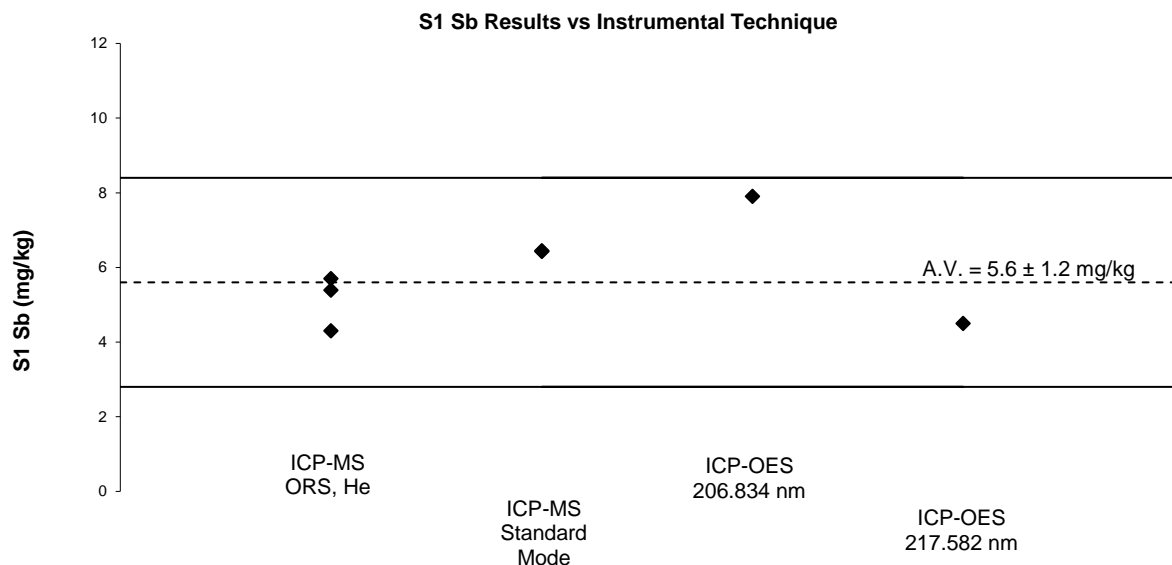
Aluminium Plots of results for Al versus instrumental technique used by participants are presented in Figure 67.



*Results from laboratories 8, 16, 17, 19 and 21 were excluded. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2

Figure 67 S2 Al Results vs. Instrumental Technique

Antimony level in S1 was low at 5.6 mg/kg which may have challenged participants' analytical techniques. The between-laboratory CV was 25%, more than double than the CV predicted by Thomson/Horwitz CV of 12%. There was no evident relationship between participants' results for Sb and the instrumental technique they used (Figure 68).



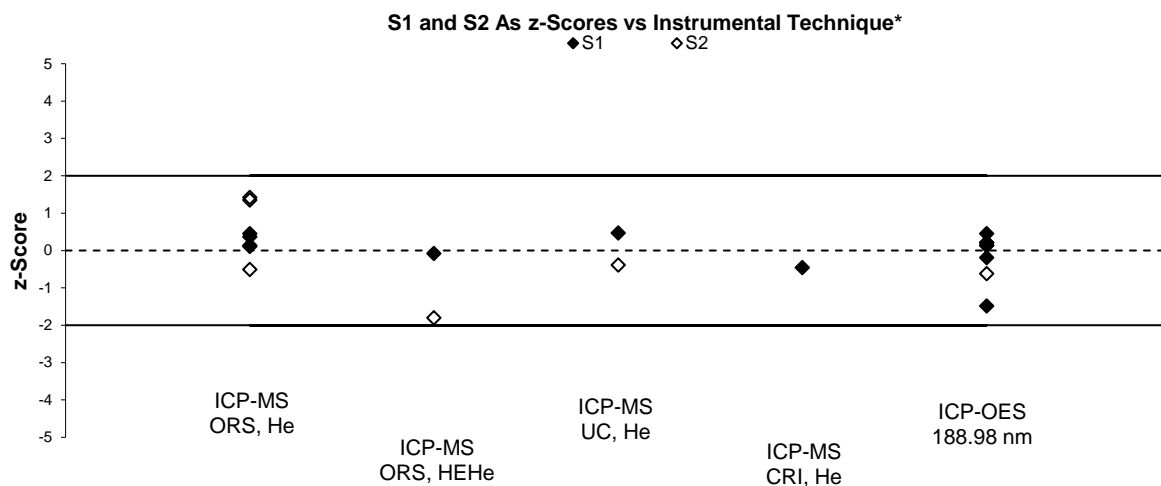
Horizontal lines on charts are the results corresponding to z-scores of 2 and -2

Figure 68 Sb Performance in S1 vs. Instrumental Technique

Arsenic level in the study samples S1 and S2 was similar, 4.12 mg/kg and 4.8 mg/kg respectively. The between-laboratory CV for As in the dried soil sample was 5.9% and almost 5 times higher in the sludge sample S2, at 24%.

Figure 69 presents plots of participants' z-scores versus instrumental technique used. Most participants used ICP-MS in collision mode or ICP-OES with a wavelength of 188.98 nm.

One laboratory reported using ICP-MS/MS in helium mode. Caution should be exercised when measuring elements at a low level in MS/MS mode combined with He as a collision gas (e.g. As in the diluted digest of the sludge sample S2). The use of MS/MS mode as opposed to single-quad mode will cause a sensitivity decrease of approximately 30%, in addition to the sensitivity loss from using a collision gas. Furthermore, the combination of He collision gas and MS/MS may have limited benefits. Consider using MS/MS with O₂ or single quad with He.



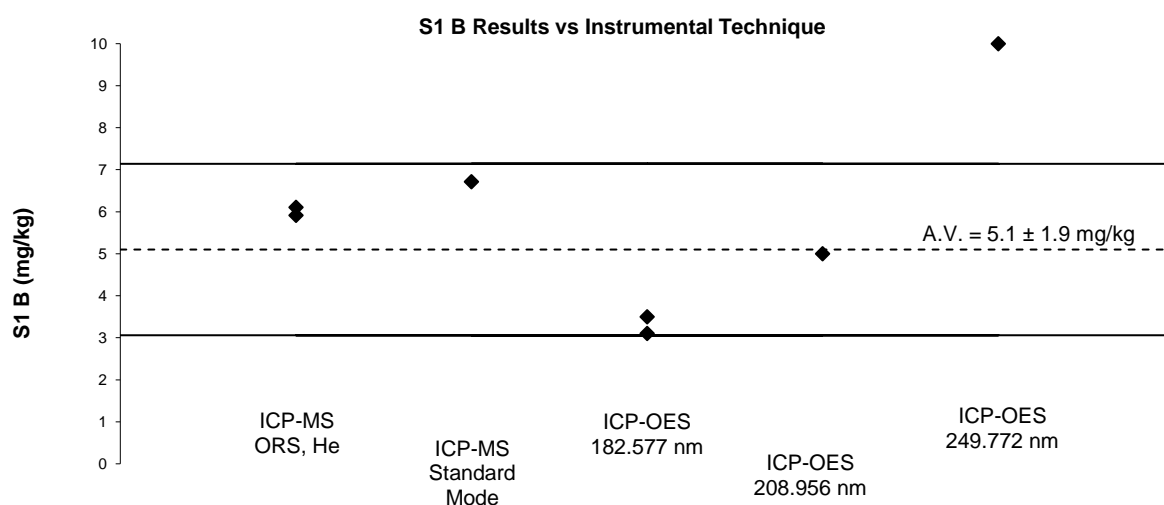
*S2-As results from Laboratories 8, 16, 17, 19 and 21 were excluded. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2

Figure 69 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%).

Caution should be exercised when B is measured by ICP-OES at 249.7 nm, because it can have significant interferences from Fe 249.771 nm if on-line inter-element correction is not used.

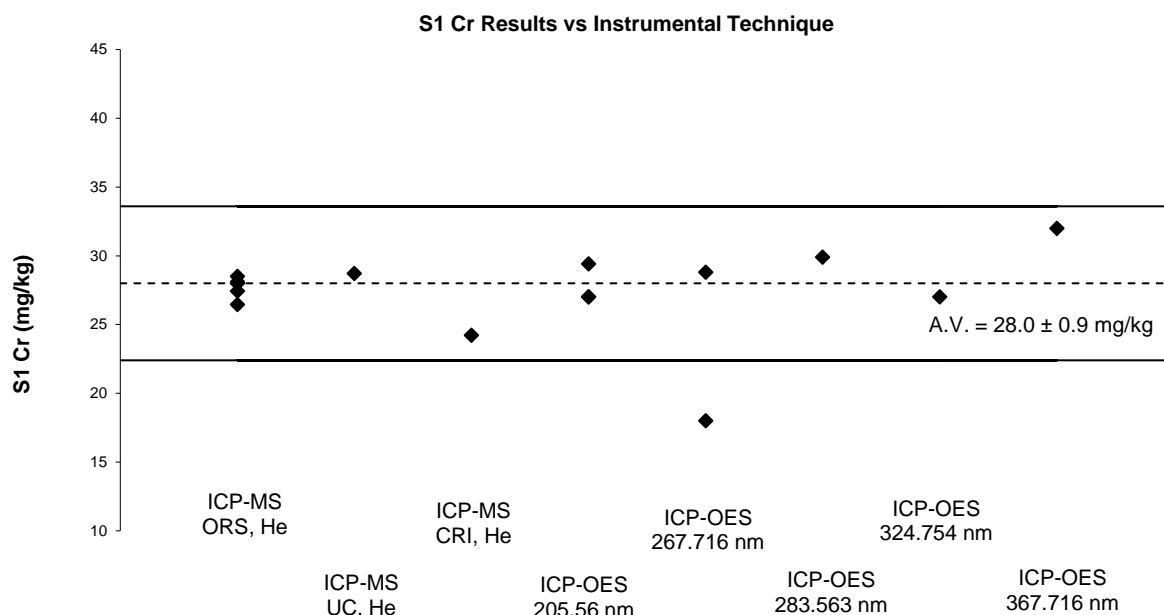
Plots of participants' results versus instrumental technique used are presented in Figure 70.



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

Figure 70 B Results in S1 vs. Instrumental Technique

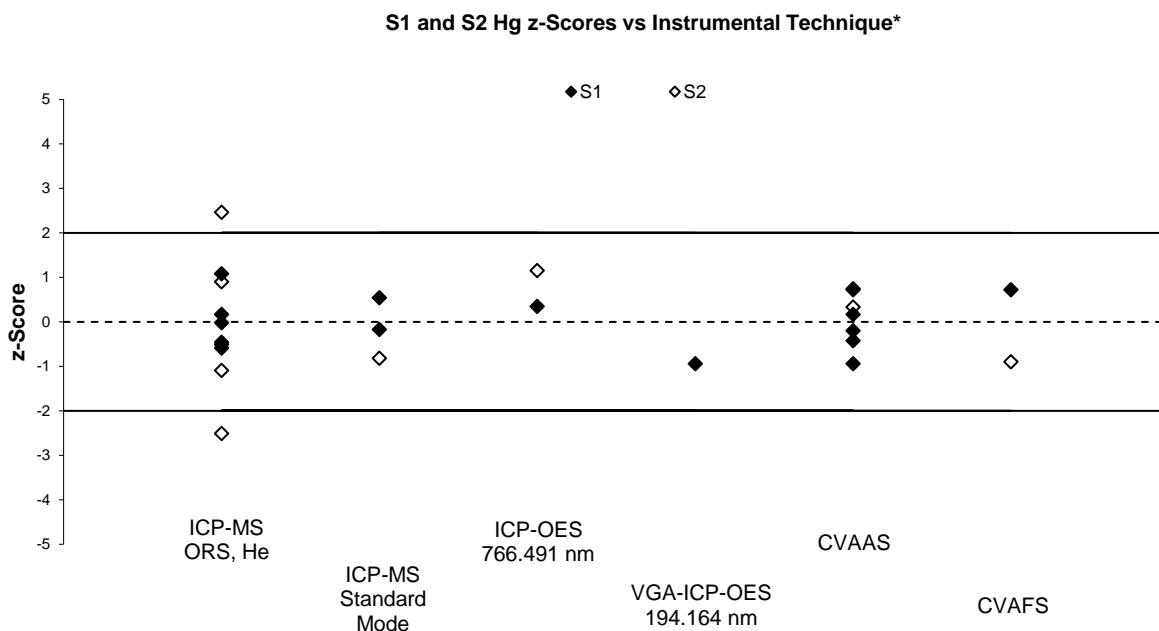
Chromium Figure 71 presents plots of participants' z-scores versus instrumental technique used. Most participants reported using ICP-OES with various wavelengths.



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

Figure 71 S1Cr Results vs. Instrumental Technique

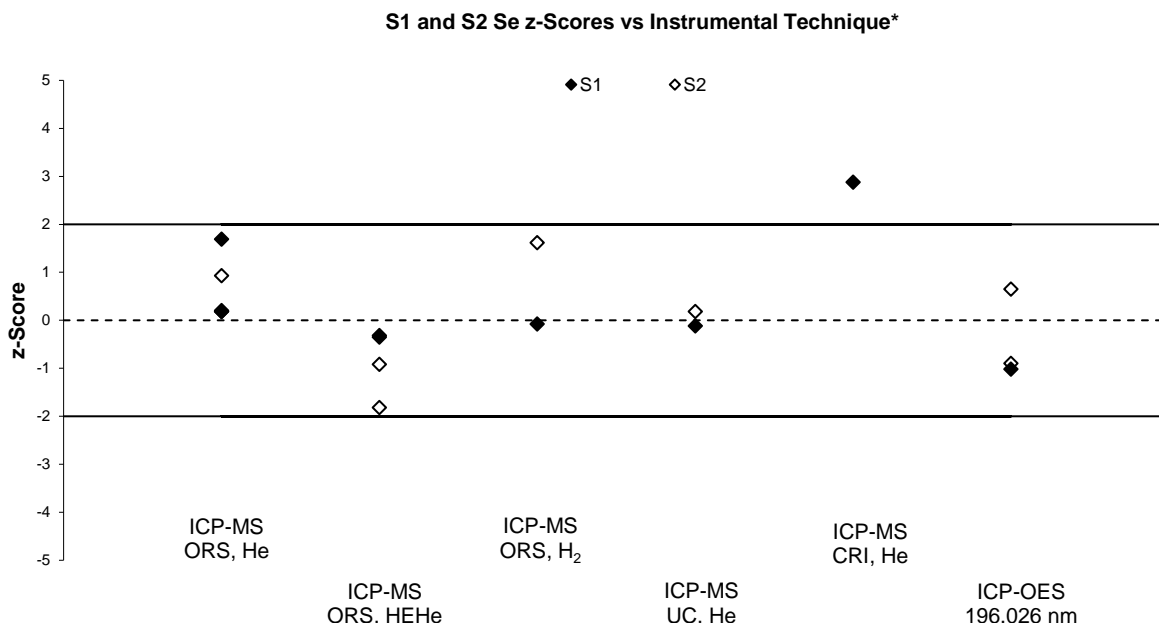
Mercury Participants used a wide variety of analytical techniques for Hg measurements, ICP-MS in collision mode and CVAAS were the preferred techniques (Figure 72).



* * z-scores of Laboratories 8, 16, 17, 19 and 21 for Se in S2 were excluded.

Figure 72 Hg Performance in S1 and S2 vs. Instrumental Technique

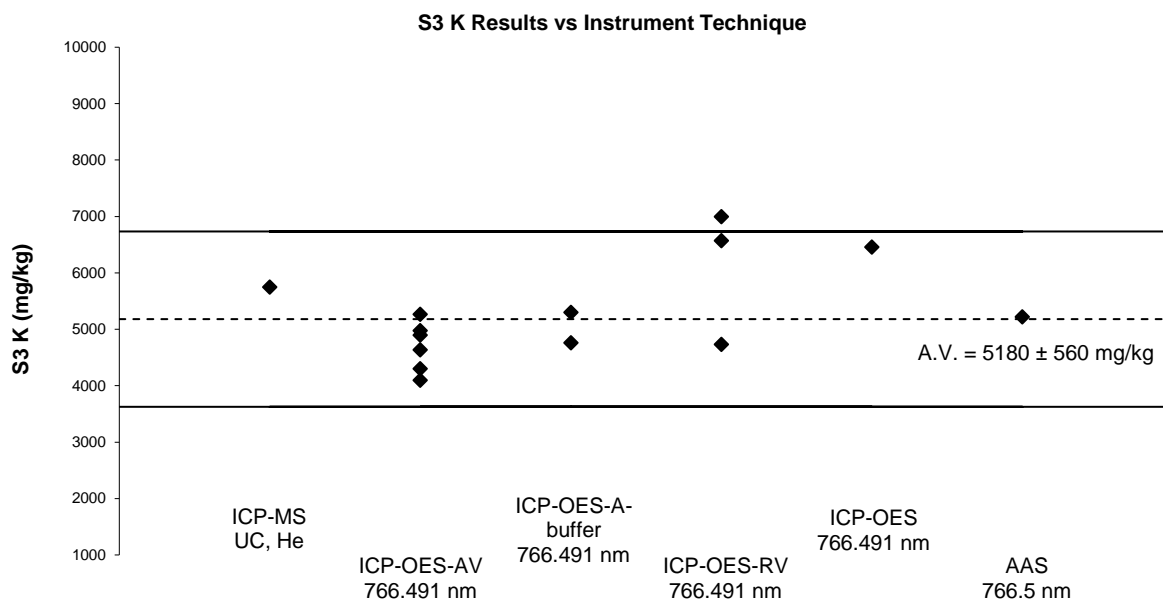
Selenium challenged participants' analytical techniques; the between-laboratory CV was high for both samples. The unsatisfactory results were higher than the assigned value which is an indication of unsolved interferences (Table 74 and Figure 73).



* z-scores of Laboratories 8, 16, 17, 19 and 21 for Se in S2 were excluded.

Figure 73 Se Performance in S1 and S2 vs. Instrumental Technique

Potassium Plots of participants' results for K versus instrumental technique used are presented in Figure 74. ICP-OES with axial view plasma (ICP-OES-AV) and wavelength 766.491 nm was the preferred instrumental technique.



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

Figure 74 K Results vs. Instrumental Technique

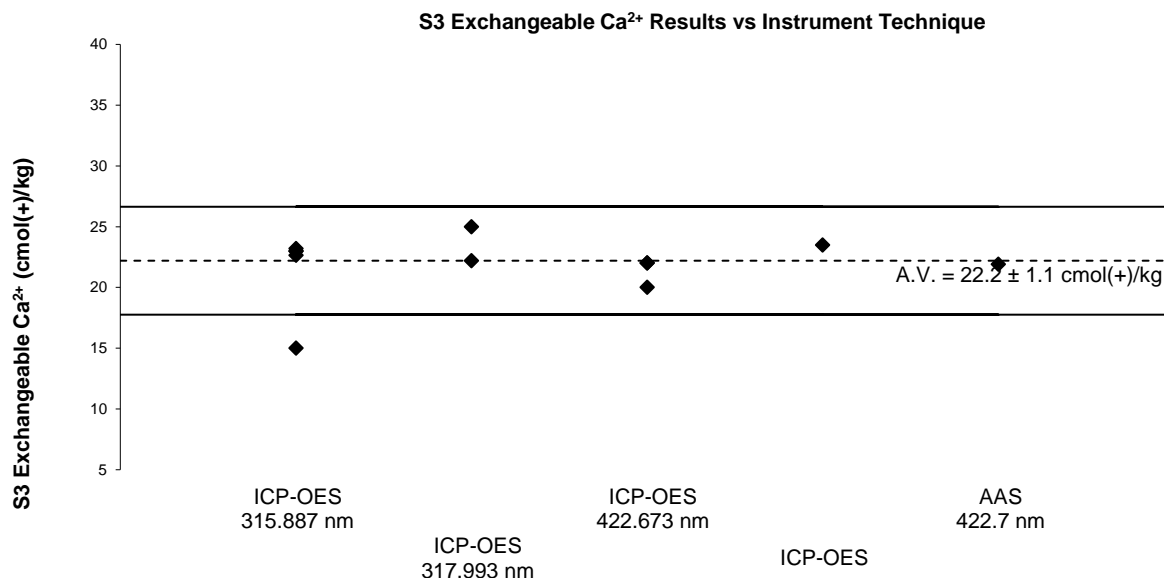
Rubidium Only 5 results were reported for Rb in S1, however the reported results were in relatively good agreement with each other centred on a value of 6.44 mg/kg value.

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

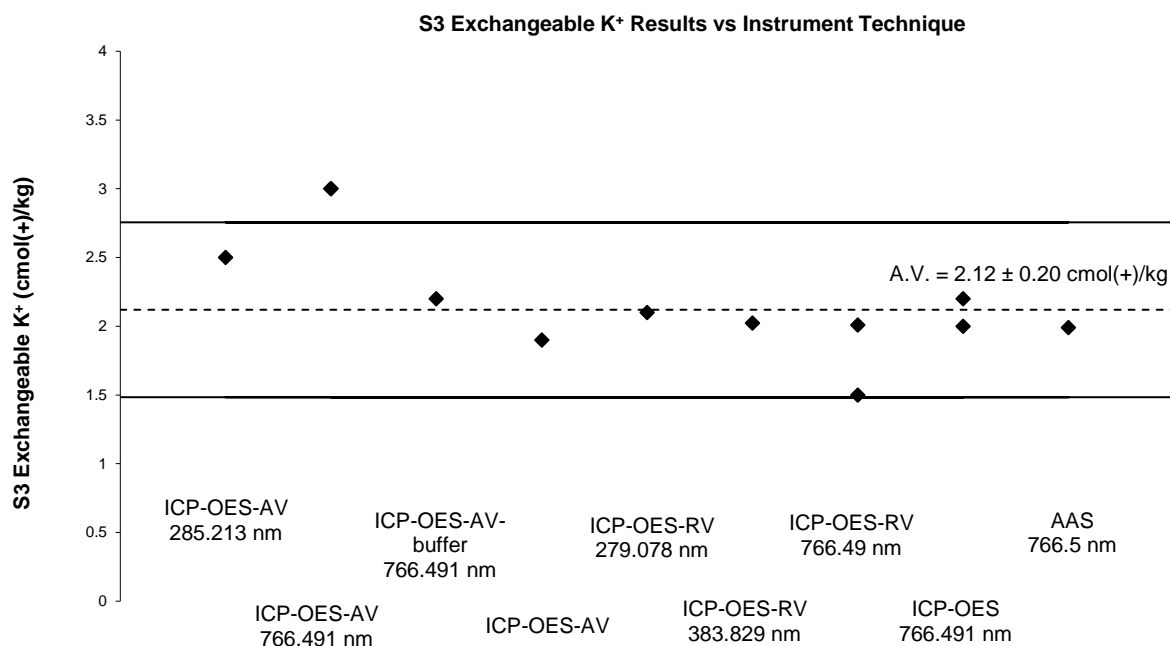
Method 15A1 as defined by Rayment, G.E. and David, J. L in “Soil Chemical Methods- Australasia”.²¹

The methods 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 to 2 min. Laboratory 8 used a ratio of 1:5 for sample mass/extraction solution and Laboratory 13 used a ratio of 1:10. No significant differences were noticed between the results reported by these participants and the results reported by participants who used a ration of 1:5. Plots of participants’ results versus the analytical methods used for the exchangeable bases measurement are presented in Figures 75 to 78.



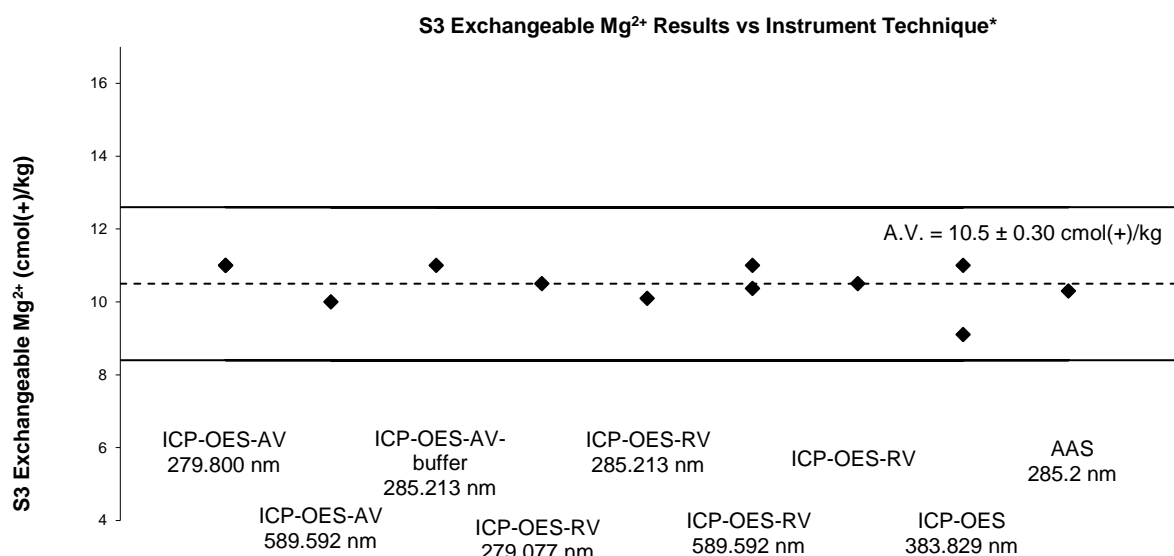
Horizontal lines on charts are the results corresponding to z-scores of 2 and -2

Figure 75 Exchangeable Ca²⁺ Results vs. Analytical Methods



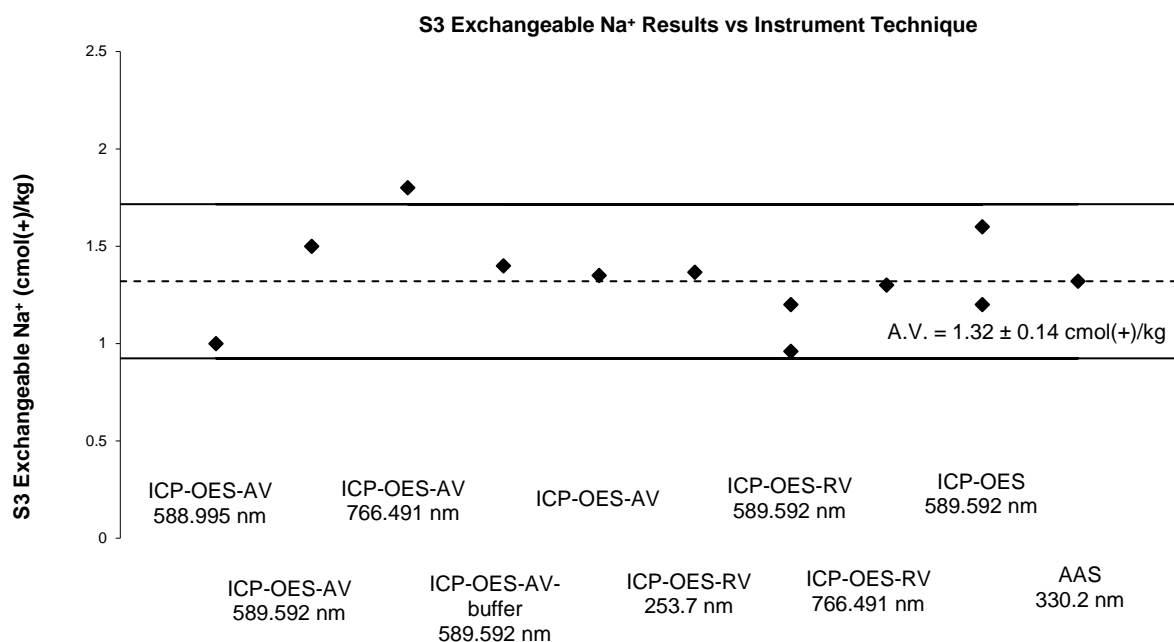
Horizontal lines on charts are the results corresponding to z-scores of 2 and -2

Figure 76 Exchangeable K⁺ Results vs. Analytical Methods



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

Figure 77 Exchangeable Mg²⁺ Results vs. Analytical Methods



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

Figure 78 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".²¹ All participants shook the sample for 16 hours and used a ratio of 1 :100 sample mass/extraction solution (Table 5).

Colwell K Five participants extracted K in S3 using 0.5 M NaHCO₃ and reported results for this test. Two laboratories used ICP-OES to measure Colwell K and one used ICP-MS. Four results were in a relative agreement with each other, (Figure 79).

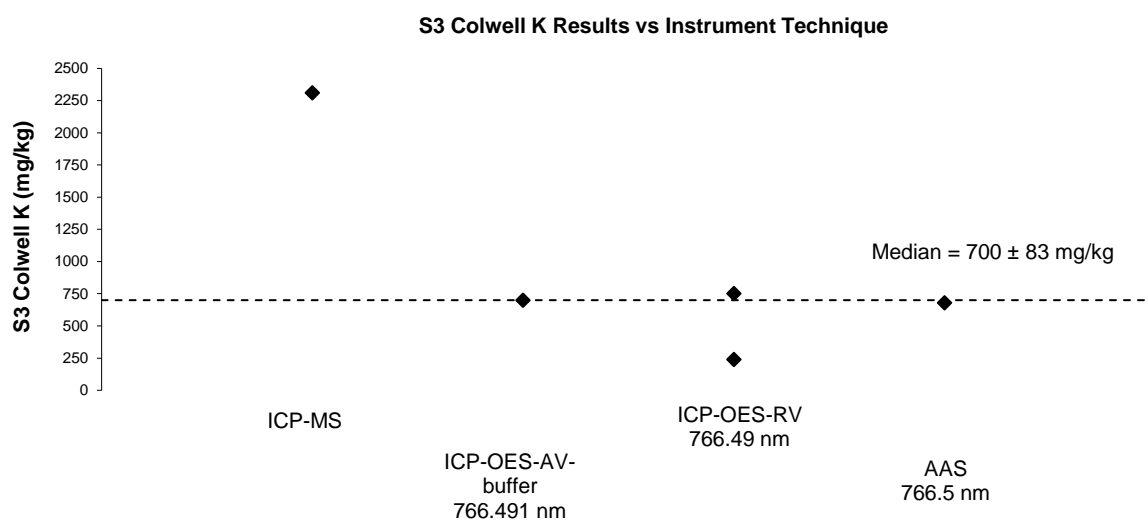
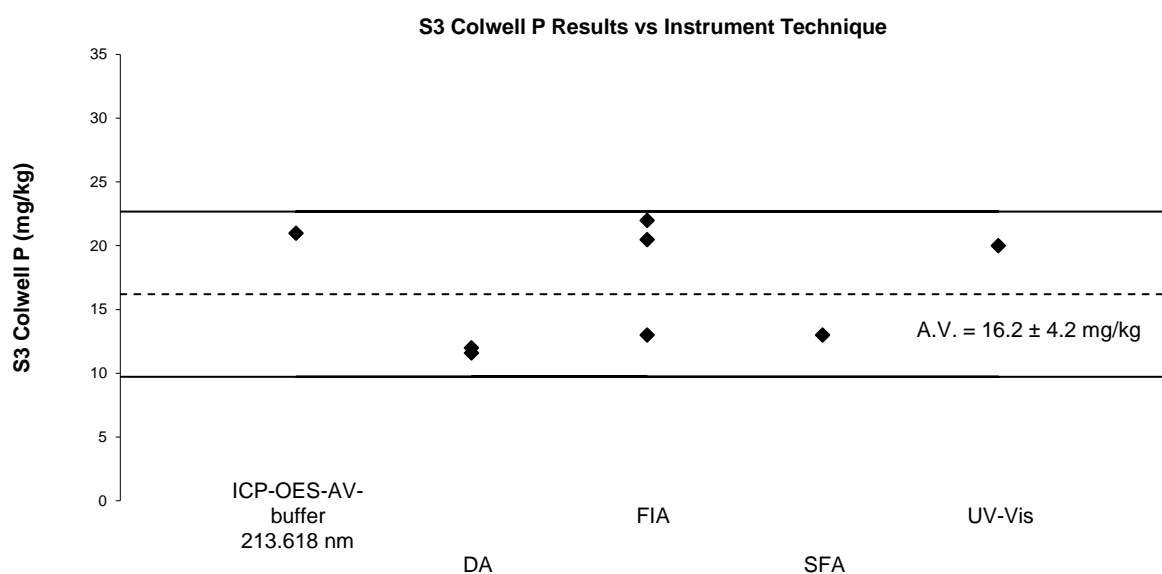


Figure 79 Colwell K Results vs. Instrumental Technique

Colwell P Eight results were reported for Colwell P in S3 and all were compatible with each other and with the assigned value of 16.2 mg/kg. Plots of participants' results versus the instrumental technique used are presented in Figure 80.



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

Figure 80 Colwell P Results vs. Instrumental Technique

6.8 Participants' Results and Analytical Methods for Phosphorus Buffer Index-PBI_{CoIP}

P Buffer Index-PBI_{CoIP} gives an indication of soil ability to fix P and make it unavailable to plant uptake. Four laboratories reported results for this test. The results were in relatively good agreement with each other (CV16%), centred on the value of 173 mg/kg (Figure 81).

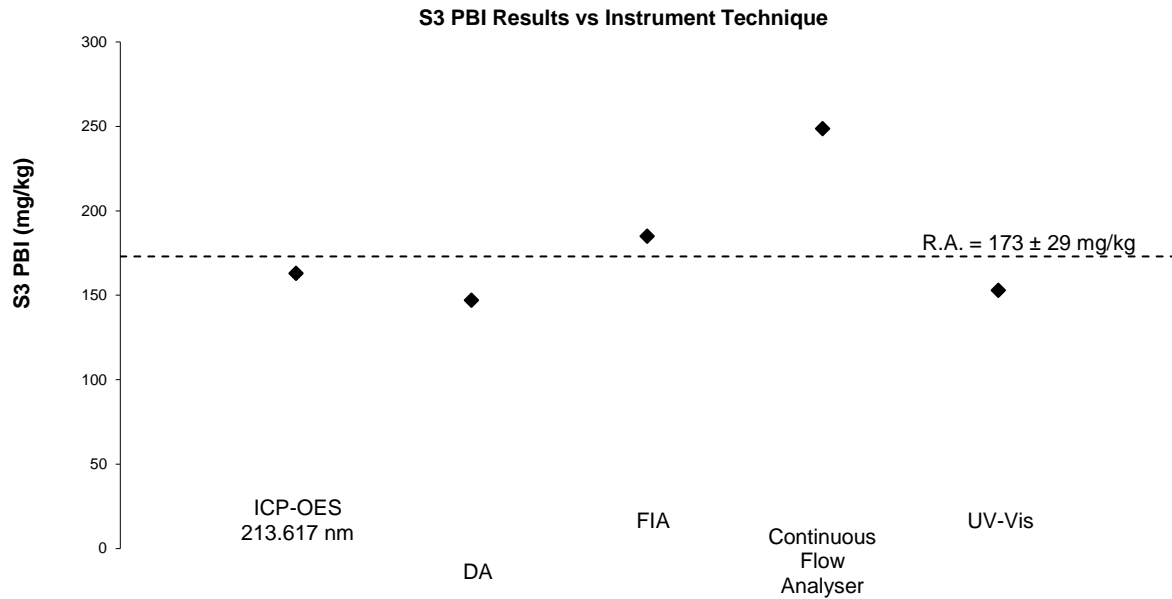
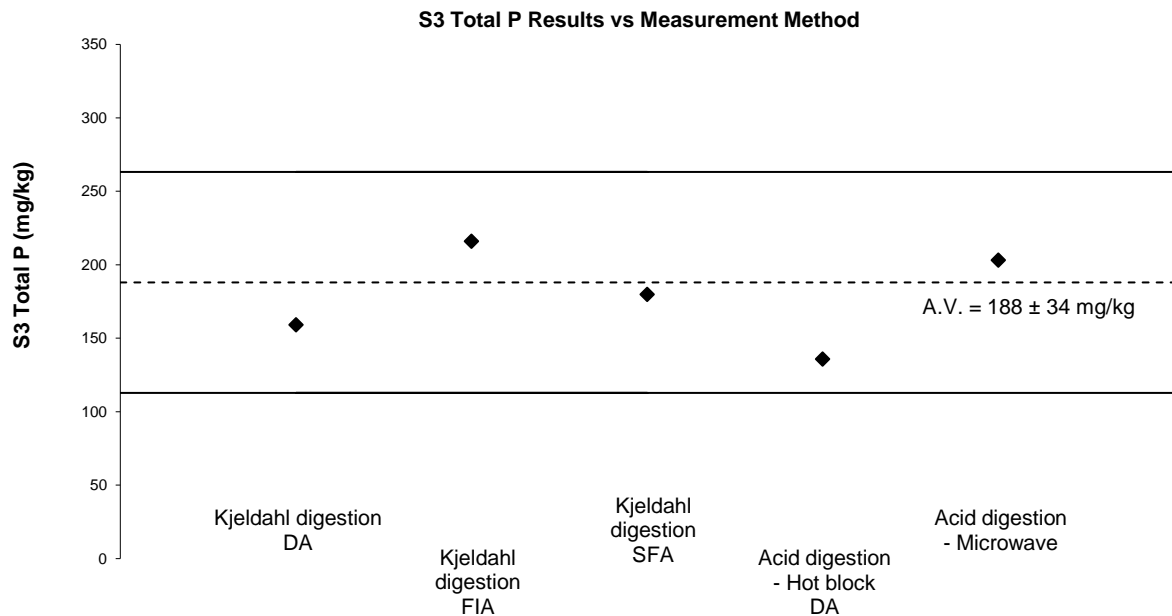


Figure 81 PBI Results vs. Instrumental Technique

6.9 Participants' Results and Analytical Methods for Total P

Total P assigned value was 188 mg/kg. Seven participants reported results for total P, and all performed satisfactorily (Figure 82).

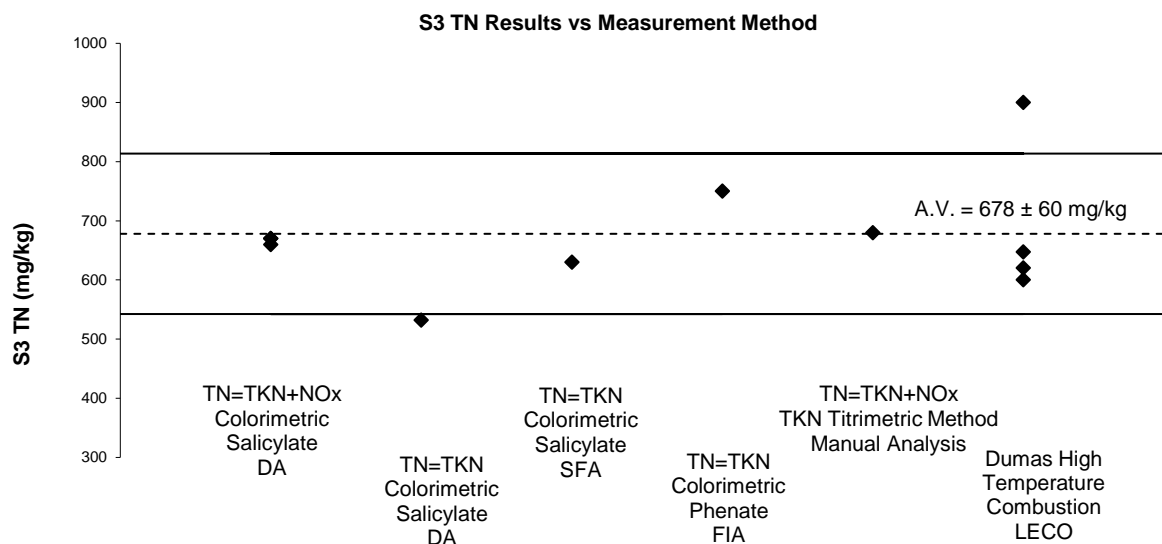


Horizontal lines on charts are the results corresponding to z-scores of 2 and -2

Figure 82 TP Results vs. Analytical Method

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 83.



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

Figure 83 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 All reported results for TC in S3 returned satisfactory z-scores. For TC measurement, Laboratory 16 used a colorimetric method based on the Rayment & Higginson Method 6B1 for TOC measurements in S3 but did not report a result for it.²⁰

Total Organic Carbon 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.²¹⁻²⁴

Laboratory 16 correctly measured TOC in S3 but reported results in the wrong units.

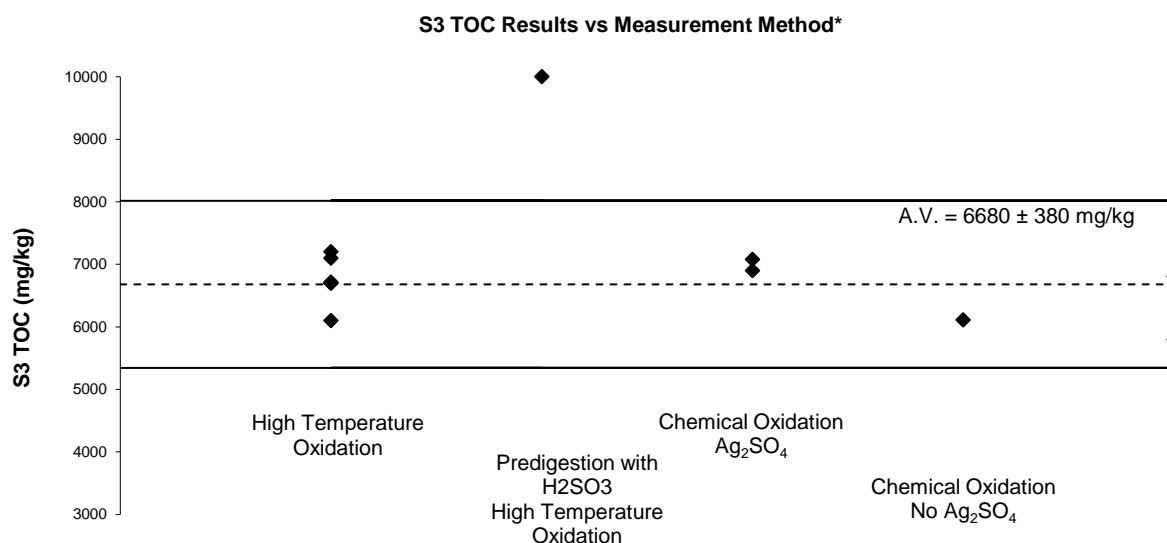
Laboratory 23 digested the sample with sulfurous acid prior LECO determination.

Two laboratories reported adding silver sulfate to remove chloride interference and one used mercuric (II) sulfate to remove chloride interference.

Seven participants used a high temperature oxidation method and five used a chemical oxidation method based on the "Walkley-Black" method (Figure 84).

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

reported: “Sample was Fizz test with 4 M HCl and no Fizzing observed. Therefore no acid treatment was carried for TOC”.



* The result reported by Laboratory 16 as 0.68 mg/kg has been plotted as 68000 mg/kg. The results reported by Laboratory 23 as 15000 mg/kg has been plotted as 10000 mg/kg. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2

Figure 84 TOC Result vs. Analytical Method

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}

6.12 Comparison with Previous NMI Proficiency Tests of Metals in Soil

AQA 23-02 is the 32nd NMI proficiency study of inorganic analytes in soil.

Participants' performance in measurement of metals in soil over the last ten years is presented in 5. 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 77).

Table 77 Control Samples Used by Participants

Lab. Code	Description of Control Samples
1	CRM – AGAL 10 & AGAL 12
2	RM – ASPAC 7098 ASPAC 7118
3	SS
6	AQA, Inhouse
7	CRM – QCS-01-05 ICP Quality Control Standard #1; High Purity Standards CCV-1 Solution A; High Purity Standards CCV-1 Solution B; NMI AGAL-12 Biosoil; Australian Chemical Reagents Multi Element Standard; Australian Chemical Reagents Mixed Anion Standard; ERA Mercury WasteWatR
8	CRM – AGAL12, LOAM B
9	RM – Previous PT program
10	SS
11	CRM – PACS3
13	SS
14	CRM – CRM036
15	CRM
16	SS
18	SS
19	SS
20	SS
21	CRM – Agal-12 Biosoil
23	RM – AGAL 12, In House AG reference
24	CRM – 10589-QC-PR9660

Matrix matched control samples taken through all steps of the analytical process, are the most valuable quality control tools for assessing a methods' performance. Some laboratories reported using certified reference materials. These materials may not meet the internationally recognised definition of a Certified Reference Material:

*' a reference material, accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceabilities, using valid procedures'*²⁶

Surplus test samples from this study are available from NMI.

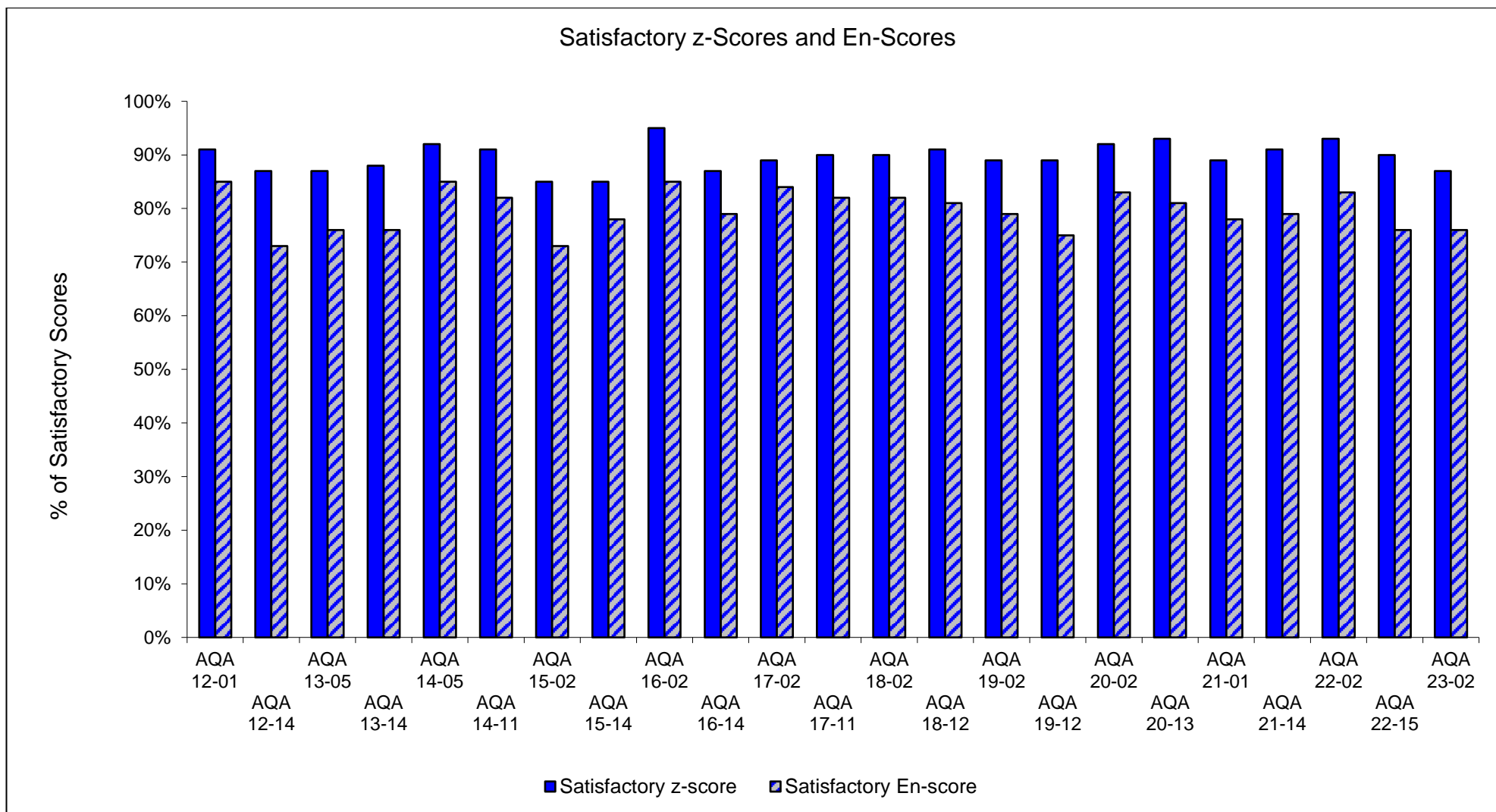


Figure 85 Participants' Performance over Time (2012-2023)

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APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING

Sample Preparation

Sample S1 was a soil material fortified for 15 elements, dried, ground and passed through a 355 µm sieve prior to being divided into portions of approximately 30 g each.

Sample S2 was soil fortified with 16 elements. After drying, the spiked soil was ground and passed through a 355 µm sieve then accurately weighed in portions of 54 g in each jar. 43 g of water were accurately weighed and further added to each jar. Each jar was sealed and tumbled for 30 min.

Sample S3 was an unfortified, dried, agricultural soil material. It was ground and sieved through a 355 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.

No homogeneity testing was carried out for S1 B, Li, Rb, Sb and V, S2 Al and Ba, and all tests in sample S3 that were not acid extractable except for S, TC, TN and TOC.

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 soil samples and 1.2 g for the sludge 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 conditions used, and the ion/wavelength monitored for each analyte is given in Table 78.

Table 78 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
Ag	ICP-MS	Rh	ORS	He	NA	800	107 m/z
As	ICP-MS	Rh	ORS	He	800	800	75 m/z
B	ICP-MS	Rh	NA	NA	800	NA	11 m/z
Be	ICP-MS	Rh	NA	NA	800	NA	9 m/z
Bi	ICP-MS	Ir	ORS	He	NA	800	209 m/z
Ca	ICP-MS	Rh	ORS	He	800	NA	43 m/z
Cd	ICP-MS	Rh	ORS	He	800	800	111 m/z
Co	ICP-MS	Rh	ORS	He	NA	800	59 m/z

Cr	ICP-MS	Rh	ORS	He	800	NA	52 m/z
Cs	ICP-MS	Rh	ORS	He	NA	800	133 m/z
Cu	ICP-MS	Rh	ORS	He	800	800	63 m/z
Fe	ICP-MS	Rh	ORS	He	800	NA	56 m/z
Hg	ICP-MS	Rh	ORS	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
Mg	ICP-MS	Rh	ORS	He	800	NA	24 m/z
Mn	ICP-MS	Rh	ORS	He	800	NA	55 m/z
Mo	ICP-MS	Rh	ORS	He	800	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	ORS	He	800	800	Average of 206, 207, 208 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	ORS	He	800	NA	118 m/z
Sr	ICP-MS	Rh	ORS	He	NA	800	88 m/z
Th	ICP-MS	Rh	ORS	He	NA	800	232 m/z
Tl	ICP-MS	Rh	ORS	He	800	NA	205 m/z
U	ICP-MS	Ir	ORS	He	NA	800	238 m/z
Zn	ICP-MS	Rh	ORS	He	800	800	64 m/z

Sample Analysis for Total Carbon and Total Organic Carbon

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

Sample Analysis for Total Nitrogen

Total Nitrogen in Sample S3 was measured as the sum of TKN +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 oC ± 5 oC 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 79.

Table 79 Uncertainty of Assigned Value for As in Sample S1

No. results (p)	15
Robust Average	4.12 mg/kg
$S_{rob\ av}$	0.24
$u_{rob\ av}$	0.08 mg/kg
k	2
$U_{rob\ av}$	0.16 mg/kg

The assigned value for As in Sample S1 is **4.12 ± 0.16 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 16).

A worked example is set out below in Table 80.

Table 80 z-Score and E_n-score for As result reported by Laboratory 1 in S1

As Result mg/kg	Assigned Value mg/kg	Set Target Standard Deviation	z-Score	E _n -Score
4.2±0.84	4.12±0.16	15% as PCV or 0.15x4.12= = 0.62 mg/kg	$z = \frac{(4.2 - 4.12)}{0.62}$ z = 0.13	$E_n = \frac{(4.2 - 4.12)}{\sqrt{0.84^2 + 0.16^2}}$ E _n = 0.09

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 2023 NMI carried out 28 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 (Tables 81 and 82).

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 9.3% respectively. Using a coverage factor of two gives relative expanded uncertainties of 24% and 19% respectively, at a level of confidence of approximately 95%.

Table 81 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
AQA 22-02	S1 – Sediment	3.57	4.02	9.5	15
AQA 22-02	S2 – Moist Soil	3.57	3.56	6.2	13
AQA 22-15	S2 – Clay	4.29	3.63	17	19
AQA 23-02	S1 – Soil	4.41	4.12	5.9	16
AQA 23-02	S2 - Sludge	4.41	4.8	24	8
Average				12*	

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

Table 82 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
AQA 21-14	S1 - Sediment	19.5	20.9	8.9	21
AQA 22-15	S1 -Sediment	58.6	56.8	7.8	22
Average				9.3**	

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

Table 83 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 83 Uncertainty of As Results Estimated Using PT Data.

Results mg/kg	Uncertainty mg/kg
1.00	0.24
5.0	1.2
20.0	3.8
75	14
100	19

The estimates of 24% and 19% relative passes the test of being reasonable, and the analysis of the 36 different PT samples over fourteen years can be assumed to include all the relevant uncertainty components (different matrices, operators, reagents, calibrators etc.), and so complies with ISO 17025.

APPENDIX 4 - ACRONYMS AND ABBREVIATIONS

AAS	Atomic Absorption Spectroscopy
APHA	American Public Health Association
A.V.	Assigned Value
CITAC	Cooperation on International Traceability in Analytical Chemistry
CRI	Collision Reaction Interface
CRM	Certified Reference Material
CV	Coefficient of Variation
CV _{rob}	Robust Coefficient of Variation
DA	Discreet Analyser
FIA	Flow Injection Analyser
GUM	Guide to the Expression of Uncertainty in Measurement
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
ISO/IEC	International Organisation for Standardisation / International Electrotechnical Commission
KED	Kinetic Energy Discrimination
Max	Maximum value in a set of results
Md	Median
Min	Minimum value in a set of results
MU	Measurement Uncertainty
M.V.	Median Value
N	Number of Participants
NATA	National Association of Testing Authorities
NMI	National Measurement Institute (of Australia)
NR	Not Reported
NT	Not Tested
ORS	Octopole Reaction System
PCV	Performance Coefficient of Variation
PFAS	Polyfluoroalkyl Substances
PT	Proficiency Test
R.A.	Robust Average
RM	Reference Material
CV _{rob}	Robust Coefficient of Variation
SD _{rob}	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
USEPA	United States Environmental Protection Agency
UV-Vis	Ultraviolet and Visible Spectroscopy

APPENDIX 5 - INSTRUMENT DETAILS

Table 84 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	Rh	ORS	He	800	NA	107
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	328.289
5	ICP-MS/MS	In	CRI	He	NA		107
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS	Rh	ORS	He	NA	200	103
8	ICP-OES-AV				NA		
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-MS	72 Ge/103 Rh/193 Ir	ORS	He	NA	100	107
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV				NA		328.068
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	NA	500	107 m/z
14	ICP-OES-AV-buffer	Y			NA	100	328.068
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			NA	83	328.068
17	ICP-MS	Y 89	KED		NA	2000	107
18	NA	NA	NA	NA	NA	NA	NA
19	ICP-MS	Rh	CRI	He	NA	500	107
20	ICP-OES-AV	Lu	NA	NA	NA	25	328.289
21					NA	100	
22	ICP-OES-AV	Lutetium	NA	NA	NA	50-1000	328.068nm
23	ICP-MS	Rh	NA	NA	NA	625	109
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Rhodium	ORS	standard mode			

Table 85 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	Y	NA	NA	800	NA	396.152
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	236.705
5	ICP-MS	In	CRI	He	NA		27
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	ORS	He	NA	200	45
8	ICP-OES-RV				NA		
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-OES-AV	214.282 Te/371.02 9 Y	NA	NA	NA	500	396.152
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV				NA		308.215
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	NA	500	27 m/z
14	ICP-OES-AV- buffer	Y			NA	100	394.401
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			NA	83	396.152
17	ICP-MS	Sc-2 45	KED		NA	2000	27
18	NA	NA	NA	NA	NA	NA	NA
19	ICP-MS	Sc	CRI	He	NA	500	27
20	ICP-OES-AV	Lu	NA	NA	NA	2500	236.705
21					NA	100	
22	ICP-OES-AV	Lutetium	NA	NA	NA	50	236.705nm
23	ICP-MS	Sc	UC	He	NA	625	27
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Scandium	ORS	standard mode			

Table 86 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	Rh	ORS	He	800	NA	75
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	188.98
5	ICP-MS	In	CRI	He			75
6	ICP-MS	72	ORS	He	100	NA	75
7	ICP-MS	Rh	ORS	He	200	200	103
8	ICP-OES-AV						
9	ICP-OES-AV					NA	
10	ICP-MS	72 Ge/103 Rh/193 Ir	ORS	He	200	100	75
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV						188.98
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	500	75 m/z
14	ICP-OES-AV-buffer	Y			100	100	188.979
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	83	188.98
17	ICP-MS	Ge-1 72	KED		100	2000	75
18	ICP-OES-AV	NA	NA	NA	49.6	NA	188.98
19	ICP-MS	Rh	CRI	He	500	500	75
20	ICP-OES-AV	Lu	NA	NA	20	25	188.98
21	ICP-MS	Rh	KED	He	50	50	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	50-1000	188.98nm
23	ICP-MS	Rh	UC	He	625	625	75
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Telerium	ORS	HEHe			

Table 87 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-MS	Rh	ORS	He	800	NA	
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	182.577
5	ICP-OES-RV	Y				NA	249.678
6						NA	
7	ICP-MS	Sc	ORS	He	200	NA	45
8	ICP-OES-AV					NA	
9						NA	
10	ICP-MS	⁷² Ge/ ¹⁰³ Rh/ ¹⁹³ Ir	ORS	He	200	NA	11
11	NA	NA	NA	NA	NA	NA	NA
12						NA	
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	NA	11 m/z
14	ICP-OES-AV-buffer	Y			100	NA	208.957
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	NA	182.577
17	ICP-MS	Y 89	KED		100	NA	11
18	ICP-OES-AV	NA	NA	NA	496	NA	249.772
19	ICP-MS	Sc	CRI	NA	500	NA	11
20	ICP-OES-AV	Lu	NA	NA	20	NA	182.577
21	ICP-MS	Sc	KED	He	50	NA	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	NA	208.956nm
23	ICP-MS	Sc	NA	NA	625	NA	10
24	NA	NA	NA	NA	NA	NA	NA
25	NA	NA	NA	NA	NA	NA	NA

Table 88 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-MS	Rh	ORS	He	800	NA	134Mini
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	230.424
5	ICP-MS	In	CRI	He	NA		137
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS	Rh	ORS	He	NA	200	103
8	ICP-OES-AV				NA		
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-MS	72 Ge/103 Rh/193 Ir	ORS	He	NA	500	137
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV				NA		455.403
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	NA	500	137 m/z
14	ICP-OES-AV-buffer	Y			NA	100	233.527
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			NA	83	493.408
17	ICP-MS	In-1 115	KED		100	2000	138
18	NA	NA	NA	NA	NA	NA	NA
19	ICP-MS	Rh	CRI	He	NA	500	135
20	ICP-OES-AV	Lu	NA	NA	NA	25	230.424
21					NA	100	
22	ICP-OES-AV	Lutetium	NA	NA	NA	50-1000	230.424nm
23	ICP-MS	Rh	NA	NA	NA	625	138
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Rhodium	ORS	standard mode			

Table 89 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	Rh	ORS	He	800	NA	9
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	313.107
5	ICP-OES-AV	Y				NA	313.042
6	ICP-OES-AV-buffer	Lu				NA	313.107
7	ICP-MS	Sc	ORS	No Gas	200	NA	45
8						NA	
9						NA	
10	ICP-MS	72 Ge/103 Rh/193 Ir	ORS	He	200	NA	9
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV					NA	
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	NA	9 m/z
14	ICP-OES-AV-buffer	Y			100	NA	313.107
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	NA	313.042
17	ICP-MS	Sc-2 45	KED		10	NA	9
18	ICP-OES-AV	NA	NA	NA	49.6	NA	234.861
19	ICP-MS	Sc	CRI	NA	500	NA	9
20	ICP-OES-AV	Lu	NA	NA	20	NA	313.107
21	ICP-MS	Sc	NA	He	100	NA	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	NA	313.042nm
23	ICP-MS	Sc	NA	NA	625	NA	9
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Scandium	ORS	standard mode			

Table 90 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							
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	315.887
5					NA		
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS	Lu	ORS	He	NA	200	175
8					NA		
9	NA	NA	NA	NA	NA	NA	NA
10	NA	NA	NA	NA	NA	NA	NA
11	NA	NA	NA	NA	NA	NA	NA
12					NA		
13					NA		
14	NT				NA		
15	NA	NA	NA	NA	NA	NA	NA
16	NA	Lu			NA	NT	NT
17	NT	NA			NA		NA
18	NA	NA	NA	NA	NA	NA	NA
19	ICP-MS	Lu	CRI	He	NA	500	209
20	ICP-MS	Lu	ORS	standard mode	200	NA	209
21	ICP-MS	Tb	NA	He	NA	100	
22	ICP-MS	Lutetium	ORS	No Gas	NA	1000	209m/z
23	ICP-MS	Ir	NA	NA	NA	625	209
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Iridium	ORS	standard mode			

Table 91 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	Y	NA	NA	NA	800	422.673
2						NA	
3	ICP-OES-AV	Lu	NA	NA	50	50	228.802
5	ICP-OES-RV	Y				NA	317.933
6	ICP-OES-AV-buffer	Lu			100	NA	430.253
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-OES-RV					NA	
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-OES-AV	214.282 Te/371.02 9 Y	NA	NA	200	NA	315.887
11						NA	
12	ICP-OES-RV					NA	318.887
13	ICP-OES	Cs, Y			500	NA	315.887 nm
14	ICP-OES-AV-buffer	Y			100	NA	315.887
15						NA	
16	ICP-OES-AV	Lu			83	NA	317.933
17	NA	NA	NA	NA	NA	NA	NA
18	AAS	NA	NA	NA	444.6	NA	422.7
19	NA	NA	NA	NA	NA	NA	NA
20	ICP-OES-AV	Lu	NA	NA	400	NA	315.887
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Lutetium	NA	NA	50	NA	315.887nm
23	ICP-MS	Sc	UC	He	625	NA	44
24	ICP-OES-RV	Yttrium	NA	NA	100	NA	315.887
25	NA	NA	NA	NA	NA	NA	NA

Table 92 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	ORS	He	800	NA	111
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	228.615
5	ICP-MS	In	CRI	He			111
6	ICP-MS	103	ORS	He	100	NA	114
7	ICP-MS	Rh	ORS	He	200	200	103
8	ICP-OES-AV						
9	ICP-OES-AV					NA	
10	ICP-MS	72 Ge/103 Rh/193 Ir	ORS	He	200	10	111
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV						214.439
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	500	111 m/z
14	ICP-OES-AV-buffer	Y			100	100	228.802
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	83	214.439
17	ICP-MS	In-1 115	KED		100	2000	111
18	ICP-OES-AV	NA	NA	NA	496	NA	226.502
19	ICP-MS	Rh	CRI	He	500	500	111
20	ICP-OES-AV	Lu	NA	NA	20	25	228.802
21	ICP-MS	Rh	KED	He	50	50	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	50-1000	214.439nm
23	ICP-MS	Rh	NA	NA	625	625	111
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Rhodium	ORS	He			

Table 93 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-MS	Rh	ORS	He	800	NA	59
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	205.56
5	ICP-MS	In	CRI	He	NA		59
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	ORS	He	NA	200	45
8	ICP-OES-AV				NA		
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-MS	72 Ge/103 Rh/193 Ir	ORS	He	NA	100	59
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV				NA		228.615
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	NA	500	59 m/z
14	ICP-OES-AV-buffer	Y			NA	100	228.616
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			NA	83	230.786
17	ICP-MS	Sc 45	KED		NA	2000	59
18	NA	NA	NA	NA	NA	NA	NA
19	ICP-MS	Sc	CRI	He	NA	500	59
20	ICP-OES-AV	Lu	NA	NA	NA	25	228.615
21					NA	100	
22	ICP-OES-AV	Lutetium	NA	NA	NA	50-1000	231.160nm
23	ICP-MS	Ge	UC	He	NA	625	59
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Rhodium	ORS	He			

Table 94 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-MS	Rh	ORS	He	800	NA	52
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	324.754
5	ICP-MS	In	CRI	He		NA	52
6	ICP-MS	103	ORS	He	100	NA	52
7	ICP-MS	Sc	ORS	He	200	NA	45
8	ICP-OES-AV					NA	
9	ICP-OES-AV					NA	
10	ICP-OES-AV	214.282 Te/371.02 9 Y	NA	NA	200	NA	267.716
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV					NA	367.716
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	NA	52 m/z
14	ICP-OES-AV- buffer	Y			100	NA	205.56
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	NA	267.716
17	ICP-MS	Sc 45	KED		100	2000	52
18	ICP-OES-AV	NA	NA	NA	496	NA	283.563
19	ICP-MS	Sc	CRI	He	500	NA	52
20	ICP-OES-AV	Lu	NA	NA	20	NA	205.56
21	ICP-MS	Sc	KED	He	50	NA	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	NA	205.560nm
23	ICP-MS	Sc	UC	He	625	NA	52
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Rhodium	ORS	He			

Table 95 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							
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-MS	Ge	ORS	standard mode	50	50	
5					NA		
6	NA	NA	NA	NA	NA	NA	NA
7	NA	NA	NA	NA	NA	NA	NA
8					NA		
9	NA	NA	NA	NA	NA	NA	NA
10	N/A	NA	NA	NA	NA	NA	NA
11	NA	NA	NA	NA	NA	NA	NA
12					NA		
13					NA		
14	NT				NA		
15	NA	NA	NA	NA	NA	NA	NA
16	NA	Lu			NA	NT	NT
17	NT	NA			NA		NA
18	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20	ICP-MS/MS	Ge	ORS	standard mode	20	20	133
21					NA	100	
22	ICP-MS		ORS	No Gas	NA	1000	133m/z
23					NA		
24	NA	NA	NA	NA	NA	NA	NA
25	NA	NA	NA	NA	NA	NA	NA

Table 96 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-MS	Rh	ORS	He	800	NA	63Mini
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	324.754
5	ICP-MS	In	CRI	He			63
6	ICP-MS	103	ORS	He	100	NA	65
7	ICP-MS	Sc	ORS	He	200	200	45
8	ICP-OES-AV						
9	ICP-OES-AV					NA	
10	ICP-OES-AV	214.282 Te/371.02 9 Y	NA	NA	200	10	217.895
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV						324.754
13	ICP-OES	Cs, Y			500	500	327.395 nm
14	ICP-OES-AV- buffer	Y			100	100	324.752
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	83	327.395
17	ICP-MS	Sc 45	KED		100	2000	63
18	ICP-OES-AV	NA	NA	NA	496	NA	324.754
19	ICP-MS	Sc	CRI	He	500	500	63
20	ICP-OES-AV	Lu	NA	NA	20	25	324.754
21	ICP-MS	Ga	KED	He	50	50	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	50-1000	324.754nm
23	ICP-MS	Ge	UC	He	625	625	63
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Rhodium	ORS	He			

Table 97 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	Y	NA	NA	NA	800	238.204
2						NA	
3	CVAAS	NA	NA	NA	50	50	253.7
5	ICP-OES-RV	Y				NA	261.187
6	ICP-OES-AV-buffer	Lu			500	NA	239.563
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-OES-RV					NA	
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-OES-AV	214.282 Te/371.02 9 Y	NA	NA	2000	NA	240.489
11						NA	
12	ICP-OES-RV					NA	234.35
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	NA	57 m/z
14	ICP-OES-AV-buffer	Y			100	NA	273.955
15						NA	
16	ICP-OES-AV	Lu			83	NA	238.204
17	NA	NA	NA	NA	NA	NA	NA
18	ICP-OES-AV	NA	NA	NA	44460	NA	238.204
19	NA	NA	NA	NA	NA	NA	NA
20	ICP-OES-AV	Lu	NA	NA	2000	NA	261.382
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Lutetium	NA	NA	50	NA	234.350nm
23	ICP-MS	Sc	UC	He	625	NA	56
24	ICP-OES-AV	Yttrium	NA	NA	100	NA	259.939
25	NA	NA	NA	NA	NA	NA	NA

Table 98 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	ORS	He	800	NA	202
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	766.491
5	ICP-MS	Tb	CRI				202
6	VGA-ICP-OES				100	NA	194.164
7	ICP-MS	Lu	ORS	He	200	200	175
8	CVAAS						
9	CVAAS					NA	
10	ICP-MS	72 Ge/103 Rh/193 Ir	ORS	He	200	100	202
11	NA	NA	NA	NA	NA	NA	NA
12	if other please type						
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	500	201 m/z
14	CVAAS				100		
15	NA	NA	NA	NA	NA	NA	NA
16	CVAAS				83	83	253.7
17	ICP-MS	Ir 193	KED		100	2000	202
18	Cold Vapour FIMS	NA	NA	NA	198.4	NA	253.7
19	ICP-MS	Lu	CRI	He	500	500	202
20	CVAAS	NA	NA	NA	200	250	253.7
21	ICP-MS	Tb	KED	He	50	50	
22	CVAAS	NA	NA	NA	500	500	253.7nm
23	ICP-MS	Ir	NA	NA	625	625	201
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Iridium	ORS	He			

Table 99 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	Y	NA	NA	NA	800	766.491
2						NA	
3	ICP-OES-AV	Lu	NA	NA	50	50	766.491
5	ICP-OES-AV	Y				NA	766.491
6	ICP-OES-AV-buffer	Lu				NA	766.491
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-OES-RV					NA	
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-OES-AV	214.282 Te/371.02 9 Y	NA	NA	200	NA	766.491
11						NA	
12	ICP-OES-RV					NA	766.491
13	ICP-OES	Cs, Y			500	NA	766.491 nm
14	ICP-OES-AV-buffer	Y			100	NA	766.49
15						NA	
16	ICP-OES-AV	Lu			83	NA	769.897
17	NA	NA	NA	NA	NA	NA	NA
18	AAS	NA	NA	NA	2223	NA	766.5
19	NA	NA	NA	NA	NA	NA	NA
20	ICP-OES-AV	Lu	NA	NA	20	NA	766.491
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Caesium	NA	NA	50	NA	766.491nm
23	ICP-MS	Sc	UC	He	625	NA	39
24	ICP-OES-RV	Yttrium	NA	NA	100	NA	766.49
25	NA	NA	NA	NA	NA	NA	NA

Table 100 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	Rh	ORS	He	800	NA	139
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-MS	Lu	ORS	standard mode	50	50	139
5					NA		
6	NA	NA	NA	NA	NA	NA	NA
7	NA	NA	NA	NA	NA	NA	NA
8					NA		
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-MS	⁷² Ge/ ¹⁰³ Rh/ ¹⁹³ Ir	ORS	He	NA	100	139
11	NA	NA	NA	NA	NA	NA	NA
12					NA		
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	NA	500	139 m/z
14	NT				NA		
15	NA	NA	NA	NA	NA	NA	NA
16	NA	Lu			NA	NT	NT
17	NT	NA			NA		NA
18	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20	ICP-MS/MS	Ge	ORS	standard mode	20	20	139
21					NA	100	
22	ICP-MS	Lutetium	ORS	No Gas	NA	1000	139m/z
23					NA		
24	NA	NA	NA	NA	NA	NA	NA
25	NA	NA	NA	NA	NA	NA	NA

Table 101 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	NA	NA	He	800	NA	7
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	670.783
5	ICP-OES-AV	Y				NA	610.36
6						NA	
7	ICP-MS	Sc	ORS	No Gas	200	NA	45
8						NA	
9						NA	
10	ICP-MS	⁷² Ge/ ¹⁰³ Rh/ ¹⁹³ Ir	ORS	He	200	NA	7
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV					NA	670.783
13	ICP-OES	Cs, Y			500	NA	670.783 nm
14	ICP-OES-AV-buffer	Y			100	NA	670.784
15	NA	NA	NA	NA	NA	NA	NA
16	NA	Lu			NT	NA	NT
17	ICP-MS	Sc - 45	KED		10	NA	7
18	NA	NA	NA	NA	NA	NA	NA
19	ICP-MS	Sc	CRI	NA	500	NA	7
20	ICP-OES-AV	Lu	NA	NA	NA	25	670.783
21					100	NA	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	NA	670.783nm
23	ICP-MS	Sc	NA	NA	625	NA	7
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Scandium	ORS	standard mode			

Table 102 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	Y	NA	NA	NA	800	285.213
2						NA	
3	ICP-OES-AV	Lu	NA	NA	50	50	279.8
5	ICP-OES-AV	Y				NA	279.078
6	ICP-OES-AV-buffer	Lu			100	NA	279.078
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-OES-RV					NA	
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-OES-AV	214.282 Te/371.02 9 Y	NA	NA	200	NA	383.829
11						NA	
12	ICP-OES-RV					NA	383.23
13	ICP-OES	Cs, Y			500	NA	383.829 nm
14	ICP-OES-AV-buffer	Y			100	NA	285.213
15						NA	
16	ICP-OES-AV	Lu			83	NA	383.829
17	NA	NA	NA	NA	NA	NA	NA
18	AAS	NA	NA	NA	2223	NA	285.2
19	NA	NA	NA	NA	NA	NA	NA
20	ICP-OES-AV	Lu	NA	NA	20	NA	279.8
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Lutetium	NA	NA	50	NA	279.800nm
23	ICP-MS	Sc	UC	He	625	NA	25
24	ICP-OES-RV	Yttrium	NA	NA	100	NA	279.077
25	NA	NA	NA	NA	NA	NA	NA

Table 103 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-MS	Rh	ORS	He	800	NA	55
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	257.61
5	ICP-MS	In	CRI	He		NA	55
6	ICP-MS	103	ORS	He	100	NA	55
7	ICP-MS	Sc	ORS	He	200	NA	45
8	ICP-OES-AV					NA	
9						NA	
10	ICP-OES-AV	214.282 Te/371.02 9 Y	NA	NA	200	NA	257.61
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV					NA	257.61
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	NA	55 m/z
14	ICP-OES-AV- buffer	Y			100	NA	257.61
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	NA	260.568
17	ICP-MS	Sc 45	KED		100	NA	55
18	ICP-OES-AV	NA	NA	NA	496	NA	294.921
19	ICP-MS	Sc	CRI	He	500	NA	55
20	ICP-OES-AV	Lu	NA	NA	20	25	257.61
21	ICP-MS	Sc	KED	He	100	NA	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	NA	257.610nm
23	ICP-MS	Sc	UC	He	625	NA	55
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Rhodium	ORS	He			

Table 104 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-MS	Rh	ORS	He	800	NA	95
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	202.032
5	ICP-MS	In	CRI	He			98
6	ICP-MS	103	ORS	He	100	NA	98
7	ICP-MS	Rh	ORS	He	200	200	103
8	ICP-OES-AV						
9						NA	
10	ICP-MS	⁷² Ge/ ¹⁰³ Rh/ ¹⁹³ Ir	ORS	He	200	500	95
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV						202.032
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	500	98 m/z
14	ICP-OES-AV-buffer	Y			100	100	202.031
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	83	202.568
17	ICP-MS	Y 89	KED		100	2000	98
18	ICP-OES-AV	NA	NA	NA	49.6	NA	202.032
19	ICP-MS	Rh	CRI	He	500	500	95
20	ICP-OES-AV	Lu	NA	NA	20	NA	202.032
21	ICP-MS	Rh	KED	He	100	100	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	50-1000	202.032nm
23	ICP-MS	Rh	NA	NA	625	625	95
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Telerium	ORS	standard mode			

Table 105 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-AV	Y	NA	NA	NA	800	
2						NA	
3	ICP-OES-AV	Lu	NA	NA	50	50	588.995
5	ICP-OES-RV	Y				NA	589.592
6	ICP-OES-AV-buffer	Lu			100	NA	589.592
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-OES-RV					NA	
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-OES-AV	214.282 Te/371.02 9 Y	NA	NA	200	NA	589.592
11						NA	
12	ICP-OES-RV					NA	216.555
13	ICP-OES	Cs, Y			500	NA	589.592 nm
14	ICP-OES-AV-buffer	Y			100	NA	589.592
15						NA	
16	ICP-OES-AV	Lu			83	NA	330.237
17	NA	NA	NA	NA	NA	NA	NA
18	AAS	NA	NA	NA	44.5	NA	330.2
19	NA	NA	NA	NA	NA	NA	NA
20	ICP-OES-AV	Lu	NA	NA	400	NA	588.995
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Lutetium	NA	NA	50	NA	588.995nm
23	ICP-MS	Sc	UC	He	625	NA	23
24	ICP-OES-RV	Yttrium	NA	NA	100	NA	589.592
25	NA	NA	NA	NA	NA	NA	NA

Table 106 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-MS	Rh	ORS	He	800	NA	60
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	231.604
5	ICP-MS	In	CRI	He			60
6	ICP-MS	103	ORS	He	100	NA	60
7	ICP-MS	Sc	ORS	He	200	200	45
8	ICP-OES-AV						
9						NA	
10	ICP-MS	⁷² Ge/ ¹⁰³ Rh/ ¹⁹³ Ir	ORS	He	200	500	60
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV						
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	500	60 m/z
14	ICP-OES-AV-buffer	Y			100	100	221.648
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	83	231.604
17	ICP-MS	Sc 45	KED		100	2000	58
18	ICP-OES-AV	NA	NA	NA	496	NA	231.604
19	ICP-MS	Sc	CRI	He	500	500	60
20	ICP-OES-AV	Lu	NA	NA	20	25	231.604
21	ICP-MS	Ga	KED	He	50	50	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	50-1000	231.604nm
23	ICP-MS	Ge	UC	He	625	625	60
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Rhodium	ORS	He			

Table 107 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-MS	Rh	ORS	HEHe	NA	800	31
2						NA	
3	ICP-OES-AV	Lu	NA	NA	50	50	182.143
5	ICP-OES-AV	Y				NA	210.623
6	ICP-OES-AV-buffer	Lu				NA	213.618
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-OES-AV					NA	
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-OES-AV	214.282 Te/371.02 9 Y	NA	NA	200	NA	177.434
11						NA	
12						NA	
13	ICP-OES	Cs, Y			500	NA	185.827 nm
14	ICP-OES-AV-buffer	Y			100	NA	214.914
15						NA	
16	ICP-OES-AV	Lu			83	NA	178.222
17	NA	NA	NA	NA	NA	NA	NA
18	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20	ICP-OES-AV	Lu	NA	NA	2000	NA	182.143
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Lutetium	NA	NA	50	NA	213.618nm
23	ICP-MS	Sc	UC	He	625	NA	31
24	ICP-OES-AV	Yttrium	NA	NA	100	NA	178.221
25	ICP-MS	Iridium	ORS	standard mode			

Table 108 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-MS	Ir	ORS	He	800	NA	208
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	220.353
5	ICP-MS	Tb	CRI				
6	ICP-MS	103	ORS	He	100	NA	208
7	ICP-MS	Lu	ORS	He	200	200	175
8	ICP-OES-AV						
9	ICP-OES-AV					NA	
10	ICP-OES-AV	214.282 Te/371.02 9 Y	NA	NA	200	10	220.353
11	NA	NA	NA	NA	NA	NA	NA
12							
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	500	206, 207, 208 m/z
14	ICP-OES-AV- buffer	Y			100	100	220.353
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	83	220.353
17	ICP-MS	NA	KED		100	2000	208
18	ICP-OES-AV	NA	NA	NA	496	NA	220.353
19	ICP-MS	Lu	CRI	He	500	500	208
20	ICP-OES-AV	Lu	NA	NA	20	NA	220.353
21	ICP-MS	Tb	KED	He	50	50	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	50-1000	220.353nm
23	ICP-MS	Ir	NA	NA	625	625	206+207+208
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Iridium	ORS	standard mode			

Table 109 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	ICP-MS	Rh	ORS	He	800	NA	85
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-MS	Ge	ORS	standard mode	50	50	85
5						NA	
6						NA	
7	NA	NA	NA	NA	NA	NA	NA
8						NA	
9						NA	
10	NA	NA	NA	NA	NA	NA	NA
11	NA	NA	NA	NA	NA	NA	NA
12						NA	
13	NT					NA	
14	NT					NA	
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	NT	NT
17	NT	NA				NA	NA
18	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20	ICP-MS	Ge	ORS	standard mode	200	NA	85
21	ICP-MS	Rh	NA	He	100	NA	
22	ICP-MS	Germanium	ORS	He	1000	NA	85m/z
23						NA	
24	NA	NA	NA	NA	NA	NA	NA
25	NA	NA	NA	NA	NA	NA	NA

Table 110 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-RV	Y	NA	NA	NA	800	181.972
2						NA	
3	ICP-OES-AV	Lu	NA	NA	50	50	181.972
5						NA	
6	ICP-OES-AV-buffer	Lu				NA	181.972
7	NA	NA	NA	NA	NA	NA	NA
8	ICP-OES-AV					NA	
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-OES-AV	214.282 Te/371.02 9 Y	NA	NA	200	NA	181.972
11						NA	
12						NA	
13	ICP-OES	Cs, Y			500	NA	181.972 nm
14	ICP-OES-AV-buffer	Y			100	NA	181.975
15						NA	
16	ICP-OES-AV	Lu			83	NA	181.972
17	NA	NA	NA	NA	NA	NA	NA
18	ICP-OES-AV	NA	NA	NA	44.5	NA	181.972
19	NA	NA	NA	NA	NA	NA	NA
20	ICP-OES-AV	Lu	NA	NA	20	NA	181.972
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Lutetium	NA	NA	50	NA	181.972nm
23	ICP-OES-AV					NA	
24	ICP-OES-AV	Yttrium	NA	NA	100	NA	181.975
25	NA	NA	NA	NA	NA	NA	NA

Table 111 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	ORS	He	800	NA	121
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	206.834
5	ICP-MS	In	CRI	He		NA	121
6	ICP-MS	103	ORS	He	100	NA	121
7	ICP-MS	Rh	ORS	He	200	NA	103
8						NA	
9						NA	
10	ICP-MS	⁷² Ge/ ¹⁰³ Rh/ ¹⁹³ Ir	ORS	He	200	NA	121
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV					NA	217.582
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	NA	121 m/z
14	NT					NA	
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	NA	206.834
17	NT	NA				NA	NA
18	ICP-OES-AV	NA	NA	NA	49.6	NA	206.834
19	ICP-MS	Rh	CRI	He	500	NA	123
20	ICP-OES-AV	Lu	NA	NA	NA	25	206.834
21	NA	NA	NA	NA	100	NA	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	NA	206.834nm
23	ICP-MS	Rh	NA	NA	625	NA	121
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Rhodium	ORS	standard mode			

Table 112 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	ORS	He	800	NA	78
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	196.026
5	ICP-MS	In	CRI	He			78
6	ICP-MS	72	ORS	He	100	NA	78
7	ICP-MS	Sc	ORS	H2	200	200	45
8							
9						NA	
10	ICP-MS	72 Ge/103 Rh/193 Ir	ORS	HEHe	200	100	78
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV						196.026
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	500	82 m/z
14	ICP-OES-AV-buffer	Y			100	100	196.026
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	83	196.026
17	ICP-MS	Ge-1 72	KED		100	2000	82
18	ICP-OES-AV	NA	NA	NA	49.6	NA	196.026
19	ICP-MS	Rh	CRI	H2	500	500	78
20	ICP-OES-AV	Lu	NA	NA	20	25	196.026
21	ICP-MS	Te	NA	He	100	100	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	50-1000	196.026nm
23	ICP-MS	Rh	UC	He	625	625	82
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Telerium	ORS	HEHe			

Table 113 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	ORS	He	800	NA	118
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	189.925
5	ICP-MS	In	CRI	He		NA	120
6	ICP-MS	103	ORS	He	100	NA	118
7	ICP-MS	Rh	ORS	He	200	NA	103
8						NA	
9						NA	
10	ICP-MS	⁷² Ge/ ¹⁰³ Rh/ ¹⁹³ Ir	ORS	He	200	NA	118
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV					NA	189.925
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir			500	NA	118 m/z
14	ICP-OES-AV-buffer	Y			100	NA	189.927
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	NA	189.925
17	ICP-MS	In-1 115	KED		100	NA	118
18	ICP-OES-AV	NA	NA	NA	49.6	NA	181.059
19	ICP-MS	Rh	CRI	He	500	NA	118
20	ICP-OES-AV	Lu	NA	NA	20	NA	189.925
21	ICP-MS	Rh	NA	He	100	NA	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	NA	189.925nm
23						NA	
24	NA	NA	NA	NA	NA	NA	NA
25	NA	NA	NA	NA	NA	NA	NA

Table 114 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-RV	Y	NA	NA	NA	800	421.552
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	421.552
5	ICP-OES-RV	Y			NA		407.77
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS	Rh	ORS	He	NA	200	103
8	ICP-OES-AV				NA		
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-MS	72 Ge/103 Rh/193 Ir	ORS	He	NA	100	88
11	NA	NA	NA	NA	NA	NA	NA
12					NA		
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	NA	500	88 m/z
14	ICP-OES-AV-buffer	Y			NA	100	421.552
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			NA	83	407.771
17	NA	NA	NA	NA	NA	200	NA
18	NA	NA	NA	NA	NA	NA	NA
19	ICP-MS	Rh	CRI	He	NA	500	88
20	ICP-OES-AV	Lu	NA	NA	NA	25	421.552
21					NA	100	
22	ICP-OES-AV	Lutetium	NA	NA	NA	50-1000	407.771nm
23	ICP-MS	Rh	NA	NA	NA	625	88
24	NA	NA	NA	NA	NA	NA	NA
25	NA	NA	NA	NA	NA	NA	NA

Table 115 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	ICP-MS	Ir	ORS	He	800	NA	232
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-MS	Lu	ORS	standard mode	50	50	232
5	ICP-MS	Tb	CRI		NA		232
6	NA	NA	NA	NA	NA	NA	NA
7	NA	NA	NA	NA	NA	NA	NA
8					NA		
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-MS	72 Ge/103 Rh/193 Ir	ORS	He	NA	100	232
11	NA	NA	NA	NA	NA	NA	NA
12					NA		
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	NA	500	232 m/z
14	NT				NA		
15	NA	NA	NA	NA	NA	NA	NA
16	NA	Lu			NA	NA	NT
17	NT	NA				NA	NA
18	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA
20	ICP-MS	Lu	ORS	standard mode	200	NA	232
21					NA	NT	
22	ICP-MS	Lutetium	ORS	No Gas	NA	1000	232m/z
23	ICP-MS	Ir	NA	NA	NA	625	232
24	NA	NA	NA	NA	NA	NA	NA
25	NA	NA	NA	NA	NA	NA	NA

Table 116 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	ORS	He	800	NA	
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-MS	Lu	ORS	standard mode	50	50	
5	ICP-MS	Tb	CRI			NA	205
6	ICP-MS	103	ORS	He	100	NA	205
7	ICP-MS	Lu	ORS	He	200	NA	175
8						NA	
9						NA	
10	ICP-MS	72 Ge/103 Rh/193 Ir	ORS	He	200	NA	205
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV					NA	190.794
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	NA	203 m/z
14	ICP-OES-AV-buffer	Y			100	NA	190.801
15	NA	NA	NA	NA	NA	NA	NA
16	NA	Lu			NT	NA	NT
17	NT	NA			NA		NA
18	ICP-OES-AV	NA	NA	NA	49.6	NA	190.794
19	ICP-MS	Lu	CRI	He	500	NA	205
20	ICP-OES-AV	Lu	NA	NA	NA	25	190.794
21					100	NA	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	NA	190.764nm
23	ICP-MS	Ir	NA	NA	625	NA	205
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Iridium	ORS	standard mode			

Table 117 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	Rh	ORS	He	800	NA	
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-MS	Lu	ORS	standard mode	50	50	
5	ICP-MS	Tb	CRI		NA		238
6	NA	NA	NA	NA	NA	NA	NA
7	ICP-MS	Lu	ORS	He	NA	200	175
8					NA		
9	NA	NA	NA	NA	NA	NA	NA
10	ICP-MS	72 Ge/103 Rh/193 Ir	ORS	He	NA	10	238
11	NA	NA	NA	NA	NA	NA	NA
12					NA		
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	NA	500	238 m/z
14	NT				NA		
15	NA	NA	NA	NA	NA	NA	NA
16	NA	Lu			NA	NT	NT
17	ICP-MS	Ir 193	KED		NA	200	238
18	NA	NA	NA	NA	NA	NA	NA
19	ICP-MS	Lu	CRI	He	NA	500	238
20	ICP-MS	Lu	ORS	standard mode	NA	250	238
21					NA	100	
22	ICP-MS	Lutetium	ORS	No Gas	NA	1000	238m/z
23	ICP-MS	Ir	NA	NA	NA	625	238
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Iridium	ORS	standard mode			

Table 118 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-MS	Rh	ORS	He	800	NA	51
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	292.401
5	ICP-MS	In	CRI	He		NA	51
6	ICP-MS	103	ORS	He	100	NA	51
7	ICP-MS	Sc	ORS	He	200	NA	45
8	ICP-OES-AV					NA	
9						NA	
10	ICP-MS	⁷² Ge/ ¹⁰³ Rh/ ¹⁹³ Ir	ORS	He	2000	NA	51
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV					NA	292.401
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	NA	51 m/z
14	ICP-OES-AV-buffer	Y			100	NA	292.402
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	NA	292.401
17	ICP-MS	Sc 45	KED		10	NA	51
18	ICP-OES-AV	NA	NA	NA	49.6	NA	292.401
19	ICP-MS	Sc	CRI	He	500	NA	51
20	ICP-OES-AV	Lu	NA	NA	20	NA	292.401
21	ICP-MS	Sc	KED	He	100	NA	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	NA	292.401nm
23	ICP-MS	Sc	UC	He	625	NA	51
24	NA	NA	NA	NA	NA	NA	NA
25	NA	NA	NA	NA	NA	NA	NA

Table 119 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-MS	Rh	ORS	He	800	NA	64Mini
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV	Lu	NA	NA	50	50	206.2
5	ICP-MS	In	CRI	He			66
6	ICP-MS	103	ORS	He	100	NA	66
7	ICP-MS	Sc	ORS	He	200	200	45
8	ICP-OES-AV						
9	ICP-OES-AV					NA	
10	ICP-OES-AV	214.282 Te/371.02 9 Y	NA	NA	200	10	202.548
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV						206.2
13	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir		He	500	500	66 m/z
14	ICP-OES-AV- buffer	Y			100	100	213.857
15	NA	NA	NA	NA	NA	NA	NA
16	ICP-OES-AV	Lu			83	83	213.857
17	ICP-MS	Sc 45	KED		100	2000	66
18	ICP-OES-AV	NA	NA	NA	496	NA	206.2
19	ICP-MS	Sc	CRI	He	500	500	66
20	ICP-OES-AV	Lu	NA	NA	20	25	206.2
21	ICP-MS	Ga	KED	He	50	50	
22	ICP-OES-AV	Lutetium	NA	NA	50-1000	50-1000	206.200nm
23	ICP-MS	Ge	UC	He	625	625	66
24	NA	NA	NA	NA	NA	NA	NA
25	ICP-MS	Rhodium	ORS	He			

Table 120 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-RV	Y	NA	NA	40	422.673
2	ICP-OES-RV					
3	ICP-OES-AV-buffer				100	422.673
5	ICP-OES-AV	Y			10	317.933
8	ICP-OES-AV				50	315.887
13	ICP-OES	Cs Y		500	NA	315.887 nm
15	ICP-OES-RV					315.887
16	ICP-OES-AV	Lu				317.933
18	AAS	NA	NA	NA	994	422.7
20	ICP-OES-AV	Lu	NA	NA	50	315.887
22	ICP-OES-AV	Lutetium	NA	NA	100	315.887
23	ICP-OES-RV	Y	NA		20	317.933
24	ICP-OES-RV	Yttrium	NA	NA	20	315.887

Table 121 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-RV	Y	NA	NA	40	279.078
2	ICP-OES-AV					
3	ICP-OES-AV-buffer				100	766.491
5	ICP-OES-AV	Y			10	766.491
8					50	279.077
13	ICP-OES	Cs Y		500	NA	766.491 nm
15	ICP-OES-RV					383.829
16	ICP-OES-AV	Lu				383.829
18	AAS	NA	NA	NA	198.8	766.5
20	ICP-OES-AV	Lu	NA	NA	5	766.491
22	ICP-OES-AV	Caesium	NA	NA	100	766.491
23	ICP-OES-RV	Y	NA		20	766.49
24	ICP-OES-RV	Yttrium	NA	NA	20	766.49

Table 122 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-RV	Y	NA	NA	40	589.592
2	ICP-OES-RV					
3	ICP-OES-AV-buffer				100	285.213
5	ICP-OES-AV	Y			10	279.078
8					50	589.592
13	ICP-OES	Cs Y		500	NA	383.829 nm
15	ICP-OES-RV					589.592
16	ICP-OES-AV	Lu				589.592
18	AAS	NA	NA	NA	397.6	422.7
20	ICP-OES-AV	Lu	NA	NA	5	279.8
22	ICP-OES-AV	Lutetium	NA	NA	100	279.8
23	ICP-OES-RV	Y	NA		20	285.213
24	ICP-OES-RV	Yttrium	NA	NA	20	279.077

Table 123 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-RV	Y	NA	NA	40	766.491
2	ICP-OES-AV					
3	ICP-OES-AV-buffer			100	589.592	
5	ICP-OES-AV	Y			10	598.592
8					50	766.49
13	ICP-OES	Cs Y		500	NA	589.592 nm
15	ICP-OES-RV					253.7
16	ICP-OES-AV	Lu				769.897
18	AAS	NA	NA	NA	19.88	330.2
20	ICP-OES-AV	Lu	NA	NA	5	589.592
22	ICP-OES-AV	Lutetium	NA	NA	100	588.995
23	ICP-OES-RV	Y	NA		20	589.592
24	ICP-OES-RV	Yttrium	NA	NA	20	589.592

END OF REPORT