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AQA 24-01

Metals, Nutrients and Exchangeable Bases in Soil

June 2024

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Accredited for compliance with ISO/IEC 17043:2023

TABLE OF CONTENTS

1	INTRODUCTION	2
1.1	NMI Proficiency Testing Program	2
1.2	Study Aims	2
1.3	Study Conduct	2
2	STUDY INFORMATION	2
2.1	Selection of Matrices and Inorganic Analytes	2
2.2	Participation	2
2.3	Test Material Specification	3
2.4	Laboratory Code	3
2.5	Sample Preparation, Analysis and Homogeneity Testing	3
2.6	Stability of Analytes	3
2.7	Sample Storage, Dispatch and Receipt	3
2.8	Instructions to Participants	3
2.9	Interim and Preliminary Reports	5
3	PARTICIPANT LABORATORY INFORMATION	6
3.1	Test Method Summaries	6
3.2	Additional Information	10
3.3	Basis of Participants' Measurement Uncertainty Estimates	10
3.4	Participant Comments on this PT Study or Suggestions for Future Studies	12
4	PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS	13
4.1	Results Summary	13
5	TABLES AND FIGURES	15
6	DISCUSSION OF RESULTS	145
6.1	Assigned Value	145
6.2	Measurement Uncertainty Reported by Participants	145
6.3	z-Score	146
6.4	E _n -score	148
6.5	Participants' Results and Analytical Methods for Acid Extractable Elements	149
6.6	Participants' Results and Analytical Methods for Exchangeable Cations	164
6.7	Participants' Results and Analytical Methods for Colwell P and Colwell K	165
6.8	Participants' Results and Analytical Methods for Phosphorus Buffer Index-PBI _{+ColP}	166
6.9	Participants' Results and Analytical Methods for Total P	166
6.10	Participants' Results and Analytical Methods for Total Nitrogen	167
6.11	Participants' Results and Analytical Methods for Total Carbon and Total Organic Carbon	167
6.12	Comparison with Previous NMI Proficiency Tests of Metals in Soil	169
6.13	Reference Materials and Certified Reference Materials	169
7	REFERENCES	171
	APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING	173

APPENDIX 2 - ASSIGNED VALUE, Z-SCORE AND E_N SCORE CALCULATION	175
APPENDIX 3 - USING PT DATA FOR UNCERTAINTY ESTIMATION	176
APPENDIX 4 - ACRONYMS AND ABBREVIATIONS	178
APPENDIX 5 - INSTRUMENT DETAILS	180

SUMMARY

This report presents the results of the proficiency test AQA 24-01 Metals, Nutrients and Exchangeable Bases in Soil. The study focused on the measurement of the following acid extractable elements: Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cs, Cu, Fe, Ga, Gd, Hg, K, La, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Rb, S, Sb, Se, Sm, 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 CaCl₂ extract, exchangeable bases (Ca²⁺, K⁺, Mg²⁺, Na⁺) - 1M NH₄Cl extract and moisture content was also included in the program.

The sample set consisted of one dried soil sample, one moist soil sample and one dried agricultural soil sample. The assigned values were the robust average of participants' results. The associated uncertainties were estimated from the robust standard deviation of the participants' results. The results reported by Laboratory 15 in Sample S2 were all approximately double the robust average of participants' results, which is an indication of laboratory bias. To avoid bias in calculation of the assigned value and unfair scoring, these results were excluded from robust average calculations; they were also excluded from the calculation of all summary statistics.

Twenty-six 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 787 z-scores, 709 (90%) returned an acceptable score of |z| ≤ 2.0.

Of 787 E_n-scores, 599 (76%) were acceptable with |E_n| < 1.0.

No laboratory reported results for all 52 tests for which a z-score was calculated.

Laboratory 1 returned the highest number of acceptable z-scores (49 out of 50 reported) and the highest number of acceptable E_n-scores (47 out of 50 reported).

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

There was no significant difference between the variability of participants' results in the dried soil Sample S1 and the variability of participants' results in the moist soil Sample S2.

Se followed by Rb and Sb in S1 were the most difficult elements to analyse.

A limited number of laboratories reported results for Cs, Gd, La and Sm in S2.

- 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 868 numerical results, 833 (96%) were reported with an expanded measurement uncertainty. The magnitude of these expanded uncertainties was within the range 0.0009% to 217% of the reported value.

- 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; and
- controlled drug assay.

AQA 24-01 is the 34th 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: 2023 as a provider of proficiency testing schemes. This study 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 65 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

26 laboratories participated and 24 submitted results.

The timetable of the study was:

Invitations issued: 12 February 2024

Samples dispatched: 11 March 2024

Results due: 12 April 2024
Interim report issued: 15 April 2024
Preliminary report issued 17 April 2024

2.3 Test Material Specification

Three samples were provided for analysis:

Sample S1 was 30 g of dried soil.

Sample S2 was 35 g of moist soil.

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. As the same preparation procedure was followed as in the previous studies only a partial homogeneity test was conducted for all analytes in S1 and S3, with the exception of calcium chloride-extractable B, Colwell K, Colwell P, electrical conductivity, exchangeable Ca, K, Mg and Na, PBI_{+ColP}, pH, S, total carbon, total nitrogen, total organic carbon and total P in S3.

Sample S2 was the same soil material as that used in the preparation of Sample S1 of AQA 23-16, to which a known amount of water was added. No homogeneity study was conducted for this sample, as it was proven to be homogeneous in the previous study.⁶ A partial homogeneity test was conducted for the moisture content in this sample.

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

Sample S2, the moist soil, was stored refrigerated prior to dispatch, whilst all other test samples were stored at ambient temperature.

The samples were dispatched by courier on 11 March 2024.

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 the 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; store this sample refrigerated.
- For Sample S3 for determination of calcium chloride – extractable B, exchangeable bases (Ca^{2+} , Mg^{2+} , Na^+ , K^+) - 1M NH_4Cl extract and of P buffer index (with Colwell P)- PBI +ColP, participants are asked to use the methods defined by Rayment, G.E. and David, J. L in “Soil Chemical Methods-Australasia”.
- 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 (Ca^{2+} , Mg^{2+} , Na^+ , K^+) - 1M NH_4Cl extract. For all other tests, except for pH and EC ($\mu\text{S}/\text{cm}$), report results on as received basis in units of mg/kg.

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	5-200	Ag	0.5-20	Ca (acid extractable)	1000-40000
B	0.5-20	Al	1000-40000	Fe (acid extractable)	1000-40000
Be	0.5-20	As	1-40	K (acid extractable)	50-2000
Cd	0.1-4	Ba	5-200	Mg (acid extractable)	50-2000
Co	5-200	Bi	0.5-20	Na (acid extractable)	50-2000
Cr	5-200	Cd	0.5-20	P (acid extractable)	100-4000
Cu	5-200	Cr	1-40	S (acid extractable)	50-2000
Hg	0.1-4	Cs	0.5-20	Colwell K	50-2000
Ga	0.5-20	Cu	1-40	Colwell P	5-200
Li	5-200	Gd	0.5-20	EC	50-2000 $\mu\text{S}/\text{cm}$
Mn	50-2000	Hg	0.5-20	Exchangeable Ca-1MNH ₄ Cl extract ²	Not Available
Mo	0.5-20	La	1-40	Exchangeable Mg-1MNH ₄ Cl extract ²	Not Available
Ni	5-200	Mn	50-2000	Exchangeable Na-1MNH ₄ Cl extract ²	Not Available
Pb	5-200	Mo	0.5-20	Exchangeable K-1MNH ₄ Cl extract ²	Not Available
Rb	0.5-20	Ni	1-40	Calcium chloride –extractable B ¹	Not Available
Sb	5-200	Se	0.5-20	P buffer index (with Colwell P)- PBI +ColP ³	Not Available
Se	0.1-4	Sm	0.5-20	pH of 1:5soil/ 0.01M CaCl ₂ extract	Not Available
Sn	0.5-20	Tl	0.1-4	Total Carbon	>40000
Sr	5-200	U	0.5-20	Total Nitrogen	1000-40000
Th	0.5-20	V	5-200	Total Organic Carbon	>40000
V	5-200	Zn	50-2000	Total P	Not Available
Zn	50-2000	Moisture Content	5-80%		

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

- 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 5 April 2024.

The due date for results was extended to 12 April 2024 due to Easter long weekend.

2.9 Interim and Preliminary Reports

An Interim Report was emailed to participants on 15 April 2024.

A Preliminary Report was issued on 17 April 2024. 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.

The mercury spike value has been changed from 1.00 ± 0.40 mg/kg in the Preliminary Report to 0.898 ± 0.018 mg/kg in the present Final Report. The revised figure no longer accounts for losses during the preparation process because these were not accurately assessed.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Method Summaries

Summaries of test methods are transcribed in Tables 1 to 9. The instruments and settings reported by participants are presented in Appendix 5.

Table 1 Methodology for Acid Extractable Elements

Lab. Code	Method Reference	Staggered Digestion	Sample Mass (g)	Temp. (°C)	Time (min)	Vol. HNO ₃ (mL)	Vol. HCl (mL)	Vol. HNO ₃ (1:1) (mL)	Vol. HCl (1:1) (mL)	Vol. H ₂ O ₂ (mL)	Other (mL)
1*	USEPA Method 200.2, Revision 2.8	Yes	1	95	60			2		2	10 (1:4 HCl)
2			2.508	95	180	4	6			3	
3		NA	2	98	60	800	2.4				800 H ₂ O
6	USEPA-6010C (Except Mercury by USEPA-7471B)		1	120		5	5			3	
7	In House, US EPA 6020B		2	90-95	60	4	12				4 H ₂ O
9	USEPA 3010		2	95-105	60	4	12				
10	USEPA 200.7/200.8	No	1	95	45	2.5	2.5				
11	Digestion block		1	96	270	4					
12	US EPA 200.2	NA	1	95	50	2	2				10 H ₂ O
13	USEPA 200.2	NA	1	95	60			2	10	2	
14	USEPA 3051A	No	0.5	180	30	9ml	3ml				
15	EPA200.2, (1:1 Nitric: Hydrochloric Acid)		0.5	96	30	1	1				
16		Yes	1	95	2			2	10	2	
17			1	100	120	3	3				
19	200.2 Revision 2.8	Yes	1	95 ± 5	60	2	10				2
20	In House - referencing APHA 3125	No	0.4	120	60	2.5	7.5				
21	US EPA METHOD 3010	No	2	90-95	60	4	12				4 H ₂ O
22*	USEPA 3050B										
23	In House		2	100	60	4	8				4 H ₂ O

*Additional information in Table 10

Table 2 Methodology for Total Carbon

Lab. Code	Method Reference	Total Carbon Test Method	Total Carbon Measurement Technique	Additional Information
4	Total Carbon (6B2b)		IR-Combustion Analyser	
7	In House		Combustion Analyser	S3: TC/TOC tested as part of validation. Not a production test currently.
8	Organic Application Note, LECO Corporation Form No. 203-821-498, "Carbon, Nitrogen and Sulfur in Soil". Instrument TruMac CNS. 2015, REV0.	High Temperature Oxidation	Combustion Analyser	

Lab. Code	Method Reference	Total Carbon Test Method	Total Carbon Measurement Technique	Additional Information
17		High Temperature Oxidation		
19	Rayment & Lyons 6B3	High Temperature Oxidation	Combustion Analyser	
20	In house	High Temperature Oxidation	Combustion Analyser	
21	N/A	In-House	Combustion;INFRARED (IR)	
22		High Temperature Oxidation	Combustion NDIR	
23	Rayment & Lyons 6B2b	High Temperature Oxidation	Dumas high temp combustion, IR detection	
26	Application Note, Elementar Form No. AN-A-030609-E-01, "CNS analysis in soil with the vario EL Cube".	High Temperature Oxidation	Combustion Analyser	

Table 3 Methodology for Total Organic Carbon

Lab. Code	Method Reference	Total Organic Carbon Test Method	Total Organic Carbon Measurement Technique	Additional Information
1	AS 1289.4.1.1	Walkely & Black	Titration	
4				Sample was Fizz test with 4 M HCl and no Fizzing observed. Therefore no acid treatment was carried for TOC
7	In House		Combustion Analyser	S3: TC/TOC tested as part of validation. Not a production test currently.
8	Organic Application Note, LECO Corporation Form No. 203-821-498, "Carbon, Nitrogen and Sulfur in Soil". Instrument TruMac CNS. 2015, REV0.	High Temperature Oxidation	Combustion Analyser	
10	NEPM 105	Chemical Oxidation (no Ag ₂ SO ₄ added)	Titration	
13	AS1289.4.1.1	Other		
16	AS 1289.4.1.1	Chemical Oxidation (Ag ₂ SO ₄ added)	Walkely & Black Method	
17		High Temperature Oxidation		
19	Rayment & Lyons 6B3	High Temperature Oxidation	Combustion Analyser	
20		High Temperature Oxidation	Combustion Analyser	TOC - sample digested with sulfurous acid prior to analysis

Lab. Code	Method Reference	Total Organic Carbon Test Method	Total Organic Carbon Measurement Technique	Additional Information
21	N/A	In House	Combustion;INFRARED (IR)	
22		High Temperature Oxidation	Combustion NDIR	
23	Rayment & Lyons 6B3	High Temperature Oxidation	Dumas high temp combustion, IR detection	Inorganic Carbon measured and subtracted from Total Carbon result to get TOC result

Table 4 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)
19	Rayment & Lyons 9B1 & 18A1	1	100	16	100	100	ICP-OES (wavelength)	UV-Vis (wavelength)
20	Colwell P 9B2, Colwell K 18A1	0.4	40	16	3280	328	ICPMS	FIA (wavelength)

Table 5 Methodology for P Buffer Index – PBI_{ColP}

Lab. Code	Method Reference*	Sample Mass (g)	Extraction Solution (P equilibrating Solution) Volume (mL)	Shake time (hours)	Instrument	Final Dilution Factor	Wavelength (nm) / Absorbance (nm)
4	9I2						
10	9I2						
17	9I2						
20	9I2b	2	20	16	ICP-OES	10	213.617
24	9I2						

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

Table 6 Methodology for Total P

Lab. Code	Method
1	Total P by Kjeldahl digestion and DA
10	Total P by Kjeldahl digestion and DA
13	Other (Persulfate digestion followed by DA)
16	Total P by Kjeldahl digestion and DA
19	Other (Total P by APHA 4500 Norg-D with Jirka modification followed by DA finish)
22	Total P by Kjeldahl digestion and SFA
25	In house method

Table 7 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)
4	12C						
10	12C						
13	12C	10	20	10	ICP-OES		
17	12C						
19	12C	10	20	0.16	ICP-OES	2	249.773
20	12C2	10	20	10	ICP-OES	2	208.889
24	12C						

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

Table 8 Methodology for Total Nitrogen

Lab. Code	Method Reference	Test Method	Measurement Method	Instrument
1	APHA 4500-Norg / 4500-NO ₃ -	Digestion TN = TKN + NO _x	Colorimetric - salicylate method	DA
4	7A5	Dumas	Dumas -High temperature combustion	Combustion Analyser
9	APHA4500-Norg-B,C,D.(4-123)(TKN)	Digestion TN = TKN + NO _x	Colorimetric - phenate method	DA
10	USEPA 351.2 for TKN	Digestion TN = TKN + NO _x	Colorimetric - salicylate method	DA
13	APHA, 4500-P J. & 4500-N C.	Digestion TN = TKN + NO _x	Other	DA
16	APHA 4500 - Norg A &D	Digestion TN = TKN + NO _x	Colorimetric - salicylate method	DA
17		Digestion and Distillation TN = TKN + NO _x	Titrimetric method	Manual Analysis
19	APHA 22nd edition 4500 Norg A & D with Jirka Modification-Jirka et al. (1976)	Digestion TN = TKN + NO _x	Colorimetric - phenate method	DA
20	In house - Dumas combustion	Combustion	Dumas -High temperature combustion	Combustion Analyser
22		Digestion and Distillation TN = TKN + NO _x	Colorimetric - phenate method	DA
25	In house method	Digestion TN = TKN + NO _x	Other (Colorimetric method)	FIA
26	Application Note, Elementar Form No. AN-A-030609-E-01, "CWNS analysis in soil with the vario EL Cube".	Combustion	High temperature combustion	Combustion Analyser

Table 9 Methodology for Exchangeable Bases

Lab. Code	Method Reference*	Sample Mass (g)	Shake time (hrs)	Extraction Solution	Extraction Solution Vol. (mL)
1	15A1	2.5	1	1M NH4Cl	50
4	15A1	2	1	1M NH4Cl	40
10	15A1	2	0.5	1M Ammonium Acetate	20ml
13	15A1	2.5	1	1M NH4Cl	50
16	15A1	2.5	1	1M NH4Cl	50
17	15A1	2	2	1M NH4Cl	40
19	15A1	2.5	1	1M NH4Cl	50
20	15A1	1	1	1M NH4Cl	20
22	15A1			1M NH4Cl	
23	15A1	5	1	1M NH4Cl	100
24	15A1			1M NH4Cl	
25	15A1			1M NH4Cl	

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

3.2 Additional Information

Participants had the option to report additional information for each sample analysed. These are transcribed in Table 10.

Table 10 Additional information

Lab Code	Additional Information
1	S3: pH (CaCl ₂)=4.9, pH (H ₂ O)=5.4
22	S3: Microwave digestion (not hot block)

3.3 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	Bottom Up (ISO/GUM, fish bone/ cause and effect diagram) Coverage factor not reported	Control Samples Duplicate Analysis	Instrument Calibration Recoveries of SS	Eurachem/CITAC Guide
2	Top Down - precision and estimates of the method and laboratory bias $k = 2$	Control Samples - RM Duplicate Analysis Instrument Calibration	CRM	ISO/GUM
3	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration Laboratory Bias from PT Studies Recoveries of SS	ASTM E2554-13
4	Top Down - reproducibility (standard deviation) from PT studies used directly Coverage factor not reported	Control Samples - CRM Duplicate Analysis		Other

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation ^a		Guide Document for Estimating MU
		Precision	Method Bias	
6	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - CRM Duplicate Analysis	CRM Instrument Calibration Recoveries of SS	NMI Uncertainty Course
7	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - SS	Recoveries of SS	ISO/GUM
8	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration	
9	Bottom Up (ISO/GUM, fish bone/ cause and effect diagram) Coverage factor not reported	Control Samples - CRM Instrument Calibration	CRM Instrument Calibration	ISO/GUM
10	Top Down - precision and estimates of the method and laboratory bias $k = 2$			Other (NATA TN33)
11	Top Down - precision and estimates of the method and laboratory bias $k = 2$	Duplicate Analysis	CRM	Eurachem/CITAC Guide
12	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control Samples - CRM Duplicate Analysis	CRM Instrument Calibration Laboratory Bias from PT Studies	Eurachem/CITAC Guide
13	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - CRM Duplicate Analysis Instrument Calibration		Eurachem/CITAC Guide
14	Top Down - precision and estimates of the method and laboratory bias $k = 2$	Control samples - SS Duplicate Analysis	Recoveries of SS	ISO/GUM
15	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Duplicate Analysis		Technical Guide: Measurement Uncertainty in Chemistry and Microbiology
16	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - CRM	CRM Recoveries of SS	Eurachem/CITAC Guide
17	Top Down - precision and estimates of the method and laboratory bias $k = 2$	Control Samples Duplicate Analysis	CRM Recoveries of SS	Nordtest Report TR537
19	Top Down - precision and estimates of the method and laboratory bias $k = 2$	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration	Eurachem/CITAC Guide
20	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - RM Duplicate Analysis	Instrument Calibration Standard Purity	Nordtest Report TR537
21*	See 'Additional Information' section below Coverage factor not reported	Control samples - CRM Instrument Calibration	CRM	See 'Additional Information' section below
22	Top Down - precision and estimates of the method and	Control Samples - CRM Duplicate Analysis	CRM	NMI Uncertainty Course

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation ^a		Guide Document for Estimating MU
		Precision	Method Bias	
	laboratory bias k = 2			
23	Standard deviation of replicate analyses multiplied by 2 or 3 k = 2	Control Samples - CRM	CRM Instrument Calibration Recoveries of SS	Other (ASTM E2587-16)
24	Top Down - reproducibility (standard deviation) from PT studies used directly Coverage factor not reported	Standard deviation from PT studies only		Eurachem/CITAC Guide
		Control Samples	CRM Laboratory Bias from PT Studies	
25	Top Down - precision and estimates of the method and laboratory bias k = 2	Standard deviation from PT studies only		ISO/GUM
		Control Samples Duplicate Analysis	CRM Instrument Calibration Recoveries of SS	
26	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - RM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration	

*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
21	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

3.4 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 77 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 66. An example chart with interpretation guide is shown in Figure 1.

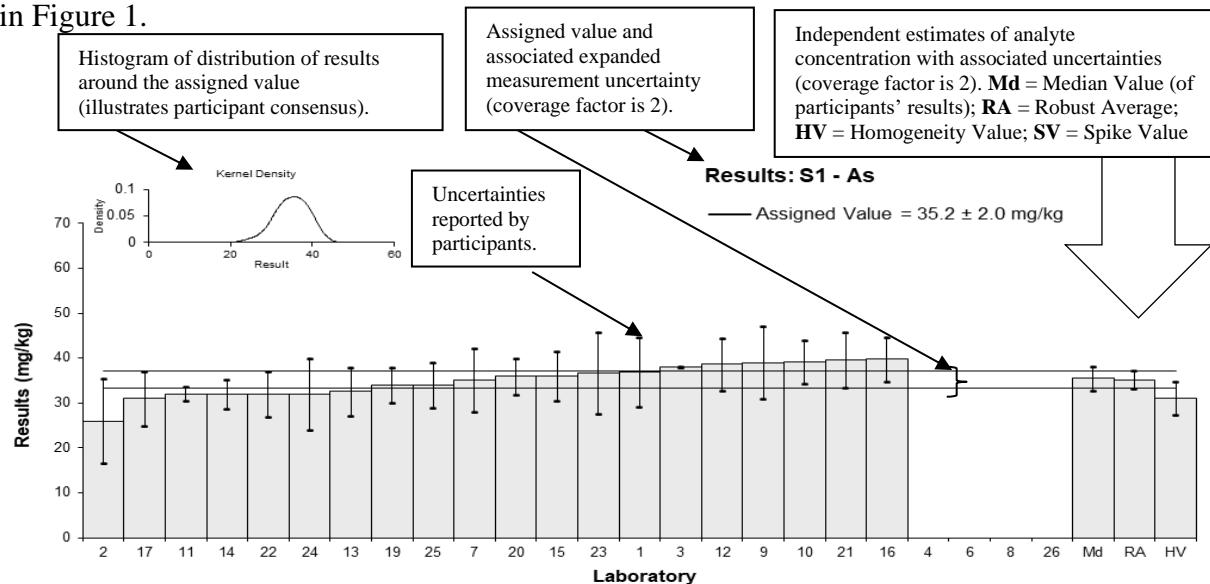


Figure 1 Guide to Presentation of Results

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. Extreme outliers (gross errors) were obvious blunders, such as those with incorrect units, decimal errors, or results from a different proficiency test item and were removed for calculation of summary statistics.^{3,4}

4.3 Assigned Value

An example of the assigned value calculation using data from the present study is given in Appendix 2. The assigned value is defined as: ‘the value attributed to a particular property of a proficiency test item.’¹ In this PT study, the property is the mass fraction of analyte. Assigned values were the robust average of participants’ results, outliers and extreme outliers removed; the expanded uncertainties were estimated from the associated robust standard deviations.^{4,7}

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.’⁷ 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 ISO13528.⁷

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.

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

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.⁸

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 acceptable;
- $2.0 < |z| < 3.0$ is questionable;
- $|z| \geq 3.0$ is unacceptable.

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 assessing laboratory performance.

E_n-score includes measurement uncertainty and is calculated according to Equation 3 below:

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

Where:

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

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

- $|E_n| < 1.0$ is acceptable;
- $|E_n| \geq 1.0$ is unacceptable.

The acceptance criteria for E_n-score has been changed from an acceptable $|E_n|$ score of ≤ 1 to an acceptable $|E_n|$ score of < 1.0 as per new ISO/IEC 17043:2023 requirements.¹

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.⁹ Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.¹⁰

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	36.9	7.65	0.48	0.21
2	26	9.5	-2.61	-0.95
3	38	0.2	0.80	1.39
4	NT	NT		
6	NT	NT		
7	35.1	7.03	-0.03	-0.01
8	NR	NR		
9	39	8	1.08	0.46
10	39.2	4.8	1.14	0.77
11	32	1.6	-0.91	-1.25
12	38.6	5.9	0.97	0.55
13	32.6	5.35	-0.74	-0.46
14	32	3.2	-0.91	-0.85
15	36	5.4	0.23	0.14
16	39.707	5.031	1.28	0.83
17	31	6	-1.19	-0.66
19	34	4	-0.34	-0.27
20	35.9	4.0	0.20	0.16
21	39.54	6.18	1.23	0.67
22	32	5	-0.91	-0.59
23	36.64	9.16	0.41	0.15
24	32.0	8	-0.91	-0.39
25	34	5	-0.34	-0.22
26	NR	NR		

Statistics

Assigned Value	35.2	2.0
Spike Value	Not Spiked	
Homogeneity Value	31.0	3.7
Robust Average	35.2	2.0
Median	35.5	2.7
Mean	35.0	
N	20	
Max	39.707	
Min	26	
Robust SD	3.6	
Robust CV	10%	

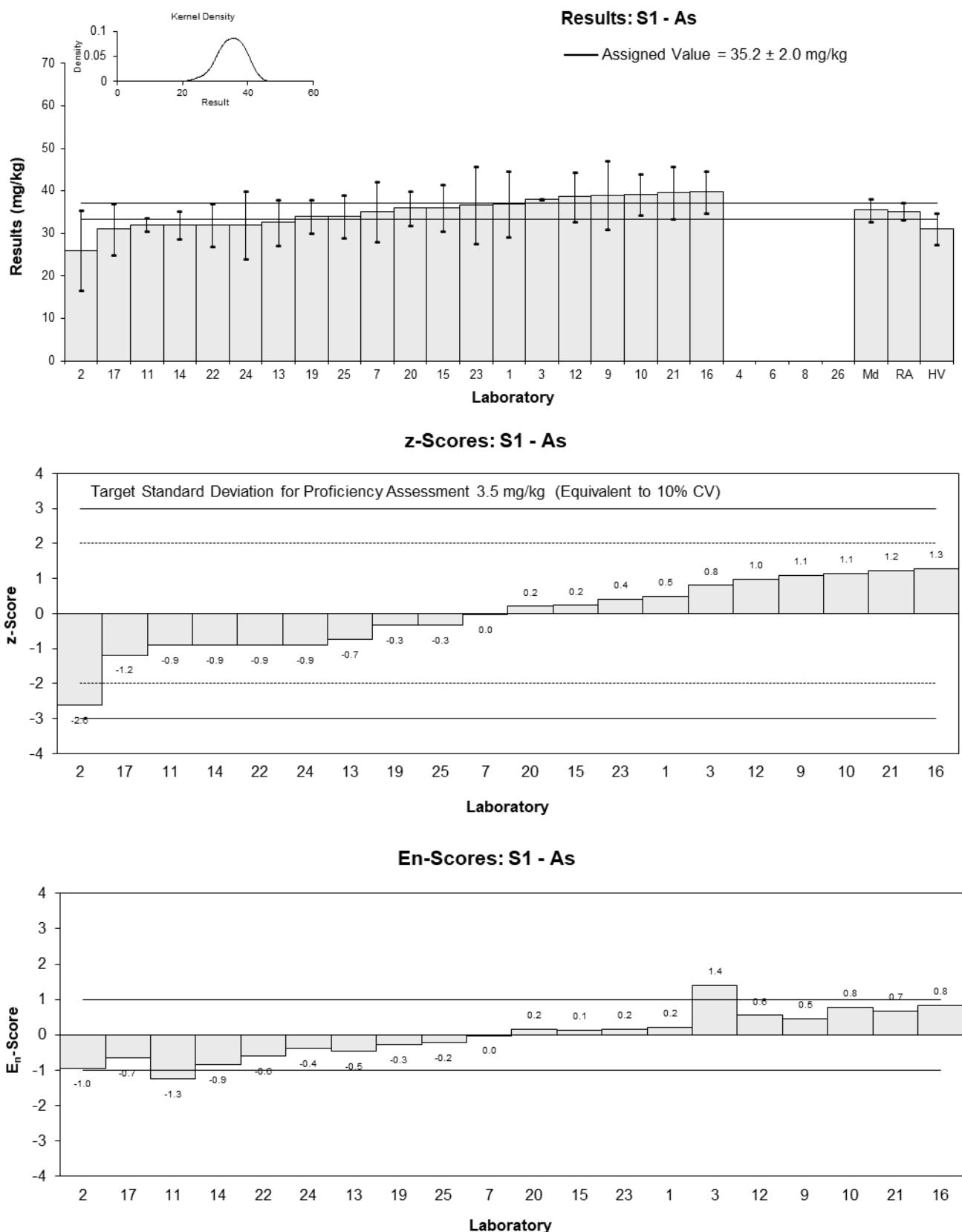


Figure 2

Table 14

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	<50	11.83
2	NT	NT
3	<10	0.2
4	NT	NT
6	NT	NT
7	<10	<2
8	NR	NR
9	< 10	NR
10	<5	5.0
11	<1	NR
12	3.0	1.4
13	NT	NR
14	4	0.4
15	<4.4	NR
16	<50	NR
17	NR	NR
19	<50	NR
20	3.24	0.7
21	< 10	NR
22	9.7	2.5
23	< 10	NR
24	<5	NR
25	5.2	1.0
26	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Homogeneity Value	4.46	0.54
Median	4.0	1.7
Mean	5.0	
N	5	
Max	9.7	
Min	3	

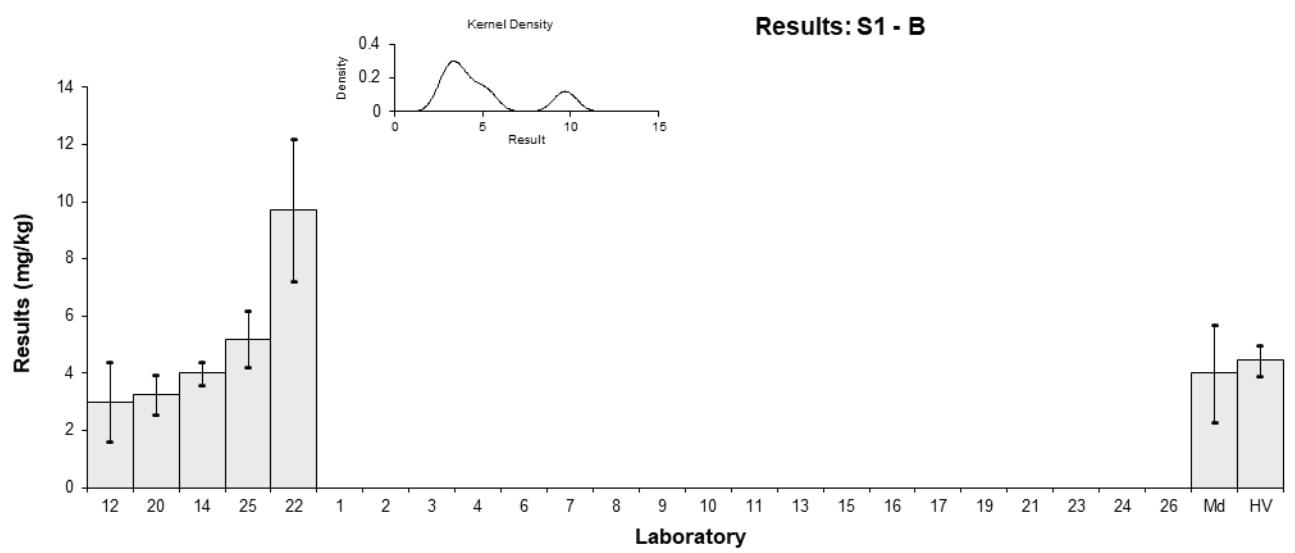


Figure 3

Table 15

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	1	0.24	-0.65	-0.28
2	NT	NT		
3	<2	0.3		
4	NT	NT		
6	1	0.076	-0.65	-0.72
7	<2	<0.4		
8	NR	NR		
9	< 2	NR		
10	0.832	0.5	-2.22	-0.47
11	1.06	0.11	-0.09	-0.08
12	1.15	0.25	0.75	0.31
13	1.0	0.2	-0.65	-0.34
14	<2	NR		
15	1.1	0.35	0.28	0.08
16	1.0984	0.195	0.27	0.14
17	1.1	0.2	0.28	0.14
19	<1	NR		
20	1.07	0.2	0.00	0.00
21	< 2	NR		
22	1.1	0.1	0.28	0.26
23	< 2	NR		
24	1.2	0.2	1.21	0.62
25	<5	NR		
26	NR	NR		

Statistics

Assigned Value	1.07	0.06
Spike Value	1.05	0.07
Homogeneity Value	1.12	0.13
Robust Average	1.07	0.06
Median	1.08	0.05
Mean	1.06	
N	12	
Max	1.2	
Min	0.832	
Robust SD	0.078	
Robust CV	7.3%	

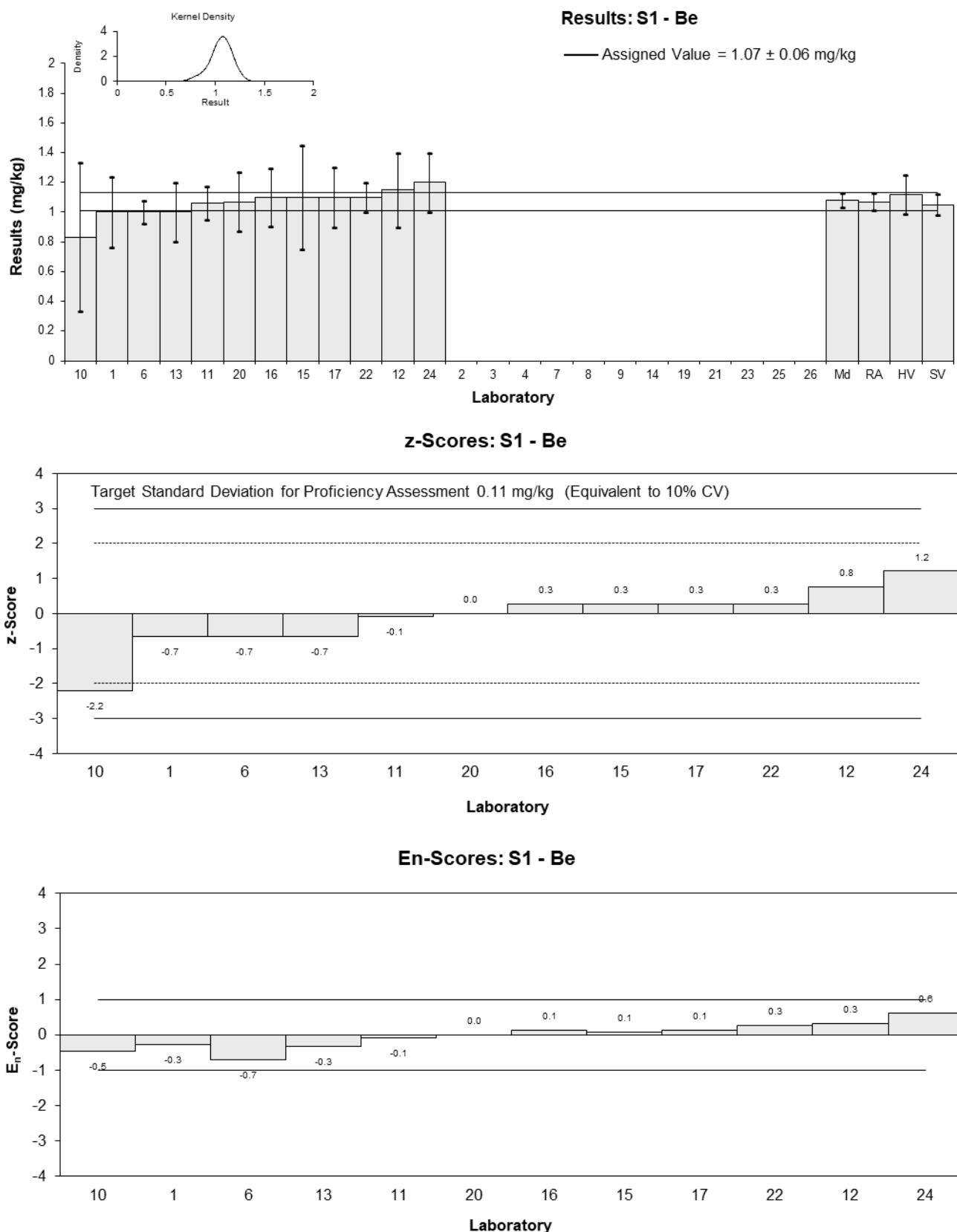


Figure 4

Table 16

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	0.7	0.15	-1.70	-1.21
2*	1.6	0.52	4.68	1.23
3	1	0.2	0.43	0.25
4	NT	NT		
6	1	0.032	0.43	0.45
7	0.984	0.197	0.31	0.19
8	NR	NR		
9	0.8	0.2	-0.99	-0.59
10	0.891	0.08	-0.35	-0.32
11	<1	NR		
12	1.23	0.19	2.06	1.26
13	0.8	0.1	-0.99	-0.85
14	0.5	0.05	-3.12	-3.16
15	0.83	0.16	-0.78	-0.53
16	1.232	0.3	2.07	0.89
17	0.91	0.18	-0.21	-0.14
19	<1	NR		
20	0.74	0.1	-1.42	-1.22
21	1.010	0.152	0.50	0.35
22	NR	NR		
23	0.8670	0.217	-0.52	-0.29
24	1.10	0.2	1.13	0.67
25	1.3	0.1	2.55	2.19
26	NR	NR		

* Outlier, see Section 4.2

Statistics

Assigned Value	0.94	0.13
Spike Value	0.91	0.16
Homogeneity Value	0.85	0.10
Robust Average	0.96	0.14
Median	0.95	0.13
Mean	0.97	
N	18	
Max	1.6	
Min	0.5	
Robust SD	0.24	
Robust CV	25%	

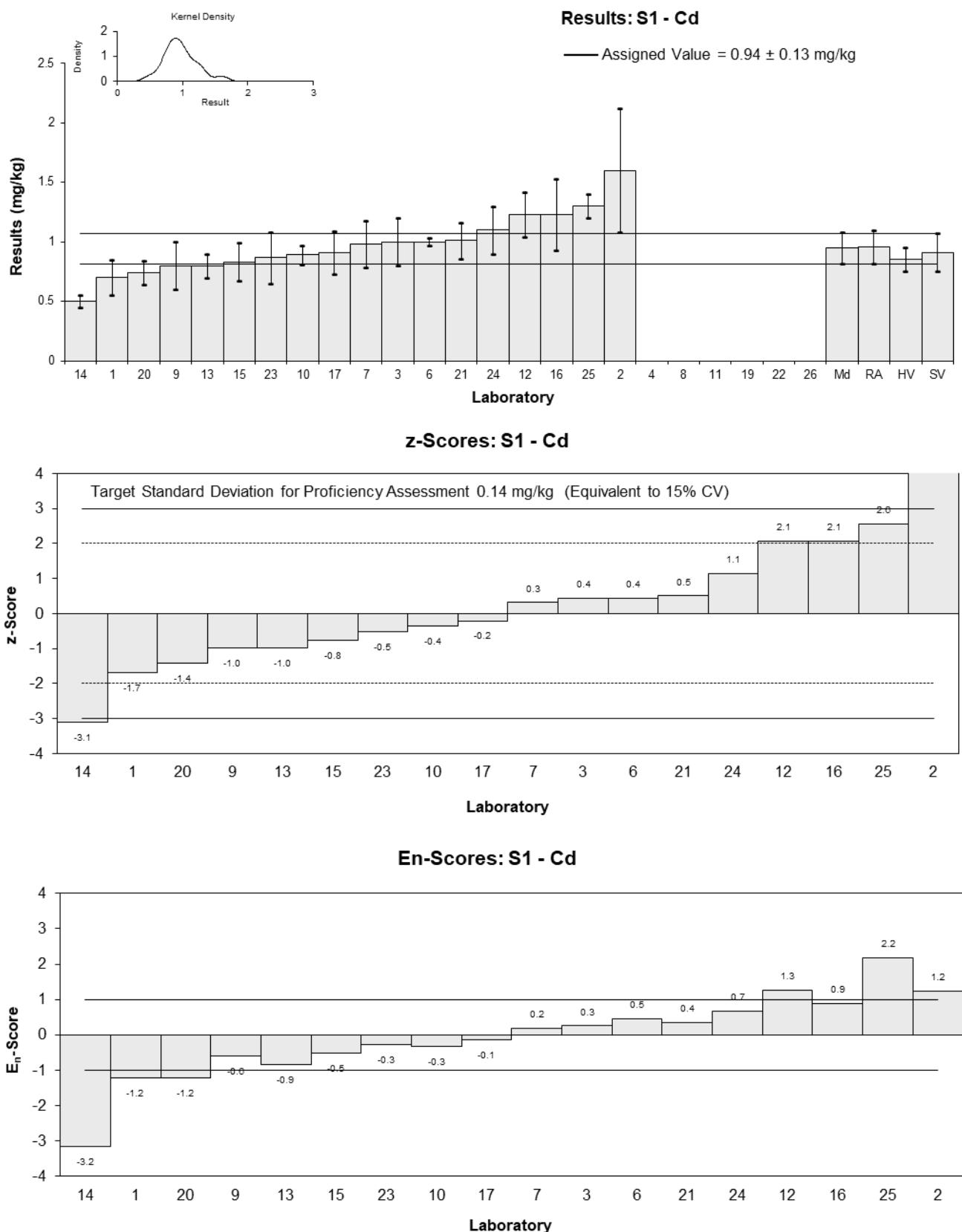


Figure 5

Table 17

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	16.6	3.86	-0.08	-0.05
2	NT	NT		
3	20	0.2	1.27	2.86
4	NT	NT		
6	15	0.588	-0.71	-1.44
7	17.1	3.42	0.12	0.08
8	NR	NR		
9	17	4	0.08	0.05
10	14.3	1.4	-0.99	-1.40
11	17	1.7	0.08	0.10
12	19.3	2.8	0.99	0.83
13	17.5	2.78	0.28	0.23
14	13	1.3	-1.51	-2.23
15	17	2.5	0.08	0.07
16	15.94	1.425	-0.34	-0.48
17	17	3	0.08	0.06
19	13	1	-1.51	-2.56
20	18.6	2.0	0.71	0.79
21	17.90	2.53	0.44	0.40
22	18	3.4	0.48	0.34
23	18.6	4.65	0.71	0.38
24	16.75	0.3	-0.02	-0.04
25	14.9	1.5	-0.75	-1.02
26	NR	NR		

Statistics

Assigned Value	16.8	1.1
Spike Value	Not Spiked	
Homogeneity Value	16.7	2.0
Robust Average	16.8	1.1
Median	17.0	0.9
Mean	16.7	
N	20	
Max	20	
Min	13	
Robust SD	1.9	
Robust CV	11%	

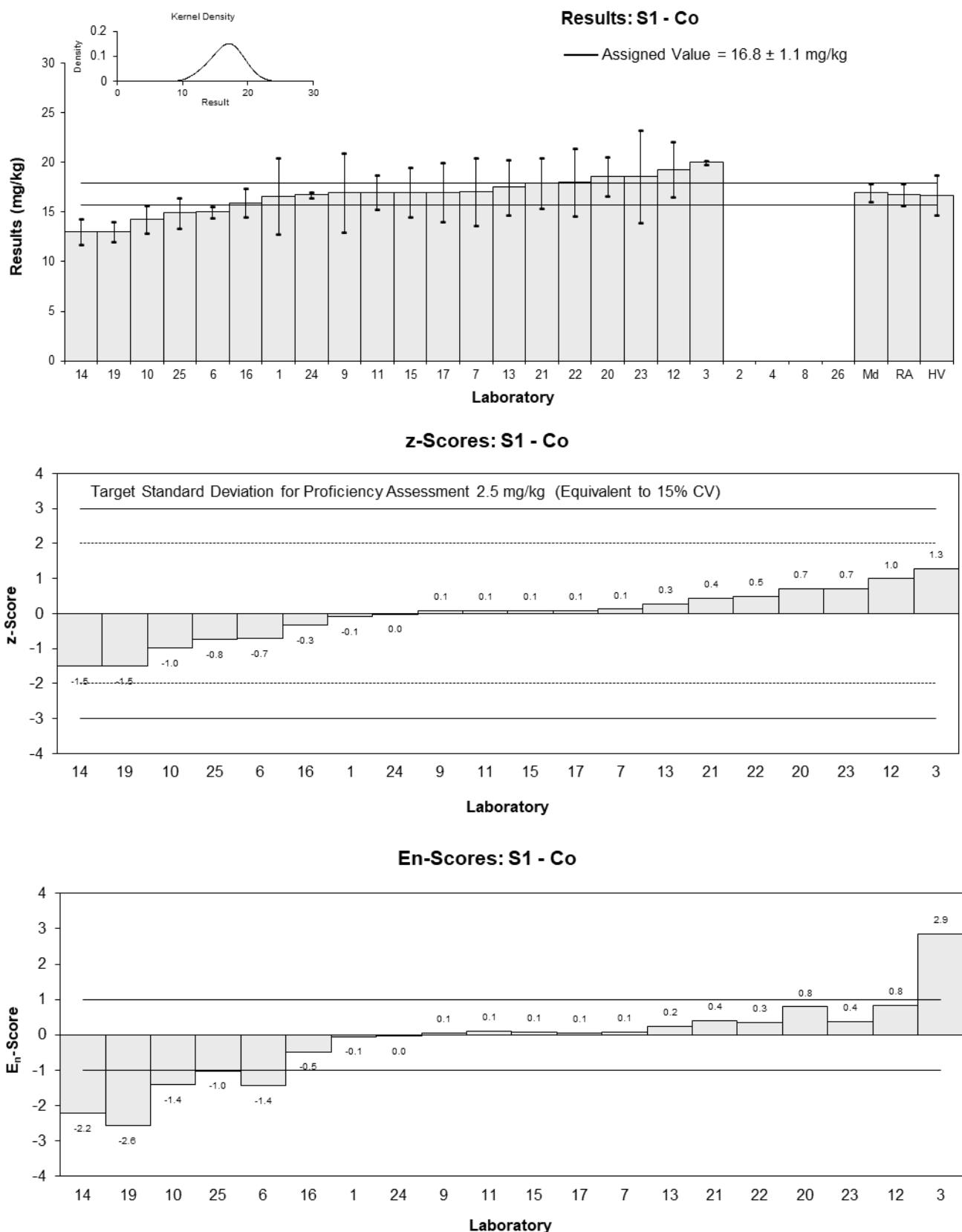


Figure 6

Table 18

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	35	12.45	-0.61	-0.27
2	29	8.5	-1.65	-1.04
3	52	0.2	2.34	3.96
4	NT	NT		
6	37	1.043	-0.26	-0.42
7	46.4	9.28	1.37	0.80
8	NR	NR		
9	38	7	-0.09	-0.06
10	32.9	5.0	-0.97	-0.93
11	35	2.1	-0.61	-0.88
12	39.6	6.3	0.19	0.15
13	38.2	10.7	-0.05	-0.03
14	40	4	0.26	0.29
15	40	6.0	0.26	0.22
16	29.08	3.554	-1.63	-1.92
17	44	9	0.95	0.57
19	29	5	-1.65	-1.57
20	37.8	4.0	-0.12	-0.13
21	41.39	6.54	0.50	0.39
22	46	5.1	1.30	1.22
23	39.42	9.86	0.16	0.09
24	43.5	0.8	0.87	1.43
25	38	4	-0.09	-0.10
26	NR	NR		

Statistics

Assigned Value	38.5	3.4
Spike Value	Not Spiked	
Homogeneity Value	41.8	5.0
Robust Average	38.5	3.4
Median	38.2	2.6
Mean	38.6	
N	21	
Max	52	
Min	29	
Robust SD	6.2	
Robust CV	16%	

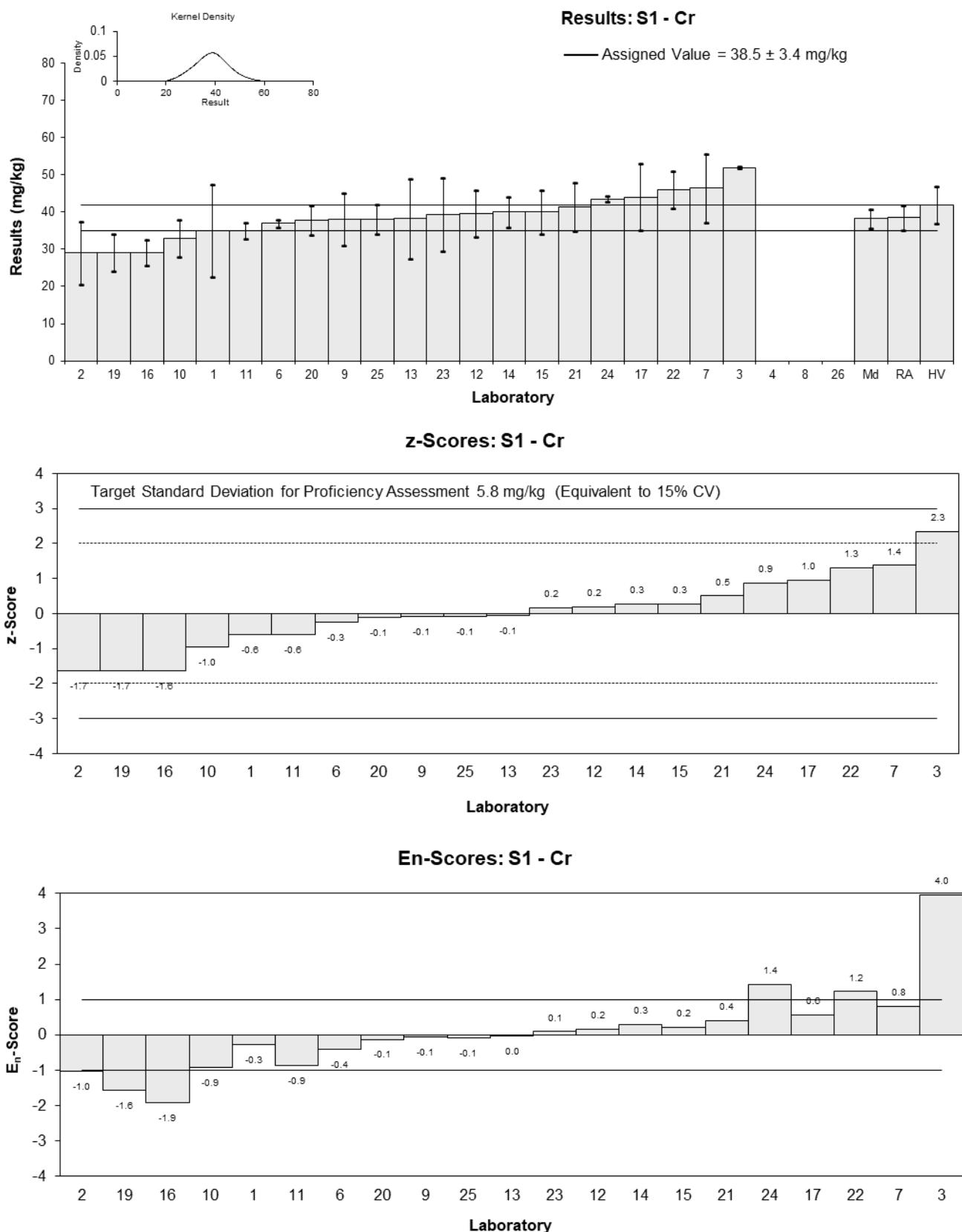


Figure 7

Table 19

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	28.2	8.12	-1.35	-0.53
2	33	5.2	0.12	0.07
3	36	0.2	1.04	2.25
4	NT	NT		
6	35	1.072	0.74	1.30
7	32.0	6.41	-0.18	-0.09
8	NR	NR		
9	31	6	-0.49	-0.26
10	26.8	4.2	-1.78	-1.30
11	40	2.4	2.27	2.61
12	31.0	4.5	-0.49	-0.34
13	33.4	5.63	0.25	0.14
14	30	3	-0.80	-0.78
15	36	5.4	1.04	0.61
16	31.22	3.054	-0.42	-0.41
17	31	6	-0.49	-0.26
19	30	3	-0.80	-0.78
20	34.7	4.0	0.64	0.49
21	33.77	5.87	0.36	0.19
22	31	3.1	-0.49	-0.46
23	34.2	8.55	0.49	0.18
24	35.0	0.7	0.74	1.45
25	33	5	0.12	0.08
26	NR	NR		

Statistics

Assigned Value	32.6	1.5
Spike Value	Not Spiked	
Homogeneity Value	29.2	3.5
Robust Average	32.6	1.5
Median	33.0	1.6
Mean	32.7	
N	21	
Max	40	
Min	26.8	
Robust SD	2.8	
Robust CV	8.6%	

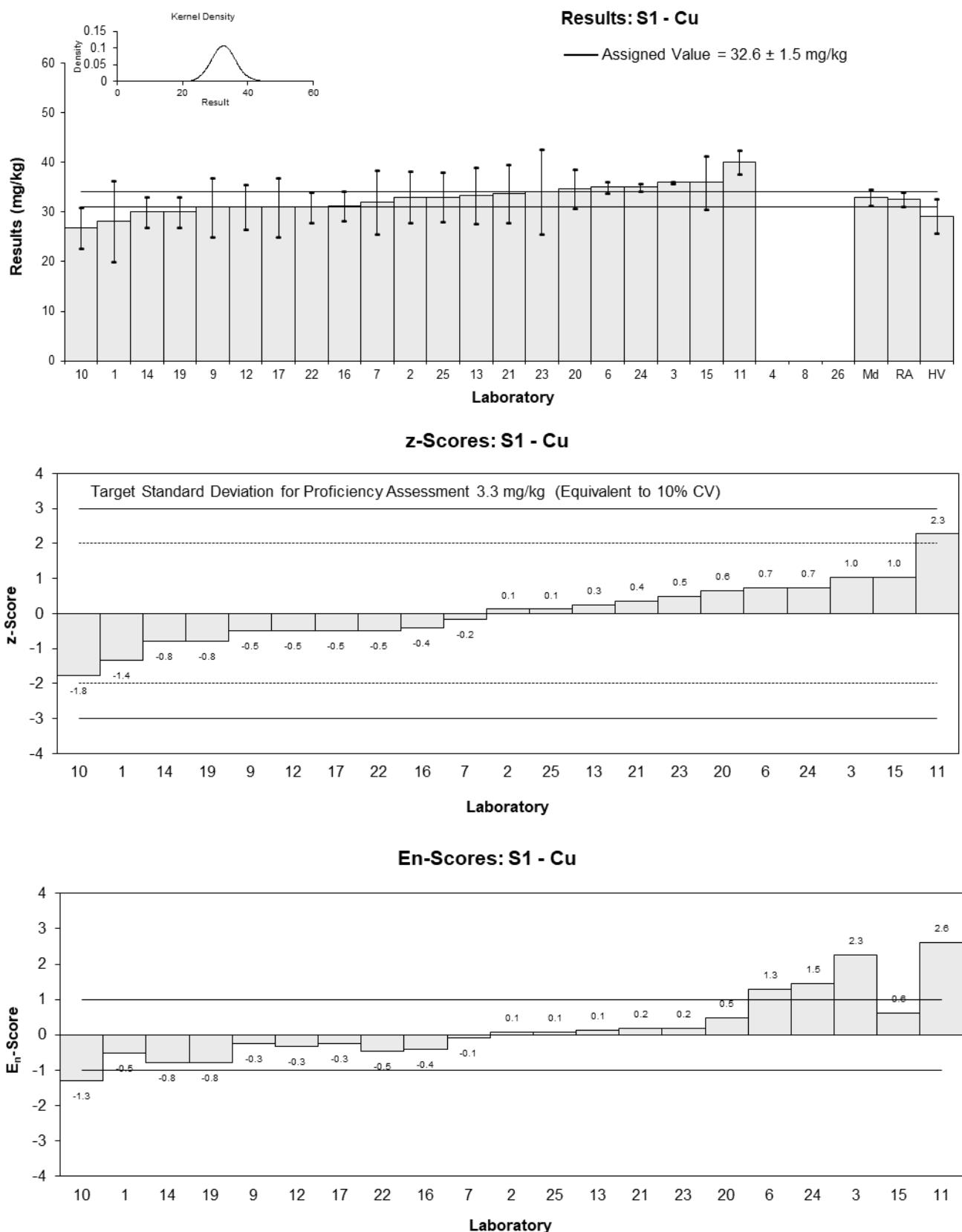


Figure 8

Table 20

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	4.1	0.93	-0.44	-0.29
2	NT	NT		
3	NT	NT		
4	NT	NT		
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	5	2	0.56	0.22
10	NR	NR		
11	NT	NT		
12	NT	NT		
13	3.7	NR	-0.89	-0.80
14	5	0.5	0.56	0.45
15	NT	NT		
16	4.256	NR	-0.27	-0.24
17	4.8	1.0	0.33	0.21
19	2.8	0.5	-1.89	-1.52
20	NT	NT		
21	NT	NT		
22	6.6	0.8	2.33	1.64
23	NT	NT		
24	NR	NR		
25	NT	NT		
26	NR	NR		

Statistics

Assigned Value	4.5	1.0
Spike Value	Not Spiked	
Homogeneity Value	4.67	0.56
Robust Average	4.5	1.0
Median	4.53	0.62
Mean	4.53	
N	8	
Max	6.6	
Min	2.8	
Robust SD	1.2	
Robust CV	26%	

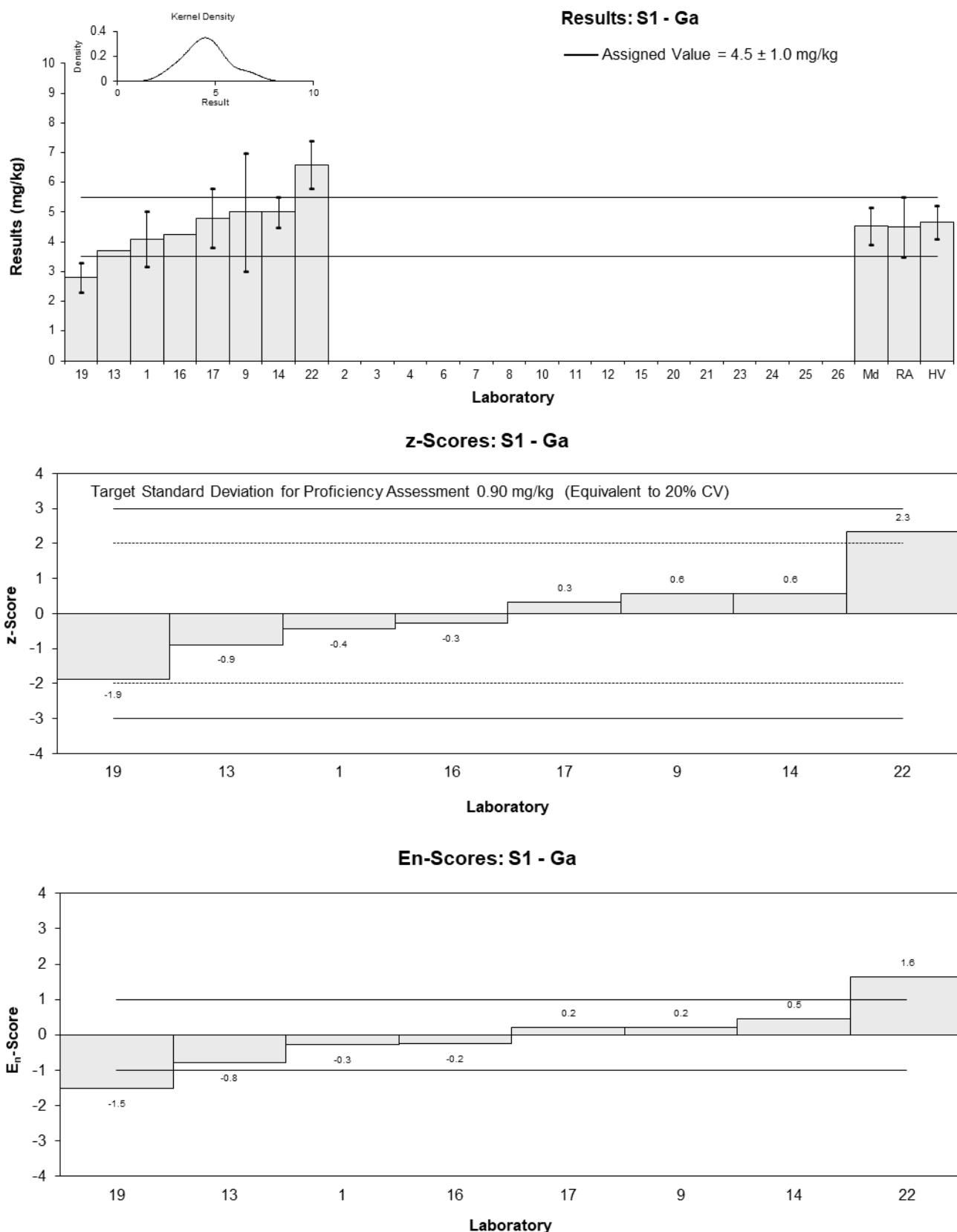


Figure 9

Table 21

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	0.6	0.17	-0.16	-0.09
2	0.61	0.2	-0.05	-0.02
3	0.67	0.4	0.60	0.14
4	NT	NT		
6	0.6	0.072	-0.16	-0.19
7	0.669	0.134	0.59	0.39
8	NR	NR		
9	0.56	0.12	-0.60	-0.44
10	0.598	0.15	-0.18	-0.11
11	<1	NR		
12	0.60	0.12	-0.16	-0.12
13	0.7	0.09	0.92	0.88
14	0.5	0.05	-1.25	-1.87
15	0.58	0.99	-0.38	-0.04
16	0.821	0.131	2.23	1.52
17	0.57	0.11	-0.49	-0.39
19	0.6	0.1	-0.16	-0.14
20	0.63	0.1	0.16	0.14
21	0.669	0.108	0.59	0.47
22	0.57	0.06	-0.49	-0.64
23	0.6	0.15	-0.16	-0.10
24	0.71	0.2	1.03	0.47
25	0.53	0.13	-0.92	-0.63
26	NR	NR		

Statistics

Assigned Value	0.615	0.036
Spike Value*	0.898	0.018
Homogeneity Value	0.602	0.072
Robust Average	0.615	0.036
Median	0.600	0.025
Mean	0.619	
N	20	
Max	0.821	
Min	0.5	
Robust SD	0.064	
Robust CV	10%	

*Spike value uncertainty does not include the losses during the preparation process.

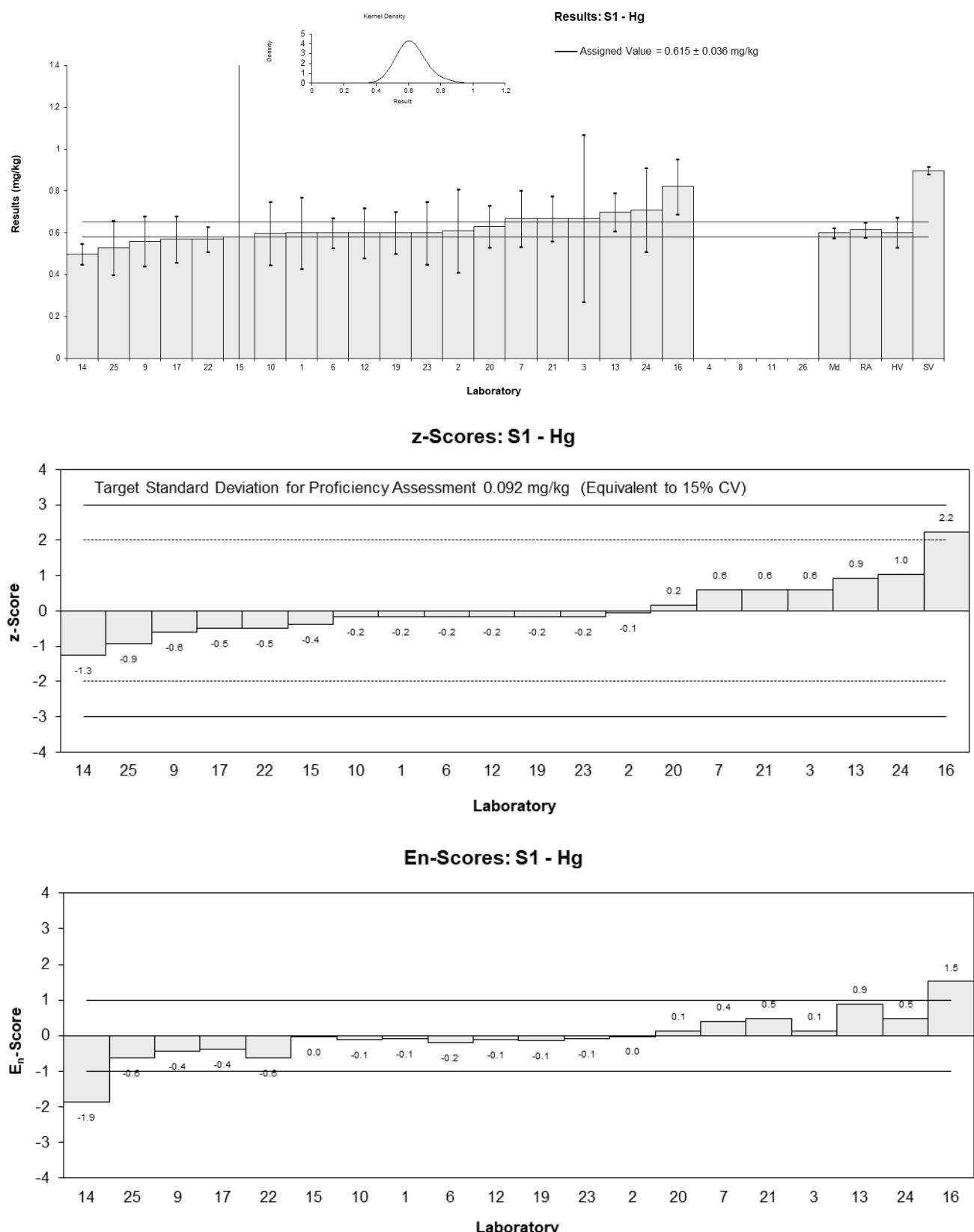


Figure 10

Table 22

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	25.5	11.56	-0.72	-0.26
2	NT	NT		
3	34	0.2	1.26	1.92
4	NT	NT		
6	NT	NT		
7	33.6	6.72	1.17	0.69
8	NR	NR		
9	30	8	0.33	0.17
10	29	8.7	0.09	0.04
11	27	2.7	-0.37	-0.41
12	29.2	3.6	0.14	0.13
13	20.4	NR	-1.91	-2.93
14	26	2.6	-0.61	-0.68
15	26	5.0	-0.61	-0.45
16	26.206	6.212	-0.56	-0.35
17	27	5	-0.37	-0.28
19	18.0	3.8	-2.47	-2.25
20	26.3	3.0	-0.54	-0.56
21	29.96	3.60	0.32	0.30
22	37	4.5	1.96	1.58
23	30.6	7.65	0.47	0.25
24	35.5	15	1.61	0.45
25	NT	NT		
26	NR	NR		

Statistics

Assigned Value	28.6	2.8
Spike Value	Not Spiked	
Homogeneity Value	28.7	3.4
Robust Average	28.6	2.8
Median	28.0	1.7
Mean	28.4	
N	18	
Max	37	
Min	18	
Robust SD	4.8	
Robust CV	17%	

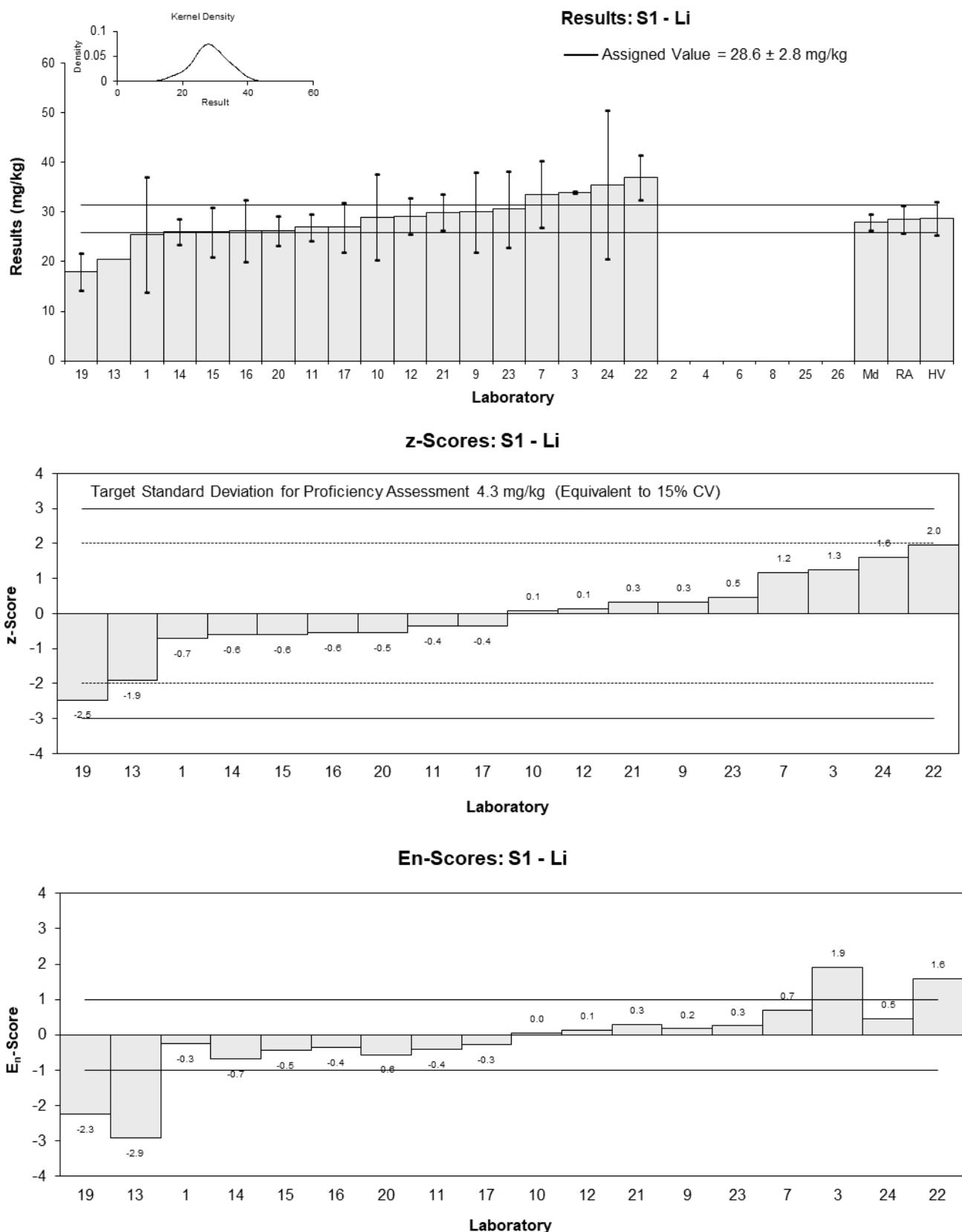


Figure 11

Table 23

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	723	194	0.76	0.26
2	NT	NT		
3	868	0.2	2.92	5.94
4	NT	NT		
6	676	18.428	0.06	0.11
7	681	136	0.13	0.06
8	NR	NR		
9	680	87	0.12	0.09
10	610	92	-0.92	-0.63
11	640	64	-0.48	-0.44
12	734	74	0.92	0.77
13	699	117	0.40	0.22
14	590	59	-1.22	-1.21
15	650	140	-0.33	-0.15
16	632.10	59.699	-0.59	-0.58
17	670	130	-0.03	-0.01
19	539	55	-1.98	-2.07
20	672	70	0.00	0.00
21	702.7	99.2	0.46	0.29
22	650	65	-0.33	-0.30
23	714.5	178.63	0.63	0.23
24	786.0	100	1.70	1.08
25	610	92	-0.92	-0.63
26	NR	NR		

Statistics

Assigned Value	672	33
Spike Value	Not Spiked	
Homogeneity Value	632	76
Robust Average	672	33
Median	674	31
Mean	676	
N	20	
Max	868	
Min	539	
Robust SD	59	
Robust CV	8.8%	

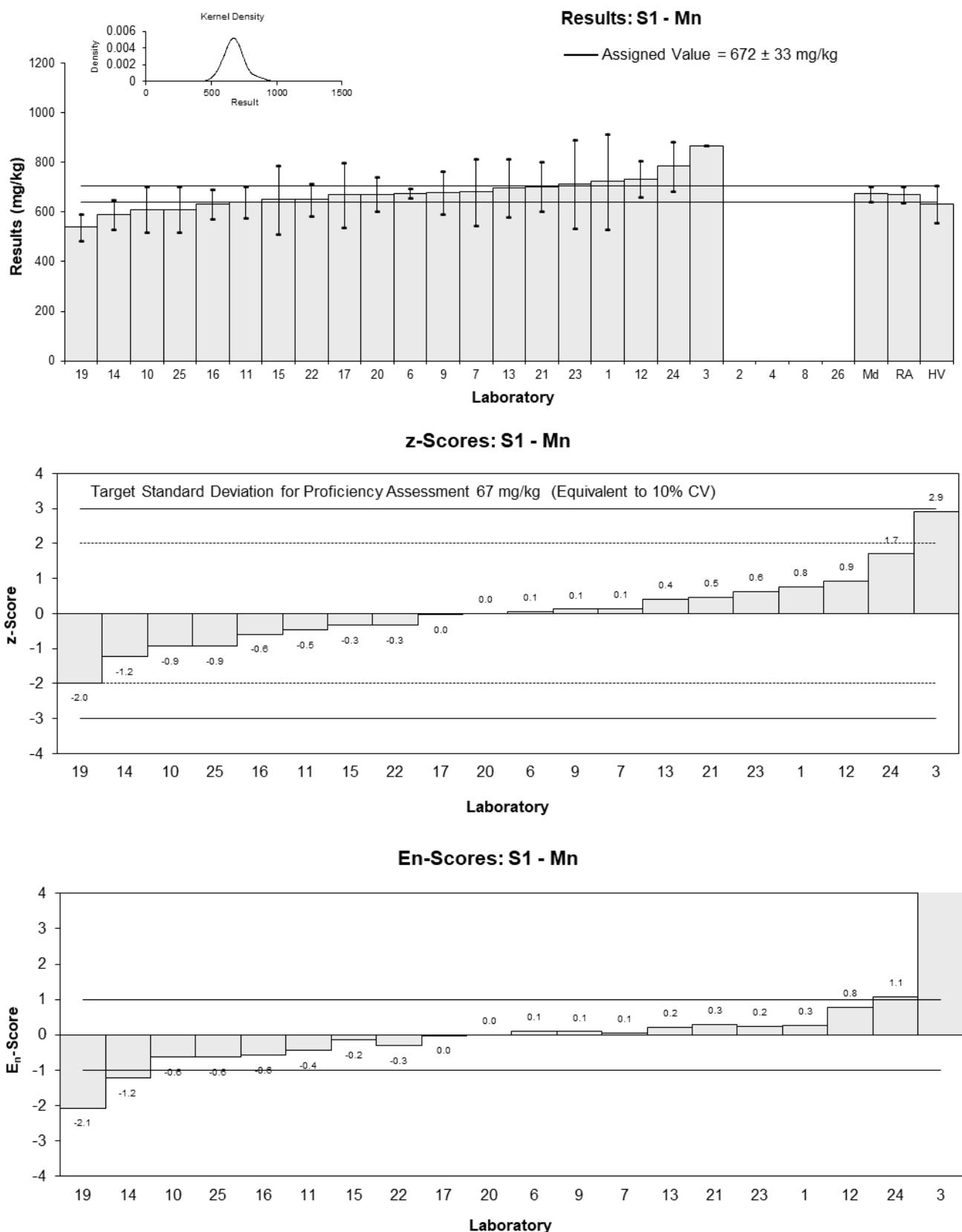


Figure 12

Table 24

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	5.0	1.94	0.11	0.04
2	NT	NT		
3	5.7	0.2	1.06	2.07
4	NT	NT		
6	4	0.647	-1.25	-1.27
7	5.36	1.07	0.60	0.39
8	NR	NR		
9	5	2	0.11	0.04
10	3.89	1.0	-1.40	-0.98
11	4.8	0.48	-0.16	-0.21
12	5.06	0.95	0.19	0.14
13	5.0	1.0	0.11	0.08
14	<5	NR		
15	4.9	0.83	-0.03	-0.02
16	5.128	1.005	0.28	0.20
17	4.7	0.9	-0.30	-0.23
19	4	1	-1.25	-0.88
20	4.83	0.6	-0.12	-0.13
21	5.105	0.977	0.25	0.18
22	NR	NR		
23	5	1.25	0.11	0.06
24	6.16	1.5	1.68	0.81
25	<5.0	NR		
26	NR	NR		

Statistics

Assigned Value	4.92	0.32
Spike Value	5.44	0.11
Homogeneity Value	4.82	0.58
Robust Average	4.92	0.32
Median	5.00	0.15
Mean	4.92	
N	17	
Max	6.16	
Min	3.89	
Robust SD	0.54	
Robust CV	11%	

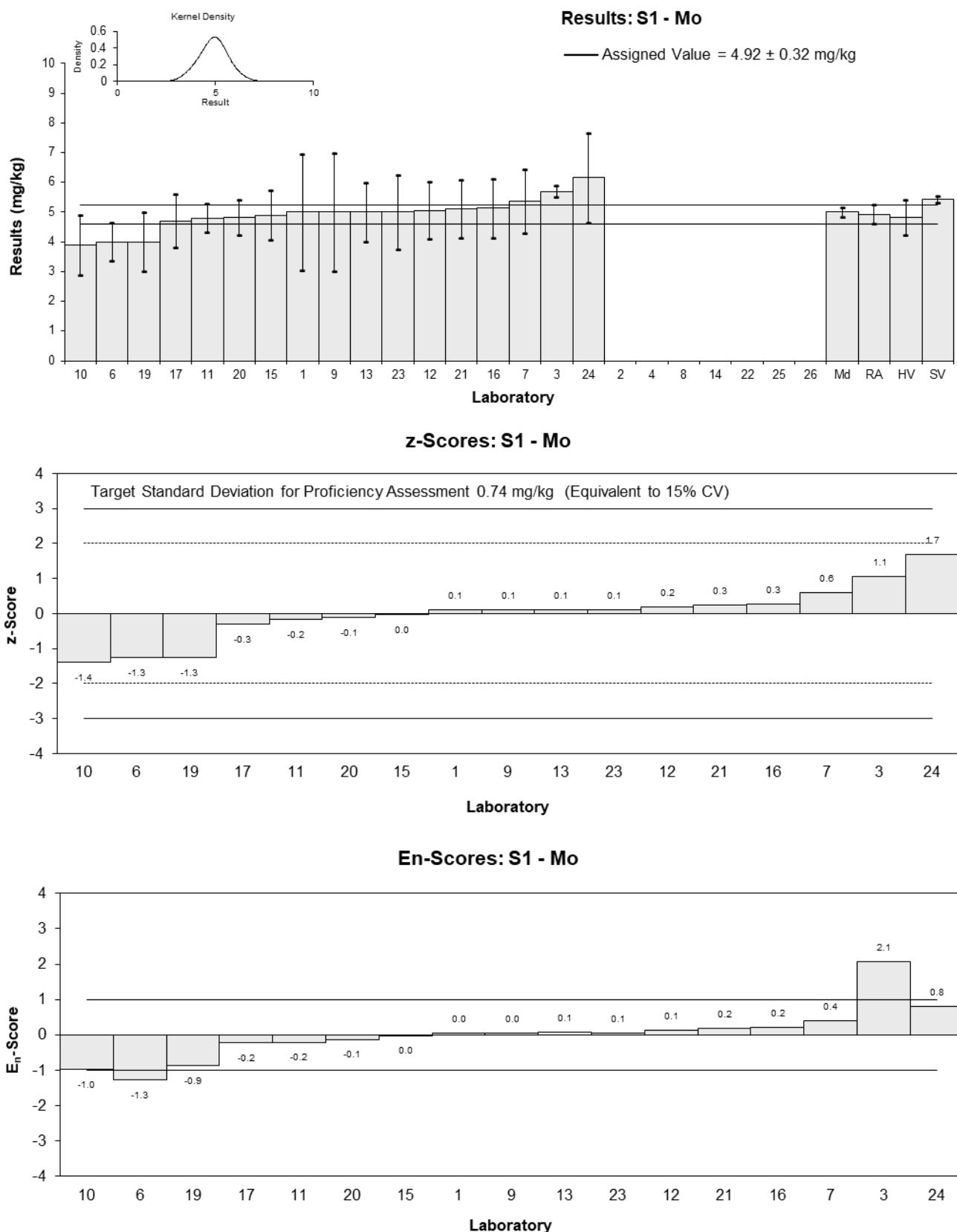


Figure 13

Table 25

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	53.6	13.34	-0.46	-0.19
2	35	11	-3.77	-1.84
3	76	0.2	3.52	5.81
4	NT	NT		
6	52	2.412	-0.75	-1.01
7	59.8	12.0	0.64	0.29
8	NR	NR		
9	59	11	0.50	0.24
10	49.7	6.0	-1.16	-0.94
11	58	5.8	0.32	0.27
12	61.5	8.0	0.94	0.61
13	62.4	12.2	1.10	0.49
14	45	4.5	-1.99	-1.99
15	58	15	0.32	0.12
16	54.831	6.261	-0.24	-0.19
17	61	12	0.85	0.38
19	47	6	-1.64	-1.33
20	57.5	5.8	0.23	0.19
21	59.44	9.67	0.58	0.32
22	NR	NR		
23	58	14.5	0.32	0.12
24	58.0	12	0.32	0.14
25	54	5	-0.39	-0.36
26	NR	NR		

Statistics

Assigned Value	56.2	3.4
Spike Value	Not Spiked	
Homogeneity Value	56.7	6.8
Robust Average	56.2	3.4
Median	58.0	2.8
Mean	56.0	
N	20	
Max	76	
Min	35	
Robust SD	6	
Robust CV	11%	

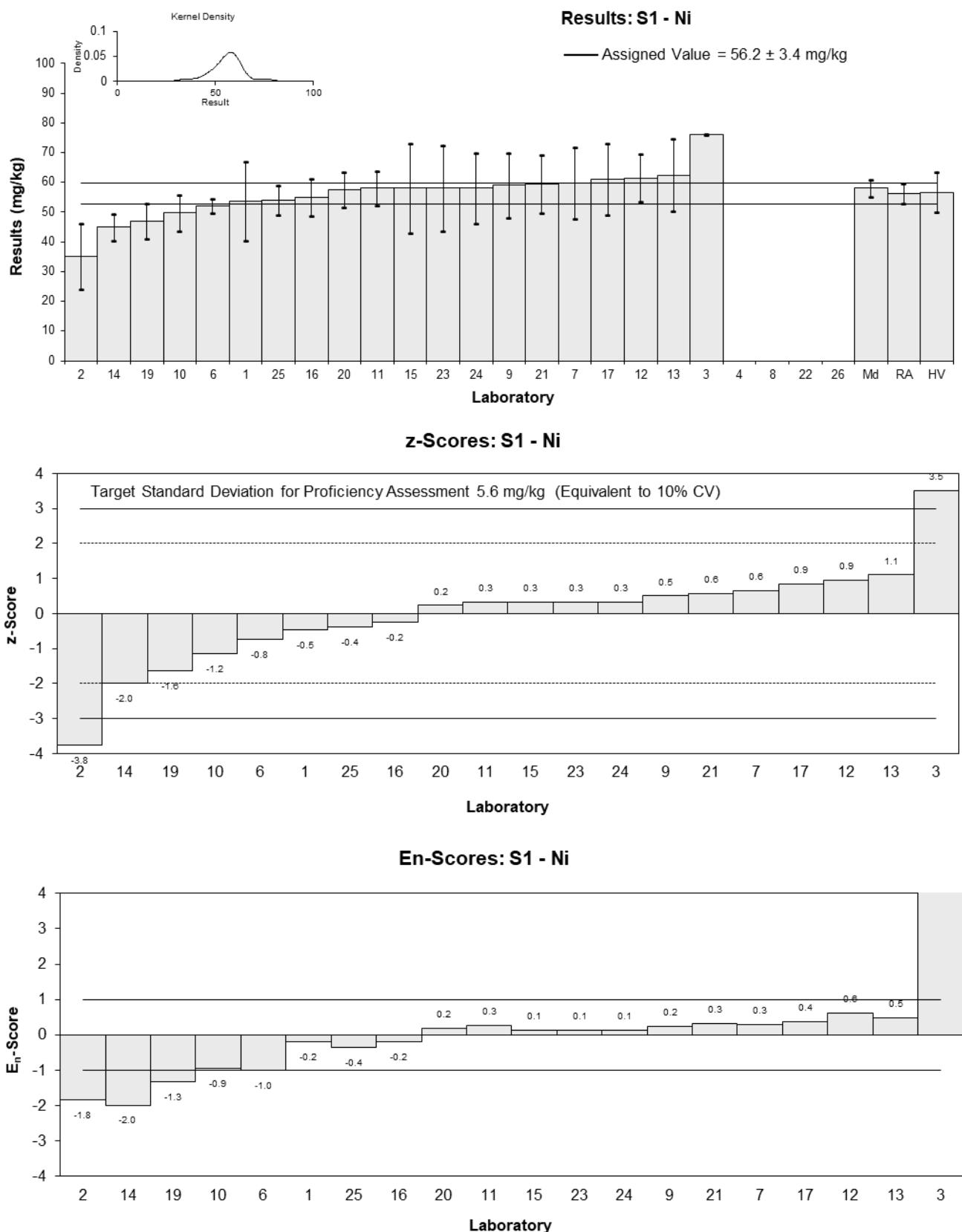


Figure 14

Table 26

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	51.4	9.37	1.00	0.66
2	29	7.1	-2.34	-1.94
3	50	0.2	0.79	1.36
4	NT	NT		
6	50	2.135	0.79	1.19
7	50.2	10.0	0.82	0.51
8	NR	NR		
9	47	8	0.34	0.26
10	39.3	5.0	-0.81	-0.85
11	43	4.3	-0.25	-0.29
12	49.1	7.4	0.66	0.53
13	47.3	7.87	0.39	0.30
14	36	3.6	-1.30	-1.64
15	38	6.1	-1.00	-0.93
16	48.11	7.726	0.51	0.39
17	47	9	0.34	0.23
19	38	5	-1.00	-1.06
20	51.5	5.5	1.01	1.01
21	53.63	9.25	1.33	0.89
22	46	6.4	0.19	0.17
23	48	12	0.49	0.26
24	32.0	8	-1.89	-1.43
25	37	4	-1.15	-1.38
26	NR	NR		

Statistics

Assigned Value	44.7	3.9
Spike Value	Not Spiked	
Homogeneity Value	47.7	5.7
Robust Average	44.7	3.9
Median	47.0	3.2
Mean	44.4	
N	21	
Max	53.63	
Min	29	
Robust SD	7.2	
Robust CV	16%	

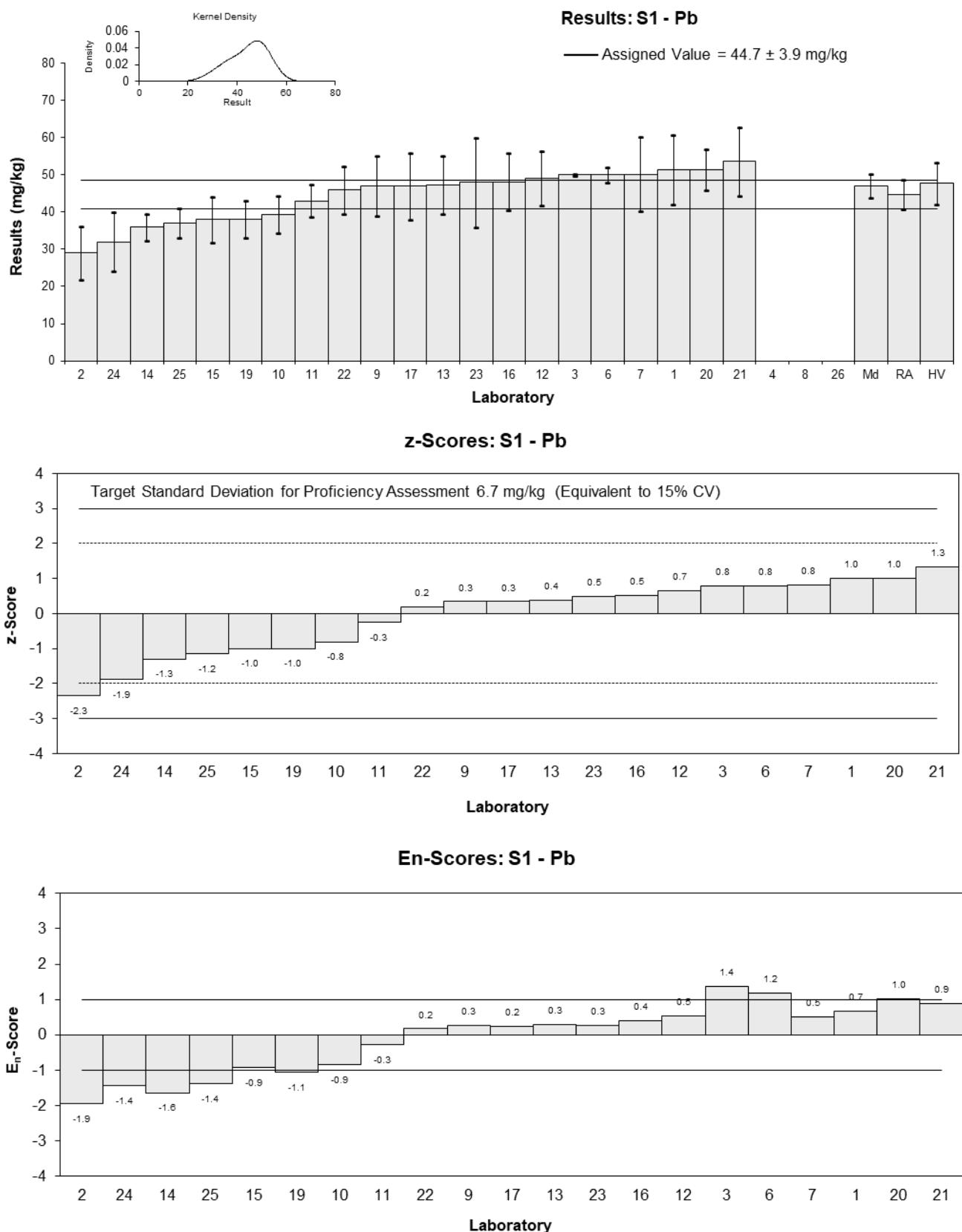


Figure 15

Table 27

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	3.8	1.45
2	NT	NT
3	NT	NT
4	NT	NT
6	NT	NT
7	NT	NT
8	NR	NR
9	NT	NT
10	NR	NR
11	NT	NT
12	4.71	0.55
13	3.7	NR
14	11	1.1
15	3.9	1.2
16	4.490	NR
17	6.2	1.2
19	2.8	0.3
20	NT	NT
21	NT	NT
22	16	1.9
23	NT	NT
24	NR	NR
25	NT	NT
26	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Homogeneity Value	6.98	0.84
Robust Average	5.7	3.0
Median	4.49	0.98
Mean	6.3	
N	9	
Max	16	
Min	2.8	
Robust SD	3.6	
Robust CV	62%	

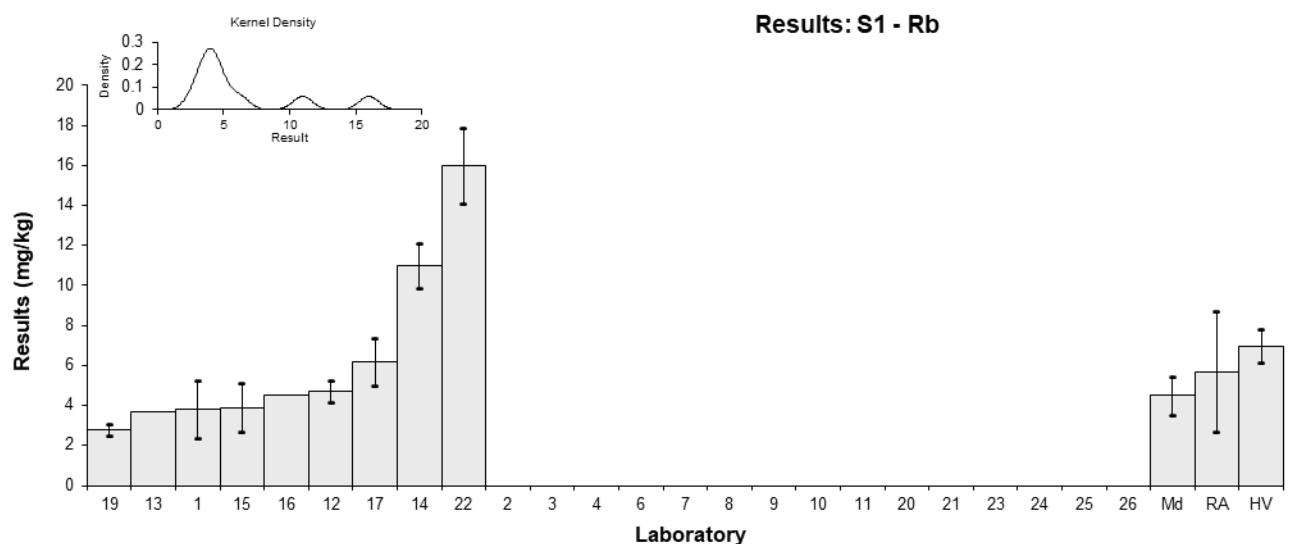


Figure 16

Table 28

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	18.5	7.00
2	NT	NT
3	31.00	0.2
4	NT	NT
6	NT	NT
7	28.8	5.77
8	NR	NR
9	30	6
10	20.5	4
11	NT	NT
12	21.0	3.8
13	9.8	3.4
14	21	2.1
15	22	7.4
16	19.87	4.022
17	13	6
19	8	2
20	36.4	4.0
21	27.76	4.42
22	15	1.2
23	32.7	8.18
24	NR	NR
25	17	NR
26	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	40.0	0.8
Homogeneity Value	22.0	2.6
Robust Average	21.9	5.5
Median	21.0	6.1
Mean	21.9	
N	17	
Max	36.4	
Min	8	
Robust SD	9.1	
Robust CV	42%	

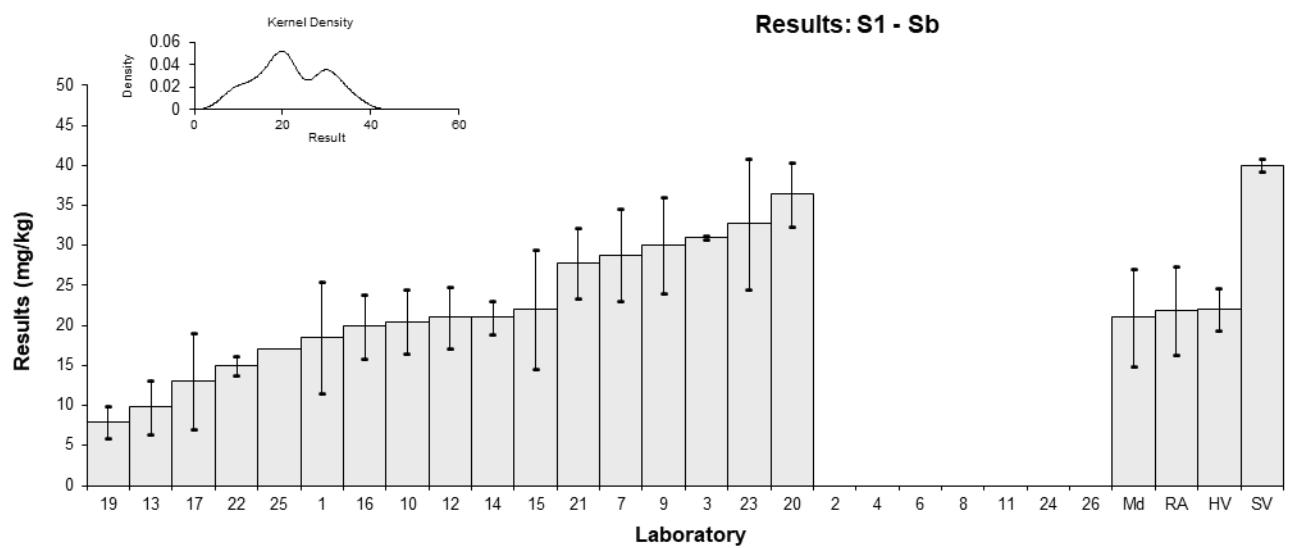


Figure 17

Table 29

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	<1	0.38
2	NT	NT
3	<2	0.2
4	NT	NT
6	0.4	0.01
7	<2	<0.4
8	NR	NR
9	<2	NR
10	3.05	1.0
11	<5	NR
12	0.6	1.3
13	5.0	1.3
14	<5	NR
15	0.6	0.11
16	<1	NR
17	0.59	0.11
19	<5	NR
20	1.15	0.2
21	< 2	NR
22	0.49	0.04
23	< 2	NR
24	NR	NR
25	<5.0	NR
26	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Homogeneity Value	0.617	0.074
Robust Average	1.3	1.2
Median	0.60	0.20
Mean	1.5	
N	8	
Max	5	
Min	0.4	
Robust SD	1.4	
Robust CV	110%	

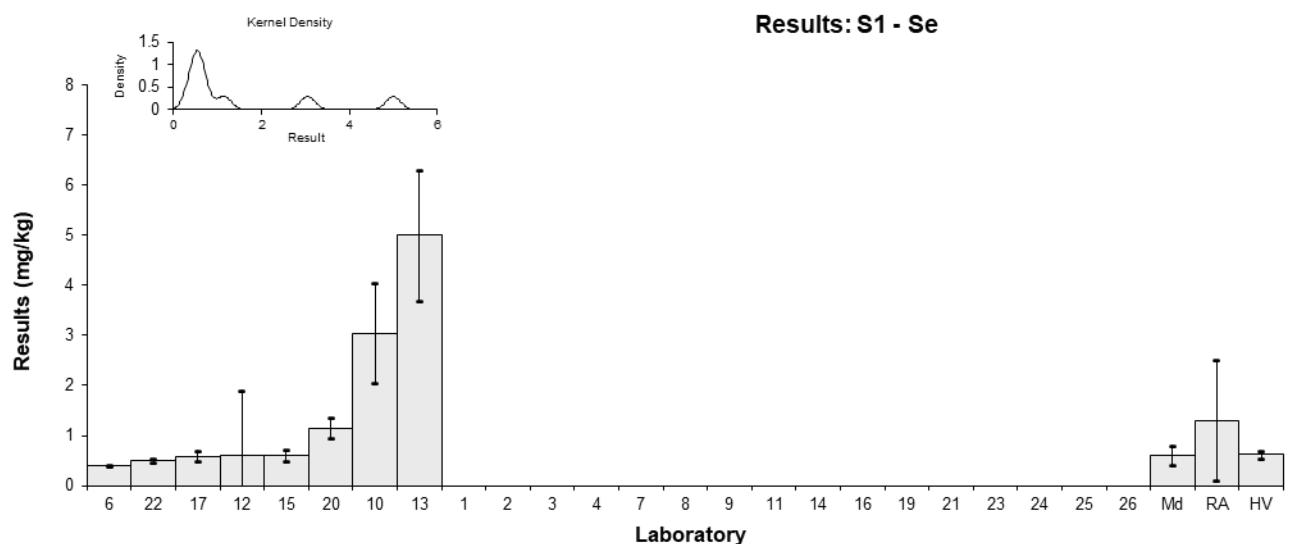


Figure 18

Table 30

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	5.8	2.19	0.28	0.07
2	NT	NT		
3	<10	0.3		
4	NT	NT		
6	NT	NT		
7	<10	<2		
8	NR	NR		
9	<10	NR		
10	5.6	3.0	-0.07	-0.01
11	6.3	0.63	1.17	0.87
12	5.8	1.4	0.28	0.11
13	5.1	0.83	-0.96	-0.58
14	5	0.5	-1.13	-0.97
15	5.3	1.9	-0.60	-0.17
16	6.149	1.483	0.90	0.33
17	5.2	1.0	-0.78	-0.40
19	<5	NR		
20	NT	NT		
21	< 10	NR		
22	6.2	0.8	0.99	0.62
23	< 10	NR		
24	NR	NR		
25	<5.0	NR		
26	NR	NR		

Statistics

Assigned Value	5.64	0.43
Spike Value	6.36	0.13
Homogeneity Value	5.47	0.66
Robust Average	5.64	0.43
Median	5.70	0.56
Mean	5.64	
N	10	
Max	6.3	
Min	5	
Robust SD	0.54	
Robust CV	9.6%	

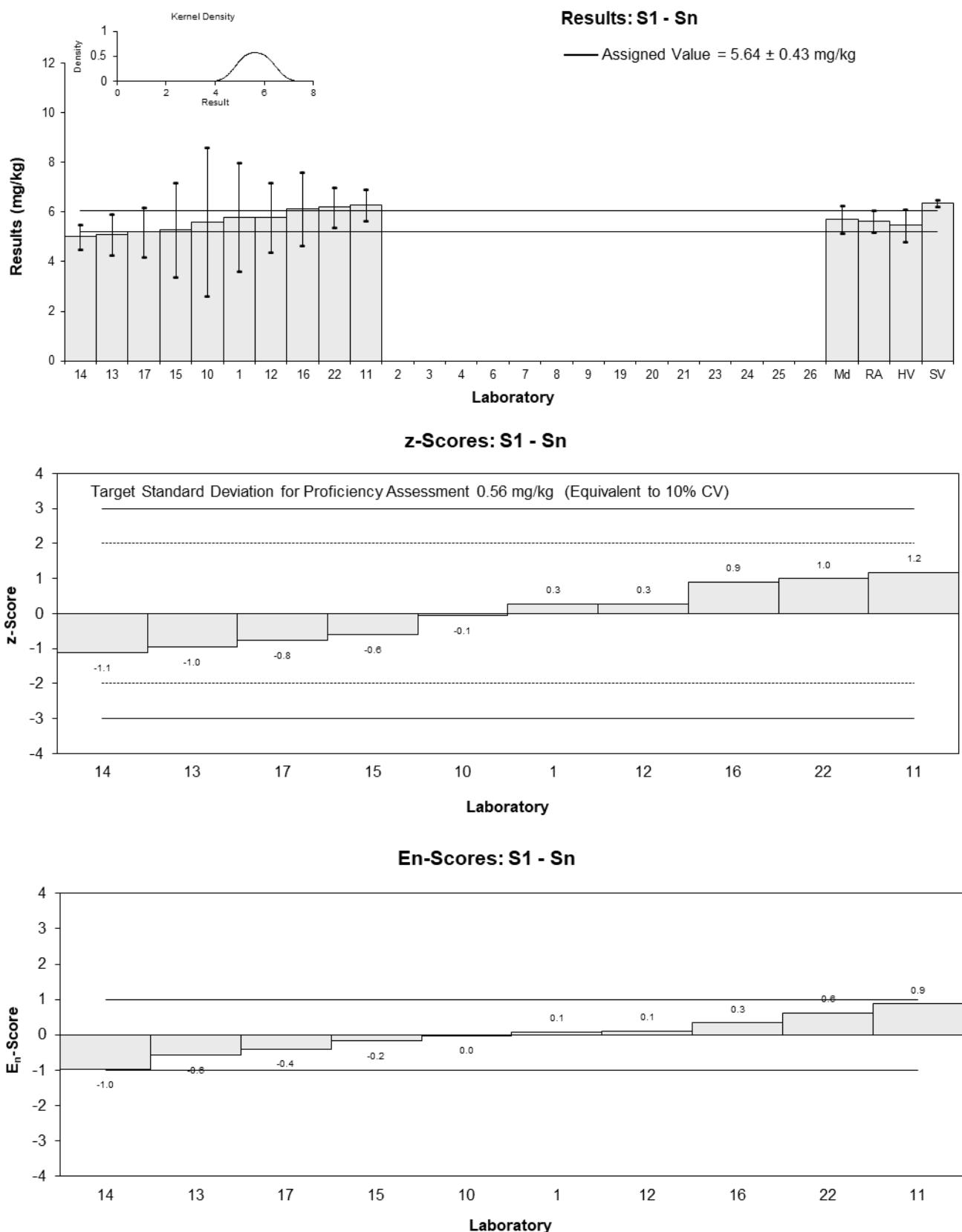


Figure 19

Table 31

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	122	29.14	-0.24	-0.10
2	NT	NT		
3	140	0.2	1.20	2.14
4	NT	NT		
6	NT	NT		
7	131	26.2	0.48	0.22
8	NR	NR		
9	130	26	0.40	0.19
10	114	30	-0.88	-0.36
11	120	12.0	-0.40	-0.36
12	138	14	1.04	0.83
13	122	22.6	-0.24	-0.13
14	110	11	-1.20	-1.15
15	120	22	-0.40	-0.22
16	131.3	14.88	0.50	0.38
17	140	30	1.20	0.49
19	98	13	-2.16	-1.83
20	111	15	-1.12	-0.85
21	132.6	19.5	0.61	0.37
22	109	11	-1.28	-1.23
23	133.1	33.28	0.65	0.24
24*	265.2	40	11.22	3.45
25	131.5	NR	0.52	0.93
26	NR	NR		

* Outlier, see Section 4.2

Statistics

Assigned Value	125	7
Spike Value	Not Spiked	
Homogeneity Value	110	13
Robust Average	126	8
Median	130	9
Mean	132	
N	19	
Max	265.2	
Min	98	
Robust SD	14	
Robust CV	11%	

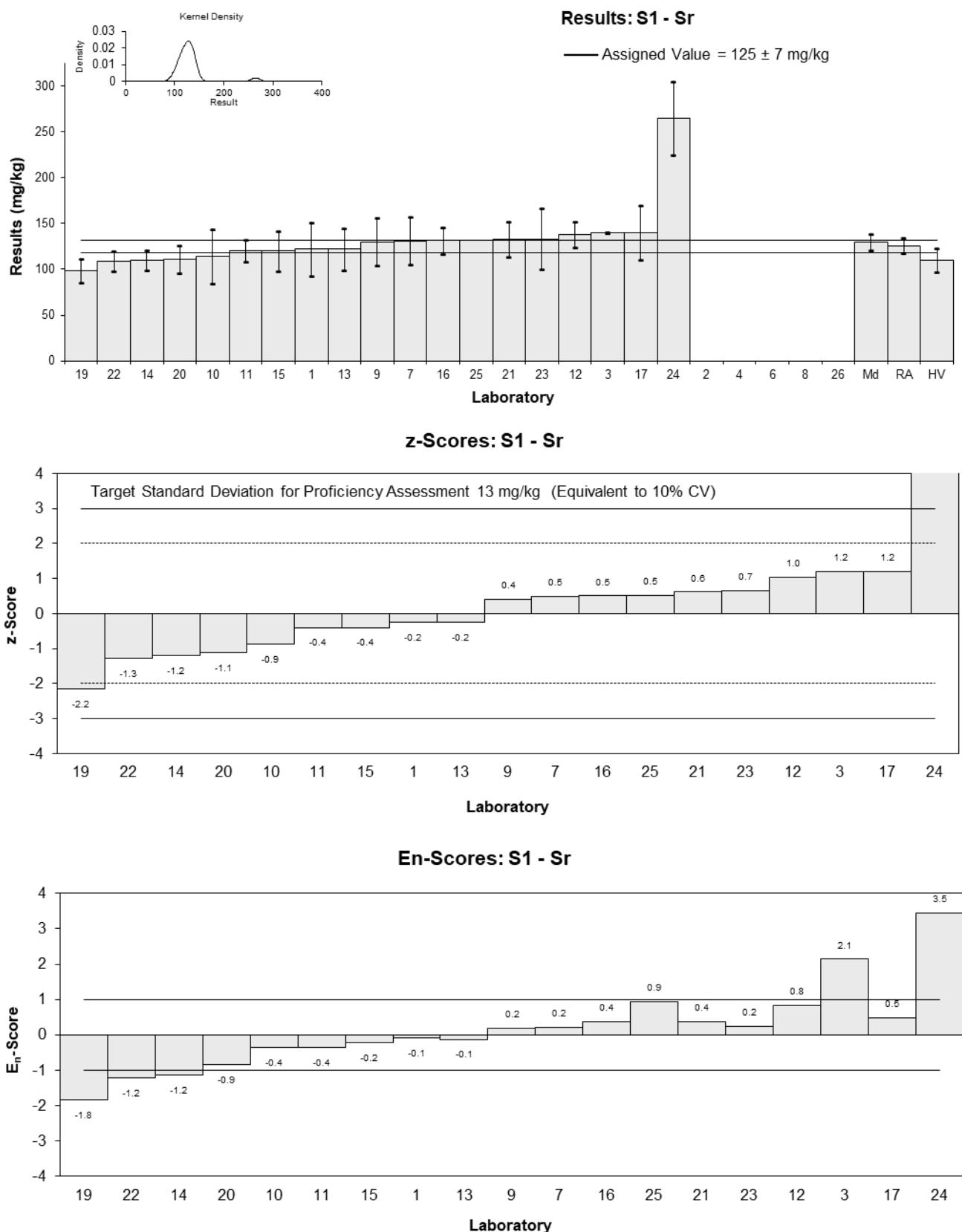


Figure 20

Table 32

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	2	0.92	-0.47	-0.16
2	NT	NT		
3	NT	NT		
4	NT	NT		
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	2.1	1	-0.16	-0.05
10	NR	NR		
11	NT	NT		
12	NT	NT		
13	2.0	NR	-0.47	-0.50
14*	5	0.5	8.84	4.89
15	NT	NT		
16	2.495	0.477	1.07	0.61
17	2.1	0.4	-0.16	-0.10
19	1.7	0.5	-1.40	-0.77
20	2.14	0.4	-0.03	-0.02
21	NT	NT		
22	2.8	0.33	2.02	1.46
23	NT	NT		
24	NR	NR		
25	NT	NT		
26	NR	NR		

* Outlier, see Section 4.2

Statistics

Assigned Value	2.15	0.30
Spike Value	Not Spiked	
Homogeneity Value	2.30	0.28
Robust Average	2.25	0.39
Median	2.10	0.12
Mean	2.48	
N	9	
Max	5	
Min	1.7	
Robust SD	0.46	
Robust CV	21%	

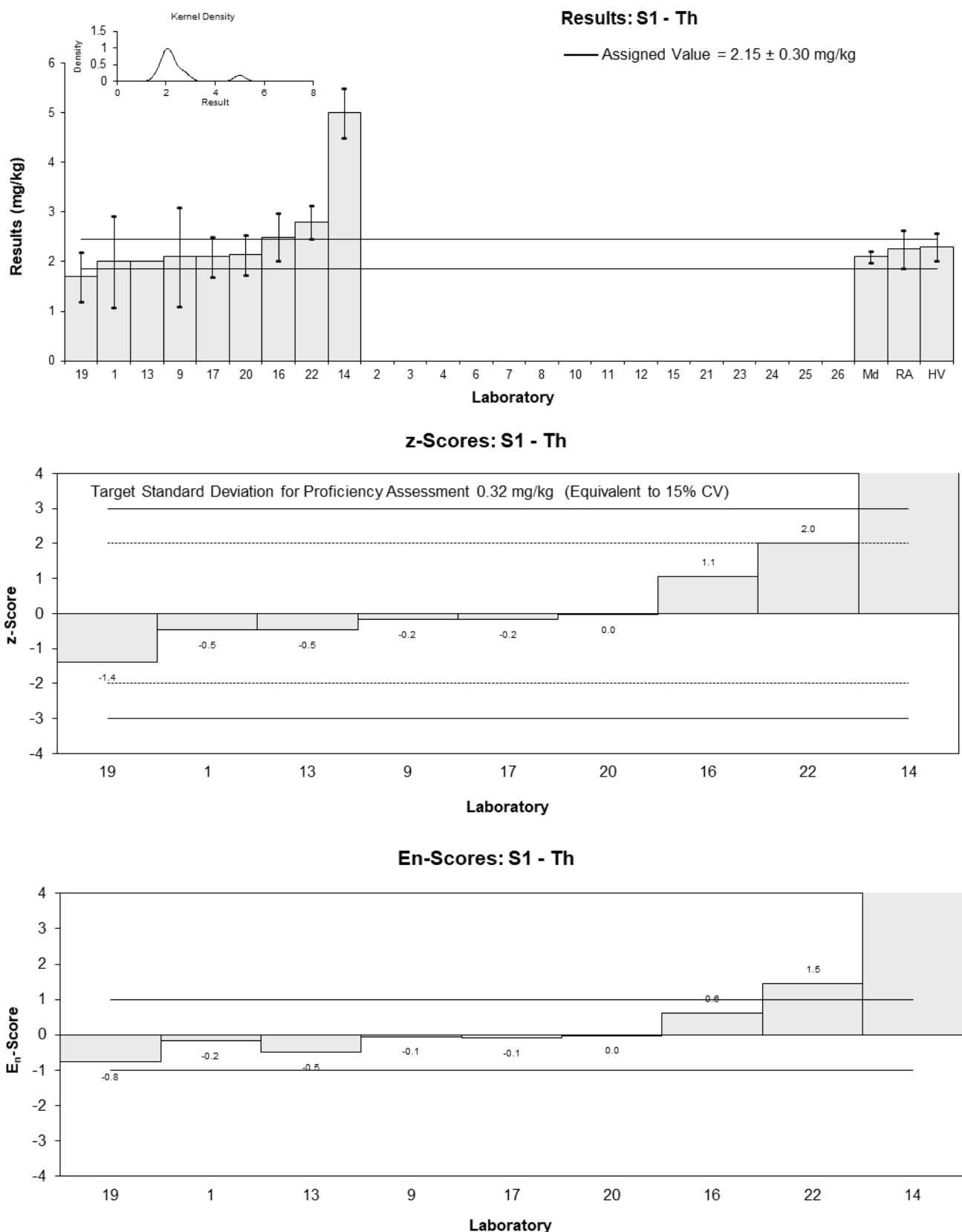


Figure 21

Table 33

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	43	12.08	-0.80	-0.46
2	NT	NT		
3	60	0.2	1.51	2.36
4	NT	NT		
6	48	0.549	-0.12	-0.19
7	56.9	11.4	1.09	0.65
8	NR	NR		
9	49	9	0.01	0.01
10	40.4	12	-1.16	-0.66
11	47	4.7	-0.26	-0.29
12	44	11	-0.67	-0.41
13	48	8.96	-0.12	-0.09
14	49	4.9	0.01	0.01
15	42	11	-0.94	-0.58
16	45.285	5.342	-0.49	-0.51
17	52	10	0.42	0.28
19	32	45	-2.30	-0.37
20	50.3	5.5	0.19	0.19
21	55.21	8.59	0.86	0.64
22	65	6.9	2.19	1.93
23	55.3	13.83	0.87	0.44
24	34.0	10	-2.03	-1.35
25	59	NR	1.38	2.15
26	NR	NR		

Statistics

Assigned Value	48.9	4.7
Spike Value	Not Spiked	
Homogeneity Value	48.2	5.8
Robust Average	48.9	4.7
Median	48.5	5.0
Mean	48.8	
N	20	
Max	65	
Min	32	
Robust SD	8.5	
Robust CV	17%	

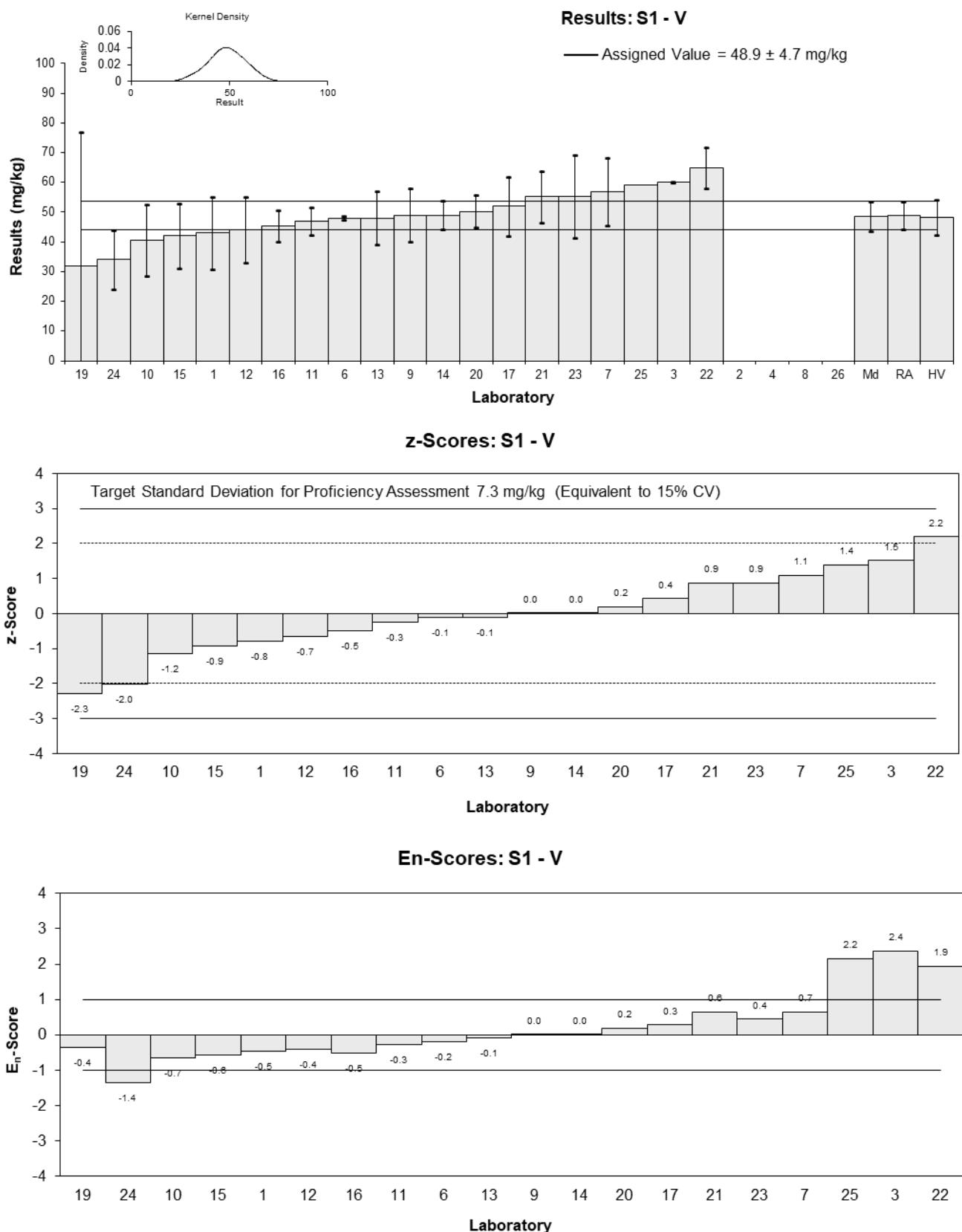


Figure 22

Table 34

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	170	60.58	-1.11	-0.54
2	220	49	0.52	0.31
3	264	0.2	1.96	3.53
4	NT	NT		
6	190	2.581	-0.46	-0.81
7	215.9	43.19	0.39	0.26
8	NR	NR		
9	190	36	-0.46	-0.35
10	184	25	-0.65	-0.66
11	200	20	-0.13	-0.15
12	247	18	1.41	1.74
13	207	32.1	0.10	0.08
14	140	14	-2.09	-2.91
15	190	28	-0.46	-0.43
16	254.8	29.59	1.66	1.49
17	210	40	0.20	0.14
19	170	17	-1.11	-1.41
20	182	20	-0.72	-0.84
21	231.0	36.7	0.88	0.67
22	193	27	-0.36	-0.34
23	207.3	51.83	0.11	0.06
24	235.0	50	1.01	0.59
25	180	29	-0.78	-0.71
26	NR	NR		

Statistics

Assigned Value	204	17
Spike Value	Not Spiked	
Homogeneity Value	194	23
Robust Average	204	17
Median	200	15
Mean	204	
N	21	
Max	264	
Min	140	
Robust SD	31	
Robust CV	15%	

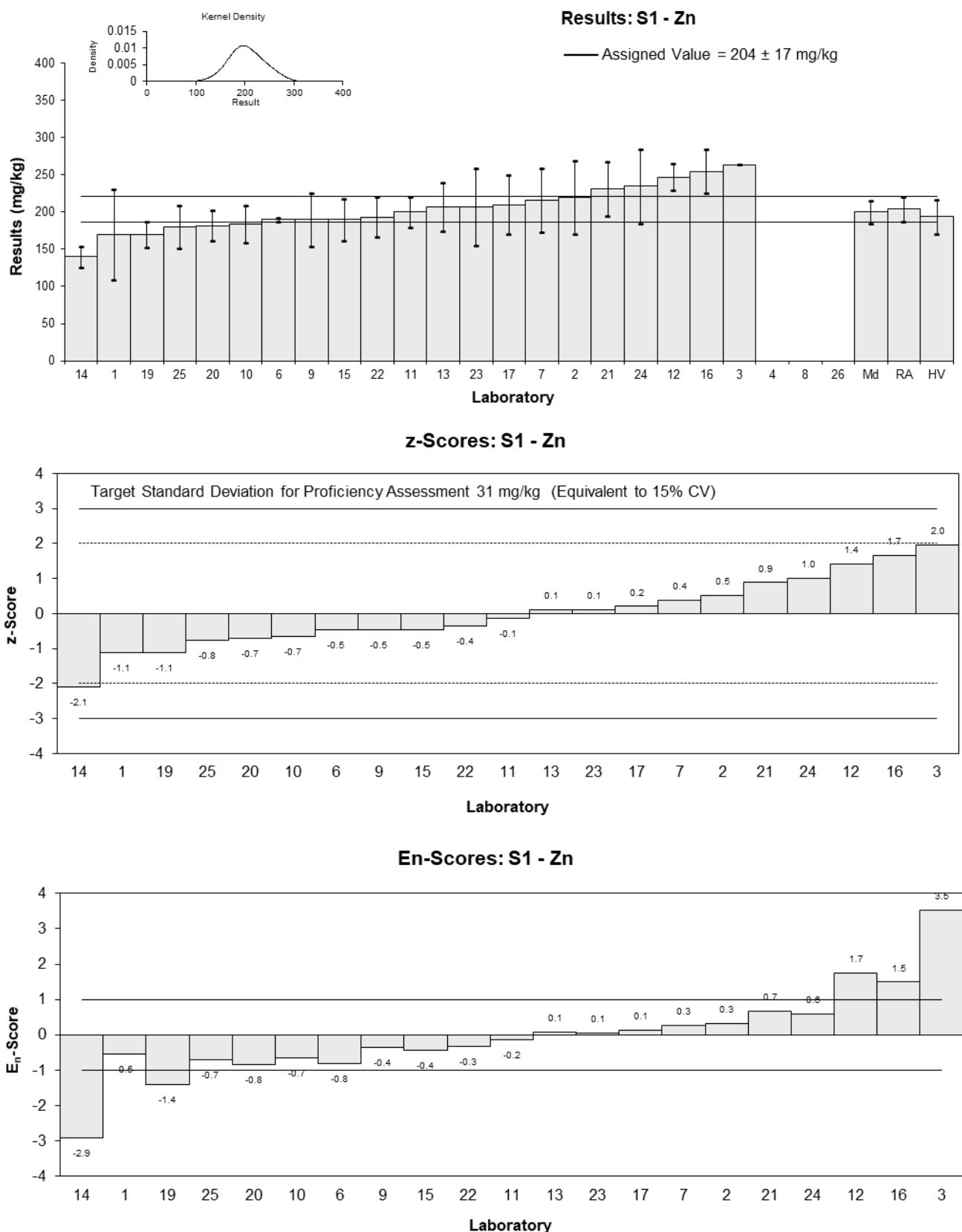


Figure 23

Table 35

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Ag
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	<2	0.23		
2	NT	NT		
3	1	0.2	0.00	0.00
4	NT	NT		
6	NT	NT		
7	<2	<0.4		
8	NR	NR		
9	< 2	NR		
10	1.05	0.3	0.33	0.16
11	<1	NR		
12	NT	NT		
13	0.8	0.2	-1.33	-0.97
14	<5	NR		
15**	2.5	0.89	10.00	1.68
16	<2	NR		
17	1.0	0.2	0.00	0.00
19	1.0	0.2	0.00	0.00
20	1.04	0.2	0.27	0.19
21	< 2	NR		
22	NT	NT		
23	< 2	NR		
24	NR	NR		
25	<5.0	NR		
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	1.00	0.05
Spike Value	Not Spiked	
Homogeneity Value	1.04	0.12
Robust Average	1.00	0.05
Median	1.00	0.03
Mean	0.982	
N	6	
Max	1.05	
Min	0.8	
Robust SD	0.046	
Robust CV	4.5%	

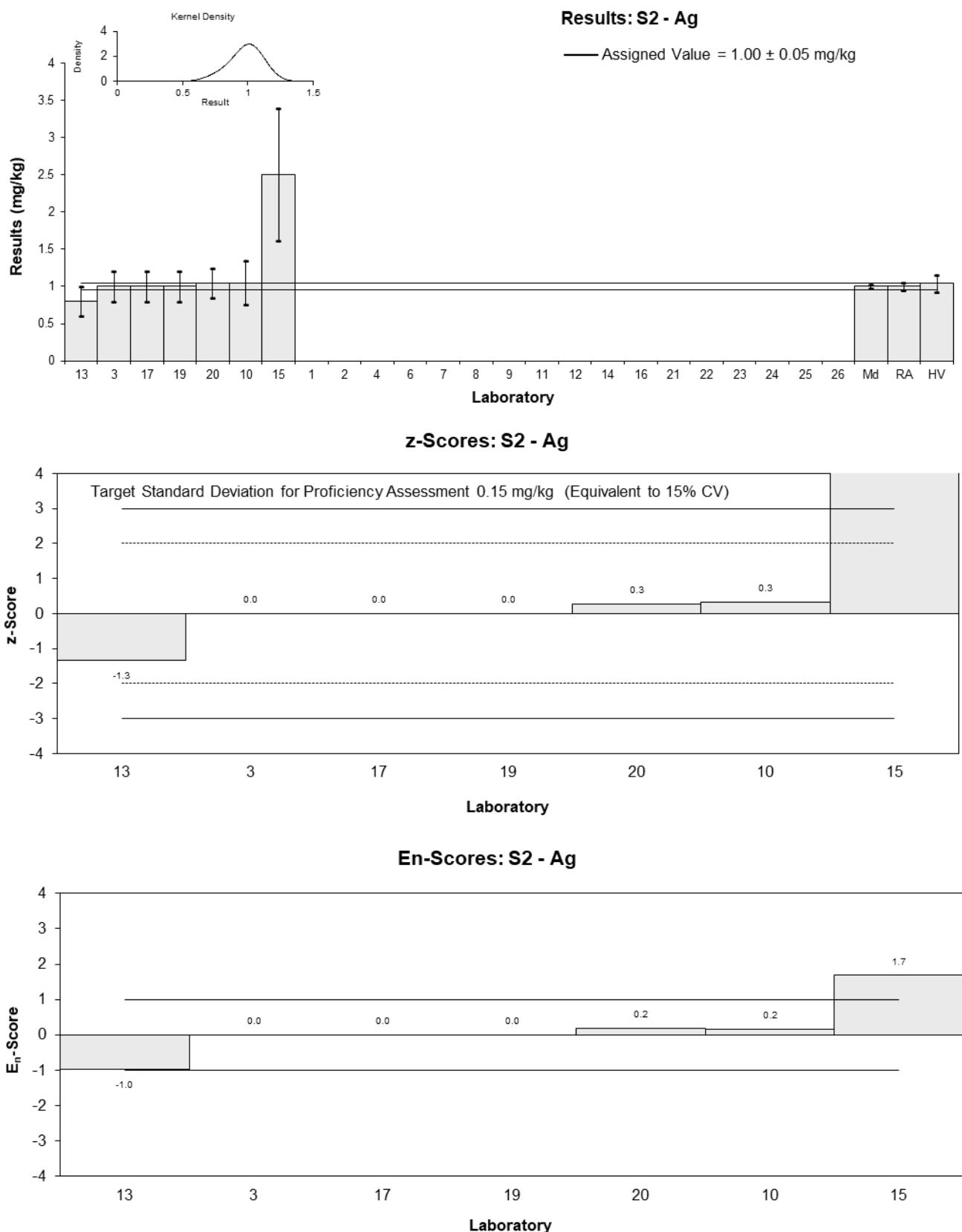


Figure 24

Table 36

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Al
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	12100	2140	-0.94	-0.89
2	NT	NT		
3	17000	0.2	0.70	0.91
4	NT	NT		
6	11520	1357.584	-1.13	-1.27
7	19900	3980	1.68	1.09
8	NR	NR		
9	14000	2800	-0.30	-0.25
10	10600	1900	-1.44	-1.44
11	14100	1410	-0.27	-0.30
12	NT	NT		
13	11500	2484	-1.14	-1.00
14	18000	1800	1.04	1.06
15**	29000	9800	4.73	1.40
16	13037	1785	-0.63	-0.64
17	15800	3200	0.30	0.23
19	9510	1090	-1.81	-2.12
20	14232	1400	-0.22	-0.25
21	15452	2622	0.19	0.16
22	NT	NT		
23	19000	4750	1.38	0.78
24	16750	2000	0.62	0.61
25	22000	4400	2.38	1.43
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	14900	2300
Spike Value	Not Spiked	
Homogeneity Value	13400	1600
Robust Average	14900	2300
Median	14200	2400
Mean	15000	
N	17	
Max	22000	
Min	9510	
Robust SD	3800	
Robust CV	25%	

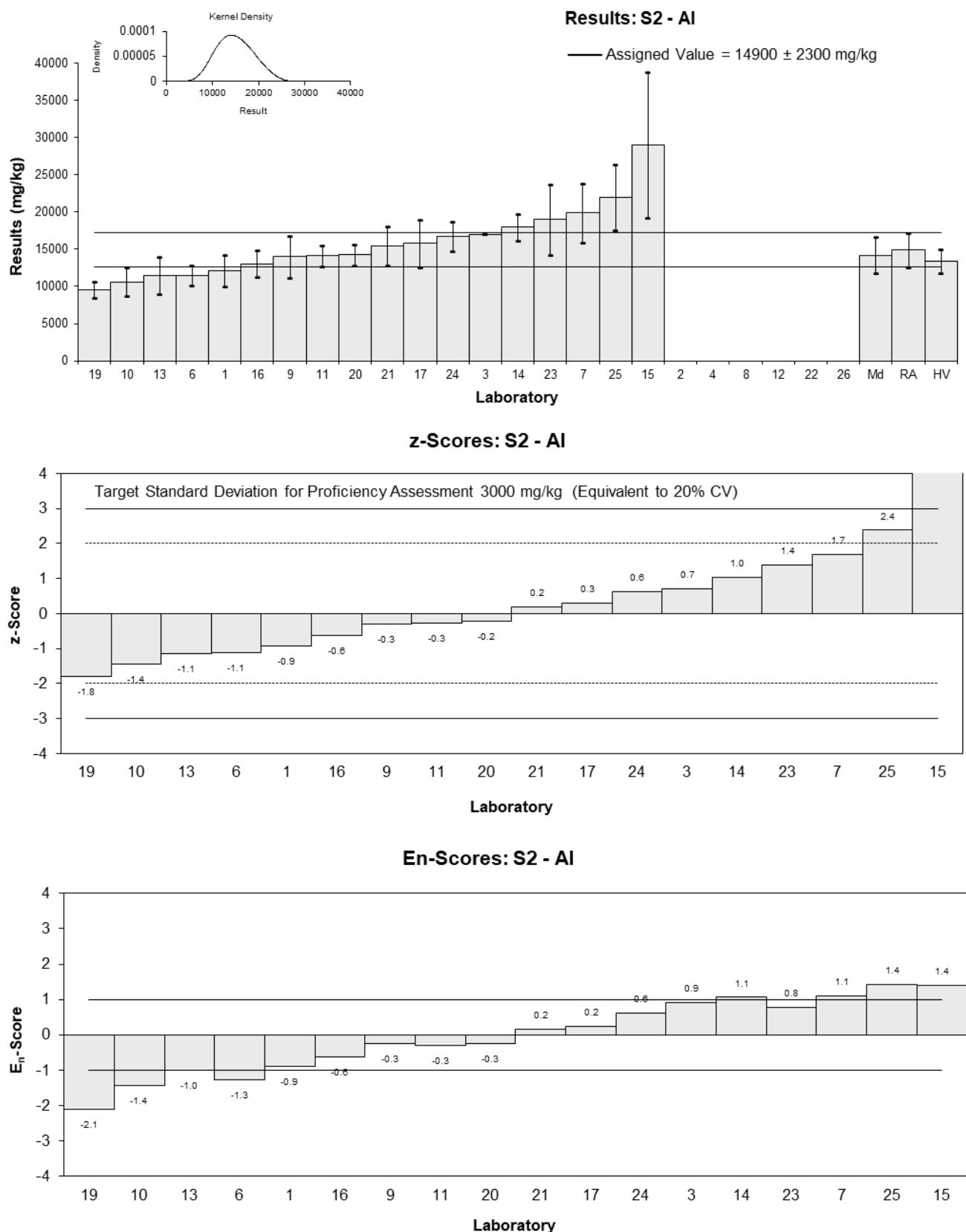


Figure 25

Table 37

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	As
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	10.4	2.16	-1.68	-0.88
2	NT	NT		
3	14	0.2	1.20	1.47
4	NT	NT		
6	11	0.935	-1.20	-1.10
7	12.7	2.55	0.16	0.07
8	NR	NR		
9	14	3	1.20	0.47
10	12.2	2.0	-0.24	-0.13
11	14	0.70	1.20	1.23
12	NT	NT		
13	10.4	1.7	-1.68	-1.06
14	11	1.1	-1.20	-1.01
15**	25	3.8	10.00	3.18
16	12.297	1.617	-0.16	-0.11
17	12	2	-0.40	-0.22
19	12	2	-0.40	-0.22
20	11.8	1.5	-0.56	-0.39
21	14.92	2.33	1.94	0.95
22	NT	NT		
23	14	3.5	1.20	0.41
24	<15	NR		
25	14	2	1.20	0.67
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	12.5	1.0
Spike Value	Not Spiked	
Homogeneity Value	12.5	1.5
Robust Average	12.5	1.0
Median	12.2	1.4
Mean	12.5	
N	16	
Max	14.92	
Min	10.4	
Robust SD	1.6	
Robust CV	13%	

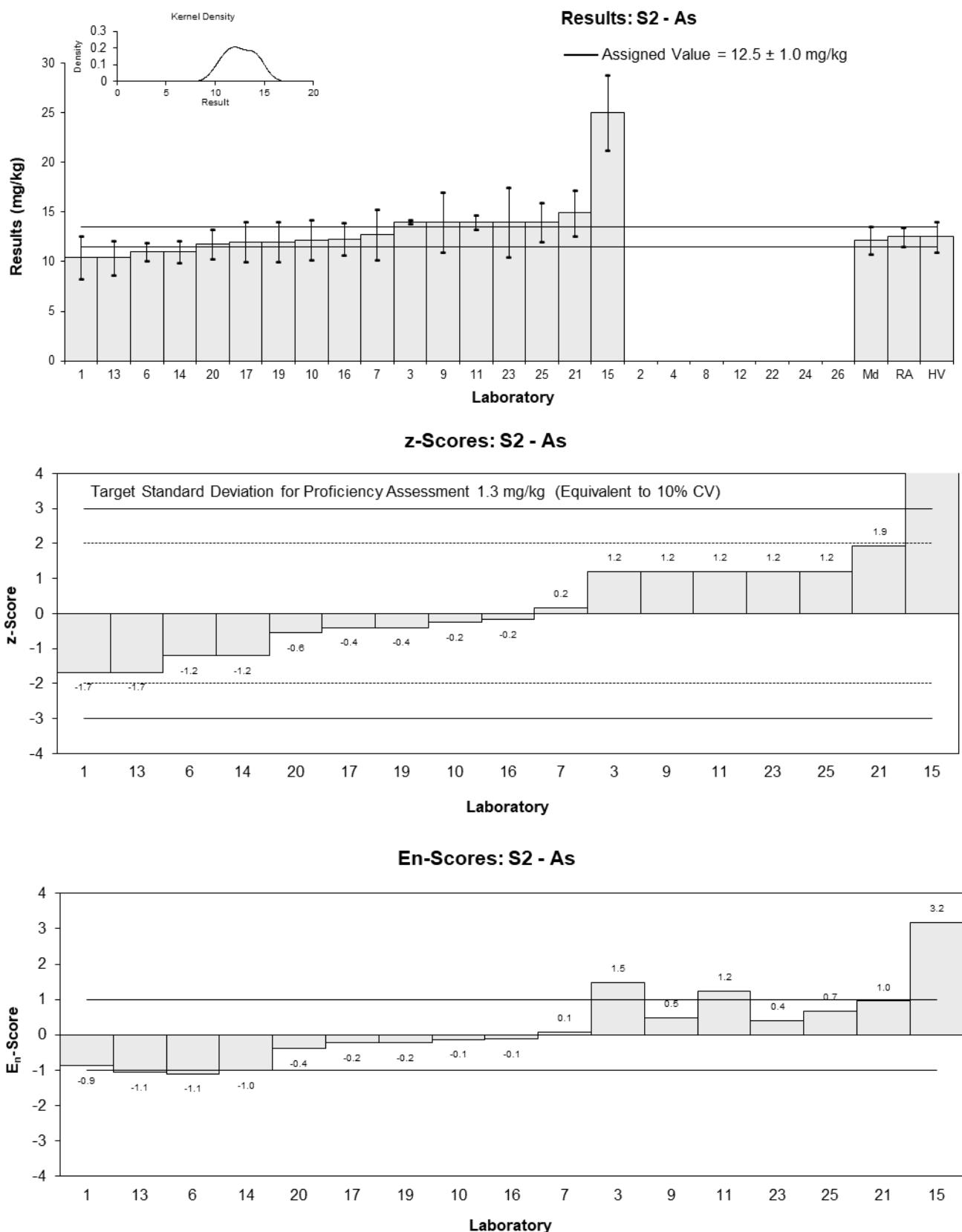


Figure 26

Table 38

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Ba
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	95.9	22.80	-0.95	-0.42
2	NT	NT		
3	120	0.2	1.32	2.00
4	NT	NT		
6	108	3.938	0.19	0.25
7	118	23.6	1.13	0.49
8	NR	NR		
9	110	22	0.38	0.17
10	99.3	24	-0.63	-0.27
11	92	9.2	-1.32	-1.21
12	NT	NT		
13	90.5	16.0	-1.46	-0.89
14	100	10	-0.57	-0.49
15**	240	58	12.64	2.29
16	100.98	11.42	-0.47	-0.37
17	110	20	0.38	0.19
19	90	11	-1.51	-1.23
20	107	10	0.09	0.08
21	113.2	16.8	0.68	0.40
22	NT	NT		
23	110	27.5	0.38	0.14
24	113.7	30	0.73	0.25
25	120	24	1.32	0.56
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	106	7
Spike Value	Not Spiked	
Homogeneity Value	102	12
Robust Average	106	7
Median	108	7
Mean	106	
N	17	
Max	120	
Min	90	
Robust SD	11	
Robust CV	11%	

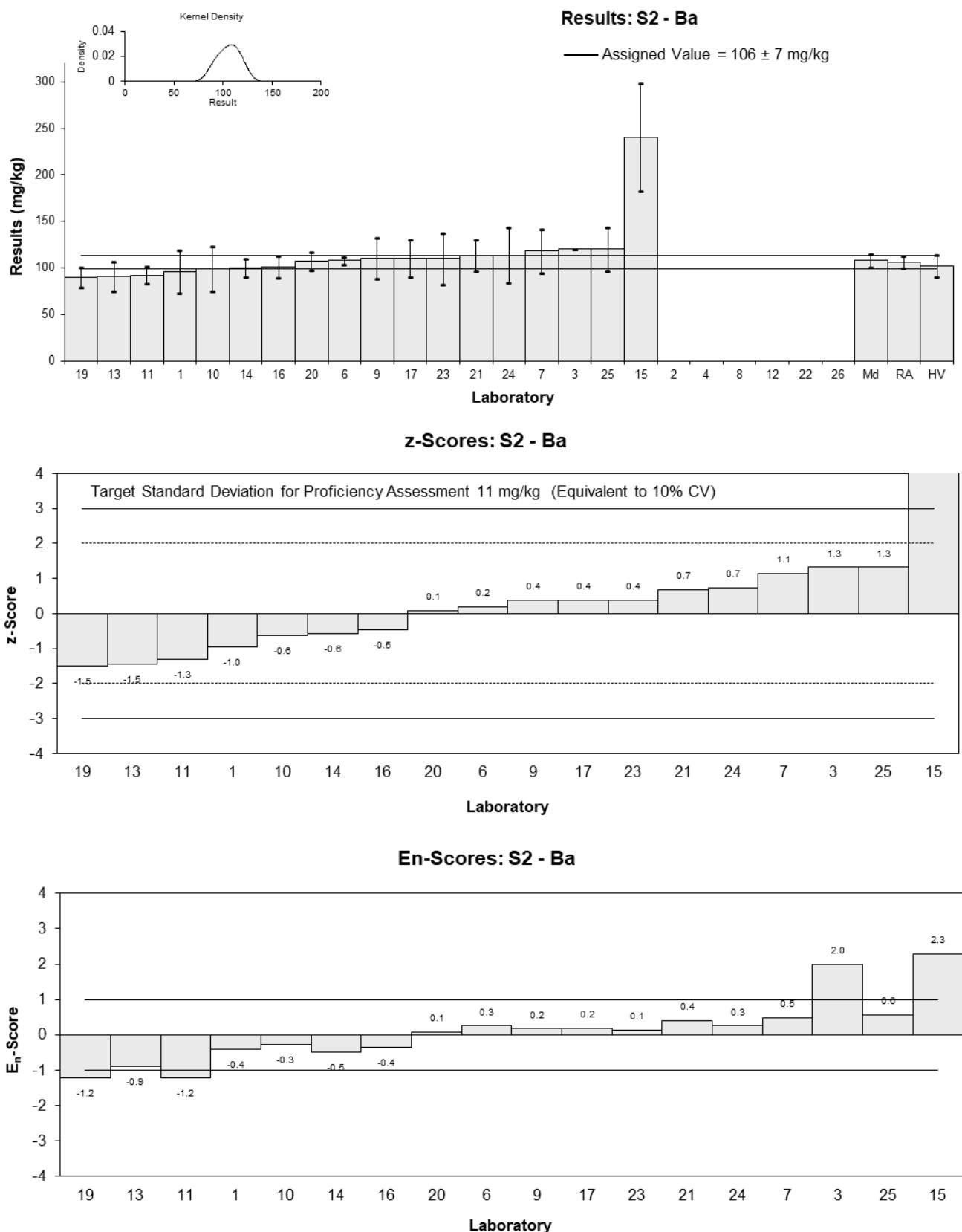


Figure 27

Table 39

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Bi
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	2.9	1.26	0.07	0.02
2	NT	NT		
3	<10	0.2		
4	NT	NT		
6	NT	NT		
7	<10	<2		
8	NR	NR		
9	< 10	NR		
10	3.07	1.1	0.66	0.17
11	NT	NT		
12	NT	NT		
13	2.6	NR	-0.97	-1.17
14	3	0.3	0.42	0.31
15**	7.1	2.2	14.65	1.91
16	3.225	0.930	1.20	0.36
17	3.0	0.6	0.42	0.19
19	2.7	0.8	-0.62	-0.22
20	2.57	0.4	-1.08	-0.66
21	< 10	NR		
22	NT	NT		
23	< 10	NR		
24	NR	NR		
25	NT	NT		
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	2.88	0.24
Spike Value	Not Spiked	
Homogeneity Value	3.11	0.37
Robust Average	2.88	0.24
Median	2.95	0.24
Mean	2.88	
N	8	
Max	3.225	
Min	2.57	
Robust SD	0.27	
Robust CV	9.3%	

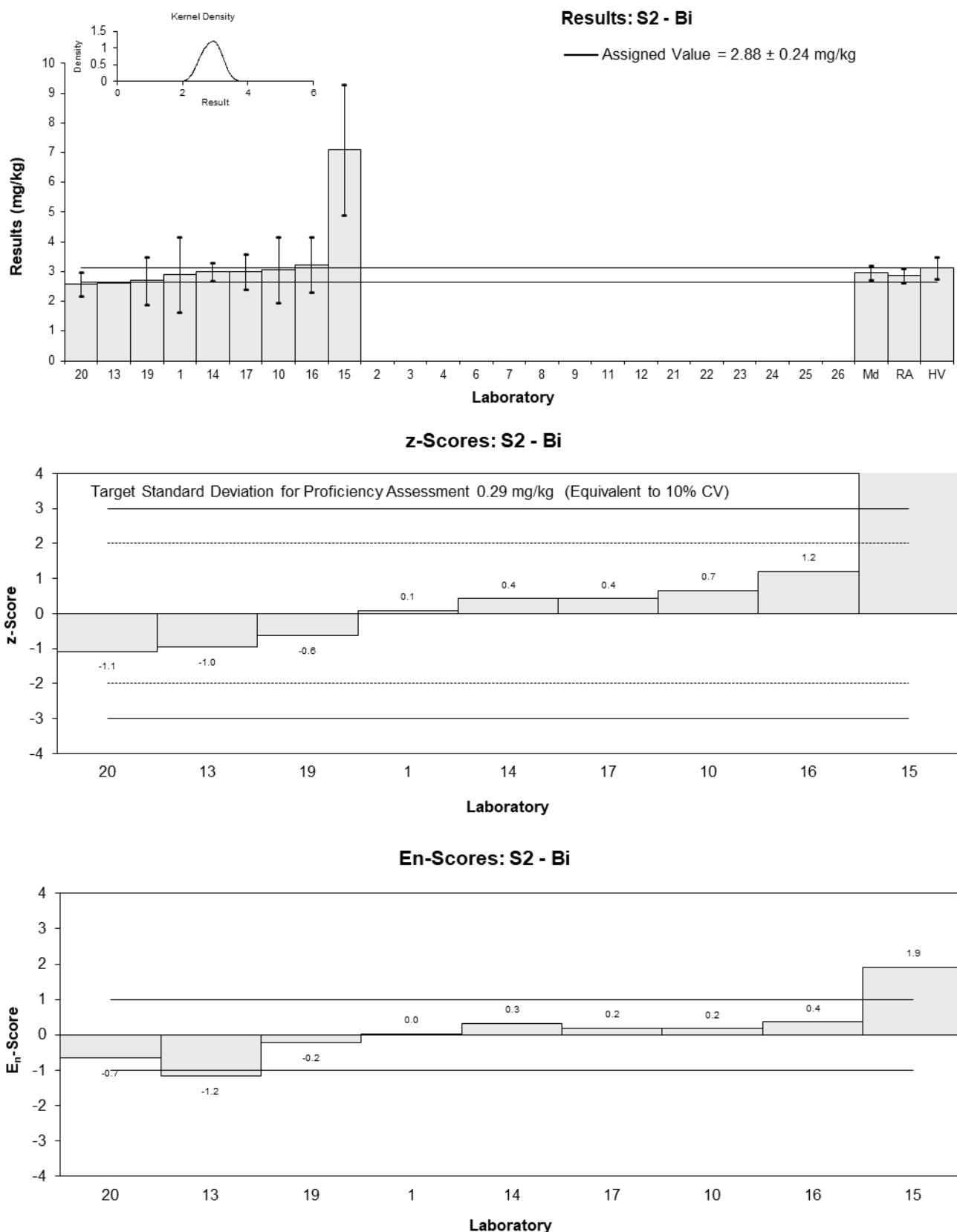


Figure 28

Table 40

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Cd
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	2.8	0.60	-0.16	-0.11
2	NT	NT		
3	3.3	0.2	1.00	1.80
4	NT	NT		
6	3	0.096	0.30	0.80
7	2.84	0.568	-0.07	-0.05
8	NR	NR		
9	2.8	0.6	-0.16	-0.11
10	2.88	0.5	0.02	0.02
11	3.0	0.15	0.30	0.65
12	NT	NT		
13	2.6	0.37	-0.63	-0.69
14*	1	0.1	-4.34	-11.40
15**	6.6	1.3	8.66	2.85
16*	4.689	1.389	4.23	1.30
17	2.7	0.5	-0.39	-0.33
19	2	1	-2.02	-0.86
20	2.78	0.3	-0.21	-0.28
21	3.111	0.469	0.56	0.50
22	NT	NT		
23	3	0.75	0.30	0.17
24	2.75	0.5	-0.28	-0.23
25	3.0	0.3	0.30	0.40
26	NR	NR		

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	2.87	0.13
Spike Value	Not Spiked	
Homogeneity Value	2.86	0.34
Robust Average	2.87	0.17
Median	2.84	0.14
Mean	2.84	
N	17	
Max	4.689	
Min	1	
Robust SD	0.27	
Robust CV	9.6%	

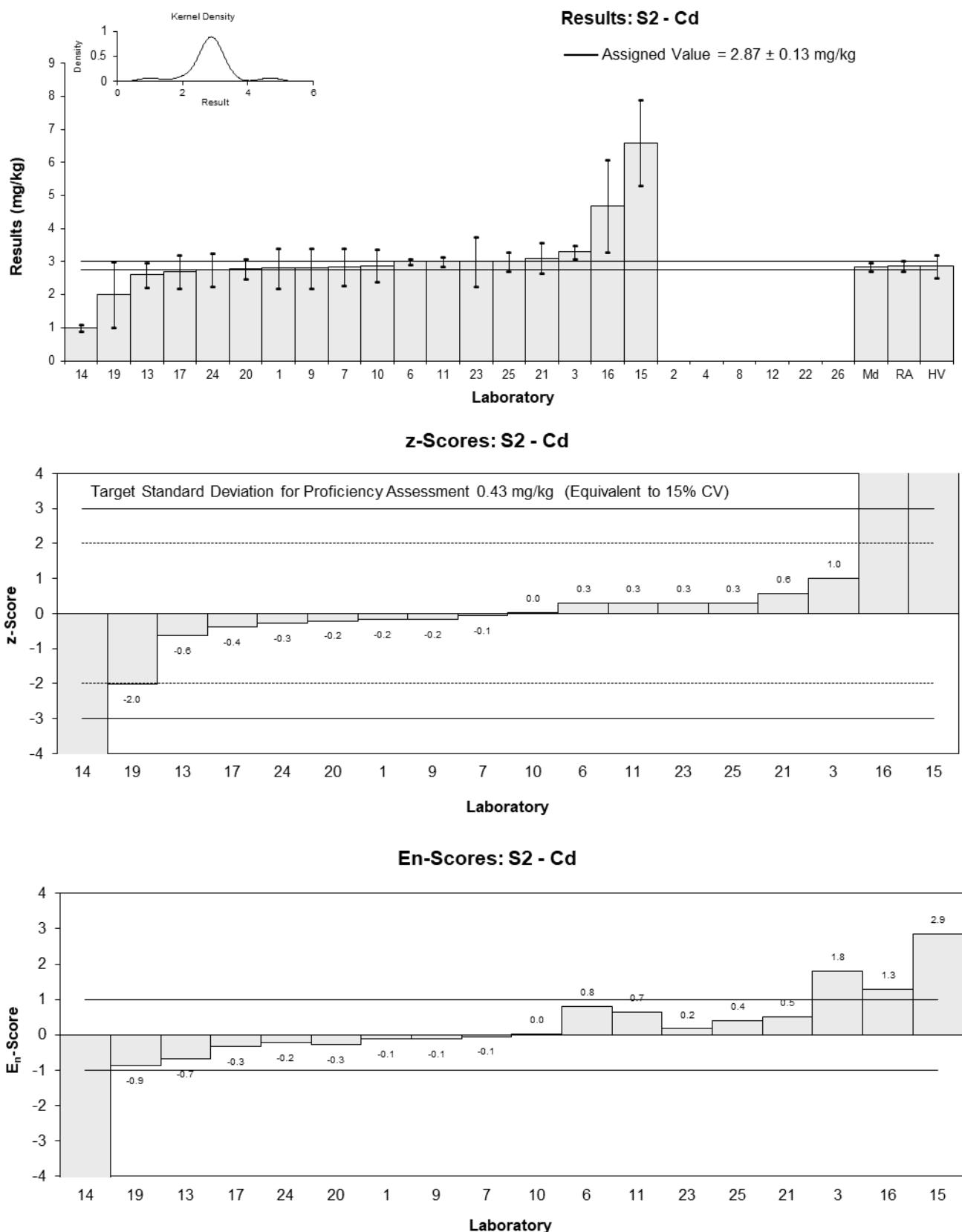


Figure 29

Table 41

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Cr
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	18.7	6.67	-0.76	-0.35
2	NT	NT		
3	27	0.2	1.86	3.45
4	NT	NT		
6	20	0.564	-0.35	-0.61
7	24.4	4.89	1.04	0.64
8	NR	NR		
9	20	4	-0.35	-0.25
10	18.2	3.6	-0.92	-0.73
11	22	1.3	0.28	0.42
12	NT	NT		
13	18.3	5.1	-0.88	-0.52
14	22	2.2	0.28	0.32
15**	46	7.0	7.87	3.46
16	18.10	2.226	-0.95	-1.07
17	22	4	0.28	0.21
19	16	3	-1.61	-1.48
20	21.2	2.5	0.03	0.03
21	23.67	3.74	0.81	0.63
22	NT	NT		
23	22	5.5	0.28	0.16
24	23.0	5	0.60	0.36
25	23.1	2.3	0.63	0.70
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	21.1	1.7
Spike Value	Not Spiked	
Homogeneity Value	20.5	2.5
Robust Average	21.1	1.7
Median	22.0	1.8
Mean	21.2	
N	17	
Max	27	
Min	16	
Robust SD	2.8	
Robust CV	13%	

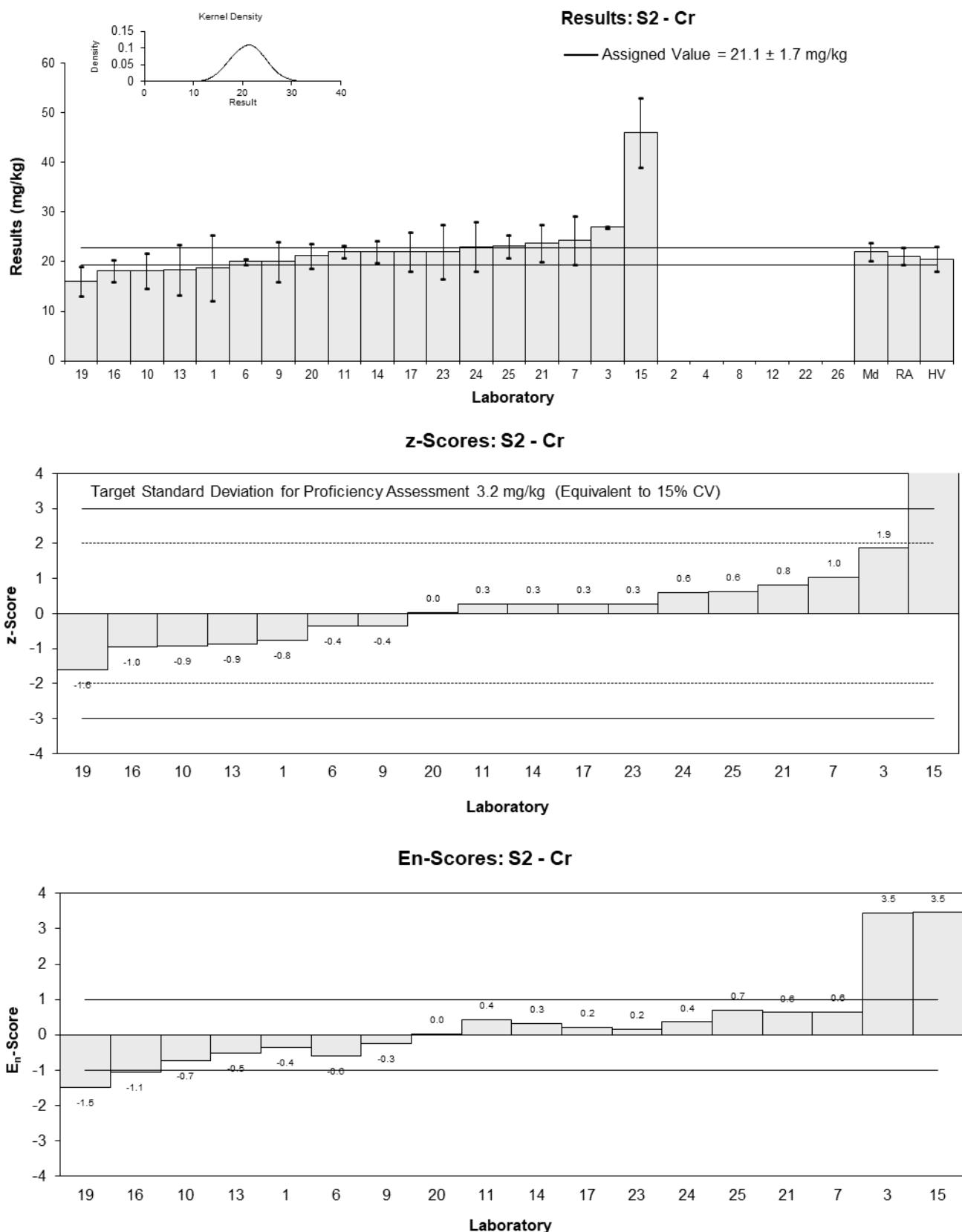


Figure 30

Table 42

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Cs
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty
1	1.1	0.36
2	NT	NT
3	NT	NT
4	NT	NT
6	NT	NT
7	NT	NT
8	NR	NR
9	NT	NT
10	NR	NR
11	NT	NT
12	NT	NT
13	0.6	NR
14	NT	NT
15**	2.6	0.52
16	1.36	NR
17	1.6	0.3
19	0.9	0.2
20	NT	NT
21	NT	NT
22	NT	NT
23	NT	NT
24	NR	NR
25	NT	NT
26	NR	NR

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Homogeneity Value	1.46	0.18
Median	1.10	0.43
Mean	1.11	
N	5	
Max	1.6	
Min	0.6	

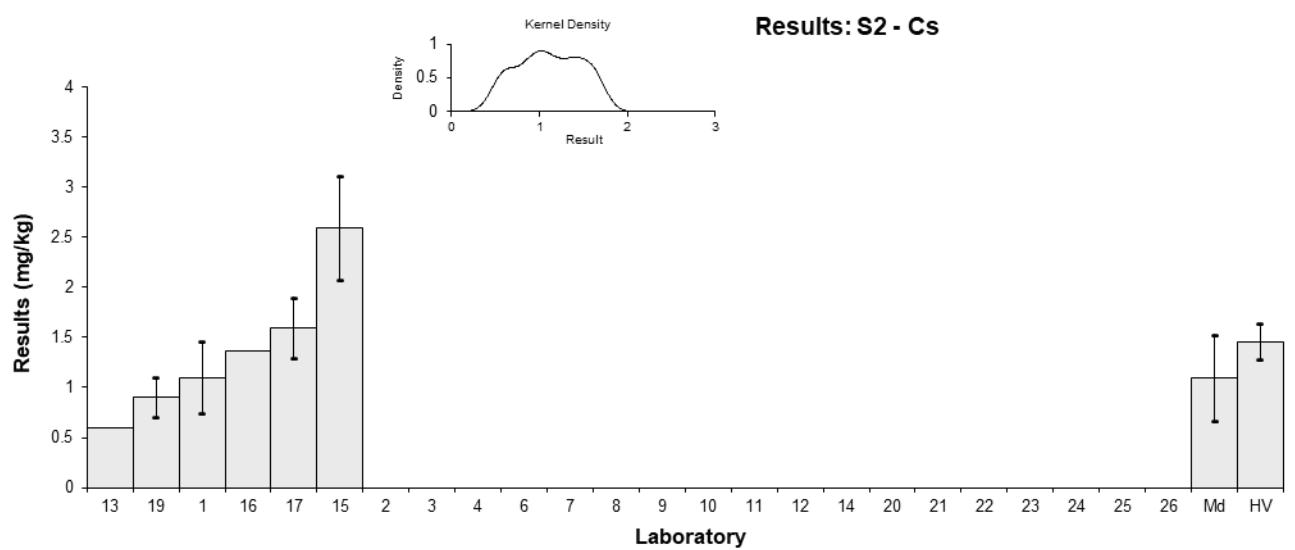


Figure 31

Table 43

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Cu
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	19.6	5.66	-1.40	-0.53
2	NT	NT		
3	27	0.2	1.84	2.09
4	NT	NT		
6	23	0.704	0.09	0.09
7	23.5	4.70	0.31	0.14
8	NR	NR		
9	21	5	-0.79	-0.33
10	18.9	2.9	-1.71	-1.11
11	34	2.04	4.91	3.92
12	NT	NT		
13	21.2	3.57	-0.70	-0.39
14	20	2	-1.23	-0.99
15**	49	7.4	11.49	3.42
16	22.50	2.23	-0.13	-0.10
17	23	5	0.09	0.04
19	18	2	-2.11	-1.70
20	22.0	2.5	-0.35	-0.25
21	27.61	4.8	2.11	0.92
22	NT	NT		
23	24	6	0.53	0.19
24	23.5	5	0.31	0.13
25	25	4	0.96	0.49
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	22.8	2.0
Spike Value	Not Spiked	
Homogeneity Value	23.8	2.9
Robust Average	22.8	2.0
Median	23.0	1.8
Mean	23.2	
N	17	
Max	34	
Min	18	
Robust SD	3.3	
Robust CV	14%	

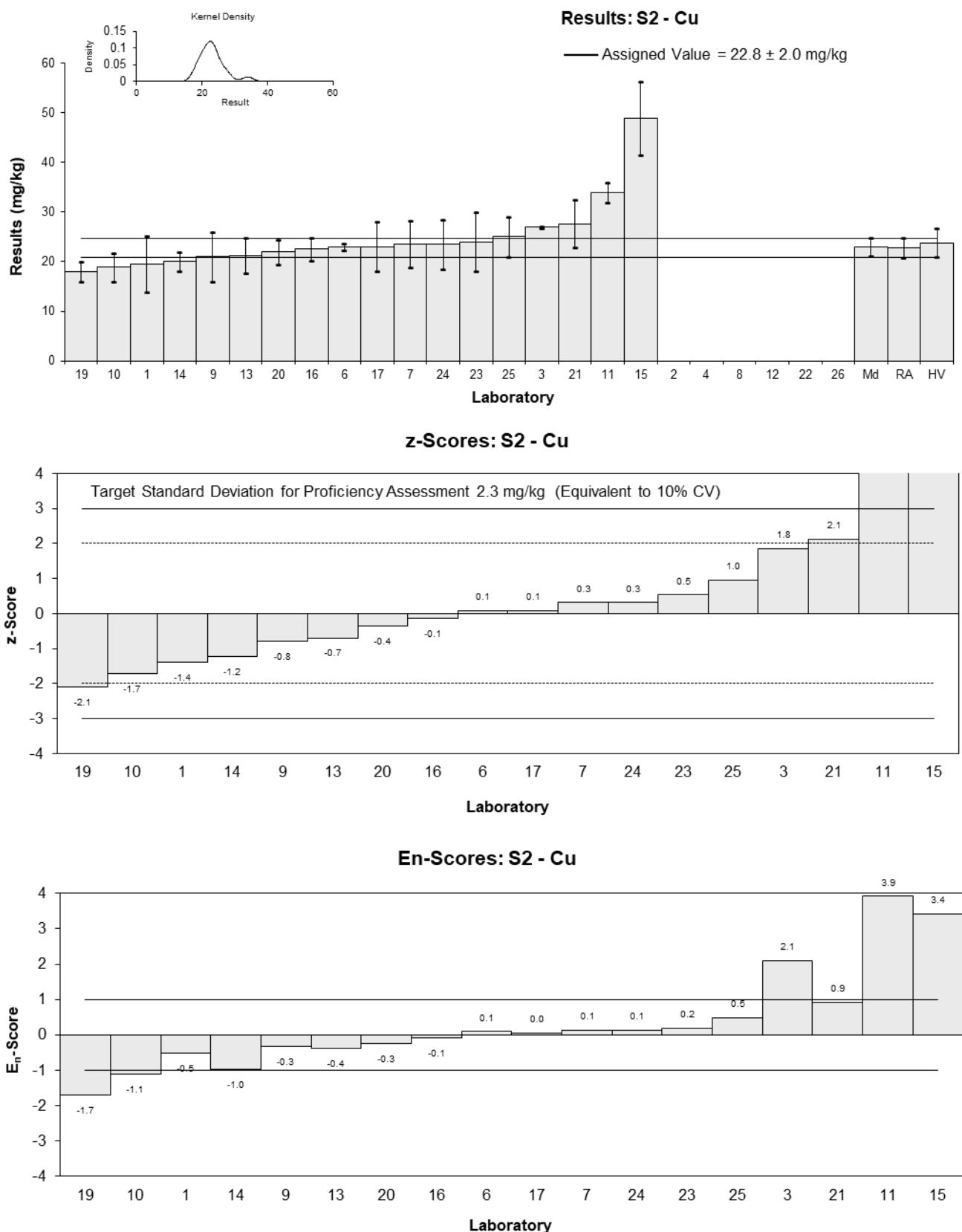


Figure 32

Table 44

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Gd
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty
1	2.2	0.73
2	NT	NT
3	NT	NT
4	NT	NT
6	NT	NT
7	NT	NT
8	NR	NR
9	NT	NT
10	NR	NR
11	NT	NT
12	NT	NT
13	1.7	NR
14	NT	NT
15	NT	NT
16	2.172	NR
17	3.8	0.8
19	1.9	0.2
20	NT	NT
21	NT	NT
22	NT	NT
23	NT	NT
24	NR	NR
25	NT	NT
26	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Homogeneity Value	3.24	0.39
Median	2.17	0.45
Mean	2.35	
N	5	
Max	3.8	
Min	1.7	

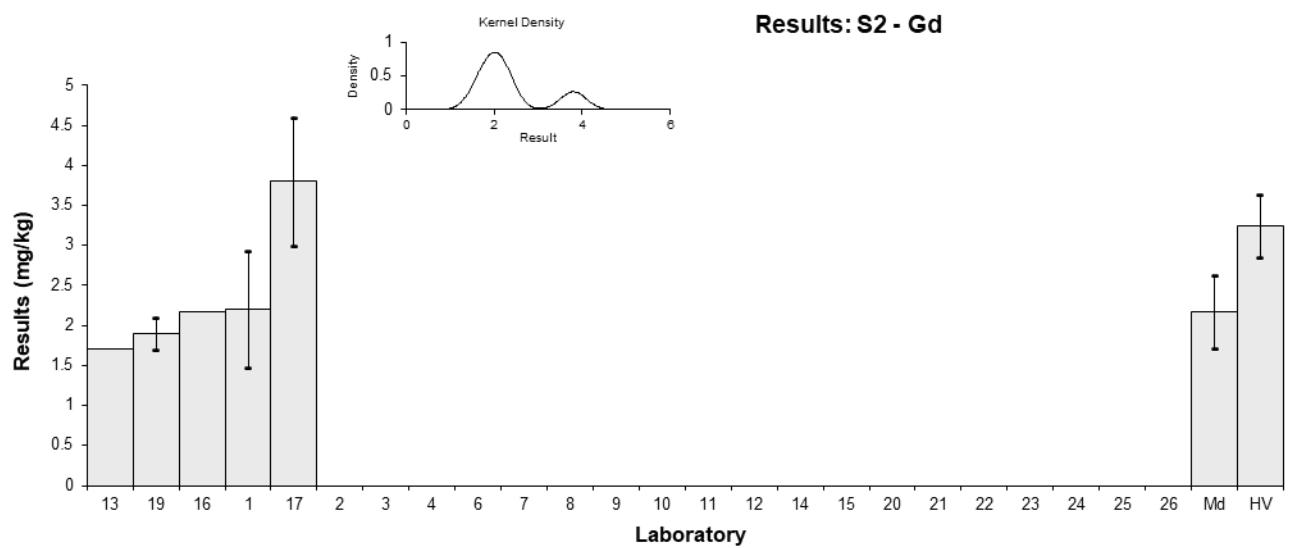


Figure 33

Table 45

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Hg
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	2	0.57	0.03	0.02
2	NT	NT		
3	2.2	0.4	0.70	0.48
4	NT	NT		
6	NT	NT		
7	2.19	0.438	0.67	0.43
8	NR	NR		
9	1.6	0.42	-1.31	-0.86
10	2.03	0.5	0.13	0.08
11	1.8	0.18	-0.64	-0.77
12	NT	NT		
13	1.9	0.25	-0.30	-0.30
14	1.7	0.17	-0.97	-1.21
15**	3.9	0.66	6.40	2.80
16	2.448	0.361	1.53	1.15
17	1.9	0.4	-0.30	-0.21
19	2.0	0.5	0.03	0.02
20	2.43	0.3	1.47	1.28
21	2.261	0.366	0.91	0.67
22	NT	NT		
23	1.9	0.475	-0.30	-0.18
24	1.75	0.5	-0.80	-0.45
25	1.8	0.4	-0.64	-0.44
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	1.99	0.17
Spike Value	2.01	0.10
Homogeneity Value	1.98	0.24
Robust Average	1.99	0.17
Median	1.95	0.16
Mean	1.99	
N	16	
Max	2.448	
Min	1.6	
Robust SD	0.28	
Robust CV	14%	

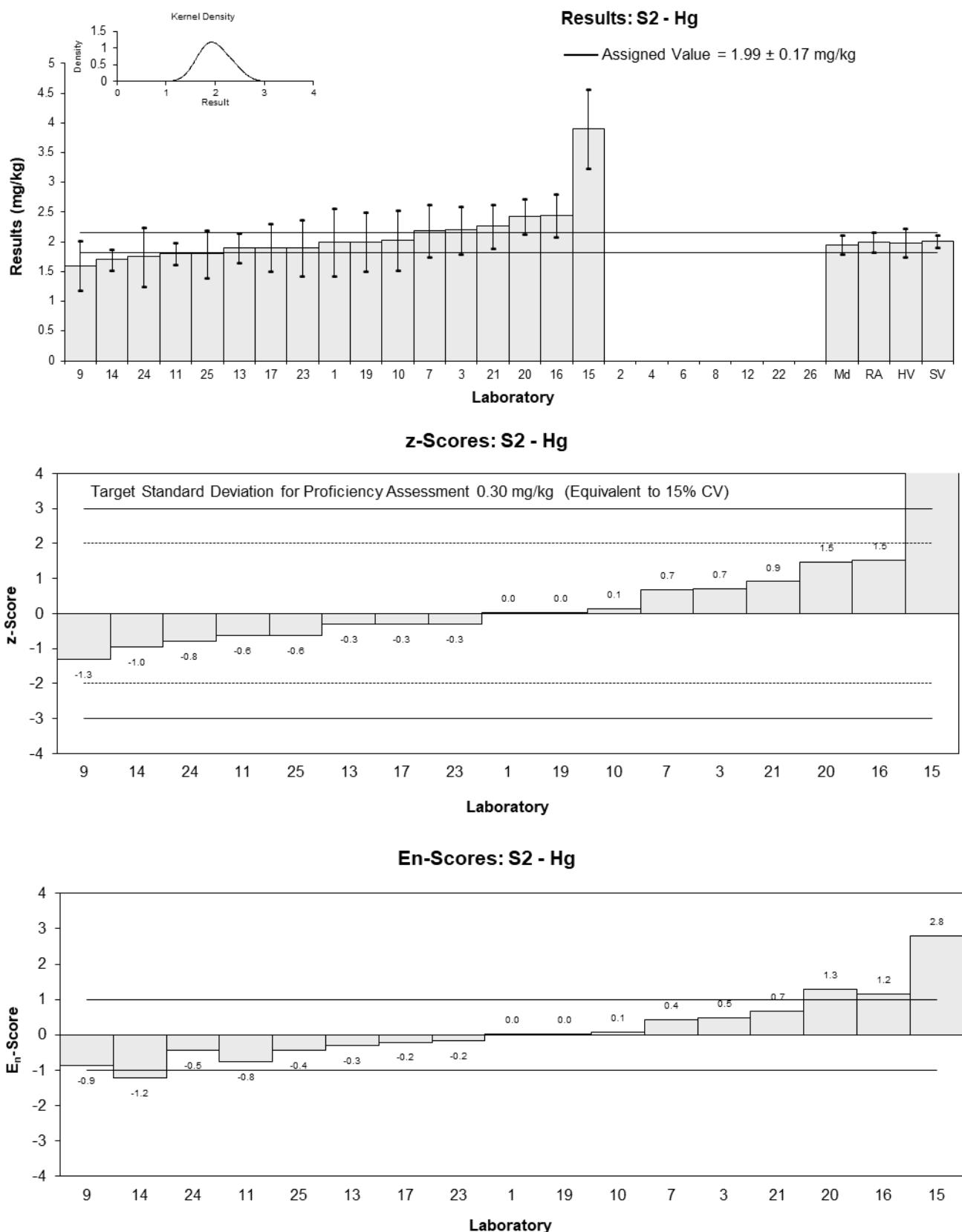


Figure 34

Table 46

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	La
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty
1	12.5	5.28
2	NT	NT
3	NT	NT
4	NT	NT
6	NT	NT
7	NT	NT
8	NR	NR
9	NT	NT
10	NR	NR
11	NT	NT
12	NT	NT
13	11.8	NR
14	NT	NT
15**	31	4.6
16	14.610	NR
17	27	5
19	9.3	1.4
20	NT	NT
21	NT	NT
22	NT	NT
23	NT	NT
24	NR	NR
25	NT	NT
26	NR	NR

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Homogeneity Value	20.6	2.5
Median	12.5	3.5
Mean	15.0	
N	5	
Max	27	
Min	9.3	

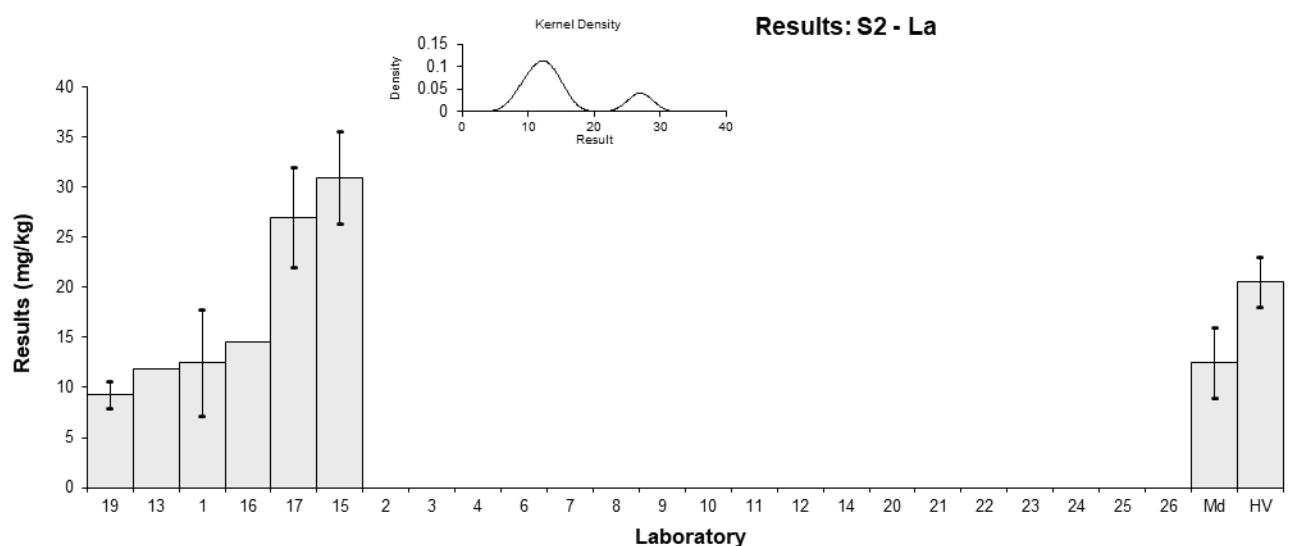


Figure 35

Table 47

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Mn
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	507	136	-0.96	-0.37
2	NT	NT		
3	670	0.2	1.94	2.02
4	NT	NT		
6	493	13.439	-1.21	-1.22
7	676	135.2	2.05	0.79
8	NR	NR		
9	530	110	-0.55	-0.25
10	464	70	-1.73	-1.10
11	510	51.0	-0.91	-0.69
12	NT	NT		
13	484	81.6	-1.37	-0.79
14	670	67	1.94	1.27
15**	1100	250	9.61	2.11
16	512.82	48.439	-0.86	-0.66
17	550	110	-0.20	-0.09
19	437	45	-2.21	-1.76
20	572	55	0.20	0.14
21	581.4	82.0	0.36	0.21
22	NT	NT		
23	670	167.5	1.94	0.62
24	607.0	90	0.82	0.44
25	610	92	0.87	0.46
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	561	54
Spike Value	Not Spiked	
Homogeneity Value	555	67
Robust Average	561	54
Median	550	51
Mean	561	
N	17	
Max	676	
Min	437	
Robust SD	89	
Robust CV	16%	

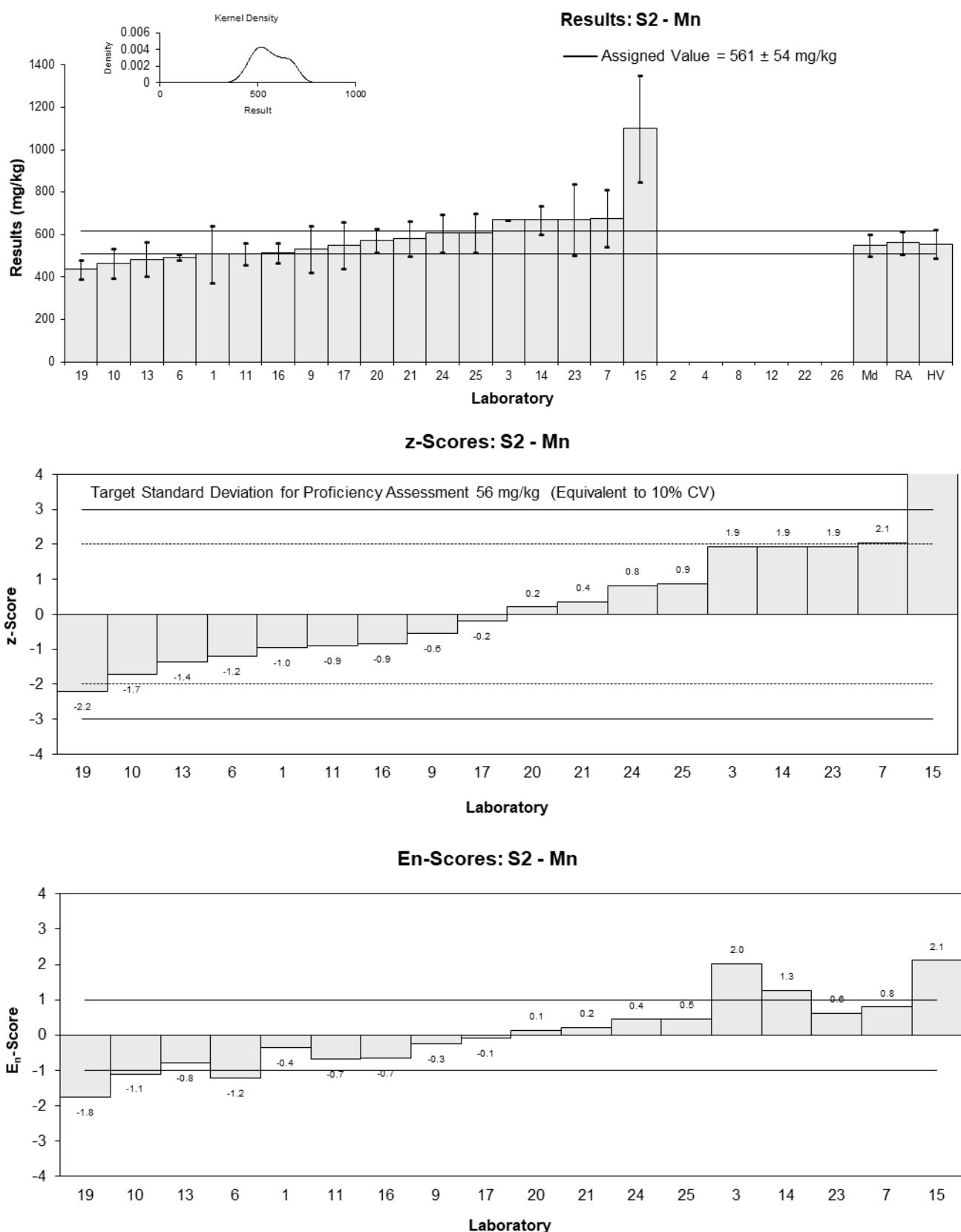


Figure 36

Table 48

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Mo
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	5.6	2.17	-0.11	-0.04
2	NT	NT		
3	7.2	0.2	1.77	2.71
4	NT	NT		
6	NT	NT		
7	6.83	1.37	1.34	0.78
8	NR	NR		
9	5	2	-0.81	-0.33
10	5.15	1.0	-0.63	-0.48
11	5.0	0.50	-0.81	-0.96
12	NT	NT		
13	5.1	1.1	-0.69	-0.48
14	6	0.6	0.36	0.39
15**	13	2.2	8.56	3.23
16	6.248	1.128	0.65	0.45
17	5.7	1.1	0.01	0.01
19	5	1	-0.81	-0.61
20	5.52	0.6	-0.20	-0.21
21	6.669	1.277	1.15	0.71
22	NT	NT		
23	6.2	1.55	0.60	0.31
24	3.54	1	-2.52	-1.91
25	5.6	0.6	-0.11	-0.11
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	5.69	0.52
Spike Value	7.02	0.14
Homogeneity Value	6.10	0.85
Robust Average	5.69	0.52
Median	5.60	0.56
Mean	5.65	
N	16	
Max	7.2	
Min	3.54	
Robust SD	0.84	
Robust CV	15%	

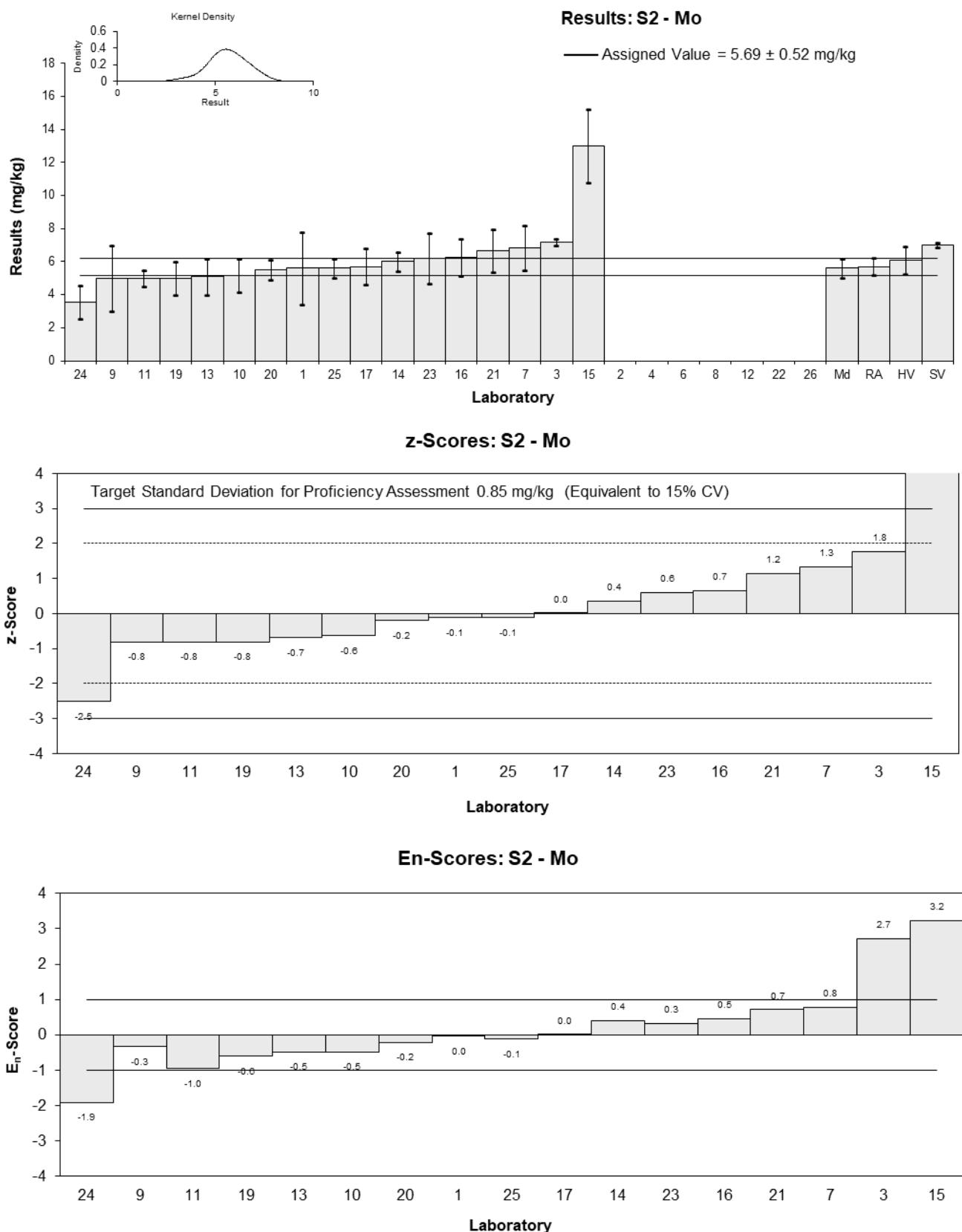


Figure 37

Table 49

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Ni
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	13.3	3.32	-0.43	-0.18
2	NT	NT		
3	18	0.2	2.95	4.97
4	NT	NT		
6	14	0.649	0.07	0.10
7	15.6	3.13	1.22	0.53
8	NR	NR		
9	14	3	0.07	0.03
10	12.4	2.0	-1.08	-0.70
11	13	1.3	-0.65	-0.59
12	NT	NT		
13	13.1	2.57	-0.58	-0.30
14	14	1.4	0.07	0.06
15**	29	7.5	10.86	2.00
16	13.138	1.527	-0.55	-0.44
17	14	3	0.07	0.03
19	12	2	-1.37	-0.88
20	12.8	1.3	-0.79	-0.72
21	14.86	2.42	0.69	0.38
22	NT	NT		
23	14	3.5	0.07	0.03
24	14.0	6	0.07	0.02
25	16	2	1.51	0.97
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	13.9	0.8
Spike Value	Not Spiked	
Homogeneity Value	11.8	1.4
Robust Average	13.9	0.8
Median	14.0	0.8
Mean	14.0	
N	17	
Max	18	
Min	12	
Robust SD	1.3	
Robust CV	9.3%	

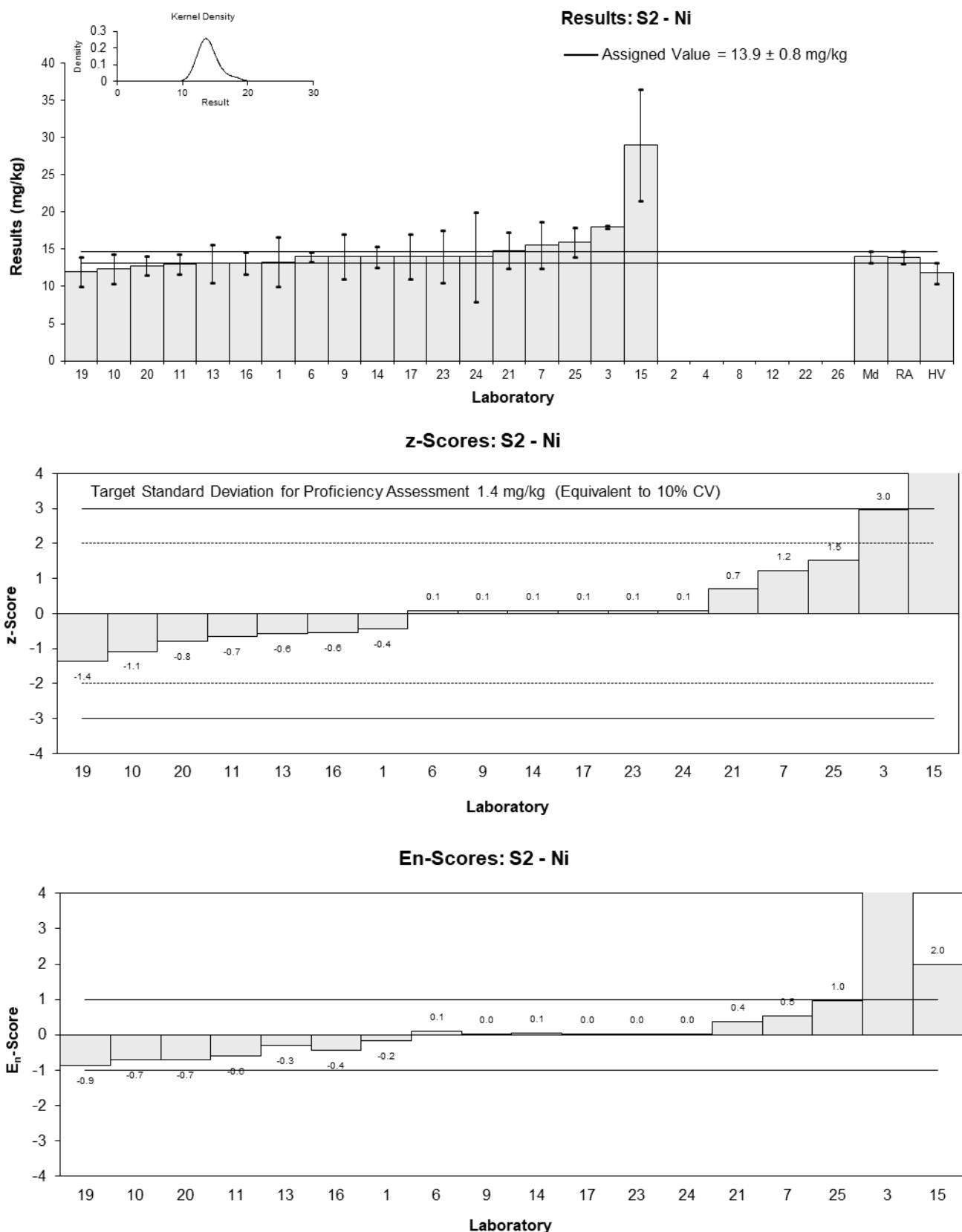


Figure 38

Table 50

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Se
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	5	1.71	0.50	0.20
2	NT	NT		
3	4.7	0.2	0.07	0.12
4	NT	NT		
6	4.0	0.119	-0.93	-1.71
7	4.23	0.846	-0.60	-0.46
8	NR	NR		
9	5	2	0.50	0.17
10*	7.59	1.5	4.22	1.91
11	<5.0	NR		
12	NT	NT		
13*	8.0	2.1	4.80	1.57
14	6	0.6	1.94	1.93
15**	9.4	1.4	6.81	3.29
16**	0.495	0.034	-5.96	-11.49
17	4.5	0.9	-0.22	-0.15
19	<5	NR		
20	4.40	0.5	-0.36	-0.41
21	4.517	0.690	-0.19	-0.17
22	NT	NT		
23	4.8	1.2	0.22	0.12
24	NR	NR		
25	<5.0	NR		
26	NR	NR		

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	4.65	0.36
Spike Value	5.12	0.11
Homogeneity Value	4.59	0.55
Robust Average	4.99	0.67
Median	4.75	0.32
Mean	5.23	
N	12	
Max	8	
Min	4	
Robust SD	0.93	
Robust CV	19%	

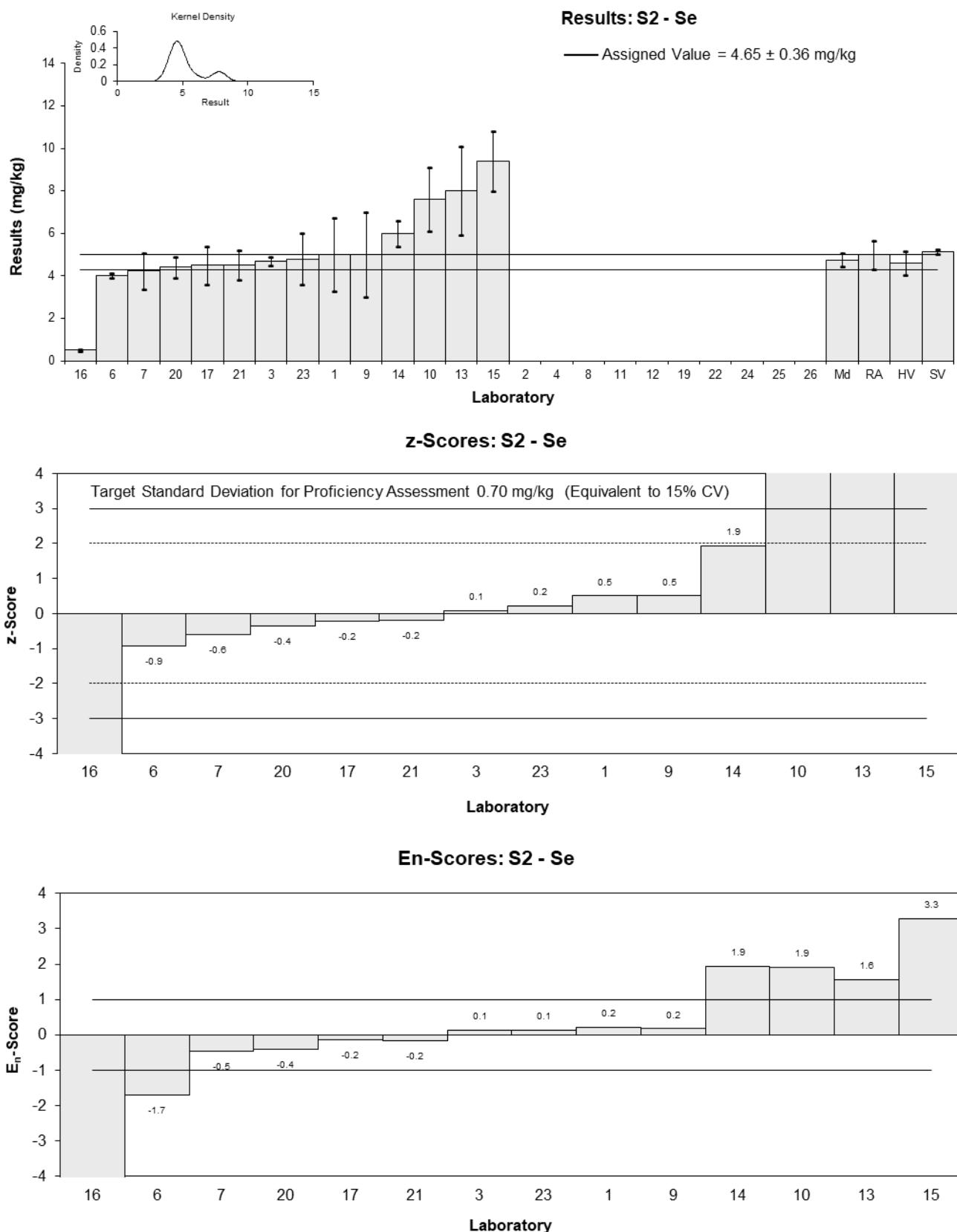


Figure 39

Table 51

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Sm
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty
1	2.4	1.07
2	NT	NT
3	NT	NT
4	NT	NT
6	NT	NT
7	NT	NT
8	NR	NR
9	NT	NT
10	NR	NR
11	NT	NT
12	NT	NT
13	2.2	NR
14	NT	NT
15	NT	NT
16	2.667	NR
17	4.7	0.9
19	1.7	0.2
20	NT	NT
21	NT	NT
22	NT	NT
23	NT	NT
24	NR	NR
25	NT	NT
26	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Homogeneity Value	3.69	0.44
Median	2.40	0.44
Mean	2.7	
N	5	
Max	4.7	
Min	1.7	

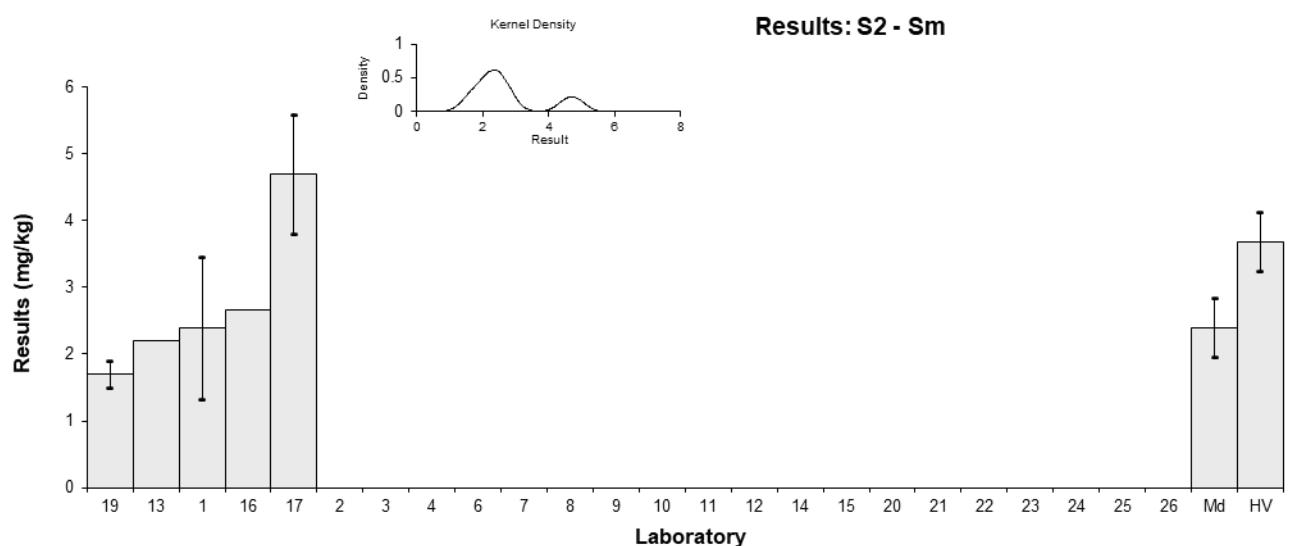


Figure 40

Table 52

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Tl
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	0.8	0.17		
2	NT	NT		
3	<1	0.2		
4	NT	NT		
6	NT	NT		
7	<10	<2		
8	NR	NR		
9	< 10	NR		
10	0.75	0.4	-0.91	-0.19
11	NT	NT		
12	NT	NT		
13	0.8	0.16	-0.30	-0.15
14	<2	NR		
15	<2.8	NR		
16	0.8697	0.1278	0.54	0.32
17	0.86	0.17	0.42	0.20
19	NT	NT		
20	0.87	0.1	0.55	0.39
21	< 10	NR		
22	NT	NT		
23	< 10	NR		
24	NR	NR		
25	<5.0	NR		
26	NR	NR		

Statistics

Assigned Value	0.825	0.057
Spike Value	0.999	0.041
Homogeneity Value	0.95	0.11
Robust Average	0.825	0.057
Median	0.830	0.053
Mean	0.825	
N	6	
Max	0.87	
Min	0.75	
Robust SD	0.056	
Robust CV	6.8%	

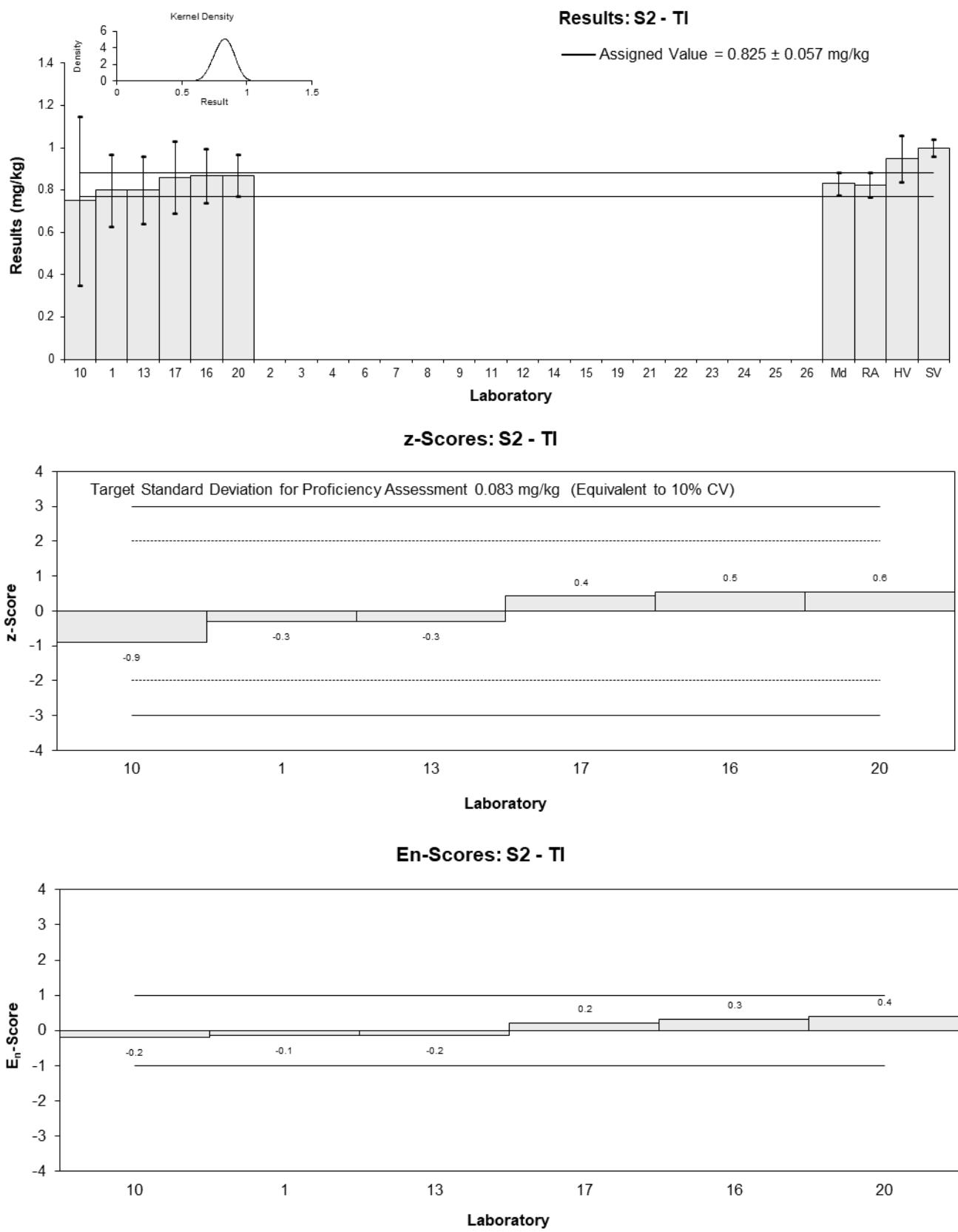


Figure 41

Table 53

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	U
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty
1	0.6	0.12
2	NT	NT
3	<10	0.2
4	NT	NT
6	NT	NT
7	<10	<2
8	NR	NR
9	< 10	NR
10	0.978	0.2
11	<1	NR
12	NT	NT
13	0.7	NR
14	<2	NR
15**	1.7	0.25
16	0.876	0.107
17	1.3	0.3
19	0.5	0.1
20	1.60	0.2
21	< 10	NR
22	NT	NT
23	< 10	NR
24	NR	NR
25	NT	NT
26	NR	NR

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Homogeneity Value	1.07	0.13
Robust Average	0.94	0.42
Median	0.88	0.39
Mean	0.94	
N	7	
Max	1.6	
Min	0.5	
Robust SD	0.45	
Robust CV	48%	

Results: S2 - U

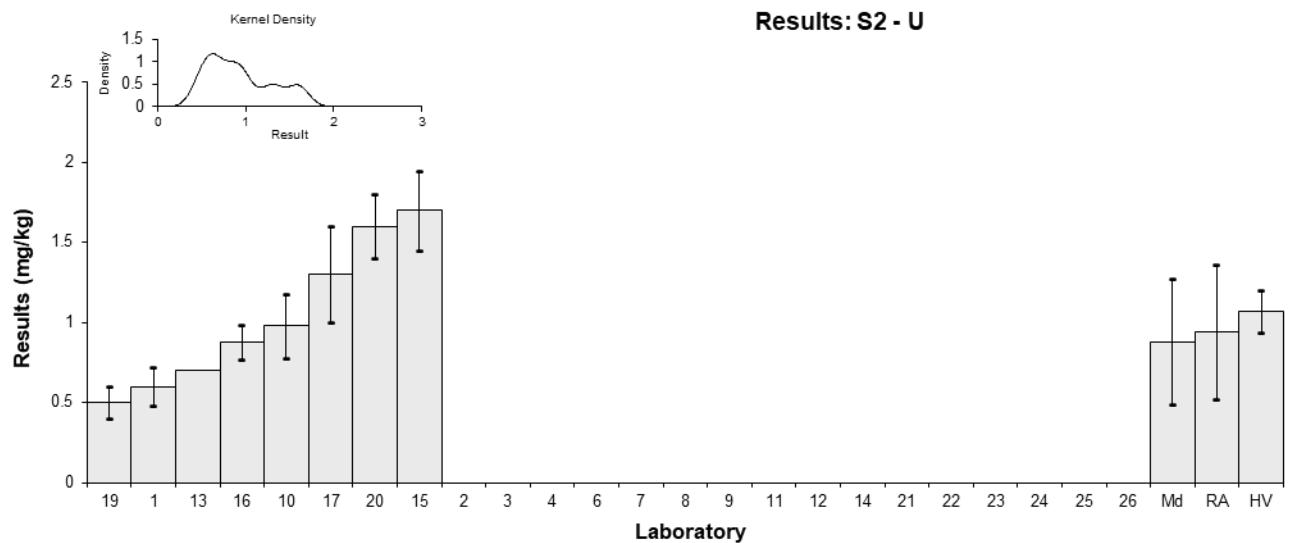


Figure 42

Table 54

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	V
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	30	8.44	-0.59	-0.32
2	NT	NT		
3	40	0.2	1.44	2.03
4	NT	NT		
6	31	0.709	-0.39	-0.53
7	36.3	7.26	0.69	0.42
8	NR	NR		
9	28	5	-0.99	-0.80
10	30.3	9.0	-0.53	-0.27
11	30	3.0	-0.59	-0.63
12	NT	NT		
13	30	5.6	-0.59	-0.44
14	34	3.4	0.22	0.23
15**	69	19	7.32	1.87
16	47.084	5.554	2.87	2.16
17	32	6	-0.18	-0.13
19	26	4	-1.40	-1.30
20	32.5	3.5	-0.08	-0.08
21	37.68	5.86	0.97	0.70
22	NT	NT		
23	34	8.5	0.22	0.12
24	22.5	7	-2.11	-1.33
25	42.8	NR	2.01	2.83
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	32.9	3.5
Spike Value	Not Spiked	
Homogeneity Value	34.8	4.2
Robust Average	32.9	3.5
Median	32.0	1.8
Mean	33.2	
N	17	
Max	47.084	
Min	22.5	
Robust SD	5.9	
Robust CV	18%	

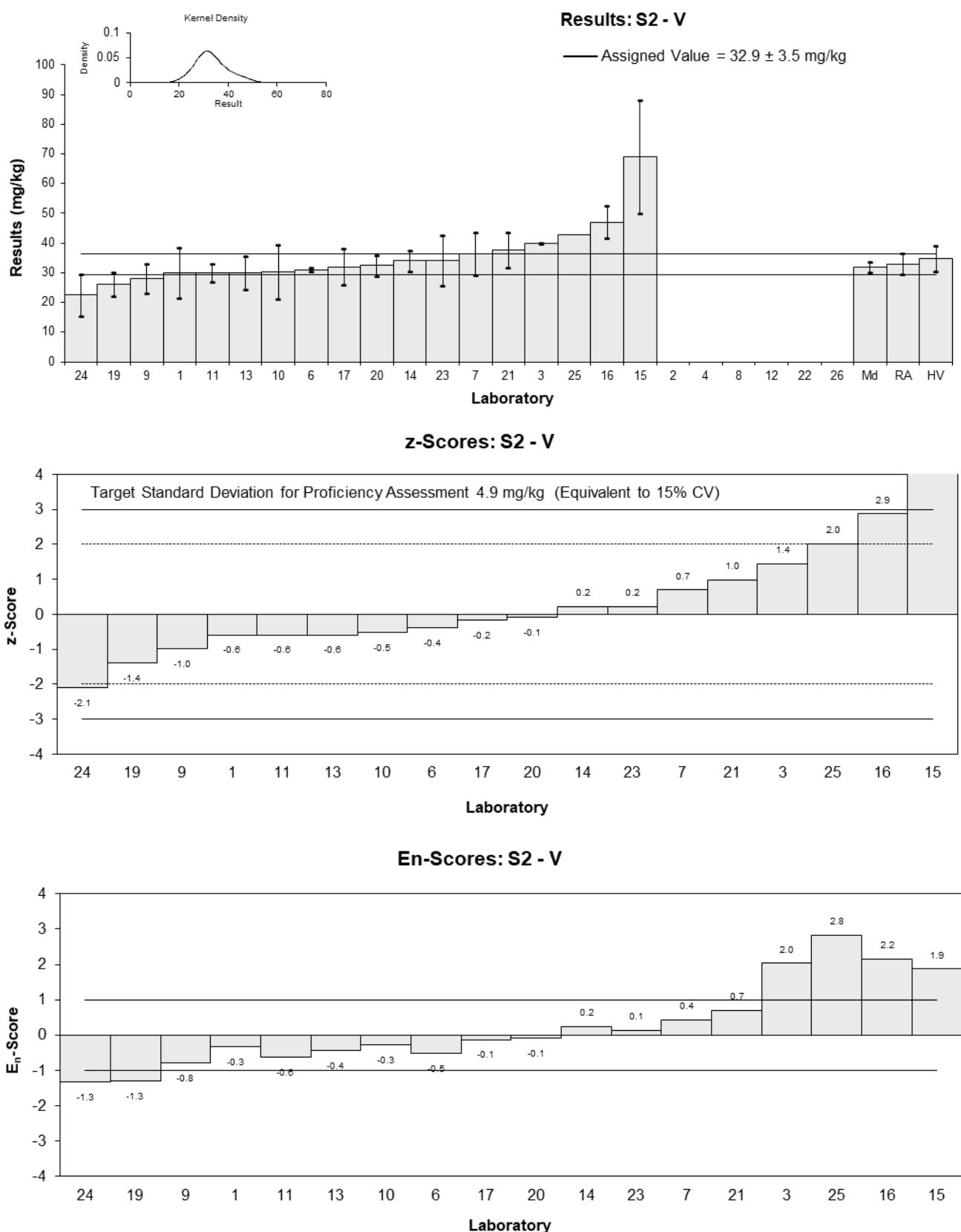


Figure 43

Table 55

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Zn
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	691	6.30	-0.72	-1.40
2	NT	NT		
3	890	0.2	1.95	3.82
4	NT	NT		
6	746	20.294	0.01	0.02
7	748	150	0.04	0.02
8	NR	NR		
9	740	140	-0.07	-0.03
10	703	70	-0.56	-0.53
11	770	77	0.34	0.29
12	NT	NT		
13	675	104.7	-0.94	-0.63
14	600	60	-1.95	-2.04
15**	1800	470	14.16	2.24
16*	1236.26	142.85	6.59	3.32
17	760	150	0.20	0.10
19	682	69	-0.85	-0.80
20	742	75	-0.04	-0.04
21	810.8	128.7	0.88	0.49
22	NT	NT		
23	780	195	0.47	0.18
24	828.0	150	1.11	0.54
25	760	38	0.20	0.28
26	NR	NR		

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	745	38
Spike Value	Not Spiked	
Homogeneity Value	776	93
Robust Average	753	42
Median	748	40
Mean	774	
N	17	
Max	1236.26	
Min	600	
Robust SD	70	
Robust CV	9.3%	

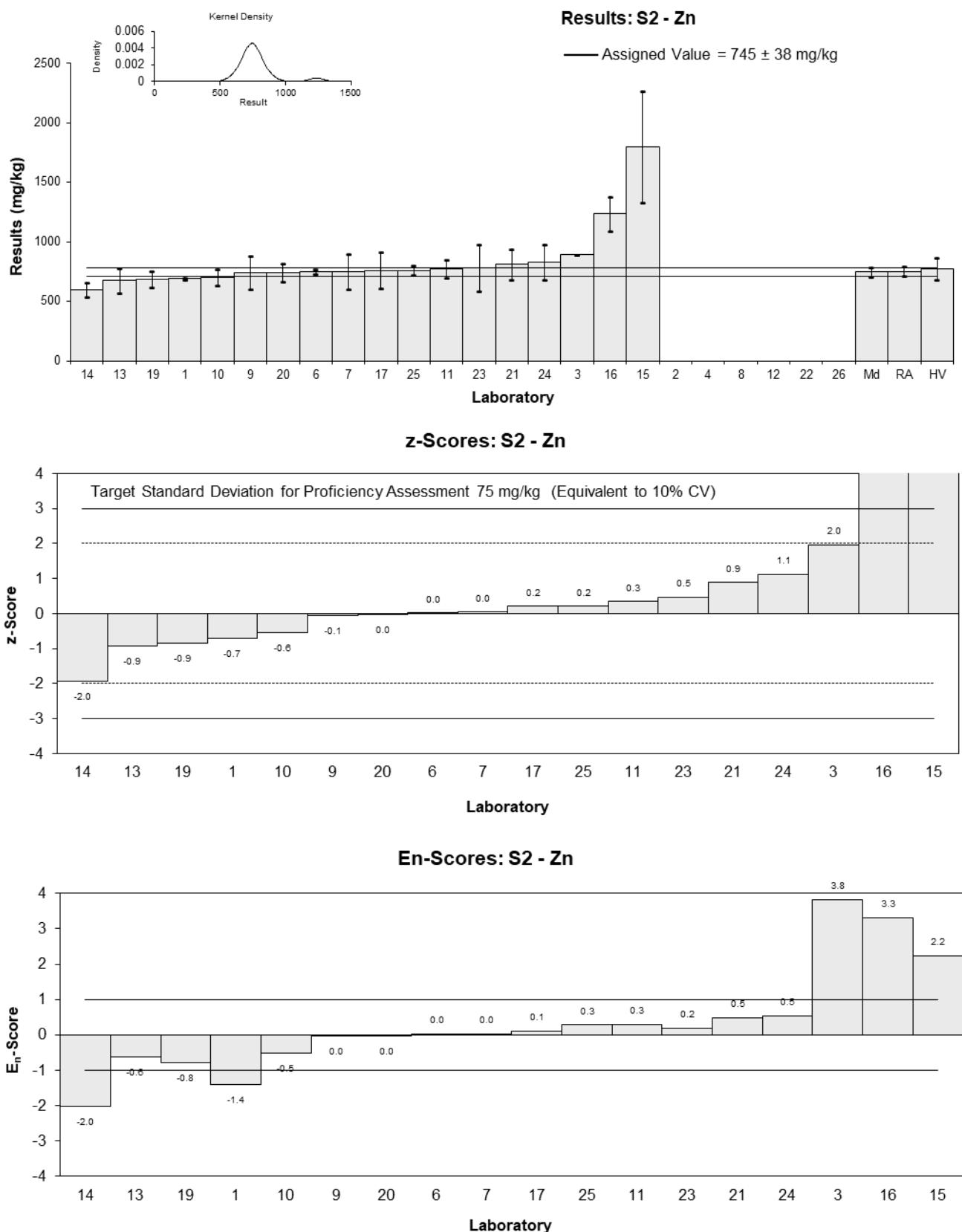


Figure 44

Table 56

Sample Details

Sample No.	S2
Matrix	Moist soil
Analyte	Moisture Content
Unit	%

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	17.5	3.5	-0.28	-0.14
2	NT	NT		
3	19	0.3	0.56	2.36
4	NT	NT		
6	19.29	NR	0.72	4.30
7	18	5.38	0.00	0.00
8	NR	NR		
9	17.9	3	-0.06	-0.03
10	18.1	3.6	0.06	0.03
11	18	1.8	0.00	0.00
12	NT	NT		
13*	8.6	NR	-5.22	-31.33
14	18.3	2.1	0.17	0.14
15**	63.4	NR	25.22	151.33
16	18.3	2.03	0.17	0.15
17	18	3	0.00	0.00
19	18.1	0.8	0.06	0.12
20	16.7	0.5	-0.72	-2.23
21	17.7	3.2	-0.17	-0.09
22	18	1.8	0.00	0.00
23	18	5.4	0.00	0.00
24	18.3	3	0.17	0.10
25	17.1	NR	-0.50	-3.00
26	NR	NR		

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	18.0	0.3
Spike Value	18.0	0.4
Homogeneity Value	18.0	0.4
Robust Average	18.0	0.3
Median	18.0	0.3
Mean	17.5	
N	18	
Max	19.29	
Min	8.6	
Robust SD	0.58	
Robust CV	3.2%	

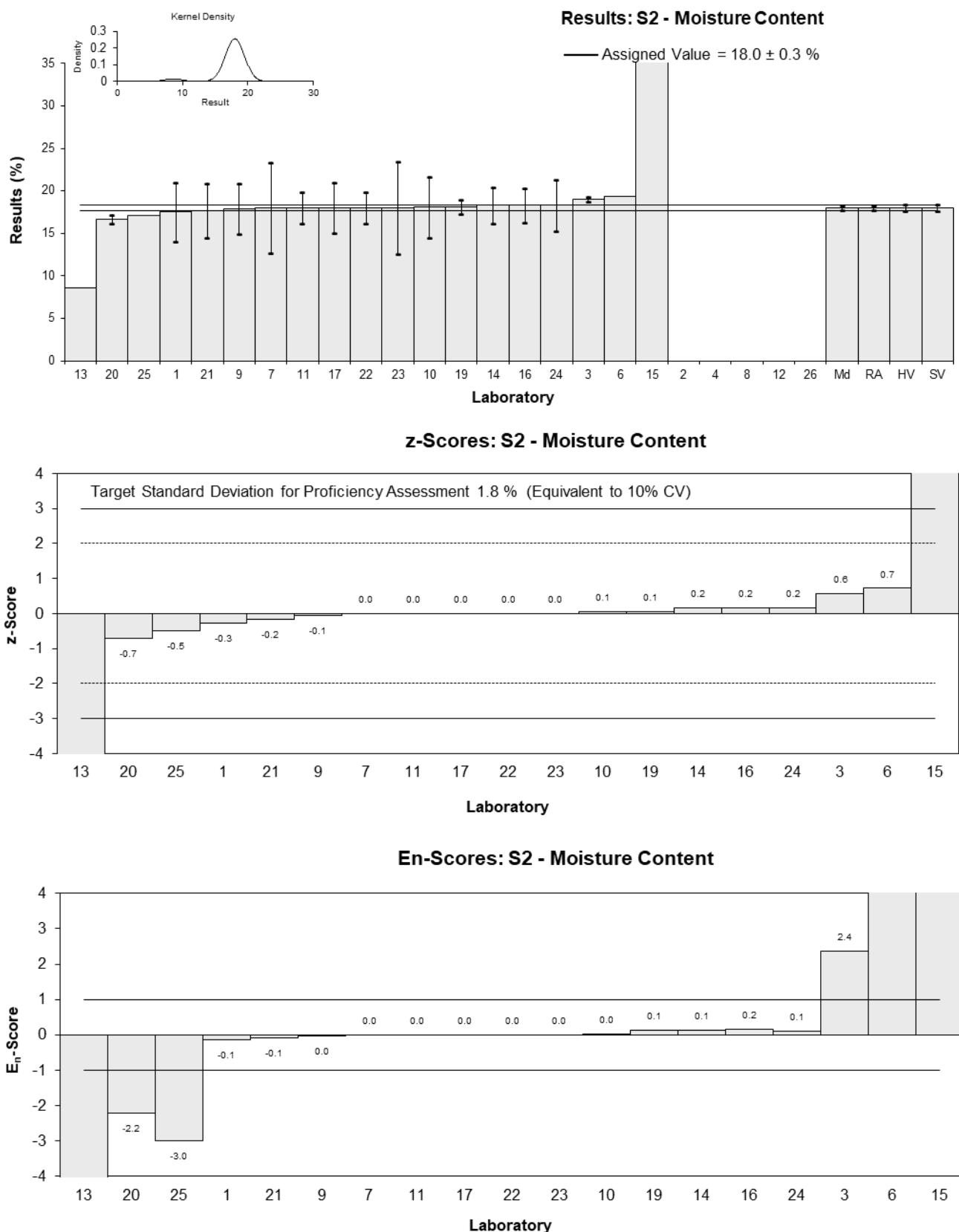


Figure 45

Table 57

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	5280	798	0.21	0.13
2	NT	NT		
3	NT	NT		
4	NR	NR		
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	4600	920	-1.10	-0.58
10	5020	600	-0.29	-0.22
11	NT	NT		
12	NT	NT		
13	5760	1099	1.14	0.51
14	NT	NT		
15	NT	NT		
16	5797.7	588.3	1.21	0.93
17	5410	1080	0.46	0.21
19	5000	982	-0.33	-0.16
20	4920	500	-0.48	-0.42
21	NT	NT		
22	4460	268	-1.37	-1.67
23	5058	1265	-0.22	-0.09
24	5405	800	0.45	0.27
25	5300	530	0.25	0.21
26	NR	NR		

Statistics

Assigned Value	5170	330
Spike Value	Not Spiked	
Homogeneity Value	5080	600
Robust Average	5170	330
Median	5170	260
Mean	5170	
N	12	
Max	5797.7	
Min	4460	
Robust SD	460	
Robust CV	8.9%	

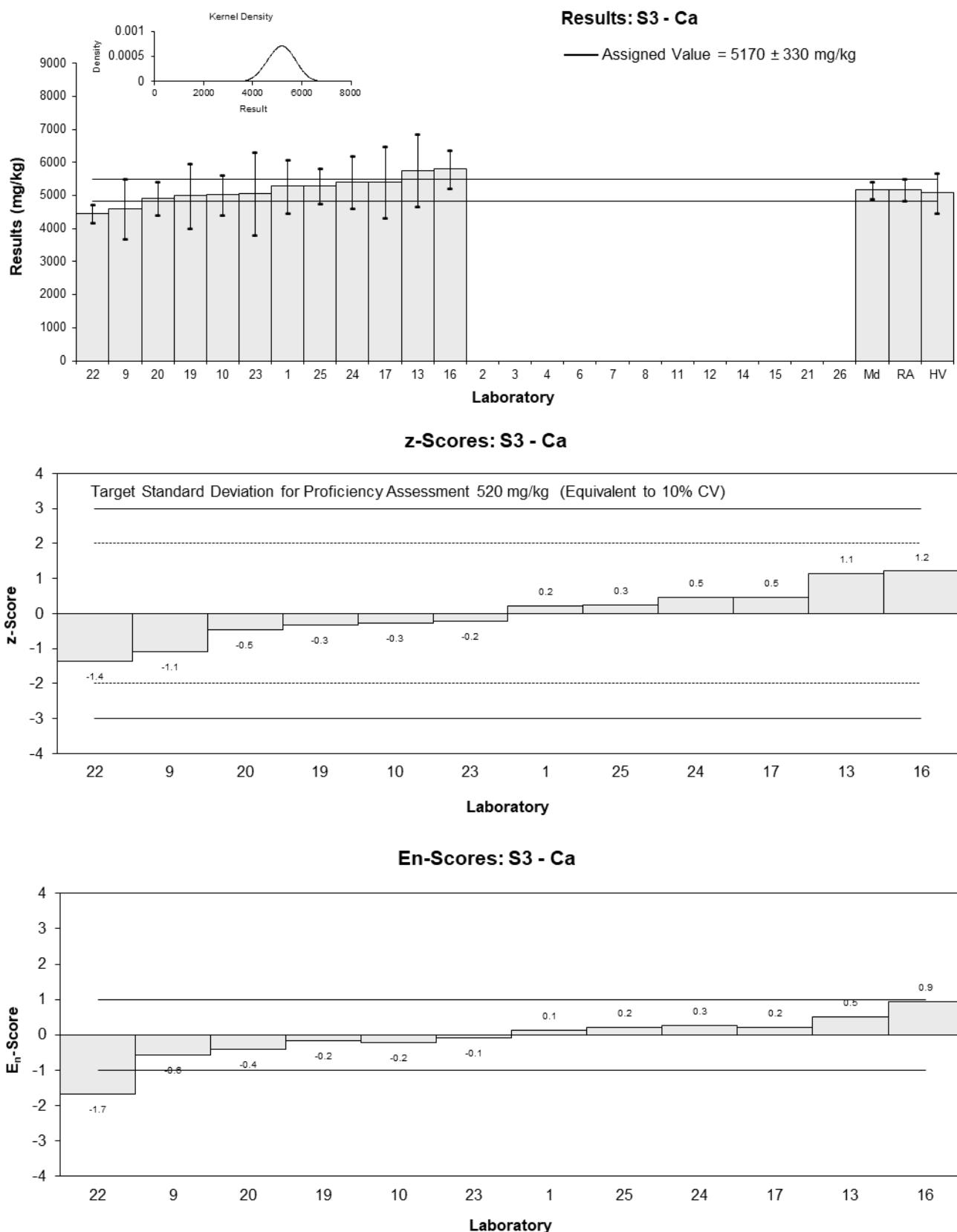


Figure 46

Table 58

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	22600	9529	0.27	0.06
2	NT	NT		
3	23000	0.2	0.45	1.11
4	NR	NR		
6	23040	4129.562	0.47	0.25
7	20700	41.4	-0.59	-1.44
8	NR	NR		
9	20000	4000	-0.91	-0.49
10	21900	4400	-0.05	-0.02
11	NT	NT		
12	NT	NT		
13	22600	7865	0.27	0.08
14	NT	NT		
15	NT	NT		
16	25000	2320	1.36	1.21
17	21700	4300	-0.14	-0.07
19	21900	2500	-0.05	-0.04
20	19604	2000	-1.09	-1.09
21	20461	3320	-0.70	-0.45
22	NR	NR		
23	22237	5560	0.11	0.04
24	23200	4500	0.55	0.26
25	23000	3450	0.45	0.28
26	NR	NR		

Statistics

Assigned Value	22000	900
Spike Value	Not Spiked	
Homogeneity Value	19700	2400
Robust Average	22000	900
Median	22200	700
Mean	22100	
N	15	
Max	25000	
Min	19604	
Robust SD	1500	
Robust CV	6.6%	

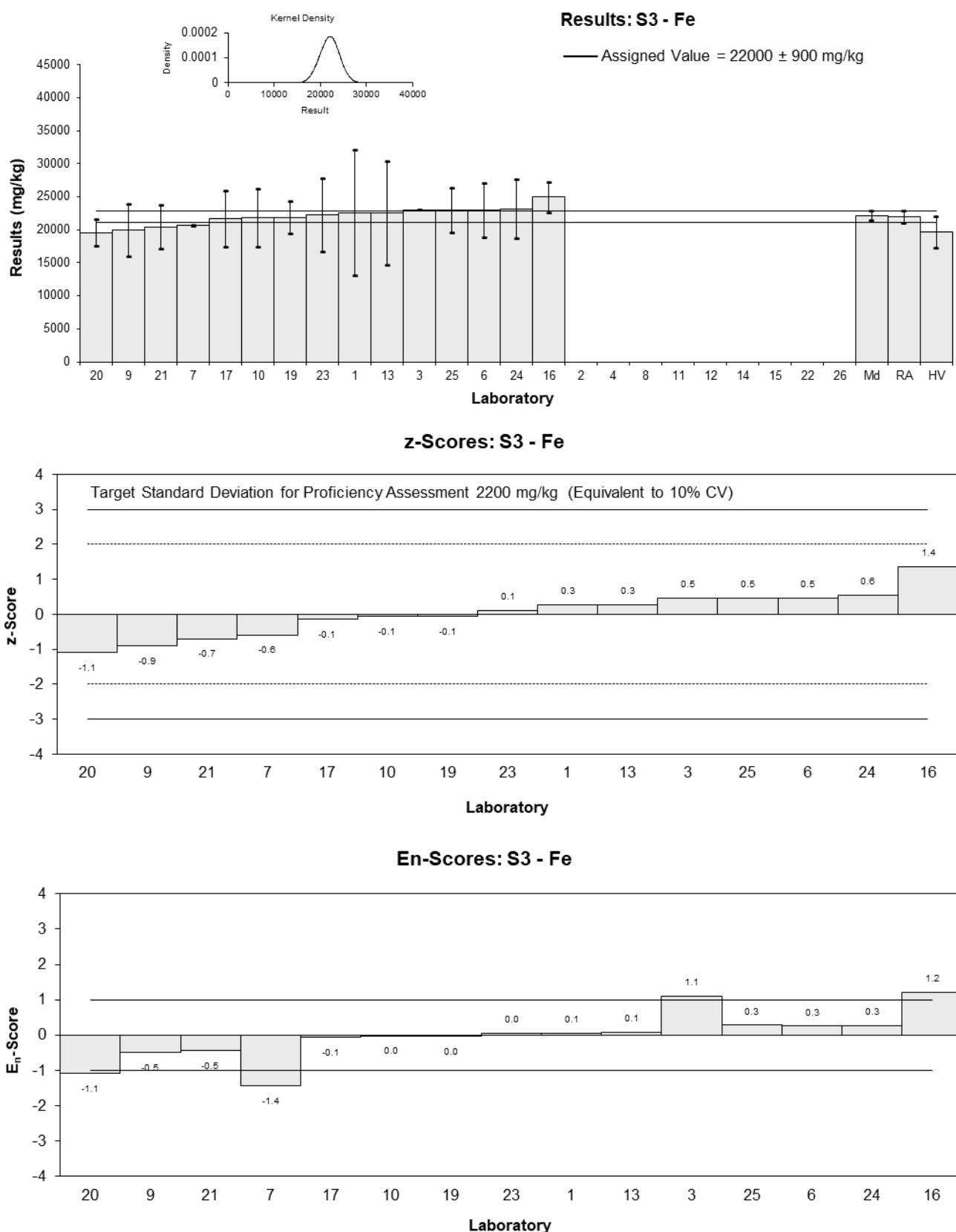


Figure 47

Table 59

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	840	118	-0.95	-0.80
2	NT	NT		
3	NT	NT		
4	NR	NR		
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	880	180	-0.68	-0.45
10	886	160	-0.64	-0.46
11	NT	NT		
12	NT	NT		
13	980	160	0.00	0.00
14	NT	NT		
15	NT	NT		
16	1066	150	0.59	0.43
17	1210	240	1.56	0.84
19	720	84	-1.77	-1.68
20	986	100	0.04	0.04
21	NT	NT		
22	NR	NR		
23	1044	261	0.44	0.22
24	922.5	250	-0.39	-0.20
25	1400	140	2.86	2.20
26	NR	NR		

Statistics

Assigned Value	980	130
Spike Value	Not Spiked	
Homogeneity Value	1200	150
Robust Average	980	130
Median	980	110
Mean	990	
N	11	
Max	1400	
Min	720	
Robust SD	180	
Robust CV	18%	

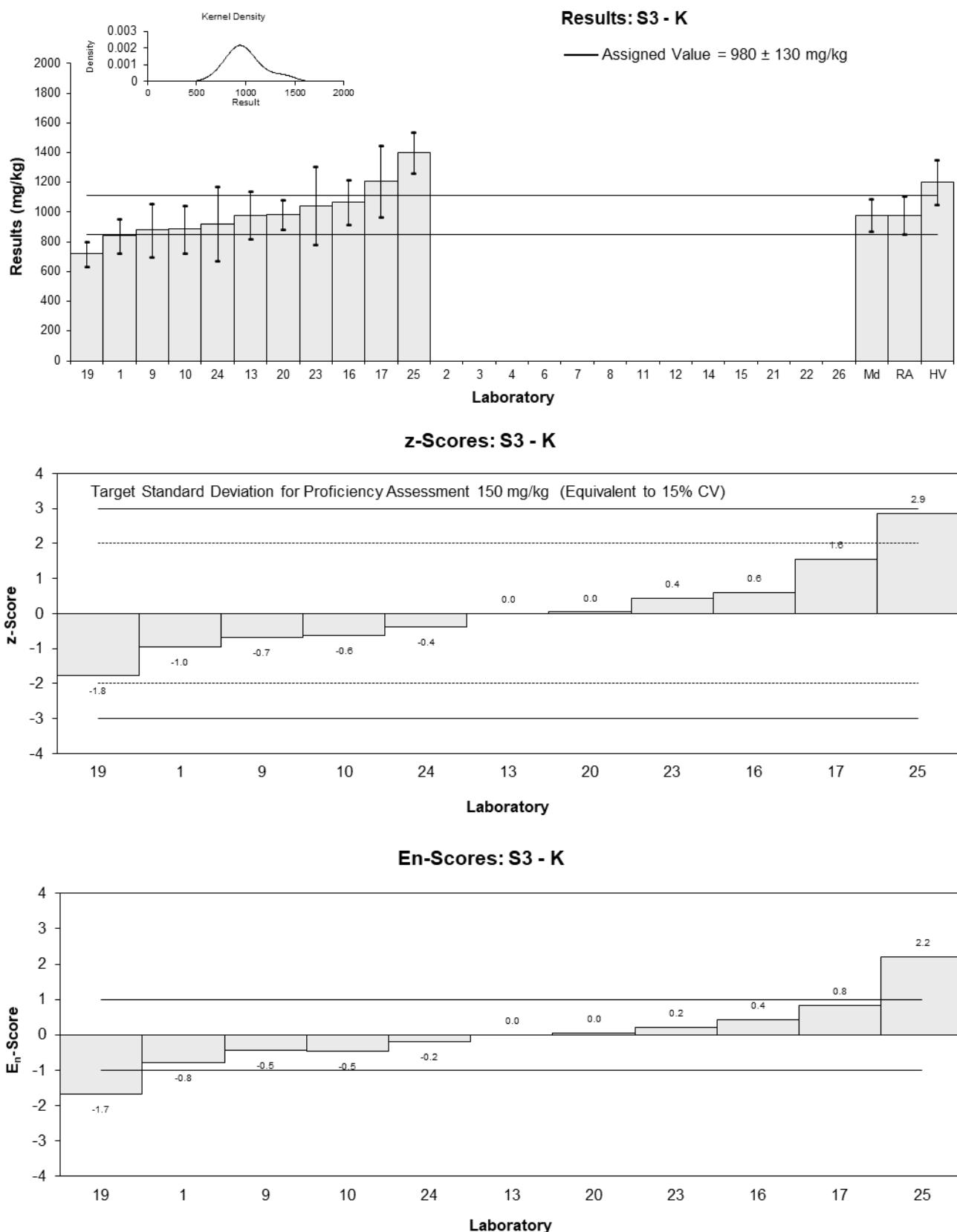


Figure 48

Table 60

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	1010	177	-1.06	-0.62
2	NT	NT		
3	NT	NT		
4	NR	NR		
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	1100	220	-0.27	-0.13
10	1030	110	-0.88	-0.74
11	NT	NT		
12	NT	NT		
13	1200	220	0.62	0.30
14	NT	NT		
15	NT	NT		
16	1217	117	0.77	0.61
17	1190	240	0.53	0.24
19	970	136	-1.42	-1.01
20	1081	100	-0.43	-0.38
21	NT	NT		
22	1260	89	1.15	1.09
23	1133	283	0.03	0.01
24	1190	200	0.53	0.28
25	1200	120	0.62	0.49
26	NR	NR		

Statistics

Assigned Value	1130	80
Spike Value	Not Spiked	
Homogeneity Value	1120	140
Robust Average	1130	80
Median	1160	60
Mean	1130	
N	12	
Max	1260	
Min	970	
Robust SD	100	
Robust CV	9.2%	

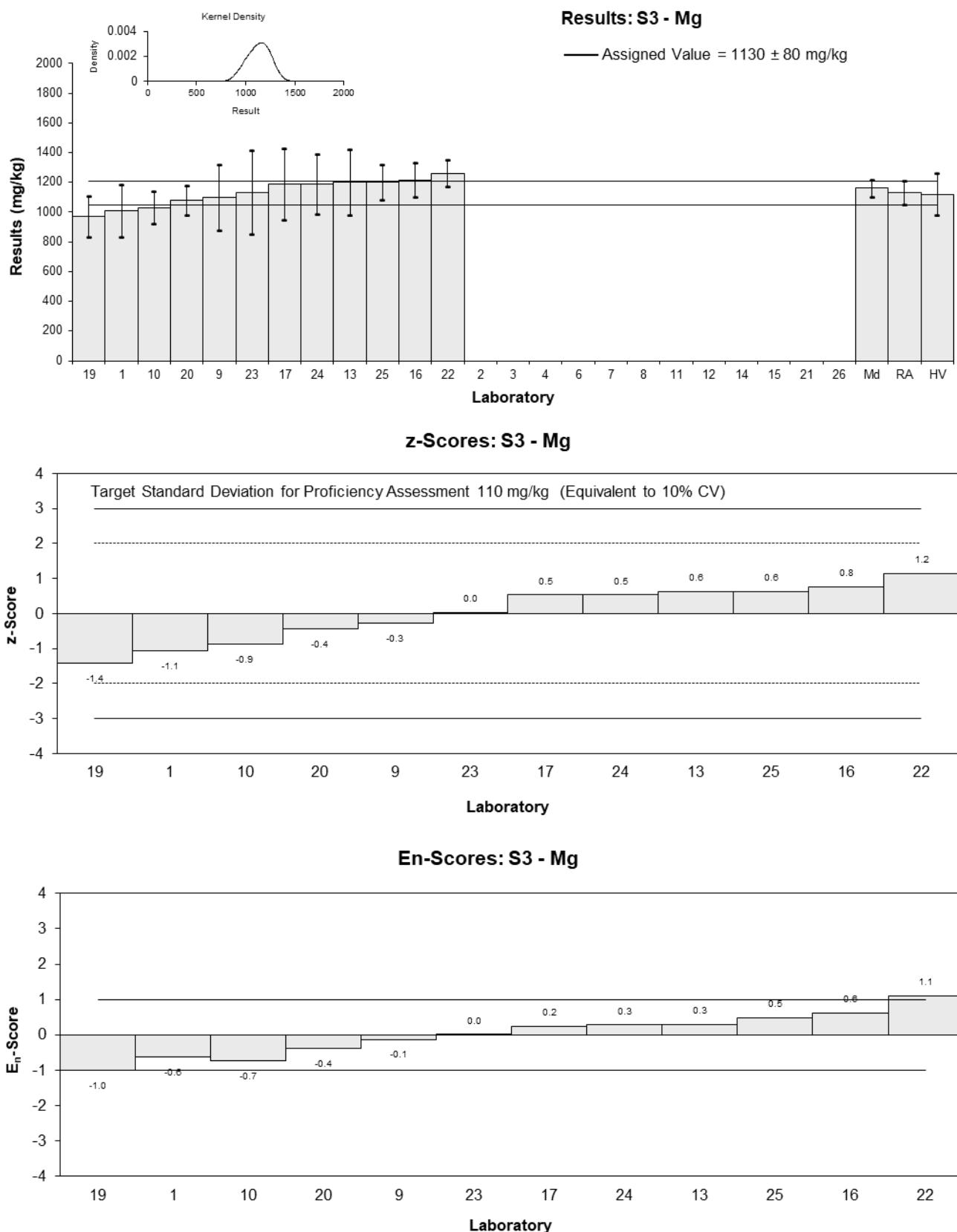


Figure 49

Table 61

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	160	55	-0.20	-0.08
2	NT	NT		
3	NT	NT		
4	NR	NR		
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	120	40	-1.82	-0.94
10	156	30	-0.36	-0.23
11	NT	NT		
12	NT	NT		
13	200	29.9	1.41	0.88
14	NT	NT		
15	NT	NT		
16	186.10	32.09	0.85	0.51
17	NR	NR		
19	150	23	-0.61	-0.43
20	133	15	-1.29	-1.07
21	NT	NT		
22	199	32	1.37	0.82
23	166	42	0.04	0.02
24	133	30	-1.29	-0.81
25	210	21	1.82	1.35
26	NR	NR		

Statistics

Assigned Value	165	26
Spike Value	Not Spiked	
Homogeneity Value	140	17
Robust Average	165	26
Median	160	30
Mean	165	
N	11	
Max	210	
Min	120	
Robust SD	35	
Robust CV	21%	

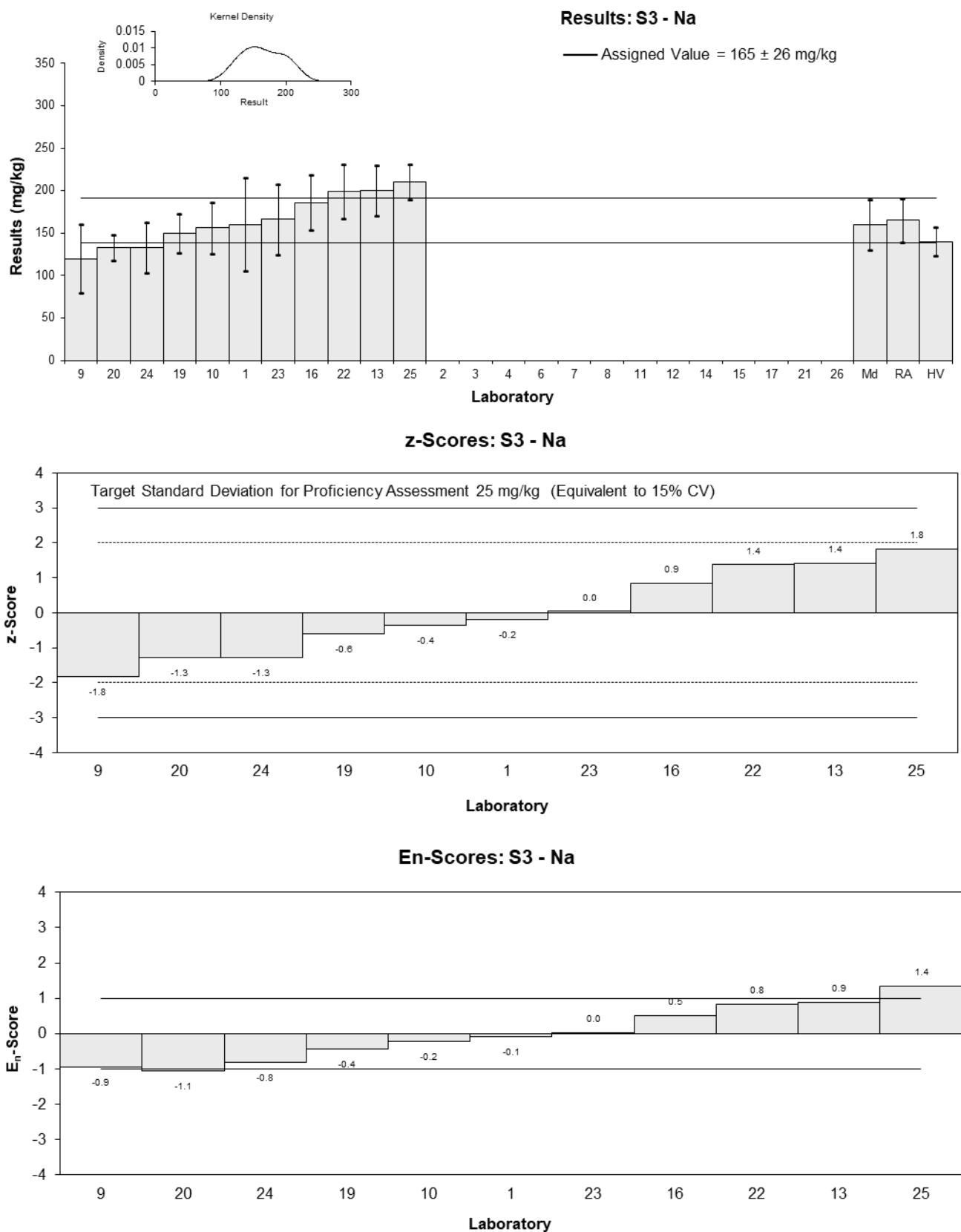


Figure 50

Table 62

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	1820	264	1.10	0.59
2	NT	NT		
3	NT	NT		
4	NR	NR		
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	1500	300	-0.85	-0.42
10	1730	350	0.55	0.24
11	NT	NT		
12	NT	NT		
13	NT	NR		
14	NT	NT		
15	NT	NT		
16	1878	183	1.45	1.01
17	1900	380	1.59	0.64
19	NT	NT		
20	1469	150	-1.04	-0.81
21	1582	303	-0.35	-0.17
22	1520	198	-0.73	-0.48
23	1660	415	0.12	0.05
24	1595	300	-0.27	-0.13
25	1400	NR	-1.46	-1.60
26	NR	NR		

Statistics

Assigned Value	1640	150
Spike Value	Not Spiked	
Homogeneity Value	1700	210
Robust Average	1640	150
Median	1600	140
Mean	1640	
N	11	
Max	1900	
Min	1400	
Robust SD	190	
Robust CV	12%	

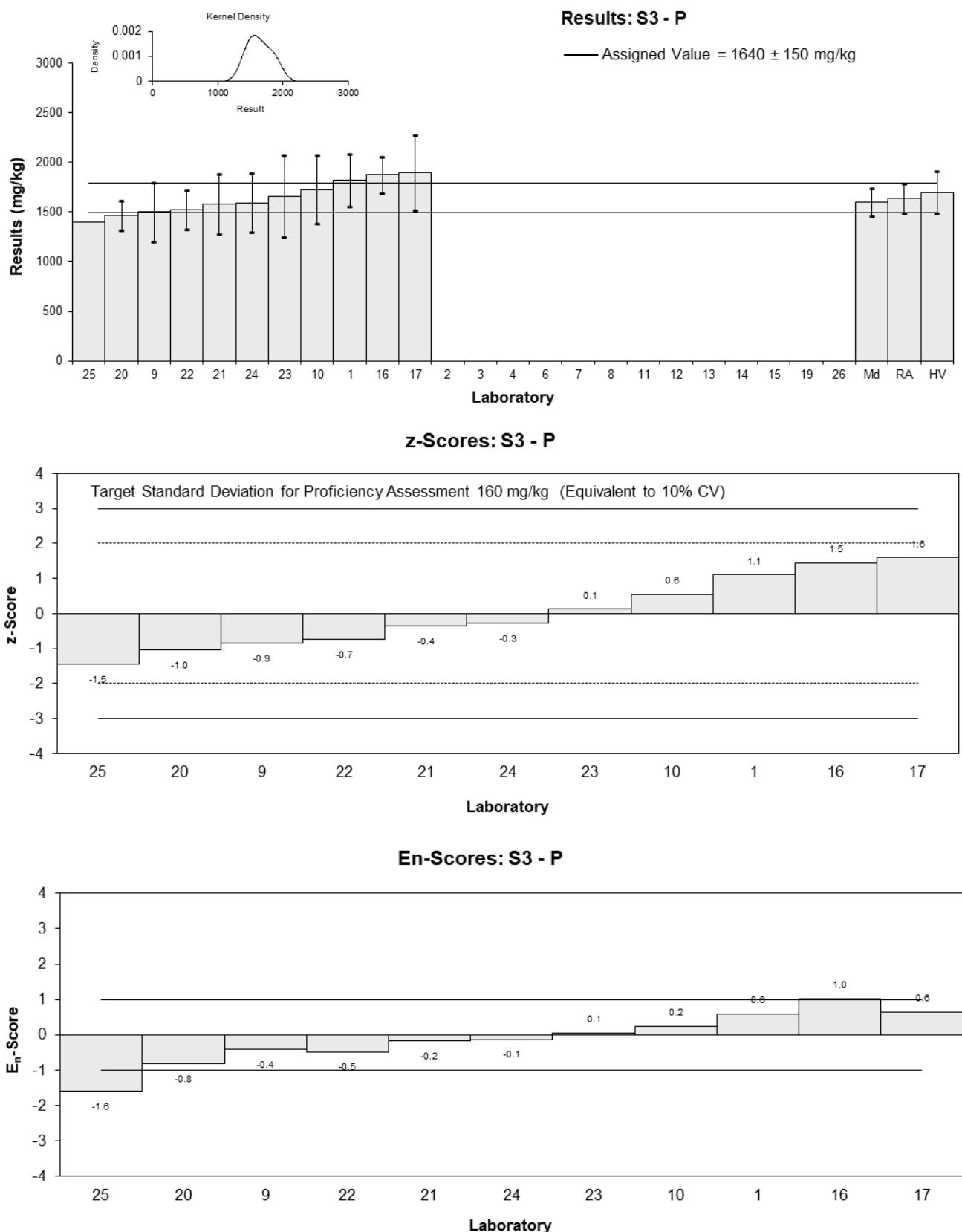


Figure 51

Table 63

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	610	122	0.84	0.47
2	NT	NT		
3	NT	NT		
4	NR	NR		
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	480	100	-0.76	-0.49
10	547	110	0.06	0.04
11	NT	NT		
12	NT	NT		
13	280	NR	-3.22	-3.36
14	NT	NT		
15	NT	NT		
16	602	65.4	0.74	0.59
17	690	140	1.82	0.92
19	NT	NT		
20	545	55	0.04	0.03
21	NT	NT		
22	500	50	-0.52	-0.45
23	410	103	-1.62	-1.02
24	583	90	0.50	0.34
25	603	NR	0.75	0.78
26	NR	NR		

Statistics

Assigned Value	542	78
Spike Value	Not Spiked	
Robust Average	542	78
Median	547	63
Mean	532	
N	11	
Max	690	
Min	280	
Robust SD	100	
Robust CV	19%	

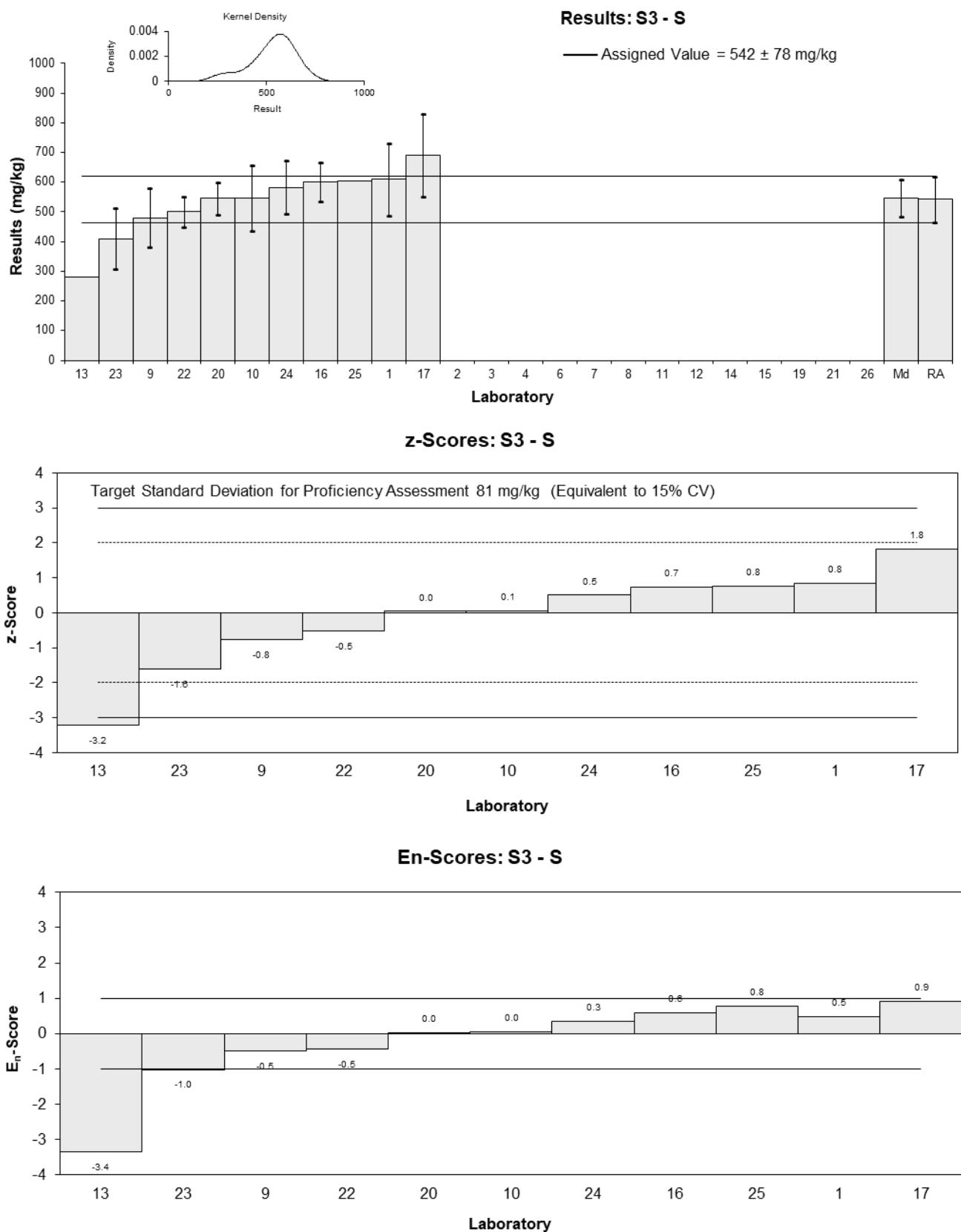


Figure 52

Table 64

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	NT	NT
2	NT	NT
3	NT	NT
4	NR	NR
6	NT	NT
7	NT	NT
8	NR	NR
9	NT	NT
10	NR	NR
11	NT	NT
12	NT	NT
13	NT	NR
14	NT	NT
15	NT	NT
16	NT	NT
17	NR	NR
19	546	165
20	302	30
21	NT	NT
22	310	62
23	NT	NT
24	NR	NR
25	NT	NT
26	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Median	310	17
Mean	390	
N	3	
Max	546	
Min	302	

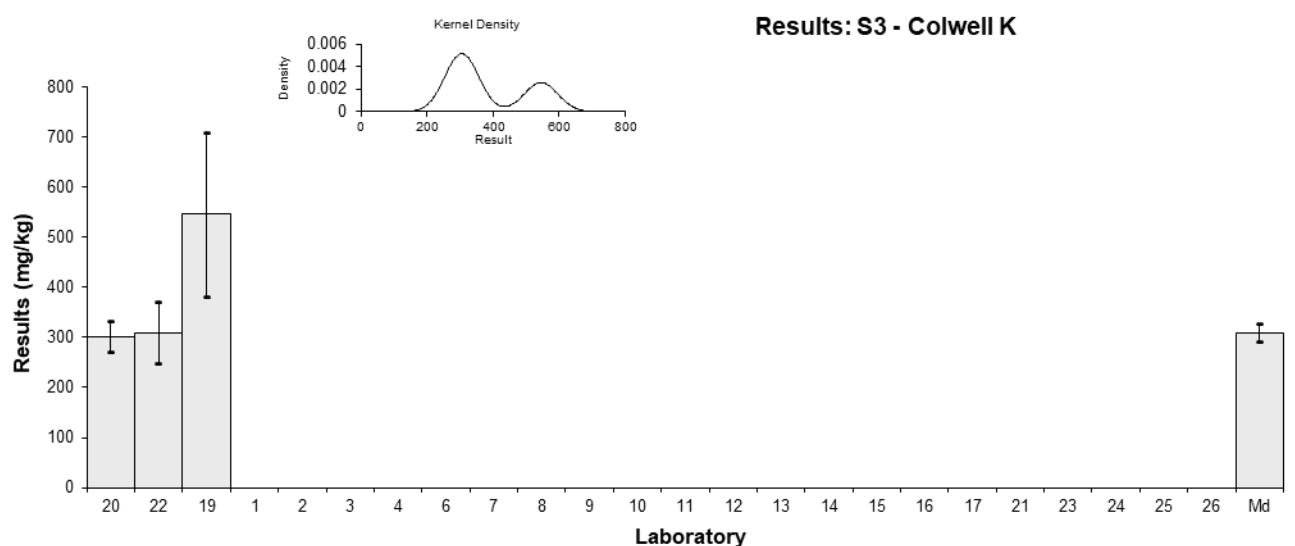


Figure 53

Table 65

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	NT	NT
2	NT	NT
3	NT	NT
4	324	6.0
6	NT	NT
7	NT	NT
8	NR	NR
9	NT	NT
10	NR	NR
11	NT	NT
12	NT	NT
13	NT	NR
14	NT	NT
15	NT	NT
16	NT	NT
17	NR	NR
19	374	72
20	355	35
21	NT	NT
22	NT	NT
23	NT	NT
24	331	60
25	NT	NT
26	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Median	343	29
Mean	346	
N	4	
Max	374	
Min	324	

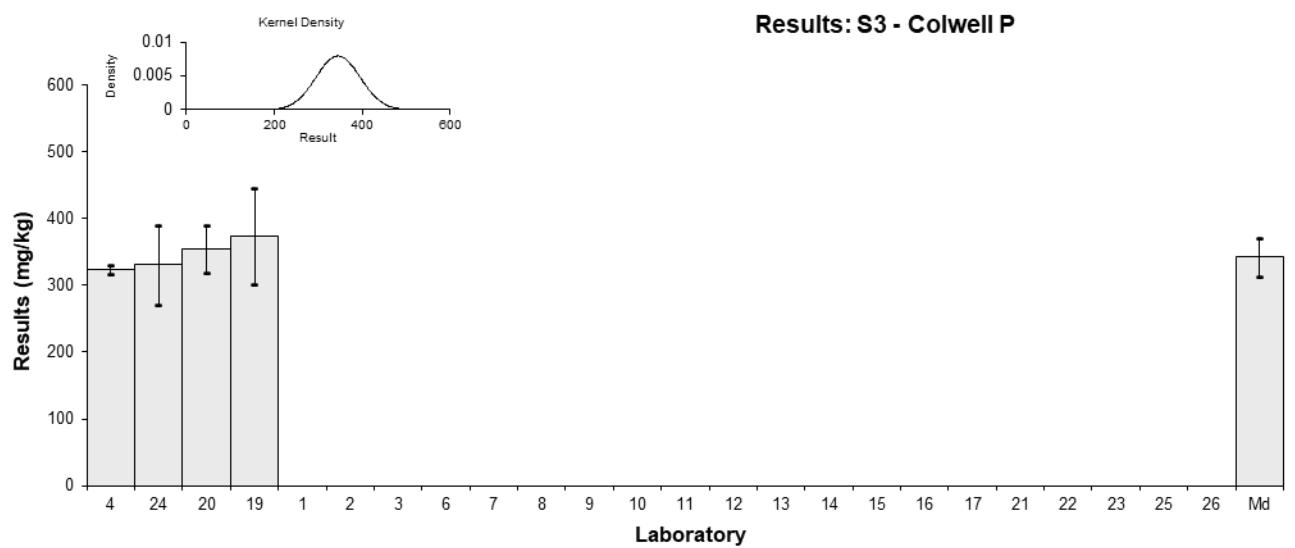


Figure 54

Table 66

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	186	10.41	0.00	0.00
2	NT	NT		
3	163	0.2	-1.24	-1.35
4	178	20	-0.43	-0.30
6	196	8	0.54	0.53
7	113.2	17.66	-3.91	-2.97
8	NR	NR		
9	130	26	-3.01	-1.80
10	209	20	1.24	0.88
11	NT	NT		
12	NT	NT		
13	209	19.45	1.24	0.89
14	NT	NT		
15	NT	NT		
16	228.7	5.2	2.30	2.40
17	210	30	1.29	0.70
19	186	5	0.00	0.00
20	182	15	-0.22	-0.18
21	151.74	10.14	-1.84	-1.73
22	175	35	-0.59	-0.28
23	189.6	56.88	0.19	0.06
24	200	20	0.75	0.53
25	210	NR	1.29	1.41
26	NR	NR		

Statistics

Assigned Value	186	17
Spike Value	Not Spiked	
Robust Average	186	17
Median	186	21
Mean	183	
N	17	
Max	228.7	
Min	113.2	
Robust SD	28	
Robust CV	15%	

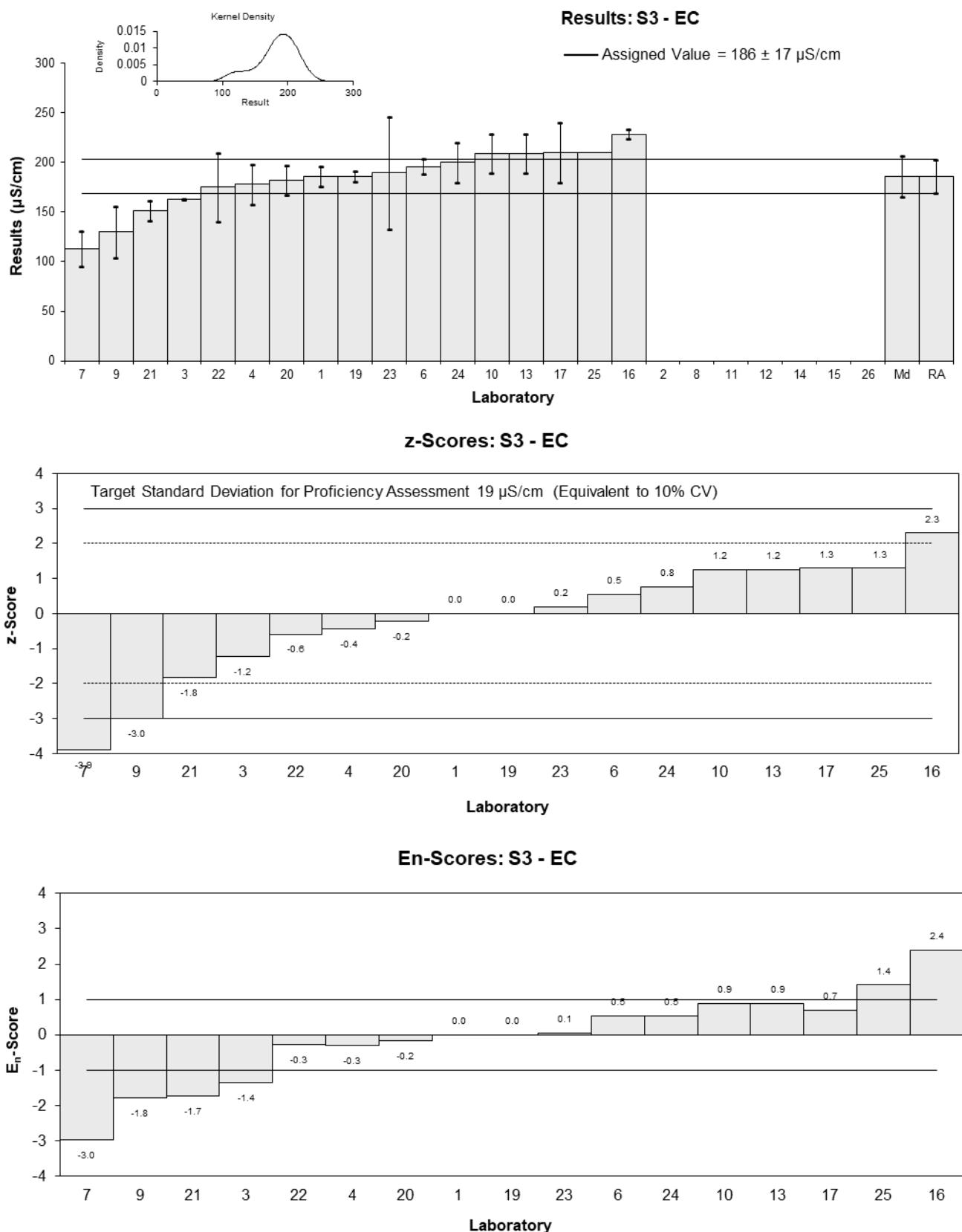


Figure 55

Table 67

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	16.1	2.75	1.03	0.49
2	NT	NT		
3	NT	NT		
4	15.13	0.56	0.36	0.35
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	NT	NT		
10	14.0	2.0	-0.41	-0.25
11	NT	NT		
12	NT	NT		
13	15.7	1.14	0.75	0.61
14	NT	NT		
15	NT	NT		
16	15.09	1.76	0.34	0.22
17	NR	NR		
19	14.1	1.6	-0.34	-0.24
20	12.6	1.5	-1.37	-0.97
21	NT	NT		
22	17.9	2.1	2.26	1.31
23	14.0	3.5	-0.41	-0.16
24	12.2	2	-1.64	-0.98
25**	0.1	NR	-9.93	-10.36
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	14.6	1.4
Spike Value	Not Spiked	
Robust Average	14.6	1.4
Median	14.6	1.0
Mean	14.7	
N	10	
Max	17.9	
Min	12.2	
Robust SD	1.8	
Robust CV	12%	

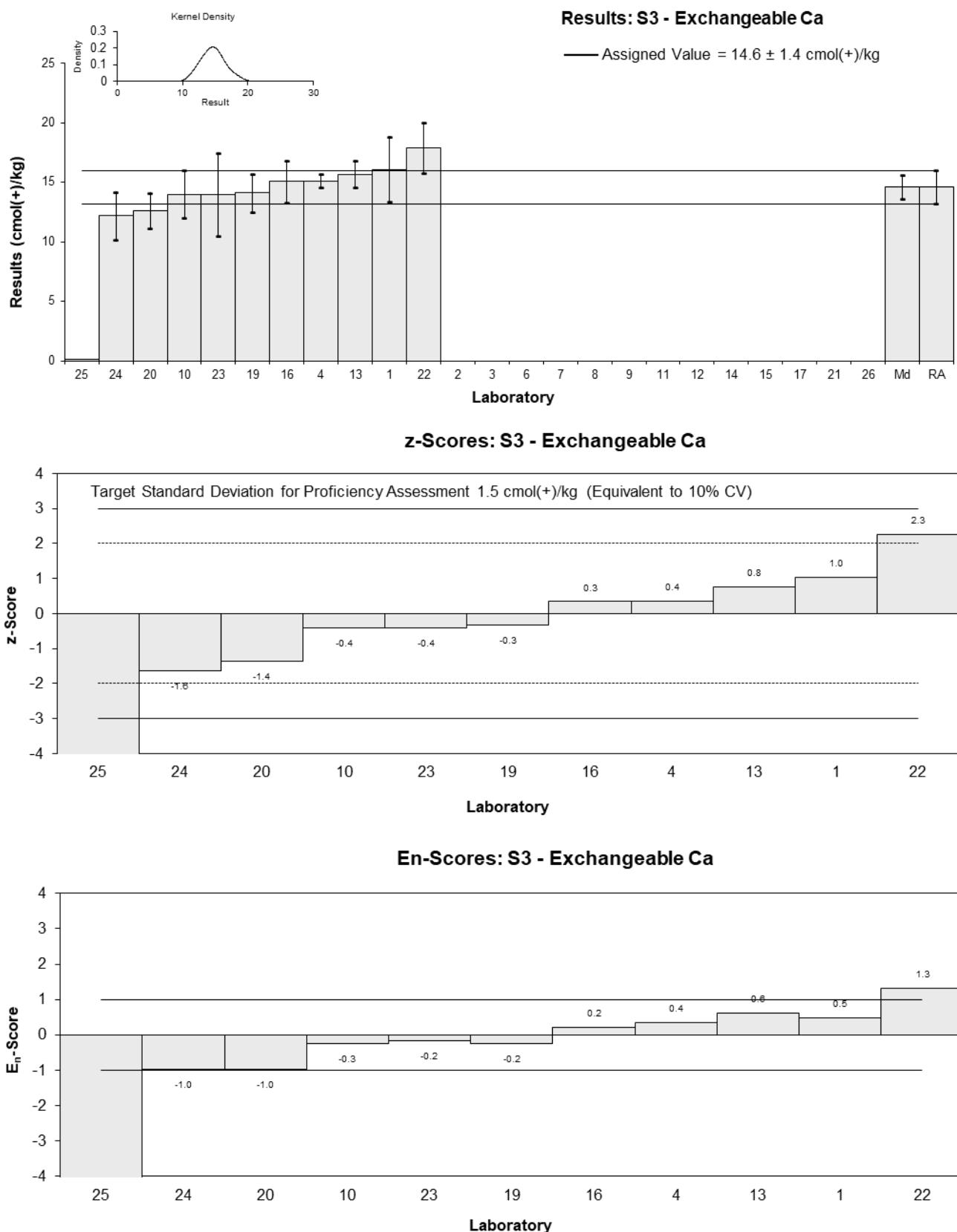


Figure 56

Table 68

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	0.8	0.2	1.55	0.69
2	NT	NT		
3	NT	NT		
4	0.65	0.07	0.01	0.01
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	NT	NT		
10	0.64	0.15	-0.09	-0.05
11	NT	NT		
12	NT	NT		
13	0.8	0.06	1.55	1.44
14	NT	NT		
15	NT	NT		
16	0.664	0.087	0.15	0.12
17	NR	NR		
19	0.7	0.1	0.52	0.39
20	0.55	0.06	-1.02	-0.94
21	NT	NT		
22	0.54	0.09	-1.12	-0.88
23	0.548	0.14	-1.04	-0.61
24	0.60	1	-0.50	-0.05
25**	0.05	NR	-6.15	-6.97
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	0.649	0.086
Spike Value	Not Spiked	
Robust Average	0.649	0.086
Median	0.645	0.088
Mean	0.649	
N	10	
Max	0.8	
Min	0.54	
Robust SD	0.11	
Robust CV	17%	

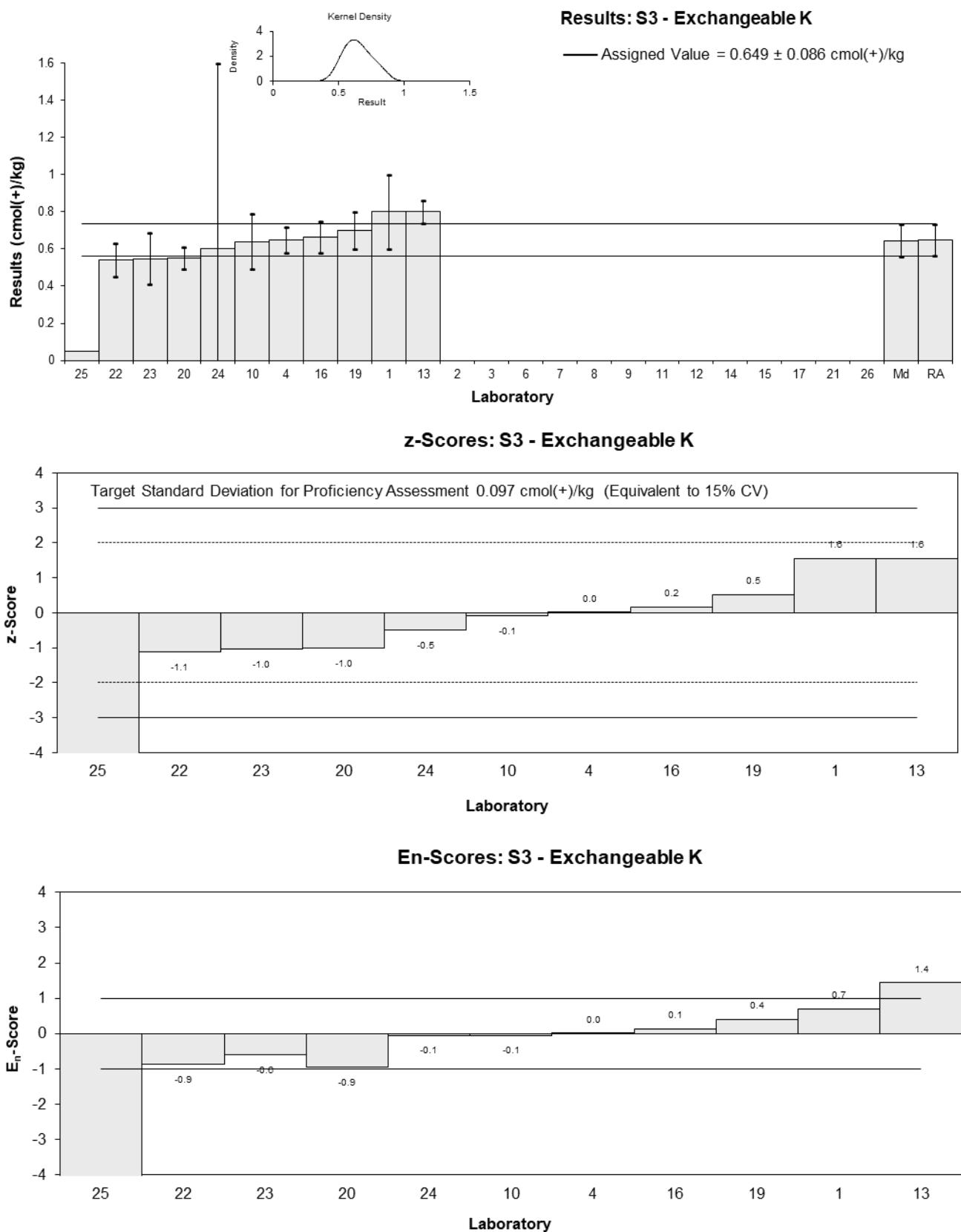


Figure 57

Table 69

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	3.0	0.44	0.68	0.38
2	NT	NT		
3	NT	NT		
4	2.88	0.34	0.25	0.17
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	NT	NT		
10	2.7	0.3	-0.39	-0.29
11	NT	NT		
12	NT	NT		
13	3.1	0.21	1.03	0.91
14	NT	NT		
15	NT	NT		
16	2.75	0.42	-0.21	-0.12
17	NR	NR		
19	2.9	0.3	0.32	0.23
20	2.47	0.3	-1.21	-0.88
21	NT	NT		
22	3.2	0.3	1.39	1.02
23	2.36	0.59	-1.60	-0.71
24	2.70	5	-0.39	-0.02
25**	0.2	NR	-9.29	-10.88
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	2.81	0.24
Spike Value	Not Spiked	
Robust Average	2.81	0.24
Median	2.82	0.18
Mean	2.81	
N	10	
Max	3.2	
Min	2.36	
Robust SD	0.3	
Robust CV	11%	

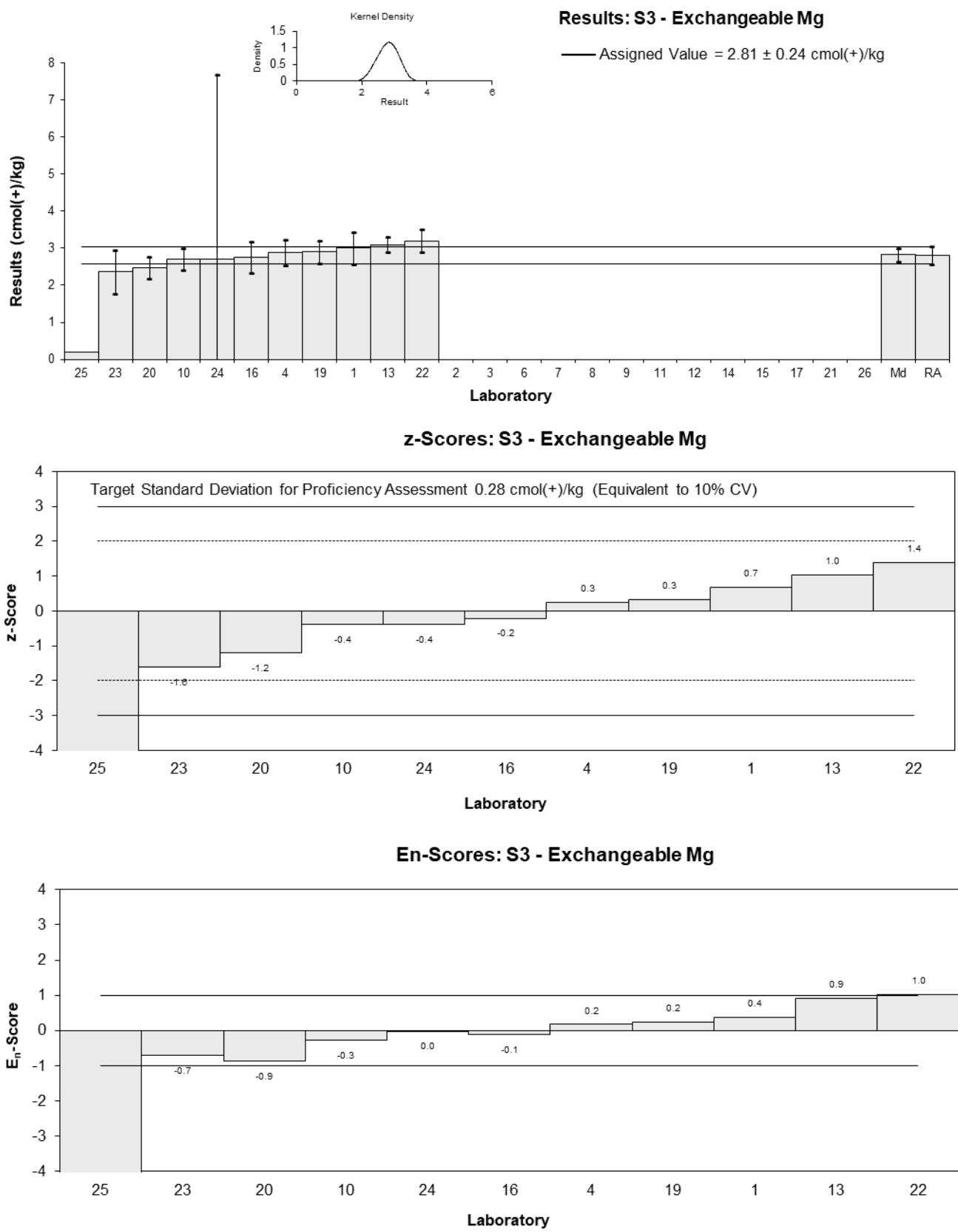


Figure 58

Table 70

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	0.2	0.07	0.43	0.21
2	NT	NT		
3	NT	NT		
4	0.15	0.18	-0.92	-0.19
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	NT	NT		
10	0.21	0.05	0.71	0.45
11	NT	NT		
12	NT	NT		
13	0.2	0.012	0.43	0.50
14	NT	NT		
15	NT	NT		
16	0.184	0.022	0.00	0.00
17	NR	NR		
19	0.2	0.1	0.43	0.15
20	0.14	0.02	-1.20	-1.22
21	NT	NT		
22	0.18	0.04	-0.11	-0.08
23	0.138	0.035	-1.25	-1.00
24	0.27	0.1	2.34	0.82
25	<0.17	NR		
26	NR	NR		

Statistics

Assigned Value	0.184	0.030
Spike Value	Not Spiked	
Robust Average	0.184	0.030
Median	0.192	0.018
Mean	0.187	
N	10	
Max	0.27	
Min	0.138	
Robust SD	0.038	
Robust CV	20%	

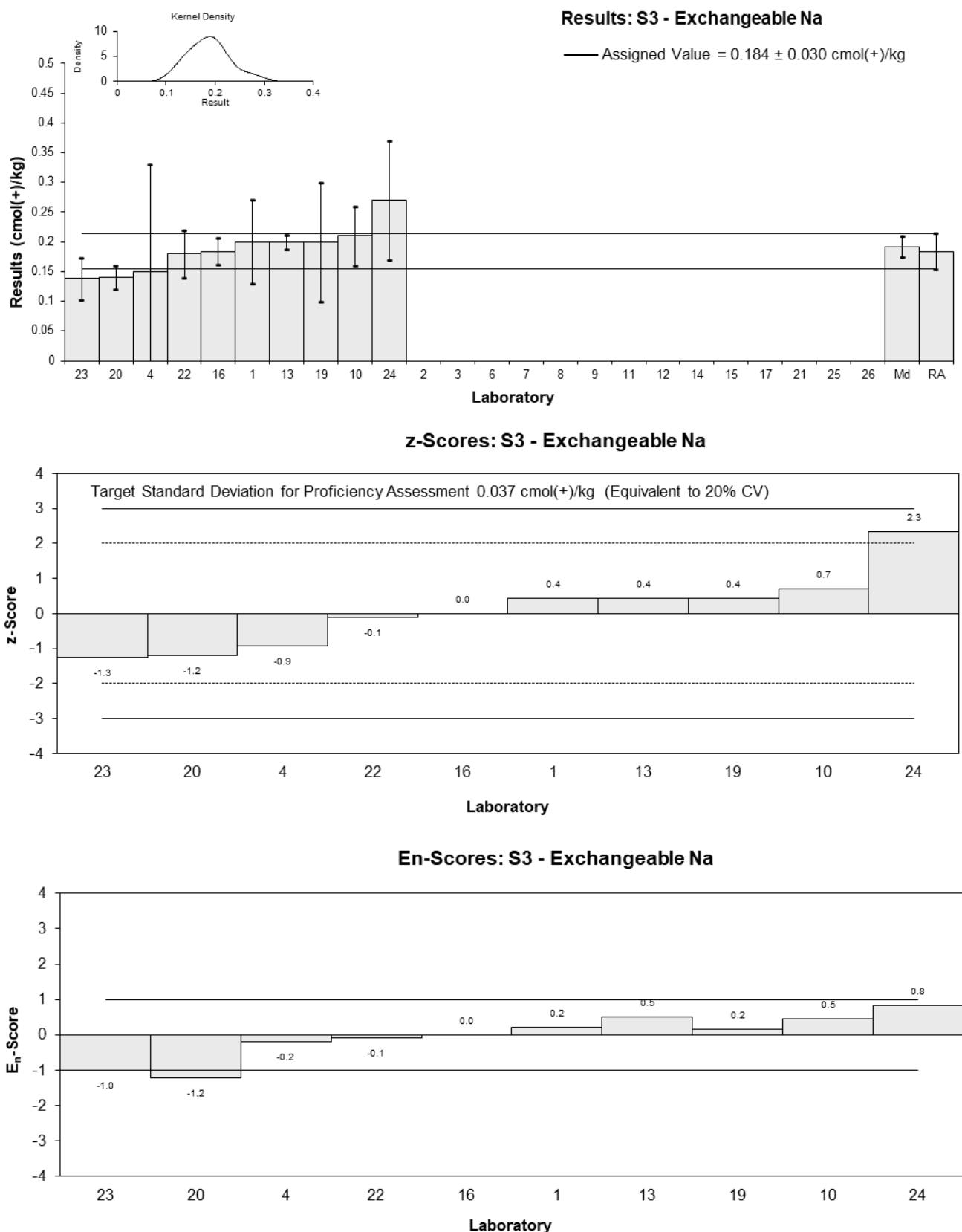


Figure 59

Table 71

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	NT	NT
2	NT	NT
3	NT	NT
4	NR	NR
6	NT	NT
7	NT	NT
8	NR	NR
9	NT	NT
10	NR	NR
11	NT	NT
12	NT	NT
13	<0.2	NR
14	NT	NT
15	NT	NT
16	NT	NT
17	NR	NR
19	0.9	0.1
20	2.95	0.3
21	NT	NT
22	NT	NT
23	NT	NT
24	1.45	0.4
25	NT	NT
26	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Median	1.5	1.2
Mean	1.8	
N	3	
Max	2.95	
Min	0.9	

Results: S3 - Extractable B

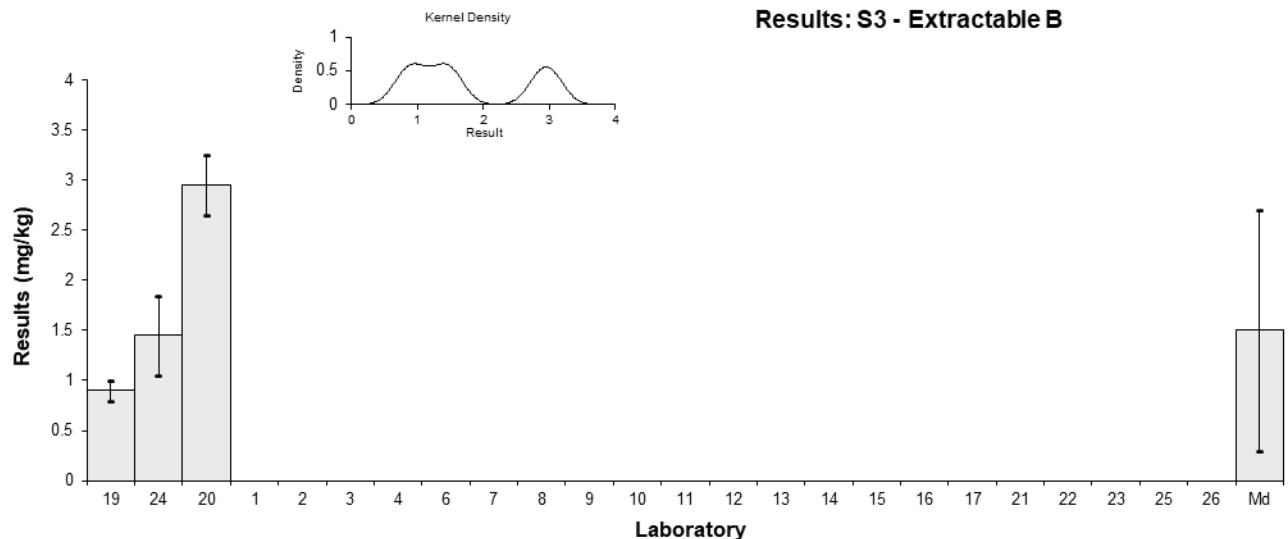


Figure 60

Table 72

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	NT	NT
2	NT	NT
3	NT	NT
4	NR	NR
6	NT	NT
7	NT	NT
8	NR	NR
9	NT	NT
10	NR	NR
11	NT	NT
12	NT	NT
13	NT	NR
14	NT	NT
15	NT	NT
16	NT	NT
17	NR	NR
19	NT	NT
20	163	20
21	NT	NT
22	NT	NT
23	NT	NT
24	169.5	34
25	NT	NT
26	NR	NR

Statistics

Assigned Value	Not Set	
Spike Value	Not Spiked	
Mean	166	
N	2	

Results: S3 - PBI

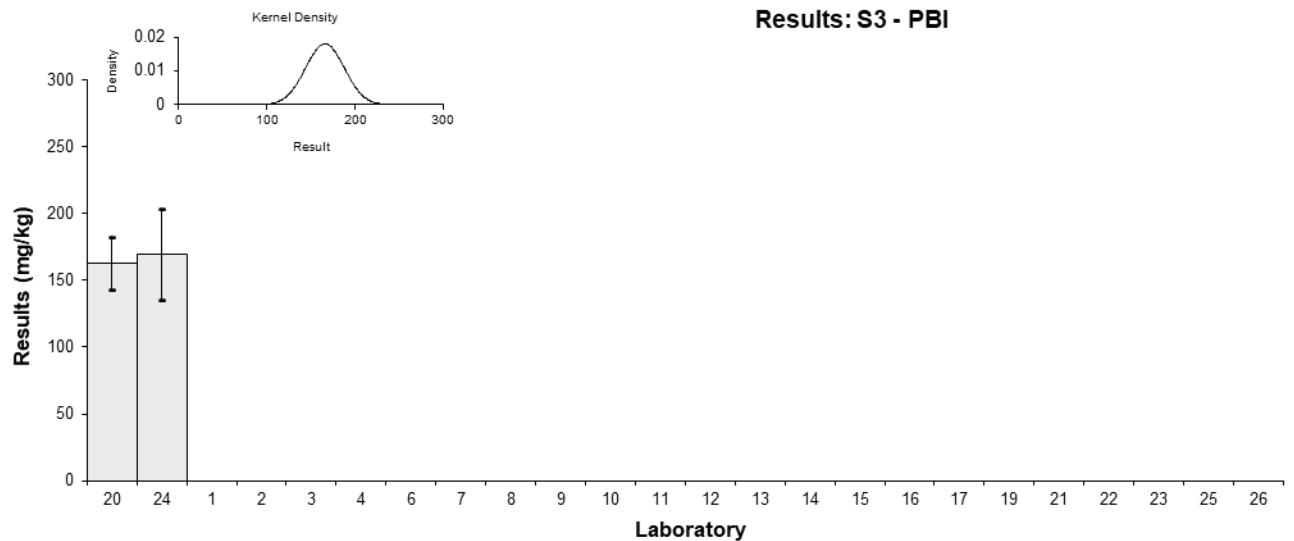


Figure 61

Table 73

Sample Details

Sample No.	S3
Matrix	Soil
Analyte	pH

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	5.4	0.06	1.16	0.96
2	NT	NT		
3	5.50	0.4	1.71	0.69
4	5.43	0.08	1.32	1.07
6	5.3	0.1	0.61	0.47
7	4.91	0.15	-1.54	-1.08
8	NR	NR		
9	5.5	0.3	1.71	0.85
10	4.76	0.2	-2.37	-1.48
11	NT	NT		
12	NT	NT		
13	5.45	0.15	1.43	1.01
14	NT	NT		
15	NT	NT		
16	5.63	0.05	2.42	2.04
17	5.0	0.1	-1.05	-0.82
19	4.87	0.05	-1.76	-1.48
20	4.95	0.2	-1.32	-0.83
21	4.92	0.15	-1.49	-1.05
22	4.9	0.2	-1.60	-1.00
23	5.5	0.2	1.71	1.07
24	5.07	0.5	-0.66	-0.22
25	NT	NT		
26	NR	NR		

Statistics

Assigned Value	5.19	0.21
Spike Value	Not Spiked	
Robust Average	5.19	0.21
Median	5.19	0.25
Mean	5.19	
N	16	
Max	5.63	
Min	4.76	
Robust SD	0.33	
Robust CV	6.4%	

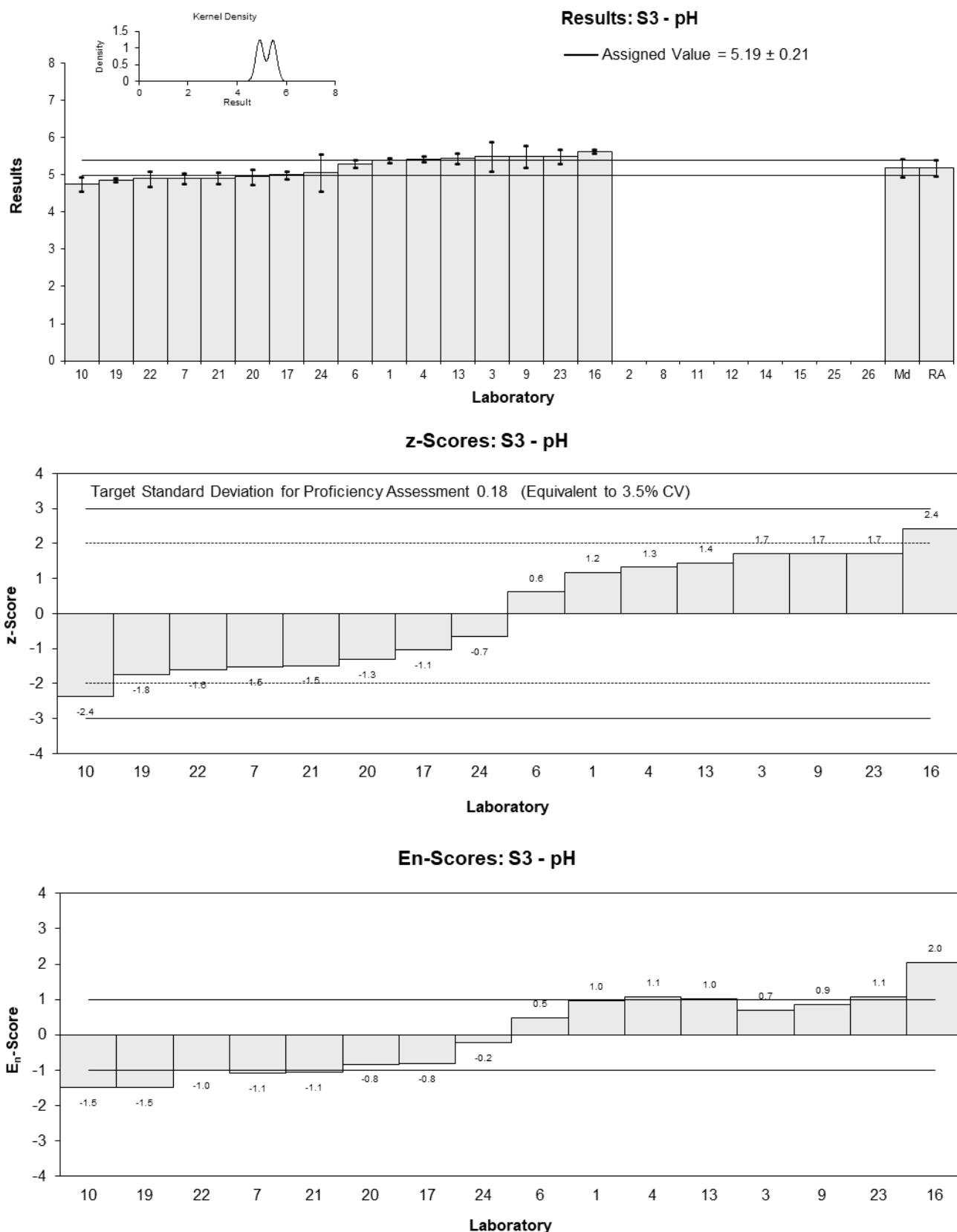


Figure 62

Table 74

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	NT	NT		
2	NT	NT		
3	NT	NT		
4	75950	800	0.09	0.10
6	NT	NT		
7**	8.90	0.124	-10.00	-11.41
8	68244	12149.7646	-0.94	-0.51
9	NT	NT		
10	NR	NR		
11	NT	NT		
12	NT	NT		
13	NT	NR		
14	NT	NT		
15	NT	NT		
16	NT	NT		
17	75000	11000	-0.04	-0.02
19	75400	2250	0.01	0.01
20	83600	8360	1.10	0.78
21	85709	11047.89	1.38	0.81
22	76100	5600	0.11	0.09
23	81970	24591	0.89	0.26
24	59500	6000	-2.10	-1.77
25	NT	NT		
26	67890	8993.3579	-0.98	-0.66

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	75300	6600
Spike Value	Not Spiked	
Robust Average	75300	6600
Median	75700	8000
Mean	74900	
N	10	
Max	85709	
Min	59500	
Robust SD	8300	
Robust CV	11%	

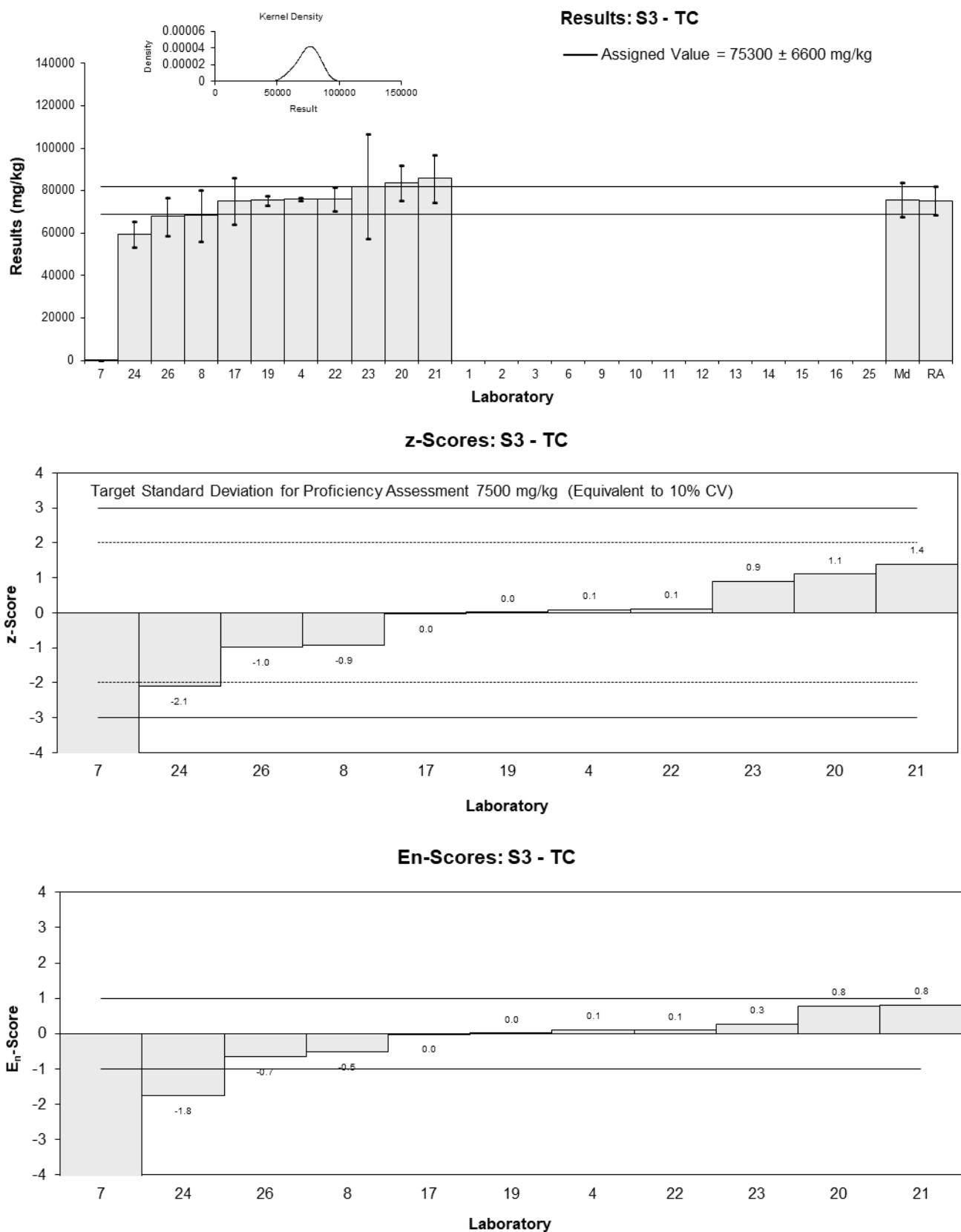


Figure 63

Table 75

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	4320	1770	-0.57	-0.14
2	NT	NT		
3	NT	NT		
4	4700	100	0.26	0.27
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	5200	1040	1.35	0.55
10	4700	600	0.26	0.16
11	NT	NT		
12	NT	NT		
13	5280	NR	1.53	1.63
14	NT	NT		
15	NT	NT		
16	4291.0	1068.3	-0.63	-0.25
17	5200	780	1.35	0.70
19	4100	846	-1.05	-0.51
20	4825	483	0.53	0.38
21	NT	NT		
22	4740	520	0.35	0.24
23	NT	NT		
24	3900	400	-1.48	-1.16
25*	710	NR	-8.45	-9.00
26	2290	421.1508	-5.00	-3.80

* Outlier, see Section 4.2

Statistics

Assigned Value	4580	430
Spike Value	Not Spiked	
Robust Average	4460	510
Median	4700	510
Mean	4170	
N	13	
Max	5280	
Min	710	
Robust SD	730	
Robust CV	16%	

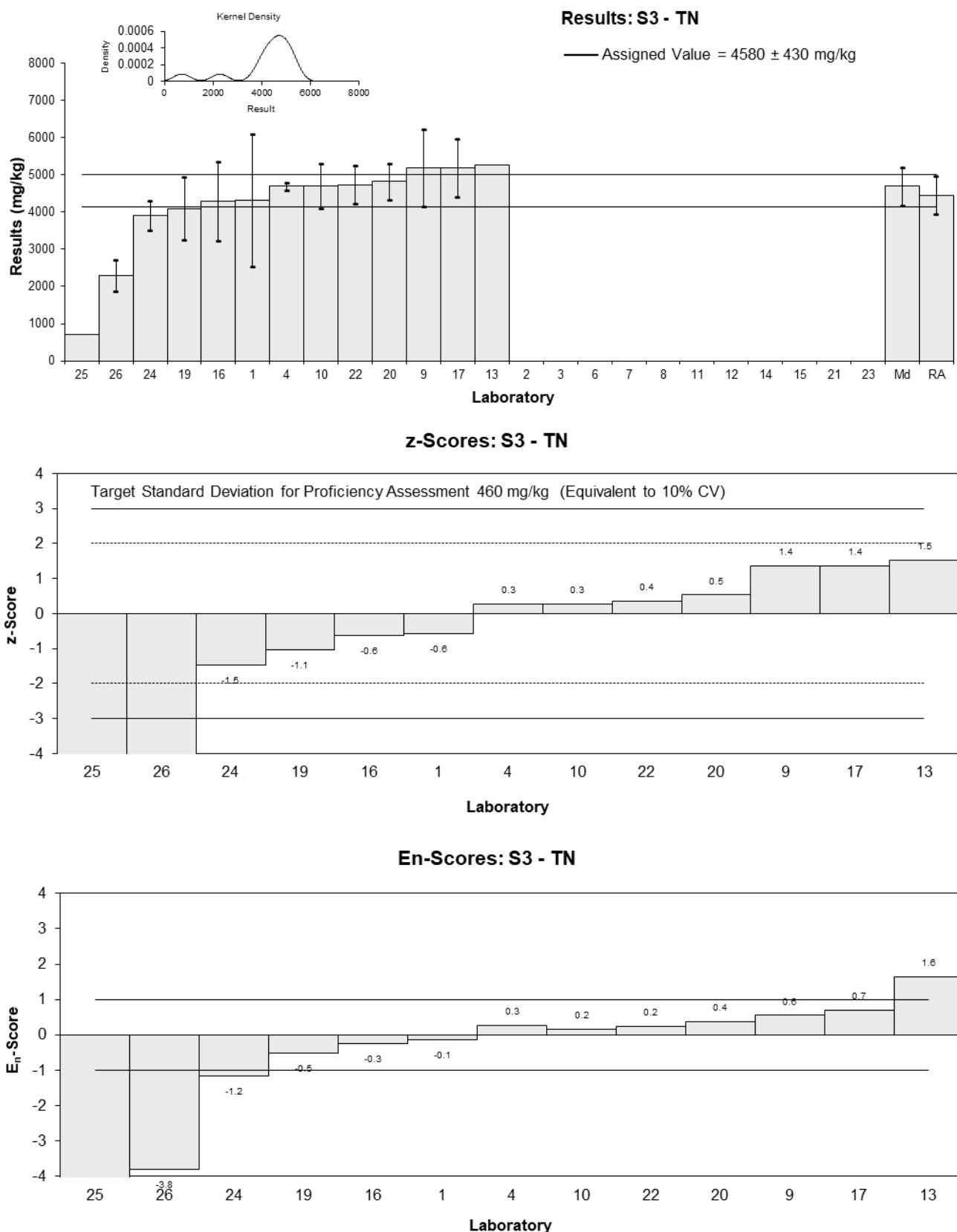


Figure 64

Table 76

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1**	7.4	1.48	-10.00	-17.30
2	NT	NT		
3	NT	NT		
4	75950	800	0.21	0.35
6	NT	NT		
7**	8.21	0.115	-10.00	-17.30
8	68721	22493.2282	-0.76	-0.25
9	NT	NT		
10	77200	13000	0.38	0.20
11	NT	NT		
12	NT	NT		
13	71000	NR	-0.46	-0.79
14	NT	NT		
15	NT	NT		
16	71900	13157	-0.34	-0.18
17	74000	11000	-0.05	-0.03
19	71700	2700	-0.36	-0.53
20	83600	8360	1.24	0.98
21	79021	10185.81	0.62	0.42
22	72500	7250	-0.26	-0.23
23	81600	24480	0.97	0.29
24	59500	12000	-2.00	-1.17
25	NT	NT		
26	NR	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	74400	4300
Spike Value	Not Spiked	
Robust Average	74400	4300
Median	73300	3600
Mean	73900	
N	12	
Max	83600	
Min	59500	
Robust SD	6000	
Robust CV	8%	

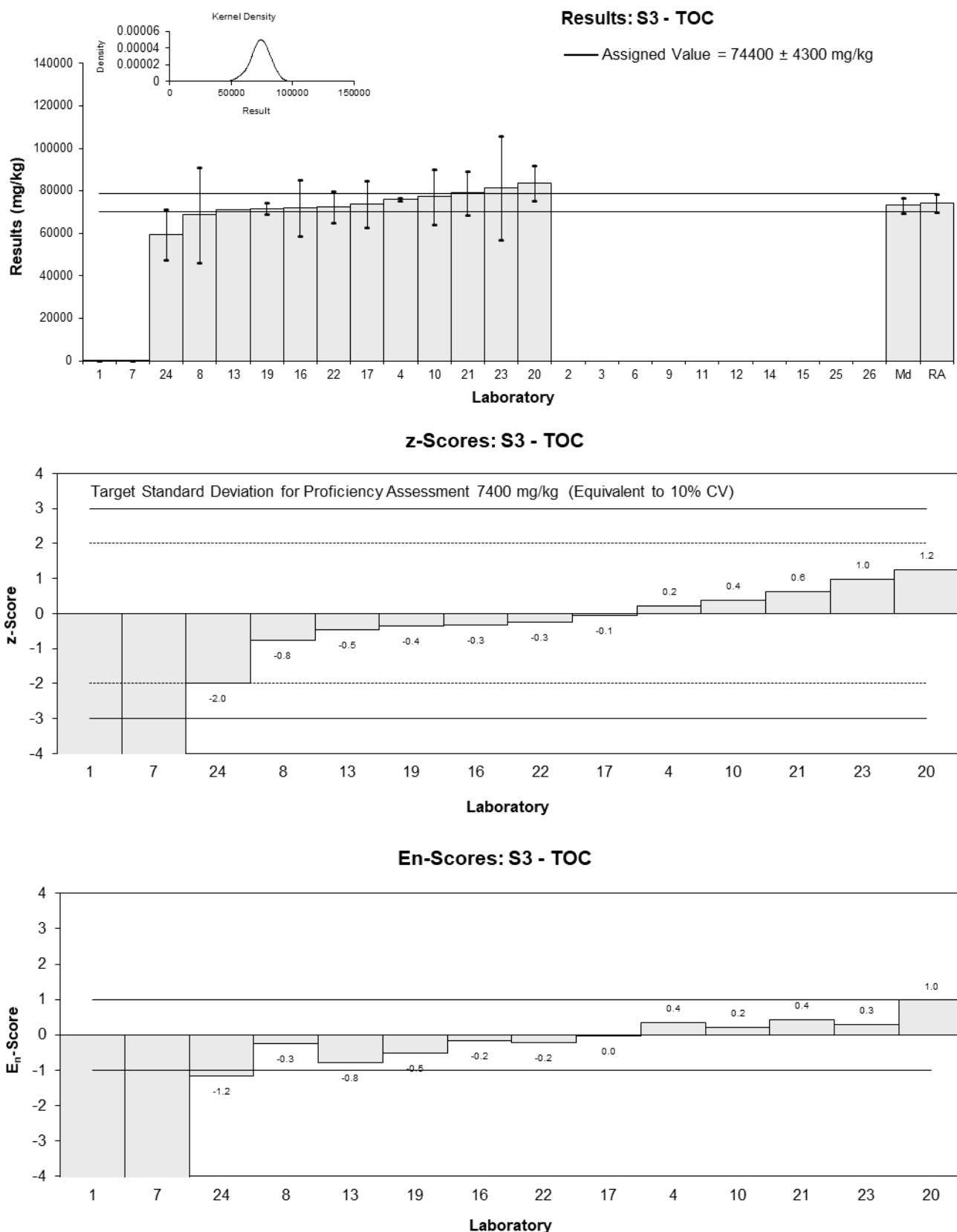


Figure 65

Table 77

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	1590	432	-0.61	-0.32
2	NT	NT		
3	NT	NT		
4	NR	NR		
6	NT	NT		
7	NT	NT		
8	NR	NR		
9	NT	NT		
10	1700	220	-0.19	-0.15
11	NT	NT		
12	NT	NT		
13	2270	644	1.98	0.75
14	NT	NT		
15	NT	NT		
16	1566.73	363.8	-0.70	-0.42
17	NR	NR		
19	1650	256	-0.38	-0.28
20	NR	NR		
21	NT	NT		
22	1900	287	0.57	0.39
23	NT	NT		
24	NR	NR		
25*	300	NR	-5.52	-5.80
26	NR	NR		

* Outlier, see Section 4.2

Statistics

Assigned Value	1750	250
Spike Value	Not Spiked	
Robust Average	1670	400
Median	1650	120
Mean	1570	
N	7	
Max	2270	
Min	300	
Robust SD	420	
Robust CV	25%	

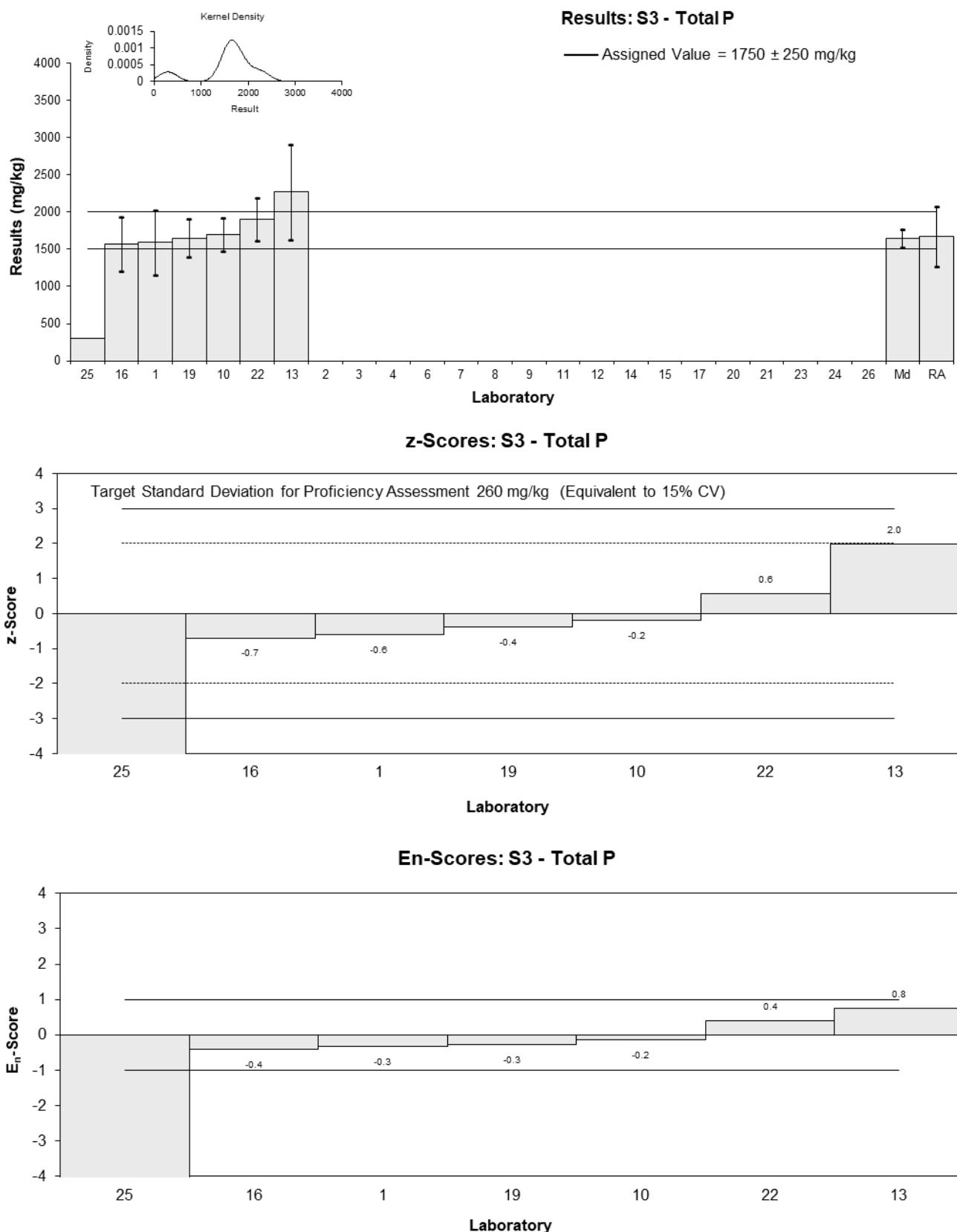


Figure 66

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 the same soil material as that used in the preparation of Sample S1 of PT study AQA 23-16, to which a known amount of water was added. Participants were asked to use their normal analytical method but to report results corrected for moisture content.⁶

Sample S3 was dried agricultural soil.

Assigned values for 52 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 13528.⁷ Extreme outliers and results less than 50% or more than 150% of the robust average were excluded prior to the calculation of each assigned value (see subchapters 4.2 and 4.3). Appendix 2 sets out the calculation of the robust average of As in Sample S1 and its associated uncertainty.

The results reported by Laboratory 15 in Sample S2 were all approximately double that of the robust average of participants' results, 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.

No assigned value was set for B, Rb, Sb and Se in S1, Cs, Gd, La, Sm and U in S2 and Colwell K, Colwell P, Extractable B and PBI in S3 because the results were too few or too variable. However, participants may still compare their reported results for some of 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.

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 868 numerical results, 833 (96%) were reported with an expanded measurement uncertainty. The magnitude of these expanded uncertainties was within the range 0.0009% to 217% 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.^{10–16}

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 66). 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, 21 laboratories reported results for Cr in S1. The uncertainty of the

assigned value estimated from the robust standard deviation of the 21 laboratories' results is 3.4 mg/kg or 8.8% (see equation 4, Appendix 2). Laboratories 3, 6 and 24 may have under-estimated their expanded measurement uncertainties for Cr in S1 reported as 2.8%, 0.4% and 1.8% respectively, as an uncertainty estimated from one measurement cannot be smaller than the uncertainty estimated from 21 measurements. Alternatively, estimates of uncertainties for Be in S1 larger than 0.274 mg/kg or 35% (the uncertainty of the assigned value of 0.06 mg/kg plus the allowable variation from the assigned value, the target standard deviation of 0.107 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 laboratory 10 for Be in S1 of 0.5 mg/kg (60%), may have been over-estimated.

Laboratory 3 should review their procedure for estimating measurement uncertainty as some of their estimated uncertainties may be under-estimated (between 0.0009% and 0.74% of the reported results).

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.¹¹ An example of estimating measurement uncertainty using proficiency testing data only is given in Appendix 3.

Laboratories 1, 3 and 10 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.¹⁰

Laboratories 4, 12, 15, 19 and 24 reported an estimate of expanded uncertainty for some measurement results 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.¹⁰

For consistency the results of “<.2 mg/kg” reported by Laboratory 13 for Extractable B has been transcribed as “<0.2 mg/kg”.

6.3 z-Score

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

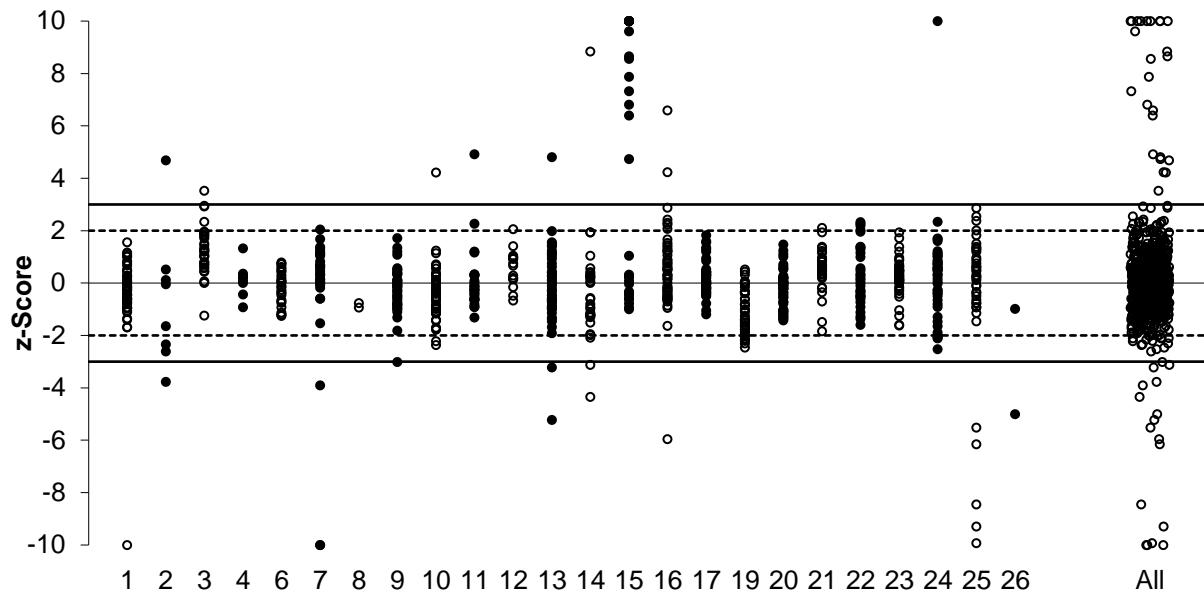
The target standard deviation defines acceptable performance in a proficiency test. Target standard deviations equivalent to 3.5% to 20% PCV were used to calculate z-scores. Unlike the standard deviation based on between laboratories CV, setting the target standard deviation as a realistic, set value enables z-scores to be used as a fixed reference value point for assessment of laboratory performance, independent of group performance.

The between laboratory coefficient of variation predicted by the Thompson-Horwitz equation⁸ and the between laboratory coefficient of variation resulted in this study are presented for comparison in Table 78. The dispersal of participants' z-scores is presented in Figure 67 (by laboratory code) and in Figure 69 (by test). Of 787 results for which z-scores were calculated, 709 (90%) returned an acceptable score of $|z| \leq 2.0$ and 36 (5%) 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 70. No laboratory reported results for all 52 tests for which a z-score was calculated.

Laboratory 1 returned the highest number of acceptable z-scores (49 out of 50 reported).

Laboratory **20** returned acceptable results for 49 results out of a total of 49 reported.



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

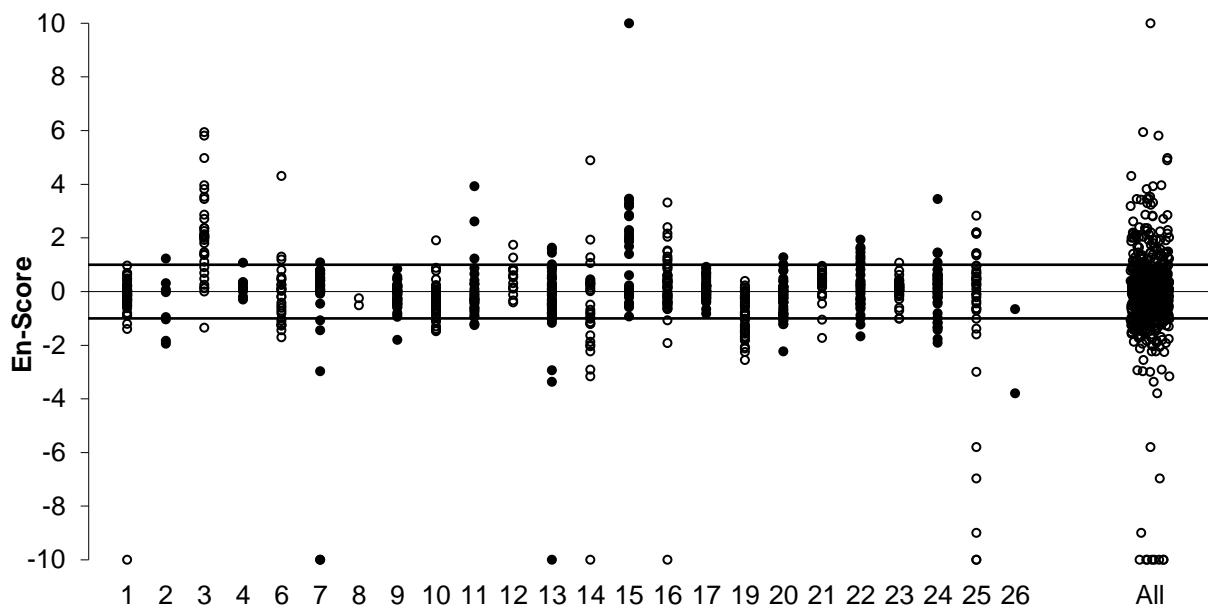
Figure 67 z-Score Dispersal by Laboratory

Table 78 Between-Laboratory CV of this Study, Thompson/Horwitz CV, and Set Target CV

Sample	Test	Assigned Value (mg/kg)	Between Laboratories CV*	Thompson/ Horwitz CV	Target SD (as PCV)
S1	As	35.2	10%	9.4%	10%
S1	B	Not Set	61%	NA	Not Set
S1	Be	1.07	7.3%	16%	10%
S1	Cd	0.94	23%	16%	15%
S1	Co	16.8	11%	10%	15%
S1	Cr	38.5	16%	9.2%	15%
S1	Cu	32.6	8.6%	9.5%	10%
S1	Ga	4.5	26%	13%	20%
S1	Hg	0.615	10%	17%	15%
S1	Li	28.6	17%	9.7%	15%
S1	Mn	672	8.8%	6%	10%
S1	Mo	4.92	11%	13%	15%
S1	Ni	56.2	11%	8.7%	10%
S1	Pb	44.7	16%	9%	15%
S1	Rb	Not Set	62%	NA	Not Set
S1	Sb	Not Set	42%	NA	Not Set
S1	Se	Not Set	107%	NA	Not Set
S1	Sn	5.64	9.6%	12%	10%
S1	Sr	125	10%	7.7%	10%
S1	Th	2.15	16%	14%	15%
S1	V	48.9	17%	8.9%	15%
S1	Zn	204	15%	7.2%	15%
S2	Ag	1.00	4.5%	16%	15%
S2	Al	14900	25%	3.8%	20%
S2	As	12.5	13%	11%	10%
S2	Ba	106	11%	7.9%	10%
S2	Bi	2.88	9.3%	14%	10%
S2	Cd	2.87	7.1%	14%	15%
S2	Cr	21.1	13%	10%	15%
S2	Cs	Not Set	40%	NA	Not Set
S2	Cu	22.8	14%	10%	10%
S2	Gd	Not Set	33%	NA	Not Set

Sample	Test	Assigned Value (mg/kg)	Between Laboratories CV*	Thompson/Horwitz CV	Target SD (as PCV)
S2	Hg	1.99	14%	14%	15%
S2	La	Not Set	47%	NA	Not Set
S2	Mn	561	16%	6.2%	10%
S2	Mo	5.69	15%	12%	15%
S2	Ni	13.9	9.3%	11%	10%
S2	Se	4.65	9.7%	13%	15%
S2	Sm	Not Set	46%	NA	Not Set
S2	Tl	0.825	6.8%	16%	10%
S2	U	Not Set	48%	NA	Not Set
S2	V	32.9	18%	9.5%	15%
S2	Zn	745	8.3%	5.9%	10%
S2	Moisture Content	18.0%	2.4%	2.6%	10%
S3	Ca	5170	8.9%	4.4%	10%
S3	Fe	22000	6.6%	3.6%	10%
S3	K	980	18%	5.7%	15%
S3	Mg	1130	9.2%	5.6%	10%
S3	Na	165	21%	7.4%	15%
S3	P	1640	12%	5.3%	10%
S3	S	542	19%	6.2%	15%
S3	Colwell P	Not Set	7.5%	NA	Not Set
S3	EC	186 µS/cm	15%	7.3%	10%
S3	Exchangeable Ca	14.6 cmol(+)/kg	12%	11%	10%
S3	Exchangeable K	0.649 cmol(+)/kg	17%	17%	15%
S3	Exchangeable Mg	2.81 cmol(+)/kg	11%	14%	10%
S3	Exchangeable Na	0.184 cmol(+)/kg	20%	21%	20%
S3	pH	5.19	6.4%	12%	3.5%
S3	TC	75300	11%	3%	10%
S3	TN	4580	13%	4.5%	10%
S3	TOC	74400	8%	3%	10%
S3	Total P	1750	14%	5.2%	15%

*Robust between Laboratories CV with outliers removed. NA = Not Applicable.



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

Figure 68 En-Score Dispersal by Laboratory

6.4 En-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 unacceptable 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 68. 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 787 results for which E_n -scores were calculated, 599 (76%) returned an acceptable score of $|E_n| < 1$ indicating agreement of the participants' results with the assigned values within their respective expanded measurement uncertainties.

Laboratory 1 had the highest number of acceptable E_n -scores at 47 out of 50 reported.

6.5 Participants' Results and Analytical Methods for Acid Extractable Elements

Sample S1 was dried soil while Sample S2 was moist soil 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. The between-laboratory CVs in the two study samples were comparable.

A summary of participants' results and performance is presented in Tables 79 to 81 and in Figures 67 to 70.

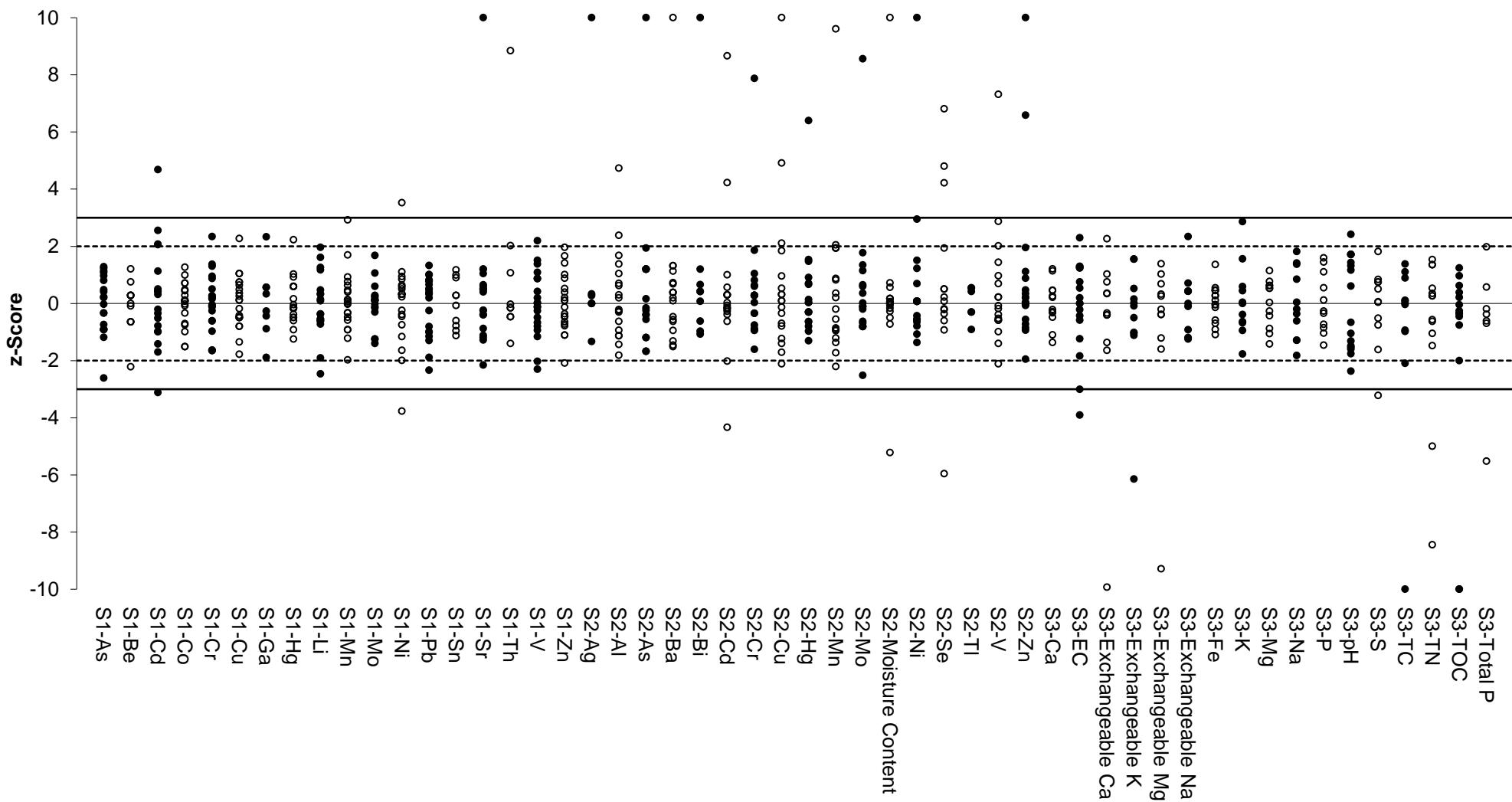
Se followed by Rb and Sb in S1 were the most difficult elements to analyse. No agreement was found between results reported by participants for these elements.

Ag and Tl in S2 were the tests that presented the least analytical difficulty to participating laboratories, with a between-laboratory CV under 7%.

The results reported by laboratory 15 in Sample S2 were consistently higher than the assigned value by the same factor of approximately 2. These results were not included in statistical calculation and in the analysis of the extraction methods and instrumental techniques employed by participants.

A limited number of laboratories reported results for Cs, Gd, La and Sm in S2 (Laboratories 1, 13, 16, 17 and 19).

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



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

Figure 69: z-Score Dispersal by Test

Summary of Participant's Performance in AQA 24-01 Samples S1, S2 and S3

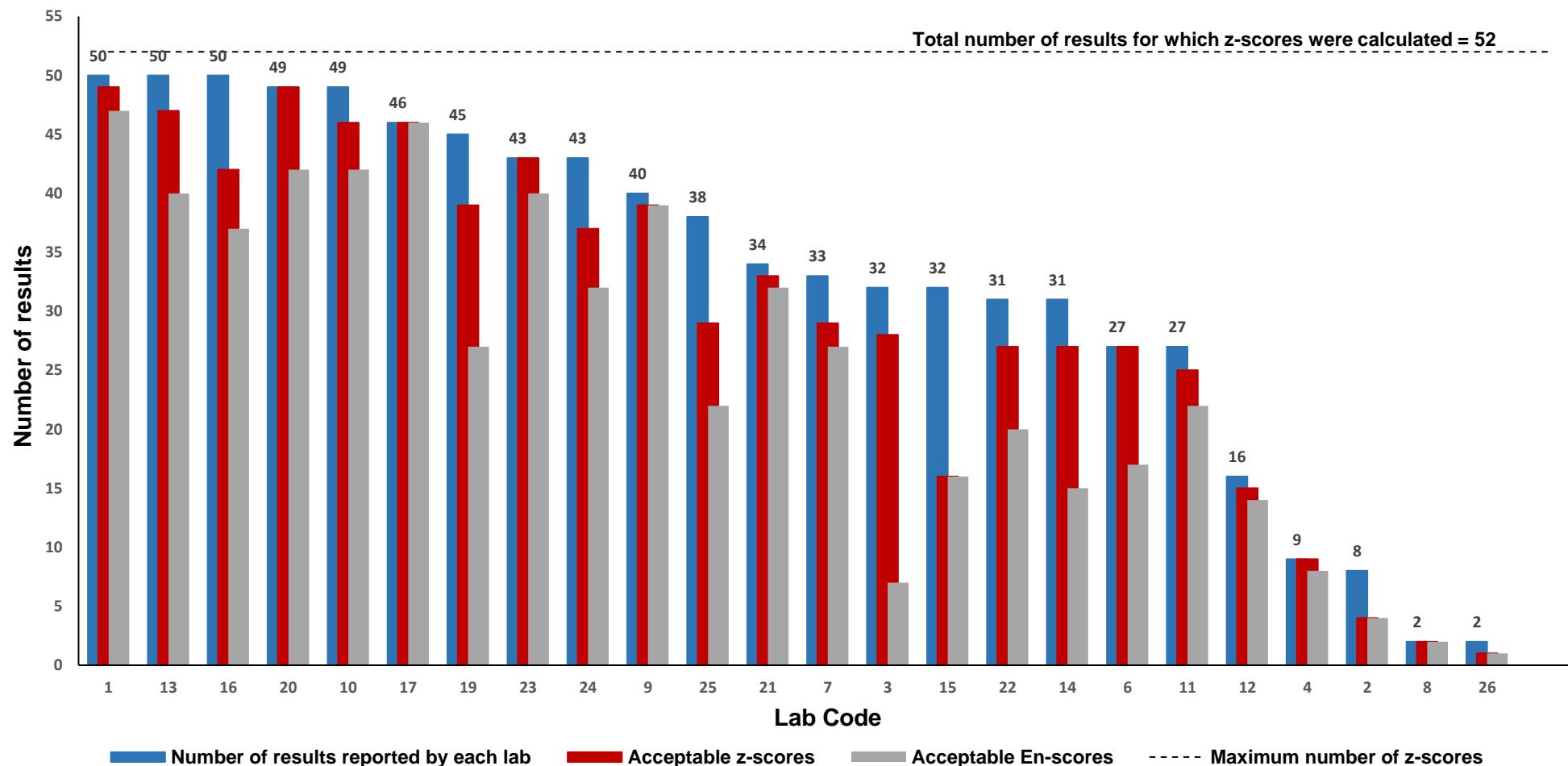


Figure 70: Summary of Participant's Performance in AQA 24-01

Table 79 Summary of Participants' Results and Performance for Sample S1

Lab Code	As (mg/kg)	B (mg/kg)	Be (mg/kg)	Cd (mg/kg)	Co (mg/kg)	Cr (mg/kg)	Cu (mg/kg)	Ga (mg/kg)	Hg (mg/kg)	Li (mg/kg)	Mn (mg/kg)
AV	35.2	Not Set	1.07	0.94	16.8	38.5	32.6	4.5	0.615	28.6	672
HV	31.0	4.46	1.12	0.85	16.7	41.8	29.2	4.67	0.602	28.7	632
1	36.9	<50	1	0.7	16.6	35	28.2	4.1	0.6	25.5	723
2	26	NT	NT	1.6	NT	29	33	NT	0.61	NT	NT
3	38	<10	<2	1	20	52	36	NT	0.67	34	868
4	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
6	NT	NT	1	1	15	37	35	NT	0.6	NT	676
7	35.1	<10	<2	0.984	17.1	46.4	32.0	NT	0.669	33.6	681
8	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
9	39	< 10	< 2	0.8	17	38	31	5	0.56	30	680
10	39.2	<5	0.832	0.891	14.3	32.9	26.8	NR	0.598	29	610
11	32	<1	1.06	<1	17	35	40	NT	<1	27	640
12	38.6	3.0	1.15	1.23	19.3	39.6	31.0	NT	0.60	29.2	734
13	32.6	NT	1.0	0.8	17.5	38.2	33.4	3.7	0.7	20.4	699
14	32	4	<2	0.5	13	40	30	5	0.5	26	590
15	36	<4.4	1.1	0.83	17	40	36	NT	0.58	26	650
16	39.707	<50	1.0984	1.232	15.94	29.08	31.22	4.256	0.821	26.206	632.10
17	31	NR	1.1	0.91	17	44	31	4.8	0.57	27	670
19	34	<50	<1	<1	13	29	30	2.8	0.6	18.0	539
20	35.9	3.24	1.07	0.74	18.6	37.8	34.7	NT	0.63	26.3	672
21	39.54	< 10	< 2	1.010	17.90	41.39	33.77	NT	0.669	29.96	702.7
22	32	9.7	1.1	NR	18	46	31	6.6	0.57	37	650
23	36.64	< 10	< 2	0.8670	18.6	39.42	34.2	NT	0.6	30.6	714.5
24	32.0	<5	1.2	1.10	16.75	43.5	35.0	NR	0.71	35.5	786.0
25	34	5.2	<5	1.3	14.9	38	33	NT	0.53	NT	610
26	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR

Shaded cells are results which returned a questionable or unacceptable z-score. AV = Assigned Value, HV = Homogeneity Value; NA = Not Available

Table 79 Summary of Participants' Results and Performance for Sample S1 (continued)

Lab Code	Mo (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Rb (mg/kg)	Sb (mg/kg)	Se (mg/kg)	Sn (mg/kg)	Sr (mg/kg)	Th (mg/kg)	V (mg/kg)	Zn (mg/kg)
AV	4.92	56.2	44.7	Not Set	Not Set	Not Set	5.64	125	2.15	48.9	204
HV	4.82	56.7	47.7	6.98	22.0	0.617	5.47	110	2.30	48.2	194
1	5.0	53.6	51.4	3.8	18.5	<1	5.8	122	2	43	170
2	NT	35	29	NT	NT	NT	NT	NT	NT	NT	220
3	5.7	76	50	NT	31.00	<2	<10	140	NT	60	264
4	NT	NT	NT								
6	4	52	50	NT	NT	0.4	NT	NT	NT	48	190
7	5.36	59.8	50.2	NT	28.8	<2	<10	131	NT	56.9	215.9
8	NR	NR	NR								
9	5	59	47	NT	30	<2	<10	130	2.1	49	190
10	3.89	49.7	39.3	NR	20.5	3.05	5.6	114	NR	40.4	184
11	4.8	58	43	NT	NT	<5	6.3	120	NT	47	200
12	5.06	61.5	49.1	4.71	21.0	0.6	5.8	138	NT	44	247
13	5.0	62.4	47.3	3.7	9.8	5.0	5.1	122	2.0	48	207
14	<5	45	36	11	21	<5	5	110	5	49	140
15	4.9	58	38	3.9	22	0.6	5.3	120	NT	42	190
16	5.128	54.831	48.11	4.490	19.87	<1	6.149	131.3	2.495	45.285	254.8
17	4.7	61	47	6.2	13	0.59	5.2	140	2.1	52	210
19	4	47	38	2.8	8	<5	<5	98	1.7	32	170
20	4.83	57.5	51.5	NT	36.4	1.15	NT	111	2.14	50.3	182
21	5.105	59.44	53.63	NT	27.76	<2	<10	132.6	NT	55.21	231.0
22	NR	NR	46	16	15	0.49	6.2	109	2.8	65	193
23	5	58	48	NT	32.7	<2	<10	133.1	NT	55.3	207.3
24	6.16	58.0	32.0	NR	NR	NR	265.2	NR	34.0	235.0	
25	<5.0	54	37	NT	17	<5.0	<5.0	131.5	NT	59	180
26	NR	NR	NR								

Shaded cells are results which returned a questionable or unacceptable z-score. AV = Assigned Value, HV = Homogeneity Value; NA = Not Available

Table 80 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)	Cr (mg/kg)	Cs (mg/kg)	Cu (mg/kg)	Gd (mg/kg)	Hg (mg/kg)
AV	1.00	14900	12.5	106	2.88	2.87	21.1	Not Set	22.8	Not Set	1.99
HV	1.04	13400	12.5	102	3.11	2.86	20.5	1.46	23.8	3.24	1.98
1	<2	12100	10.4	95.9	2.9	2.8	18.7	1.1	19.6	2.2	2
2	NT										
3	1	17000	14	120	<10	3.3	27	NT	27	NT	2.2
4	NT										
6	NT	11520	11	108	NT	3	20	NT	23	NT	NT
7	<2	19900	12.7	118	<10	2.84	24.4	NT	23.5	NT	2.19
8	NR										
9	< 2	14000	14	110	< 10	2.8	20	NT	21	NT	1.6
10	1.05	10600	12.2	99.3	3.07	2.88	18.2	NR	18.9	NR	2.03
11	<1	14100	14	92	NT	3.0	22	NT	34	NT	1.8
12	NT										
13	0.8	11500	10.4	90.5	2.6	2.6	18.3	0.6	21.2	1.7	1.9
14	<5	18000	11	100	3	1	22	NT	20	NT	1.7
15	2.5	29000	25	240	7.1	6.6	46	2.6	49	NT	3.9
16	<2	13037	12.297	100.98	3.225	4.689	18.10	1.36	22.50	2.172	2.448
17	1.0	15800	12	110	3.0	2.7	22	1.6	23	3.8	1.9
19	1.0	9510	12	90	2.7	2	16	0.9	18	1.9	2.0
20	1.04	14232	11.8	107	2.57	2.78	21.2	NT	22.0	NT	2.43
21	< 2	15452	14.92	113.2	< 10	3.111	23.67	NT	27.61	NT	2.261
22	NT										
23	< 2	19000	14	110	< 10	3	22	NT	24	NT	1.9
24	NR	16750	<15	113.7	NR	2.75	23.0	NR	23.5	NR	1.75
25	<5.0	22000	14	120	NT	3.0	23.1	NT	25	NT	1.8
26	NR										

Shaded cells are results which returned a questionable or unacceptable z-score. AV = Assigned Value, HV = Homogeneity Value; NA = Not Available

Table 80 Summary of Participants' Results and Performance for Sample S2 (continued)

Lab Code	La (mg/kg)	Mn (mg/kg)	Mo (mg/kg)	Ni (mg/kg)	Se (mg/kg)	Sm (mg/kg)	Tl (mg/kg)	U (mg/kg)	V (mg/kg)	Zn (mg/kg)	Moisture Content (%)
AV	Not Set	561	5.69	13.9	4.65	Not Set	0.825	Not Set	32.9	745	18.0
HV	20.6	555	6.1	11.8	4.59	3.69	0.95	1.07	34.8	776	18.0
1	12.5	507	5.6	13.3	5	2.4	0.8	0.6	30	691	17.5
2	NT	NT	NT	NT	NT						
3	NT	670	7.2	18	4.7	NT	<1	<10	40	890	19
4	NT	NT	NT	NT	NT						
6	NT	493	NT	14	4.0	NT	NT	NT	31	746	19.29
7	NT	676	6.83	15.6	4.23	NT	<10	<10	36.3	748	18
8	NR	NR	NR	NR	NR						
9	NT	530	5	14	5	NT	<10	<10	28	740	17.9
10	NR	464	5.15	12.4	7.59	NR	0.75	0.978	30.3	703	18.1
11	NT	510	5.0	13	<5.0	NT	NT	<1	30	770	18
12	NT	NT	NT	NT	NT						
13	11.8	484	5.1	13.1	8.0	2.2	0.8	0.7	30	675	8.6
14	NT	670	6	14	6	NT	<2	<2	34	600	18.3
15	31	1100	13	29	9.4	NT	<2.8	1.7	69	1800	63.4
16	14.610	512.82	6.248	13.138	0.495	2.667	0.8697	0.876	47.084	1236.26	18.3
17	27	550	5.7	14	4.5	4.7	0.86	1.3	32	760	18
19	9.3	437	5	12	<5	1.7	NT	0.5	26	682	18.1
20	NT	572	5.52	12.8	4.40	NT	0.87	1.60	32.5	742	16.7
21	NT	581.4	6.669	14.86	4.517	NT	<10	<10	37.68	810.8	17.7
22	NT	NT	NT	NT	18						
23	NT	670	6.2	14	4.8	NT	<10	<10	34	780	18
24	NR	607.0	3.54	14.0	NR	NR	NR	NR	22.5	828.0	18.3
25	NT	610	5.6	16	<5.0	NT	<5.0	NT	42.8	760	17.1
26	NR	NR	NR	NR	NR						

Shaded cells are results which returned a questionable or unacceptable z-score. AV = Assigned Value, HV = Homogeneity Value; NA = Not Available

Table 81 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)
AV	5170	22000	980	1130	165	1640	542	Not Set	Not Set	186
HV	5080	19700	1200	1120	140	1700	NA	NA	NA	NA
1	5280	22600	840	1010	160	1820	610	NT	NT	186
2	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
3	NT	23000	NT	NT	NT	NT	NT	NT	NT	163
4	NR	NR	NR	NR	NR	NR	NR	NR	324	178
6	NT	23040	NT	NT	NT	NT	NT	NT	NT	196
7	NT	20700	NT	NT	NT	NT	NT	NT	NT	113.2
8	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
9	4600	20000	880	1100	120	1500	480	NT	NT	130
10	5020	21900	886	1030	156	1730	547	NR	NR	209
11	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
12	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
13	5760	22600	980	1200	200	NT	280	NT	NT	209
14	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
15	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
16	5797.7	25000	1066	1217	186.10	1878	602	NT	NT	228.7
17	5410	21700	1210	1190	NR	1900	690	NR	NR	210
19	5000	21900	720	970	150	NT	NT	546	374	186
20	4920	19604	986	1081	133	1469	545	302	355	182
21	NT	20461	NT	NT	NT	1582	NT	NT	NT	151.74
22	4460	NR	NR	1260	199	1520	500	310	NT	175
23	5058	22237	1044	1133	166	1660	410	NT	NT	189.6
24	5405	23200	922.5	1190	133	1595	583	NR	331	200
25	5300	23000	1400	1200	210	1400	603	NT	NT	210
26	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR

Shaded cells are results which returned a questionable or unacceptable z-score. AV = Assigned Value, HV = Homogeneity Value; NA = Not Available

Table 81 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+ColP (mg/kg)	pH	TC (mg/kg)	TN (mg/kg)	TOC (mg/kg)	Total P (mg/kg)
AV	14.6	0.649	2.81	0.184	Not Set	Not Set	5.19	75300	4580	74400	1750
HV	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
1	16.1	0.8	3.0	0.2	NT	NT	5.4	NT	4320	7.4	1590
2	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
3	NT	NT	NT	NT	NT	NT	5.50	NT	NT	NT	NT
4	15.13	0.65	2.88	0.15	NR	NR	5.43	75950	4700	75950	NR
6	NT	NT	NT	NT	NT	NT	5.3	NT	NT	NT	NT
7	NT	NT	NT	NT	NT	NT	4.91	8.90	NT	8.21	NT
8	NR	NR	NR	NR	NR	NR	NR	68244	NR	68721	NR
9	NT	NT	NT	NT	NT	NT	5.5	NT	5200	NT	NT
10	14.0	0.64	2.7	0.21	NR	NR	4.76	NR	4700	77200	1700
11	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
12	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
13	15.7	0.8	3.1	0.2	<.2	NT	5.45	NT	5280	71000	2270
14	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
15	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
16	15.09	0.664	2.75	0.184	NT	NT	5.63	NT	4291.0	71900	1566.73
17	NR	NR	NR	NR	NR	NR	5.0	75000	5200	74000	NR
19	14.1	0.7	2.9	0.2	0.9	NT	4.87	75400	4100	71700	1650
20	12.6	0.55	2.47	0.14	2.95	163	4.95	83600	4825	83600	NR
21	NT	NT	NT	NT	NT	NT	4.92	85709	NT	79021	NT
22	17.9	0.54	3.2	0.18	NT	NT	4.9	76100	4740	72500	1900
23	14.0	0.548	2.36	0.138	NT	NT	5.5	81970	NT	81600	NT
24	12.2	0.60	2.70	0.27	1.45	169.5	5.07	59500	3900	59500	NR
25	0.1	0.05	0.2	<0.17	NT	NT	NT	NT	710	NT	300
26	NR	NR	NR	NR	NR	NR	NR	67890	2290	NR	NR

Shaded cells are results which returned a questionable or unacceptable z-score. AV = Assigned Value, HV = Homogeneity Value; NA = Not Available

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, 17-20} 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 95°C.

One laboratory used dilute HNO₃ and hydrogen peroxide and digested their samples at 95°C for 60 min, while one digested their sample for 270 min at 96°C using HNO₃ only. Most of the results reported by these laboratories were in good agreement with the assigned value.

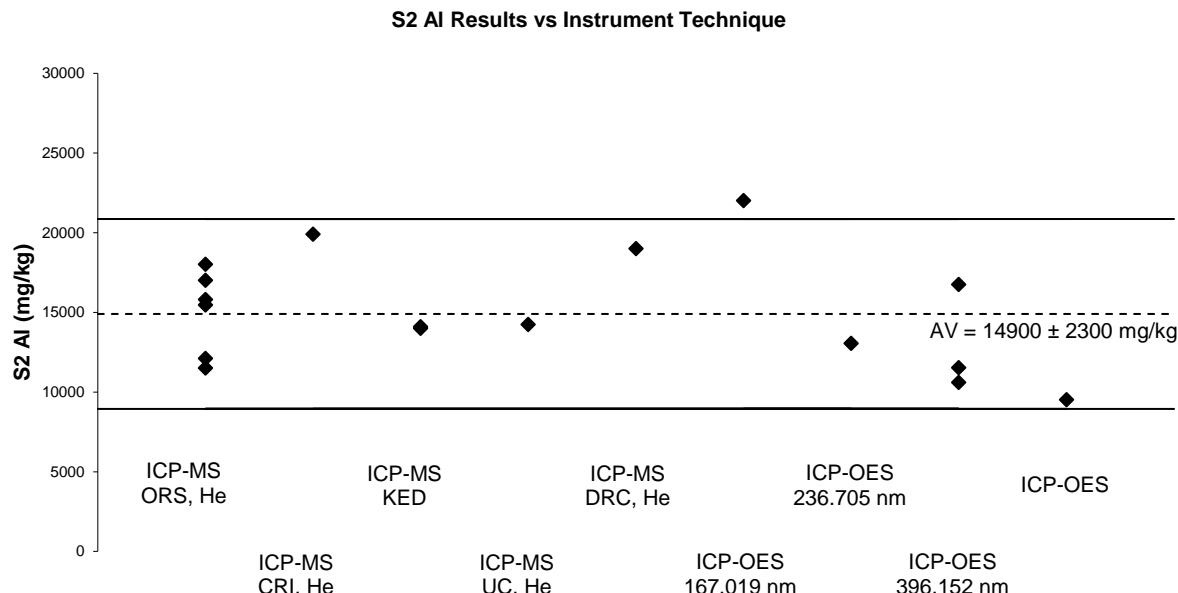
Laboratory 14 used a digestion temperature of 180°C for 30 min and a large ratio of acid to sample size of 12 mL to 0.5 g of dried soil. All results reported by this laboratory for acid extractable elements returned acceptable z-scores with the exception of three.

Dilution/calculation problems may explain some of the unacceptable results.

Individual Element Commentary

Aluminium level in S2 was high, at 14 900 mg/kg. Plots of Al results versus instrumental technique used by participants are presented in Figure 71.

ICP-OES with wavelength 167.019 nm may not be the appropriate analytical technique for Al measurements at a high level.



*Results from laboratory 15 were excluded. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2

Figure 71 S2 Al Results vs. Instrumental Technique

Antimony spike value in S1 was 40 mg/kg. No assigned value could be set for this test because the results were too variable (between 8 mg/kg to 36.4 mg/kg).

Laboratory 20 recovered 91% of the spiked Sb in the soil sample S1. Their digestion regime involved a digestion temperature of 120°C over 60 min, no water addition, and a ratio of concentrated acid ($\text{HNO}_3 + \text{HCl}$) to sample size of 25 to 1.

Plots of participants' results versus instrumental technique are presented in Figures 72. There was no evident relationship between participants' results for Sb and the instrumental technique used.

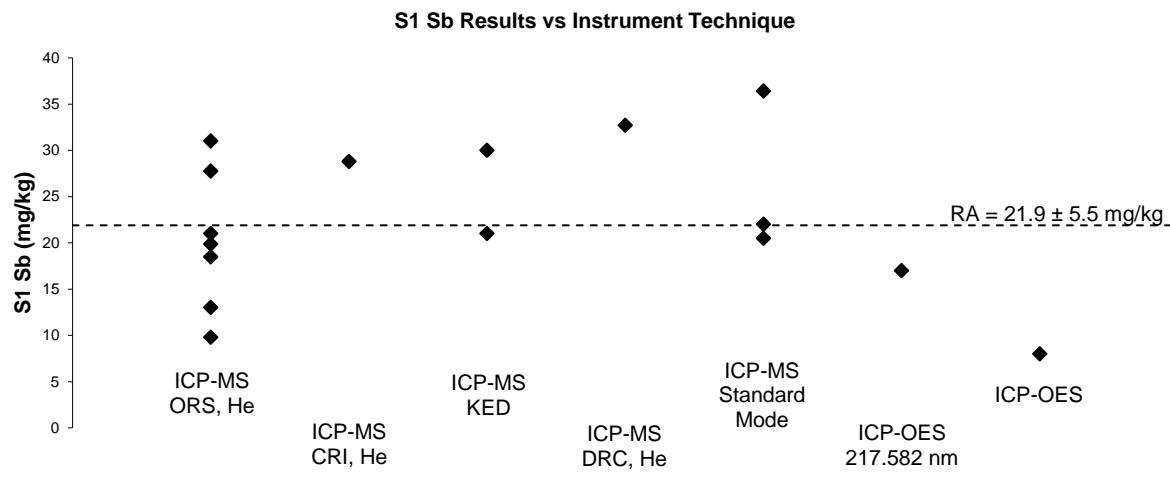
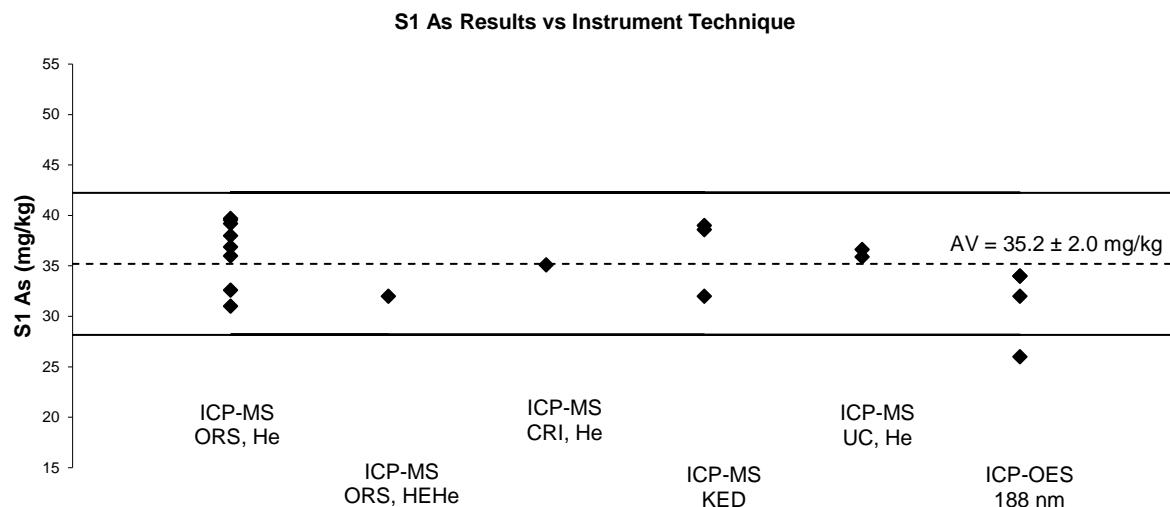


Figure 72 S1 Sb Results vs. Instrumental Technique

Arsenic All results reported for As in S1 returned acceptable z-scores but one.

The result reported by Laboratory 2 returned an unacceptable z-score but was in good agreement with the assigned value considering the respective uncertainties (the E_n -score was acceptable). Eurachem recommends for laboratories to assess their method and laboratory bias and if significant to correct for it.¹⁰

Figure 73 presents plots of participants' z-scores versus instrumental technique used. Participants used ICP-MS in collision mode or ICP-OES with a wavelength of 188 nm.



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

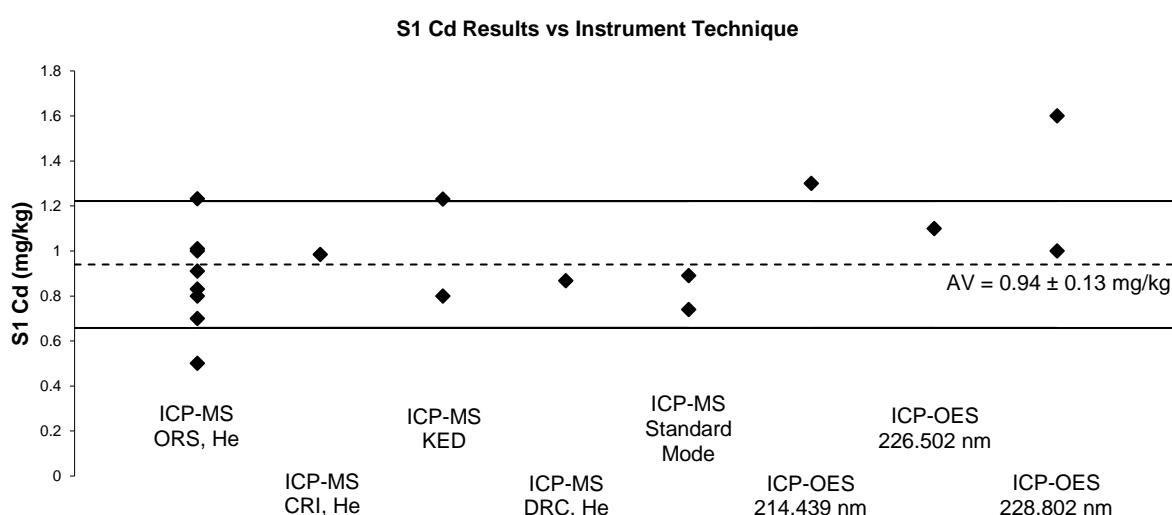
Figure 73 As Results in S1 vs. Instrumental Technique

Boron level in Sample S1 was below the reporting level of most laboratories. Of 5 reported results 4 were in relatively good agreement with each-other, the median value (4.0 mg/kg) and the homogeneity value (4.46 mg/kg).

For measurement of B two participants reported using ICP-MS in collision mode, one used ICP-MS in standard mode, and one ICP-OES with wavelength 249 nm. Caution should be exercised when B is measured by ICP-OES at 249 nm, because it can have significant interferences from Fe 249.771 nm if on-line inter-element correction is not used.

Cadmium was one of the tests with the largest number of unacceptable z-scores.

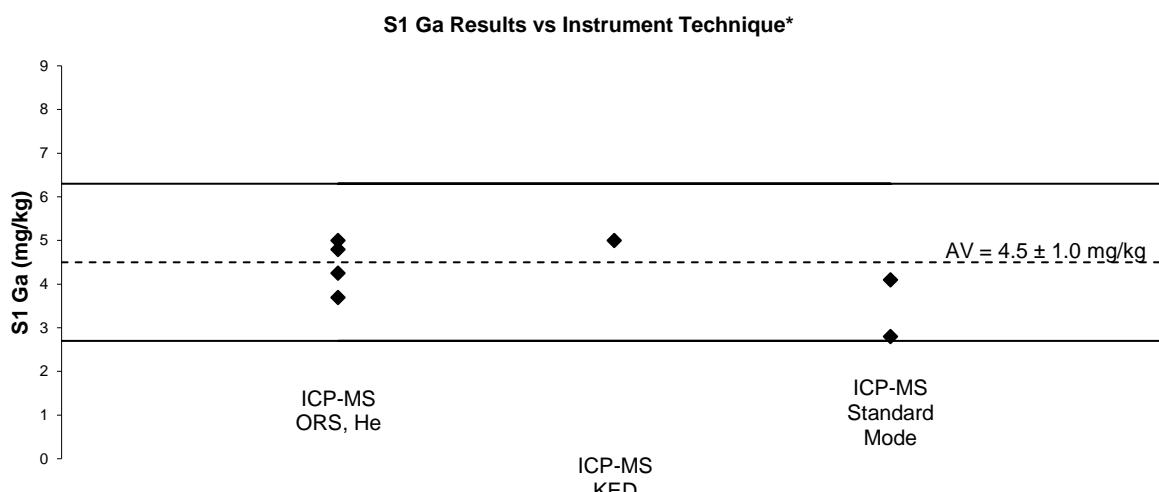
Figure 74 presents plots of participants' z-scores versus instrumental technique used. Cd 228.802 nm and Cd 214.439 nm can have significant spectral interferences from Fe 228.804 nm and Fe 214.445 nm respectively. If ICP-OES is used with one of these two wavelengths, then online inter-element correction may be required as soil is usually rich in Fe.



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

Figure 74 S1 Cd Results vs. Instrumental Technique

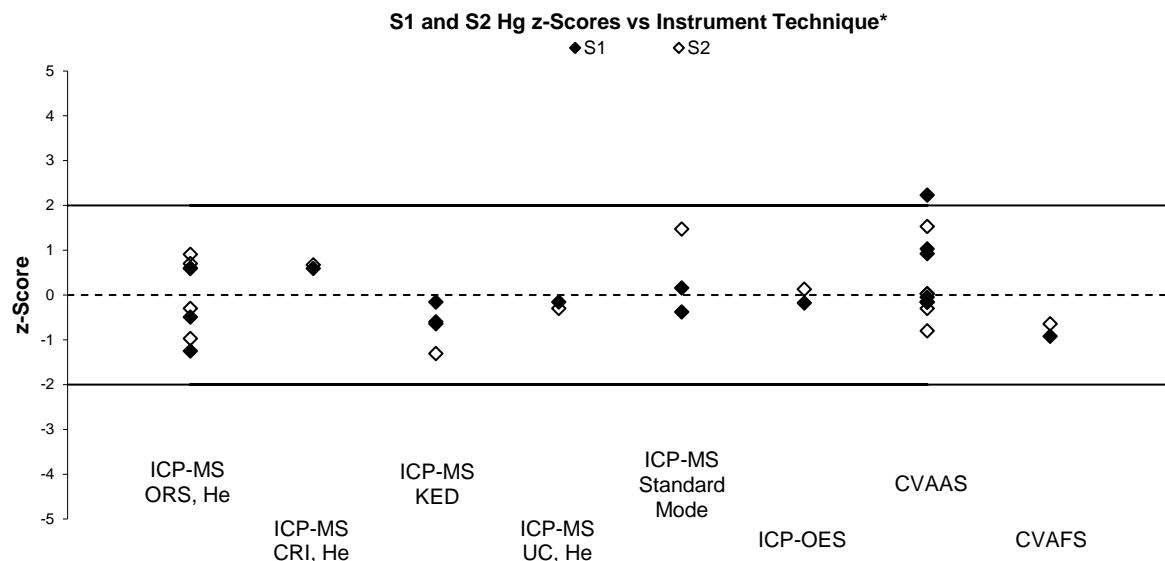
Gallium Although a limited number of participants reported results for Ga in S1 all performed acceptably but one (Figure 75 and Table 79).



*The laboratory that has not performed acceptably is not charted because it did not report an instrumental technique.
Horizontal lines on charts are the results corresponding to z-scores of 2 and -2

Figure 75 S1 Ga 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 76).



*Laboratory 15 result was excluded

Figure 76 Hg Performance in S1 and S2 vs. Instrumental Technique

Selenium analysis is challenging due to there being multiple sources of significant interference. This is especially problematic at low levels where any unresolved interference can have a more significant effect on results. Only 8 laboratories reported results for Se in S1; results were too variable, and hence no assigned value could be set.

Plots of participants' results versus instrumental technique used are presented in Figure 77. Laboratories used 4 different instrumental techniques: ICP-MS in collision mode with He, ICP-MS in collision mode with high energy helium, ICP-MS in reaction mode with H₂, and hydride generation ICP-OES. Except for two, all results reported for Se were in good agreement with each other, with the homogeneity value of 0.617 mg/kg and with median value of 0.600 mg/kg.

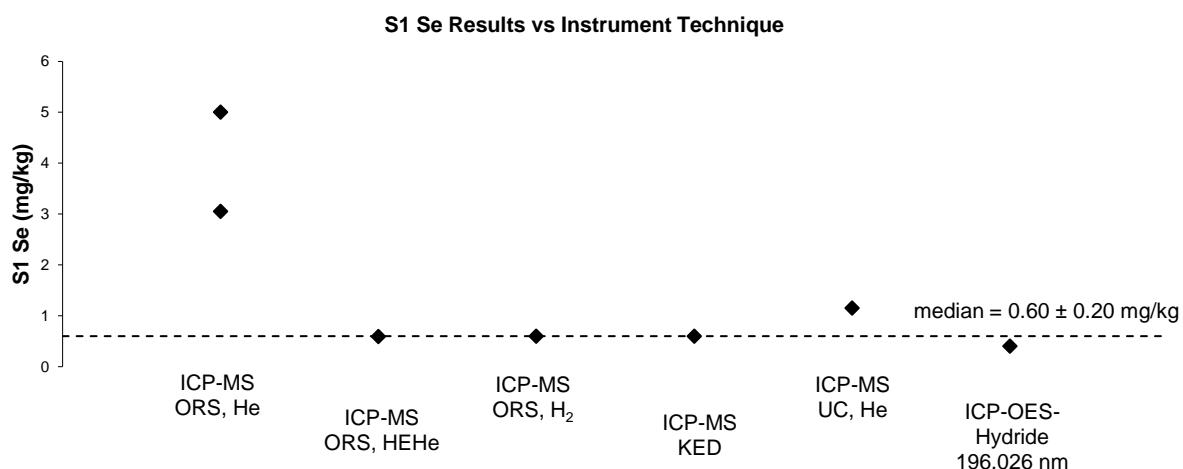
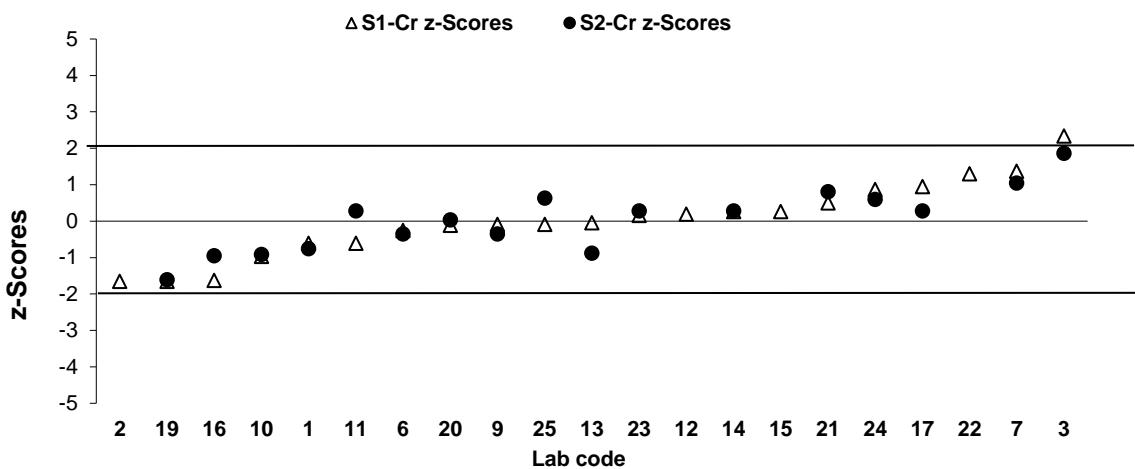


Figure 77 Se Results in S1 vs. Instrumental Technique

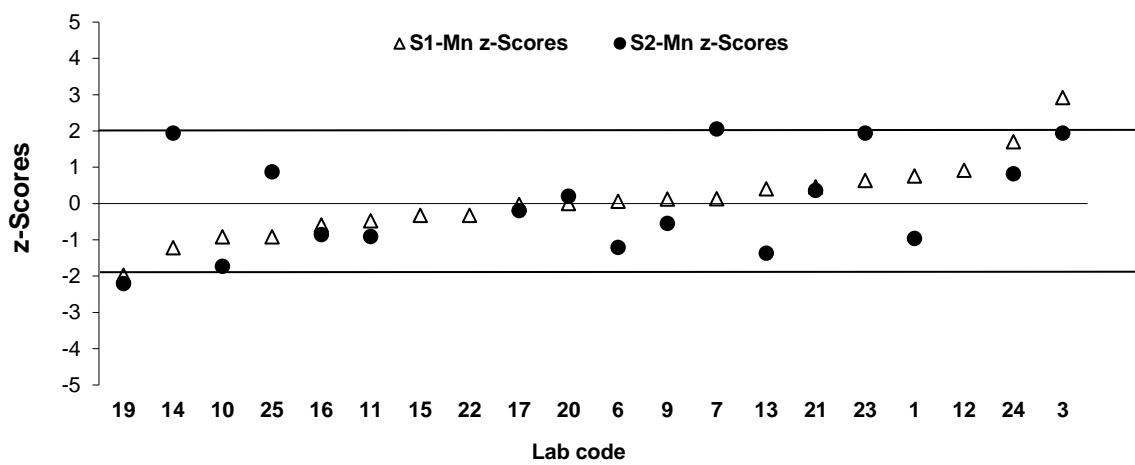
Chromium, Nickel, Manganese and Vanadium are four elements which are strongly dependent on the digestion regime. Participants' performance for these elements in the two study samples are presented in Figures 78 to 81.

Laboratories 2 and 19 should check for method or laboratory bias as their reported results were consistently low. Laboratory 3 should also check for method or laboratory bias as their result was consistently high.



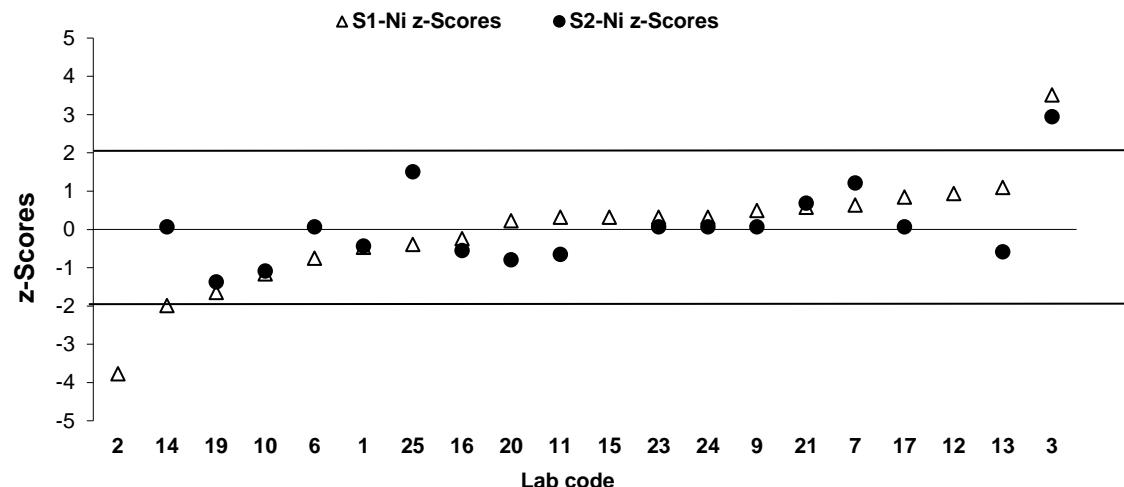
*Laboratory 15 result was excluded

Figure 78 S1 and S2 Cr z-Scores vs Laboratory Code



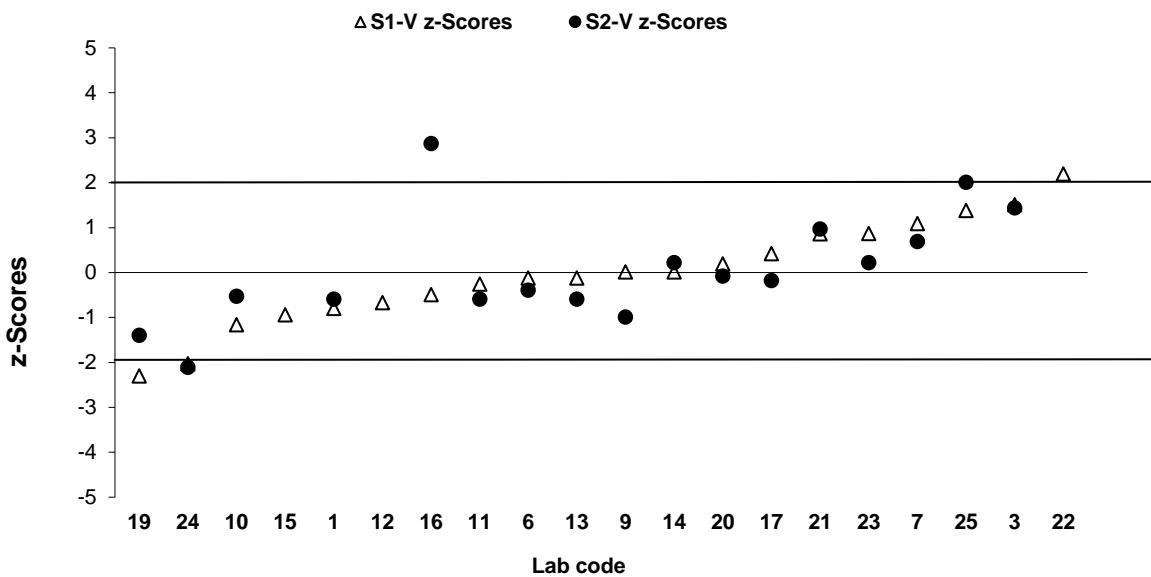
*Laboratory 15 result was excluded

Figure 79 S1 and S2 Mn z-Scores vs Laboratory Code



*Laboratory 15 result was excluded

Figure 80 S1 and S2 Ni z-Scores vs Laboratory Code



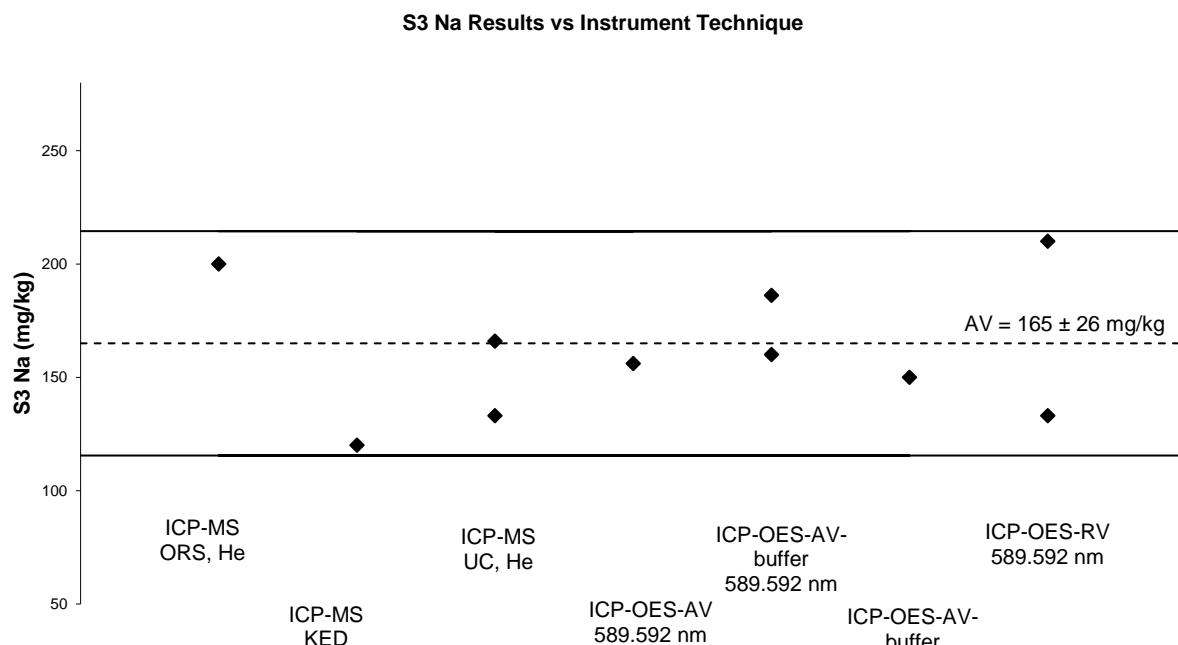
*Laboratory 15 result was excluded

Figure 81 S1 and S2 V z-Scores vs Laboratory Code

Laboratory 24 should also check their method used for V measurements as their reported results in both test samples S1 and S2 were too low (Figure 81).

Sodium level in S3 was low (165 mg/kg) which may have challenged participants' analytical techniques. The between laboratory CV was large (21%) three times higher than the CV predicted by Thompson and Horwitz (7.4%).

Results for Na versus instrumental technique used are presented in Figure 82. Participants used a wide variety of instrumental techniques, including ICP-MS in collision mode ICP-OES with axial view, ICP-OES with axial view and buffer or ICP-OES with radial view.



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

Figure 82 Na Results vs. Instrumental Technique

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 9. With one exception, all participants used a ratio sample mass/extraction solution of 1 to 20 and shook their sample for 1 to 2 hours. Laboratory 10 used a ratio of 1:10 for sample mass/extraction solution and shook their sample for half an hour. No significant differences were noticed between the results reported by this participant and the results reported by participants who used a ration of 1:20. Plots of participants' results versus the analytical methods used for the exchangeable bases measurement are presented in Figures 83 to 86.

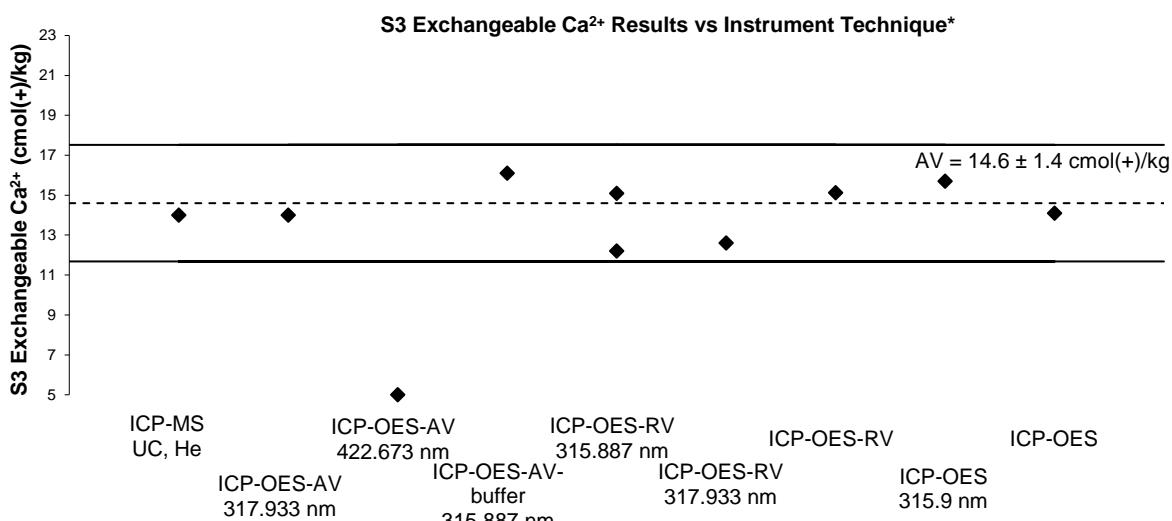


Figure 83 Exchangeable Ca²⁺ Results vs. Analytical Methods

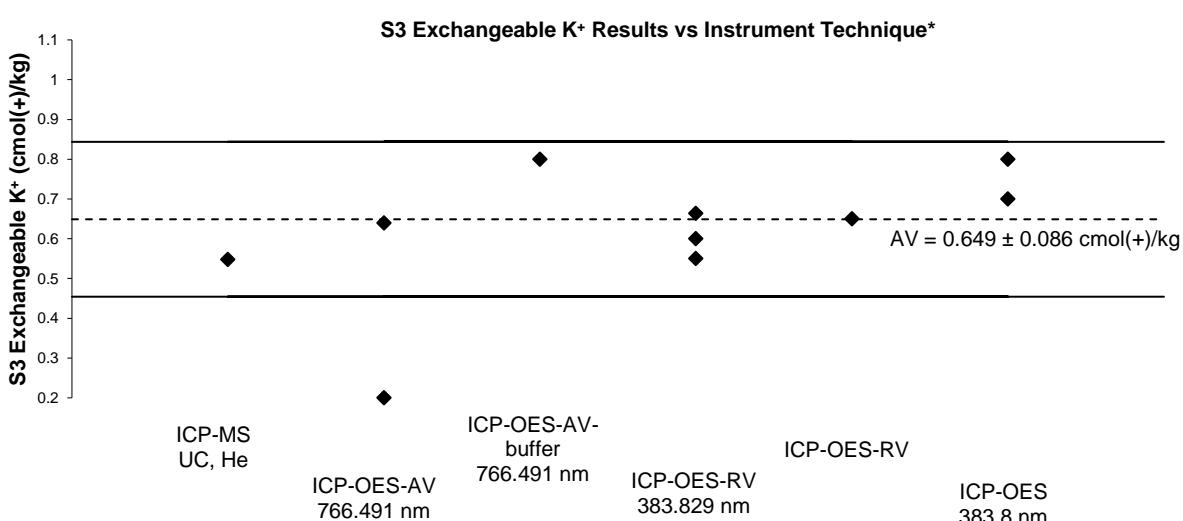


Figure 84 Exchangeable K⁺ Results vs. Analytical Methods

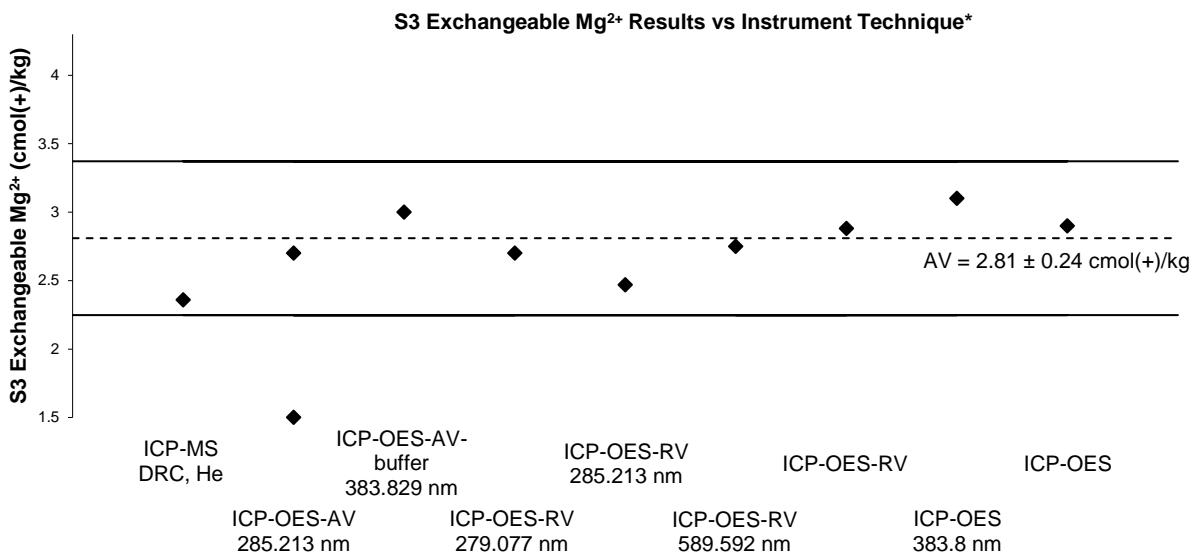


Figure 85 Exchangeable Mg²⁺ Results vs. Analytical Methods

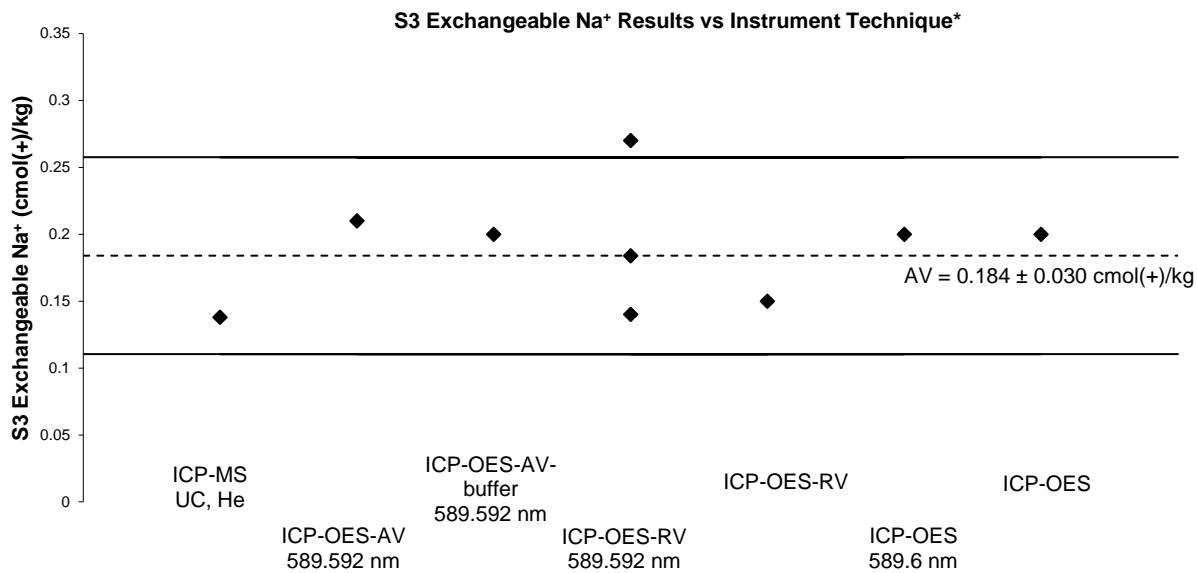


Figure 86 Exchangeable Na⁺ Results vs. Analytical Methods

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 Only two participants reported the method used for Colwell P and Colwell K; measurements in S3, both shook the sample for 16 hours and used a ratio of 1 :100 sample mass/extraction solution (Table 4).

Colwell K Two participants reported results for Colwell K in S3, one used ICP-OES for K measurements and one used ICP-MS (Figure 87).

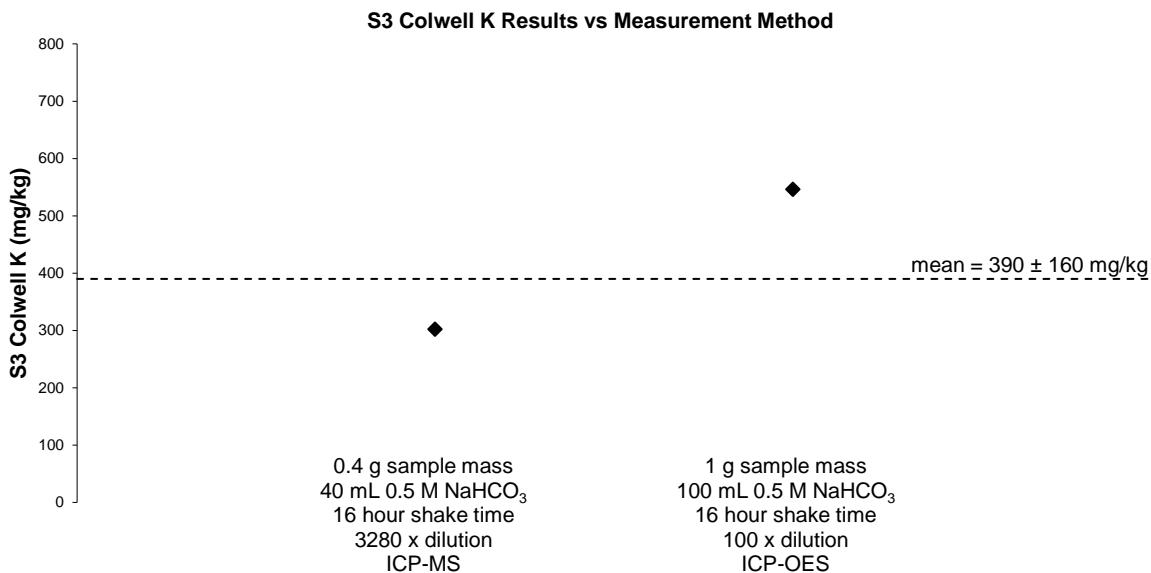
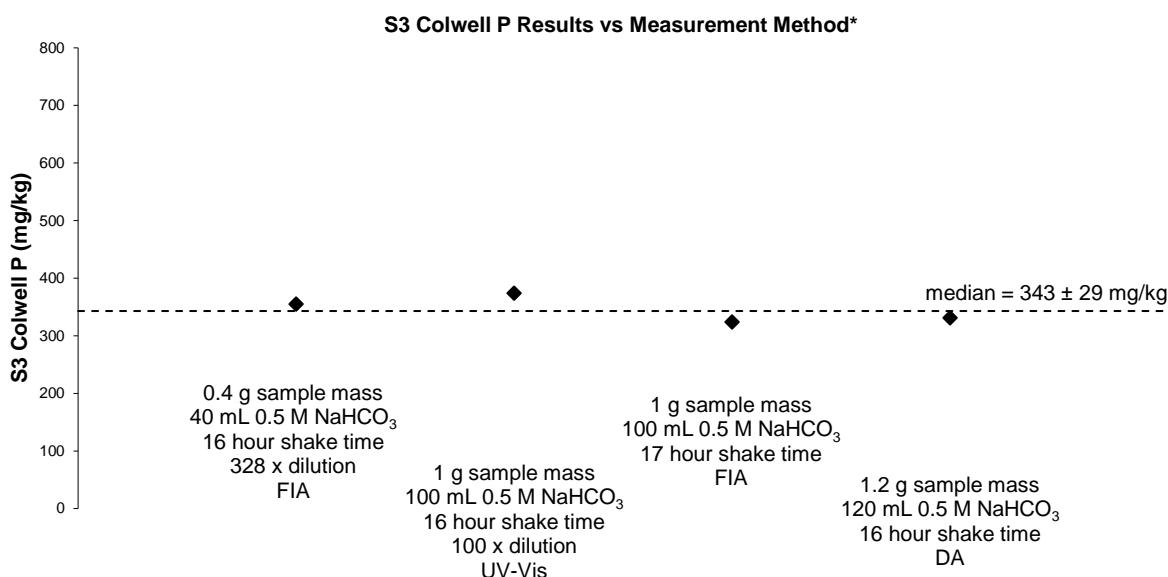


Figure 87 Colwell K Results vs. Method

Colwell P Four laboratories reported results for this test. The results were in relatively good agreement with each other, centred on the value of 343 mg/kg (Figure 88).



*The methodologies used in AQA 23-02 by laboratories 4 and 24 were plotted.

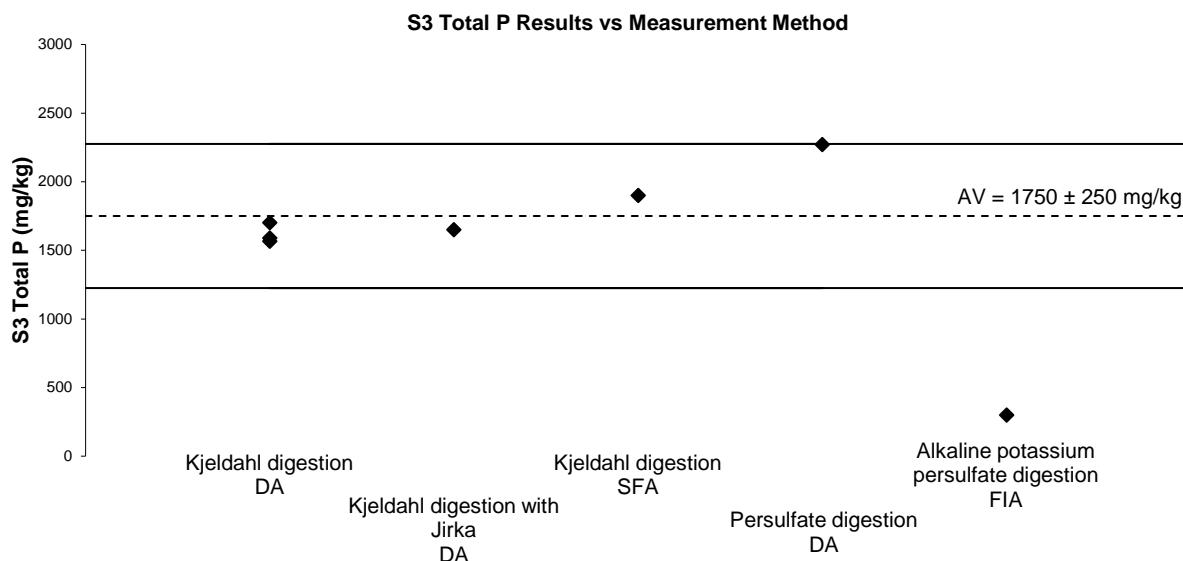
Figure 88 Colwell P Results vs. Instrumental Technique

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. Two laboratories reported results for this test. The results were in excellent good agreement with each other.

6.9 Participants' Results and Analytical Methods for Total P

Total P assigned value was 1750 mg/kg. Seven participants reported results for total P, and all performed acceptably but one (Figure 89).

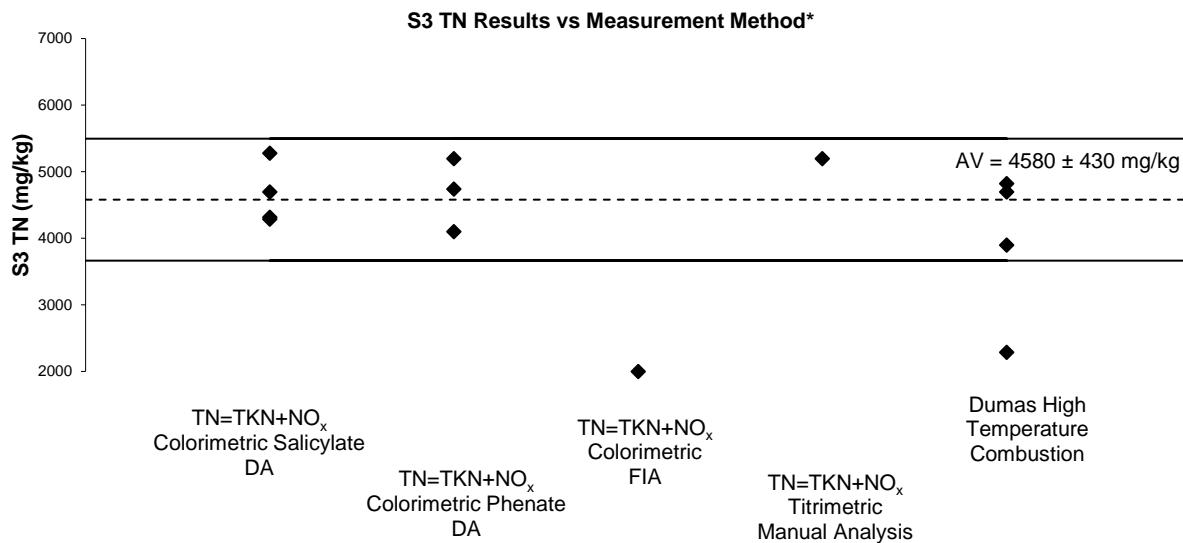


Horizontal lines on charts are the results corresponding to z-scores of 2 and -2

Figure 89 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 8. A plot of participants' results versus analytical method and measurement technique used for TN analysis in S3 is presented in Figure 90.



*Laboratory 25 result of 710 has plotted as 2000 mg/kg. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2.

Figure 90 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 2 and 3.

Total Carbon With the exception of two results, all other reported results for TC in S3 returned acceptable z-scores.

Laboratory 7 correctly measured TC in S3 but reported results in the wrong units.

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.²²⁻²⁵

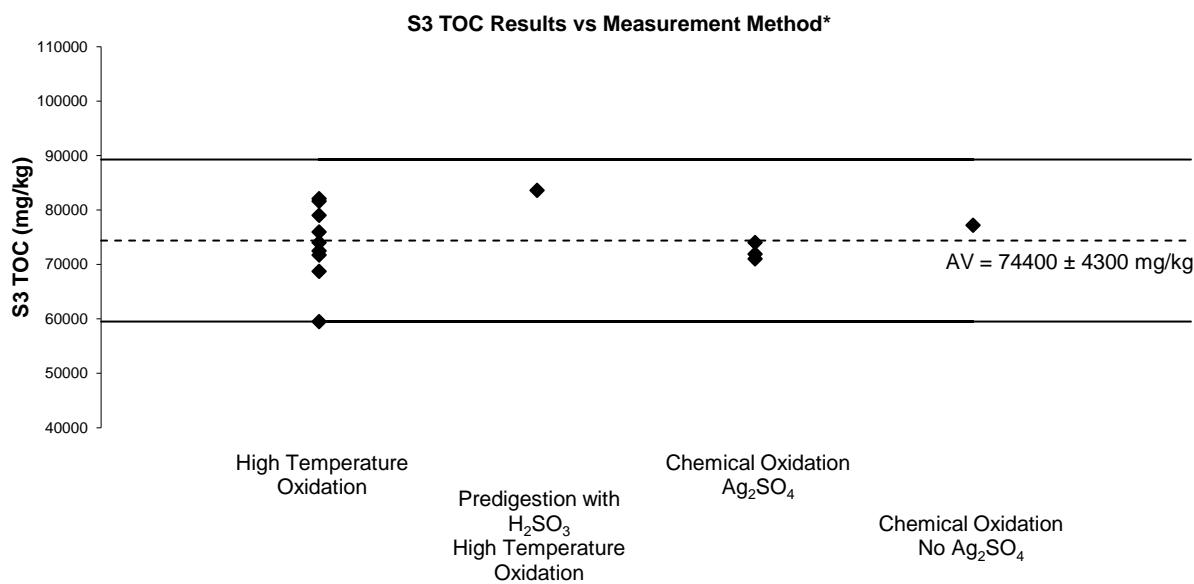
Laboratories 1 and 7 correctly measured TOC in S3 but reported results in the wrong units.

One laboratory digested the sample with sulfurous acid prior LECO determination.

Two laboratories reported adding silver sulfate to remove chloride interferences.

Ten participants used a high temperature oxidation method and four used a chemical oxidation method based on the “Walkley-Black” method (Figure 91).

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 1 as 7.4 mg/kg has been plotted as 74000 mg/kg. The result reported by Laboratory 7 as 8.21 mg/kg has been plotted as 82100 mg/kg. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2

Figure 91 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.^{25, 26}

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.^{25, 26}

6.12 Comparison with Previous NMI Proficiency Tests of Metals in Soil

AQA 24-01 is the 34th NMI proficiency study of inorganic analytes in soil.

Participants' performance in measurement of metals in soil over the last twelve years is presented in Figure 92. 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 82).

Table 82 Control Samples Used by Participants

Lab. Code	Description of Control Samples
2	RM
3	CRM – ICV1-11, ICV-1, AGAL-12, CRM540, CRM Hg
4	CRM – ASPAC ASS6052, ASPAC 7468-QC, ASPAC 651-C-1
6	CRM – AGAL-12
7	SS
8	CRM – BBOT LCRM for Total Carbon and Total Organic Carbon
9	CRM
12	CRM – Agal-12 Biosoil
13	CRM
14	SS and CRM
16	CRM
17	CRM – agal10 & agal12
19	CRM
20	RM – AGAL 12 (metals) In house AG reference
21	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
22	CRM – PACS3
23	CRM
24	CRM – AGAL 12, LOAM B
26	RM: Sulfamethazine is for Total Nitrogen and Total Carbon

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'²⁷

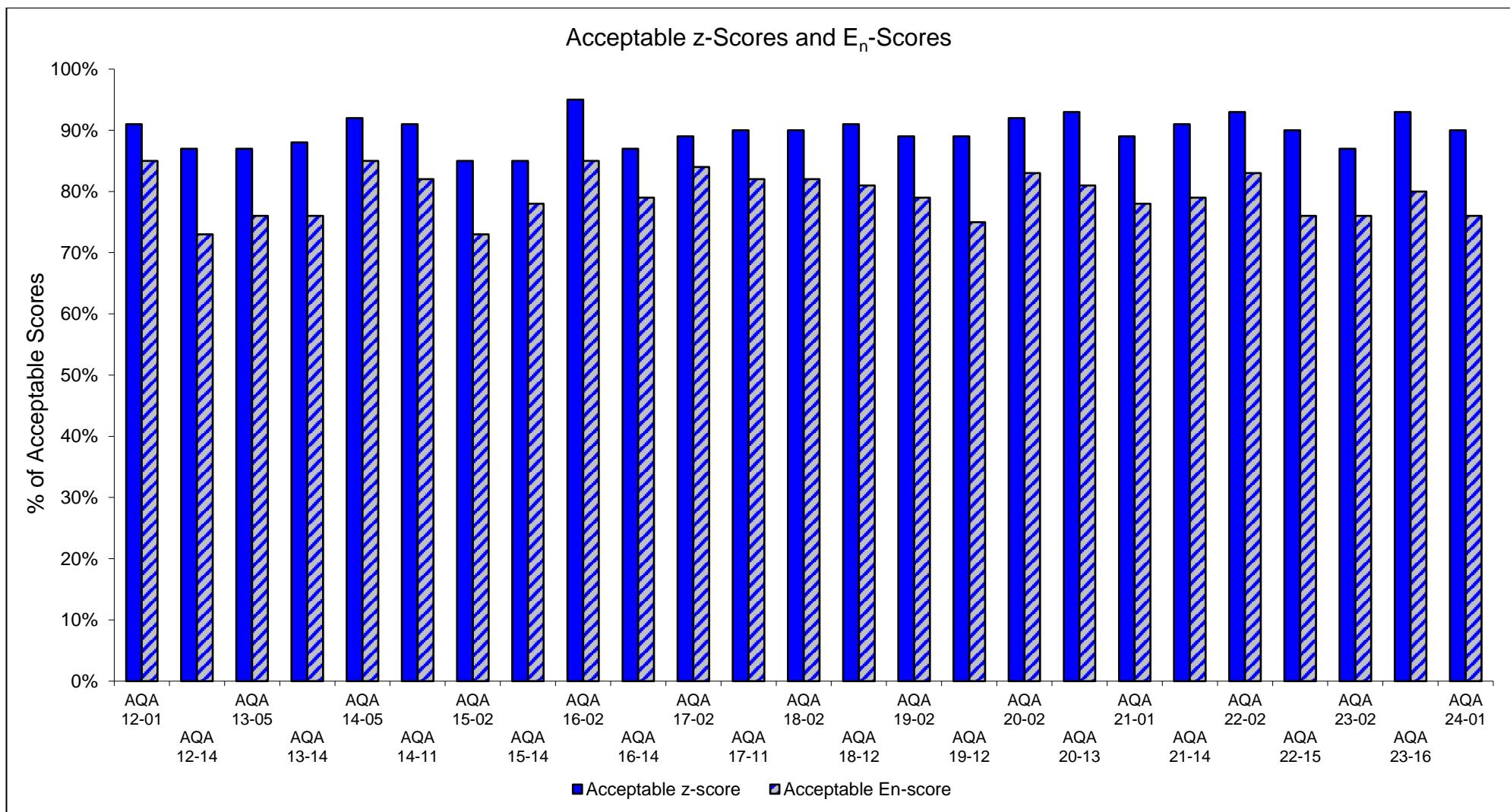


Figure 92 Participants' Performance over Time (2012-2024)

7 REFERENCES

Note: For all undated references, the latest edition of the referenced document (including any amendments) applies.

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APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING

Sample Preparation

Sample S1 was a sediment soil material fortified for 6 elements, dried, ground and sieved prior to being divided into portions of approximately 30 g each.

Sample S2 was the same soil material as that used in the preparation of Sample S1 of PT study AQA 23-16, to which a known amount of water was added before the moist soil was mixed and divided into portions of 35 g each.⁶

Sample S3 was an agricultural soil material that was ground, sieved, mixed and divided into portions of 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 in Samples S1 and S3, with the exception of calcium chloride-extractable B, Colwell K, Colwell P, electrical conductivity, exchangeable Ca, K, Mg and Na, PBI +ColP, pH, S, total carbon, total nitrogen, total organic carbon and total P in S3. A partial homogeneity test was also conducted for moisture content in S2. Three bottles were analysed in duplicate, and the average of the results was reported as the homogeneity value. Measurements were made under repeatability conditions in random order.

Sample S2 was the same soil material as that used in the preparation of Sample S1 of AQA 23-16, to which a known amount of water was added. A full homogeneity test was conducted for this material in AQA 23-16 S1.⁶

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 were weighed into 50 mL graduated polypropylene centrifuge tubes. 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 83.

Table 83 Instrumental Technique used for Acid Extractable Elements

Analyte	Instrument	Internal Standard	Reaction/Collision Cell (if applicable)	Cell Mode/Gas (if applicable)	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Ion (m/z) / Wavelength (nm)
Ag	ICP-MS	Rh	ORS	He	NA	800	107 m/z
Al	ICP-MS	Rh	NA	NA	NA	800	27 m/z
As	ICP-MS	Rh	ORS	He	800	800	75 m/z
B	ICP-MS	Rh	NA	NA	800	NA	11 m/z
Ba	ICP-MS	Rh	ORS	He	NA	800	137 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-OES	Y	NA	NA	800	NA	422.673 nm
Cd	ICP-MS	Rh	ORS	He	800	800	111 m/z
Co	ICP-MS	Rh	ORS	He	800	NA	59 m/z
Cr	ICP-MS	Rh	ORS	He	800	800	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-OES	Y	NA	NA	800	NA	238.204 nm
Ga	ICP-MS	Rh	ORS	He	800	NA	71 m/z
Gd	ICP-MS	Rh	ORS	He	NA	800	157 m/z
Hg	ICP-MS	Rh	ORS	He	800	800	201 m/z
K	ICP-OES	Y	NA	NA	800	NA	766.491 nm
La	ICP-MS	Rh	ORS	He	NA	800	139 m/z
Li	ICP-MS	Rh	ORS	He	800	NA	7 m/z
Mg	ICP-OES	Y	NA	NA	800	NA	279.078 nm
Mn	ICP-MS	Rh	ORS	He	800	800	55 m/z
Mo	ICP-MS	Rh	ORS	He	800	800	95 m/z
Na	ICP-OES	Y	NA	NA	800	NA	588.995 nm
Ni	ICP-MS	Rh	ORS	He	800	800	60 m/z
P	ICP-OES	Y	NA	NA	800	NA	177.434 nm
Pb	ICP-MS	Ir	ORS	He	800	NA	Average of 206, 207, 208 m/z
Rb	ICP-MS	Rh	ORS	He	800	NA	85 m/z
Sb	ICP-MS	Rh	ORS	He	800	NA	121 m/z
Se	ICP-MS	Rh	ORS	HEHe	800	800	78 m/z
Sm	ICP-MS	Rh	ORS	He	NA	800	147 m/z
Sn	ICP-MS	Rh	ORS	He	800	NA	118 m/z
Sr	ICP-MS	Rh	ORS	He	800	NA	88 m/z
Th	ICP-MS	Rh	ORS	He	800	NA	232 m/z
Tl	ICP-MS	Rh	ORS	He	NA	800	205 m/z
U	ICP-MS	Ir	ORS	He	NA	800	238 m/z
V	ICP-MS	Rh	ORS	He	800	800	51 m/z
Zn	ICP-MS	Rh	ORS	He	800	800	64 m/z

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 13258’⁷; 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 84.

Table 84 Uncertainty of Assigned Value for As in Sample S1

No. results (p)	20
Robust Average	35.2 mg/kg
$S_{rob\ av}$	3.6
$u_{rob\ av}$	1.0 mg/kg
k	2
$U_{rob\ av}$	2.0 mg/kg

The assigned value for As in Sample S1 is **35.2 ± 2.0 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 14).

A worked example is set out below in Table 85.

Table 85 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
36.9 ± 7.65	35.2 ± 2.0	10% as PCV or 0.10 x 35.2 = = 3.52 mg/kg	$z = \frac{(36.9 - 35.2)}{3.52}$ z = 0.48	$E_n = \frac{(36.9 - 35.2)}{\sqrt{7.65^2 + 2.0^2}}$ E _n = 0.21

APPENDIX 3 - USING PT DATA FOR UNCERTAINTY ESTIMATION

When a laboratory has successfully participated in at least 6 proficiency testing studies (e.g. is demonstrating control of bias and verification of repeatability), the standard deviation from proficiency testing studies (the reproducibility between laboratories variation) can also be used to estimate the uncertainty of their measurement results.^{11, 13} An example is given.

Between 2009 and 2024 NMI carried out 29 proficiency tests of metals in soil. These studies involved analyses of acid-extractable elements at low and high levels in dried soil, moist soil, biosolid, clay, compost, sediment and sludge.

Laboratory X submitted results for As in all of these PTs. All reported results returned acceptable z-scores. This data can usefully be separated into two ranges of results 1 to 10 mg/kg and 10 to 100 mg/kg (Tables 86 and 87).

The pooled standard deviation of the robust CV over these PT samples for each concentration range gives estimates of the relative standard uncertainty of 13% and 9.6% respectively. Using a coverage factor of two gives relative expanded uncertainties of 26% and 20% respectively, at a level of confidence of approximately 95%.

Table 86 Laboratory X Reported Results for As at 1 to 10 mg/kg Level.

Study No.	Sample	Laboratory result mg/kg	Assigned value mg/kg	Number of laboratories	Robust CV of all results (%)
AQA 09-13	S1 – Biosolid	4.091	3.64	11	16
AQA 09-13	S2 – Soil	4.29	4.57	12	15
AQA 11-01	S1 – Biosolid	3.54	3.57	18	20
AQA 13-05	S1 – Soil	9.22	9.21	22	14
AQA 14-11	S1 – Sediment	7.91	7.37	21	12
AQA 15-02	S1 – Moist Sludge	8.29	7.02	22	13
AQA 15-02	S2 – Moist Sludge	7.42	7.02	17	11
AQA 15-14	S1 – Sediment	10	9.95	17	6.7
AQA 15-14	S2 – Soil	4.53	4.47	14	6.4
AQA 16-02	S2 – Clay	2.67	2.11	20	14
AQA 16-14	S1 – Soil	6.03	5.61	17	20
AQA 17-02	S1 – Soil	3.71	3.76	13	10
AQA 17-02	S2 – Soil	2.92	3.01	13	4
AQA 18-02	S1 – Compost	2.22	2.73	17	11
AQA 19-02	S1 – Soil	2.83	2.65	24	11
AQA 19-12	S1 – Soil	2.32	2.12	16	16
AQA 20-13	S1 – Biosolid	2.85	3.29	17	11
AQA 21-01	S1 – Sediment	7.02	6.26	18	6.9
AQA 21-01	S2 – Moist Sludge	3.99	3.58	13	13
AQA 22-02	S1 – Sediment	4.32	4.02	15	9.5
AQA 22-02	S2 – Moist Soil	3.57	3.56	13	6.2
AQA 22-15	S2 – Clay	4.29	3.63	19	17
AQA 23-02	S1 – Soil	4.41	4.12	16	5.9
AQA 23-02	S2 – Sludge	4.43	4.8	8	24
Average					12%*
$pooled s\% = \sqrt{\frac{(11-1)x16^2 + (12-1)x15^2 + \dots + (8-1)x24^2}{393-24}}$					13%

* The pooled standard deviation was used.

Table 87 Laboratory X Reported Results for As at 10 to 75 mg/kg Level.

Study No.	Sample	Laboratory result mg/kg	Assigned value mg/kg	Number of Laboratories	Robust CV of all results (%)
AQA 10-12	S1 – Soil	16.6	14.4	19	8.5
AQA 11-12	S1 – Moist Sludge	25	21.6	13	15
AQA 12-01	S1 – Sediment	18.4	17.3	21	8.1
AQA 12-14	S2 – Soil	16.6	14.8	20	11
AQA 13-14	S1 – Sandy Soil	16.6	15.1	21	10
AQA 14-05	S1 – Soil	13.2	12.3	25	7.8
AQA 17-11	S1 – Sediment	18.1	17.4	22	11
AQA 18-12	S2 – Soil	10.4	9.6	20	8
AQA 19-12	S2 – Sediment	21	19.9	19	9
AQA 20-02	S1 – Soil	18.8	21.6	23	8.8
	S2 – Moist Soil	16.5	17.8	24	6.7
AQA 21-14	S1 – Sediment	19.5	20.9	21	8.9
AQA 22-15	S2 – Sediment	58.6	56.8	22	7.8
AQA 23-16	S1 – Soil	10.9	12.3	18	9.7
	S2 – Soil	12.4	12.3	17	9.4
AQA 24-01	S1 – Soil	35.9	35.2	20	10
	S2 – Moist Soil	11.8	12.5	16	13
Average					9.6%*
$pooled\ s\% = \sqrt{\frac{(18x8.5^2 + 12x15^2 + \dots + 15x13^2)}{341 - 17}}$					9.6%

* The pooled standard deviation was used

Table 88 sets out the expanded uncertainty for results of the measurement of As in soil, biosolid, clay, sediment, sludge, sandy soil, moist soil, compost and agricultural soil over the ranges 1 to 10 mg/kg and 10 to 100 mg/kg.

Table 88 Uncertainty of As Results Estimated Using PT Data.

Results mg/kg	Uncertainty mg/kg
1.00	0.26
5.0	1.3
10.0	2.6
20.0	4.0
75	15
100	20

The estimates of 26% and 20% relative passes the test of being reasonable, and the analysis of the 41 different PT samples over sixteen years can be assumed to include all the relevant uncertainty components (different matrices, operators, reagents, calibrators etc.), and so complies with ISO 17025 requirements.⁹

APPENDIX 4 - ACRONYMS AND ABBREVIATIONS

AAS	Atomic Absorption Spectroscopy
APHA	American Public Health Association
AV	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
HV	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
RA	Robust Average
RM	Reference Material
CV _{rob}	Robust Coefficient of Variation
SD _{rob}	Robust Standard Deviation
SV	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 89 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	50	50	107
3	ICP-MS	Rh	ORS	He	500	500	107
7	ICP-MS	Rh	CRI	He	NA	500	107
9	ICP-MS	Rh		He	NA		
10	ICP-MS				NA		
11	ICP-MS	Y 89	KED		NA	5000	107
13	ICP-MS	103 Rh	ORS	He	N/A	50	107
14	ICP-MS	Rh	ORS	He	NA	1000	107
15	ICP-MS	103	ORS	He	NA	10	107
16	ICP-MS	Sc,Ir,Rh	ORS	He	NA		107
17	ICP-MS	Rh	ORS	He	800	NA	107
19	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	328.069nm
20	ICP-MS	Rh	NA	NA	NA	625	109
21	ICP-MS	Rh	ORS	He	NA	500	107 (m/z)
23	ICP-MS	103 Rh	DRC	He	NA	20	107
25	ICP-OES-RV				NA		328.068

Table 90 Instrument Conditions Al

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	Sc	ORS	He	50	50	27
3	ICP-MS	Sc	ORS	He	500	500	27
6	ICP-OES-AV				NA		396.153
7	ICP-MS	Sc	CRI	He	NA	500	27
9	ICP-MS	Sc		He	NA		
10	ICP-OES-AV				NA		
11	ICP-MS	Sc-2 45	KED		NA	5000	27
13	ICP-MS	45 Sc	ORS	He	N/A	50	27
14	ICP-MS	Sc	ORS	He	NA	20000	27
15	ICP-MS	72	ORS	Standard Mode	NA	50	27
16	ICP-OES-AV	Eu			NA		236.705
17	ICP-MS	Rh	ORS	He	800	NA	27
19	ICP-OES	Eu & Cs	NA	NA	NA	50	236.707, 308.215, 396.15nm
20	ICP-MS	Sc	UC	He	NA	625	27
21	ICP-MS	Sc	ORS	He	NA	500	27 (m/z)
23	ICP-MS	45 Ge	DRC	He	NA	20	27
25	ICP-OES-RV				NA		167.019

Table 91 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	50	50	75
2	ICP-OES-AV					NA	
3	ICP-MS	Y	ORS	He	500	500	75
6	ICP-OES-AV						188.982
7	ICP-MS	Rh	CRI	He	500	500	75
9	ICP-MS	Rh		He			
10	ICP-MS						
11	ICP-MS	Ge-1 72	KED		5000	5000	75
12	ICP-MS	Rh	KED	He	1000	NA	75
13	ICP-MS	103 Rh	ORS	He	50	N/A	75
14	ICP-MS	Rh	ORS	HEHe	1000	1000	75
15	ICP-MS	72	ORS	He	50	50	75
16	ICP-MS	Sc,Ir,Rh	ORS	He			75
17	ICP-MS	Rh	ORS	He	800	NA	75
19	ICP-OES	Eu & Cs	NA	NA	50	50	188.89nm
20	ICP-MS	Rh	UC	He	625	625	75
21	ICP-MS	Rh	ORS	He	500	500	75 (m/z)
23	ICP-MS	72 Ge	DRC	He	20	20	75
25	ICP-OES-RV						188.98

Table 92 Instrument Conditions B

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV-buffer	Eu			50	50	249.772nm
3	ICP-MS	Sc	ORS	No gas	500	500	10
6	ICP-OES-AV					NA	208.957
7	ICP-MS	Sc	CRI	NA	500	NA	11
9	ICP-MS	Sc		He		NA	
10	ICP-OES-AV					NA	
11	ICP-MS	Y 89	KED		5000	NA	11
12	ICP-MS	Sc	KED	He	400	NA	11
13	ICP-MS	45 Sc	NA		N/A	50	11
14	ICP-MS	Sc	ORS	He	1000	NA	11
15	ICP-MS	89	ORS	Standard Mode	10	NA	11
16	ICP-MS	Sc,Ir,Rh	ORS	He		NA	11
19	ICP-OES	Eu & Cs	NA	NA	50	NA	249.773nm
20	ICP-MS	Sc	NA	NA	625	NA	10
21	ICP-MS	Sc	ORS	He	500	NA	11 (m/z)
23	ICP-MS	45 Sc	DRC	Other (No gas)	20	NA	11
25	ICP-OES-RV					NA	249.678

Table 93 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	50	50	137
3	ICP-MS	NA	ORS	NA	NA	500	135
6	ICP-OES-AV				NA		233.527
7	ICP-MS	Rh	CRI	He	NA	500	135
9	ICP-MS	Rh		He	NA		
10	ICP-OES-AV				NA		
11	ICP-MS	In-1 115	KED		NA	5000	138
13	ICP-MS	103 Rh	ORS	He	50	50	137
14	ICP-MS	Rh	ORS	He	NA	1000	137
15	ICP-MS	159	ORS	He	NA	50	137
16	ICP-MS	Sc,Ir,Rh	ORS	He	NA		137
17	ICP-MS	Rh	ORS	He	800	NA	134Mini
19	ICP-OES	Eu & Cs	NA	NA	NA	50	585.369nm
20	ICP-MS	Rh	NA	NA	NA	625	138
21	ICP-MS	Rh	ORS	He	NA	500	135 (m/z)
23	ICP-MS	103 Rh	DRC	He	NA	20	137
25	ICP-OES-RV				NA		455.403

Table 94 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	Sc	ORS	NA	50	50	9
3	ICP-MS	Sc	ORS	He	500	500	9
6	ICP-OES-AV					NA	313.107
7	ICP-MS	Sc	CRI	NA	500	NA	9
9	ICP-MS	Sc		NA		NA	
10	ICP-OES-AV					NA	
11	ICP-MS	Sc-2 45	KED		5000	NA	9
12	ICP-MS	Sc	KED	He	2000	NA	9
13	ICP-MS	45 Sc	NA		50	N/A	9
14	ICP-MS	Sc	ORS	He	1000	NA	9
15	ICP-MS	72	ORS	Standard Mode	50	NA	9
16	ICP-MS	Sc,Ir,Rh	ORS	He		NA	9
17	ICP-MS	Rh	ORS	He	800	NA	9
19	ICP-OES	Eu & Cs	NA	NA	50	NA	313.042nm
20	ICP-MS	Sc	NA	NA	625	NA	9
21	ICP-MS	Sc	ORS	Standard Mode	500	NA	9 (m/z)
23	ICP-MS	45 Sc	DRC	Other (No gas)	20	NA	9

Table 95 Instrument Conditions Bi

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Ir	ORS	He	50	50	209
3	ICP-MS	NA	ORS	NA	NA	500	209
7	ICP-MS	Lu	CRI	He	NA	500	209
9	ICP-MS	Lu		He	NA		
10	ICP-OES-AV				NA		
13	ICP-MS	193 Y	ORS	He	N/A	50	209
14	ICP-MS	Ir	ORS	He	NA	1000	209
15	ICP-MS	159	ORS	Standard Mode	NA	50	209
16	ICP-MS	Sc,Ir,Rh	ORS	He	NA		209
17	ICP-MS	Ir	ORS	He	800	NA	209
19	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	209 m/z
20	ICP-MS	Ir	NA	NA	NA	625	209
21	ICP-MS	Lu	ORS	He	NA	500	209 (m/z)
23	ICP-MS	175 Lu	DRC	He	NA	20	209

Table 96 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-buffer	Eu			50	50	315.887nm
3	NA	NA	NA	NA	NA	NA	40
6	ICP-OES-AV					NA	317.933
7	NA	NA	NA	NA	500	NA	NA
9	ICP-MS	Sc		H2		NA	
10	ICP-OES-AV					NA	
13	ICP-MS	45 Sc	ORS	He	N/A	50	44
16	ICP-OES-AV	Eu				NA	315.885
17	ICP-MS	Rh	ORS	He	800	800	43Mini
19	ICP-OES	Eu & Cs	NA	NA	50	NA	315.887, 370.602nm
20	ICP-MS	Sc	UC	He	625	NA	44
23	ICP-MS	72 Ge	DRC	He	100	NA	40
25	ICP-OES-RV					NA	315.887

Table 97 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	50	50	111
2	ICP-OES-AV					NA	
3	ICP-MS	Rh	ORS	He	500	500	112
6	ICP-OES-AV						228.802
7	ICP-MS	Rh	CRI	He	500	500	111
9	ICP-MS	Rh		He			
10	ICP-MS						
11	ICP-MS	In-1 115	KED		5000	5000	111
12	ICP-MS	Rh	KED	He	1000	NA	111
13	ICP-MS	103 Rh	ORS	He	50	N/A	111
14	ICP-MS	Rh	ORS	He	1000	1000	111
15	ICP-MS	103	ORS	He	50	50	111
16	ICP-MS	Sc,Ir,Rh	ORS	He			111
17	ICP-MS	Rh	ORS	He	800	NA	111Mini
19	ICP-OES	Eu & Cs	NA	NA	50	50	226.502nm
20	ICP-MS	Rh	NA	NA	625	625	111
21	ICP-MS	Rh	ORS	He	500	500	111 (m/z)
23	ICP-MS	103 Rh	DRC	He	20	20	111&114
25	ICP-OES-RV						214.439

Table 98 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	50	50	59
3	ICP-MS	Sc	ORS	He	500	500	58
6	ICP-OES-AV					NA	228.616
7	ICP-MS	Sc	CRI	He	500	NA	59
9	ICP-MS	Sc		He		NA	
10	ICP-OES-AV					NA	
11	ICP-MS	Sc 45	KED		5000	NA	59
12	ICP-MS	Ga	KED	He	2000	NA	59
13	ICP-MS	103 Rh	ORS	He	N/A	50	59
14	ICP-MS	Sc	ORS	He	1000	NA	59
15	ICP-MS	103	ORS	He	50	NA	59
16	ICP-MS	Sc,Ir,Rh	ORS	He		NA	59
17	ICP-MS	Rh	ORS	He	800	NA	59
19	ICP-OES	Eu & Cs	NA	NA	50	NA	228.616nm
20	ICP-MS	Ge	UC	He	625	NA	59
21	ICP-MS	Sc	ORS	He	500	NA	59 (m/z)
23	ICP-MS	45 Sc	DRC	He	20	NA	59
25	ICP-OES-RV					NA	228.615

Table 99 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	Sc	ORS	He	50	50	52
2	ICP-OES-AV					NA	
3	ICP-MS	Sc	ORS	He	500	500	52
6	ICP-OES-AV						267.716
7	ICP-MS	Sc	CRI	He	500	500	52
9	ICP-MS	Sc		He			
10	ICP-OES-AV						
11	ICP-MS	Sc 45	KED		5000	5000	52
12	ICP-MS	Sc	KED	He	1000	NA	52
13	ICP-MS	45 Sc	ORS	He	50	N/A	52
14	ICP-MS	Sc	ORS	He	1000	1000	52 + 53
15	ICP-MS	103	ORS	He	50	50	52
16	ICP-MS	Sc,Ir,Rh	ORS	He			52
17	ICP-MS	Rh	ORS	He	800	NA	52
19	ICP-OES	Eu & Cs	NA	NA	50	50	267.716nm
20	ICP-MS	Sc	UC	He	625	625	52
21	ICP-MS	Sc	ORS	He	500	500	52 (m/z)
23	ICP-MS	45 Sc	DRC	He	20	20	52
25	ICP-OES-RV						267.716

Table 100 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	ICP-MS	Rh	ORS	He	50	50	133
3	NA	NA	NA	NA	NA	Na	132
7	NA	NA	NA	NA	NA	500	NA
10	ICP-OES-AV				NA		
15	ICP-MS	159	ORS	He	NA	10	133
16	ICP-MS	Sc,Ir,Rh	ORS	He	NA		133
17	ICP-MS	Rh	ORS	He	800	NA	133
19	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	133 m/z

Table 101 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	50	50	63
2	ICP-OES-AV					NA	
3	ICP-MS	Sc	ORS	He	500	500	63
6	ICP-OES-AV						324.752
7	ICP-MS	Sc	CRI	He	500	500	63
9	ICP-MS	Sc		He			
10	ICP-OES-AV						
11	ICP-MS	Sc 45	KED		5000	5000	63
12	ICP-MS	Ga	KED	He	1000	NA	63
13	ICP-MS	103 Rh	ORS	He	50	N/A	63
14	ICP-MS	Sc	ORS	He	1000	1000	63
15	ICP-MS	103	ORS	He	50	50	63
16	ICP-MS	Sc,Ir,Rh	ORS	He			63
17	ICP-MS	Rh	ORS	He	800	NA	63Mini
19	ICP-OES	Eu & Cs	NA	NA	50	50	327.395nm
20	ICP-MS	Ge	UC	He	625	625	63
21	ICP-MS	Sc	ORS	He	500	500	63 (m/z)
23	ICP-MS	72 Ge	DRC	He	20	20	63
25	ICP-OES-RV						324.754

Table 102 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-buffer	Eu			50	50	258.588nm
3	ICP-MS	Sc	ORS	He	500	500	55
6	ICP-OES-RV					NA	238.204
7	ICP-MS	Sc	CRI	He	500	NA	56
9	ICP-MS	Sc		He		NA	
10	ICP-OES-AV					NA	
13	ICP-MS	103 Rh	ORS	He	N/A	50	133
16	ICP-OES-AV	Eu				NA	258.588
17	ICP-MS	Rh	ORS	He	800	800	56
19	ICP-OES	Eu & Cs	NA	NA	50	NA	238.204, 258.588, 259.940nm
20	ICP-MS	Sc	UC	He	625	NA	56
21	ICP-MS	Sc	ORS	He	500	NA	56 (m/z)
23	ICP-MS	45 Sc	DRC	He	20	NA	56
25	ICP-OES-RV					NA	234.35

Table 103 Instrument Conditions Ga

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	NA	50	50	71
3	NA	NA	NA	NA	NA	NA	69
9	ICP-MS	Sc		He		NA	
10	ICP-OES-AV					NA	
12	KED	He	2000	NA	59		
14	ICP-MS	Sc	ORS	He	1000	NA	69
16	ICP-MS	Sc,Ir,Rh	ORS	He		NA	71
17	ICP-MS	Rh	ORS	He	800	NA	71
19	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	71 m/z

Table 104 Instrument Conditions Gd

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	NA	50	50	157
3	NA	NA	NA	NA	NA	500	157
10	ICP-OES-AV				NA		
16	ICP-MS	Sc,Ir,Rh	ORS	He	NA		157
17	ICP-MS	Rh	ORS	He	800	NA	157
19	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	157 m/z

Table 105 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	CVAAS				50	50	253.7nm
2	CVAAS					NA	
3	ICP-MS	Lu	ORS	He	500	500	200
6	FIMS						253.7
7	ICP-MS	Lu	CRI	He	500	500	202
9	ICP-MS	Lu		He			
10	ICP-OES-AV						
11	ICP-MS	Ir 193	KED		5000	5000	202
12	ICP-MS	Tb	KED	He	1000	NA	201
13	CVAAS	Eu	NA		N/A	50	258.3
14	ICP-MS	Ir	ORS	He	1000	1000	202
15	ICP-MS	193	ORS	Standard Mode	10	50	202
16	AAS	NA					253.7
17	ICP-MS	Ir	ORS	He	800	NA	202
19	CETAC	NA	NA	NA	50	50	253.7nm
20	ICP-MS	Ir	NA	NA	625	625	201
21	ICP-MS	Lu	ORS	He	500	500	202 (m/z)
23	ICP-MS	175 Lu	DRC	He	20	20	201&202

Table 106 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-buffer	Eu			50	50	766.491nm
3	NA	NA	NA	NA	NA	NA	39
6	ICP-OES-RV					NA	766.49
7	NA	NA	NA	NA	500	NA	NA
9	ICP-MS	Sc		He		NA	
10	ICP-OES-AV					NA	
13	ICP-OES-AV-equation	103 Rh	NA		50	N/A	253.7
16	ICP-OES-AV	Eu				NA	766.491
17	ICP-MS	Rh	ORS	He	800	800	39
19	ICP-OES	Eu & Cs	NA	NA	50	NA	404.721nm, 766.491nm
20	ICP-MS	Sc	UC	He	625	NA	39
23	ICP-MS	72 Ge	DRC	He	100	NA	39
25	ICP-OES-RV					NA	766.491

Table 107 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	NA	50	50	140
3	NA	NA	NA	NA	NA	NA	138
7	NA	NA	NA	NA	NA	500	NA
10	ICP-OES-AV				NA		
15	ICP-MS	159	ORS	He	NA	50	139
16	ICP-MS	Sc,Ir,Rh	ORS	He	NA		139
17	ICP-MS	Rh	ORS	He	800	NA	139
19	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	139 m/z

Table 108 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	Sc	ORS	NA	50	50	7
3	ICP-MS	Sc	ORS	No gas	500	500	6
7	ICP-MS	Sc	CRI	NA	500	NA	7
9	ICP-MS	Sc		NA		NA	
10	ICP-OES-AV					NA	
11	ICP-MS	Sc - 45	KED		5000	NA	7
12	ICP-MS	Sc	KED	He	2000	NA	7
13	ICP-MS	45 Sc	ORS	He	N/A	50	39
14	ICP-MS	Sc	ORS	He	1000	NA	7
15	ICP-MS	72	ORS	H2	50	NA	7
16	ICP-MS	Sc,Ir,Rh	ORS	He		NA	7
17	ICP-MS	Rh	ORS	He	800	NA	7
19	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	7 m/z
20	ICP-MS	Sc	NA	NA	625	NA	7
21	ICP-MS	Sc	ORS	Standard Mode	500	NA	7 (m/z)
23	ICP-MS	45 Sc	DRC	Other (No gas)	20	NA	7
25	ICP-OES-RV					NA	670.783

Table 109 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-AV-buffer	Eu			50	50	383.829nm
3	NA	NA	NA	NA	NA	NA	24
6	ICP-OES-RV					NA	279.077
7	NA	NA	NA	NA	500	NA	NA
9	ICP-MS	Sc		He		NA	
10	ICP-OES-AV					NA	
13	ICP-MS	103 Rh	ORS	He	N/A	50	139
16	ICP-OES-AV	Eu				NA	383.33
17	ICP-MS	Rh	ORS	He	800	800	24
19	ICP-OES	Eu & Cs	NA	NA	50	NA	383.829nm
20	ICP-MS	Sc	UC	He	625	NA	25
23	ICP-MS	72 Ge	DRC	He	100	NA	24
25	ICP-OES-RV					NA	383.23

Table 110 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	Sc	ORS	He	50	50	55
3	ICP-MS	Sc	ORS	He	500	500	54
6	ICP-OES-AV						257.61
7	ICP-MS	Sc	CRI	He	500	500	55
9	ICP-MS	Sc		He			
10	ICP-OES-AV						
11	ICP-MS	Sc 45	KED		5000	5000	55
12	ICP-MS	Sc	KED	He	2000	NA	55
13	ICP-MS	45 Sc	NA		N/A	50	7
14	ICP-MS	Sc	ORS	He	10000	20000	55
15	ICP-MS	72	ORS	Standard Mode	50	50	55
16	ICP-MS	Sc,Ir,Rh	ORS	He			55
17	ICP-MS	Rh	ORS	He	800	NA	55
19	ICP-OES	Eu & Cs	NA	NA	50	50	261.021nm
20	ICP-MS	Sc	UC	He	625	625	55
21	ICP-MS	Sc	ORS	He	500	500	55 (m/z)
23	ICP-MS	45 Sc	DRC	He	20	20	55
25	ICP-OES-RV						257.61

Table 111 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	50	50	95
3	ICP-MS	Y	ORS	He	500	500	95
6	ICP-OES-AV						202.031
7	ICP-MS	Rh	CRI	He	500	500	95
9	ICP-MS	Rh		He			
10	ICP-OES-AV						
11	ICP-MS	Y 89	KED		5000	5000	98
12	ICP-MS	Rh	KED	He	2000	NA	98
13	ICP-MS	45 Sc	ORS	He	N/A	50	24
14	ICP-MS	Rh	ORS	He	1000	1000	95
15	ICP-MS	89	ORS	He	50	50	95
16	ICP-MS	Sc,Ir,Rh	ORS	He			95
17	ICP-MS	Rh	ORS	He	800	NA	95
19	ICP-OES	Eu & Cs	NA	NA	50	50	202.032nm
20	ICP-MS	Rh	NA	NA	625	625	95
21	ICP-MS	Rh	ORS	He	500	500	95 (m/z)
23	ICP-MS	103 Rh	DRC	He	20	20	95
25	ICP-OES-RV						202.032

Table 112 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-buffer	Eu			50	50	589.592nm
3	NA	NA	NA	NA	NA	NA	22
6	ICP-OES-AV					NA	589.592
7	NA	NA	NA	NA	500	NA	NA
9	ICP-MS	Sc		He		NA	
10	ICP-OES-AV					NA	
13	ICP-MS	45 Sc	ORS	He	50	N/A	55
16	ICP-OES-AV	Eu				NA	589.592
19	ICP-OES	Eu & Cs	NA	NA	50	NA	330.237, 589.592nm
20	ICP-MS	Sc	UC	He	625	NA	23
23	ICP-MS	72 Ge	DRC	He	100	NA	23
25	ICP-OES-RV					NA	589.592

Table 113 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	50	50	60
2	ICP-OES-AV					NA	
3	ICP-MS	Sc	ORS	He	500	500	58
6	ICP-OES-AV						231.604
7	ICP-MS	Sc	CRI	He	500	500	60
9	ICP-MS	Sc		He			
10	ICP-OES-AV						
11	ICP-MS	Sc 45	KED		5000	5000	58
12	ICP-MS	Ga	KED	He	1000	NA	60
13	ICP-MS	103 Rh	ORS	He	50	N/A	95
14	ICP-MS	Sc	ORS	He	1000	1000	60
15	ICP-MS	103	ORS	He	50	50	60
16	ICP-MS	Sc,Ir,Rh	ORS	He			60
17	ICP-MS	Rh	ORS	He	800	NA	60
19	ICP-OES	Eu & Cs	NA	NA	50	50	231.604nm
20	ICP-MS	Ge	UC	He	625	625	60
21	ICP-MS	Sc	ORS	He	500	500	60 (m/z)
23	ICP-MS	72 Ge	DRC	He	20	20	60
25	ICP-OES-RV						216.555

Table 114 Instrument Conditions P

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV-buffer	Eu			50	50	185.827nm
3	NA	NA	NA	NA	NA	NA	30
7	NA	NA	NA	NA	500	NA	NA
9	ICP-OES-AV	Sc		NA		NA	
10	ICP-OES-AV					NA	
13	ICP-MS	45 Sc	ORS	He	N/A	50	23
16	ICP-OES-AV	Eu				NA	185.827
17	ICP-MS	Rh	ORS	HEHe	800	800	31
20	ICP-MS	Sc	UC	He	625	NA	31
21	ICP-MS	Sc	ORS	He	500	NA	31 (m/z)
23	ICP-MS	72 ge	DRC	He	100	NA	31

Table 115 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	50	50	208
2	ICP-OES-AV					NA	
3	ICP-MS	Lu	ORS	He	500		207
6	ICP-OES-AV					NA	220.353
7	ICP-MS	Lu	CRI	He	500	NA	208
9	ICP-MS	Lu		He		NA	
10	ICP-OES-AV					NA	
11	ICP-MS				5000	NA	208
12	ICP-MS	Tb	KED	He	1000	NA	206+207+208
13	ICP-MS	103 Rh	ORS	He	50	N/A	60
14	ICP-MS	Ir	ORS	He	1000	NA	206 + 207 + 208
15	ICP-MS	159	ORS	Standard Mode	50	NA	208
16	ICP-MS	Sc,Ir,Rh	ORS	He		NA	208
17	ICP-MS	Ir	ORS	He	800	NA	208
19	ICP-OES	Eu & Cs	NA	NA	50	NA	185.827nm
20	ICP-MS	Ir	NA	NA	625	NA	206+207+208
21	ICP-MS	Lu	ORS	He	500	NA	208 (m/z)
23	ICP-MS	175 Lu	DRC	He	20	NA	208
25	ICP-OES-RV					NA	220.353

Table 116 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	50	50	85
3	NA	NA	NA	NA	NA	NA	85
7	NA	NA	NA	NA	500	NA	NA
10	ICP-OES-AV					NA	
12	ICP-MS	Rh	KED	He	2000	NA	85
14	ICP-MS	Rh	ORS	He	1000	NA	85
15	ICP-MS	89	ORS	He	50	NA	85
16	ICP-MS	Sc,Ir,Rh	ORS	He		NA	85
17	ICP-MS	Rh	ORS	He	800	NA	85
19	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	220.353nm

Table 117 Instrument Conditions S

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV-buffer	Eu			50	50	181.972nm
3	NA	NA	NA	NA	NA	NA	32
7	NA	NA	NA	NA	500	NA	NA
9	ICP-OES-AV	Sc		NA		NA	
10	ICP-OES-AV					NA	
13	ICP-OES-AV-equation	Eu	NA		N/A	50	185.8
16	ICP-OES-AV	Eu				NA	181.972
17	ICP-OES-RV	Y	NA	NA	NA	800	181.972
20	ICP-OES-AV				625	NA	181.975
23	ICP-MS	72 Ge	DRC	H2	100	NA	33&34

Table 118 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	50	50	121
3	ICP-MS	In	ORS	He	500		121
6	ICP-OES-AV					NA	206.836
7	ICP-MS	Rh	CRI	He	500	NA	123
9	ICP-MS	Rh		He		NA	
10	ICP-MS					NA	
12	ICP-MS	Rh	KED	He	2000	NA	121
13	ICP-MS	193 Y	ORS	He	50	N/A	208
14	ICP-MS	Rh	ORS	He	1000	NA	121
15	ICP-MS	193	ORS	Standard Mode	50	NA	121
16	ICP-MS	Sc,Ir,Rh	ORS	He		NA	121
17	ICP-MS	Rh	ORS	He	800	NA	121
19	ICP-OES	Eu & Cs	NA	NA	50	NA	178.165,181.972nm
20	ICP-MS	Rh	NA	NA	625	NA	121
21	ICP-MS	Rh	ORS	He	500	NA	123 (m/z)
23	ICP-MS	103 Rh	DRC	He	20	NA	121
25	ICP-OES-RV					NA	217.582

Table 119 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	HEHe	50	50	78
3	ICP-MS	Ge	ORS	H2	500	500	78
6	ICP-OES-Hydride						196.026
7	ICP-MS	Rh	CRI	H2	500	500	78
9	ICP-MS	Rh		H2			
10	ICP-MS						
11	ICP-MS	Ge-1 72	KED		5000	5000	82
12	ICP-MS	Te	KED	He	400	NA	82
13	ICP-MS	103 Rh	ORS	He	N/A	50	85
14	ICP-MS	Rh	ORS	HEHe	1000	1000	78
15	ICP-MS	72	ORS	H2	10	10	78
16	ICP-MS	Sc,Ir,Rh	ORS	He			78
17	ICP-MS	Rh	ORS	HEHe	800	NA	78
19	ICP-OES	Eu & Cs	NA	NA	50	50	206.834nm
20	ICP-MS	Rh	UC	He	625	625	82
21	ICP-MS	Rh	ORS	H2	500	500	78 (m/z)
23	ICP-MS	72 Ge	DRC	HEHe	20	20	78
25	ICP-OES-RV						196.026

Table 120 Instrument Conditions Sm

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	NA	50	50	147
3	NA	NA	NA	NA	NA	NA	150
10	ICP-OES-AV				NA		
16	ICP-MS	Sc,Ir,Rh	ORS	He	NA		147
17	ICP-MS	Rh	ORS	He	800	NA	147
19	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	147 m/z

Table 121 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	50	50	118
3	ICP-MS	Rh	ORS	He	500	500	118
7	ICP-MS	Rh	CRI	He	500	NA	118
9	ICP-MS	Rh		He		NA	
10	ICP-OES-AV					NA	
11	ICP-MS	In-1115	KED		5000	NA	118
12	ICP-MS	Rh	KED	He	2000	NA	120
13	ICP-OES-AV-equation	Eu	NA		N/A	50	178.2
14	ICP-MS	Rh	ORS	He	1000	NA	118
15	ICP-MS	103	ORS	He	50	NA	120
16	ICP-MS	Sc,Ir,Rh	ORS	He		NA	118
17	ICP-MS	Rh	ORS	He	800	NA	118
19	ICP-OES	Eu & Cs	NA	NA	50	NA	189.926nm
21	ICP-MS	Rh	ORS	He	500	NA	118 (m/z)
23	ICP-MS	103 Rh	DRC	He	20	NA	118
25	ICP-OES-RV					NA	189.925

Table 122 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-MS	Rh	ORS	He	50	50	88
3	ICP-MS	Ge	ORS	He	500	500	87
7	ICP-MS	Rh	CRI	He	500	NA	88
9	ICP-MS	Rh		He		NA	
10	ICP-OES-AV					NA	
11	ICP-MS				5000	NA	88
12	ICP-MS	Rh	KED	He	2000	NA	88
13	ICP-MS	103 Rh	ORS	He	50	50	78
14	ICP-MS	Rh	ORS	He	1000	NA	88
15	ICP-MS	89	ORS	He	50	NA	88
16	ICP-MS	Sc,Ir,Rh	ORS	He		NA	88
17	ICP-MS	Rh	ORS	He	800	NA	88
19	ICP-OES	Eu & Cs	NA	NA	50	NA	430.545nm
20	ICP-MS	Rh	NA	NA	625	NA	88
21	ICP-MS	Rh	ORS	He	500	NA	88 (m/z)
23	ICP-MS	72 Ge	DRC	He	20	NA	88

Table 123 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	50	50	232
3	NA	NA	NA	NA	NA	NA	232
7	NA	NA	NA	NA	500	NA	NA
9	ICP-MS	Lu		He		NA	
10	ICP-OES-AV					NA	
14	ICP-MS	Ir	ORS	He	1000	NA	232
16	ICP-MS	Sc,Ir,Rh	ORS	He		NA	232
17	ICP-MS	Ir	ORS	He	800	NA	232
19	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	232 m/z
20	ICP-MS	Ir	NA	NA	625	NA	232

Table 124 Instrument Conditions Tl

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	50	50	205
3	ICP-MS	Lu	ORS	He	500	500	204
7	ICP-MS	Lu	CRI	He	NA	500	205
9	ICP-MS	Lu		He	NA		
10	ICP-OES-AV				NA		
13	ICP-MS	103 Rh	ORS	He	50	50	118
14	ICP-MS	Ir	ORS	He	NA	1000	205
15	ICP-MS	159	ORS	Standard Mode	NA	10	205
16	ICP-MS	Sc,Ir,Rh	ORS	He	NA		205
17	ICP-MS	Ir	ORS	He	800	NA	205
20	ICP-MS	Ir	NA	NA	NA	625	205
21	ICP-MS	Lu	ORS	He	NA	500	205 (m/z)
23	ICP-MS	175 Lu	DRC	He	NA	20	205
25	ICP-OES-RV				NA		190.794

Table 125 Instrument Conditions U

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS	Ir	ORS	He	50	50	238
3	ICP-MS	Lu	ORS	He	500	500	238
7	ICP-MS	Lu	CRI	He	NA	500	238
9	ICP-MS	Lu		He	NA		
10	ICP-OES-AV				NA		
11	ICP-MS	Ir 193	KED		NA	5000	238
13	ICP-MS	103 Rh	ORS	He	N/A	50	88
14	ICP-MS	Ir	ORS	He	NA	1000	238
15	ICP-MS	159	ORS	Standard Mode	NA	50	238
16	ICP-MS	Sc,Ir,Rh	ORS	He	NA		238
17	ICP-MS	Ir	ORS	He	800	NA	238
19	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	238 m/z
20	ICP-MS	Ir	NA	NA	NA	625	238
21	ICP-MS	Lu	ORS	He	NA	500	238 (m/z)
23	ICP-MS	175 Lu	DRC	He	NA	20	238

Table 126 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	Sc	ORS	He	50	50	51
3	ICP-MS	Sc	ORS	He	500	500	50
6	ICP-OES-AV						292.402
7	ICP-MS	Sc	CRI	He	500	500	51
9	ICP-MS	Sc		He			
10	ICP-OES-AV						
11	ICP-MS	Sc 45	KED		5000	5000	51
12	ICP-MS	Sc	KED	He	400	NA	51
13	ICP-MS	193 Y	ORS	He	N/A	50	232
14	ICP-MS	Sc	ORS	He	1000	1000	51
15	ICP-MS	103	ORS	He	50	50	51
16	ICP-MS	Sc,Ir,Rh	ORS	He			51
17	ICP-MS	Rh	ORS	He	800	NA	51
19	ICP-OES	Eu & Cs	NA	NA	50	50	311.837nm
20	ICP-MS	Sc	UC	He	625	625	51
21	ICP-MS	Sc	ORS	He	500	500	51 (m/z)
23	ICP-MS	72 Ge	DRC	He	20	20	51
25	ICP-OES-RV						292.401

Table 127 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	50	50	68
2	ICP-OES-AV					NA	
3	ICP-MS	Sc	ORS	He	500	500	65
6	ICP-OES-AV						213.857
7	ICP-MS	Sc	CRI	He	500	500	66
9	ICP-MS	Sc		He			
10	ICP-OES-AV						
11	ICP-MS	Sc 45	KED		5000	5000	66
12	ICP-MS	Ga	KED	He	1000	NA	66
13	ICP-MS	193 Y	ORS	He	N/A	50	205
14	ICP-MS	Sc	ORS	He	1000	1000	66
15	ICP-MS	72	ORS	He	50	50	66
16	ICP-MS	Sc,Ir,Rh	ORS	He			66
17	ICP-MS	Rh	ORS	He	800	NA	64Mini
19	ICP-OES	Eu & Cs	NA	NA	50	50	206.2, 334.502nm
20	ICP-MS	Ge	UC	He	625	625	66
21	ICP-MS	Sc	ORS	He	500	500	66 (m/z)
23	ICP-MS	72 Ge	DRC	He	20	20	66
25	ICP-OES-RV						206.2

Table 128 Instrument Conditions Exchangeable Ca²⁺

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm) /Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV-buffer	Eu			20	315.887nm
4	ICP-OES-RV					
10	ICP-OES-AV	Y	NA			
13	ICP-OES	Eu	NA		1	315.9
16	ICP-OES-RV					315.887
17	ICP-OES-RV	Y	NA	NA	40	422.673
19	ICP-OES	Eu & Cs	NA	NA	500	315.887, 370.602nm
20	ICP-OES-RV	Y	NA		20	317.933
23	ICP-MS	72 Ge	DRC	He	200	40
25	ICP-OES-AV					422.673

Table 129 Instrument Conditions Exchangeable K⁺

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/Ion(m/z)/Absorbance(nm)
1	ICP-OES-AV-buffer	Eu			20	766.491nm
4	ICP-OES-RV					
10	ICP-OES-AV	Y	NA			
13	ICP-OES	Eu	NA		1	383.8
16	ICP-OES-RV					383.829
17	ICP-OES-RV	Y	NA	NA	40	766.491
19	ICP-OES	Eu & Cs	NA	NA	500	383.829nm
20	ICP-OES-RV	Y	NA		20	766.49
23	ICP-MS	72 Ge	DRC	He	200	39
25	ICP-OES-AV					766.491

Table 130 Instrument Conditions Exchangeable Mg²⁺

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/Ion(m/z)/Absorbance(nm)
1	ICP-OES-AV-buffer	Eu			20	383.829nm
4	ICP-OES-RV					
10	ICP-OES-AV	Y	NA			
13	ICP-OES	Eu	NA		1	766.5
16	ICP-OES-RV					589.592
17	ICP-OES-RV	Y	NA	NA	40	279.078
19	ICP-OES	Eu & Cs	NA	NA	500	330.237, 589.592nm
20	ICP-OES-RV	Y	NA		20	285.213
23	ICP-MS	72 Ge	DRC	He	200	24
25	ICP-OES-AV					285.213

Table 131 Instrument Conditions Exchangeable Na⁺

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/Ion(m/z)/Absorbance(nm)
1	ICP-OES-AV-buffer	Eu			20	589.592nm
4	ICP-OES-RV					
10	ICP-OES-AV	Y	NA			
13	ICP-OES	Eu	NA		1	589.6
16	ICP-OES-RV					253.7
17	ICP-OES-RV	Y	NA	NA	40	588.995
19	ICP-OES	Eu & Cs	NA	NA	500	404.721nm, 766.491nm
20	ICP-OES-RV	Y	NA		20	589.592
23	ICP-MS	72 Ge	DRC	He	200	23
25	ICP-OES-AV					589.592

END OF REPORT