



# Proficiency Test Final Report

## AQA 22-02

# Metals, Nutrients and Exchangeable Bases in Soil

June 2022



## **ACKNOWLEDGMENTS**

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

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

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

Luminita Antin

Andrew Evans

Hamish Lenton

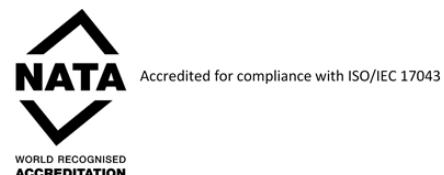
Isaac Schipp

Raluca Iavetz

Manager, Chemical Reference Values

Phone: 61-2-9449 0111

[proficiency@measurement.gov.au](mailto:proficiency@measurement.gov.au)



## TABLE OF CONTENTS

1	INTRODUCTION	5
1.1	NMI Proficiency Testing Program	5
1.2	Study Aims	5
1.3	Study Conduct	5
2	STUDY INFORMATION	5
2.1	Selection of Matrices and Inorganic Analytes	5
2.2	Participation	6
2.3	Test Material Specification	6
2.4	Laboratory Code	6
2.5	Sample Preparation, Analysis and Homogeneity Testing	6
2.6	Stability of Analytes	6
2.7	Sample Storage, Dispatch and Receipt	6
2.8	Instructions to Participants	7
2.9	Interim Report	8
3	PARTICIPANT LABORATORY INFORMATION	8
3.1	Test Method Summaries	8
3.2	Basis of Participants' Measurement Uncertainty Estimates	14
3.3	Participant Comments on this PT Study or Suggestions for Future Studies	16
4	PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS	17
4.1	Results Summary	17
5	TABLES AND FIGURES	19
6	DISCUSSION OF RESULTS	133
6.1	Assigned Value	133
6.2	Measurement Uncertainty Reported by Participants	133
6.3	E <sub>n</sub> -score	134
6.4	z-Score	134
6.5	Participants' Results and Analytical Methods for Acid Extractable Elements	137
6.6	Participants' Results and Analytical Methods for Exchangeable Cations	150
6.7	Participants' Results and Analytical Methods for Colwell P and Colwell K	151
6.8	Participants' Results and Analytical Methods for Phosphorus Buffer Index-PBI <sub>+ColP</sub>	152
6.9	Participants' Results and Analytical Methods for Total P	153
6.10	Participants' Results and Analytical Methods for Total Nitrogen	153
6.11	Participants' Results and Analytical Methods for Total Carbon and Total Organic Carbon	154
6.12	Comparison with Previous NMI Proficiency Tests of Metals in Soil	155
6.13	Reference Materials and Certified Reference Materials	155
7	REFERENCES	158

APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING	160
APPENDIX 2 - ASSIGNED VALUE, Z-SCORE AND $E_N$ SCORE CALCULATION	163
APPENDIX 3 - USING PT DATA FOR UNCERTAINTY ESTIMATION	164
APPENDIX 4 - ACRONYMS AND ABBREVIATIONS	166
APPENDIX 5 - INSTRUMENT DETAILS	168

## SUMMARY

This report presents the results of the proficiency test AQA 22-02, metals, nutrients and exchangeable bases in sediment and soil. The study focused on the measurement of the following acid extractable elements: Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Rb, S, Sb, Se, Sn, Sr, Th, Tl, U, V and Zn. Measurement of total P, P buffer index (with Colwell P)- PBI<sub>ColP</sub>, calcium chloride-extractable B, total carbon (TC), total organic carbon (TOC), total nitrogen (TN), Colwell P, Colwell K, EC, pH of 1:5 soil / 0.01 M CaCl<sub>2</sub> extract, exchangeable bases (Ca<sup>2+</sup>, Mg<sup>2+</sup>, Na<sup>+</sup>, K<sup>+</sup>) - 1M NH<sub>4</sub>Cl extract and moisture content was also included in the program.

The sample set consisted of one dried sediment sample, one moist sediment sample and one agricultural soil sample. The assigned values were the robust average of participants' results. The associated uncertainties were estimated from the robust standard deviation of the participants' results.

30 laboratories enrolled and 28 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<sub>n</sub>-scores.

Of 890 z-scores, 828 (93%) returned a satisfactory score of |z| ≤ 2.0.

Of 890 E<sub>n</sub>-scores, 740 (83%) were satisfactory with |E<sub>n</sub>| ≤ 1.0.

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

The tests that presented the most analytical difficulty to participating laboratories were: Th and Li and B. Measurement of high-level Ca and Fe in agricultural soil sample S3 also challenged participating laboratories. When a large sample size is taken for analysis, solubility limitations are expected if these analytes are present in a sample at a percentage level.

Calculation errors or reporting results in the wrong units were the main causes of some participants' poor performance.

- 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 923 numerical results, 881 were reported with an expanded measurement uncertainty. An example of estimating measurement uncertainty using only proficiency testing data is given in Appendix 3. A large number of participants reported for some results an estimate of expanded uncertainty which was larger than the results themselves.

- 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."<sup>1</sup> NMI PT studies target chemical testing in areas of high public significance such as trade, environment and food safety. NMI offers studies in:

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

AQA 22-02 is the 30<sup>th</sup> 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 and sediment;
- 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.<sup>2</sup> The statistical methods used are described in the NMI Chemical Proficiency Statistical Manual.<sup>3</sup> These documents have been prepared with reference to ISO Standard 17043<sup>1</sup> and The International Harmonized Protocol for Proficiency Testing of (Chemical) Analytical Laboratories.<sup>4</sup>

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

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

## **2 STUDY INFORMATION**

### **2.1 Selection of Matrices and Inorganic Analytes**

The 57 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)<sup>5</sup> and from analytes commonly measured in soil.

## 2.2 Participation

Thirty laboratories participated and twenty-eight submitted results.

The timetable of the study was:

Invitations issued: 23 February 2022  
Samples dispatched: 21 March 2022  
Results due: 22 April 2022  
Interim report issued: 28 April 2022

## 2.3 Test Material Specification

Three samples were provided for analysis:

**Sample S1** was 30 g of dried sediment;

**Sample S2** was 35 g of the dried sediment sample S1 to which a known amount of water was added.

**Sample S3** was 75 g of dried agricultural soil.

## 2.4 Laboratory Code

All participant laboratories were assigned a confidential code number.

## 2.5 Sample Preparation, Analysis and Homogeneity Testing

Test samples from previous studies have been demonstrated to be sufficiently homogeneous for the evaluation of participants' performance. Therefore, only a partial homogeneity test was conducted for all analytes with the exception of Sb, calcium chloride-extractable B, colwell K and PBI.<sup>1</sup> The results of the partial homogeneity testing for these samples are reported in the present study as the homogeneity value.

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

## 2.6 Stability of Analytes

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

## 2.7 Sample Storage, Dispatch and Receipt

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

The samples were dispatched by courier on 21 March 2022.

The following items were packaged with the samples:

- a covering letter which included a description of the test samples and instructions for participants; and
- a form to confirm the receipt and condition of the samples.

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

## 2.8 Instructions to Participants

Participants were instructed as follows:

- Quantitatively analyse the samples using your normal test method.
- Sample S2, the moist sample, should be thoroughly mixed before removing a test portion. To avoid loss of moisture, do not leave the sample uncovered.
- For Sample S3 for determination of calcium chloride – extractable B,<sup>1</sup> exchangeable bases ( $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Na}^+$ ,  $\text{K}^+$ ) - 1M  $\text{NH}_4\text{Cl}$  extract<sup>2</sup> and of P buffer index (with Colwell P)- PBI<sub>+ColP</sub><sup>3</sup>, 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 ( $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Na}^+$ ,  $\text{K}^+$ ) - 1M  $\text{NH}_4\text{Cl}$  extract. Except for pH and EC, for all the other tests, report results on as received basis in units of mg/kg. EC results are to be reported in units of  $\mu\text{S}/\text{cm}$ .

SAMPLE S1		SAMPLE S2		SAMPLE S3	
Test acid extractable	Approximate Conc. Range (as received basis) mg/kg	Test acid extractable	Approximate Conc. Range (dry weight basis) mg/kg	Test	Approximate Conc. Range (as received basis) mg/kg
As	0.5-20	Ag	0.5-20	Ca (acid extractable)	500-20000
B	0.5-20	As	0.5-20	Fe (acid extractable)	2500-100000
Be	0.5-20	Al	500-20000	K (acid extractable)	250-10000
Bi	0.5-20	Ba	10-400	Mg (acid extractable)	2500-100000
Cd	0.5-20	Cd	0.5-20	Na (acid extractable)	250-10000
Cr	10-400	Co	0.5-20	P (acid extractable)	250-10000
Cu	10-400	Cu	10-400	S (acid extractable)	Not Available
Hg	0.5-20	Hg	0.5-20	Calcium chloride –extractable B <sup>1</sup>	Not Available
Mn	10-400	Li	0.5-20	Exchangeable Ca-1M $\text{NH}_4\text{Cl}$ extract <sup>2</sup>	>0.5 cmol(+)/kg
Mo	0.5-20	Mn	10-400	Exchangeable Mg-1M $\text{NH}_4\text{Cl}$ extract <sup>2</sup>	>0.10 cmol(+)/kg
Ni	10-400	Ni	10-400	Exchangeable Na-1M $\text{NH}_4\text{Cl}$ extract <sup>2</sup>	>0.05 cmol(+)/kg
Pb	10-400	Sb	10-400	Exchangeable K-1M $\text{NH}_4\text{Cl}$ extract <sup>2</sup>	>0.05 cmol(+)/kg
Rb	0.5-20	Se	0.5-20	P buffer index (with Colwell P)- PBI <sub>+ColP</sub> <sup>3</sup>	25-500
Se	0.5-20	Sr	0.5-20	Colwell P	Not Available
Sn	0.5-20	Tl	0.5-20	Colwell K	Not Available
Th	0.5-20	U	0.5-20	Total P	Not Available

V	10-400	Zn	10-400	pH of 1:5soil/0.01M $\text{CaCl}_2$ extract	Not Available
Zn	10-400	Moisture Content	10-60%	EC	>500 $\mu\text{S}/\text{cm}$
				Total Carbon	2500-50000
				Total Organic Carbon	2500-50000
				Total Nitrogen	250-5000

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

- Report results using the electronic results sheet emailed to you;
- Report results as you would report to a client. For each analyte, report the expanded measurement uncertainty.
- Please send us all the requested details regarding the test method.
- Return the completed results sheet by e-mail (proficiency@measurement.gov.au), by 8 April 2022.

The due date for results was extended to 22 April 2022 due to delays in sample delivery to some of our overseas participants.

## 2.9 Interim Report

An interim report was emailed to participants on 28 April 2022.

## 3 PARTICIPANT LABORATORY INFORMATION

### 3.1 Test Method Summaries

Summaries of test methods are transcribed in Tables 1 to 10. The instruments and settings reported by participants are presented in Appendix 5.

Table 1 Methodology for Acid Extractable Elements

Lab. Code	Method Reference	Sample Mass (g)	Temp. (°C)	Time (min)	Vol. HNO <sub>3</sub> (mL)	Vol. HCl (mL)	Vol. HNO <sub>3</sub> (1:1) (mL)	Vol. HCl (1:1) (mL)	Vol. H <sub>2</sub> O <sub>2</sub> (mL)	Other (mL)
1	USEPA 3051A (Modification)	1	170	15			8	2		
2	200.2	0.5	95	30			2			5 (HCl (1+4))
3	USEPA3050/6010/6020/200.7/200.8	1 to 3	90-98	120	3	3				
4	USEPA method 200.2 Revision 2.8	1	95	60			2	10	2	
5	US EPA 200.8	0.5	95	120	2	2				10 (H <sub>2</sub> O)
6	In House, US EPA 6020B	2	90-95	60	4	12			4	
8	In House Method	1	112.5	120	2.5	7.5				
9	In-house	2	95	90	5	5			5	
10*	USEPA Method 3050A and B	1	100	120	3	3				10 mL H <sub>2</sub> O
11	US EPA Methods 3050B, 3051A and 6020B	2	100	60	4	12				
12	In house method - referencing APHA 3125	0.4	120	60	2.5	7.5				
13	USEPA Method 6010c, USEPA Methods 7471B, 7470A, 7471B	2.5	90 - 98	90	3	3				
14	USEPA 200.8	1	95	30	2.5	2.5				

Lab. Code	Method Reference	Sample Mass (g)	Temp. (°C)	Time (min)	Vol. HNO <sub>3</sub> (mL)	Vol. HCl (mL)	Vol. HNO <sub>3</sub> (1:1) (mL)	Vol. HCl (1:1) (mL)	Vol. H <sub>2</sub> O <sub>2</sub> (mL)	Other (mL)
15	EPA 200.2, (1:1 Nitric:Hydrochloric Acid	0.5	96	30	1	1				
16	Acid Digestion of sediment, sludges and soil-USEPA 3050	5	95	90	3	3				
17	200.2 Revision 2.8	1	95 ± 5	60	2	10			2	
18*	USEPA 3050	3	85	120	10	5	10		6	
19	US EPA 3050B	1	98	150	5	5				
21	US-EPA Method 200.2	1	95	50	2	2				10 (H <sub>2</sub> O)
22		2	90	90	2	6				
24	USEPA Method 200.2 Revision 2.8	1	95	60			2			10 (HCl 20%)
25	EPA3050B, 6020B	2	95-105	60	4	12				
26	US EPA 3050B	0.5	95	120	7.5	5			1.5	
27	LTM-MET-3040	2	95-105	60						20 (Aqua regia)
28	USEPA 200.2	1	95	60			2	10	2	
30		1			2.5	7.5				

\*See Additional Information for Methodology in Table 2

Table 2 Additional Information for Acid Extractable Elements

Lab Code	Additional Information
10	Made to 20mL after one hour, final hour with additional vortexing and final vol 40mL.
18	Instrument for Hg: Hg Analyser

Table 3 Methodology for Total Carbon

Lab. Code	Method Reference	Test Method	Measurement Technique
1		High Temperature Oxidation	
3	Combustion	High Temperature Oxidation	Dumas combustion - NDIR
4	AS 1289.4.1.1	High Temperature Oxidation	
7	6B2b	High Temperature Oxidation	IR-Leco
10		High Temperature Oxidation	
12	In house	High Temperature Oxidation	LECO
16	Combustion	High Temperature Oxidation	Dumas combustion - NDIR
17	Rayment & Lyons 6B3	High Temperature Oxidation	Carbon/Sulphur Analyser
19		High Temperature Oxidation	
27	LTM-INO-4060	High Temperature Oxidation	Combustion and IR

**Table 4 Methodology for Total Organic Carbon**

Lab. Code	Method Reference	Test Method	Measurement Technique	Additional Information
3	Combustion	High Temperature Oxidation Chemical Oxidation no $\text{Ag}_2\text{SO}_4$	Dumas combustion - NDIR	
4	AS1289.4.1.1	Chemical Oxidation with $\text{Ag}_2\text{SO}_4$	Walkely & Black Method	
7	6B3	High Temperature Oxidation	Leco	Sample was Fizz test with 4 M HCl and no Fizzing observed. Therefore no acid treatment was carried for TOC
8		Chemical Oxidation with $\text{Ag}_2\text{SO}_4$	Manual UV- VIS at 600nm	
10		High Temperature Oxidation		
12	In house	High Temperature Oxidation	LECO	Sample digested with sulphurous acid prior to analysis on LECO
13	Australian Laboratory Handbook of Soil and Water Chemical Methods	Chemical Oxidation with $\text{Ag}_2\text{SO}_4$	Titration	AS1289.4.1.1 2019 Methods of testing soils for engineering purposes
14	NEPM 105 / RAYMENT LYONS 6A1	Chemical Oxidation with $\text{Ag}_2\text{SO}_4$	TITRATION	
16	Combustion	High Temperature Oxidation	Dumas combustion - NDIR	
17	Rayment & Lyons 6B3	High Temperature Oxidation	Carbon/Sulphur Analyser	
18	TOC by Heanes	Chemical Oxidation no $\text{Ag}_2\text{SO}_4$	Discrete Analyser	
19		High Temperature Oxidation		
22	Heanes method	Chemical Oxidation no $\text{Ag}_2\text{SO}_4$	UV-Vis	
24	Australian Standard AS 1289.4.1.1	Chemical Oxidation no $\text{Ag}_2\text{SO}_4$	Walkely & Black method	Oxidation by dichromate, followed by calculation from organic matter for total organic carbon
27	LTM-INO-4060	High Temperature Oxidation		
28		Chemical Oxidation with $\text{Ag}_2\text{SO}_4$	Titration	

**Table 5 Methodology for Colwell P and Colwell K**

Lab. Code	Method Reference	Sample Mass (g)	Extraction Solution 0.5 M NaHCO <sub>3</sub> Volume (mL)	Shake time (hours)	Final Dilution Factor (Colwell K)	Final Dilution Factor (Colwell P)	Measurement Technique (Colwell K)	Measurement Technique (Colwell P)
1		1	100	16	100	100	ICP-OES-RV 766.491nm	UV-Vis 882 nm
7								FIA
10		0.5	50	16		1000		DA 880 nm
12	Colwell P 9B2, Colwell K 18A1	0.4	40	16	3280	328	ICPMS 31 m/z	FIA 880 nm
17	Rayment & Lyons 9B1 & 18A1	1	100	16	100	100	ICP-OES 404.721 nm 766.491 nm	UV-Vis 882 nm
18	Rayment and Lyons 9B1	1	100	16	NT		NT	DA 880 nm
19	Method 9B1: George E. Rayment and David J. Lyons	1.2	120	16	NA	260	NA	DA

**Table 6 Methodology for P Buffer Index – PBI<sub>+ColP</sub>**

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)
7	9I2	7	70	17	FIA	100	880
12	912b	2	20	16	ICP-OES	10	213.617
19	9I2	2	20	17	ICP-OES	100	213.617

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

**Table 7 Methodology for Total P**

Lab. Code	Method
1	Total P by Kjeldahl digestion and SFA
4	Total P by Kjeldahl digestion and DA
5	Alkaline potassium persulfate digestion and FIA
8	Total P by Kjeldahl digestion and Ascorbic Acid Colorimetric detection by Discrete Analyser
14	APHA4500-P E/ USEPA 365.4
17	Total P by APHA 4500 Norg-D with Jirka modification followed by DA finish
18	Total P by Kjeldahl digestion and DA
19	US EPA 3050B
24	Total P by Kjeldahl digestion and DA
27	Total P by UV digestion and SFA
28	Persulfate digestion followed by DA

**Table 8 Methodology for Calcium Chloride Extractable B**

Lab. Code	Method Reference*	Sample Mass (g)	Extraction Solution (0.01 M CaCl <sub>2</sub> ) Volume (mL)	Reflux Time (min)	Instrument	Final Dilution Factor	Wavelength (nm) / Absorbance (nm)
12	12C	10	20	10	ICP-OES	2	208.889
16	12C	10	20	10	ICP-OES	2	208.956
17	12C	10	20	10	ICP-OES	2	249.773
19	12C	2	20	10	ICP-OES	100	249.677
28	12C	10	20	10	ICP-OES		

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

**Table 9 Methodology for Total Nitrogen**

Lab. Code	Method Reference	Test Method	Measurement Method	Instrument	Additional Information
1	Rayment & Lyons 7A2a	Digestion TN=TKN	Colorimetric – salicylate method	SFA	
3		Digestion TN=TKN+NOx	Colorimetric – salicylate method	DA	
4	APHA 4500 –Norg. A & D	Digestion	Colorimetric – salicylate method	DA	
7	7A5	Dumas	Dumas –High temperature combustion	LECO	
8	Soil Chem Methods Australasia (Rayment and Lyons) and USEPA Methods 351.2 and 365.3	Digestion TN=TKN+NOx	Colorimetric – salicylate method	DA	
10		TN=TKN+NOx	Titrimetric method	Manual Analysis	
12	In house – Dumas combustion	Combustion	Dumas –High temperature combustion	LECO	
13	TKN: APHA 4500-Inorg-D / Nox: APHA 4500-N C	Digestion TN=TKN+NOx	Colorimetric- salicylate method	DA	
14	APHA 4500-NORG D / US EPA 351.2	Digestion TN=TKN+NOx	Colorimetric – salicylate method	DA	NO2-N by DA, NO3-N by IC, NOx by calc.
16	Lyons, G.E.R.a.D.J,2011 Total soil N-Dumas high temperature combustion	Combustion	Dumas –High temperature combustion	TN-analyser and SSM 5000A	
17	APHA 22nd edition 4500 Norg A & D with Jirka Modification-Jirka et al. (1976) and the appropriate Discrete Analyser method.	Digestion TN=TKN+NOx	Colorimetric – phenate method	DA	
18	ASTM D2216-98	Digestion TN=TKN+NOx	Colorimetric – salicylate method	DA	
19	Method 7A5: Rayment, G.E. and David J. Lyons	Combustion	Dumas –High temperature combustion	LECO	
22	APHA4500-N	Digestion TN=TKN+NOx	Colorimetric – phenate method	DA	
24	Total Kjeldahl Nitrogen: APHA 4500 Norg A & D	Digestion TN=TKN+NOx	Colorimetric – salicylate method	DA	Nox needed for TN calculation is performed

Lab. Code	Method Reference	Test Method	Measurement Method	Instrument	Additional Information
	with Jirka modification 1976				based on 1:5 soil:water extraction for 1h
27	LTM-INO-4310 and LTM-INO-4120	Combustion Digestion TN=TKN+NOx	Colorimetric – salicylate method	FIA	
28	APHA, 4500-P J. & 4500-N C.	Digestion		DA	

Table 10 Methodology for Exchangeable Bases

Lab. Code	Method Reference*	Sample Mass (g)	Shake time (hrs)	Extraction Solution	Extraction Solution Vol. (mL)
3	Rayment and Lyons 15B1	2.5	2	1M NH <sub>4</sub> CL	50
4	15A1	2.5	1	1M NH <sub>4</sub> Cl	
5	15A1	2	1	1M NH <sub>4</sub> Cl	40
7	15A1	2	1	1M NH <sub>4</sub> Cl	40
10	15A1	2	2	1M NH <sub>4</sub> Cl	40
12	15A1	1	1	1M NH <sub>4</sub> Cl	20
14	15A1	2	1	NH <sub>4</sub> Acetate	20
16	15A1	2.5	1	1M NH <sub>4</sub> Cl	50
17	15A1	2.5	1	1M NH <sub>4</sub> Cl	50
18	Rayment and Lyons 15D3 & 15N1	5	1	1M NH <sub>4</sub> Cl	100
19	15A1	2	1	1M NH <sub>4</sub> Cl	10
22	15A1	5	1	1M NH <sub>4</sub> Cl	100
24	15A1	2.5	1	1M NH <sub>4</sub> Cl	50
27	15A1	5	1	1M NH <sub>4</sub> Cl	100
28	ED007	2.5	1	1M NH <sub>4</sub> Cl	50

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

### 3.2 Basis of Participants' Measurement Uncertainty Estimates

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

Table 11 Basis of Uncertainty Estimate

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation <sup>a</sup>		Guide Document for Estimating MU
		Precision	Method Bias	
1	Top Down - precision and estimates of the method and laboratory bias	Control Samples Duplicate Analysis Instrument Calibration	CRM	NMI Uncertainty Course
2	Top Down - precision and estimates of the method and laboratory bias		CRM Recoveries of SS	Nordtest Report TR537
3	Top Down - precision and estimates of the method and laboratory bias	Control Samples	Recoveries of SS	NATA General Accreditation Guidance Estimating and Reporting Measurement Uncertainty of Chemical Test Results
4	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM	CRM Recoveries of SS	Eurachem/CITAC Guide
5	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM Duplicate Analysis	CRM Recoveries of SS	NATA General Accreditation, Guidance, Estimating and Reporting MU
6	Top Down - precision and estimates of the method and laboratory bias	Control Samples - SS	Recoveries of SS	ISO/GUM
7	Top Down - reproducibility (standard deviation) from PT studies used directly	Control Samples - CRM Duplicate Analysis		
8	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration Recoveries of SS	NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33)
9	Top Down - precision and estimates of the method and laboratory bias	Control Samples - RM	CRM	NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33)
10	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM Duplicate Analysis	CRM Variation in Sample Moisture Content Recoveries of SS	Nordtest Report TR537
11	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration Laboratory Bias from PT Studies Recoveries of SS	ASTM E2554-13

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation <sup>a</sup>		Guide Document for Estimating MU
		Precision	Method Bias	
12	Top Down - precision and estimates of the method and laboratory bias	Control Samples - RM Duplicate Analysis	Instrument Calibration Standard Purity	Nordtest Report TR537
13	Top Down - precision and estimates of the method and laboratory bias	Control Samples - SS	Recoveries of SS	NATA Technical Note 33
14	Top Down - precision and estimates of the method and laboratory bias	Control Samples - RM Instrument Calibration		ISO/GUM
15	Top Down - precision and estimates of the method and laboratory bias	Control Samples - SS Duplicate Analysis Instrument Calibration	CRM Instrument Calibration Recoveries of SS Standard Purity	IANZ Technical Guide
16	Top Down - precision and estimates of the method and laboratory bias	Control Samples	Recoveries of SS	ISO/GUM
17	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM Duplicate Analysis	CRM Instrument Calibration	Eurachem/CITAC Guide
18	Top Down - precision and estimates of the method and laboratory bias	Duplicate Analysis Instrument Calibration	CRM Instrument Calibration	NATA Technical Note 33
19	Top Down - reproducibility (standard deviation) from PT studies used directly	Standard deviation from PT studies only		Eurolab Technical Report No1/2007
		Control Samples - CRM	CRM Laboratory Bias from PT Studies	
21	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples - CRM Duplicate Analysis	CRM Instrument Calibration Laboratory Bias from PT Studies	Eurachem/CITAC Guide
22	Bottom Up (ISO/GUM, fish bone/ cause and effect diagram)	Control Samples - CRM Duplicate Analysis Instrument Calibration		
24	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration Laboratory Bias from PT Studies Recoveries of SS	Eurachem/CITAC Guide
25	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	Control Samples Duplicate Analysis	CRM	ASTM E2554-13

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation <sup>a</sup>		Guide Document for Estimating MU
		Precision	Method Bias	
26	Top Down - precision and estimates of the method and laboratory bias	Control Samples - CRM Duplicate Analysis	CRM Instrument Calibration Recoveries of SS	Top Down
27	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples - SS	Instrument Calibration	
28*	Top Down - precision and estimates of the method and laboratory bias	Control Samples - RM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration Laboratory Bias from PT Studies	Eurachem/CITAC Guide
29	Top Down - reproducibility (standard deviation) from PT studies used directly	Standard deviation from PT studies only		ISO/GUM
			Instrument Calibration	

\*Additional information in Table 12. <sup>a</sup>RM = Reference Material, CRM = Certified Reference Material, SS = Spiked samples.

Table 12 Additional Information for Basis of Uncertainty Estimate

Lab Code	Additional Information
28	Macro MU Calculation Pack based on QC Data

### 3.3 Participant Comments on this PT Study or Suggestions for Future Studies

The study co-ordinator welcomes comments or suggestions from participants about this study or possible future studies. Such feedback may be useful in improving future studies. Participants' comments are reproduced in Table 13.

Table 13 Participants' Comments

Participants' Comments	Study Co-ordinator's Response
Thanks for running. Great program.	Thank you for your feedback.
A more realistic approximate range would be helpful. A number of our methods have standard level, low level, ultra-trace level and super ultra-trace level options. It would save a lot of time and rework and prevent us from running out of sample if we could determine which of these options the best choice from the approximate range information was before we started.	Thank you for your feedback.  We will look into how we can provide a "friendlier" range. The allowed variation for a result is usually +/- 20% of the assigned value, so the range we give you has to be wider. We usually use a +/- 40% range but as you can imagine the expected value is not always in the middle. Another option is to give a range which is less than some specified value. Please let me know if this would suit you better. The other participants are also invited to comment.

## 4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

### 4.1 Results Summary

Participant results are listed Tables 14 to 70 with resultant summary statistics: robust average, median, maximum, minimum, robust standard deviation ( $SD_{rob}$ ) and robust coefficient of variation ( $CV_{rob}$ ). Bar charts of results and performance scores are presented in Figures 2 to 58. An example chart with interpretation guide is shown in Figure 1.

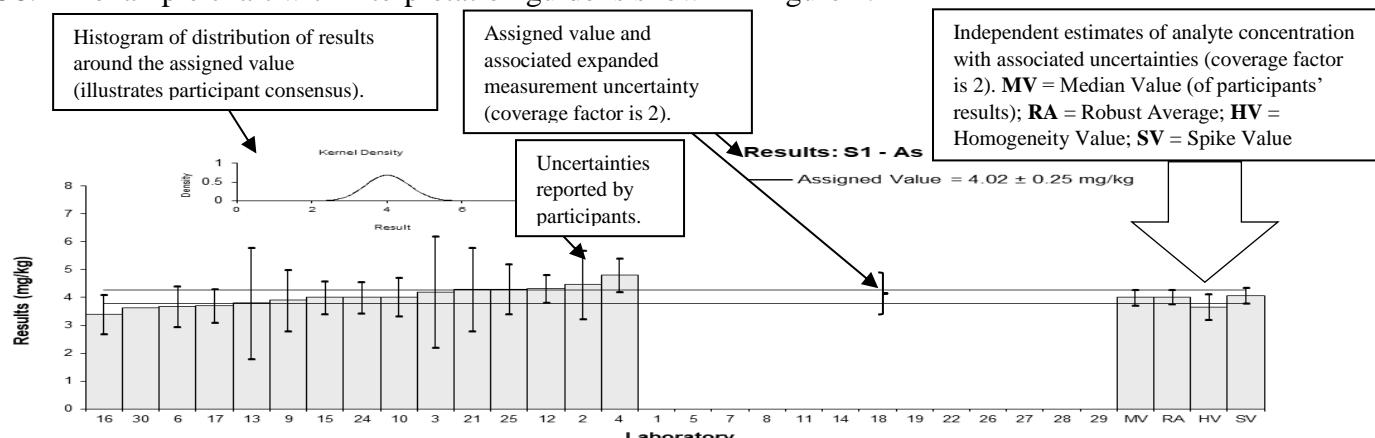


Figure 1 Guide to Presentation of Results

### 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 were obvious blunders, such as those with incorrect units, decimal errors, or results from a different proficiency test item (gross errors) and were removed for calculation of summary statistics.<sup>3, 4, 6</sup>

### 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.’<sup>1</sup> In this study the property is the mass fraction of analyte. Assigned values were the robust average of participants’ results, outliers removed; the expanded uncertainties were estimated from the associated robust standard deviations.<sup>4, 6</sup>

### 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, ISO13528:2015(E)’.<sup>6</sup>

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

### 4.5 Target Standard Deviation for Proficiency Assessment

The target standard deviation for proficiency assessment ( $\sigma$ ) is the product of the assigned value ( $X$ ) and the performance coefficient of variation (PCV). This value is used for calculation of participant z-score and provides scaling for laboratory deviation from the assigned value.

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

It is important to note that the PCV is a fixed value and is not the standard deviation of participants' results. The fixed value set for PCV is based on the existing regulation, the acceptance criteria indicated by the methods, the matrix, the concentration level of analyte and on experience from previous studies. It is backed up by mathematical models such as the Thompson Horwitz equation.<sup>7</sup>

#### **4.6 z-Score**

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

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

Where:

- $z$  is z-score;
- $\chi$  is participant's result;
- $X$  is the study assigned value;
- $\sigma$  is the target standard deviation.

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

- $|z| \leq 2.0$  is satisfactory;
- $2.0 < |z| < 3.0$  is questionable;
- $|z| \geq 3.0$  is unsatisfactory.

#### **4.7 E<sub>n</sub>-Score**

An example of E<sub>n</sub>-score calculation using data from the present study is given in Appendix 2. The E<sub>n</sub>-score is complementary to the z-score in assessment of laboratory performance. E<sub>n</sub>-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<sub>n</sub>-score;
- $\chi$  is a participant's result;
- $X$  is the study assigned value;
- $U_\chi$  is the expanded uncertainty of the participant's result;
- $U_X$  is the expanded uncertainty of the assigned value.

An E<sub>n</sub>-score with absolute value ( $|E_n|$ ):

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

#### **4.8 Traceability and Measurement Uncertainty**

Laboratories accredited to ISO/IEC Standard 17025:2018<sup>8</sup> must establish and demonstrate the traceability and measurement uncertainty associated with their test results. Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.<sup>9</sup>

## 5 TABLES AND FIGURES

Table 14

### Sample Details

<b>Sample</b>	S1
<b>Analyte</b>	As
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

### Participant Results

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	4.47	1.23	0.75	0.36
3	4.2	2	0.30	0.09
4	4.8	0.6	1.29	1.20
5	<5	NR		
6	3.67	0.73	-0.58	-0.45
7	NT	NT		
8	NT	NT		
9	3.9	1.1	-0.20	-0.11
10	4.02	0.69	0.00	0.00
11	NT	NT		
12	4.32	0.5	0.50	0.54
13	3.8	2	-0.36	-0.11
14	NT	NT		
15	4.0	0.6	-0.03	-0.03
16	3.4	0.7	-1.03	-0.83
17	3.7	0.6	-0.53	-0.49
18	NT	NT		
19	<25	NR		
21	4.3	1.5	0.46	0.18
22	NT	NT		
24	4.0	0.57	-0.03	-0.03
25	4.3	0.9	0.46	0.30
26	<3	NR		
27	NT	NT		
28	<5	0.84		
29	NT	1		
30	3.62	NR	-0.66	-1.60

### Statistics

<b>Assigned Value</b>	4.02	0.25
<b>Spike Value</b>	4.07	0.28
<b>Homogeneity Value</b>	3.66	0.46
<b>Robust Average</b>	4.02	0.25
<b>Median</b>	4.00	0.29
<b>Mean</b>	4.03	0.19
<b>N</b>	15	
<b>Max</b>	4.8	
<b>Min</b>	3.4	
<b>Robust SD</b>	0.38	
<b>Robust CV (%)</b>	9.5	

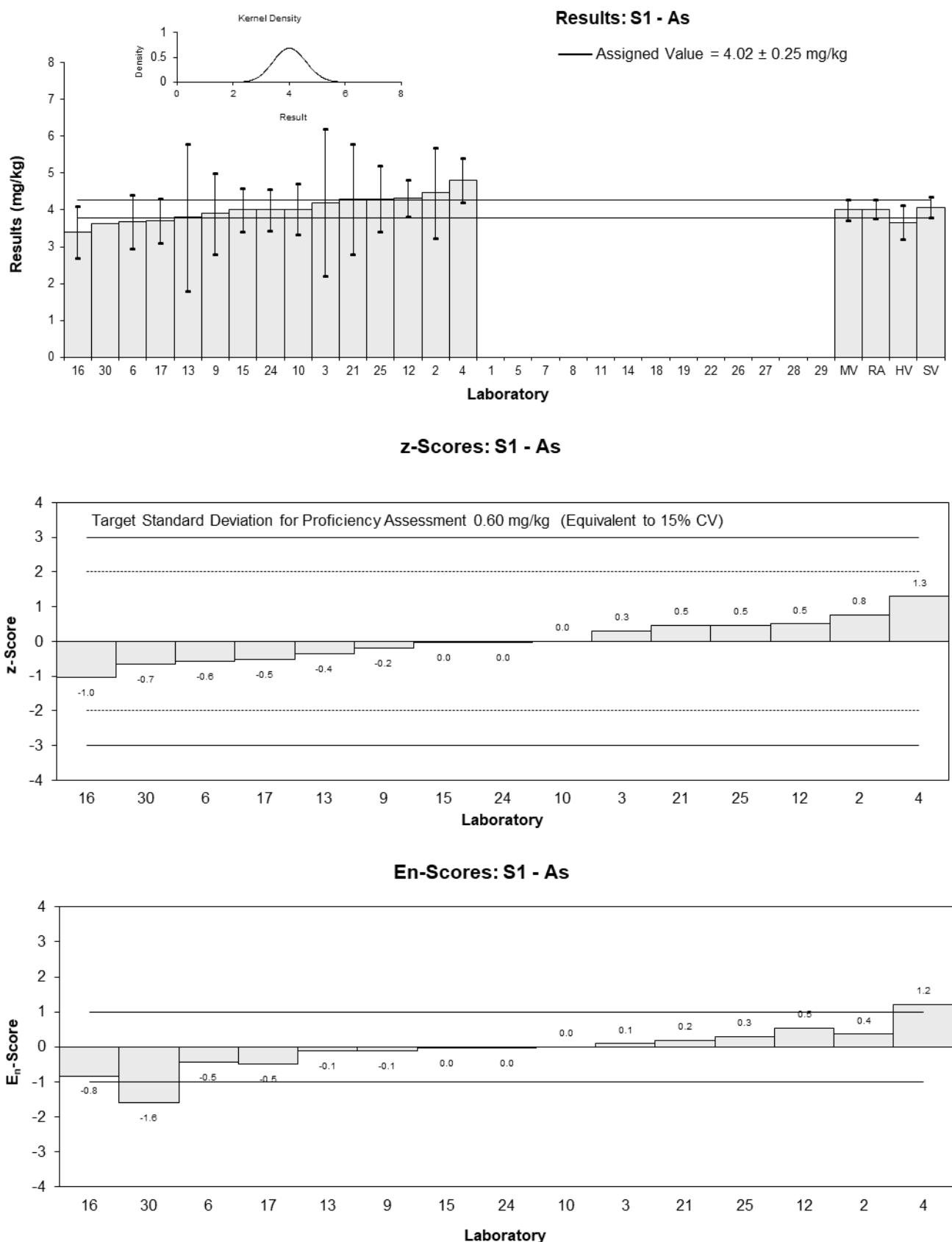


Figure 2

Table 15

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	B
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	9.90	1.06	0.10	0.10
3	7.3	2.5	-1.24	-0.78
4	<50	NR		
5	10	NR	0.15	0.17
6	12	3	1.19	0.66
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	10.7	2.1	0.52	0.36
11	NT	NT		
12	10.6	1.5	0.46	0.38
13	7.3	2	-1.24	-0.89
14	NT	NT		
15	<23	NR		
16	6	1	-1.91	-1.80
17	<50	NR		
18	NT	NT		
19	7.50	3.7	-1.13	-0.53
21	< 20	3.6		
22	NT	NT		
24	<50	6.2566		
25	11	2.3	0.67	0.45
26	11	3	0.67	0.37
27	NT	NT		
28	<50	NR		
29*	4.04	1	-2.92	-2.75
30	13.9	NR	2.16	2.33

\* Outlier

**Statistics**

<b>Assigned Value</b>	9.7	1.8
<b>Spike Value</b>	10.1	0.2
<b>Homogeneity Value</b>	8.12	0.97
<b>Robust Average</b>	9.4	2.0
<b>Median</b>	10.0	2.1
<b>Mean</b>	9.3	1.5
<b>N</b>	13	
<b>Max</b>	13.9	
<b>Min</b>	4.04	
<b>Robust SD</b>	2.9	
<b>Robust CV (%)</b>	30	

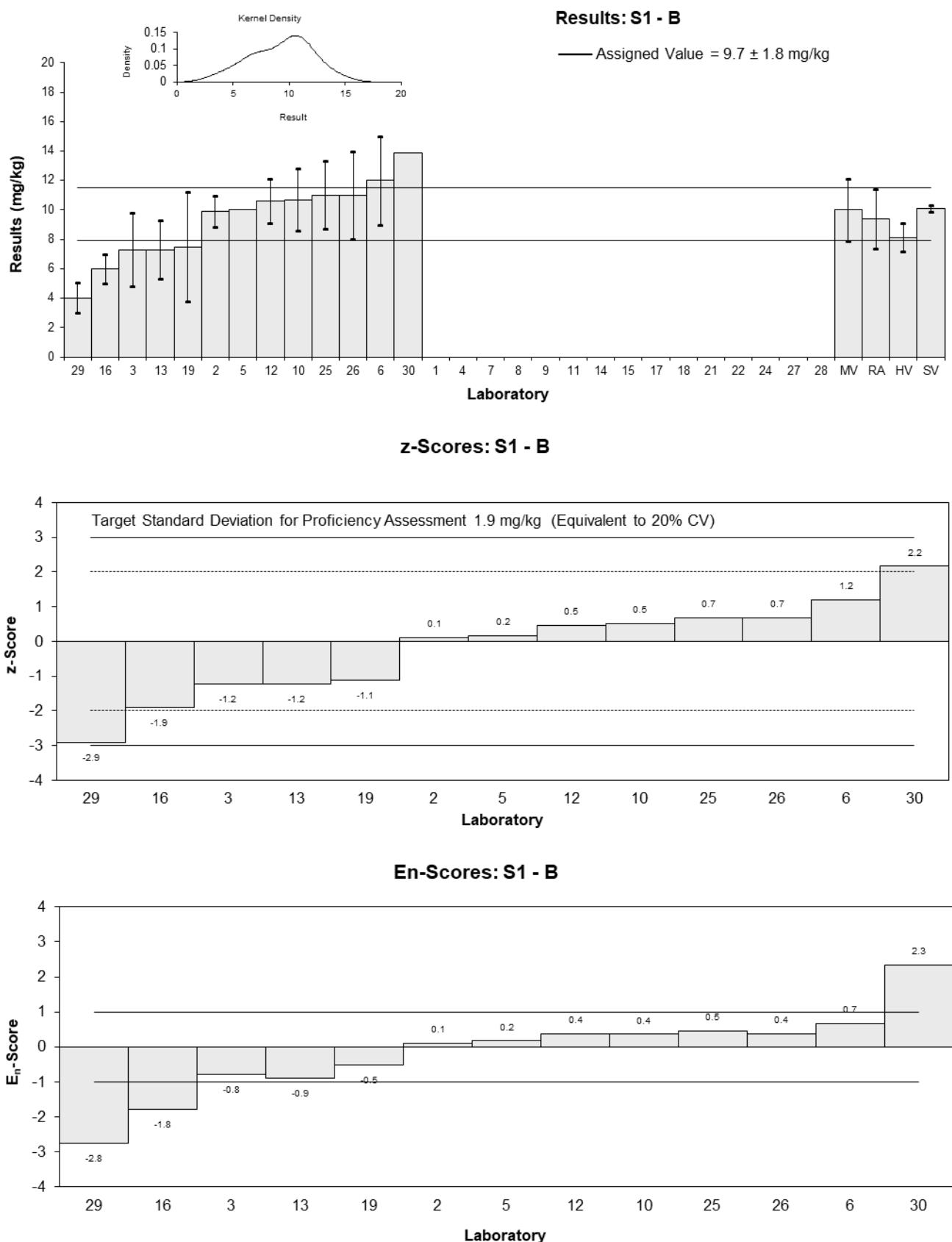


Figure 3

Table 16

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Be
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	0.96	0.16	0.47	0.34
3	1.2	1	2.25	0.30
4	0.74	0.17	-1.17	-0.82
5	0.84	0.084	-0.42	-0.47
6	<2	NT		
7	NT	NT		
8	NT	NT		
9	<5.0	NR		
10	0.924	0.185	0.20	0.13
11	NT	NT		
12	1.03	0.2	0.99	0.61
13	0.87	1	-0.20	-0.03
14	NT	NT		
15	0.89	0.29	-0.05	-0.02
16	0.76	0.2	-1.02	-0.63
17	0.8	0.2	-0.72	-0.44
18	NT	NT		
19	NT	NT		
21	1.06	0.23	1.21	0.66
22	NT	NT		
24	0.9	0.6277	0.02	0.00
25	<2	0.42		
26	<1	NR		
27	NT	NT		
28	<1	0.14		
29	0.8	1	-0.72	-0.10
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	0.897	0.089
<b>Spike Value</b>	0.898	0.024
<b>Homogeneity Value</b>	0.84	0.10
<b>Robust Average</b>	0.897	0.089
<b>Median</b>	0.890	0.093
<b>Mean</b>	0.906	0.073
<b>N</b>	13	
<b>Max</b>	1.2	
<b>Min</b>	0.74	
<b>Robust SD</b>	0.13	
<b>Robust CV (%)</b>	14	

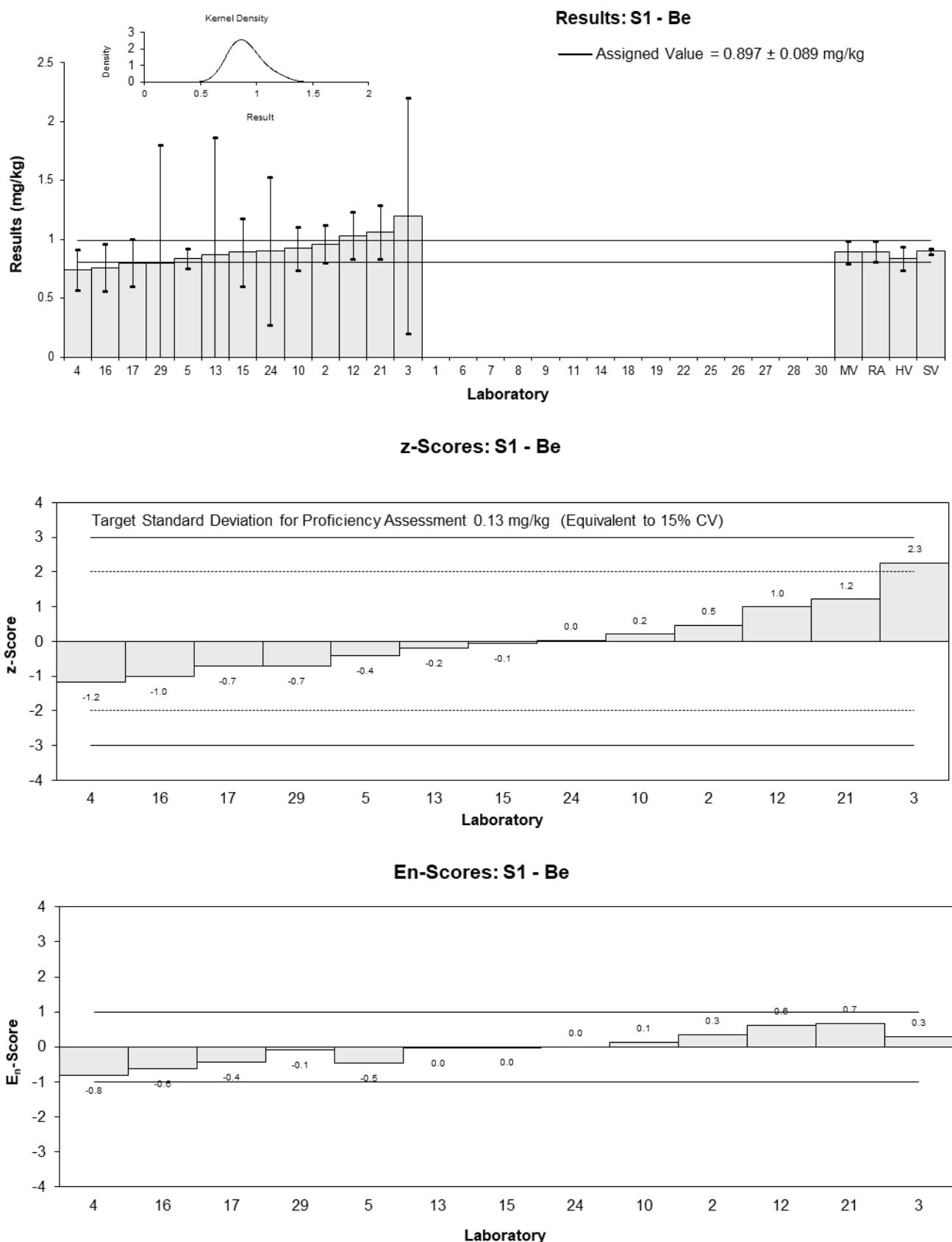


Figure 4

Table 17

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Bi
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	1.17	0.04	-0.52	-0.79
3	1.1	1	-0.89	-0.17
4	1.47	0.44	1.05	0.44
5	NT	NT		
6	<10	NT		
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	1.27	0.25	0.00	0.00
11	NT	NT		
12	1.11	0.2	-0.84	-0.69
13	1.2	1	-0.37	-0.07
14	NT	NT		
15	1.4	0.43	0.68	0.29
16	1.1	0.2	-0.89	-0.73
17	1.3	0.4	0.16	0.07
18	NT	NT		
19	NT	NT		
21	1.40	0.33	0.68	0.37
22	NT	NT		
24	1.6	1.6	1.73	0.21
25	<10	2		
26	NT	NT		
27	NT	NT		
28	1.24	NR	-0.16	-0.25
29*	0.25	1	-5.35	-1.01
30	NT	NT		

\* Outlier

**Statistics**

<b>Assigned Value</b>	1.27	0.12
<b>Spike Value</b>	1.15	0.03
<b>Homogeneity Value</b>	1.05	0.13
<b>Robust Average</b>	1.25	0.13
<b>Median</b>	1.24	0.14
<b>Mean</b>	1.20	0.18
<b>N</b>	13	
<b>Max</b>	1.6	
<b>Min</b>	0.25	
<b>Robust SD</b>	0.19	
<b>Robust CV (%)</b>	15	

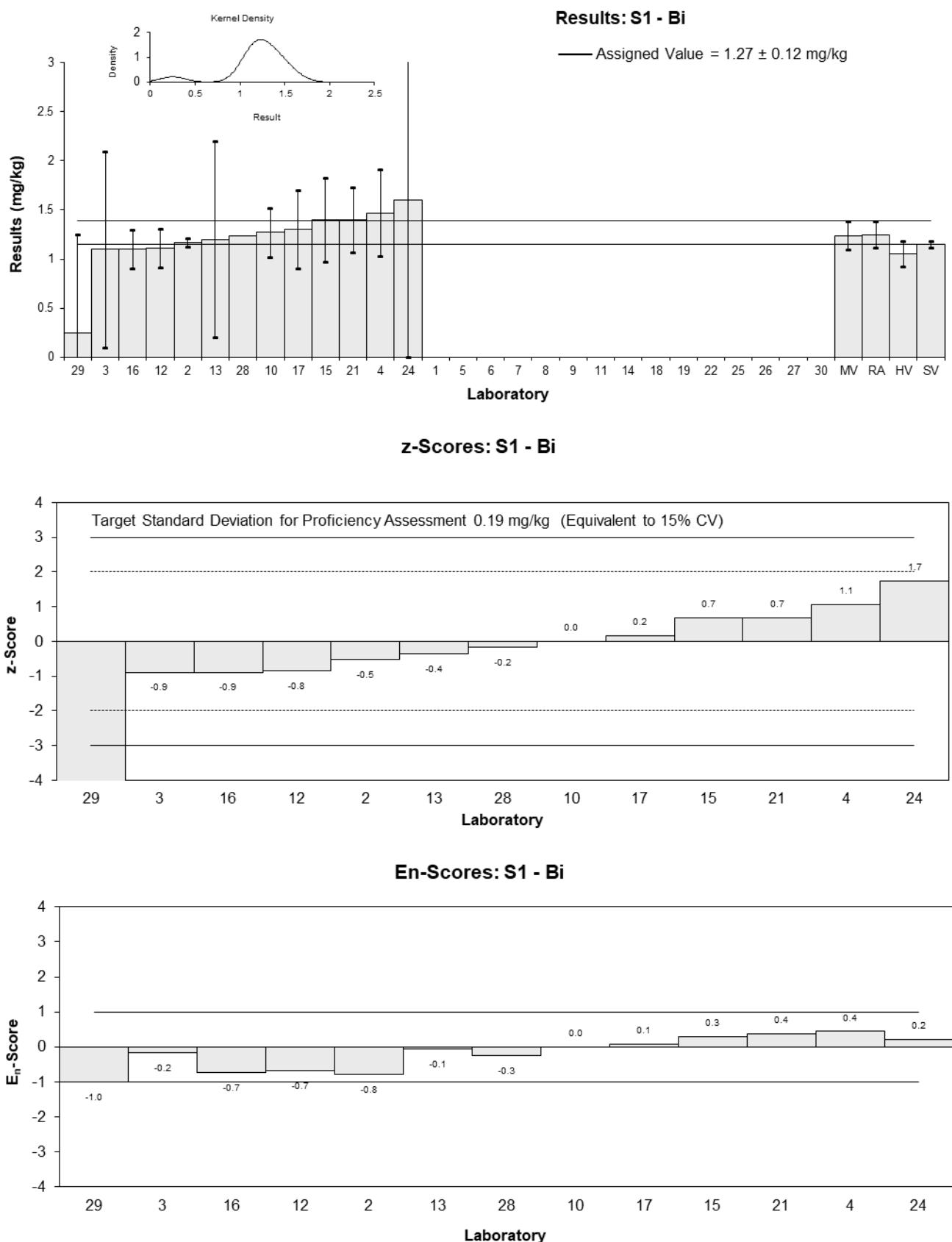


Figure 5

Table 18

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Cd
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	1.68	0.36	0.37	0.16
3	1.5	1	-0.74	-0.12
4	1.78	0.52	0.99	0.30
5	1.7	0.102	0.49	0.59
6	1.51	0.3	-0.68	-0.35
7	NT	NT		
8	NT	NT		
9	1.6	0.17	-0.12	-0.10
10	1.61	0.22	-0.06	-0.04
11	NT	NT		
12	1.65	0.2	0.19	0.14
13	1.5	1	-0.74	-0.12
14	NT	NT		
15	1.7	0.32	0.49	0.24
16	1.4	0.3	-1.36	-0.70
17	1.5	0.2	-0.74	-0.55
18	NT	NT		
19	<1.0	NR		
21	1.79	0.26	1.05	0.62
22	NT	NT		
24	1.6	0.06284	-0.12	-0.18
25	1.6	0.34	-0.12	-0.06
26	2	1	2.35	0.38
27	NT	NT		
28	1.6	0.25	-0.12	-0.08
29	1.42	1	-1.23	-0.20
30	1.91	NR	1.79	3.22

**Statistics**

<b>Assigned Value</b>	1.62	0.09
<b>Spike Value</b>	1.62	0.03
<b>Homogeneity Value</b>	1.57	0.19
<b>Robust Average</b>	1.62	0.09
<b>Median</b>	1.60	0.09
<b>Mean</b>	1.63	0.07
<b>N</b>	19	
<b>Max</b>	2	
<b>Min</b>	1.4	
<b>Robust SD</b>	0.15	
<b>Robust CV (%)</b>	9.4	

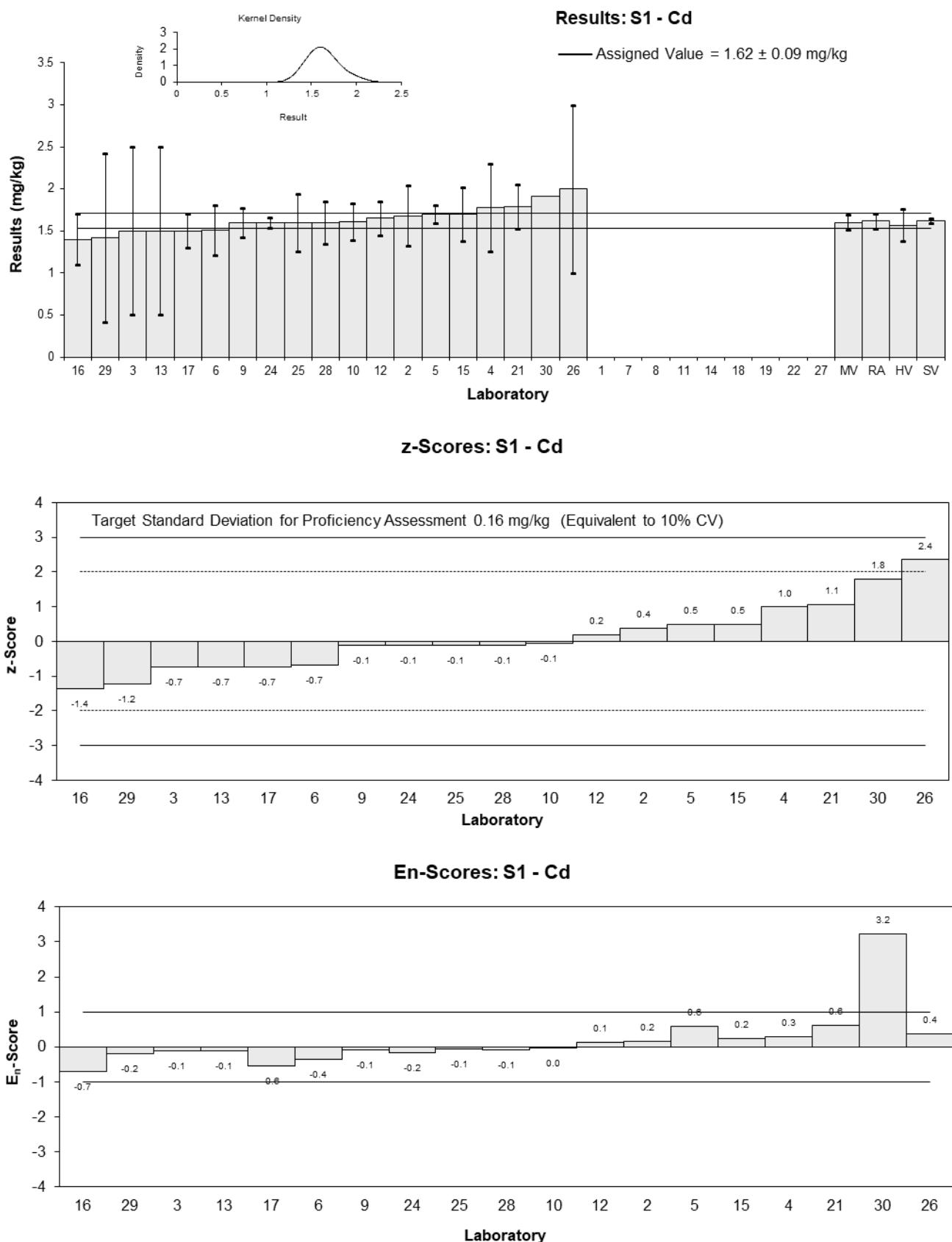


Figure 6

Table 19

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Cr
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	52.5	5.9	0.54	0.43
3	48	15	-0.36	-0.12
4	53.97	11.48	0.84	0.36
5	53	5.3	0.64	0.56
6	49.8	10	0.00	0.00
7	NT	NT		
8	NT	NT		
9	50	13	0.04	0.02
10	49.7	7.9	-0.02	-0.01
11	NT	NT		
12	50.6	7.0	0.16	0.11
13	46	10	-0.76	-0.37
14	NT	NT		
15	51	7.6	0.24	0.15
16	41	8	-1.77	-1.06
17	45.4	10.6	-0.88	-0.41
18	NT	NT		
19	51.0	15.3	0.24	0.08
21	54.6	8.6	0.96	0.54
22	NT	NT		
24	46.5	7.0962	-0.66	-0.44
25	52	11	0.44	0.20
26	57	6	1.45	1.13
27	NT	NT		
28	49.8	14.34	0.00	0.00
29	47.7	1	-0.42	-0.87
30	43.8	NR	-1.20	-2.73

**Statistics**

<b>Assigned Value</b>	49.8	2.2
<b>Spike Value</b>	50.4	1.4
<b>Homogeneity Value</b>	44.8	5.6
<b>Robust Average</b>	49.8	2.2
<b>Median</b>	49.9	2.0
<b>Mean</b>	49.7	1.7
<b>N</b>	20	
<b>Max</b>	57	
<b>Min</b>	41	
<b>Robust SD</b>	3.9	
<b>Robust CV (%)</b>	7.7	

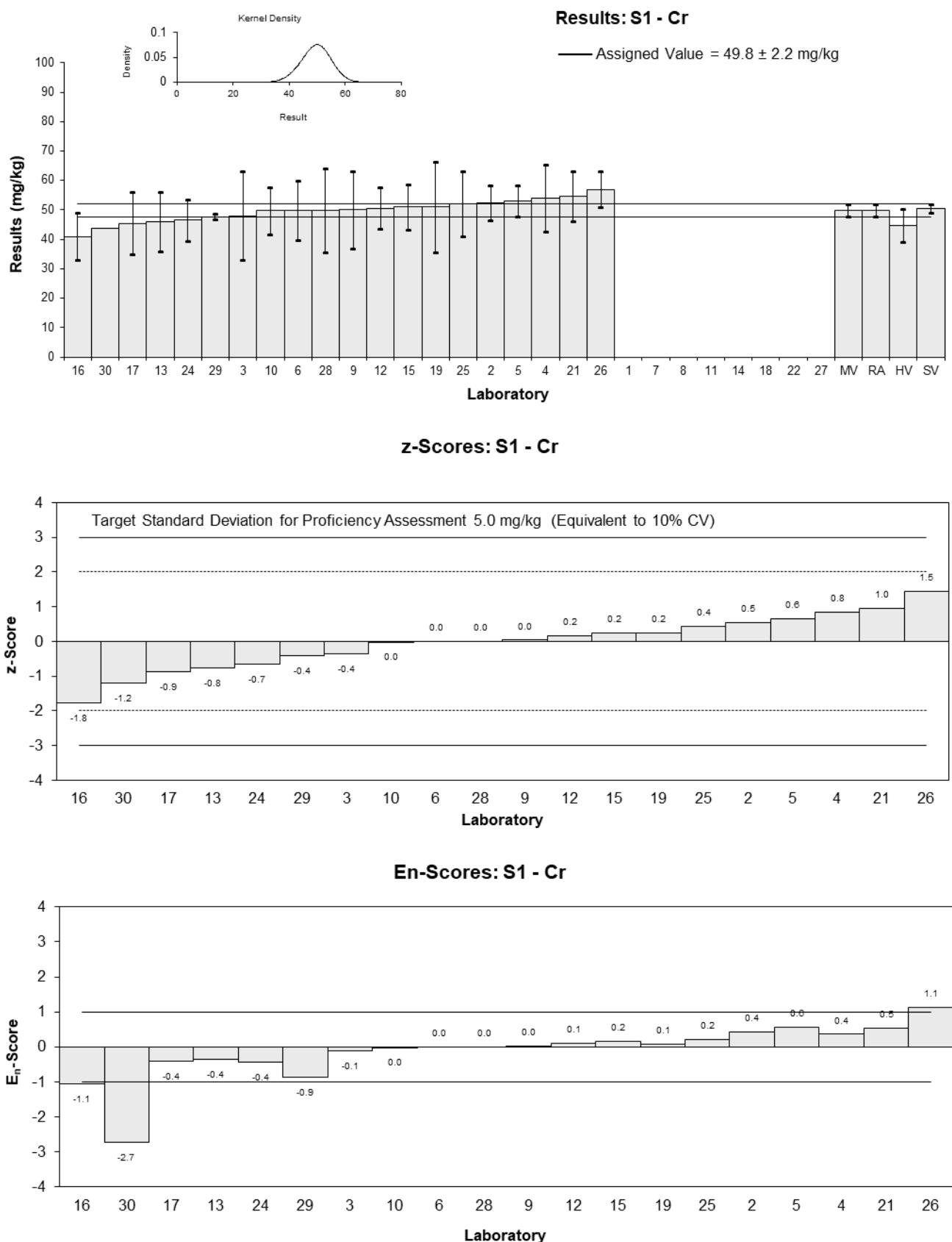


Figure 7

Table 20

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Cu
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	69.3	8.7	0.25	0.19
3	64	21	-0.53	-0.17
4	81.72	11.95	2.09	1.17
5	68	9.52	0.06	0.04
6	64.5	12.9	-0.46	-0.24
7	NT	NT		
8	NT	NT		
9	69	16	0.21	0.09
10	69.7	11.2	0.31	0.18
11	NT	NT		
12	69.7	7.0	0.31	0.29
13	61	10	-0.98	-0.65
14	NT	NT		
15	68	10	0.06	0.04
16	56	10	-1.72	-1.14
17	66.3	10.6	-0.19	-0.12
18	NT	NT		
19	70.5	14.1	0.43	0.20
21	70.4	9.7	0.41	0.28
22	NT	NT		
24	63.2	5.2971	-0.65	-0.78
25	68	14	0.06	0.03
26	67	8	-0.09	-0.07
27	NT	NT		
28	65.8	10.6	-0.27	-0.17
29	74.5	1	1.02	3.09
30	67.7	NR	0.01	0.05

**Statistics**

<b>Assigned Value</b>	67.6	2.0
<b>Spike Value</b>	70.3	4.3
<b>Homogeneity Value</b>	61.2	7.3
<b>Robust Average</b>	67.6	2.0
<b>Median</b>	68.0	1.6
<b>Mean</b>	67.7	2.3
<b>N</b>	20	
<b>Max</b>	81.72	
<b>Min</b>	56	
<b>Robust SD</b>	3.7	
<b>Robust CV (%)</b>	5.4	

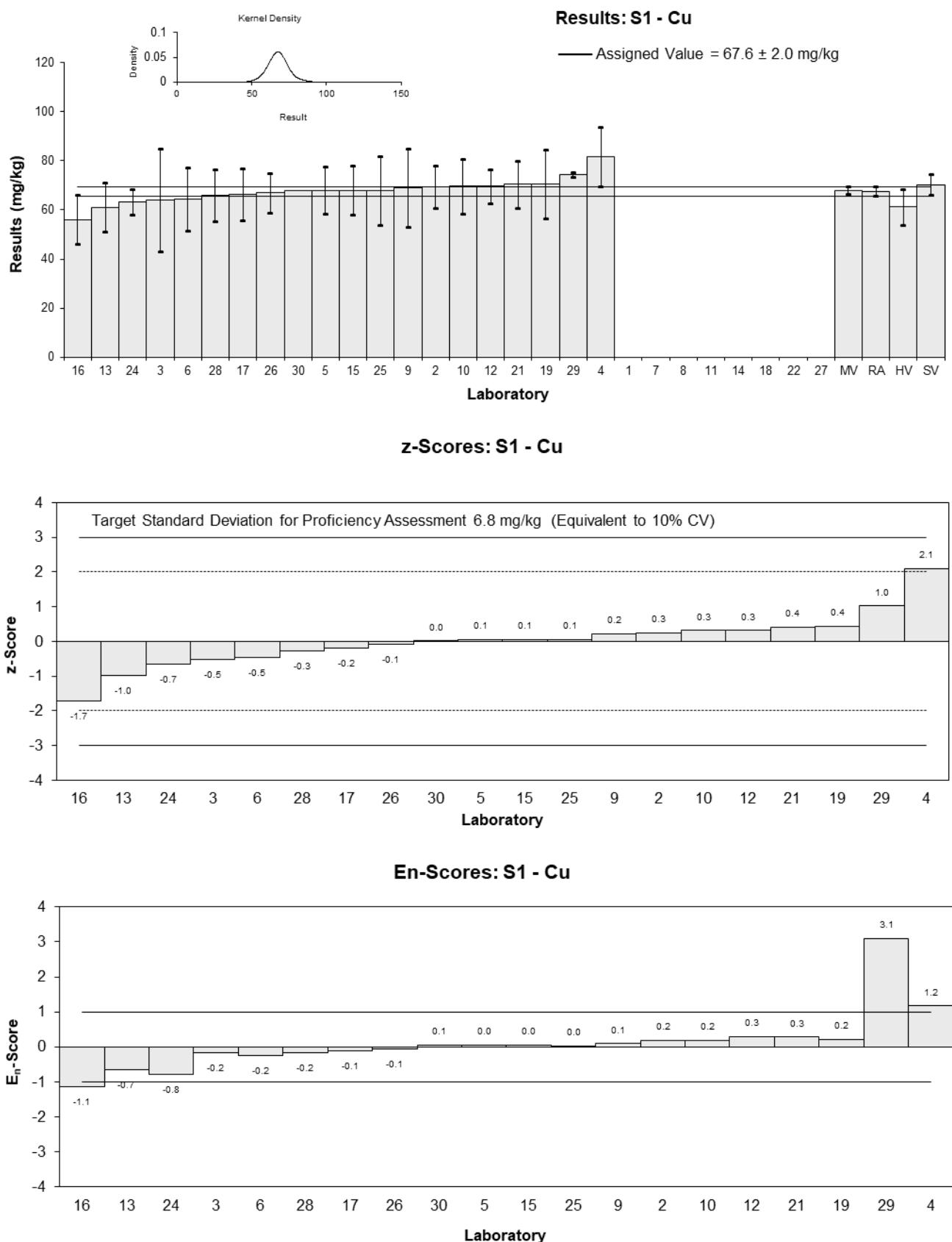


Figure 8

Table 21

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Hg
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	0.63	0.22	-0.46	-0.21
3	0.65	1	-0.27	-0.03
4	0.67	0.1	-0.07	-0.06
5	0.5	0.015	-1.74	-2.82
6	0.63	0.13	-0.46	-0.33
7	NT	NT		
8	NT	NT		
9	0.73	0.10	0.52	0.45
10	0.65	0.13	-0.27	-0.19
11	NT	NT		
12	0.67	0.1	-0.07	-0.06
13	0.78	1	1.01	0.10
14	NT	NT		
15	0.62	0.12	-0.56	-0.42
16	0.8	0.2	1.21	0.59
17	0.5	0.1	-1.74	-1.51
18	NT	NT		
19	0.95	0.29	2.69	0.92
21	0.75	0.14	0.72	0.48
22	NT	NT		
24	0.6	0.171	-0.76	-0.42
25	0.7	0.15	0.23	0.14
26	0.68	0.13	0.03	0.02
27	NT	NT		
28	0.59	0.08	-0.86	-0.86
29	NT	1		
30	1.01	NR	3.28	5.46

**Statistics**

<b>Assigned Value</b>	0.677	0.061
<b>Spike Value</b>	0.80	0.16
<b>Homogeneity Value</b>	0.633	0.079
<b>Robust Average</b>	0.677	0.061
<b>Median</b>	0.670	0.051
<b>Mean</b>	0.690	0.059
<b>N</b>	19	
<b>Max</b>	1.01	
<b>Min</b>	0.5	
<b>Robust SD</b>	0.11	
<b>Robust CV (%)</b>	16	

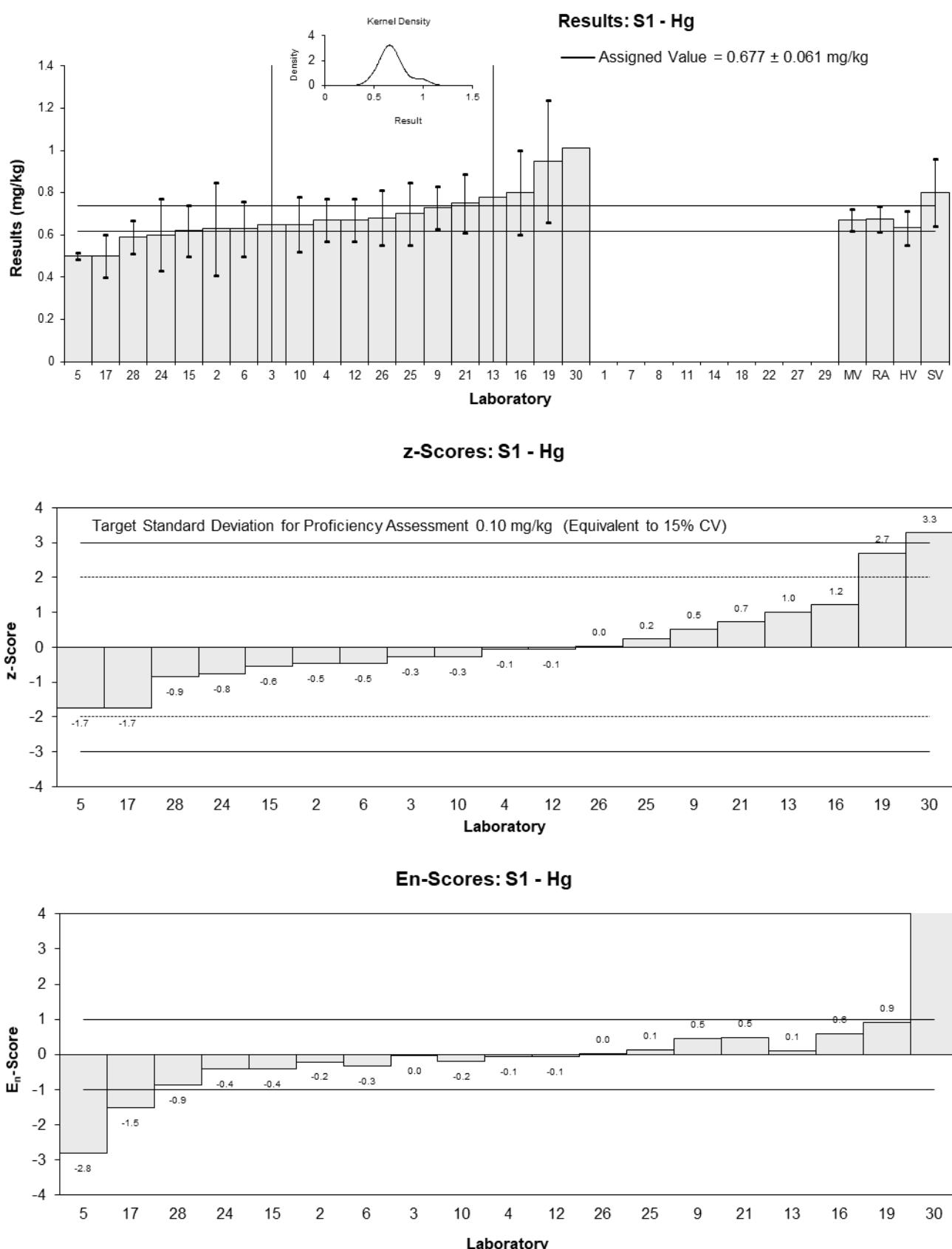


Figure 9

Table 22

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Mn
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	72.3	14.0	-0.15	-0.08
3	69	23	-0.60	-0.19
4	84.05	18.7	1.45	0.56
5	76	11.4	0.35	0.22
6	67.6	13.5	-0.79	-0.42
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	71.1	12.4	-0.31	-0.18
11	NT	NT		
12	76	8.0	0.35	0.30
13	70	20	-0.46	-0.17
14	NT	NT		
15	78	17	0.63	0.27
16	68	10	-0.74	-0.51
17	62.4	10.6	-1.50	-0.99
18	NT	NT		
19	75.5	11.3	0.29	0.18
21	81.7	8.2	1.13	0.93
22	NT	NT		
24	67.3	5.7972	-0.83	-0.90
25	76	16	0.35	0.16
26	75	8	0.22	0.18
27	NT	NT		
28	72.3	11.17	-0.15	-0.09
29	69.7	1	-0.50	-1.02
30	85.7	NR	1.68	3.51

**Statistics**

<b>Assigned Value</b>	73.4	3.5
<b>Spike Value</b>	74.9	5.0
<b>Homogeneity Value</b>	66.3	8.3
<b>Robust Average</b>	73.4	3.5
<b>Median</b>	72.3	3.1
<b>Mean</b>	73.6	2.8
<b>N</b>	19	
<b>Max</b>	85.7	
<b>Min</b>	62.4	
<b>Robust SD</b>	6.1	
<b>Robust CV (%)</b>	8.3	

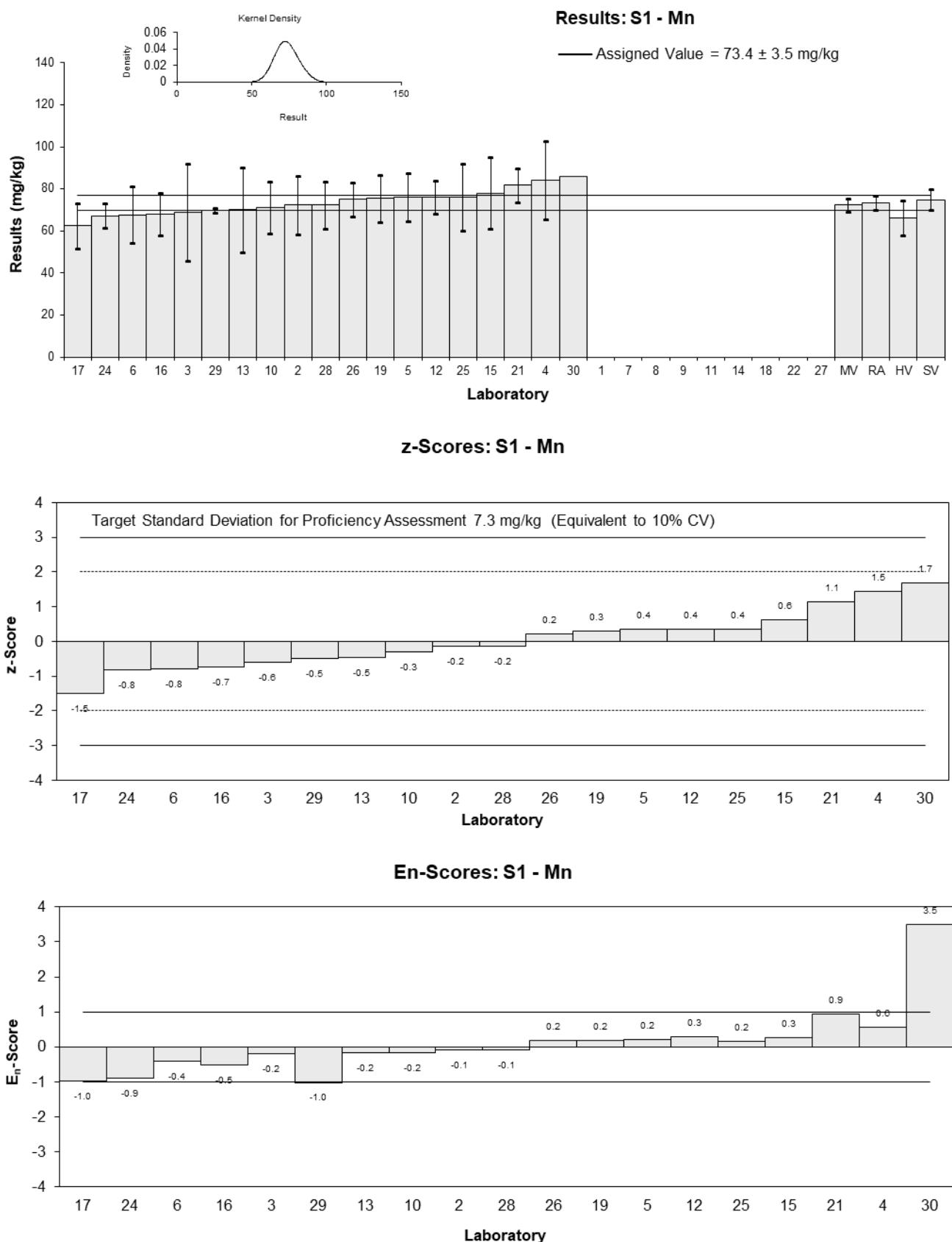


Figure 10

Table 23

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Mo
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	14.9	1.3	-0.26	-0.25
3	15	5	-0.20	-0.06
4	18.55	3.61	2.12	0.87
5	15	1.65	-0.20	-0.16
6	14.6	2.91	-0.46	-0.23
7	NT	NT		
8	NT	NT		
9	17	4.9	1.11	0.34
10	14.3	2.2	-0.65	-0.42
11	NT	NT		
12	15.9	2.0	0.39	0.27
13	14	4	-0.85	-0.32
14	NT	NT		
15	16	2.7	0.46	0.25
16	12	2	-2.16	-1.50
17	14.7	2.7	-0.39	-0.21
18	NT	NT		
19	13.0	4.5	-1.50	-0.50
21	17.1	3.1	1.18	0.56
22	NT	NT		
24	16.2	1.4438	0.59	0.53
25	16	3.4	0.46	0.20
26	17	3	1.11	0.54
27	NT	NT		
28	16.4	3.54	0.72	0.30
29	14.1	1	-0.78	-0.89
30	14.2	NR	-0.72	-1.22

**Statistics**

<b>Assigned Value</b>	15.3	0.9
<b>Spike Value</b>	15.1	0.3
<b>Homogeneity Value</b>	14.3	1.8
<b>Robust Average</b>	15.3	0.9
<b>Median</b>	15.0	0.8
<b>Mean</b>	15.3	0.7
<b>N</b>	20	
<b>Max</b>	18.55	
<b>Min</b>	12	
<b>Robust SD</b>	1.5	
<b>Robust CV (%)</b>	10	

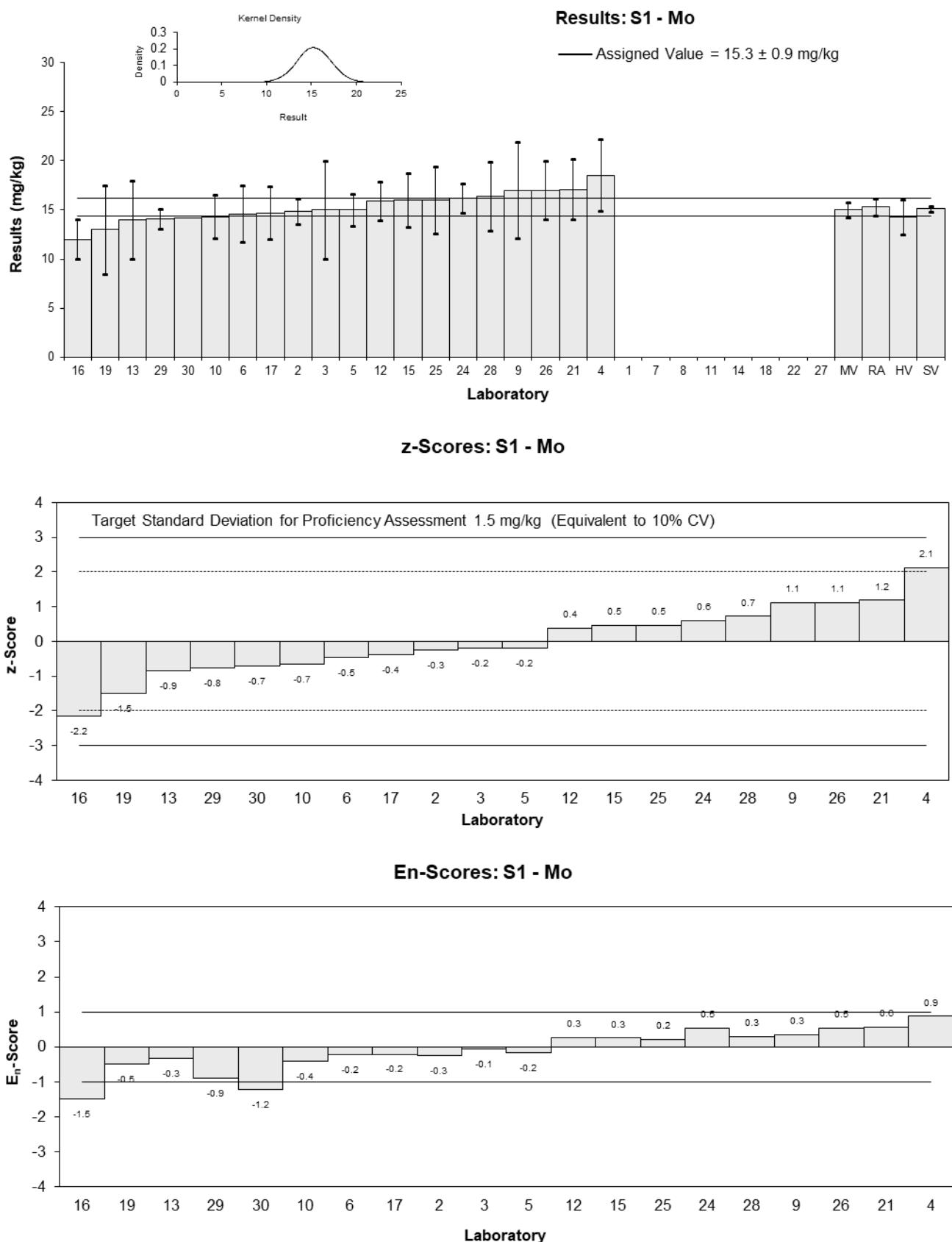


Figure 11

Table 24

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Ni
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	40.5	3.9	0.31	0.29
3	37	13	-0.59	-0.18
4	46.91	4.6	1.94	1.57
5	41	3.69	0.43	0.43
6	38.7	7.75	-0.15	-0.08
7	NT	NT		
8	NT	NT		
9	39	11	-0.08	-0.03
10	40.5	7.1	0.31	0.17
11	NT	NT		
12	41.3	4.5	0.51	0.42
13	36	10	-0.84	-0.33
14	NT	NT		
15	40	10	0.18	0.07
16	32	6	-1.86	-1.18
17	36.5	6.0	-0.71	-0.45
18	NT	NT		
19	41.5	16.5	0.56	0.13
21	42.3	5.6	0.76	0.52
22	NT	NT		
24	37.7	3.7065	-0.41	-0.40
25	42	8.9	0.69	0.30
26	39	5	-0.08	-0.06
27	NT	NT		
28	39.6	6.71	0.08	0.04
29	39.4	1	0.03	0.06
30	34.6	NR	-1.20	-3.13

**Statistics**

<b>Assigned Value</b>	39.3	1.5
<b>Spike Value</b>	40.0	0.9
<b>Homogeneity Value</b>	38.9	4.9
<b>Robust Average</b>	39.3	1.5
<b>Median</b>	39.5	1.5
<b>Mean</b>	39.3	1.4
<b>N</b>	20	
<b>Max</b>	46.91	
<b>Min</b>	32	
<b>Robust SD</b>	2.7	
<b>Robust CV (%)</b>	6.8	

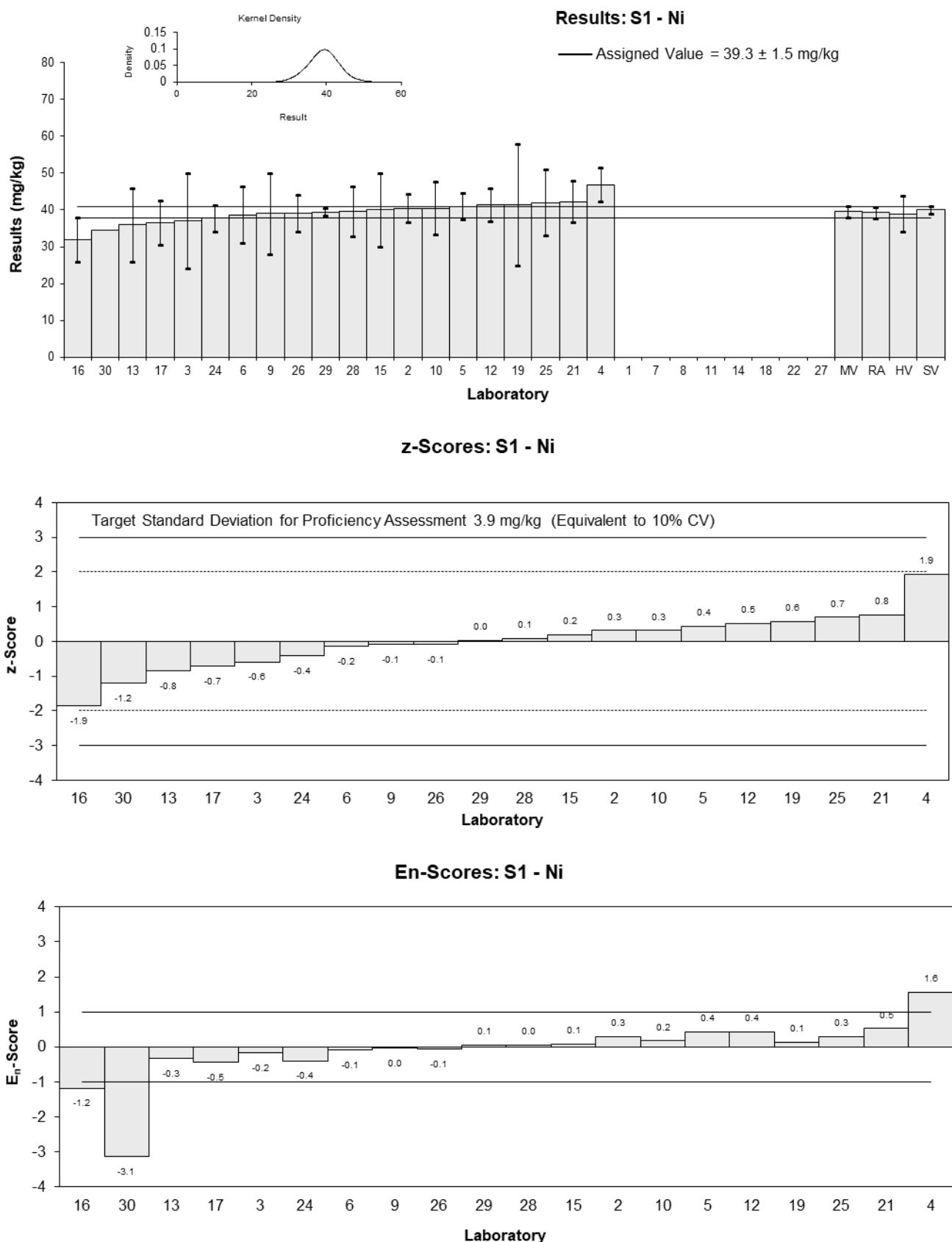


Figure 12

Table 25

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Pb
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	94.0	5.5	0.49	0.67
3	87	30	-0.29	-0.09
4	99.8	12.58	1.14	0.78
5	90	9	0.04	0.04
6	84.7	16.9	-0.55	-0.28
7	NT	NT		
8	NT	NT		
9	87	22	-0.29	-0.12
10	94.0	12.5	0.49	0.34
11	NT	NT		
12	87.2	9.0	-0.27	-0.25
13	84	20	-0.62	-0.28
14	NT	NT		
15	96	15	0.71	0.42
16	83	20	-0.74	-0.33
17	89.0	17.2	-0.07	-0.03
18	NT	NT		
19	89.5	22.4	-0.01	0.00
21	97	15	0.83	0.48
22	NT	NT		
24	89.7	5.9378	0.01	0.01
25	92	19	0.27	0.12
26	97	10	0.83	0.70
27	NT	NT		
28	92.1	16.75	0.28	0.15
29	79	1	-1.18	-2.91
30	72.4	NR	-1.92	-4.91

**Statistics**

<b>Assigned Value</b>	89.6	3.5
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	89	11
<b>Robust Average</b>	89.6	3.5
<b>Median</b>	89.6	3.6
<b>Mean</b>	89.2	3.0
<b>N</b>	20	
<b>Max</b>	99.8	
<b>Min</b>	72.4	
<b>Robust SD</b>	6.3	
<b>Robust CV (%)</b>	7	

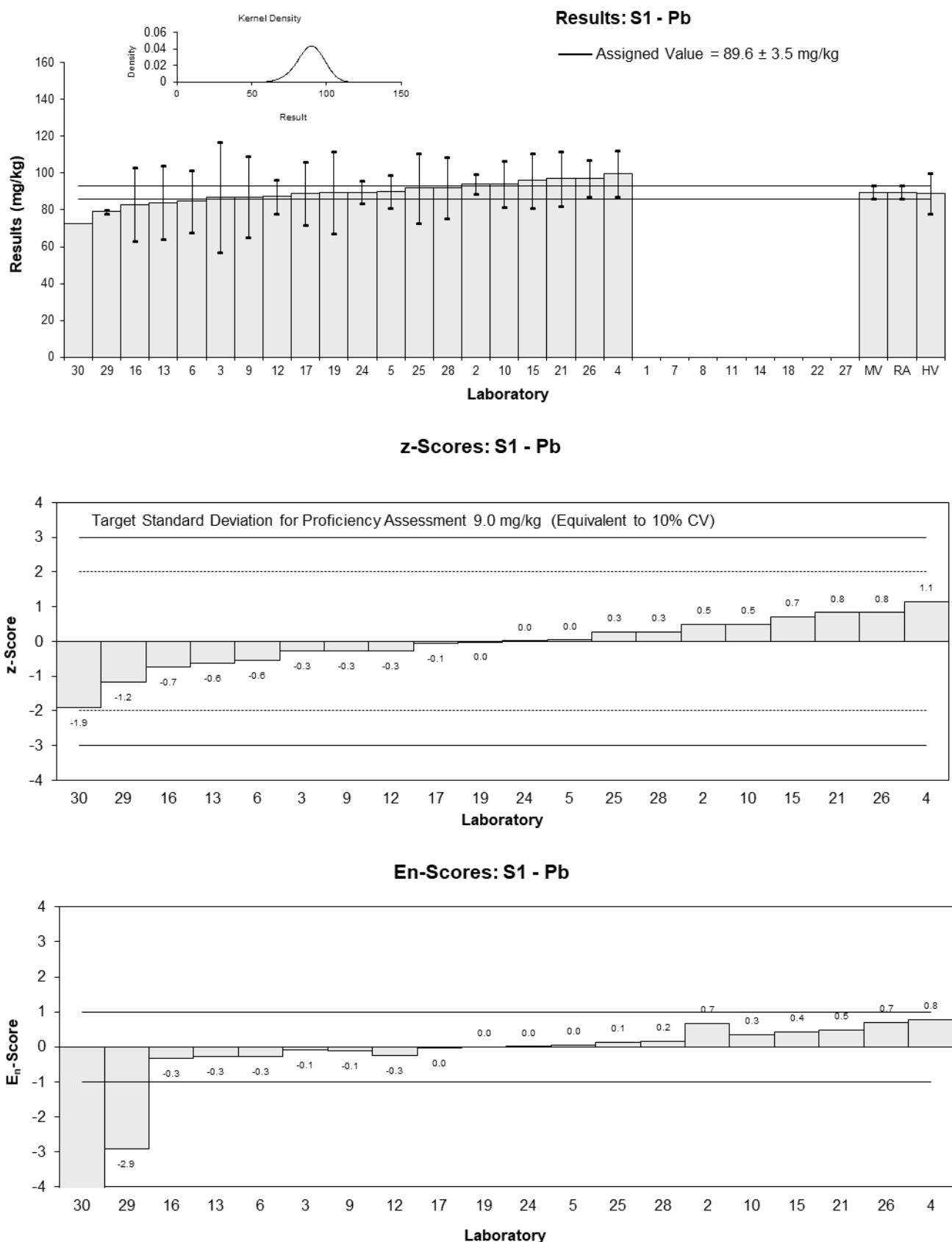


Figure 13

Table 26

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Rb
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	4.11	0.54	0.42	0.38
3	3.3	2	-0.65	-0.23
4	4.78	NR	1.31	1.55
5	NT	NT		
6	NT	NT		
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	4.94	0.99	1.52	0.98
11	NT	NT		
12	NT	NT		
13	4.0	2	0.28	0.10
14	NT	NT		
15	3.1	1	-0.91	-0.58
16	3.0	0.6	-1.04	-0.90
17	2.8	0.3	-1.31	-1.40
18	NT	NT		
19	NT	NT		
21	4.35	0.51	0.74	0.68
22	NT	NT		
24	3.2	0.3446	-0.78	-0.81
25	NT	NT		
26	NT	NT		
27	NT	NT		
28	4.11	NR	0.42	0.50
29	NT	1		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	3.79	0.64
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	4.01	0.50
<b>Robust Average</b>	3.79	0.64
<b>Median</b>	4.00	0.87
<b>Mean</b>	3.79	0.45
<b>N</b>	11	
<b>Max</b>	4.94	
<b>Min</b>	2.8	
<b>Robust SD</b>	0.84	
<b>Robust CV (%)</b>	22	

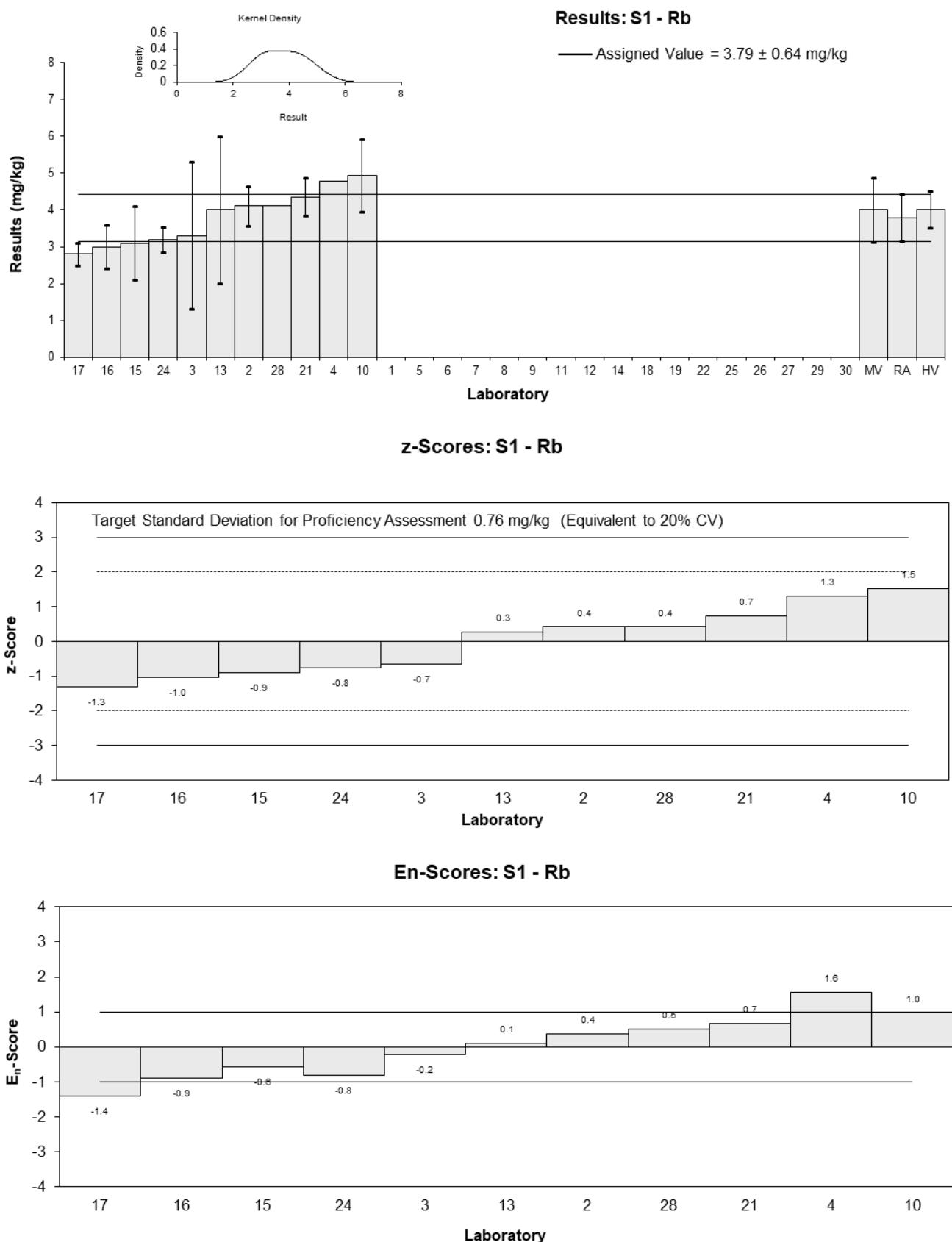


Figure 14

Table 27

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Se
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	2.57	0.67	0.36	0.18
3	2.4	2	-0.11	-0.02
4	3.19	NR	2.05	2.78
5	<5	NR		
6	2.23	0.45	-0.57	-0.40
7	NT	NT		
8	NT	NT		
9	<5.0	NR		
10	2.50	0.48	0.16	0.11
11	NT	NT		
12	2.45	0.3	0.03	0.02
13	2.1	2	-0.93	-0.17
14	NT	NT		
15	1.9	0.35	-1.48	-1.22
16	2.7	0.5	0.71	0.46
17	2	0.2	-1.20	-1.31
18	NT	NT		
19	<100	NR		
21	< 20	14		
22	NT	NT		
24	3	0.2212	1.53	1.60
25	2.4	0.5	-0.11	-0.07
26	<4	NR		
27	NT	NT		
28	<5	1.46		
29	2.5	1	0.16	0.06
30*	1.01	NR	-3.91	-5.30

\* Outlier

**Statistics**

<b>Assigned Value</b>	2.44	0.27
<b>Spike Value</b>	2.52	0.05
<b>Homogeneity Value</b>	2.33	0.28
<b>Robust Average</b>	2.40	0.29
<b>Median</b>	2.43	0.23
<b>Mean</b>	2.35	0.28
<b>N</b>	14	
<b>Max</b>	3.19	
<b>Min</b>	1.01	
<b>Robust SD</b>	0.43	
<b>Robust CV (%)</b>	18	

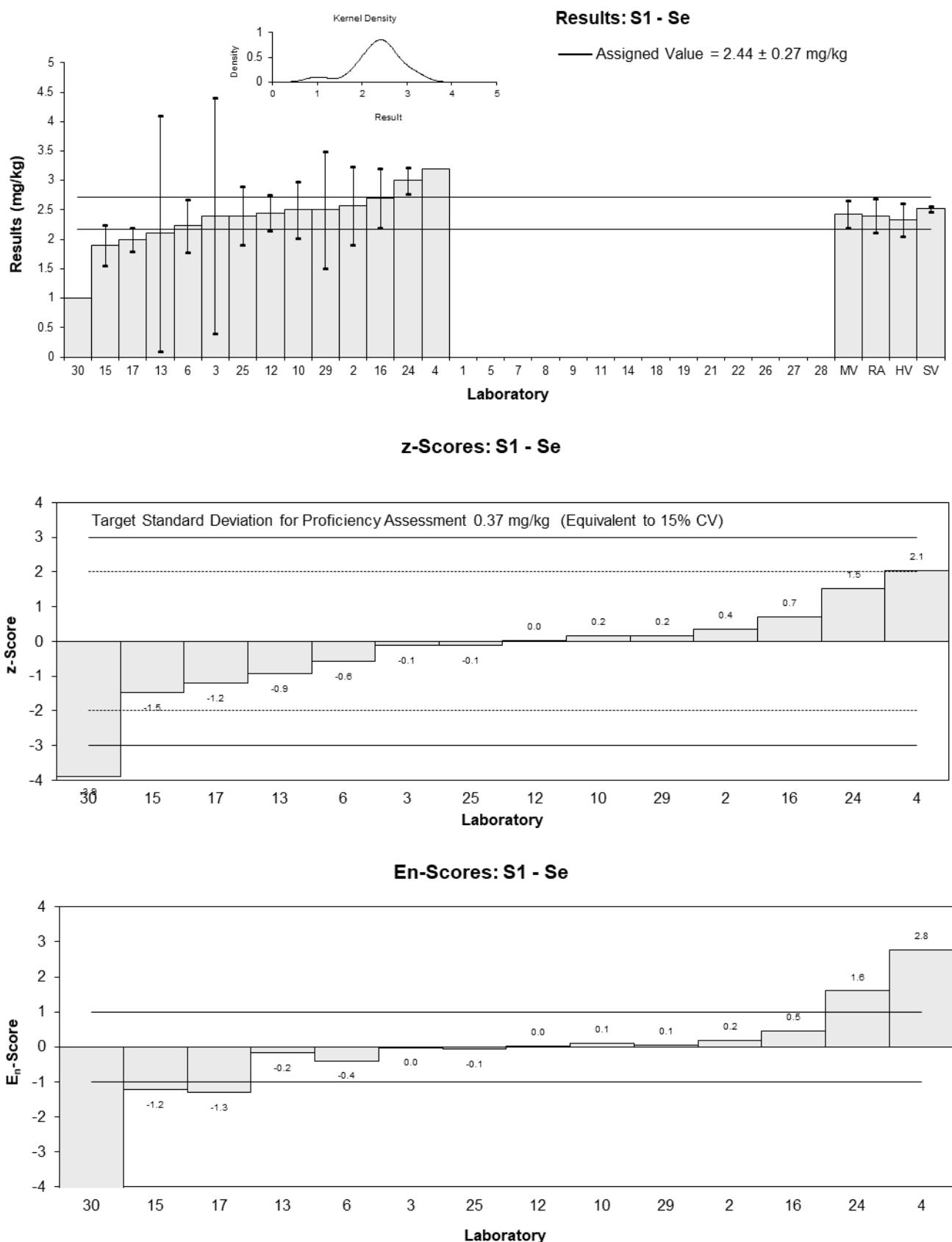


Figure 15

Table 28

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Sn
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	10.2	1.47	-1.74	-1.90
3	14	4	0.10	0.05
4	17.08	3.63	1.58	0.86
5	13	1.04	-0.39	-0.50
6	13.2	2.64	-0.29	-0.21
7	NT	NT		
8	NT	NT		
9	14	4.2	0.10	0.05
10	13.2	2.8	-0.29	-0.20
11	NT	NT		
12	14.3	2.0	0.24	0.21
13	11	3	-1.35	-0.87
14	NT	NT		
15	14	4.9	0.10	0.04
16	11	2	-1.35	-1.20
17	13.0	3.9	-0.39	-0.20
18	NT	NT		
19	<50	NR		
21	15.7	3.3	0.92	0.54
22	NT	NT		
24	15.6	1.9117	0.87	0.80
25	16	3.4	1.06	0.61
26	14	2	0.10	0.09
27	NT	NT		
28	14.3	2.32	0.24	0.19
29	NT	1		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	13.8	1.2
<b>Spike Value</b>	12.0	0.6
<b>Homogeneity Value</b>	11.7	1.4
<b>Robust Average</b>	13.8	1.2
<b>Median</b>	14.0	0.9
<b>Mean</b>	13.7	0.9
<b>N</b>	17	
<b>Max</b>	17.08	
<b>Min</b>	10.2	
<b>Robust SD</b>	1.9	
<b>Robust CV (%)</b>	14	

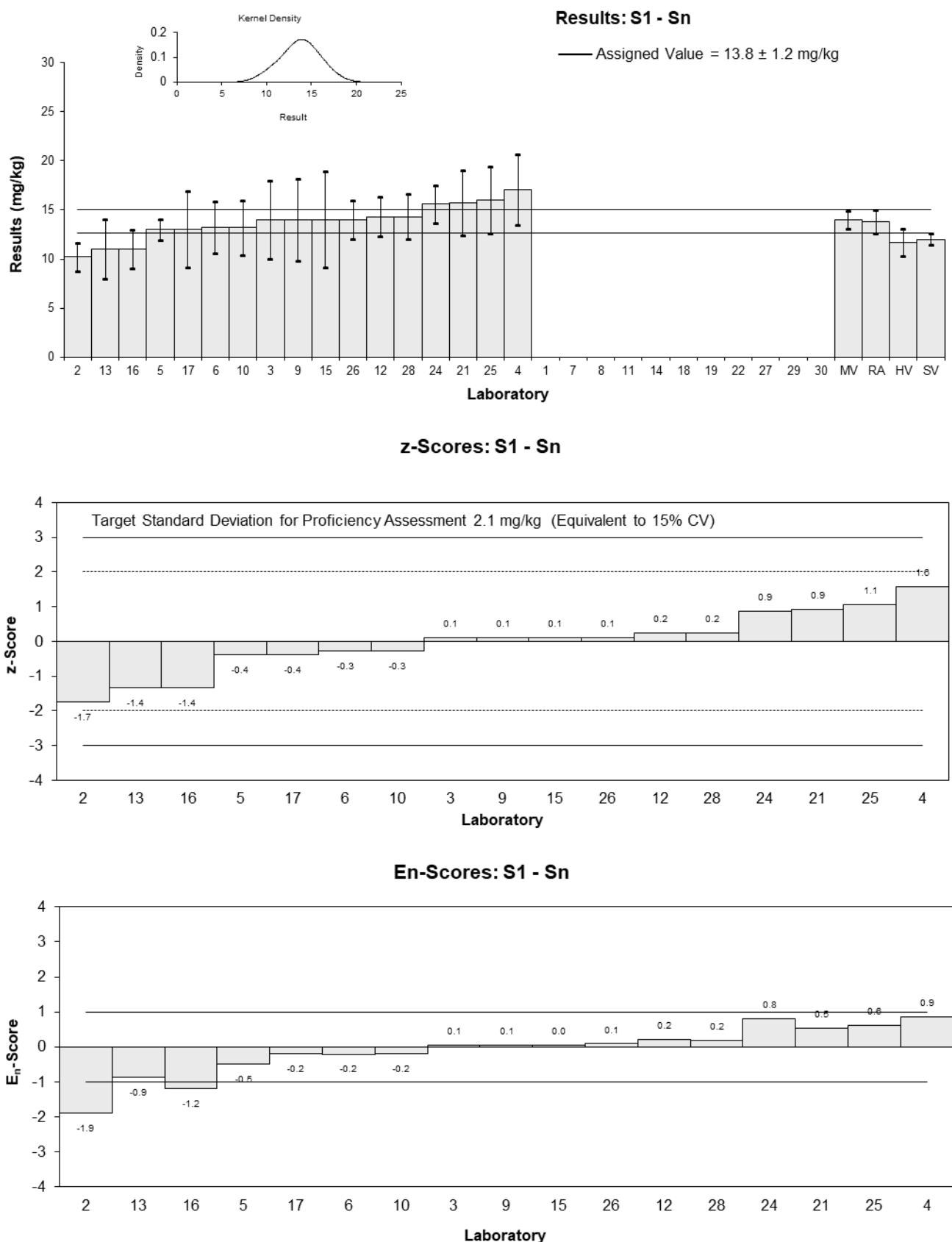


Figure 16

Table 29

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Th
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U
1	NT	NT
2	0.68	0.11
3	0.76	2
4	0.87	0.19
5	NT	NT
6	NT	NT
7	NT	NT
8	NT	NT
9	NT	NT
10	1.37	0.27
11	NT	NT
12	3.41	0.4
13	1.3	2
14	NT	NT
15	<9.1	NR
16	1.1	0.2
17	0.8	0.2
18	NT	NT
19	NT	NT
21	NT	NT
22	NT	NT
24	1.5	0.10801
25	NT	NT
26	NT	NT
27	NT	NT
28	0.83	NR
29	NT	1
30	NT	NT

**Statistics**

<b>Assigned Value</b>	Not Set	
<b>Spike Value</b>	4.47	0.10
<b>Homogeneity Value</b>	1.60	0.19
<b>Robust Average</b>	1.09	0.32
<b>Median</b>	0.99	0.31
<b>Mean</b>	1.26	0.51
<b>N</b>	10	
<b>Max</b>	3.41	
<b>Min</b>	0.68	
<b>Robust SD</b>	0.40	
<b>Robust CV (%)</b>	37	

**Results: S1 - Th**

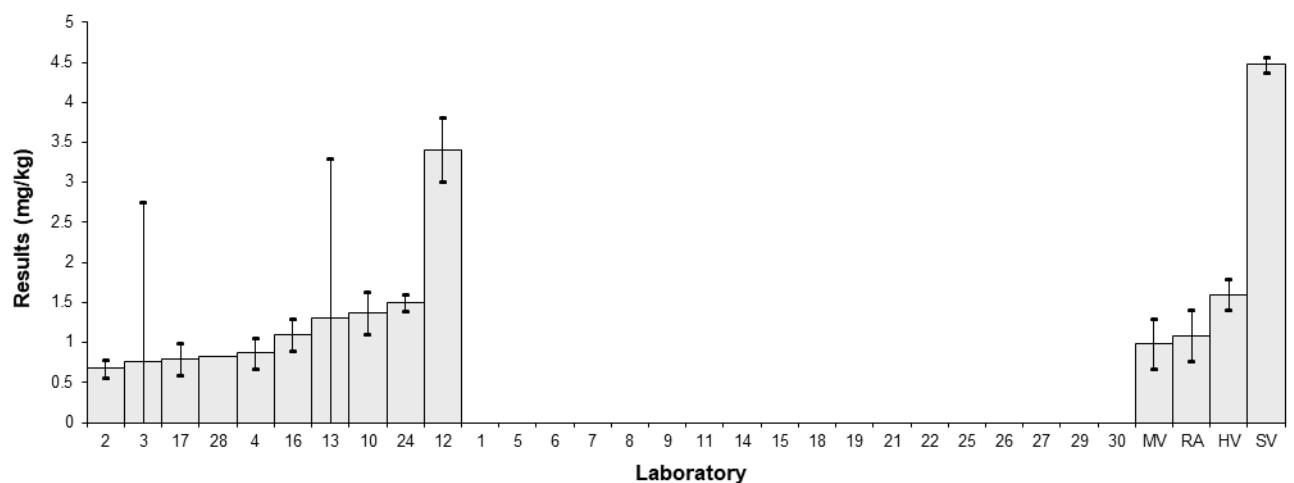


Figure 17

Table 30

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	V
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	25.4	2.2	-0.04	-0.04
3	25	8	-0.20	-0.06
4	27.99	4.86	0.98	0.50
5	27	1.35	0.59	0.89
6	25.6	5.13	0.04	0.02
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	25.1	5.4	-0.16	-0.07
11	NT	NT		
12	27.2	3.5	0.67	0.47
13	26	5	0.20	0.10
14	NT	NT		
15	24	3.7	-0.59	-0.39
16	21	4	-1.76	-1.09
17	22	5	-1.37	-0.69
18	NT	NT		
19	24.5	7.3	-0.39	-0.14
21	< 100	67		
22	NT	NT		
24	24	1.7939	-0.59	-0.73
25	27	5.7	0.59	0.26
26	27	7	0.59	0.21
27	NT	NT		
28	26.2	3.85	0.27	0.18
29	25.5	1	0.00	0.00
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	25.5	1.0
<b>Spike Value</b>	25.2	1.3
<b>Homogeneity Value</b>	21.7	2.6
<b>Robust Average</b>	25.5	1.0
<b>Median</b>	25.5	1.3
<b>Mean</b>	25.3	0.9
<b>N</b>	17	
<b>Max</b>	27.99	
<b>Min</b>	21	
<b>Robust SD</b>	1.7	
<b>Robust CV (%)</b>	6.6	

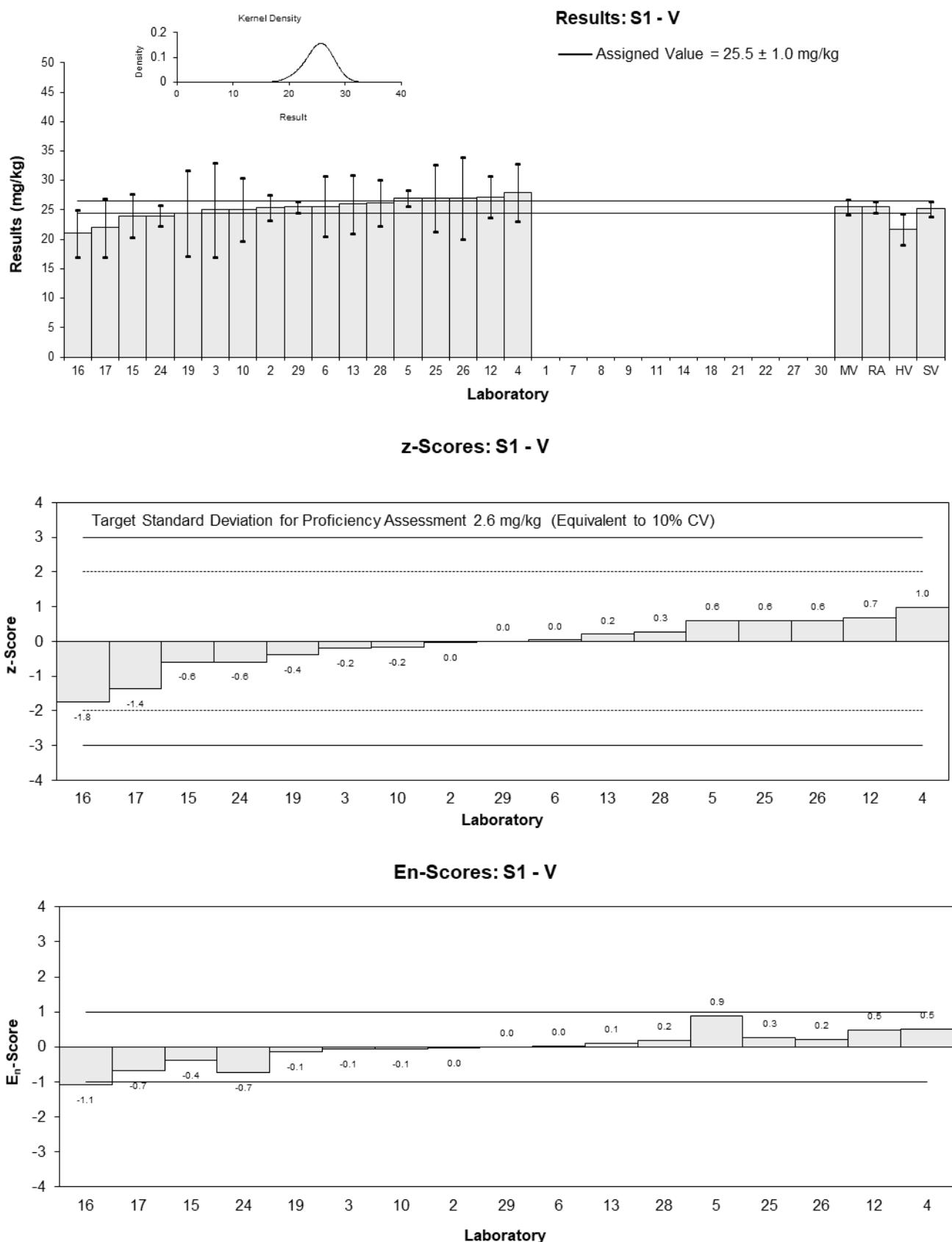


Figure 18

Table 31

**Sample Details**

<b>Sample</b>	S1
<b>Analyte</b>	Zn
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	63.3	10.2	-0.09	-0.06
3	59	20	-0.77	-0.24
4	73.97	9.11	1.58	1.07
5	62	3.72	-0.30	-0.42
6	61.2	12.2	-0.42	-0.22
7	NT	NT		
8	NT	NT		
9	66	16	0.33	0.13
10	66.5	9.2	0.41	0.27
11	NT	NT		
12	64.1	7.0	0.03	0.03
13	56	10	-1.24	-0.77
14	NT	NT		
15	68	10	0.64	0.40
16	50	10	-2.18	-1.35
17	61.1	10.4	-0.44	-0.26
18	NT	NT		
19	64.5	13	0.09	0.05
21	67.1	5.5	0.50	0.53
22	NT	NT		
24	60.4	5.0065	-0.55	-0.63
25	67	14	0.49	0.22
26	70	8	0.95	0.73
27	NT	NT		
28	64.8	10.92	0.14	0.08
29	NT	1		
30	63.9	NR	0.00	0.00

**Statistics**

<b>Assigned Value</b>	63.9	2.5
<b>Spike Value</b>	65.3	3.7
<b>Homogeneity Value</b>	60.0	7.2
<b>Robust Average</b>	63.9	2.5
<b>Median</b>	64.1	2.5
<b>Mean</b>	63.6	2.4
<b>N</b>	19	
<b>Max</b>	73.97	
<b>Min</b>	50	
<b>Robust SD</b>	4.4	
<b>Robust CV (%)</b>	6.9	

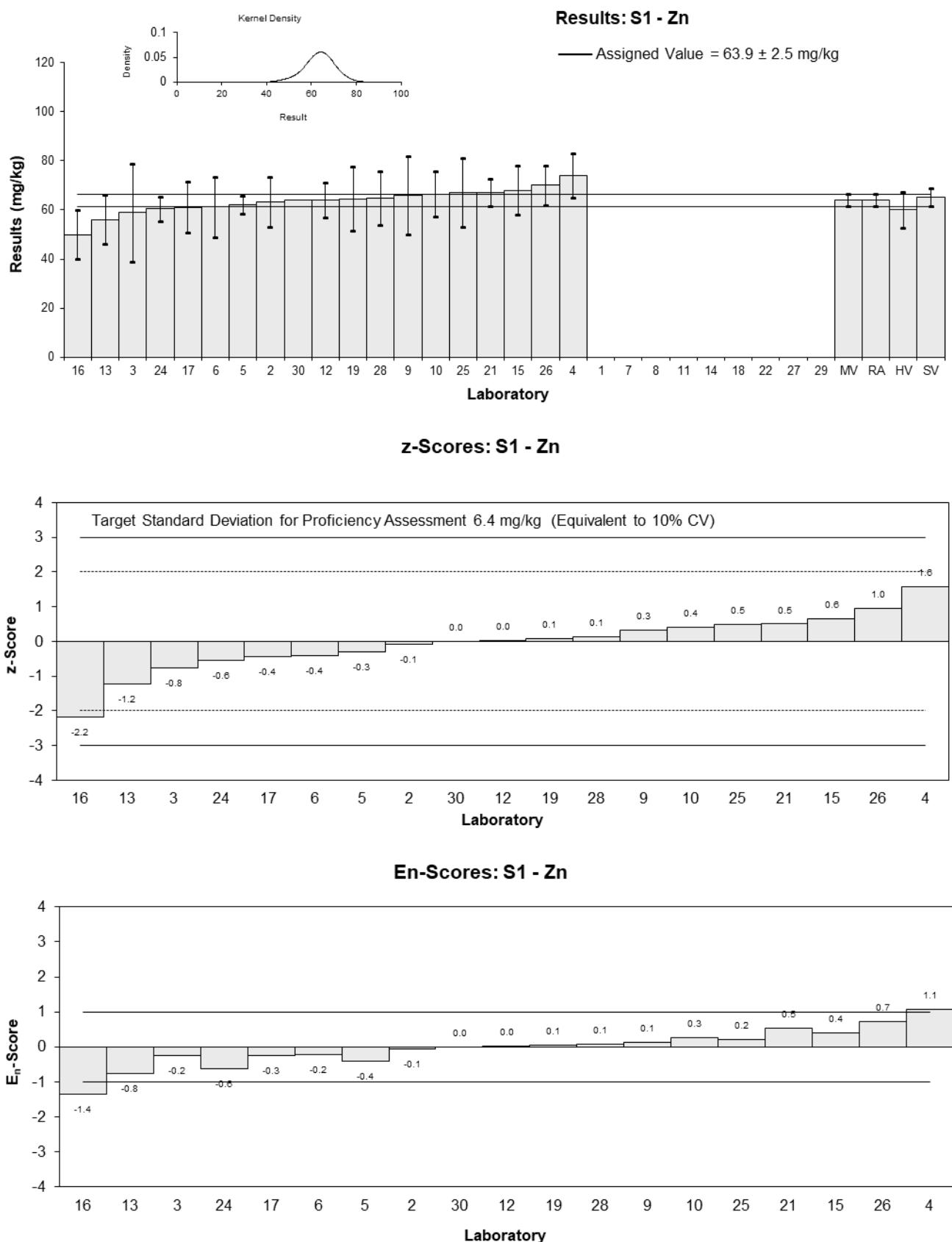


Figure 19

Table 32

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Ag
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	6.12	1.87	1.63	0.45
3	4.7	2	-1.06	-0.28
4	5	1.9	-0.49	-0.13
5	4.6	0.322	-1.25	-1.33
6	5.52	1.38	0.49	0.18
7	NT	NT		
8	NT	NT		
9	<5.0	NR		
10	5.18	0.95	-0.15	-0.08
11	5.25	0.41	-0.02	-0.02
12	4.55	0.6	-1.35	-1.00
13	5.7	2	0.84	0.22
14	NT	NT		
15	5.3	1.8	0.08	0.02
16	5.0	1	-0.49	-0.24
17	5.0	1.0	-0.49	-0.24
18	NT	NT		
19	NT	NT		
21	NT	NT		
22*	8	1.2	5.21	2.18
24	6.2	0.8238	1.79	1.04
25	4.9	1	-0.68	-0.34
26*	2.5	1	-5.25	-2.58
27	6.19	1.24	1.77	0.72
28	5.0	1.13	-0.49	-0.22
29*	2.04	1	-6.12	-3.01
30	NT	NT		

\* Outlier

**Statistics**

<b>Assigned Value</b>	5.26	0.38
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	5.29	0.66
<b>Robust Average</b>	5.19	0.45
<b>Median</b>	5.00	0.34
<b>Mean</b>	5.09	0.59
<b>N</b>	19	
<b>Max</b>	8	
<b>Min</b>	2.04	
<b>Robust SD</b>	0.79	
<b>Robust CV (%)</b>	15	

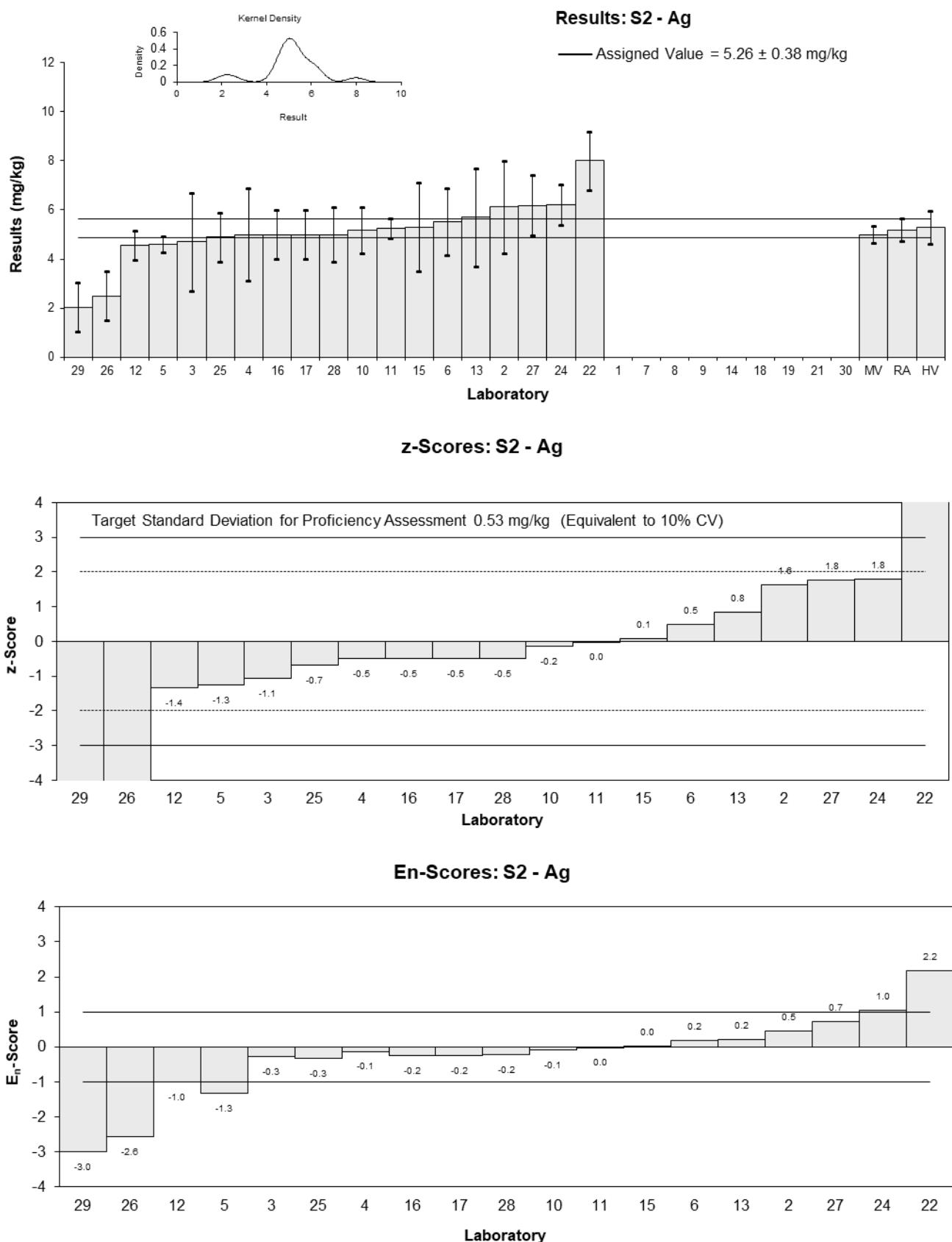


Figure 20

Table 33

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Al
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	3329	518	0.76	0.53
3**	4.1	2	-6.66	-8.07
4	2370	47.4	-1.38	-1.66
5	3700	296	1.58	1.50
6	3570	892	1.29	0.60
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	3230	670	0.54	0.31
11	2610	420	-0.85	-0.68
12	3484	350	1.10	0.97
13	3100	500	0.25	0.18
14	NT	NT		
15	2200	740	-1.76	-0.95
16**	3.2	0.6	-6.66	-8.07
17	1800	208	-2.65	-2.80
18	NT	NT		
19	3300	495	0.69	0.50
21	NT	NT		
22	3400	510	0.91	0.65
24	2280	356.01	-1.58	-1.38
25	3500	74	1.14	1.35
26	2700	340	-0.65	-0.58
27	3270	654	0.62	0.37
28	2663	526.7	-0.73	-0.51
29	NT	1		
30	NT	NT		

\*\* Gross Error

**Statistics**

<b>Assigned Value</b>	2990	370
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	2910	350
<b>Robust Average</b>	2990	370
<b>Median</b>	3230	310
<b>Mean</b>	2970	270
<b>N</b>	17	
<b>Max</b>	3700	
<b>Min</b>	1800	
<b>Robust SD</b>	600	
<b>Robust CV (%)</b>	20	

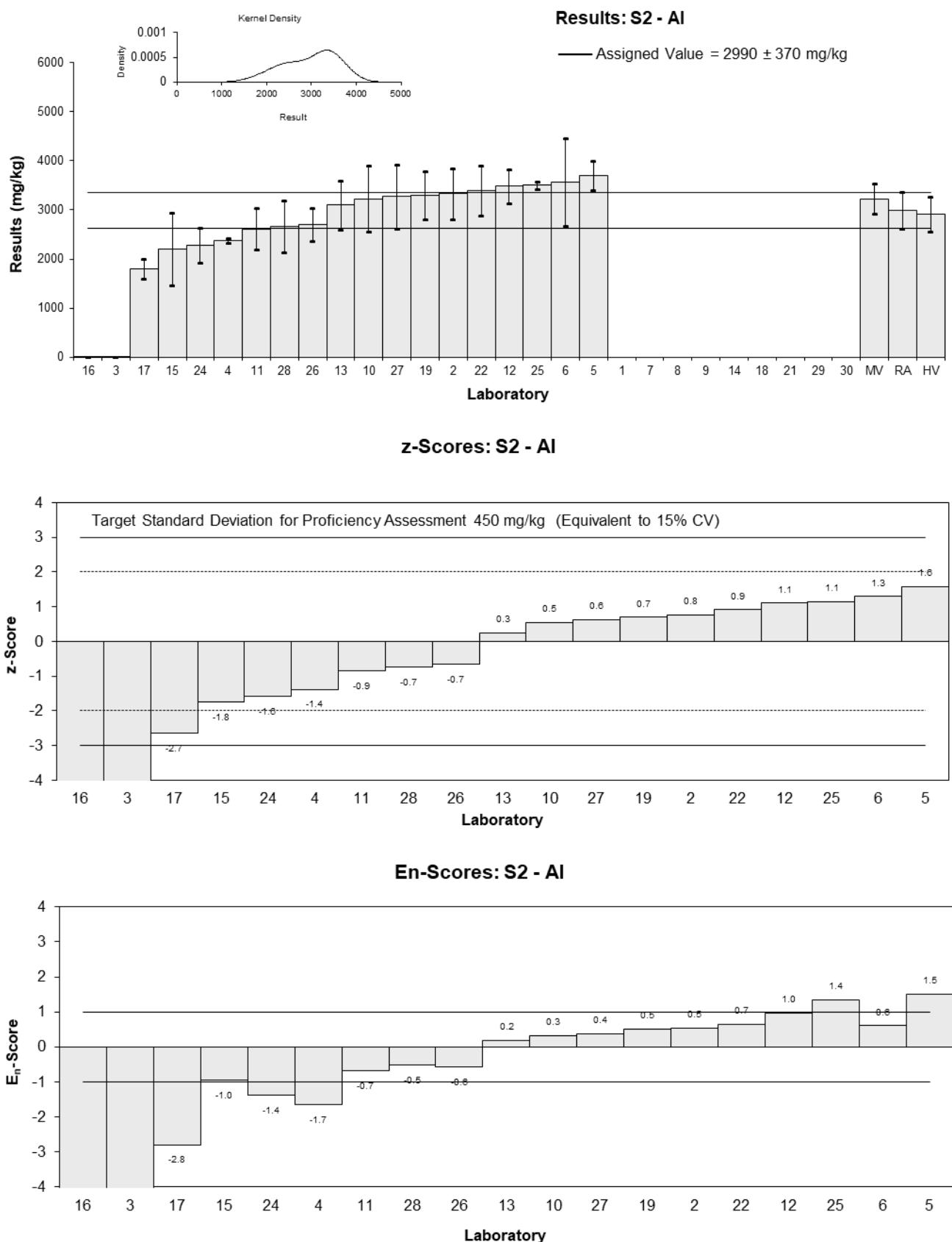


Figure 21

Table 34

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	As
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	4.10	0.63	1.52	0.83
3**	3000	1100	8,416.97	2.72
4	3.34	0.42	-0.62	-0.49
5	<5	NR		
6	3.71	0.93	0.42	0.16
7	NT	NT		
8	NT	NT		
9	3.0	0.85	-1.57	-0.65
10	3.49	0.60	-0.20	-0.11
11	3.77	0.44	0.59	0.45
12	3.57	0.6	0.03	0.02
13	3.5	2	-0.17	-0.03
14	NT	NT		
15	3.8	0.58	0.67	0.40
16**	2800	600	7,855.17	4.66
17	3.4	0.5	-0.45	-0.31
18	NT	NT		
19	<25	NR		
21	NT	NT		
22*	6	1	6.85	2.41
24	3.4	0.3555	-0.45	-0.41
25	3.6	0.76	0.11	0.05
26	<4	NR		
27	3.58	0.716	0.06	0.03
28	<5	0.84		
29	NT	1		
30	NT	NT		

\* Outlier, \*\* Gross Error

**Statistics**

<b>Assigned Value</b>	3.56	0.15
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	3.35	0.42
<b>Robust Average</b>	3.60	0.18
<b>Median</b>	3.58	0.17
<b>Mean</b>	3.73	0.37
<b>N</b>	14	
<b>Max</b>	6	
<b>Min</b>	3	
<b>Robust SD</b>	0.27	
<b>Robust CV (%)</b>	7.5	

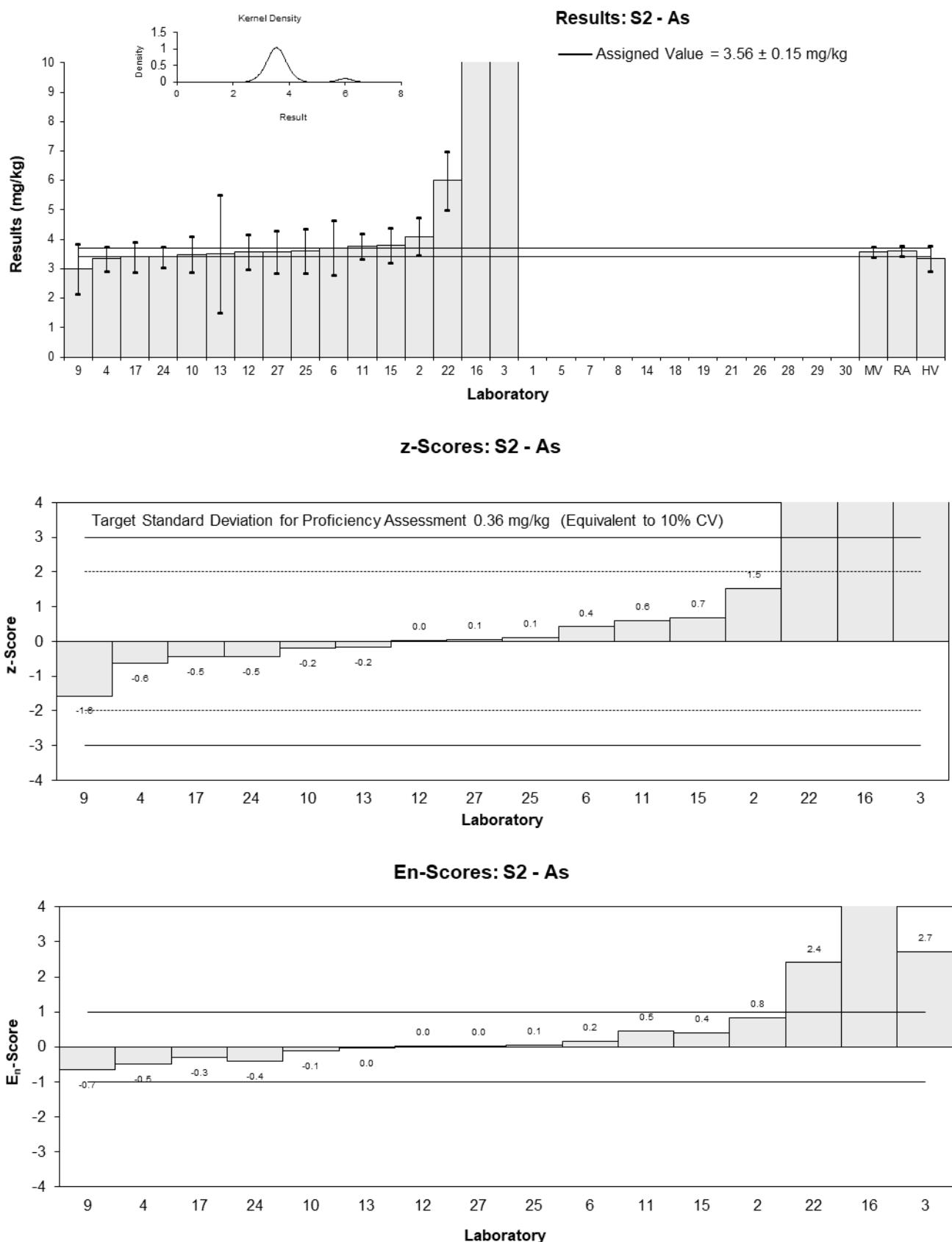


Figure 22

Table 35

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Ba
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	32.7	2.68	0.79	0.69
3	34	11	1.22	0.33
4	25.43	3.74	-1.61	-1.12
5	35	6.3	1.55	0.70
6	34.5	8.63	1.39	0.47
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	29.7	4.6	-0.20	-0.12
11	28.1	5.5	-0.73	-0.37
12	33.4	4.0	1.02	0.68
13	30	5	-0.10	-0.05
14	NT	NT		
15	25	6	-1.75	-0.83
16	27	5	-1.09	-0.60
17	25.8	4.4	-1.49	-0.91
18	NT	NT		
19	35.5	16.0	1.72	0.32
21	NT	NT		
22	32	4.8	0.56	0.32
24	25.8	2.0155	-1.49	-1.51
25	32	6.7	0.56	0.24
26	34	5	1.22	0.68
27	30.9	6.18	0.20	0.09
28	27.3	4.69	-0.99	-0.58
29	28.5	1	-0.59	-0.74
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	30.3	2.2
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	30.0	3.6
<b>Robust Average</b>	30.3	2.2
<b>Median</b>	30.5	2.7
<b>Mean</b>	30.3	1.6
<b>N</b>	20	
<b>Max</b>	35.5	
<b>Min</b>	25	
<b>Robust SD</b>	4.0	
<b>Robust CV (%)</b>	13	

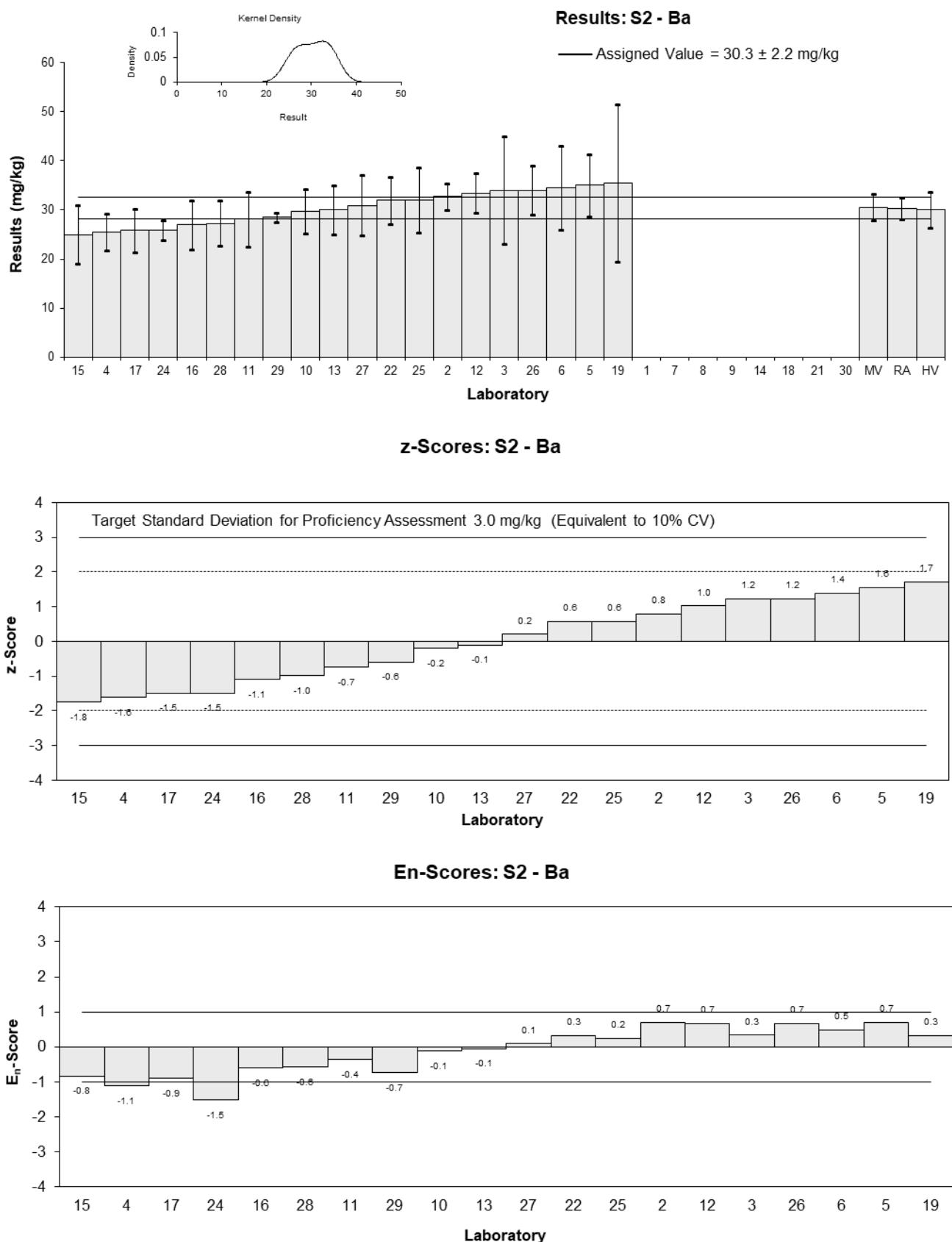


Figure 23

Table 36

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Cd
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	1.72	0.55	2.46	0.61
3	1.4	1	0.14	0.02
4	1.26	0.37	-0.87	-0.32
5	1.4	0.084	0.14	0.18
6	1.45	0.36	0.51	0.19
7	NT	NT		
8	NT	NT		
9	1.3	0.14	-0.58	-0.51
10	1.45	0.20	0.51	0.33
11	1.38	0.13	0.00	0.00
12	1.38	0.2	0.00	0.00
13	1.4	1	0.14	0.02
14	NT	NT		
15	1.5	0.28	0.87	0.42
16	1.3	0.3	-0.58	-0.26
17	1.4	0.2	0.14	0.09
18	NT	NT		
19	1.10	0.2	-2.03	-1.32
21	NT	NT		
22*	2.1	0.32	5.22	2.20
24	1.5	0.05891	0.87	1.31
25	1.4	0.29	0.14	0.07
26	1	1	-2.75	-0.38
27	1.55	0.31	1.23	0.53
28	1.3	0.21	-0.58	-0.36
29	1.29	1	-0.65	-0.09
30	NT	NT		

\* Outlier

**Statistics**

<b>Assigned Value</b>	1.38	0.07
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	1.47	0.18
<b>Robust Average</b>	1.39	0.07
<b>Median</b>	1.40	0.08
<b>Mean</b>	1.41	0.10
<b>N</b>	21	
<b>Max</b>	2.1	
<b>Min</b>	1	
<b>Robust SD</b>	0.13	
<b>Robust CV (%)</b>	9.3	

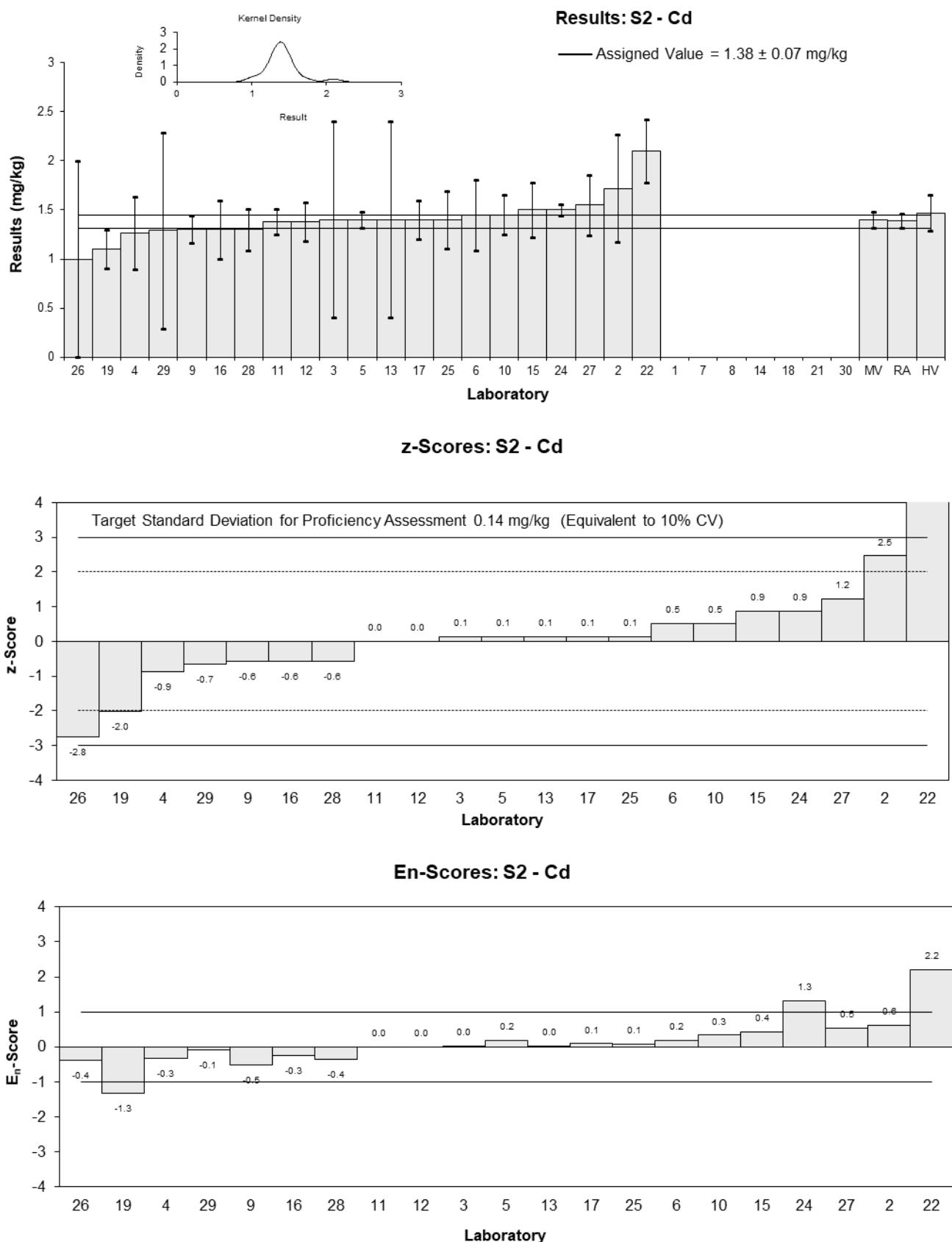


Figure 24

Table 37

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Co
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	11.1	3.6	1.76	0.46
3	9.8	3	0.38	0.12
4	8.55	0.99	-0.94	-0.81
5	9.6	NR	0.17	0.34
6	10.0	2.50	0.59	0.22
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	9.14	1.4	-0.32	-0.20
11	9.59	1.05	0.16	0.13
12	9.14	1.0	-0.32	-0.27
13	9.5	2	0.06	0.03
14	NT	NT		
15	8.7	1.3	-0.78	-0.54
16	8.7	2	-0.78	-0.36
17	8.3	1.3	-1.21	-0.82
18	NT	NT		
19	10.0	2.5	0.59	0.22
21	NT	NT		
22	10	1.5	0.59	0.36
24	9.3	0.5853	-0.15	-0.19
25	9.4	2	-0.04	-0.02
26	9	2	-0.47	-0.21
27	10.9	2.18	1.55	0.65
28	8.2	1.18	-1.31	-0.98
29	10.5	1	1.12	0.96
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	9.44	0.47
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	9.5	1.2
<b>Robust Average</b>	9.44	0.47
<b>Median</b>	9.45	0.46
<b>Mean</b>	9.47	0.36
<b>N</b>	20	
<b>Max</b>	11.1	
<b>Min</b>	8.2	
<b>Robust SD</b>	0.85	
<b>Robust CV (%)</b>	9	

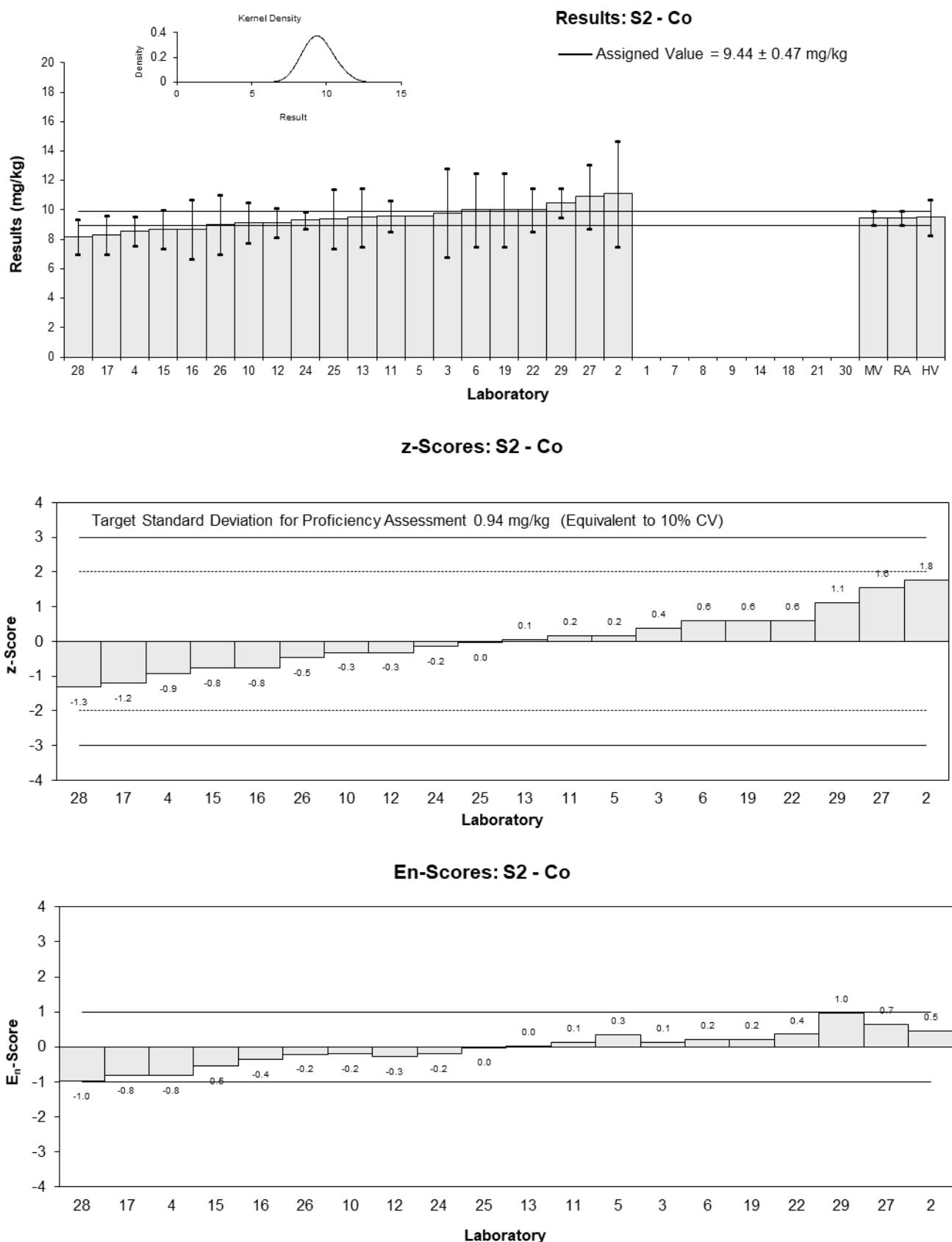


Figure 25

Table 38

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Cu
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	66.4	12.7	1.29	0.59
3	60	20	0.20	0.06
4	56.32	8.25	-0.42	-0.29
5	56	7.84	-0.48	-0.34
6	62.6	15.7	0.65	0.24
7	NT	NT		
8	NT	NT		
9	56	13	-0.48	-0.21
10	60.0	9.6	0.20	0.12
11	61.8	6.8	0.51	0.42
12	57.7	7.5	-0.19	-0.14
13	56	10	-0.48	-0.27
14	NT	NT		
15	53	8	-0.99	-0.70
16	54	10	-0.82	-0.47
17	60.4	9.6	0.27	0.16
18	NT	NT		
19	69.5	14	1.82	0.75
21	NT	NT		
22	61	9.15	0.37	0.23
24	57	4.7775	-0.31	-0.34
25	57	12	-0.31	-0.15
26	58	7	-0.14	-0.11
27	59.6	11.9	0.14	0.07
28	53.0	8.54	-0.99	-0.66
29	71.1	1	2.09	4.90
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	58.8	2.3
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	58.8	7.1
<b>Robust Average</b>	58.8	2.3
<b>Median</b>	58.0	1.6
<b>Mean</b>	59.4	2.1
<b>N</b>	21	
<b>Max</b>	71.1	
<b>Min</b>	53	
<b>Robust SD</b>	4.3	
<b>Robust CV (%)</b>	7.3	

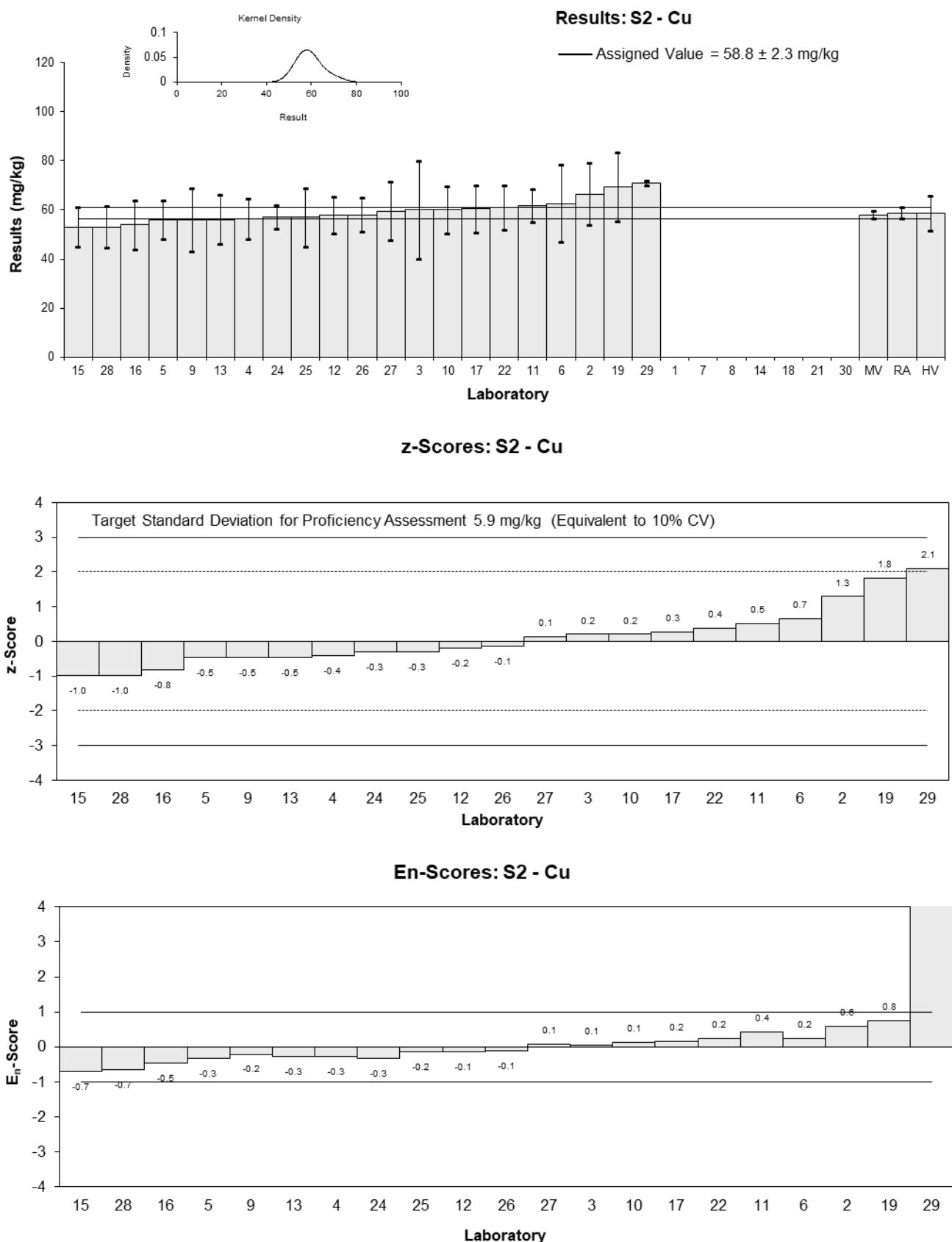


Figure 26

Table 39

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Hg
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	0.54	0.17	-0.36	-0.18
3	0.6	1	0.34	0.03
4	0.51	0.08	-0.71	-0.69
5	0.5	0.015	-0.83	-1.70
6	0.65	0.16	0.92	0.48
7	NT	NT		
8	NT	NT		
9	0.60	0.083	0.34	0.32
10	0.59	0.12	0.22	0.15
11	0.583	0.06	0.14	0.17
12	0.52	0.1	-0.60	-0.48
13	0.72	2	1.74	0.07
14	NT	NT		
15	0.5	0.09	-0.83	-0.72
16	0.8	0.2	2.67	1.12
17	0.5	0.1	-0.83	-0.66
18	NT	NT		
19*	0.92	0.29	4.07	1.19
21	NT	NT		
22	0.63	0.095	0.69	0.57
24	0.6	0.171	0.34	0.17
25	0.6	0.13	0.34	0.21
26	0.54	0.1	-0.36	-0.29
27	0.54	0.108	-0.36	-0.27
28	0.50	0.07	-0.83	-0.89
29	NT	1		
30	NT	NT		

\* Outlier

**Statistics**

<b>Assigned Value</b>	0.571	0.039
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	0.530	0.064
<b>Robust Average</b>	0.579	0.043
<b>Median</b>	0.587	0.046
<b>Mean</b>	0.597	0.049
<b>N</b>	20	
<b>Max</b>	0.92	
<b>Min</b>	0.5	
<b>Robust SD</b>	0.077	
<b>Robust CV (%)</b>	13	

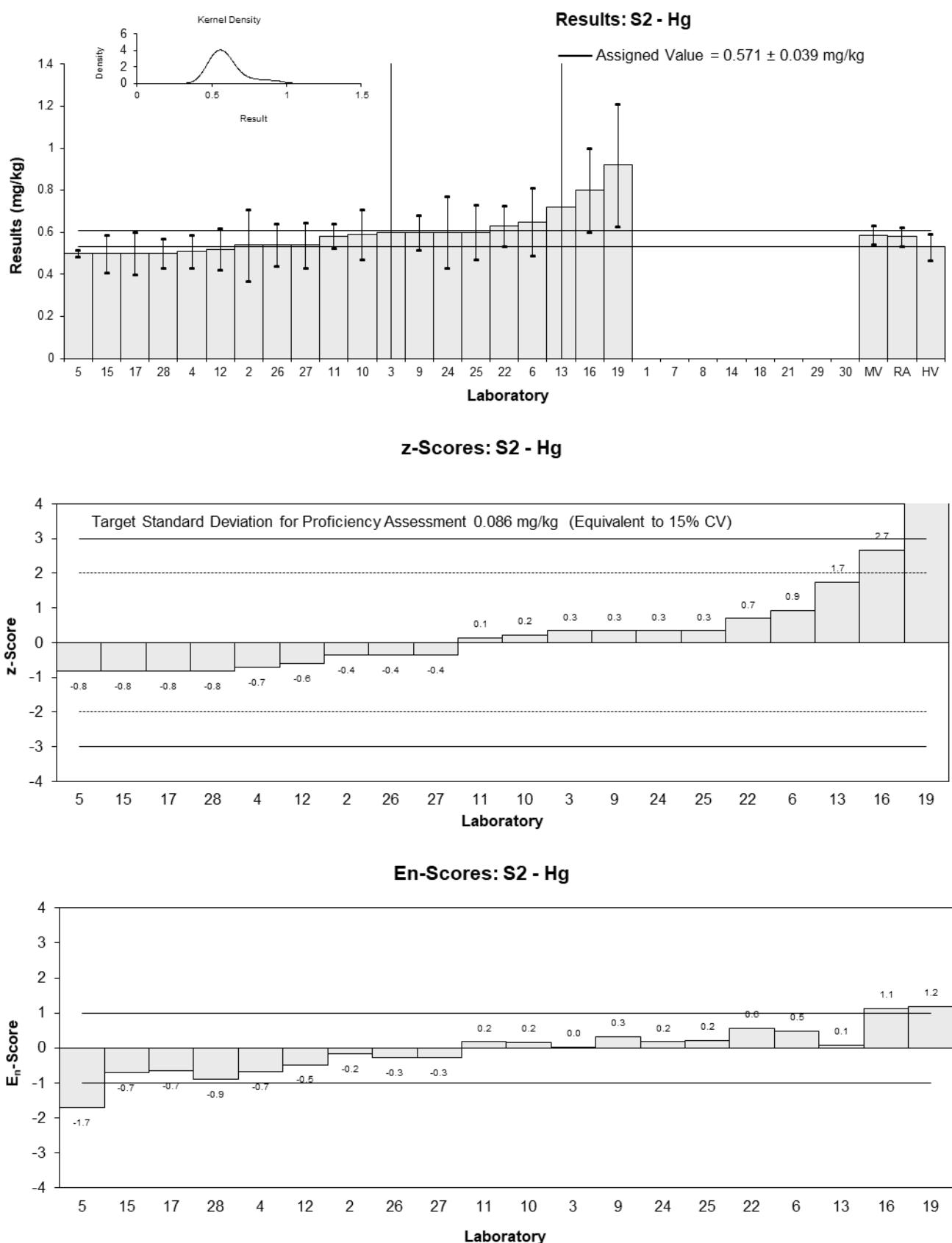


Figure 27

Table 40

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Li
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U
1	NT	NT
2	2.21	0.87
3	2.8	3
4	1.12	0.29
5	NT	NT
6	<5	NT
7	NT	NT
8	NT	NT
9	NT	NT
10	2.42	0.48
11	<5	NT
12	3.11	0.4
13	2.4	3
14	NT	NT
15	1.4	0.29
16	2.4	0.5
17	1.2	0.3
18	NT	NT
19	2.80	1.2
21	NT	NT
22	4	0.6
24	1.8	0.1849
25	<5	1
26	2	1
27	<5	NR
28	1.8	NR
29	NT	1
30	NT	NT

**Statistics**

<b>Assigned Value</b>	Not Set	
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	2.47	0.30
<b>Robust Average</b>	2.20	0.52
<b>Median</b>	2.31	0.50
<b>Mean</b>	2.25	0.42
<b>N</b>	14	
<b>Max</b>	4	
<b>Min</b>	1.12	
<b>Robust SD</b>	0.79	
<b>Robust CV (%)</b>	36	

**Results: S2 - Li**

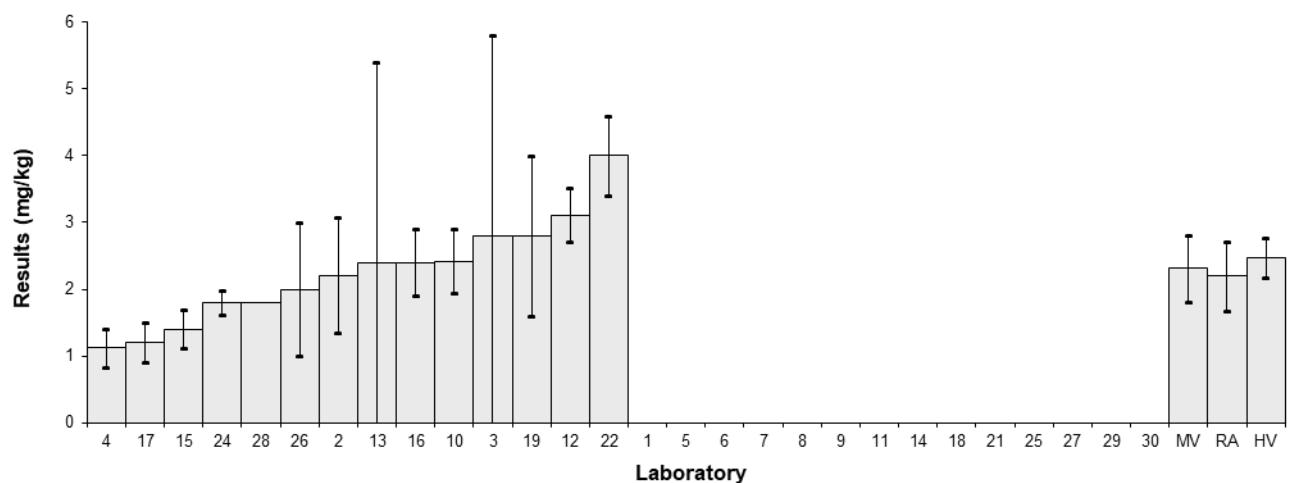


Figure 28

Table 41

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Mn
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	73.6	30.2	1.68	0.35
3	65	21	0.32	0.09
4	56.14	12.49	-1.09	-0.53
5	66	9.9	0.48	0.29
6	64.5	16.1	0.24	0.09
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	61.5	10.7	-0.24	-0.13
11	63.2	6.3	0.03	0.03
12*	114	12	8.10	4.08
13	62	10	-0.16	-0.09
14	NT	NT		
15	58	13	-0.79	-0.37
16	62	10	-0.16	-0.09
17	53.6	9.1	-1.49	-0.96
18	NT	NT		
19	73.5	11	1.67	0.91
21	NT	NT		
22	67	10	0.63	0.38
24	60.2	5.1857	-0.44	-0.45
25	65	14	0.32	0.14
26	65	7	0.32	0.26
27	70.5	14.1	1.19	0.52
28	54.9	8.52	-1.29	-0.88
29	58.5	1	-0.71	-1.24
30	NT	NT		

\* Outlier

**Statistics**

<b>Assigned Value</b>	63.0	3.5
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	61.7	7.7
<b>Robust Average</b>	63.7	3.8
<b>Median</b>	63.9	2.8
<b>Mean</b>	65.7	5.6
<b>N</b>	20	
<b>Max</b>	114	
<b>Min</b>	53.6	
<b>Robust SD</b>	6.8	
<b>Robust CV (%)</b>	11	

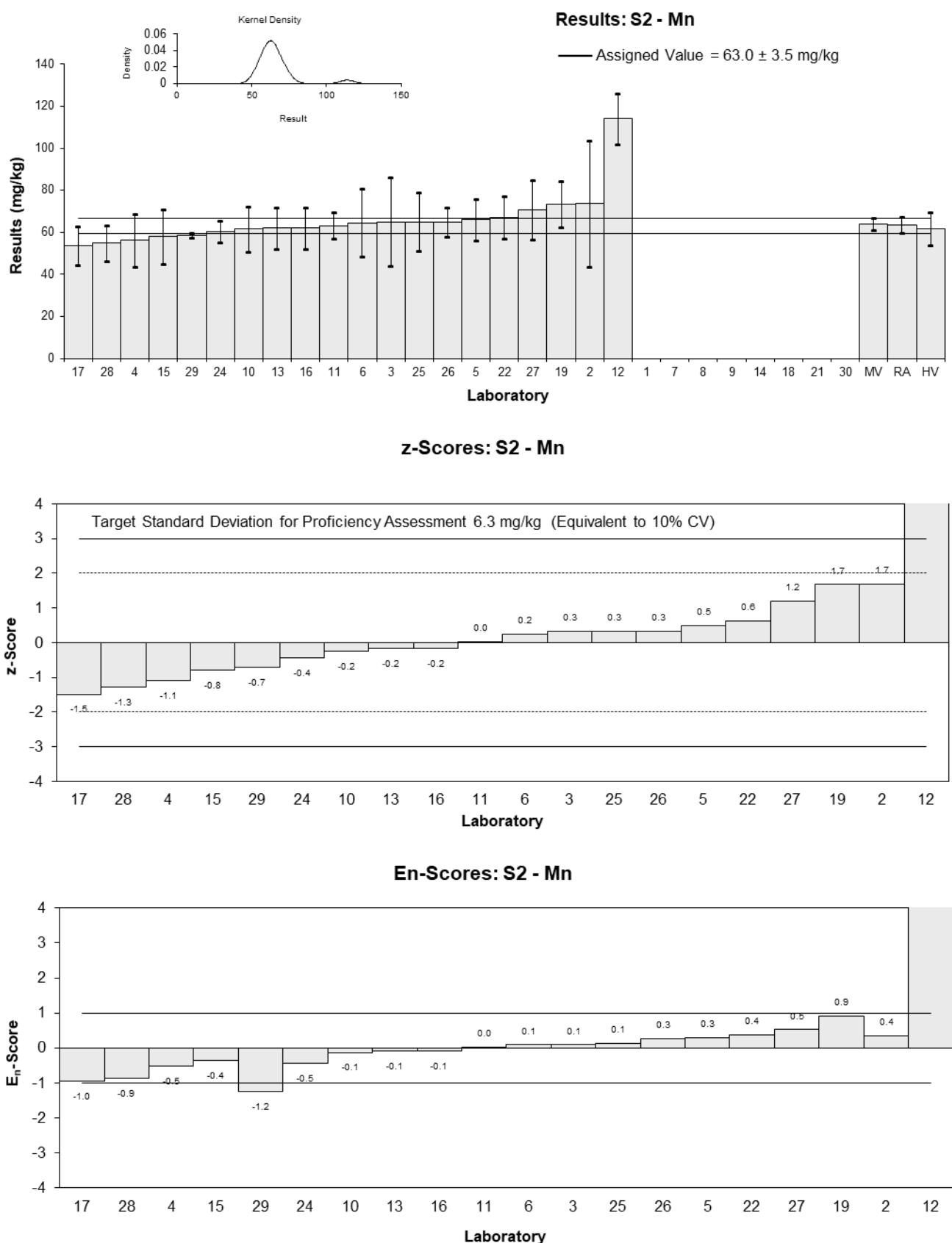


Figure 29

Table 42

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Moisture Content
<b>Matrix</b>	Moist soil
<b>Unit</b>	%

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	19.9	0.7	0.93	1.72
3	17.2	4	-0.55	-0.25
4*	5.1	0.6	-7.20	-14.21
5	18.4	2.944	0.11	0.07
6	20	3.3	0.99	0.53
7	NT	NT		
8	NT	NT		
9	19.0	2.9	0.44	0.27
10	18.7	3.7	0.27	0.13
11	17	NT	-0.66	-1.71
12	18.4	1.0	0.11	0.16
13	18	4	-0.11	-0.05
14	NT	NT		
15	17.3	NR	-0.49	-1.29
16	16	3	-1.21	-0.71
17	16.6	0.8	-0.88	-1.51
18	NT	NT		
19	18.9	3.8	0.38	0.18
21	NT	NT		
22	19	2.9	0.44	0.27
24	18.8	1.771	0.33	0.32
25	18	2.7	-0.11	-0.07
26	18	1	-0.11	-0.16
27	19	3.8	0.44	0.21
28	15.8	NR	-1.32	-3.43
29	19.2	1	0.55	0.82
30	NT	NT		

\* Outlier

**Statistics**

<b>Assigned Value</b>	18.2	0.7
<b>Spike Value</b>	19.6	0.4
<b>Homogeneity Value</b>	19.2	0.4
<b>Robust Average</b>	18.1	0.8
<b>Median</b>	18.4	0.5
<b>Mean</b>	17.5	1.3
<b>N</b>	21	
<b>Max</b>	20	
<b>Min</b>	5.1	
<b>Robust SD</b>	1.4	
<b>Robust CV (%)</b>	7.8	

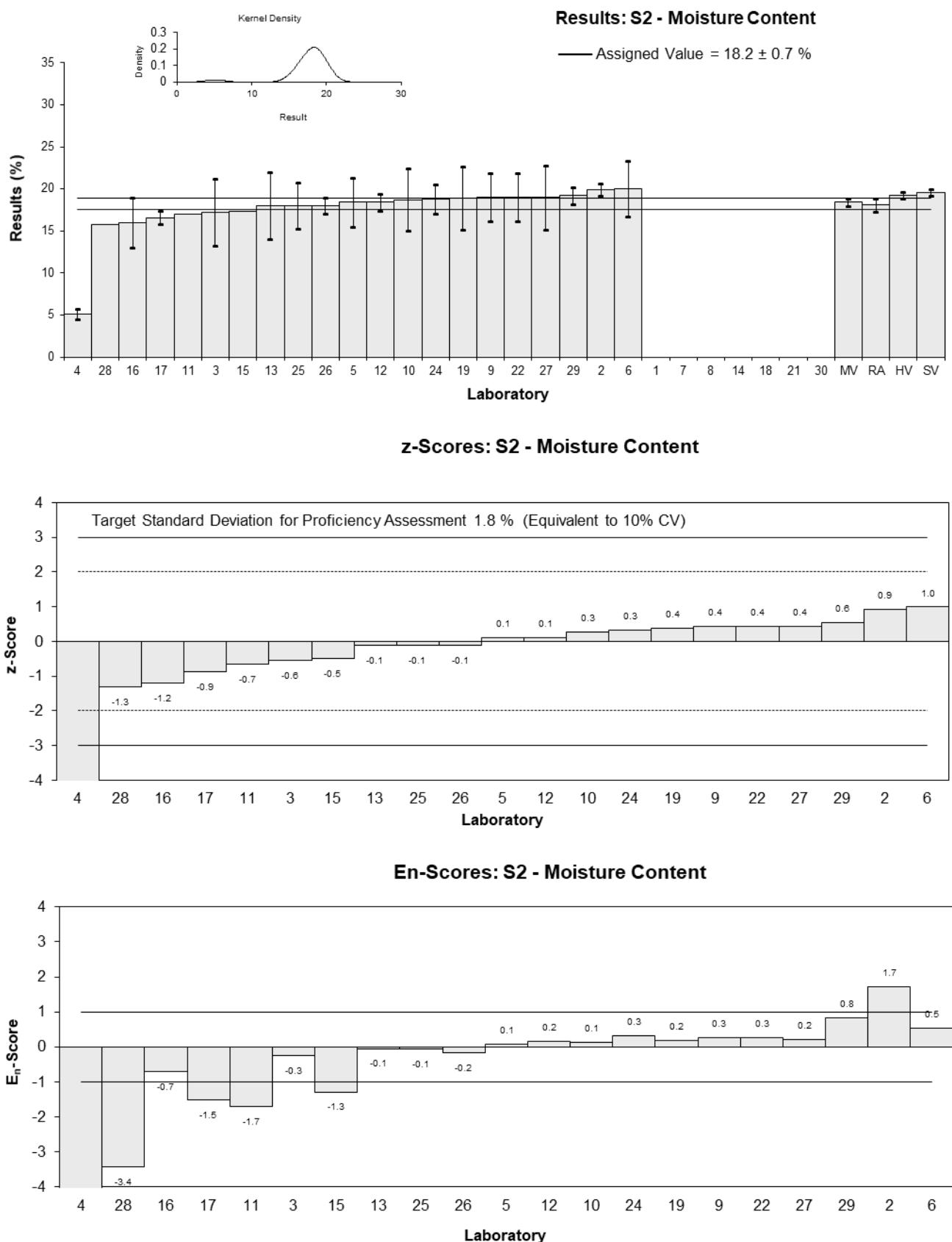


Figure 30

Table 43

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Ni
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	46.0	6.08	0.65	0.44
3	45	18	0.42	0.10
4	40.52	3.98	-0.62	-0.60
5	44	3.96	0.19	0.18
6	37.5	9.38	-1.32	-0.59
7	NT	NT		
8	NT	NT		
9	40	11	-0.74	-0.29
10	46.7	8.2	0.81	0.41
11	45.8	4.8	0.60	0.50
12	43.9	5.0	0.16	0.13
13	40	10	-0.74	-0.31
14	NT	NT		
15	40	10	-0.74	-0.31
16	38	8	-1.20	-0.63
17	38.6	6.3	-1.06	-0.69
18	NT	NT		
19	50.5	15.5	1.69	0.47
21	NT	NT		
22	47	7.1	0.88	0.51
24	43.6	4.2866	0.09	0.08
25	46	9.7	0.65	0.28
26	42	5	-0.28	-0.22
27	46.8	9.36	0.83	0.38
28	39.8	6.75	-0.79	-0.48
29	46.1	1	0.67	1.25
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	43.2	2.1
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	41.3	5.0
<b>Robust Average</b>	43.2	2.1
<b>Median</b>	43.9	2.3
<b>Mean</b>	43.2	1.6
<b>N</b>	21	
<b>Max</b>	50.5	
<b>Min</b>	37.5	
<b>Robust SD</b>	3.9	
<b>Robust CV (%)</b>	9.1	

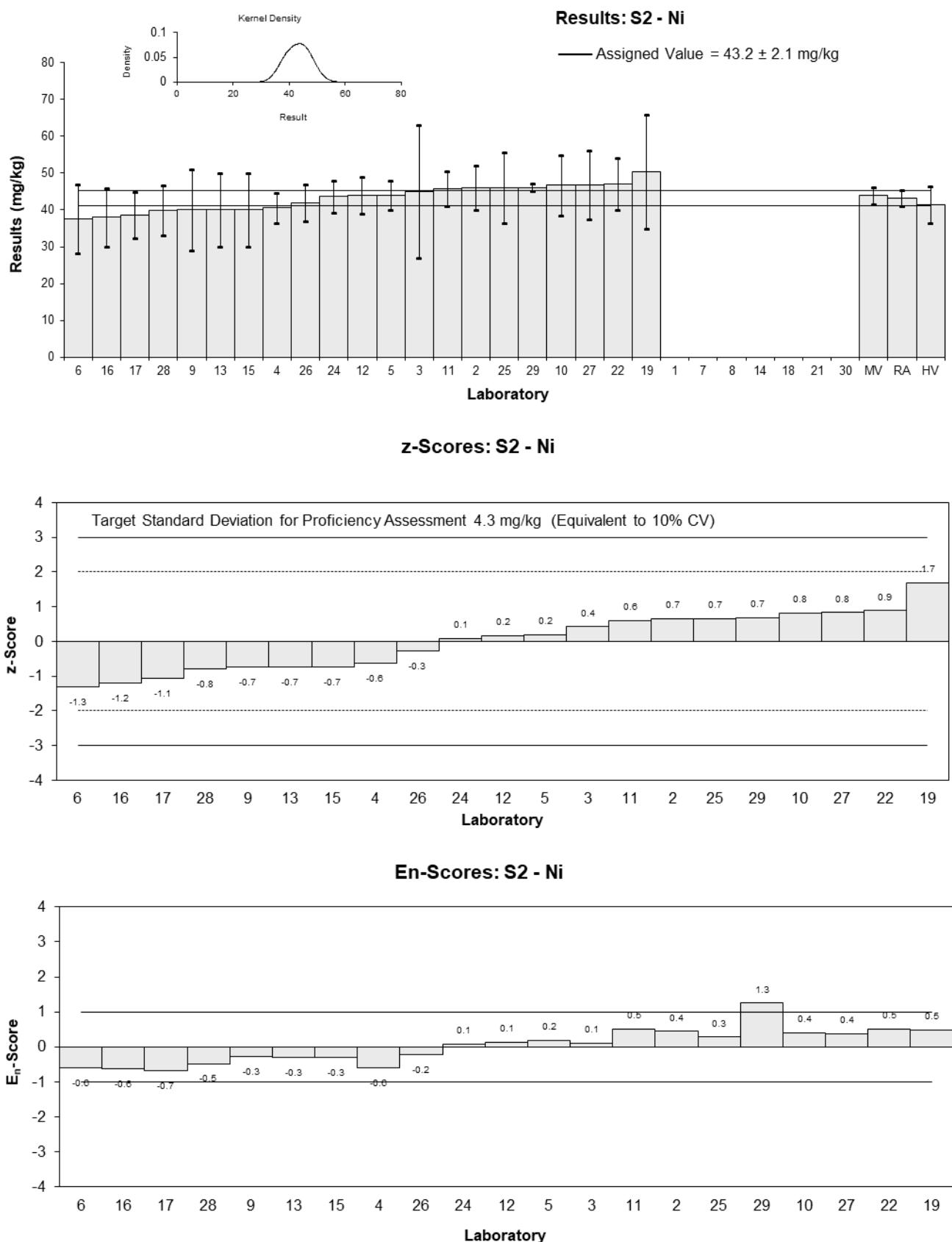


Figure 31

Table 44

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Sb
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	14.2	1.88	0.00	0.00
3	15	5	0.38	0.15
4	16.13	3.82	0.91	0.47
5	14	1.26	-0.09	-0.10
6	15.0	3.74	0.38	0.20
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	11.8	2.1	-1.13	-0.93
11	14.3	1.5	0.05	0.05
12	13.7	1.5	-0.23	-0.24
13	13	10	-0.56	-0.12
14	NT	NT		
15	10	3.4	-1.97	-1.13
16	15	3	0.38	0.24
17	16.3	4.2	0.99	0.47
18	NT	NT		
19	<100	NR		
21	NT	NT		
22	10	1.5	-1.97	-1.98
24	15.4	2.2734	0.56	0.44
25	18	3.8	1.78	0.93
26	NT	NT		
27	14.3	2.86	0.05	0.03
28	18.3	6.44	1.92	0.62
29	11.2	1	-1.41	-1.66
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	14.2	1.5
<b>Spike Value</b>	Not Spiked	
<b>Robust Average</b>	14.2	1.5
<b>Median</b>	14.3	1.0
<b>Mean</b>	14.2	1.1
<b>N</b>	18	
<b>Max</b>	18.3	
<b>Min</b>	10	
<b>Robust SD</b>	2.5	
<b>Robust CV (%)</b>	18	

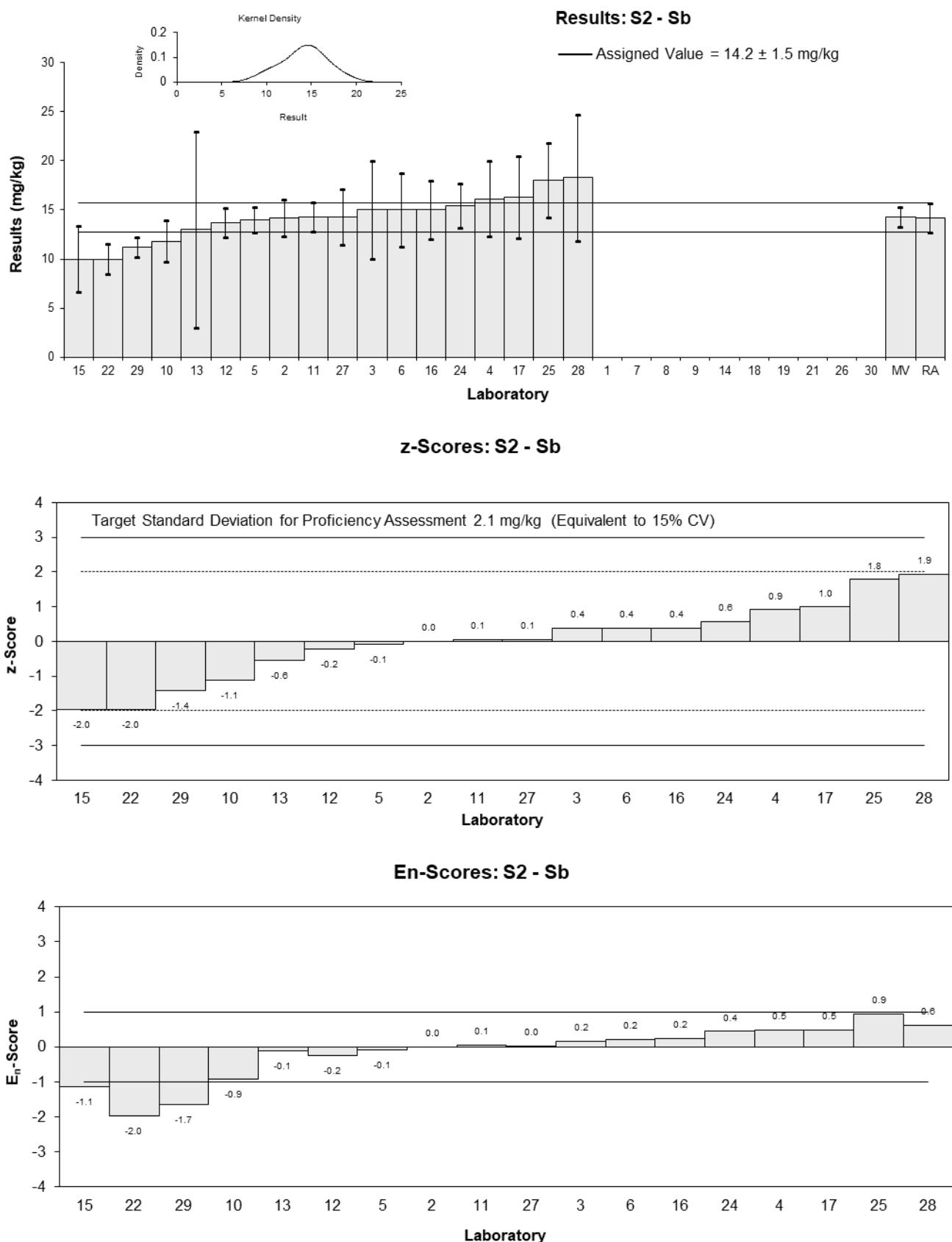


Figure 32

Table 45

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Se
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	2.34	0.43	0.62	0.45
3	2.1	2	-0.12	-0.02
4	2.1	NR	-0.12	-0.31
5	<5	NR		
6	2.2	0.55	0.19	0.11
7	NT	NT		
8	NT	NT		
9	<5.0	NR		
10	2.12	0.41	-0.06	-0.05
11	2.38	0.23	0.75	0.91
12	2.27	0.3	0.40	0.40
13	2.0	2	-0.44	-0.07
14	NT	NT		
15	1.7	0.3	-1.37	-1.35
16	2.5	0.5	1.12	0.70
17	2	0.2	-0.44	-0.59
18	NT	NT		
19	<100	NR		
21	NT	NT		
22*	4	0.6	5.79	3.03
24	2	0.1478	-0.44	-0.71
25	2	0.42	-0.44	-0.32
26	<5	NR		
27	2.19	0.438	0.16	0.11
28	<5	1.46		
29	NT	1		
30	NT	NT		

\* Outlier

**Statistics**

<b>Assigned Value</b>	2.14	0.13
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	2.13	0.26
<b>Robust Average</b>	2.17	0.14
<b>Median</b>	2.12	0.11
<b>Mean</b>	2.26	0.27
<b>N</b>	15	
<b>Max</b>	4	
<b>Min</b>	1.7	
<b>Robust SD</b>	0.22	
<b>Robust CV (%)</b>	10	

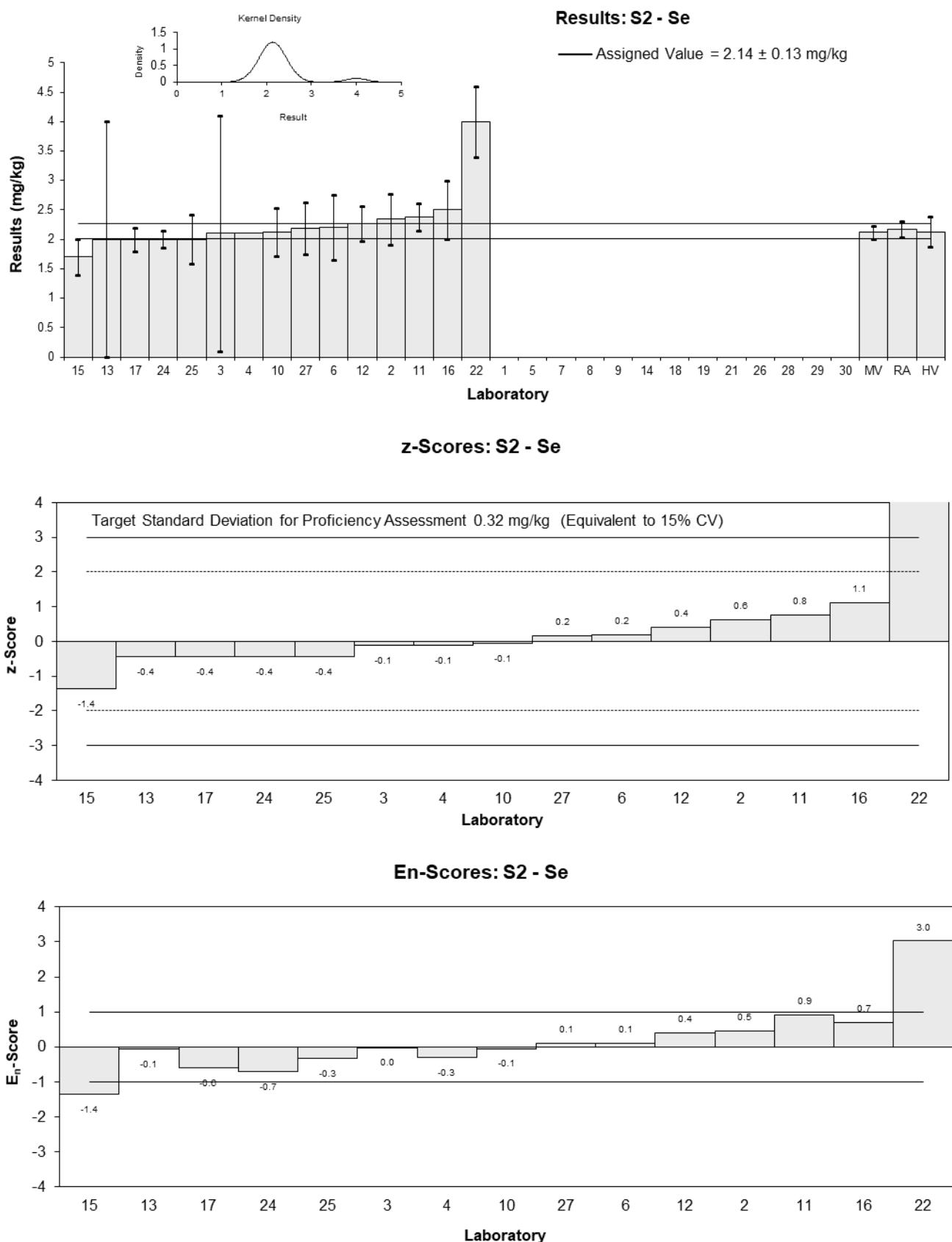


Figure 33

Table 46

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Sr
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	15.1	6.56	1.35	0.27
3	14	4	0.53	0.17
4	11.35	1.33	-1.47	-1.13
5	15	NR	1.28	1.55
6	15.0	3.74	1.28	0.44
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	14.5	2.8	0.90	0.40
11	12.9	3.2	-0.30	-0.12
12	13.6	1.5	0.23	0.16
13	14	2	0.53	0.31
14	NT	NT		
15	11	2.1	-1.73	-0.97
16	12	2	-0.98	-0.57
17	11.7	1.9	-1.20	-0.73
18	NT	NT		
19	NT	NT		
21	NT	NT		
22	10	1.5	-2.48	-1.77
24	12.4	1.234	-0.68	-0.54
25	16	3.4	2.03	0.76
26	13	5	-0.23	-0.06
27	15.7	3.14	1.80	0.72
28	11.8	1.91	-1.13	-0.68
29	12.4	1	-0.68	-0.61
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	13.3	1.1
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	12.7	1.5
<b>Robust Average</b>	13.3	1.1
<b>Median</b>	13.0	1.1
<b>Mean</b>	13.2	0.8
<b>N</b>	19	
<b>Max</b>	16	
<b>Min</b>	10	
<b>Robust SD</b>	1.9	
<b>Robust CV (%)</b>	14	

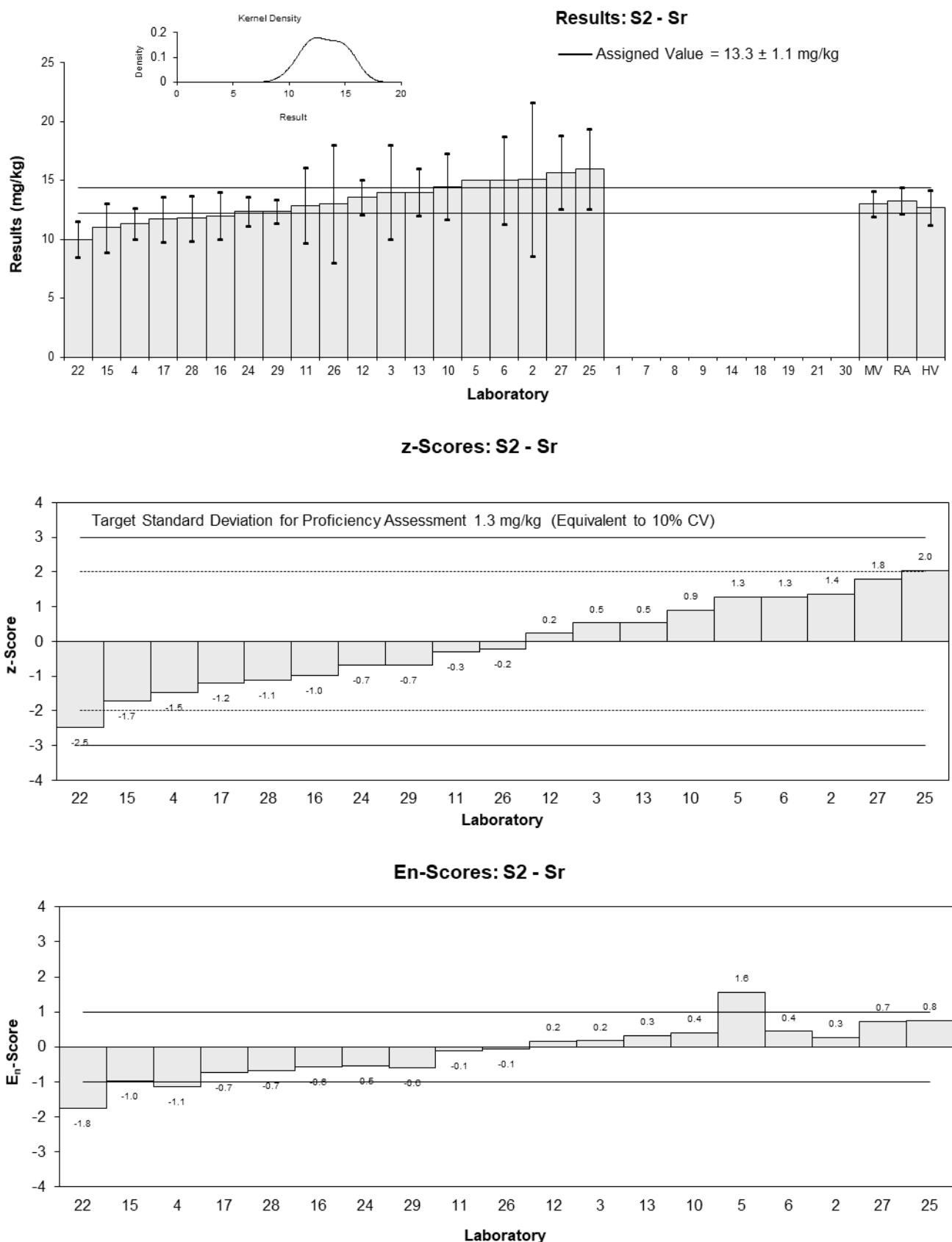


Figure 34

Table 47

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Tl
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	2.62	0.58	2.11	1.01
3	1.8	3	-0.64	-0.06
4	1.21	0.17	-2.61	-2.73
5	1.1	NR	-2.98	-3.87
6	<10	NT		
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	2.26	0.45	0.90	0.53
11	2.15	0.25	0.54	0.47
12	2.14	0.3	0.50	0.40
13	1.7	3	-0.97	-0.10
14	NT	NT		
15	2.1	NR	0.37	0.48
16	1.8	0.4	-0.64	-0.41
17	2.2	0.2	0.70	0.69
18	NT	NT		
19	NT	NT		
21	NT	NT		
22*	4	.6	6.73	3.13
24	2.2	0.1395	0.70	0.78
25	<10	2		
26	2	1	0.03	0.01
27	<10	NR		
28	2.09	0.41	0.34	0.21
29	NT	1		
30	NT	NT		

\* Outlier

**Statistics**

<b>Assigned Value</b>	1.99	0.23
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	1.94	0.24
<b>Robust Average</b>	2.04	0.27
<b>Median</b>	2.10	0.15
<b>Mean</b>	2.09	0.34
<b>N</b>	15	
<b>Max</b>	4	
<b>Min</b>	1.1	
<b>Robust SD</b>	0.41	
<b>Robust CV (%)</b>	20	

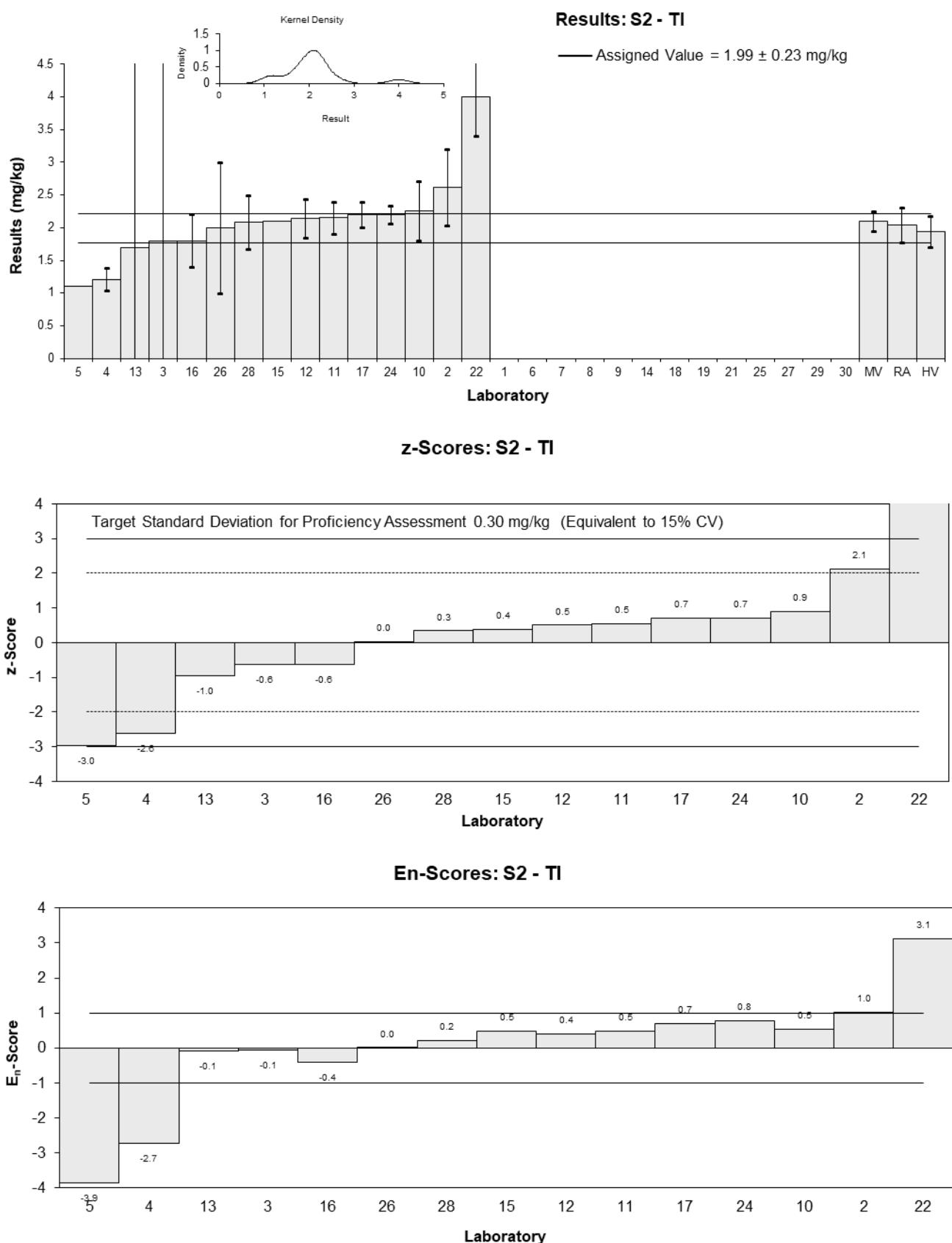


Figure 35

Table 48

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	U
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	5.28	0.88	1.35	0.66
3	5	2	0.75	0.17
4	3.92	0.45	-1.57	-1.24
5	NT	NT		
6	<10	NT		
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	4.48	0.90	-0.37	-0.17
11	<10	NT		
12	4.40	0.5	-0.54	-0.40
13	5.1	1	0.97	0.42
14	NT	NT		
15	4.4	0.67	-0.54	-0.32
16	5.6	1	2.04	0.89
17	4.9	1.2	0.54	0.20
18	NT	NT		
19	NT	NT		
21	NT	NT		
22	4.0	1.1	-1.40	-0.56
24	4.8	0.3860	0.32	0.28
25	4.4	0.92	-0.54	-0.25
26	NT	NT		
27	<10	NR		
28	4.28	NR	-0.80	-0.97
29	NT	1		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	4.65	0.38
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	4.33	0.52
<b>Robust Average</b>	4.65	0.38
<b>Median</b>	4.48	0.43
<b>Mean</b>	4.66	0.28
<b>N</b>	13	
<b>Max</b>	5.6	
<b>Min</b>	3.92	
<b>Robust SD</b>	0.55	
<b>Robust CV (%)</b>	12	

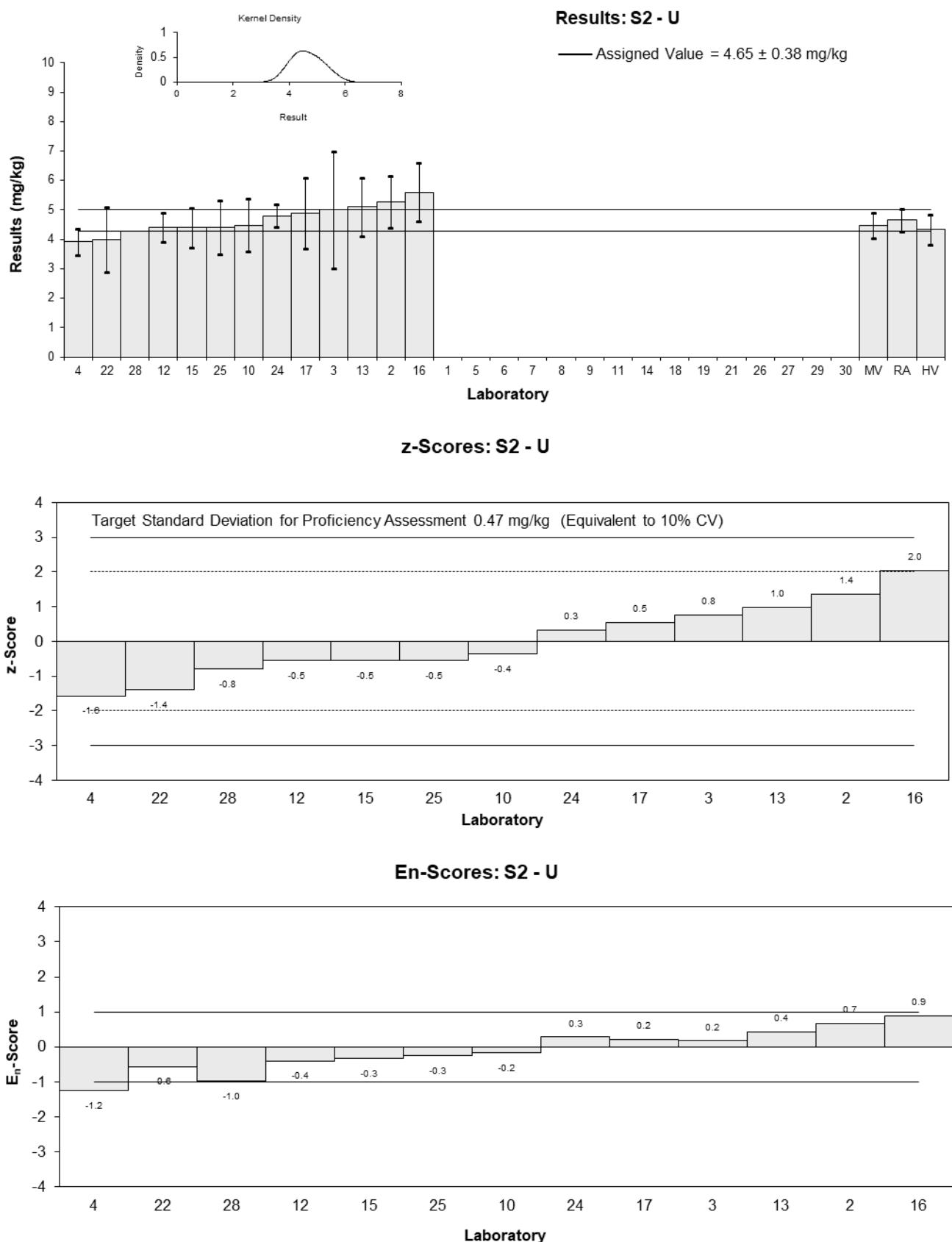


Figure 36

Table 49

**Sample Details**

<b>Sample</b>	S2
<b>Analyte</b>	Zn
<b>Matrix</b>	Moist soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	NT	NT		
2	64.6	24	1.56	0.36
3	56	18	0.02	0.01
4	52.6	6.52	-0.59	-0.47
5	54	3.24	-0.34	-0.46
6	57.8	14.4	0.34	0.13
7	NT	NT		
8	NT	NT		
9	53	13	-0.52	-0.22
10	59.2	8.2	0.59	0.38
11	56.6	7.0	0.13	0.09
12	54.9	6.0	-0.18	-0.15
13	50	10	-1.06	-0.57
14	NT	NT		
15	54	8.2	-0.34	-0.22
16	45	9	-1.95	-1.17
17	53.5	9.1	-0.43	-0.25
18	NT	NT		
19	62.0	12.4	1.09	0.48
21	NT	NT		
22	57	8.6	0.20	0.12
24	55.8	4.6253	-0.02	-0.02
25	57	12	0.20	0.09
26	61	8	0.91	0.61
27	64.5	12.9	1.54	0.65
28	49.62	8.36	-1.12	-0.72
29	55.2	1	-0.13	-0.26
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	55.9	2.5
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	55.5	6.7
<b>Robust Average</b>	55.9	2.5
<b>Median</b>	55.8	1.9
<b>Mean</b>	55.9	2.1
<b>N</b>	21	
<b>Max</b>	64.6	
<b>Min</b>	45	
<b>Robust SD</b>	4.6	
<b>Robust CV (%)</b>	8.3	

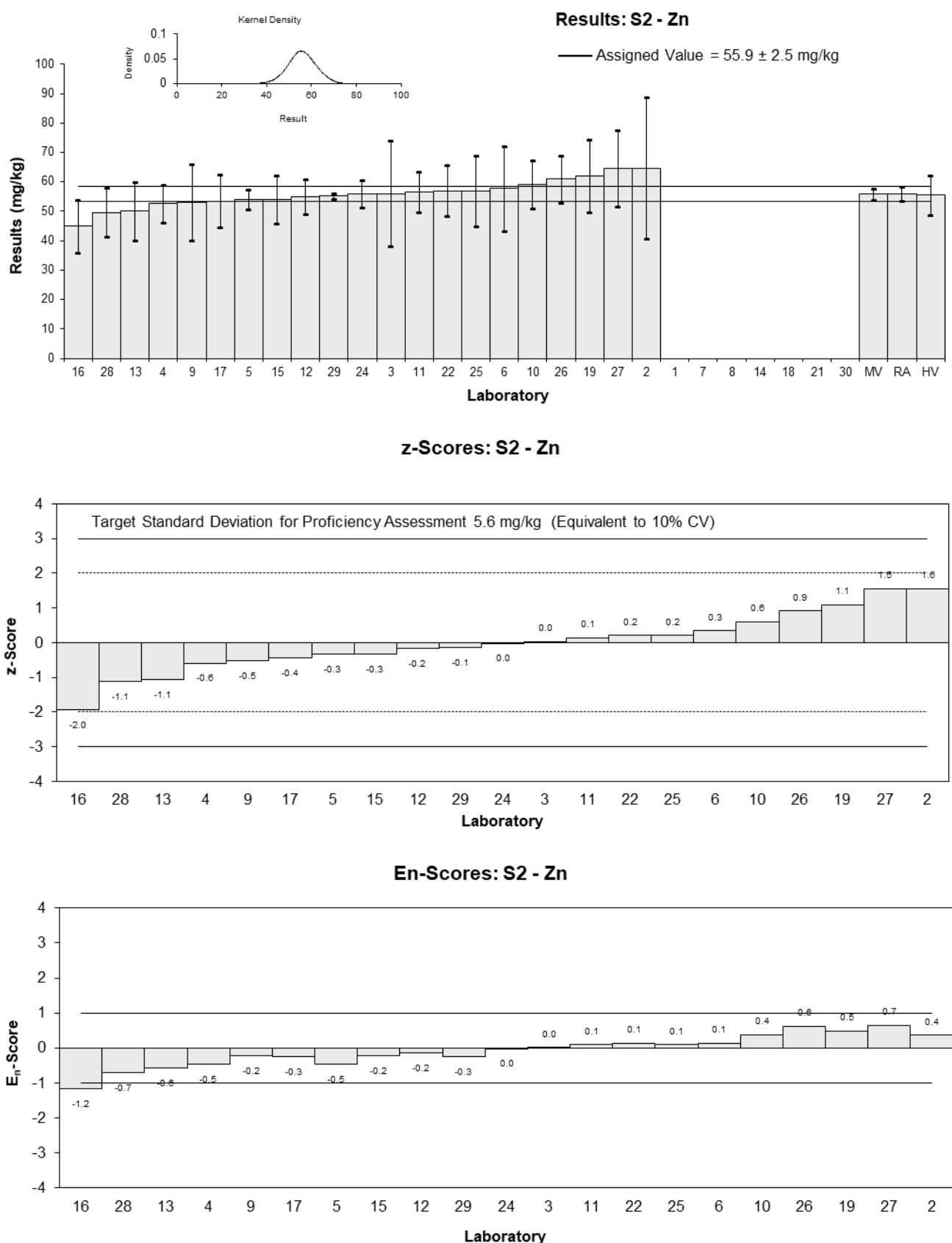


Figure 37

Table 50

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	Ca
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	11000	630	0.50	0.74
2	NT	NT		
3	9300	3000	-0.35	-0.22
4	10600	1922	0.30	0.26
5	12000	1440	1.00	1.07
6	NT	NT		
7	NR	NR		
8	13600	2600	1.80	1.26
9	NT	NT		
10	11500	2040	0.75	0.63
11	NT	NT		
12	11142	1120	0.57	0.70
13	7200	2000	-1.40	-1.20
14	8820	970	-0.59	-0.76
15	7900	1700	-1.05	-1.01
16	7500	2000	-1.25	-1.07
17	8150	1600	-0.92	-0.92
18	NT	NT		
19	12800	1920	1.40	1.24
21	NT	NT		
22	8400	1260	-0.80	-0.92
24	9130	1409	-0.44	-0.47
25	NT	NT		
26	11000	790	0.50	0.70
27	11000	2200	0.50	0.40
28	9920.6	1892.9	-0.04	-0.04
29	NT	NT		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	10000	1200
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	11100	1300
<b>Robust Average</b>	10000	1200
<b>Median</b>	10300	1200
<b>Mean</b>	10100	900
<b>N</b>	18	
<b>Max</b>	13600	
<b>Min</b>	7200	
<b>Robust SD</b>	2000	
<b>Robust CV (%)</b>	20	

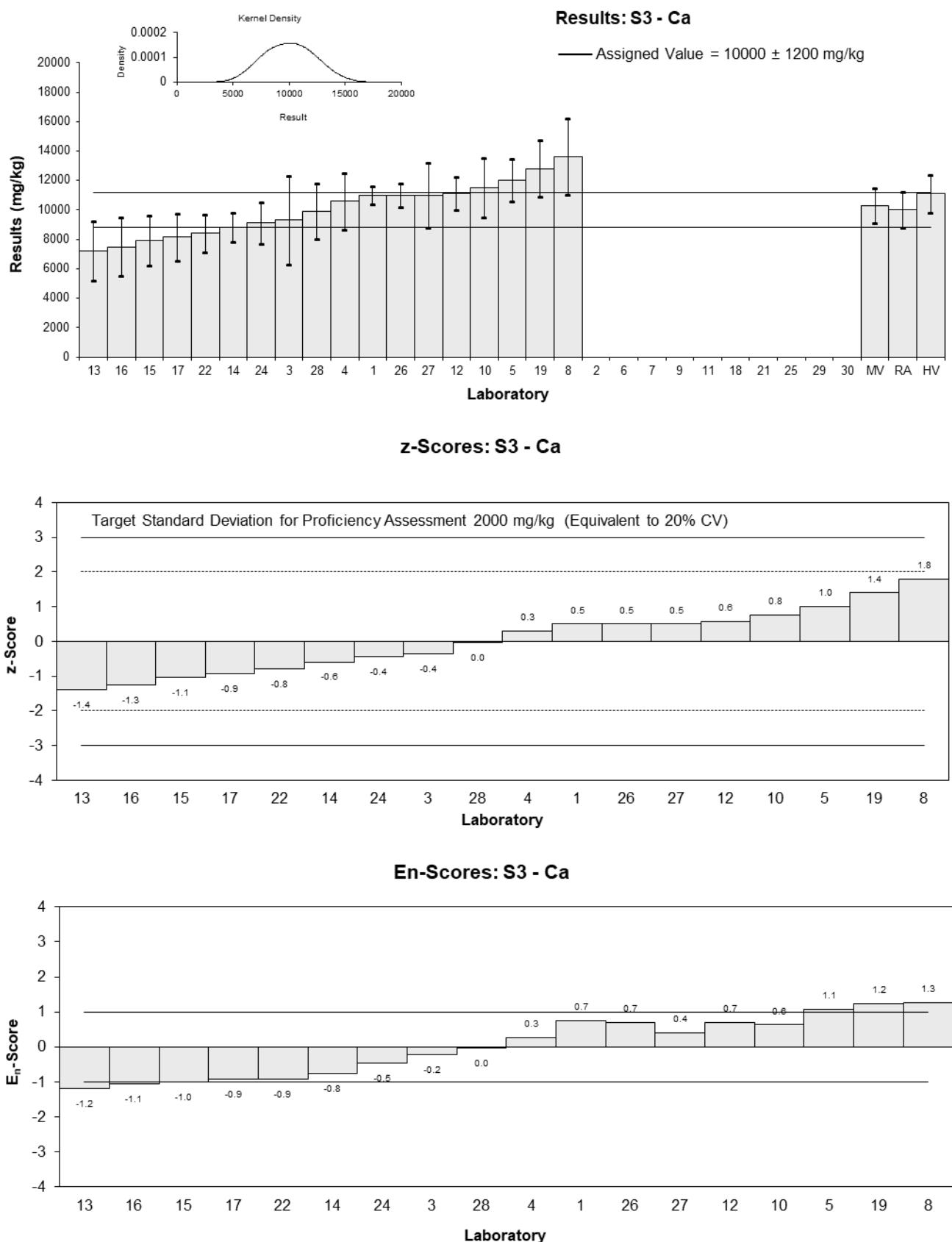


Figure 38

Table 51

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	Colwell K
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U
1	217	43
2	NT	NT
3	NT	NT
4	NT	NR
5	NT	NT
6	NT	NT
7	NR	NR
8	NT	NT
9	NT	NT
10	NR	NR
11	NT	NT
12	118	15
13	NT	NT
14	NT	NT
15	NR	NR
16	NT	NT
17	369	111
18	NT	NT
19	NT	NT
21	NT	NT
22	NT	NT
24	NR	NR
25	NT	NT
26	NT	NT
27	NT	NT
28	NR	NR
29	NT	NT
30	NT	NT

**Statistics**

<b>Assigned Value</b>	Not Set	
<b>Spike Value</b>	Not Spiked	
<b>Median</b>	220	210
<b>Mean</b>	230	150
<b>N</b>	3	
<b>Max</b>	369	
<b>Min</b>	118	

**Results: S3 - Colwell K**

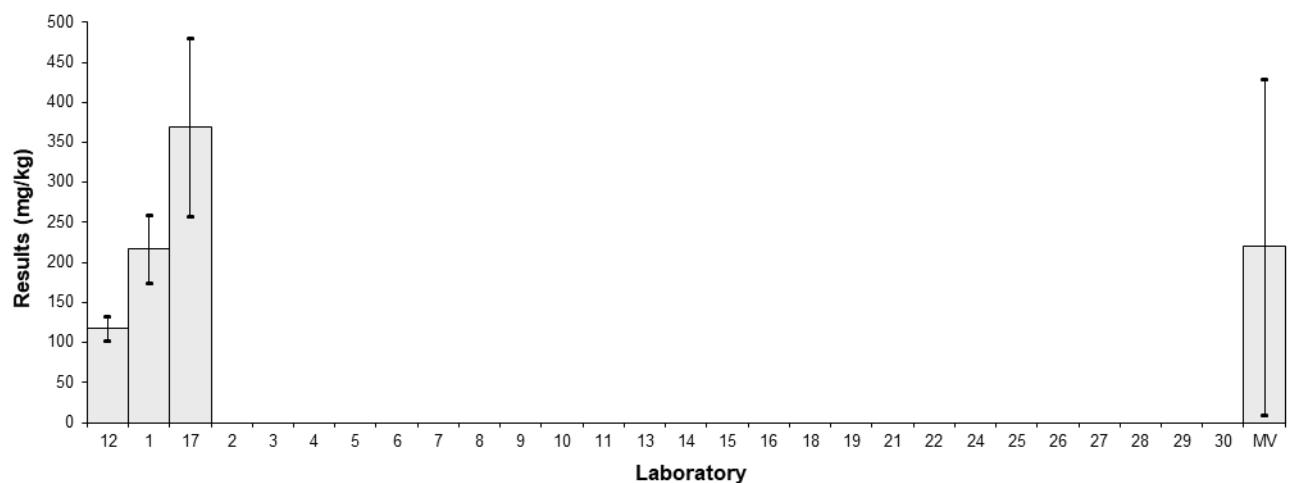


Figure 39

Table 52

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	Colwell P
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	45	6.7	0.03	0.02
2	NT	NT		
3	NT	NT		
4	NT	NR		
5	NT	NT		
6	NT	NT		
7	50	6.0	0.77	0.54
8	NT	NT		
9	NT	NT		
10	39	8	-0.86	-0.53
11	NT	NT		
12	47	5.0	0.33	0.24
13	NT	NT		
14	NT	NT		
15	NR	NR		
16	NT	NT		
17	45	11	0.03	0.01
18	33	2.9	-1.76	-1.45
19	54.5	16.4	1.44	0.54
21	NT	NT		
22	NT	NT		
24	NR	NR		
25	NT	NT		
26	NT	NT		
27	NT	NT		
28	NR	NR		
29	NT	NT		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	44.8	7.6
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	41.3	4.9
<b>Robust Average</b>	44.8	7.6
<b>Median</b>	45.0	7.0
<b>Mean</b>	44.8	5.3
<b>N</b>	7	
<b>Max</b>	54.5	
<b>Min</b>	33	
<b>Robust SD</b>	8.0	
<b>Robust CV (%)</b>	18	

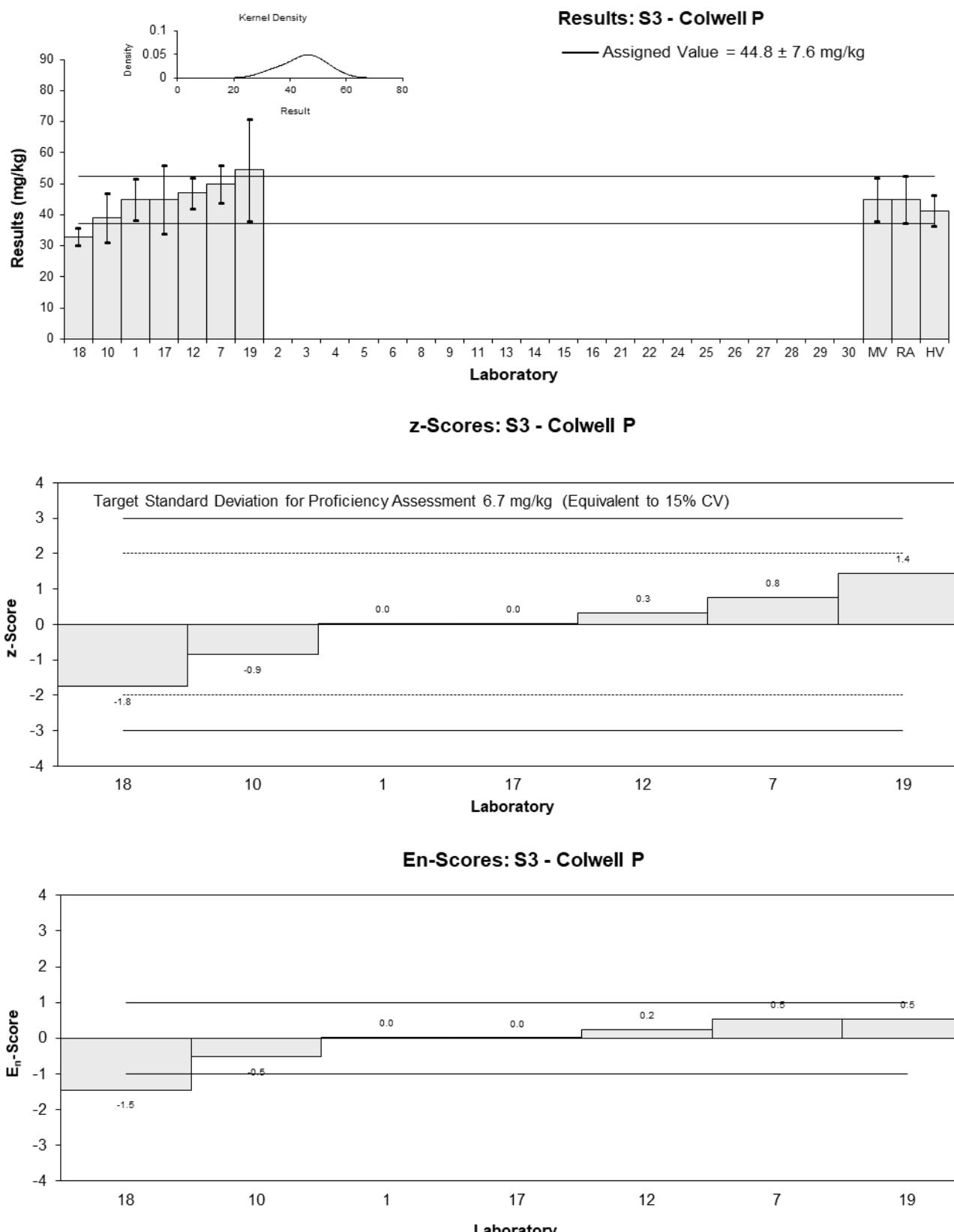


Figure 40

Table 53

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	EC
<b>Matrix</b>	Soil
<b>Unit</b>	µS/cm

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	41.8	4.2	-1.52	-1.36
2	NT	NT		
3	48	20	-0.26	-0.06
4	69	2.2	4.00	4.67
5	NT	NT		
6	NT	NT		
7	47	NR	-0.47	-0.64
8	45	9	-0.87	-0.44
9	NT	NT		
10	45	9	-0.87	-0.44
11	NT	NT		
12	47.7	5.0	-0.32	-0.26
13	48	15	-0.26	-0.08
14	54.2	5.0	0.99	0.80
15	NR	NR		
16	64	10	2.98	1.38
17	54	2	0.95	1.14
18	47	3.008	-0.47	-0.49
19	50	2.5	0.14	0.16
21	NT	NT		
22**	580	87	107.65	6.09
24	56	3.18	1.36	1.39
25	37.7	14.291	-2.35	-0.79
26	46	NR	-0.67	-0.92
27	46.4	13.9	-0.59	-0.20
28	54.4	5.44	1.03	0.78
29	NT	NT		
30	NT	NT		

\*\* Gross Error

**Statistics**

<b>Assigned Value</b>	49.3	3.6
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	42.8	5.1
<b>Robust Average</b>	49.3	3.6
<b>Median</b>	47.9	2.5
<b>Mean</b>	50.1	3.6
<b>N</b>	18	
<b>Max</b>	69	
<b>Min</b>	37.7	
<b>Robust SD</b>	6.2	
<b>Robust CV (%)</b>	13	

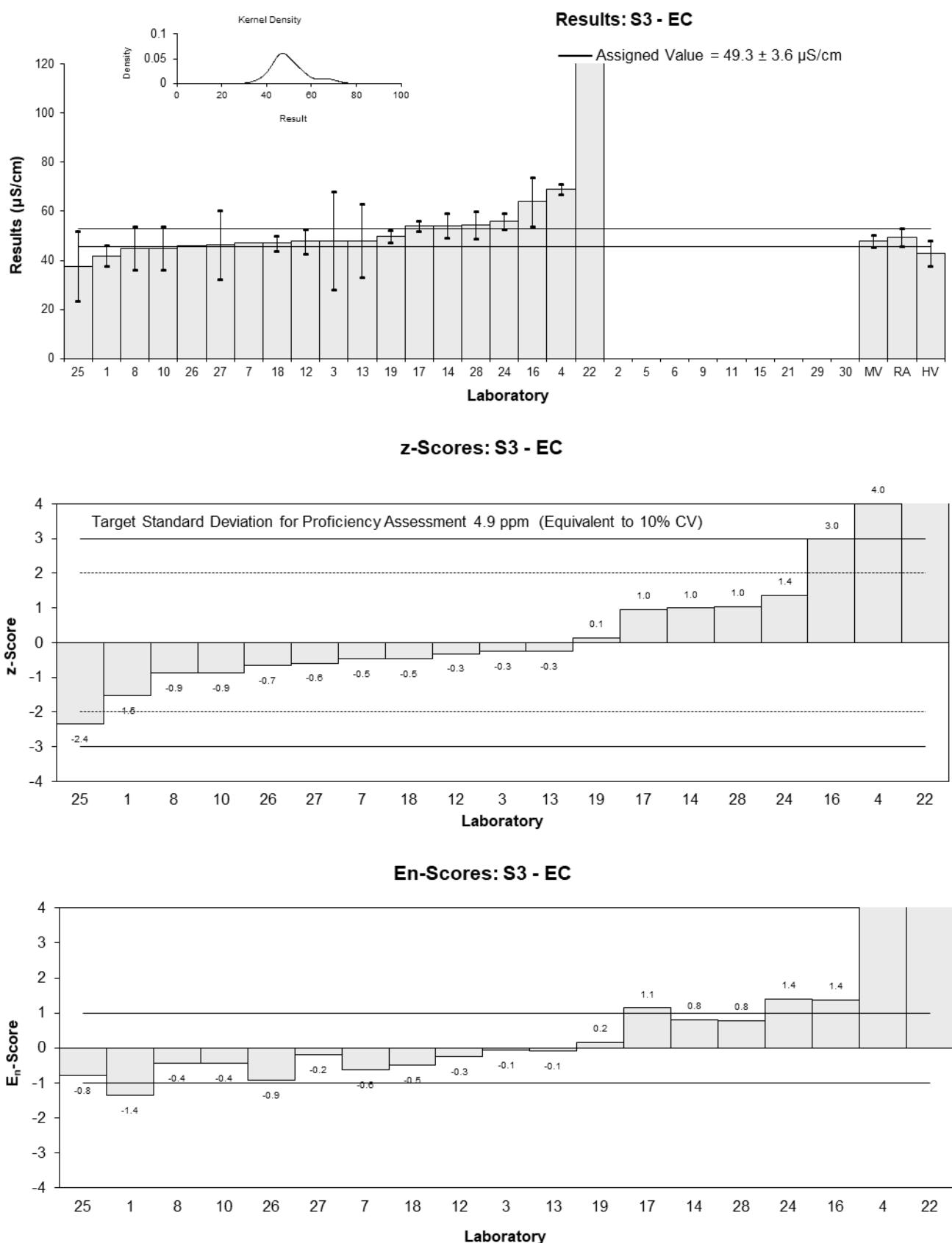


Figure 41

Table 54

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	Exchangeable Ca
<b>Matrix</b>	Soil
<b>Unit</b>	cmol(+)/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	25.4	2.9	0.54	0.42
2	NT	NT		
3	24	8	-0.04	-0.01
4	24.2	2.8	0.04	0.03
5	24	1.2	-0.04	-0.06
6	NT	NT		
7	25.7	0.56	0.66	1.30
8	NT	NT		
9	NT	NT		
10	23	4	-0.46	-0.27
11	NT	NT		
12	21.4	2.5	-1.12	-0.99
13	24	5	-0.04	-0.02
14	25.3	3.9	0.50	0.30
15	NR	NR		
16	22	4	-0.87	-0.51
17	17.1	5.5	-2.90	-1.25
18	26.0	1.9	0.79	0.87
19	24.7	4.9	0.25	0.12
21	NT	NT		
22	NT	NT		
24	23.6	4.035	-0.21	-0.12
25	NT	NT		
26	NT	NT		
27	26.7	5.34	1.08	0.48
28	24.0	NR	-0.04	-0.09
29	NT	NT		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	24.1	1.1
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	25.5	3.1
<b>Robust Average</b>	24.1	1.1
<b>Median</b>	24.0	1.1
<b>Mean</b>	23.8	1.1
<b>N</b>	16	
<b>Max</b>	26.7	
<b>Min</b>	17.1	
<b>Robust SD</b>	1.8	
<b>Robust CV (%)</b>	7.3	

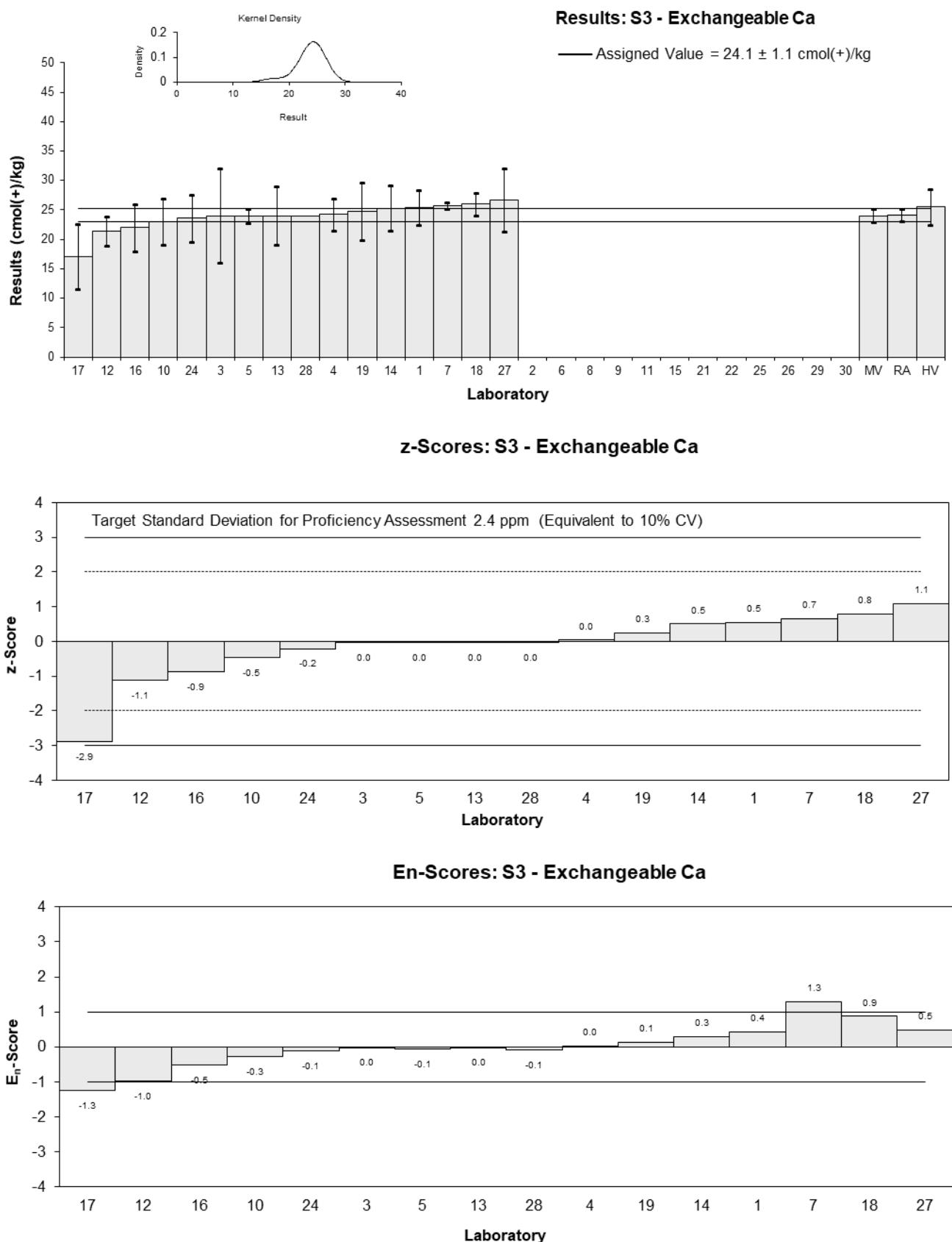


Figure 42

Table 55

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	Exchangeable K
<b>Matrix</b>	Soil
<b>Unit</b>	cmol(+)/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	0.44	0.07	0.38	0.32
2	NT	NT		
3	0.41	1	-0.10	-0.01
4	0.47	0.06	0.87	0.83
5	0.4	0.012	-0.26	-0.56
6	NT	NT		
7	0.40	0.07	-0.26	-0.21
8	NT	NT		
9	NT	NT		
10	0.38	0.06	-0.58	-0.55
11	NT	NT		
12	0.37	0.06	-0.74	-0.70
13	0.41	2	-0.10	0.00
14	0.469	0.093	0.85	0.55
15	NR	NR		
16	0.40	0.08	-0.26	-0.19
17	0.3	0.1	-1.86	-1.12
18	0.44	0.015	0.38	0.80
19	0.39	0.14	-0.42	-0.18
21	NT	NT		
22	NT	NT		
24	0.5	0.128	1.35	0.64
25	NT	NT		
26	NT	NT		
27	0.441	0.088	0.40	0.27
28	0.4	NR	-0.26	-0.62
29	NT	NT		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	0.416	0.026
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	0.413	0.050
<b>Robust Average</b>	0.416	0.026
<b>Median</b>	0.405	0.028
<b>Mean</b>	0.414	0.023
<b>N</b>	16	
<b>Max</b>	0.5	
<b>Min</b>	0.3	
<b>Robust SD</b>	0.042	
<b>Robust CV (%)</b>	10	

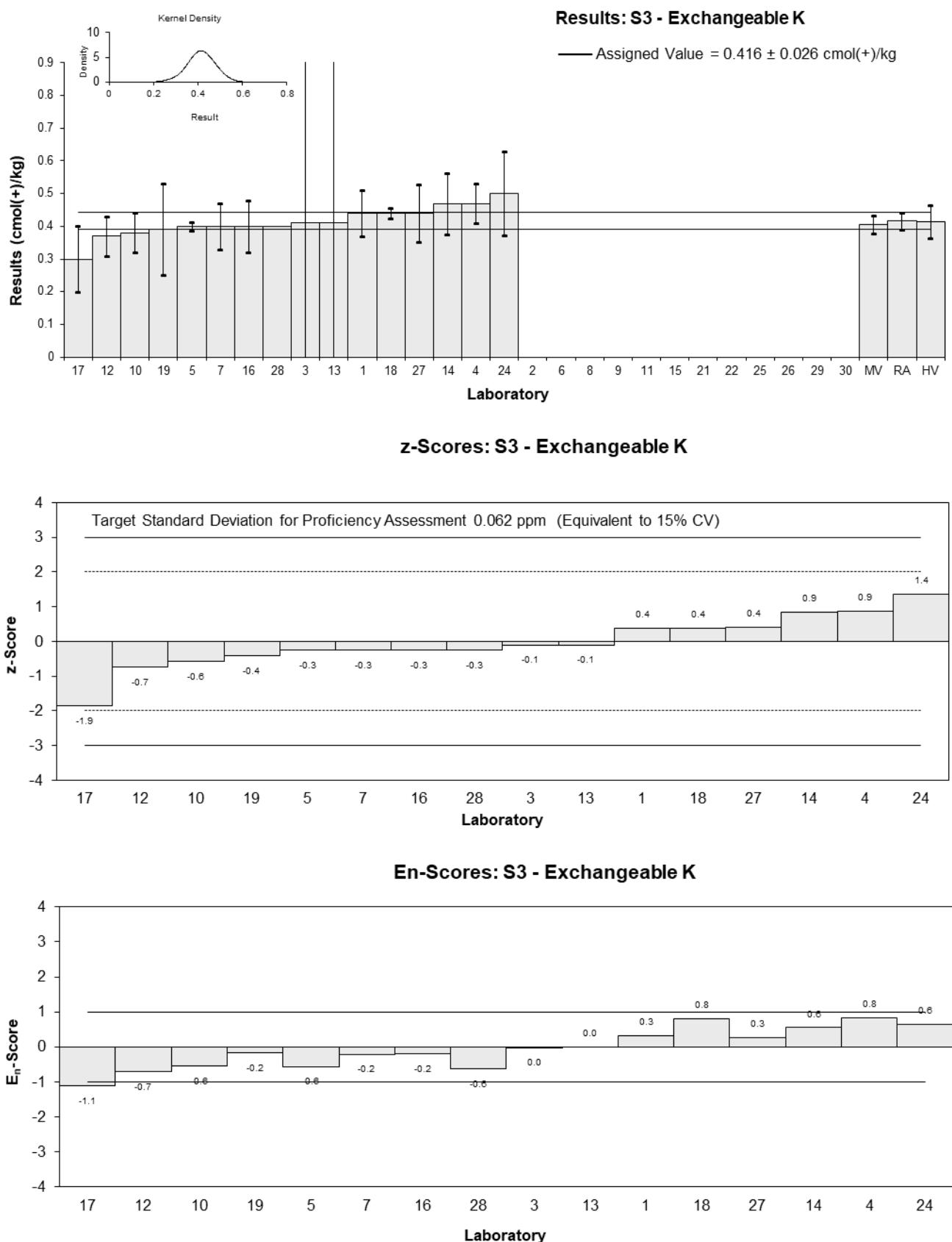


Figure 43

Table 56

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	Exchangeable Mg
<b>Matrix</b>	Soil
<b>Unit</b>	cmol(+)/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	10.6	0.87	0.78	0.74
2	NT	NT		
3	9.2	3	-0.64	-0.21
4	10.31	1.6	0.49	0.28
5	10	0.8	0.17	0.17
6	NT	NT		
7	10.3	0.34	0.48	0.72
8	NT	NT		
9	NT	NT		
10	9.9	1.5	0.07	0.04
11	NT	NT		
12	8.32	1.0	-1.54	-1.32
13	9.5	3	-0.34	-0.11
14	9.62	1.1	-0.21	-0.17
15	NR	NR		
16	8.8	2	-1.05	-0.50
17	5.6	1.5	-4.30	-2.64
18	10.8	0.32	0.99	1.50
19	11.0	2.2	1.19	0.52
21	NT	NT		
22	NT	NT		
24	9.7	1.424	-0.13	-0.08
25	NT	NT		
26	NT	NT		
27	10.7	2.14	0.89	0.39
28	9.9	NR	0.07	0.13
29	NT	NT		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	9.83	0.56
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	10.2	1.2
<b>Robust Average</b>	9.83	0.56
<b>Median</b>	9.90	0.51
<b>Mean</b>	9.64	0.65
<b>N</b>	16	
<b>Max</b>	11	
<b>Min</b>	5.6	
<b>Robust SD</b>	0.90	
<b>Robust CV (%)</b>	9.1	

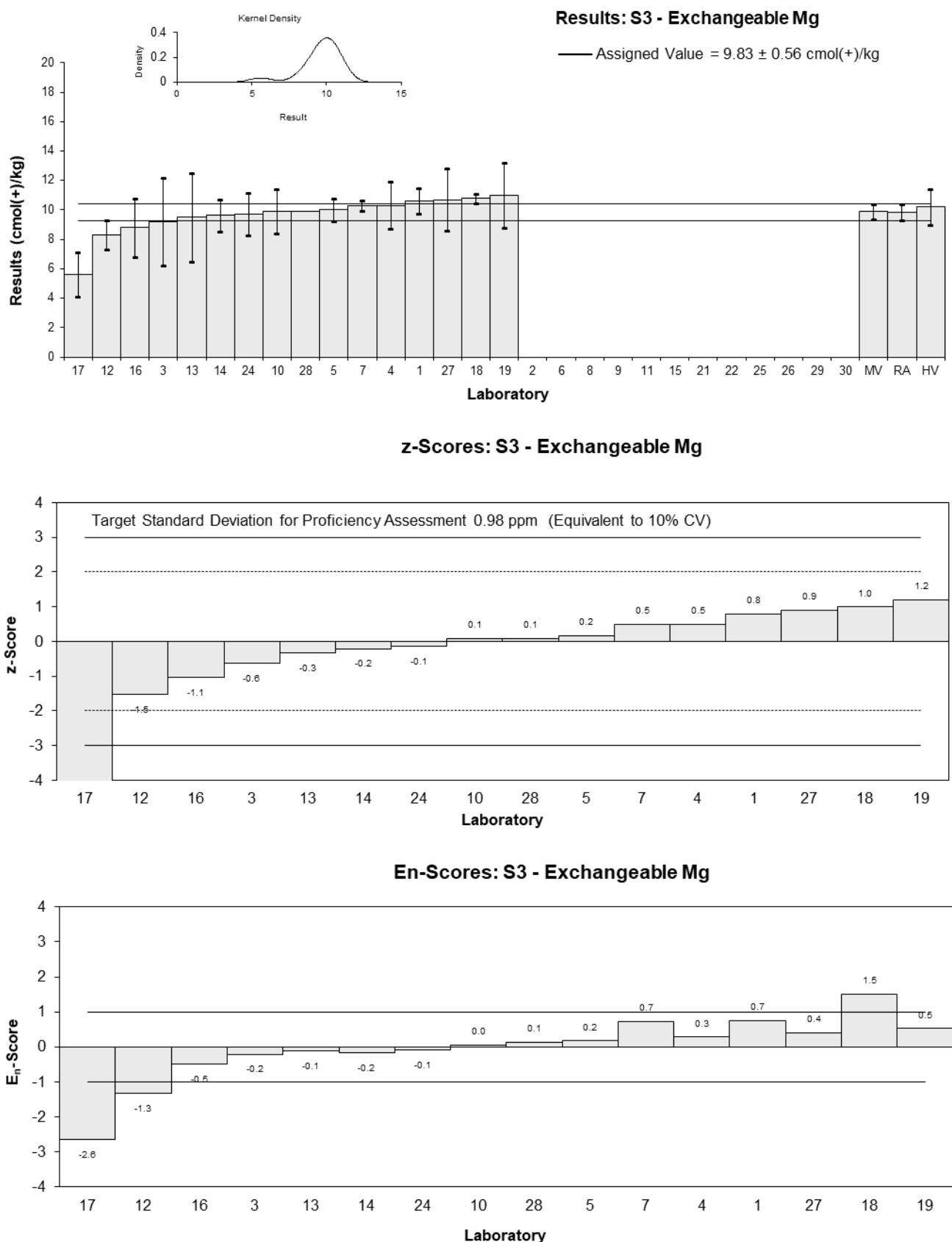


Figure 44

Table 57

**Sample Details**

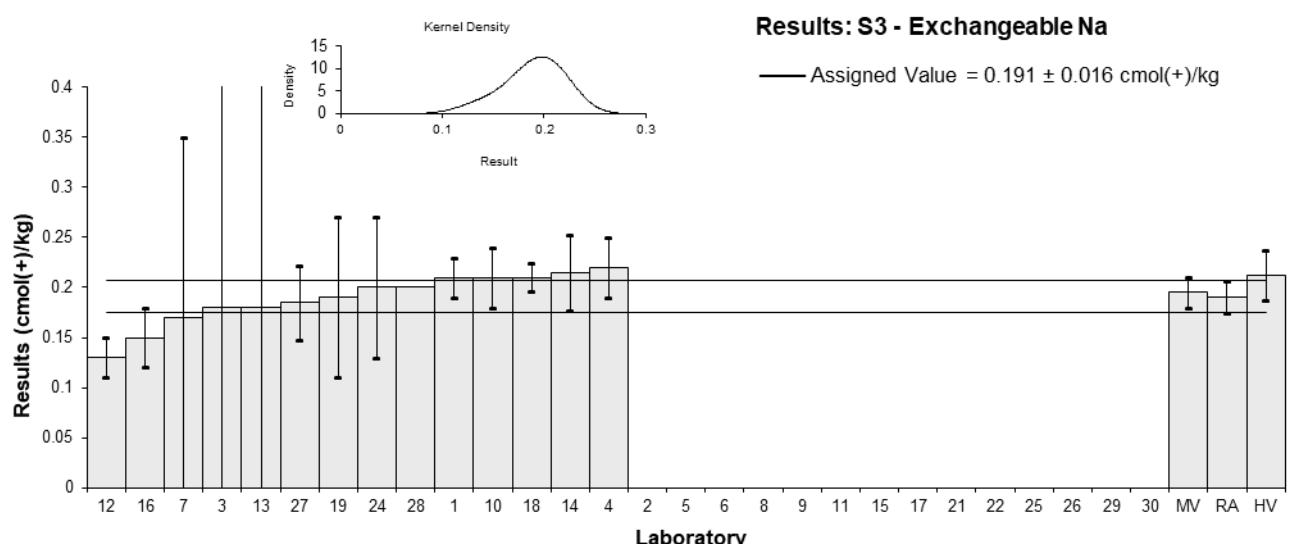
<b>Sample</b>	S3
<b>Analyte</b>	Exchangeable Na
<b>Matrix</b>	Soil
<b>Unit</b>	cmol(+)/kg

**Participant Results**

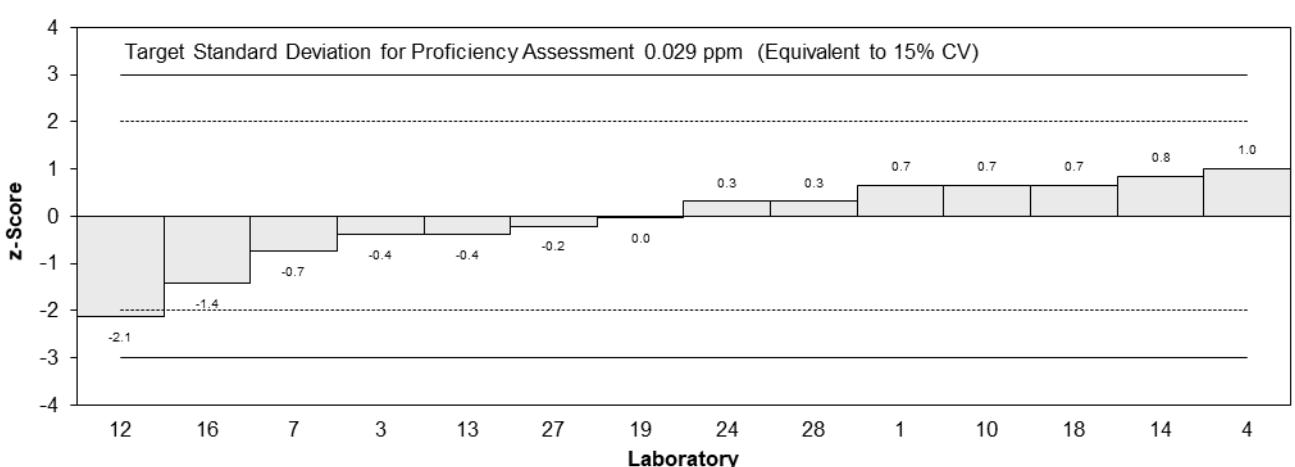
<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	0.21	0.02	0.66	0.74
2	NT	NT		
3	0.18	1	-0.38	-0.01
4	0.22	0.03	1.01	0.85
5	<0.17	NR		
6	NT	NT		
7	0.17	0.18	-0.73	-0.12
8	NT	NT		
9	NT	NT		
10	0.21	0.03	0.66	0.56
11	NT	NT		
12	0.13	0.02	-2.13	-2.38
13	0.18	2	-0.38	-0.01
14	0.215	0.038	0.84	0.58
15	NR	NR		
16	0.15	0.03	-1.43	-1.21
17	<0.2	NR		
18	0.21	0.014	0.66	0.89
19	0.19	0.08	-0.03	-0.01
21	NT	NT		
22	NT	NT		
24	0.2	0.070	0.31	0.13
25	NT	NT		
26	NT	NT		
27	0.185	0.037	-0.21	-0.15
28	0.2	NR	0.31	0.56
29	NT	NT		
30	NT	NT		

**Statistics**

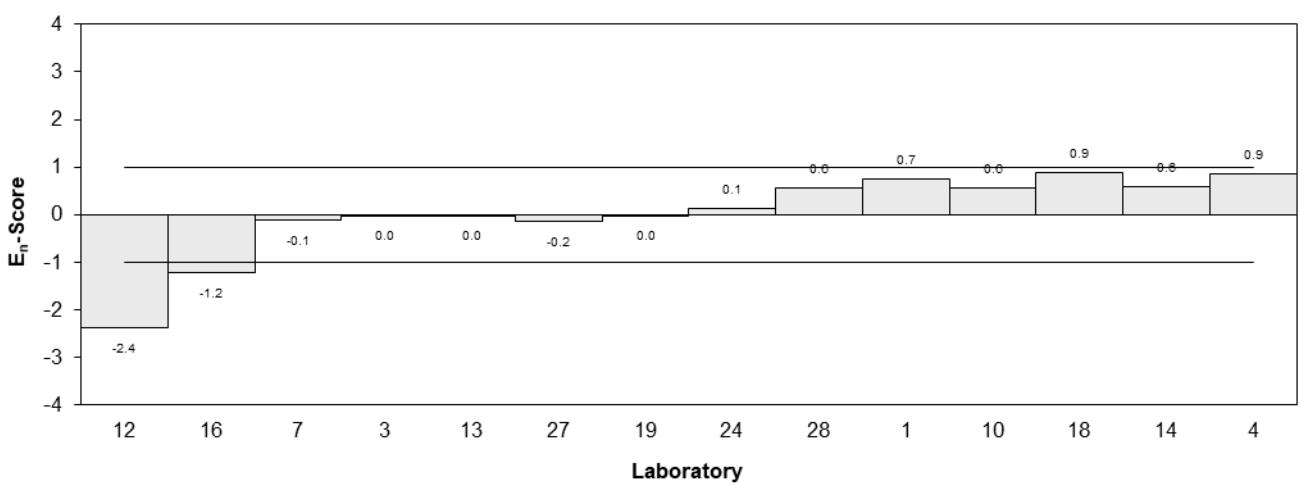
<b>Assigned Value</b>	0.191	0.016
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	0.212	0.025
<b>Robust Average</b>	0.191	0.016
<b>Median</b>	0.195	0.015
<b>Mean</b>	0.189	0.014
<b>N</b>	14	
<b>Max</b>	0.22	
<b>Min</b>	0.13	
<b>Robust SD</b>	0.024	
<b>Robust CV (%)</b>	13	



**z-Scores: S3 - Exchangeable Na**



**En-Scores: S3 - Exchangeable Na**



**Figure 45**

Table 58

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	Extractable B
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U
1	NT	NT
2	NT	NT
3	NT	NT
4	NT	NR
5	NT	NT
6	NT	NT
7	NR	NR
8	NT	NT
9	NT	NT
10	NR	NR
11	NT	NT
12	<0.1	NR
13	NT	NT
14	NT	NT
15	NR	NR
16	0.45	0.09
17	<0.2	NR
18	NT	NT
19	<1.00	NR
21	NT	NT
22	NT	NT
24	NR	NR
25	NT	NT
26	NT	NT
27	NT	NT
28	0.404	NR
29	NT	NT
30	NT	NT

**Statistics**

<b>Assigned Value</b>	Not Set	
<b>Spike Value</b>	Not Spiked	
<b>Mean</b>	0.427	0.046
<b>N</b>	2	
<b>Max</b>	0.45	
<b>Min</b>	0.404	

**Results: S3 - Extractable B**

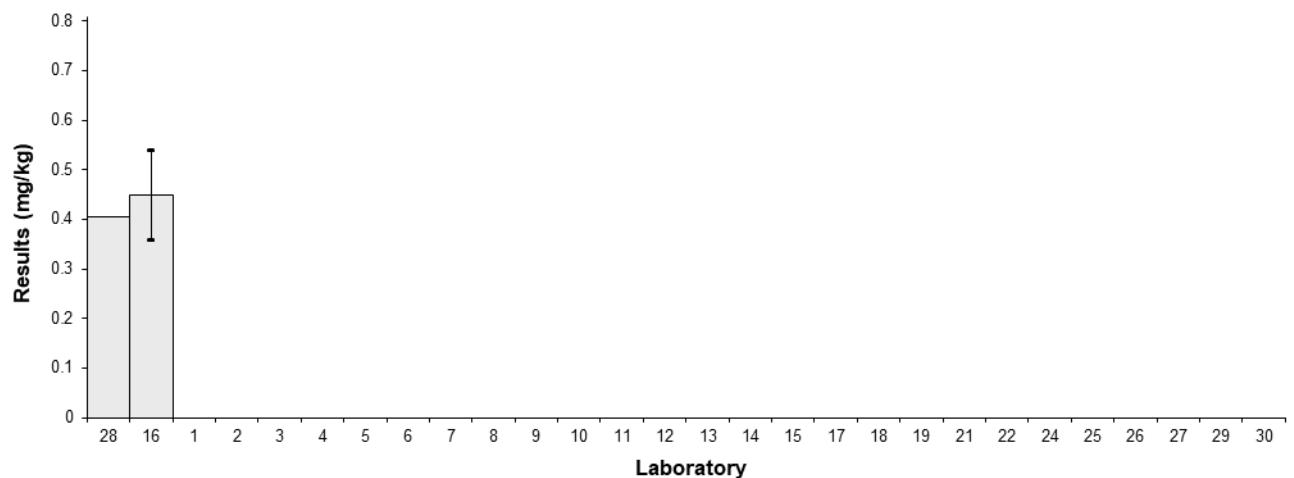


Figure 46

Table 59

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	Fe
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	NT	NT		
3	50000	17000	0.49	0.25
4	45100	5898	-0.04	-0.05
5	48000	5760	0.27	0.31
6	NT	NT		
7	NR	NR		
8	55600	10600	1.11	0.84
9	NT	NT		
10	50900	12000	0.59	0.41
11	NT	NT		
12	53645	5500	0.90	1.04
13	28000	7000	-1.92	-1.95
14	40800	3900	-0.52	-0.69
15	33000	7200	-1.37	-1.37
16	36000	7000	-1.04	-1.06
17	35900	4090	-1.05	-1.38
18	NT	NT		
19	55750	11150	1.13	0.82
21	NT	NT		
22	50000	7500	0.49	0.48
24	37400	7951	-0.89	-0.83
25	47000	9400	0.16	0.14
26	55000	2600	1.04	1.54
27	54300	10860	0.97	0.72
28	38600	7720	-0.76	-0.72
29	NT	NT		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	45500	5600
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	55100	6600
<b>Robust Average</b>	45500	5600
<b>Median</b>	47500	6200
<b>Mean</b>	45300	4100
<b>N</b>	18	
<b>Max</b>	55750	
<b>Min</b>	28000	
<b>Robust SD</b>	9500	
<b>Robust CV (%)</b>	21	

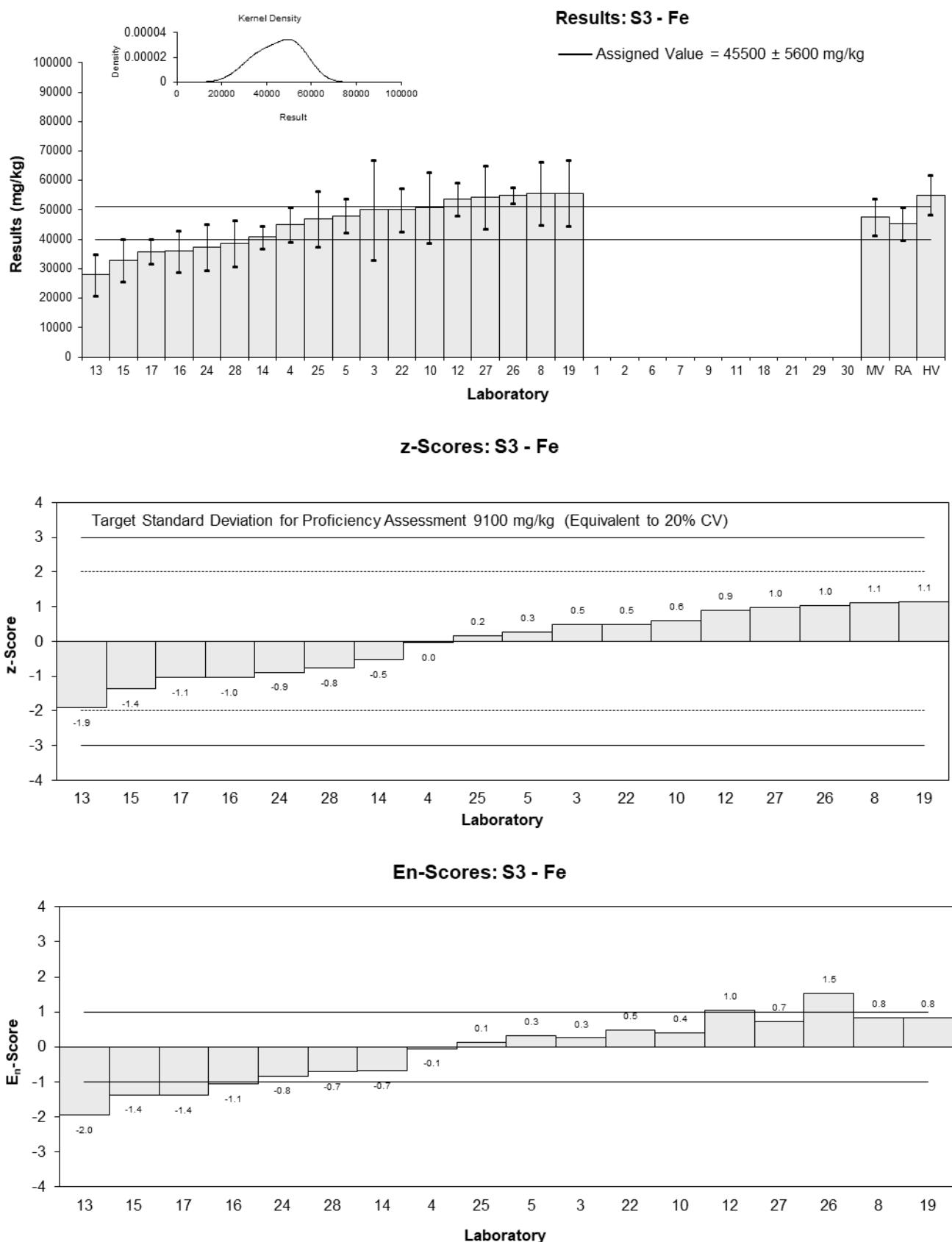


Figure 47

Table 60

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	K
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	NT	NT		
3	700	220	-0.61	-0.31
4	830	113	0.51	0.45
5*	1500	75	6.30	7.15
6	NT	NT		
7	NR	NR		
8*	1270	240	4.31	2.00
9	NT	NT		
10	830	166	0.51	0.33
11	NT	NT		
12	841	85	0.61	0.64
13	630	200	-1.22	-0.67
14	817	160	0.40	0.26
15	540	81	-2.00	-2.17
16	760	200	-0.10	-0.05
17	730	85	-0.35	-0.37
18	NT	NT		
19	872	260	0.87	0.38
21	NT	NT		
22	640	96	-1.13	-1.11
24	720	129	-0.44	-0.35
25	NT	NT		
26	890	210	1.03	0.54
27	877	175.4	0.92	0.56
28	815.5	133.6	0.38	0.30
29	NT	NT		
30	NT	NT		

\* Outlier

**Statistics**

<b>Assigned Value</b>	771	69
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	652	98
<b>Robust Average</b>	796	80
<b>Median</b>	817	66
<b>Mean</b>	840	110
<b>N</b>	17	
<b>Max</b>	1500	
<b>Min</b>	540	
<b>Robust SD</b>	130	
<b>Robust CV (%)</b>	17	

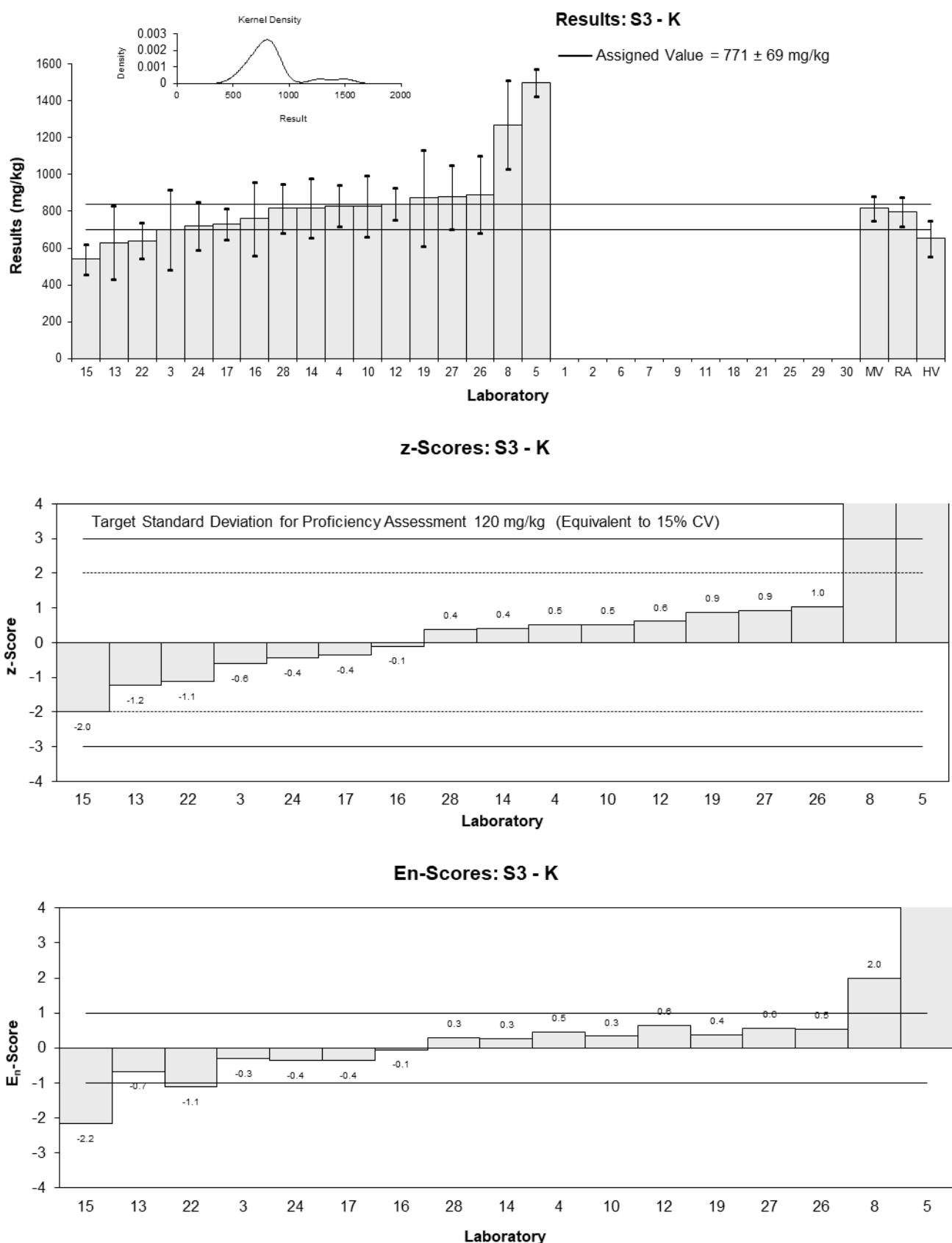


Figure 48

Table 61

**Sample Details**

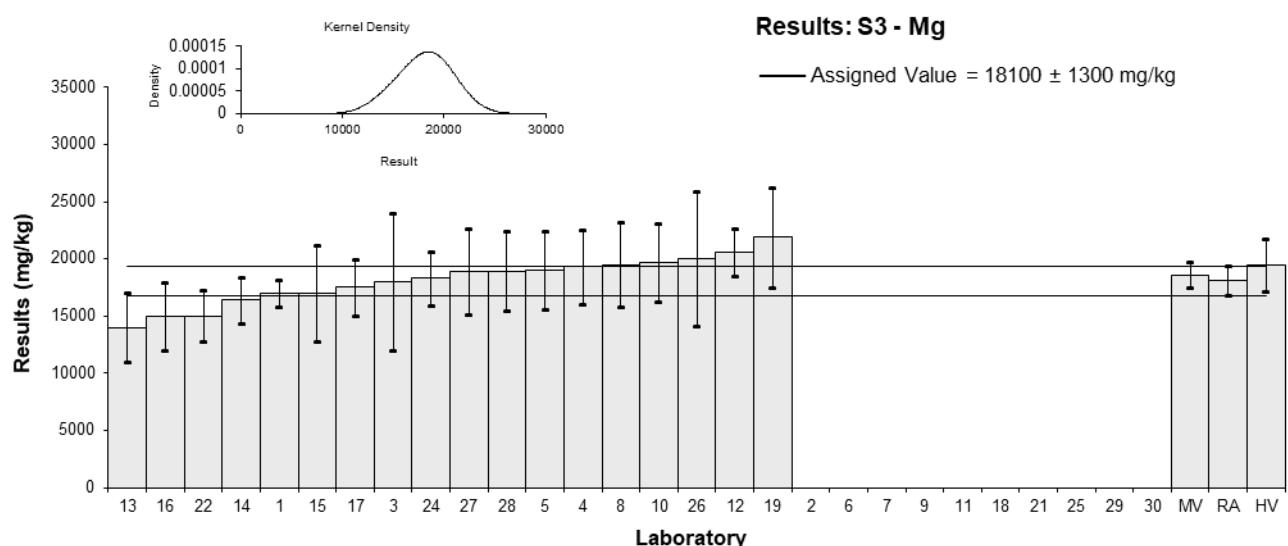
<b>Sample</b>	S3
<b>Analyte</b>	Mg
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

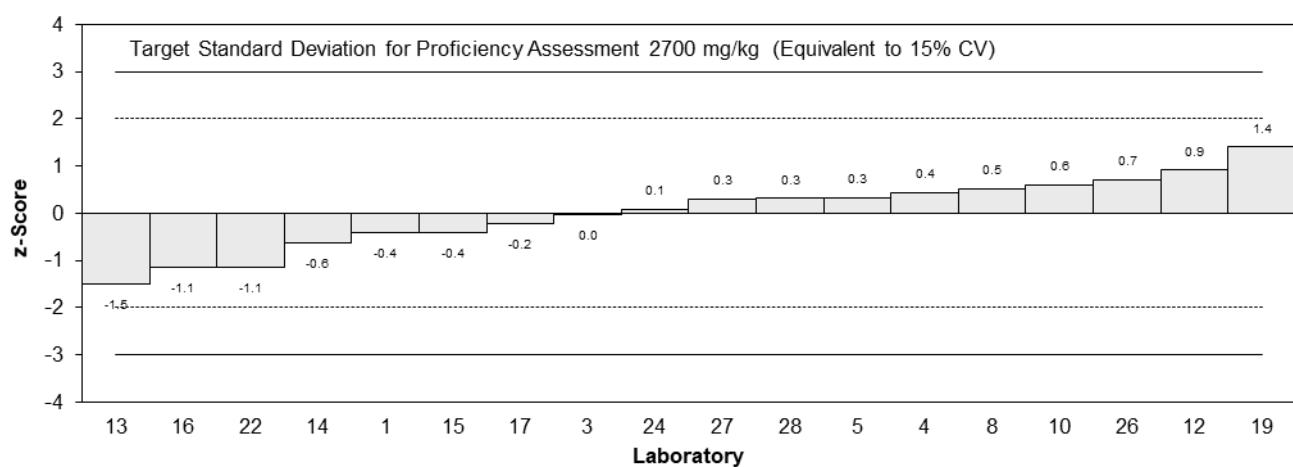
Lab. Code	Result	U	z	E <sub>n</sub>
1	17000	1200	-0.41	-0.62
2	NT	NT		
3	18000	6000	-0.04	-0.02
4	19300	3214	0.44	0.35
5	19000	3420	0.33	0.25
6	NT	NT		
7	NR	NR		
8	19500	3700	0.52	0.36
9	NT	NT		
10	19700	3420	0.59	0.44
11	NT	NT		
12	20563	2100	0.91	1.00
13	14000	3000	-1.51	-1.25
14	16400	2000	-0.63	-0.71
15	17000	4200	-0.41	-0.25
16	15000	3000	-1.14	-0.95
17	17500	2430	-0.22	-0.22
18	NT	NT		
19	21900	4380	1.40	0.83
21	NT	NT		
22	15000	2250	-1.14	-1.19
24	18300	2393	0.07	0.07
25	NT	NT		
26	20000	5900	0.70	0.31
27	18900	3780	0.29	0.20
28	18948	3478.9	0.31	0.23
29	NT	NT		
30	NT	NT		

**Statistics**

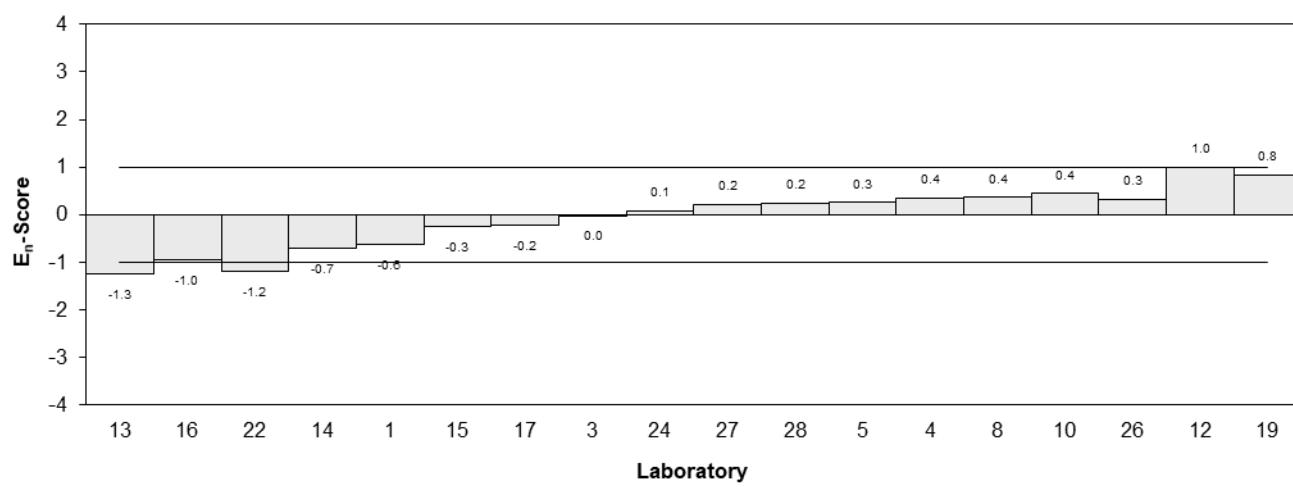
<b>Assigned Value</b>	18100	1300
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	19500	2300
<b>Robust Average</b>	18100	1300
<b>Median</b>	18600	1100
<b>Mean</b>	18100	1000
<b>N</b>	18	
<b>Max</b>	21900	
<b>Min</b>	14000	
<b>Robust SD</b>	2200	
<b>Robust CV (%)</b>	12	



**z-Scores: S3 - Mg**



**En-Scores: S3 - Mg**



**Figure 49**

Table 62

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	Na
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	630	100	2.82	1.92
2	NT	NT		
3	330	110	-0.91	-0.58
4	430	74	0.33	0.28
5*	960	76.8	6.91	5.61
6	NT	NT		
7	NR	NR		
8*	1610	310	14.98	3.82
9	NT	NT		
10	435	91	0.40	0.29
11	NT	NT		
12	469	50	0.82	0.82
13	300	100	-1.28	-0.87
14	422	30	0.24	0.27
15	270	44	-1.65	-1.73
16	290	60	-1.40	-1.30
17	360	51	-0.53	-0.53
18	NT	NT		
19	435	130	0.40	0.22
21	NT	NT		
22	360	54	-0.53	-0.52
24	380	70	-0.29	-0.24
25	NT	NT		
26	350	59	-0.66	-0.61
27	578	115.6	2.17	1.33
28	509.7	76.2	1.32	1.08
29	NT	NT		
30	NT	NT		

\* Outlier

**Statistics**

<b>Assigned Value</b>	403	63
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	360	43
<b>Robust Average</b>	435	81
<b>Median</b>	426	70
<b>Mean</b>	510	150
<b>N</b>	18	
<b>Max</b>	1610	
<b>Min</b>	270	
<b>Robust SD</b>	140	
<b>Robust CV (%)</b>	32	

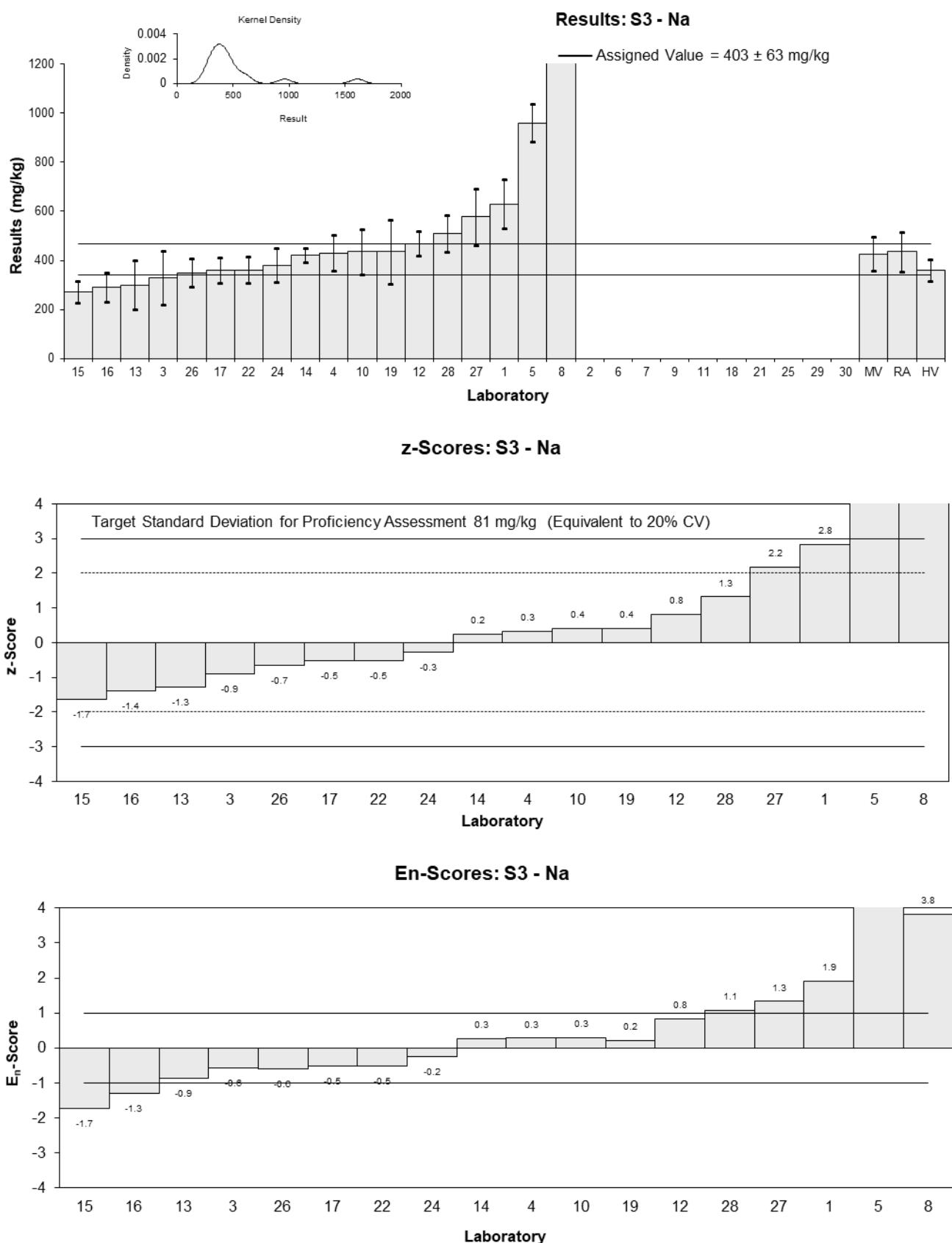


Figure 50

Table 63

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	P
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	820	110	-0.85	-0.61
2	NT	NT		
3	800	250	-1.07	-0.37
4	930	NR	0.38	0.58
5	NT	NT		
6	NT	NT		
7	NR	NR		
8	949	180	0.59	0.28
9	NT	NT		
10	988	128	1.03	0.65
11	NT	NT		
12	986	95	1.00	0.80
13	780	200	-1.29	-0.56
14	844	130	-0.58	-0.36
15	940	140	0.49	0.29
16	760	200	-1.52	-0.65
17	840	200	-0.62	-0.27
18	NT	NT		
19	1045	210	1.66	0.68
21	NT	NT		
22	780	117	-1.29	-0.89
24	910	121	0.16	0.10
25	950	190	0.60	0.27
26	920	100	0.27	0.21
27	1020	204	1.38	0.58
28	865	180.0	-0.35	-0.16
29	NT	NT		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	896	59
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	950	110
<b>Robust Average</b>	896	59
<b>Median</b>	915	63
<b>Mean</b>	896	41
<b>N</b>	18	
<b>Max</b>	1045	
<b>Min</b>	760	
<b>Robust SD</b>	100	
<b>Robust CV (%)</b>	11	

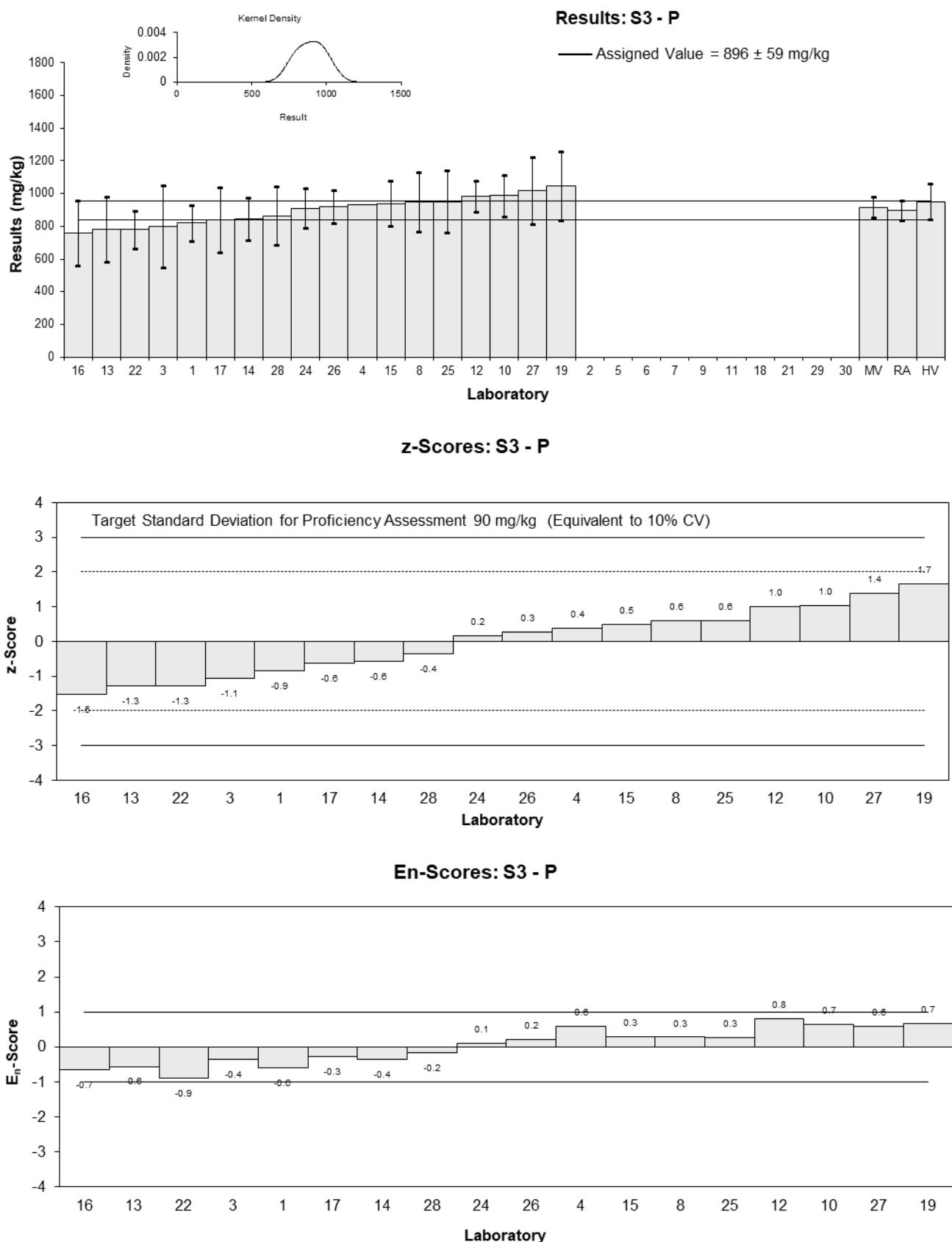


Figure 51

Table 64

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	PBI
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U
1	109	7.6
2	NT	NT
3	NT	NT
4	NT	NR
5	NT	NT
6	NT	NT
7	117.4	NR
8	NT	NT
9	NT	NT
10	NR	NR
11	NT	NT
12	140	15
13	NT	NT
14	NT	NT
15	NR	NR
16	NT	NT
17	NT	NT
18	NT	NT
19	113	23
21	NT	NT
22	NT	NT
24	NR	NR
25	NT	NT
26	NT	NT
27	NT	NT
28	NR	NR
29	NT	NT
30	NT	NT

**Statistics**

<b>Assigned Value</b>	Not Set	
<b>Spike Value</b>	Not Spiked	
<b>Median</b>	115	8
<b>Mean</b>	120	14
<b>N</b>	4	
<b>Max</b>	140	
<b>Min</b>	109	

**Results: S3 - PBI**

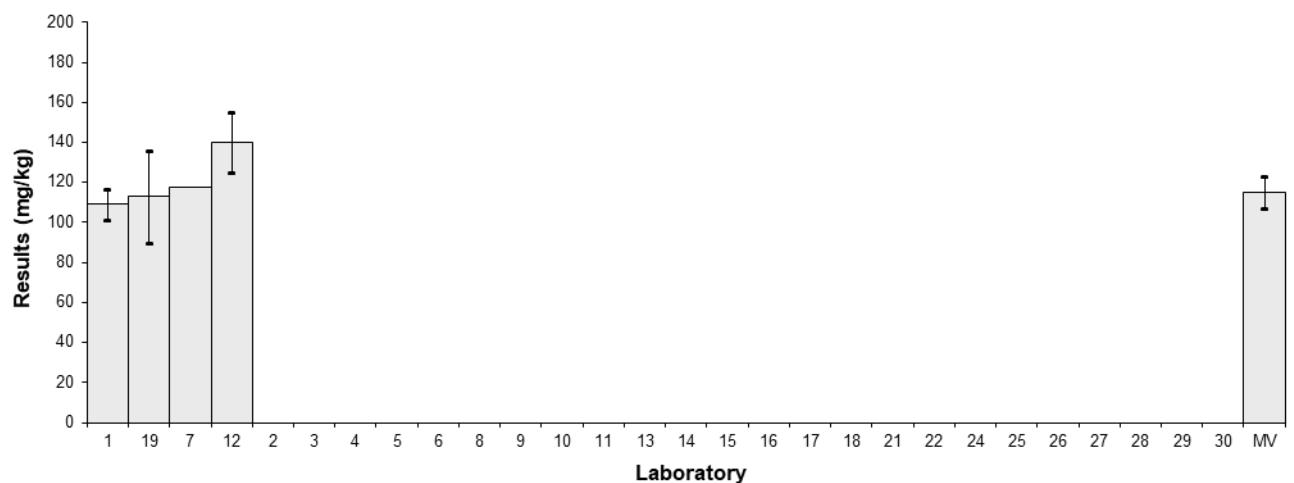


Figure 52

Table 65

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	pH
<b>Matrix</b>	Soil

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	6.72	0.22	-1.06	-0.71
2	NT	NT		
3	NT	NT		
4	7.54	0.1	2.29	1.83
5	NT	NT		
6	NT	NT		
7	7.43	0.08	1.84	1.50
8	NT	NT		
9	NT	NT		
10	6.8	0.2	-0.74	-0.51
11	NT	NT		
12	6.57	0.2	-1.68	-1.16
13	6.6	0.2	-1.56	-1.08
14	6.38	0.2	-2.46	-1.70
15	NR	NR		
16	6.6	0.2	-1.56	-1.08
17	6.8	0.1	-0.74	-0.59
18	6.8	0.048	-0.74	-0.61
19	7.25	0.4	1.11	0.55
21	NT	NT		
22	6.6	0.2	-1.56	-1.08
24	7.4	0.167	1.72	1.26
25	7.57	0.01988	2.42	2.03
26	6.6	NR	-1.56	-1.31
27	7.56	0.1	2.37	1.89
28	7.41	0.206	1.76	1.21
29	NT	NT		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	6.98	0.29
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	7.50	0.38
<b>Robust Average</b>	6.98	0.29
<b>Median</b>	6.80	0.21
<b>Mean</b>	6.98	0.21
<b>N</b>	17	
<b>Max</b>	7.57	
<b>Min</b>	6.38	
<b>Robust SD</b>	0.48	
<b>Robust CV (%)</b>	6.9	

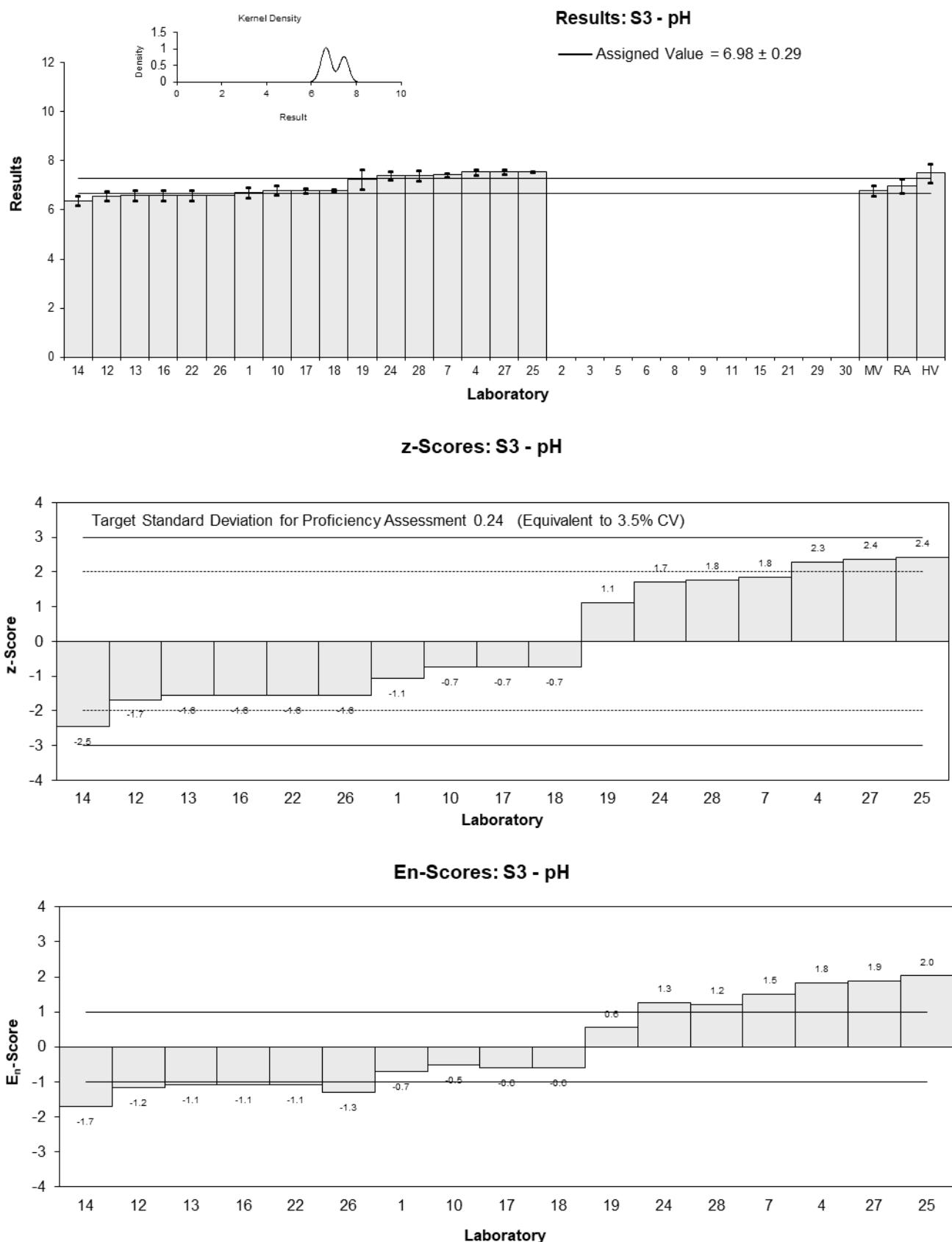


Figure 53

Table 66

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	S
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	98	10	0.29	0.23
2	NT	NT		
3	93	30	-0.23	-0.07
4	90	9.8	-0.55	-0.43
5	108	6.48	1.34	1.32
6	NT	NT		
7	NR	NR		
8	93	18	-0.23	-0.11
9	NT	NT		
10	114	23	1.97	0.78
11	NT	NT		
12	NT	NT		
13	93	30	-0.23	-0.07
14	92.3	9.4	-0.30	-0.24
15	81	NR	-1.49	-1.97
16	88	20	-0.76	-0.34
17	80	26	-1.60	-0.56
18	NT	NT		
19	108	16	1.34	0.73
21	NT	NT		
22*	22	3.3	-7.69	-9.24
24	90	11	-0.55	-0.40
25	NT	NT		
26*	330	18	24.66	12.11
27	108	21.6	1.34	0.56
28	93.2	NR	-0.21	-0.28
29	NT	NT		
30	NT	NT		

\* Outlier

**Statistics**

<b>Assigned Value</b>	95.2	7.2
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	117	14
<b>Robust Average</b>	95.3	8.1
<b>Median</b>	93.0	4.5
<b>Mean</b>	105	30
<b>N</b>	17	
<b>Max</b>	330	
<b>Min</b>	22	
<b>Robust SD</b>	13	
<b>Robust CV (%)</b>	14	

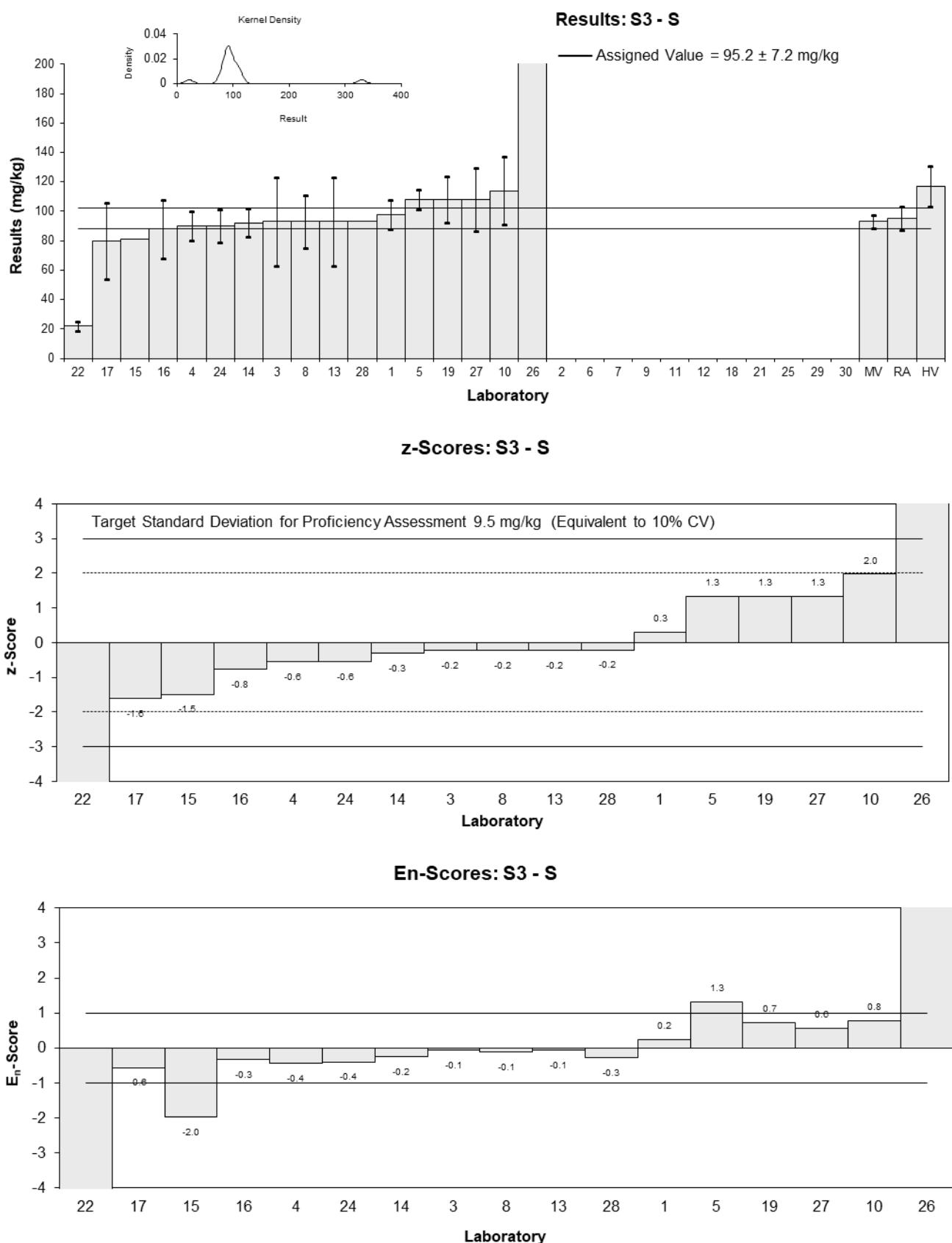


Figure 54

Table 67

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	TC
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	10600	770	0.00	0.00
2	NT	NT		
3	9800	3000	-0.75	-0.26
4	NT	NR		
5	NT	NT		
6	NT	NT		
7	10600	800	0.00	0.00
8	NT	NT		
9	NT	NT		
10	10000	1500	-0.57	-0.37
11	NT	NT		
12	12200	1220	1.51	1.18
13	NT	NT		
14	NT	NT		
15	NR	NR		
16	10000	2000	-0.57	-0.29
17	10500	313	-0.09	-0.15
18	NT	NT		
19	11500	250	0.85	1.38
21	NT	NT		
22	NT	NT		
24	NR	NR		
25	NT	NT		
26	NT	NT		
27	10600	3180	0.00	0.00
28	NR	NR		
29	NT	NT		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	10600	600
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	10300	1200
<b>Robust Average</b>	10600	600
<b>Median</b>	10600	700
<b>Mean</b>	10600	500
<b>N</b>	9	
<b>Max</b>	12200	
<b>Min</b>	9800	
<b>Robust SD</b>	740	
<b>Robust CV (%)</b>	7	

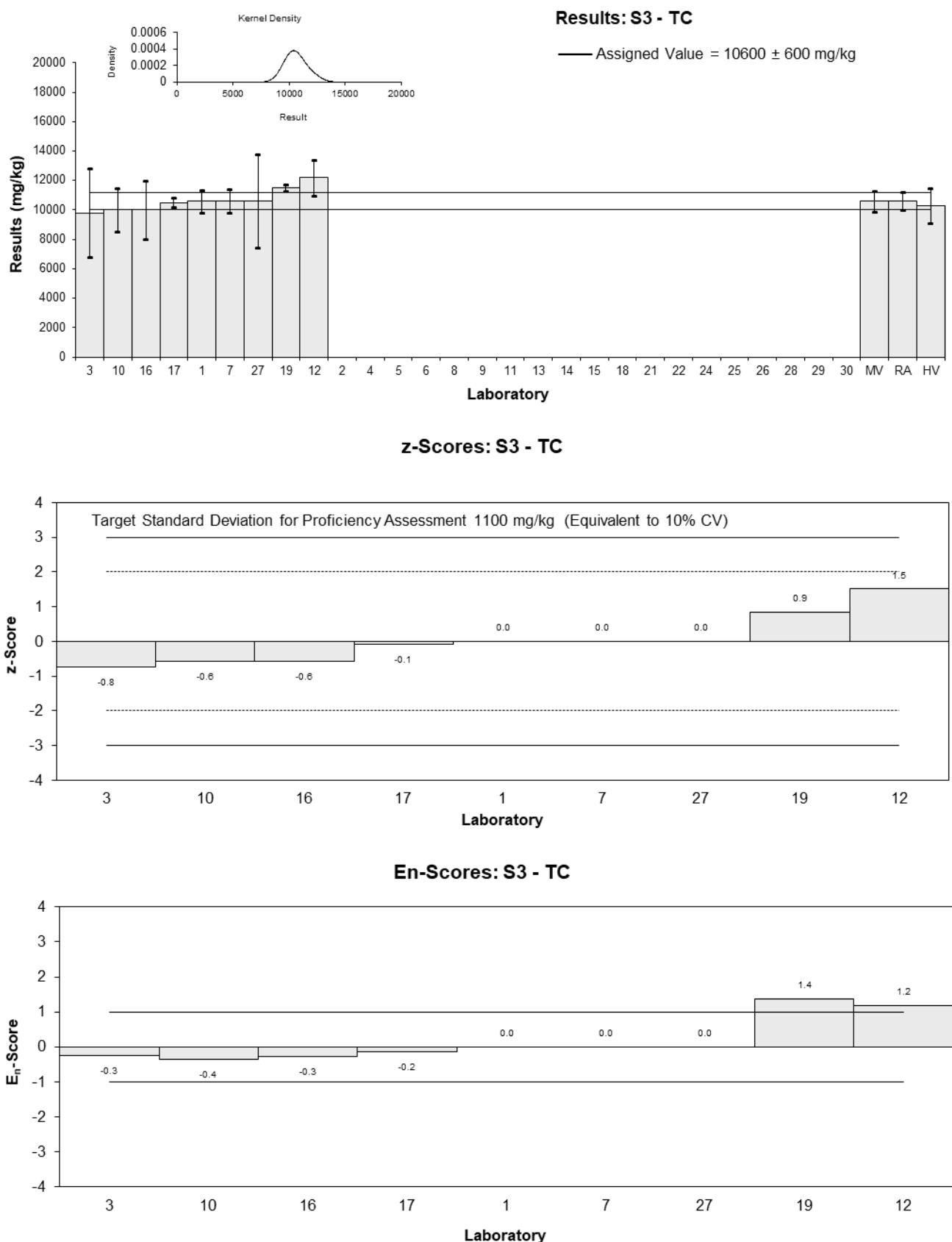


Figure 55

Table 68

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	TN
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	740	80	-1.23	-1.04
2	NT	NT		
3	940	300	1.14	0.31
4	910	230	0.78	0.28
5	NT	NT		
6	NT	NT		
7	850	100	0.07	0.05
8	850	128	0.07	0.04
9	NT	NT		
10	700	100	-1.71	-1.23
11	NT	NT		
12	790	79	-0.64	-0.54
13	940	300	1.14	0.31
14	870	250	0.31	0.10
15	NR	NR		
16	780	200	-0.76	-0.31
17	870	156	0.31	0.16
18	1020	140	2.09	1.16
19	650	70	-2.30	-2.10
21	NT	NT		
22	900	135	0.66	0.38
24*	100	34.6	-8.82	-10.74
25	NT	NT		
26	NT	NT		
27	820	164	-0.28	-0.14
28	853.3	163.3	0.11	0.05
29	NT	NT		
30	NT	NT		

\* Outlier

**Statistics**

<b>Assigned Value</b>	844	60
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	840	100
<b>Robust Average</b>	832	66
<b>Median</b>	850	54
<b>Mean</b>	799	98
<b>N</b>	17	
<b>Max</b>	1020	
<b>Min</b>	100	
<b>Robust SD</b>	110	
<b>Robust CV (%)</b>	13	

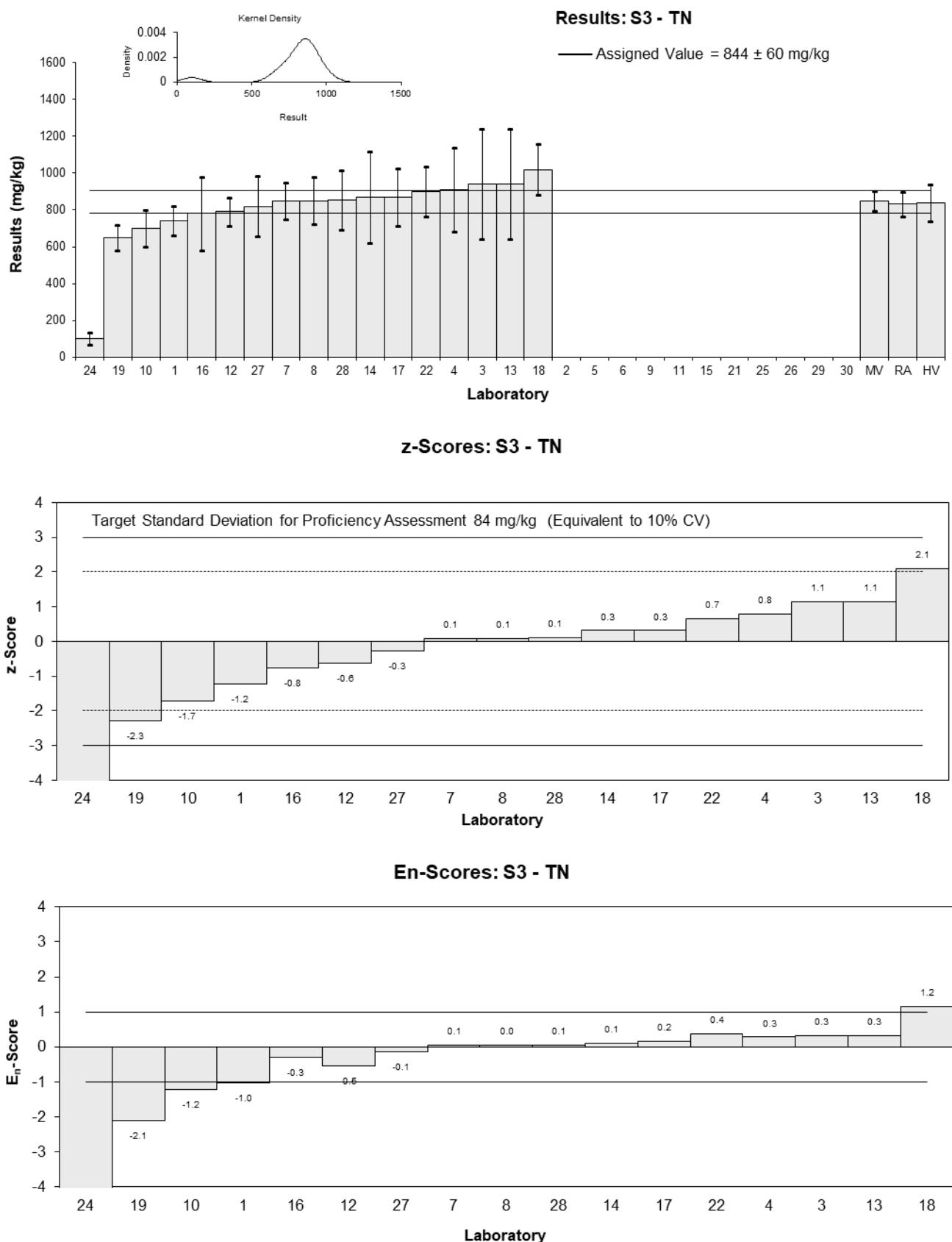


Figure 56

Table 69

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	TOC
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

Lab. Code	Result	U	z	E <sub>n</sub>
1	NT	NT		
2	NT	NT		
3	9000	3000	-1.51	-0.53
4	11000	2015	0.38	0.19
5	NT	NT		
6	NT	NT		
7	10600	853	0.00	0.00
8	11000	2800	0.38	0.14
9	NT	NT		
10	10000	1500	-0.57	-0.38
11	NT	NT		
12	11000	1100	0.38	0.33
13	10600	3000	0.00	0.00
14	10800	1700	0.19	0.11
15	NR	NR		
16	9500	2000	-1.04	-0.53
17	10000	380	-0.57	-0.96
18	10900	470	0.28	0.44
19	12500	250	1.79	3.40
21	NT	NT		
22	10000	1545	-0.57	-0.37
24	12000	2400	1.32	0.57
25	NT	NT		
26	NT	NT		
27	10600	3180	0.00	0.00
28	10440	NR	-0.15	-0.32
29	NT	NT		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	10600	500
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	10300	1200
<b>Robust Average</b>	10600	500
<b>Median</b>	10600	400
<b>Mean</b>	10600	400
<b>N</b>	16	
<b>Max</b>	12500	
<b>Min</b>	9000	
<b>Robust SD</b>	770	
<b>Robust CV (%)</b>	7.2	

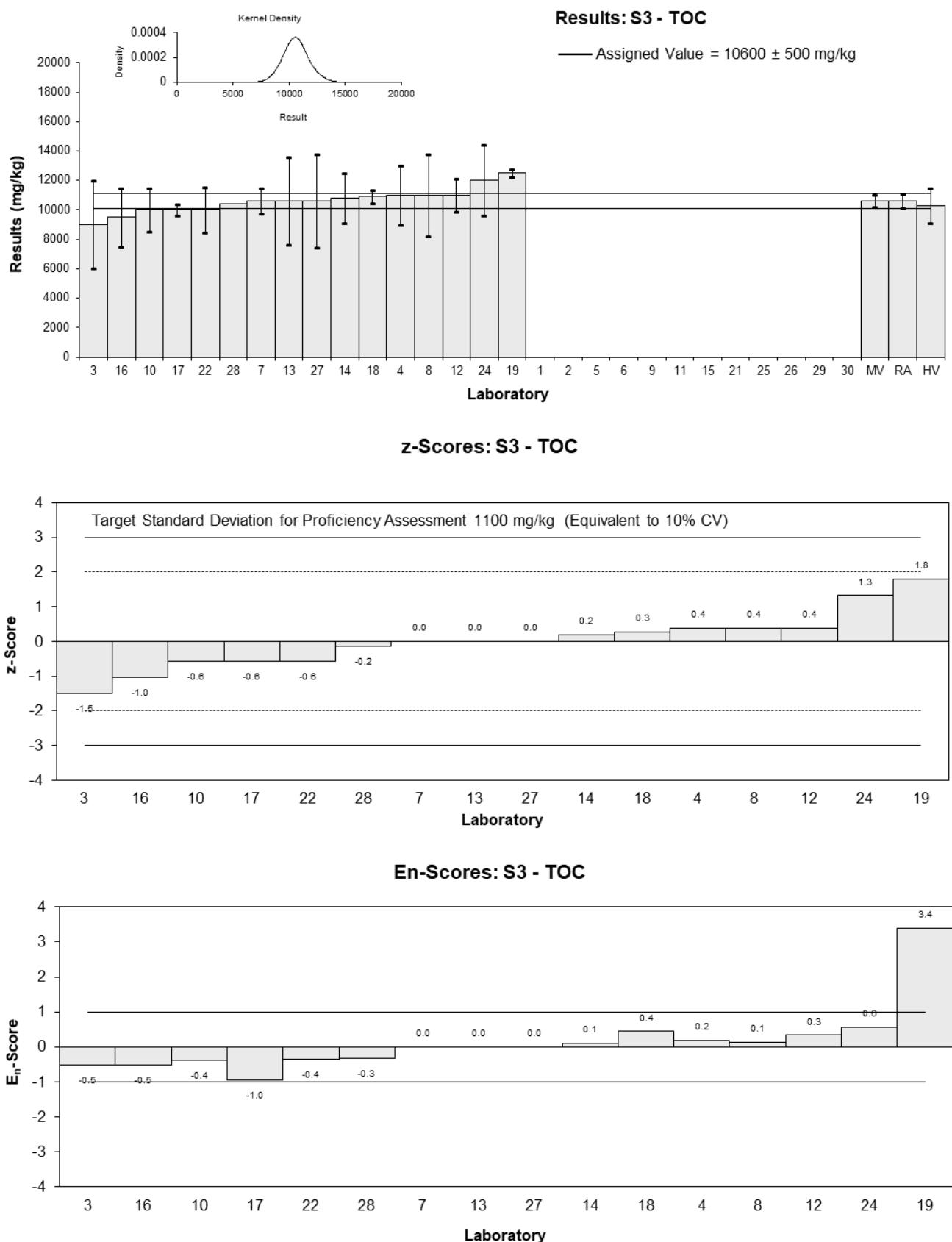


Figure 57

Table 70

**Sample Details**

<b>Sample</b>	S3
<b>Analyte</b>	Total P
<b>Matrix</b>	Soil
<b>Unit</b>	mg/kg

**Participant Results**

<b>Lab. Code</b>	<b>Result</b>	<b>U</b>	<b>z</b>	<b>E<sub>n</sub></b>
1	890	130	0.31	0.23
2	NT	NT		
3	NT	NT		
4	749	174	-0.79	-0.49
5	510	NR	-2.67	-3.09
6	NT	NT		
7	NR	NR		
8	920	138	0.55	0.40
9	NT	NT		
10	NR	NR		
11	NT	NT		
12	NR	NR		
13	NT	NT		
14	910	160	0.47	0.31
15	NR	NR		
16	NT	NT		
17	805	125	-0.35	-0.27
18	1060	170	1.65	1.04
19	1045	210	1.53	0.82
21	NT	NT		
22	780	117	-0.55	-0.44
24	747	155.6	-0.81	-0.54
25	NT	NT		
26	NT	NT		
27	NT	NT		
28	830.9	173	-0.15	-0.09
29	NT	NT		
30	NT	NT		

**Statistics**

<b>Assigned Value</b>	850	110
<b>Spike Value</b>	Not Spiked	
<b>Homogeneity Value</b>	950	110
<b>Robust Average</b>	850	110
<b>Median</b>	831	92
<b>Mean</b>	841	92
<b>N</b>	11	
<b>Max</b>	1060	
<b>Min</b>	510	
<b>Robust SD</b>	150	
<b>Robust CV (%)</b>	17	

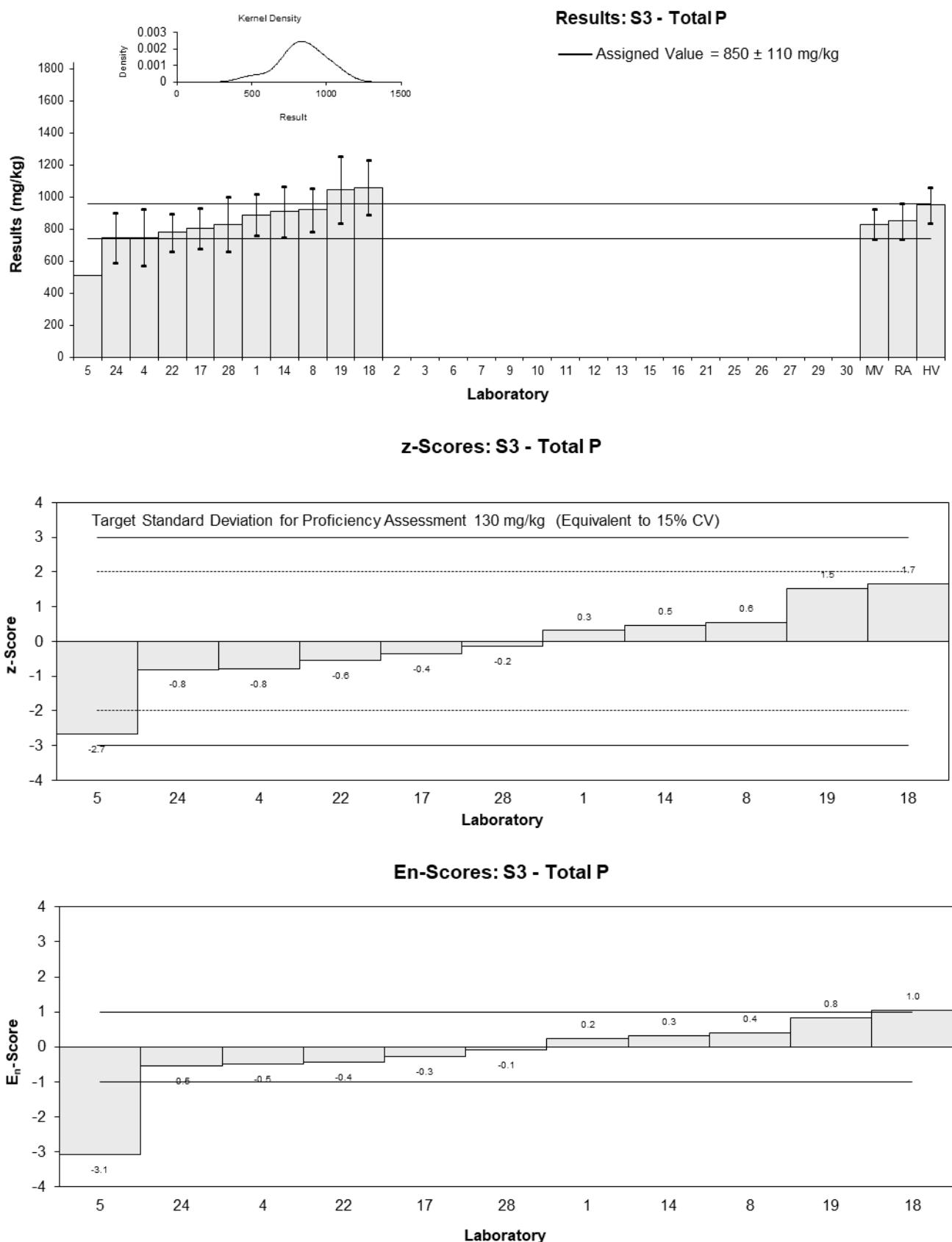


Figure 58

## 6 DISCUSSION OF RESULTS

### 6.1 Assigned Value

**Sample S1** was dried sediment fortified for 24 elements. Participants were asked to report results for Sample S1 on an as-received basis.

**Sample S2** was the dried sediment sample S1 to which a known amount of water was added. Participants were asked to use their normal method but to report results corrected for moisture content.

**Sample S3** was dried agricultural soil.

**Assigned values** for 52 tests in the study samples were the robust averages of participants' results. The robust averages and their associated expanded uncertainties were calculated using the procedures described in ISO 13528:2015(E). Results less than 50% and more than 150% of the robust average were removed before calculation of the assigned value.<sup>6</sup> Appendix 2 sets out the calculation of the robust average of As in Sample S1 and its associated uncertainty.

No assigned value was set for Th, Li, Colwell K, extractable B and phosphorus buffer index because the reported results were either too variable or too few. However, participants may still compare their reported results for these elements with the robust average of participants' results and/or the homogeneity value. Descriptive statistics for these elements are still presented in Section 5.

**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 923 numerical results, 881 (95%) were reported with an expanded measurement uncertainty. The magnitude of these expanded uncertainties was within the range 0.3% to 1111% 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.<sup>9–15</sup>

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 58). 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, 20 laboratories reported results for Cu in S1. The uncertainty of the assigned value estimated from the robust standard deviation of the 20 laboratories' results is 2 mg/kg (see equation 4, Appendix 2). Laboratory 29 might have under-estimated its expanded measurement uncertainties reported for Cu in S1 (1 mg/kg) as an uncertainty estimated from one measurement cannot be smaller than the uncertainty estimated from 20 measurements. Alternatively, estimates of uncertainties for Ba in S2 larger than 8.2 mg/kg (the uncertainty of

the assigned value, 2.2 mg/kg plus the allowable variation from the assigned value, the target standard deviation of 3.0 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 **19** for Ba in S2 (16 mg/kg) might have been over-estimated.

Laboratory **29** should review their procedure for estimating measurement uncertainty as most of their estimated uncertainties were under or over-estimated.

When a laboratory has successfully participated in at least 6 proficiency testing studies, the standard deviation from proficiency testing studies only, can also be used to estimate the uncertainty of their measurement results.<sup>10</sup> An example of estimating measurement uncertainty using proficiency testing data only is given in Appendix 3.

Laboratories **21**, **24**, **25** and **28** 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.<sup>9</sup>

Laboratories **3**, **13**, **7** and **29** 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  $2495.52 \pm 374.33$  mg/kg, it is better to report  $2500 \pm 370$  mg/kg or instead of  $9910 \pm 1486.50$  mg/kg, it is better to report  $9910 \pm 1500$  mg/kg.<sup>9</sup>

### 6.3 E<sub>n</sub>-score

E<sub>n</sub>-score should be interpreted only in conjunction with z-scores. The E<sub>n</sub>-score indicates how closely a result agrees with the assigned value taking into account the respective uncertainties. An unsatisfactory E<sub>n</sub> 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<sub>n</sub>-scores is graphically presented in Figure 59. Where a laboratory did not report an expanded uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the E<sub>n</sub>-score.

Of 890 results for which E<sub>n</sub>-scores were calculated, 740 (83%) returned a satisfactory score of  $|E_n| \leq 1$  indicating agreement of the participants' results with the assigned values within their respective expanded measurement uncertainties.

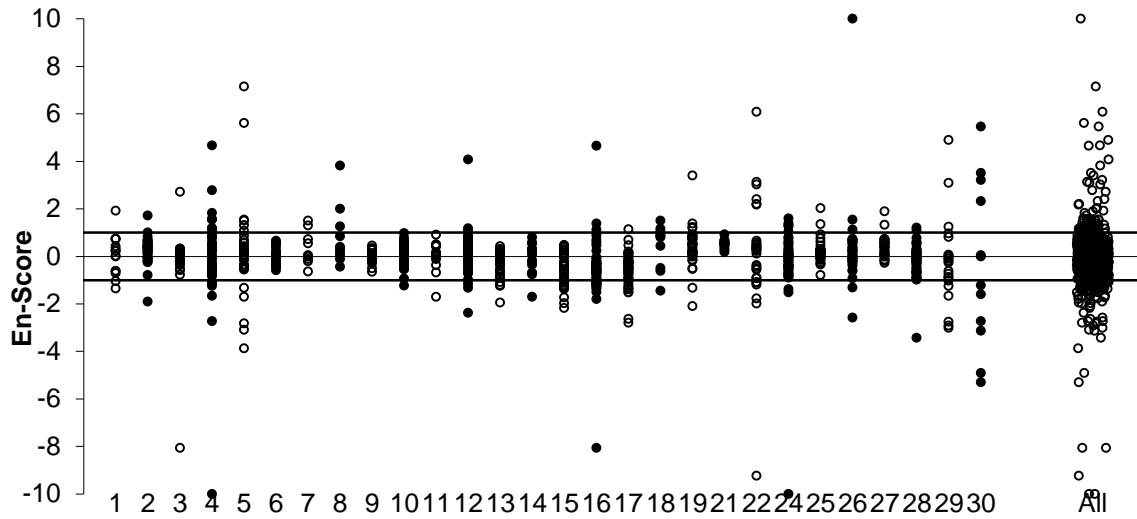
### 6.4 z-Score

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

The target standard deviation defines satisfactory performance in a proficiency test. Target standard deviations equivalent to 3.5%, 10%, 15% and 20% PCV were used to calculate z-scores. Unlike the standard deviation based on between laboratories CV, setting the target standard deviation as a realistic, set value enables z-scores to be used as a fixed reference value point for assessment of laboratory performance, independent of group performance.

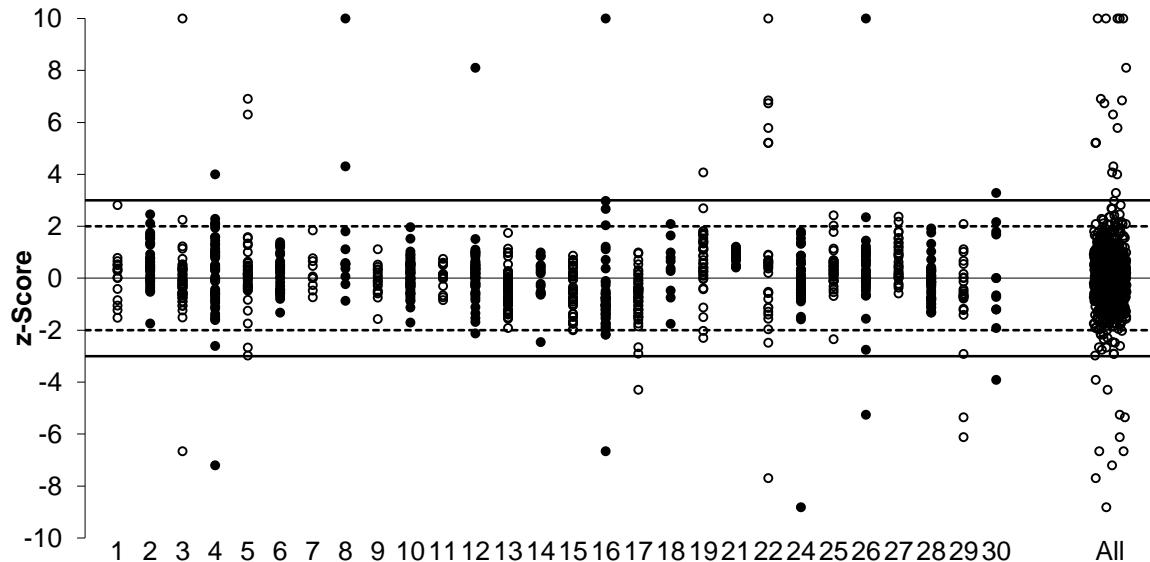
The between laboratory coefficient of variation predicted by the Thompson equation<sup>7</sup> and the between laboratory coefficient of variation resulted in this study are presented for comparison in Table 71. The dispersal of participants' z-scores is presented in Figure 60 (by laboratory code) and in Figure 61 (by test). Of 890 results for which z-scores were calculated, 828 (93%)

returned a satisfactory score of  $|z| \leq 2.0$  and 35 (4%) 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.



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

Figure 59  $E_n$ -Score Dispersal by Laboratory



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

Figure 60 z-Score Dispersal by Laboratory

Summary of participants' performance is presented in Figure 62. No laboratory reported results for all 52 tests for which a z-score was calculated.

**Laboratory 10** returned the highest number of satisfactory z-scores (51 out of 51 reported). Laboratory 10 also had the highest number of satisfactory  $E_n$ -scores at 50.

Laboratory **13** returned satisfactory results for 49 results out of a total of 49 reported and laboratory **24** for 48. Laboratories 17 and 12 reported results for 50 and 49 tests respectively and performed satisfactorily in 47 of them.

All results reported by **laboratories 28** (44), **15** (40), **6** (29), **9** (17), **11** (16), **21** (14) and **7** (10) returned satisfactory z scores.

All results reported by **laboratories 6** (29), **9** (17) and **21** (14) returned satisfactory  $E_n$  scores.

Table 71 Between-Laboratory CV of this Study, Thompson CV and Set Target CV

Sample	Test	Assigned Value (mg/kg)	Between Laboratories CV*	Thompson/ Horwitz CV	Target SD (as PCV)
S1	As	4.02	9.5%	13%	15%
S1	B	9.7	26%	11%	20%
S1	Be	0.897	14%	16%	15%
S1	Bi	1.27	13%	15%	15%
S1	Cd	1.62	9.4%	15%	10%
S1	Cr	49.8	7.7%	8.9%	10%
S1	Cu	67.6	5.4%	8.5%	10%
S1	Hg	0.677	16%	17%	15%
S1	Mn	73.4	8.3%	8.4%	10%
S1	Mo	15.3	10%	11%	10%
S1	Ni	39.3	6.8%	9.2%	10%
S1	Pb	89.6	7%	8.1%	10%
S1	Rb	3.79	22%	13%	20%
S1	Se	2.44	16%	14%	15%
S1	Sn	13.8	14%	11%	15%
S1	Th	Not Set	37%	NA	Not Set
S1	V	25.5	6.6%	9.8%	10%
S1	Zn	63.9	6.9%	8.6%	10%
S2	Ag	5.26	12%	12%	10%
S2	Al	2990	20%	4.8%	15%
S2	As	3.56	6.2%	13%	10%
S2	Ba	30.3	13%	9.6%	10%
S2	Cd	1.38	8.5%	15%	10%
S2	Co	9.44	9%	11%	10%
S2	Cu	58.8	7.3%	8.7%	10%
S2	Hg	0.571	12%	17%	15%
S2	Li	Not Set	36%	NA	Not Set
S2	Mn	63.0	9.6%	8.6%	10%
S2	Moisture Content	18.2 %	6.9%	2.3%	10%
S2	Ni	43.2	9.1%	9.1%	10%
S2	Sb	14.2	18%	11%	15%
S2	Se	2.14	8.9%	14%	15%
S2	Sr	13.3	14%	11%	10%
S2	Tl	1.99	17%	14%	15%
S2	U	4.65	12%	13%	10%
S2	Zn	55.9	8.3%	8.7%	10%
S3	Ca	10000	20%	4%	20%
S3	Colwell K	Not Set	NA	NA	Not Set
S3	Colwell P	44.8	18%	9%	15%
S3	EC	49.3 $\mu$ S/cm	13%	8.9%	10%
S3	Exchangeable Ca	24.1 cmol(+)/kg	7.3%	9.9%	10%
S3	Exchangeable K	0.416 cmol(+)/kg	10%	18%	15%
S3	Exchangeable Mg	9.83 cmol(+)/kg	9.1%	11%	10%
S3	Exchangeable Na	0.191 cmol(+)/kg	13%	21%	15%
S3	Extractable B	Not Set	NA	NA	Not Set
S3	Fe	45500	21%	3.2%	20%
S3	K	771	14%	5.9%	15%
S3	Mg	18100	12%	3.7%	15%
S3	Na	403	25%	6.5%	20%
S3	P	896	11%	5.8%	10%
S3	PBI	Not Set	13%	NA	Not Set

Sample	Test	Assigned Value (mg/kg)	Between Laboratories CV*	Thompson/ Horwitz CV	Target SD (as PCV)
S3	pH	6.98	6.9%	12%	3.5**
S3	S	95.2	12%	8.1%	10%
S3	TC	10600	7%	4%	10%
S3	TN	844	11%	5.8%	10%
S3	TOC	10600	7.2%	4%	10%
S3	Total P	850	17%	5.8%	15%

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

## 6.5 Participants' Results and Analytical Methods for Acid Extractable Elements

A summary of participants' results and performance is presented in Tables 72 to 74 and in Figures 60 and 61.

Th in S1 and Li in S2 were the most difficult elements to analyse. No agreement was found between results reported by participants for these elements.

Measurement of Ca, Fe and Na in S3 also presented difficulty to participating laboratories. The between-laboratory coefficient of variation for these tests was high, ranging from 20% to 25%, while the CV predicted by Thomson and Horwitz was 3.2% to 6.5%.

Laboratories **2**, **4** and **16** may need to check their calculation procedures. The unsatisfactory results reported by Laboratory **16** in all sediment samples S1 were below the assigned value by approximately the same factor of 1.25. A positive bias was also noticed for Laboratory 4 in S1 and for Laboratory **2** in S2; most of the unsatisfactory results they reported were higher than the assigned value by a factor of approximately 1.25.

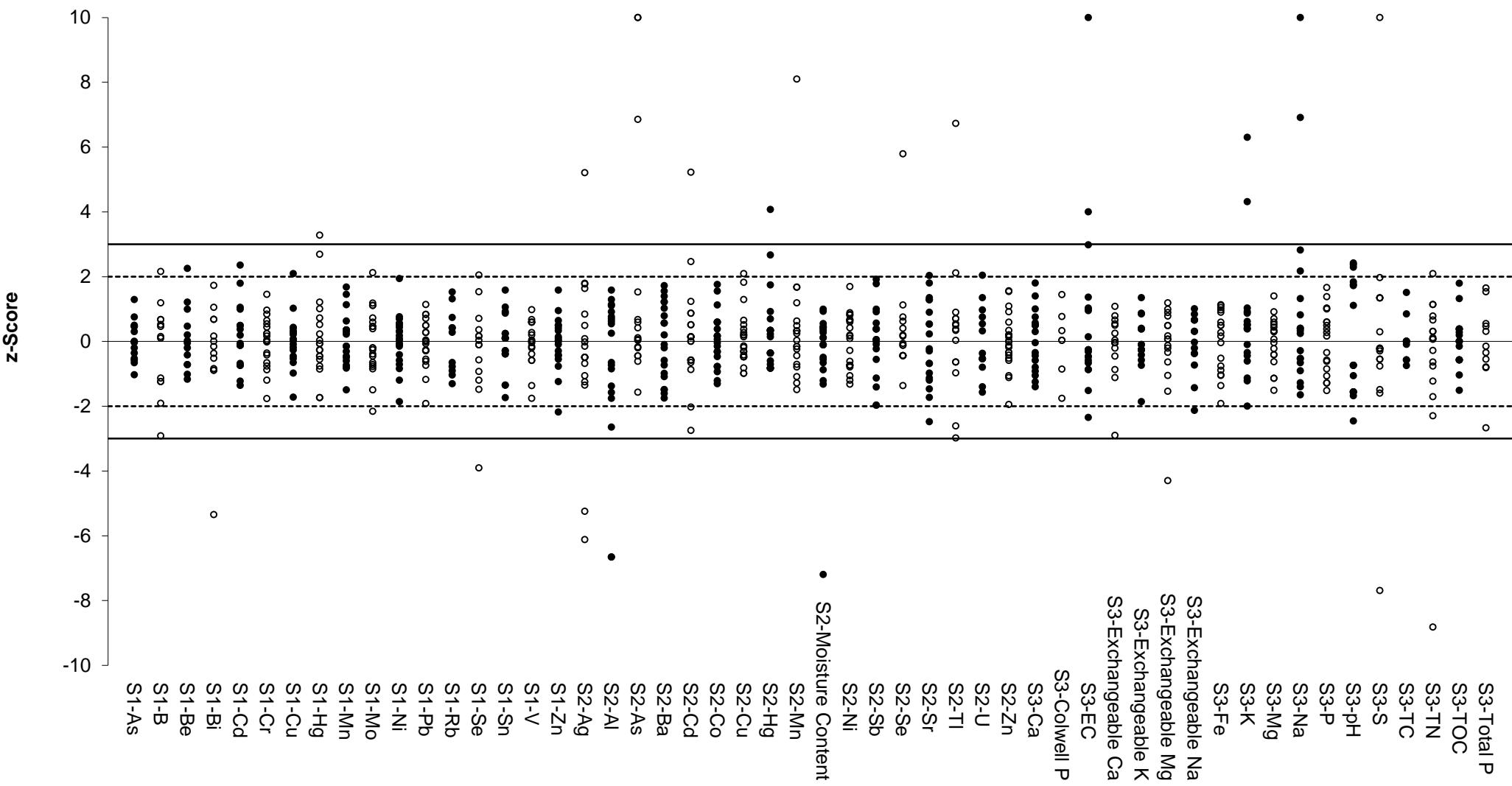
The unsatisfactory results reported by Laboratories **3** and **16** for Al and As in S2 were lower or higher than the assigned value by approximately 1,000 times. These laboratories might have an issue with their calculation procedure or may have reported results in the wrong units. The results reported by these laboratories were not included in the analysis of the extraction methods and instrumental techniques employed by participants.

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

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

### Extraction Methods

The request was for acid extractable elements; NMI PT studies of metals in soil focus on 'pseudo-total' analyses of elements in soil rather than on true total metal content because when an assessment of the anthropogenic impact of the metal content in a soil sample is made, aggressive digestion regimes (HF, high digestion temperature) can lead to misleading conclusions – since metals can be extracted from the fraction naturally present in the soil matrix.<sup>5, 16-19</sup> While an aggressive digestion regime can produce high, misleading results, weak digestion regimes (low digestion temperature, reduced digestion time, diluted acids and/or a low ratio of acid to sample size) may extract just a fraction of the contaminants from the soil. There is no standardisation of methods for acid extractable elements. In general methods are conventionally defined by procedures involving extractions: with aqua regia or



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

Figure 61: z-Score Dispersal by Element

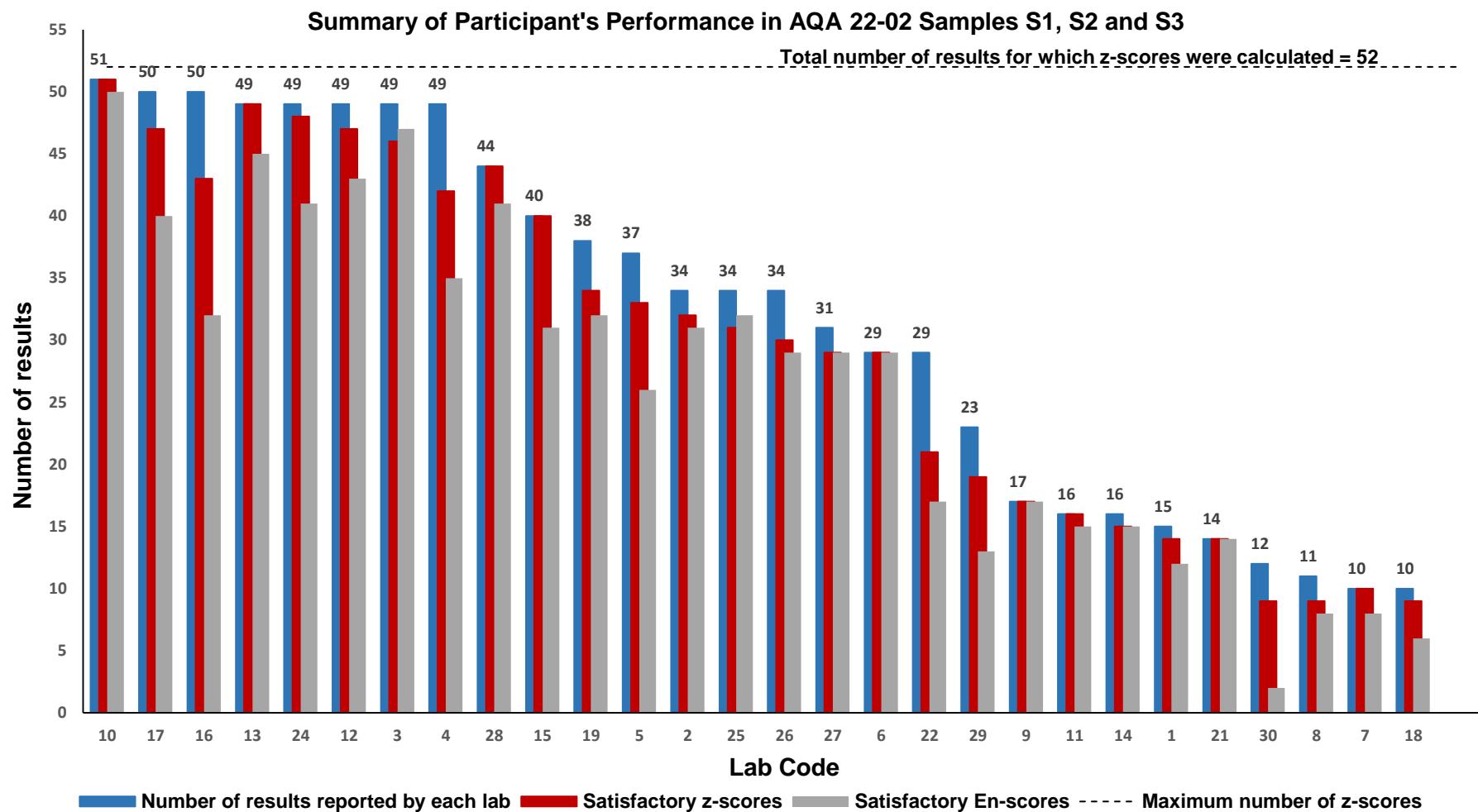


Figure 62: Summary of Participants Performance in AQA 22-02

Table 72 Summary of Participants' Results and Performance for Sample S1

Lab Code	As (mg/kg)	B (mg/kg)	Be (mg/kg)	Bi (mg/kg)	Cd (mg/kg)	Cr (mg/kg)	Cu (mg/kg)	Hg (mg/kg)	Mn (mg/kg)	Mo (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Rb (mg/kg)	Se (mg/kg)	Sn (mg/kg)	Th (mg/kg)	V (mg/kg)	Zn (mg/kg)
A.V.	4.02	9.7	0.897	1.27	1.62	49.8	67.6	0.677	73.4	15.3	39.3	89.6	3.79	2.44	13.8	Not Set	25.5	63.9
H.V.	3.66	8.12	0.84	1.05	1.57	44.8	61.2	0.633	66.3	14.3	38.9	89	4.01	2.33	11.7	1.60	21.7	60.0
1	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
2	4.47	9.90	0.96	1.17	1.68	52.5	69.3	0.63	72.3	14.9	40.5	94.0	4.11	2.57	10.2	0.68	25.4	63.3
3	4.2	7.3	1.2	1.1	1.5	48	64	0.65	69	15	37	87	3.3	2.4	14	0.76	25	59
4	4.8	<50	0.74	1.47	1.78	53.97	81.72	0.67	84.05	18.55	46.91	99.8	4.78	3.19	17.08	0.87	27.99	73.97
5	<5	10	0.84	NT	1.7	53	68	0.5	76	15	41	90	NT	<5	13	NT	27	62
6	3.67	12	<2	<10	1.51	49.8	64.5	0.63	67.6	14.6	38.7	84.7	NT	2.23	13.2	NT	25.6	61.2
7	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
8	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
9	3.9	NT	<5.0	NT	1.6	50	69	0.73	NT	17	39	87	NT	<5.0	14	NT	NT	66
10	4.02	10.7	0.924	1.27	1.61	49.7	69.7	0.65	71.1	14.3	40.5	94.0	4.94	2.50	13.2	1.37	25.1	66.5
11	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
12	4.32	10.6	1.03	1.11	1.65	50.6	69.7	0.67	76	15.9	41.3	87.2	NT	2.45	14.3	3.41	27.2	64.1
13	3.8	7.3	0.87	1.2	1.5	46	61	0.78	70	14	36	84	4.0	2.1	11	1.3	26	56
14	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
15	4.0	<23	0.89	1.4	1.7	51	68	0.62	78	16	40	96	3.1	1.9	14	<9.1	24	68
16	3.4	6	0.76	1.1	1.4	41	56	0.8	68	12	32	83	3.0	2.7	11	1.1	21	50
17	3.7	<50	0.8	1.3	1.5	45.4	66.3	0.5	62.4	14.7	36.5	89.0	2.8	2	13.0	0.8	22	61.1
18	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
19	<25	7.50	NT	NT	<1.0	51.0	70.5	0.95	75.5	13.0	41.5	89.5	NT	<100	<50	NT	24.5	64.5
21	4.3	<20	1.06	1.40	1.79	54.6	70.4	0.75	81.7	17.1	42.3	97	4.35	<20	15.7	NT	<100	67.1
22	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
24	4.0	<50	0.9	1.6	1.6	46.5	63.2	0.6	67.3	16.2	37.7	89.7	3.2	3	15.6	1.5	24	60.4
25	4.3	11	<2	<10	1.6	52	68	0.7	76	16	42	92	NT	2.4	16	NT	27	67
26	<3	11	<1	NT	2	57	67	0.68	75	17	39	97	NT	<4	14	NT	27	70
27	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
28	<5	<50	<1	1.24	1.6	49.8	65.8	0.59	72.3	16.4	39.6	92.1	4.11	<5	14.3	0.83	26.2	64.8
29	NT	4.04	0.8	0.25	1.42	47.7	74.5	NT	69.7	14.1	39.4	79	NT	2.5	NT	NT	25.5	NT
30	3.62	13.9	NT	NT	1.91	43.8	67.7	1.01	85.7	14.2	34.6	72.4	NT	1.01	NT	NT	NT	63.9

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

Table 73 Summary of Participants' Results and Performance for Sample S2

Lab Code	Ag (mg/kg)	Al (mg/kg)	As (mg/kg)	Ba (mg/kg)	Cd (mg/kg)	Co (mg/kg)	Cu (mg/kg)	Hg (mg/kg)	Li (mg/kg)	Mn (mg/kg)	Moisture Content (%)	Ni (mg/kg)	Sb (mg/kg)	Se (mg/kg)	Sr (mg/kg)	Tl (mg/kg)	U (mg/kg)	Zn (mg/kg)
A.V.	5.26	2990	3.56	30.3	1.38	9.44	58.8	0.571	Not Set	63.0	18.2	43.2	14.2	2.14	13.3	1.99	4.65	55.9
H.V.	5.29	2910	3.35	30.0	1.47	9.5	58.8	0.530	2.47	61.7	19.2	41.3	N/A	2.13	12.7	1.94	4.33	55.5
1	NT	NT	NT	NT	NT	NT	NT	NT										
2	6.12	3329	4.10	32.7	1.72	11.1	66.4	0.54	2.21	73.6	19.9	46.0	14.2	2.34	15.1	2.62	5.28	64.6
3	4.7	4.1	3000	34	1.4	9.8	60	0.6	2.8	65	17.2	45	15	2.1	14	1.8	5	56
4	5	2370	3.34	25.43	1.26	8.55	56.32	0.51	1.12	56.14	5.1	40.52	16.13	2.1	11.35	1.21	3.92	52.6
5	4.6	3700	<5	35	1.4	9.6	56	0.5	NT	66	18.4	44	14	<5	15	1.1	NT	54
6	5.52	3570	3.71	34.5	1.45	10.0	62.6	0.65	<5	64.5	20	37.5	15.0	2.2	15.0	<10	<10	57.8
7	NT	NT	NT	NT	NT	NT	NT	NT										
8	NT	NT	NT	NT	NT	NT	NT	NT										
9	<5.0	NT	3.0	NT	1.3	NT	56	0.60	NT	NT	19.0	40	NT	<5.0	NT	NT	NT	53
10	5.18	3230	3.49	29.7	1.45	9.14	60.0	0.59	2.42	61.5	18.7	46.7	11.8	2.12	14.5	2.26	4.48	59.2
11	5.25	2610	3.77	28.1	1.38	9.59	61.8	0.583	<5	63.2	17	45.8	14.3	2.38	12.9	2.15	<10	56.6
12	4.55	3484	3.57	33.4	1.38	9.14	57.7	0.52	3.11	114	18.4	43.9	13.7	2.27	13.6	2.14	4.40	54.9
13	5.7	3100	3.5	30	1.4	9.5	56	0.72	2.4	62	18	40	13	2.0	14	1.7	5.1	50
14	NT	NT	NT	NT	NT	NT	NT	NT										
15	5.3	2200	3.8	25	1.5	8.7	53	0.5	1.4	58	17.3	40	10	1.7	11	2.1	4.4	54
16	5.0	3.2	2800	27	1.3	8.7	54	0.8	2.4	62	16	38	15	2.5	12	1.8	5.6	45
17	5.0	1800	3.4	25.8	1.4	8.3	60.4	0.5	1.2	53.6	16.6	38.6	16.3	2	11.7	2.2	4.9	53.5
18	NT	NT	NT	NT	NT	NT	NT	NT										
19	NT	3300	<25	35.5	1.10	10.0	69.5	0.92	2.80	73.5	18.9	50.5	<100	<100	NT	NT	NT	62.0
21	NT	NT	NT	NT	NT	NT	NT	NT										
22	8	3400	6	32	2.1	10	61	0.63	4	67	19	47	10	4	10	4	4.0	57
24	6.2	2280	3.4	25.8	1.5	9.3	57	0.6	1.8	60.2	18.8	43.6	15.4	2	12.4	2.2	4.8	55.8
25	4.9	3500	3.6	32	1.4	9.4	57	0.6	<5	65	18	46	18	2	16	<10	4.4	57
26	2.5	2700	<4	34	1	9	58	0.54	2	65	18	42	NT	<5	13	2	NT	61
27	6.19	3270	3.58	30.9	1.55	10.9	59.6	0.54	<5	70.5	19	46.8	14.3	2.19	15.7	<10	<10	64.5
28	5.0	2663	<5	27.3	1.3	8.2	53.0	0.50	1.8	54.9	15.8	39.8	18.3	<5	11.8	2.09	4.28	49.62
29	2.04	NT	NT	28.5	1.29	10.5	71.1	NT	NT	58.5	19.2	46.1	11.2	NT	12.4	NT	NT	55.2
30	NT	NT	NT	NT	NT	NT	NT	NT										

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

Table 74 Summary of Participants' Results and Performance for Sample S3

Lab Code	Ca (mg/kg)	Colwell-K (mg/kg)	Colwell-P (mg/kg)	EC (µS/cm)	Exchangeable-Ca (cmol(+)/kg)	Exchangeable-K (cmol(+)/kg)	Exchangeable-Mg (cmol(+)/kg)	Exchangeable-Na (cmol(+)/kg)	Extractable B (mg/kg)	Fe (mg/kg)	K (mg/kg)
A.V.	10000	Not Set	44.8	49.3	24.1	0.416	9.83	0.191	Not Set	45500	771
H.V.	11100	N/A	41.3	42.8	25.5	0.413	10.2	0.212	N/A	55100	652
1	11000	217	45	41.8	25.4	0.44	10.6	0.21	NT	NT	NT
2	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
3	9300	NT	NT	48	24	0.41	9.2	0.18	NT	50000	700
4	10600	NT	NT	69	24.2	0.47	10.31	0.22	NT	45100	830
5	12000	NT	NT	NT	24	0.4	10	<0.17	NT	48000	1500
6	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
7	NR	NR	50	47	25.7	0.40	10.3	0.17	NR	NR	NR
8	13600	NT	NT	45	NT	NT	NT	NT	NT	55600	1270
9	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
10	11500	NR	39	45	23	0.38	9.9	0.21	NR	50900	830
11	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
12	11142	118	47	47.7	21.4	0.37	8.32	0.13	<0.1	53645	841
13	7200	NT	NT	48	24	0.41	9.5	0.18	NT	28000	630
14	8820	NT	NT	54.2	25.3	0.469	9.62	0.215	NT	40800	817
15	7900	NR	NR	NR	NR	NR	NR	NR	NR	33000	540
16	7500	NT	NT	64	22	0.40	8.8	0.15	0.45	36000	760
17	8150	369	45	54	17.1	0.3	5.6	<0.2	<0.2	35900	730
18	NT	NT	33	47	26.0	0.44	10.8	0.21	NT	NT	NT
19	12800	NT	54.5	50	24.7	0.39	11.0	0.19	<1.00	55750	872
21	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
22	8400	NT	NT	580	NT	NT	NT	NT	NT	50000	640
24	9130	NR	NR	56	23.6	0.5	9.7	0.2	NR	37400	720
25	NT	NT	NT	37.7	NT	NT	NT	NT	NT	47000	NT
26	11000	NT	NT	46	NT	NT	NT	NT	NT	55000	890
27	11000	NT	NT	46.4	26.7	0.441	10.7	0.185	NT	54300	877
28	9920.6	NR	NR	54.4	24.0	0.4	9.9	0.2	0.404	38600	815.5
29	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
30	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT

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

Table 74 Summary of Participants' Results and Performance for Sample S3 (continued)

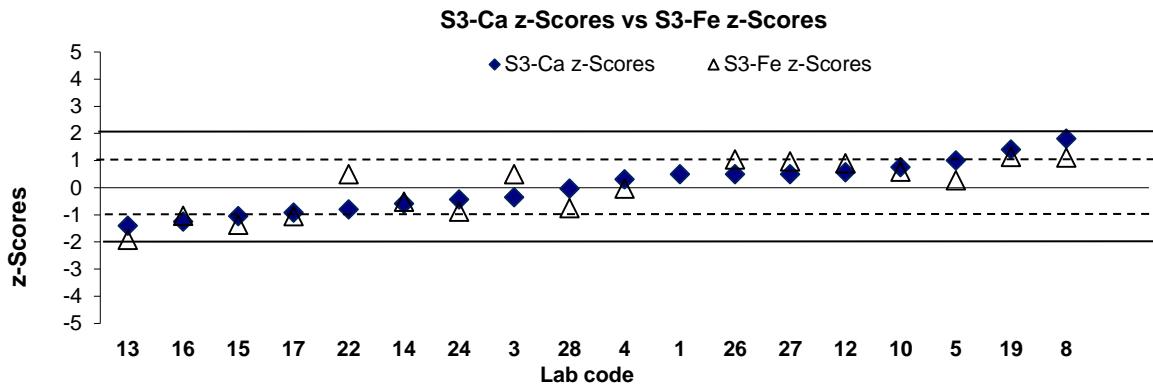
Lab Code	Mg (mg/kg)	Na (mg/kg)	P (mg/kg)	PBI <sub>ColP</sub> (mg/kg)	pH	S (mg/kg)	TC (mg/kg)	TN (mg/kg)	TOC (mg/kg)	Total P (mg/kg)
A.V.	18100	403	896	Not Set	6.98	95.2	10600	844	10600	850
H.V.	19500	360	950	N/A	7.50	117	10300	840	10300	950
1	17000	630	820	109	6.72	98	10600	740	NT	890
2	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
3	18000	330	800	NT	NT	93	9800	940	9000	NT
4	19300	430	930	NT	7.54	90	NT	910	11000	749
5	19000	960	NT	NT	NT	108	NT	NT	NT	510
6	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
7	NR	NR	NR	117.4	7.43	NR	10600	850	10600	NR
8	19500	1610	949	NT	NT	93	NT	850	11000	920
9	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
10	19700	435	988	NR	6.8	114	10000	700	10000	NR
11	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
12	20563	469	986	140	6.57	NT	12200	790	11000	NR
13	14000	300	780	NT	6.6	93	NT	940	10600	NT
14	16400	422	844	NT	6.38	92.3	NT	870	10800	910
15	17000	270	940	NR	NR	81	NR	NR	NR	NR
16	15000	290	760	NT	6.6	88	10000	780	9500	NT
17	17500	360	840	NT	6.8	80	10500	870	10000	805
18	NT	NT	NT	NT	6.8	NT	NT	1020	10900	1060
19	21900	435	1045	113	7.25	108	11500	650	12500	1045
21	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
22	15000	360	780	NT	6.6	22	NT	900	10000	780
24	18300	380	910	NR	7.4	90	NR	100	12000	747
25	NT	NT	950	NT	7.57	NT	NT	NT	NT	NT
26	20000	350	920	NT	6.6	330	NT	NT	NT	NT
27	18900	578	1020	NT	7.56	108	10600	820	10600	NT
28	18948	509.7	865	NR	7.41	93.2	NR	853.3	10440	830.9
29	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
30	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT

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

with various amounts of  $\text{HNO}_3$ ,  $\text{HCl}$ , in combination or alone and most of these methods produce comparable results.

In previous studies conducted by NMI for trace elements in garden soil, compost, sediment or clay, relationships were found to exist between the results reported for Al, Cr, Ni, V and extraction regime employed. In the present study, the samples were dried sediment and moist sediment. Participants used various sample sizes, digestion temperatures and digestion times. No relationship was evident between the results reported for these elements and the digestion regime used.

**Calcium and Iron** level in the agricultural soil sample S3 was very high at 10,000 mg/kg and 45,500 mg/kg respectively and may explain the big variation between laboratories' results, which was much larger than that predicted by Thomson and Horwitz or from previous studies. Laboratories **13** and **16** used a large sample size (of 2.5 g and 5 g respectively), while Laboratory **15** digested their sample only for 30 min. Their extraction regimes may have extracted only a fraction of the acid extractable Ca and Fe from the soil sample and this may explain the low results they reported by them for both tests (Figure 63). When a large sample size is taken for analysis, solubility limitations are expected if these analytes are present in a sample at percentage level.



Lab. Code	S3 Ca mg/kg	S3-Fe mg/kg	Digestion Regime			
			Sample Size (g)	Temp (°C)	Time (min)	Reagents Used
13	7200	NT	2.5	90 - 98	90	3 mL $\text{HNO}_3$ , 3 mL HCl
16	7500	28000	5	95	90	3 mL $\text{HNO}_3$ , 3 mL HCl
15	7900	36000	0.5	96	30	1 mL $\text{HNO}_3$ , 1 mL HCl
17	8150	33000	1	95 ± 5	60	2 mL $\text{HNO}_3$ , 10 mL HCl, 2 mL $\text{H}_2\text{O}_2$
22	8400	35900	2	90	90	2 mL $\text{HNO}_3$ , 6 mL HCl
14	8820	50000	1	95	30	2.5 mL $\text{HNO}_3$ , 2.5 mL HCl
24	9130	40800	1	95	60	2 mL $\text{HNO}_3$ , 10 mL HCl (20%)
3	9300	37400	1 to 3	90-98	120	3 mL $\text{HNO}_3$ , 3 mL HCl
28	9920.6	50000	1	95	60	2 mL $\text{HNO}_3$ (1:1), 10 mL HCl (1:1), 2 mL $\text{H}_2\text{O}_2$
4	10600	38600	1	95	60	2 mL $\text{HNO}_3$ (1:1), 10 mL HCl (1:1), 2 mL $\text{H}_2\text{O}_2$
1	11000	45100	1	170	15	8 mL $\text{HNO}_3$ (1:1), 2 mL HCl (1:1)
26	11000	55000	0.5	95	120	7.5 mL $\text{HNO}_3$ , 5 mL HCl, 1.5 mL $\text{H}_2\text{O}_2$
27	11000	54300	2	95-105	60	20 (Aqua regia)
12	11142	53645	0.4	120	60	2.5 mL $\text{HNO}_3$ , 7.5 mL HCl
10	11500	50900	1	100	120	3 mL $\text{HNO}_3$ , 3 mL HCl, 10mL $\text{H}_2\text{O}_2$
5	12000	48000	0.5	95	120	2 mL $\text{HNO}_3$ , 2 mL HCl, 10mL $\text{H}_2\text{O}_2$
19	12800	55750	1	98	150	5 mL $\text{HNO}_3$ , 5 mL HCl
8	13600	55600	1	112.5	120	2.5 mL $\text{HNO}_3$ , 7.5 mL HCl

Figure 63 Participants' Results/Performance for Ca and Fe in S3 versus Digestion Regime

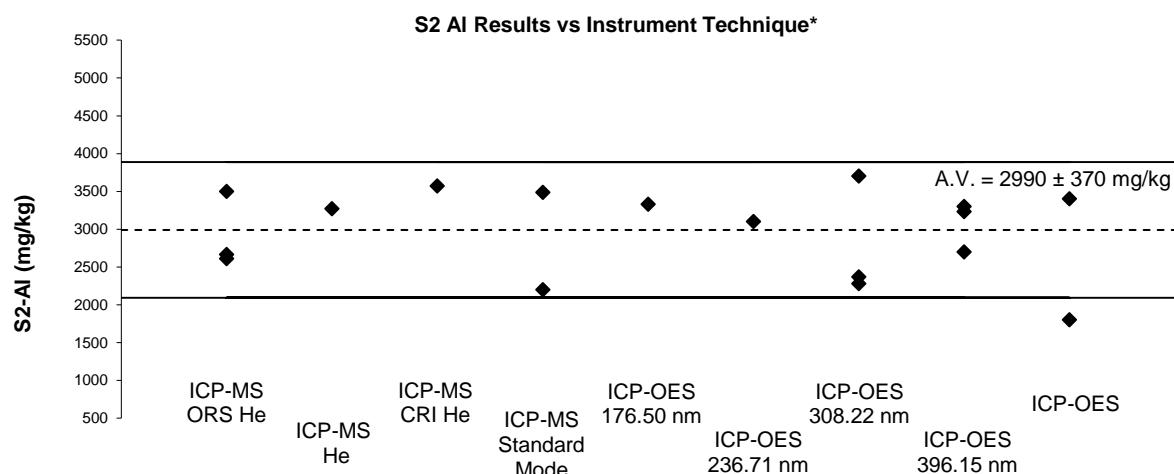
Most of the results reported for Ca and Fe that returned z-scores within the range (-1 to 1) were from participants who used dilute acids or reported using a staggered digestion regime (added water during the extraction process). Al, Ca, Fe and P can form insoluble oxides in hot concentrated  $\text{HNO}_3$ .<sup>16-19</sup>

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

### Individual Element Commentary

**Aluminium** Calculation error and/or reporting results in the wrong units may explain the unsatisfactory results reported by Laboratories **3** and **16**. The results they reported were lower than the assigned value by a factor of approximately 1,000.

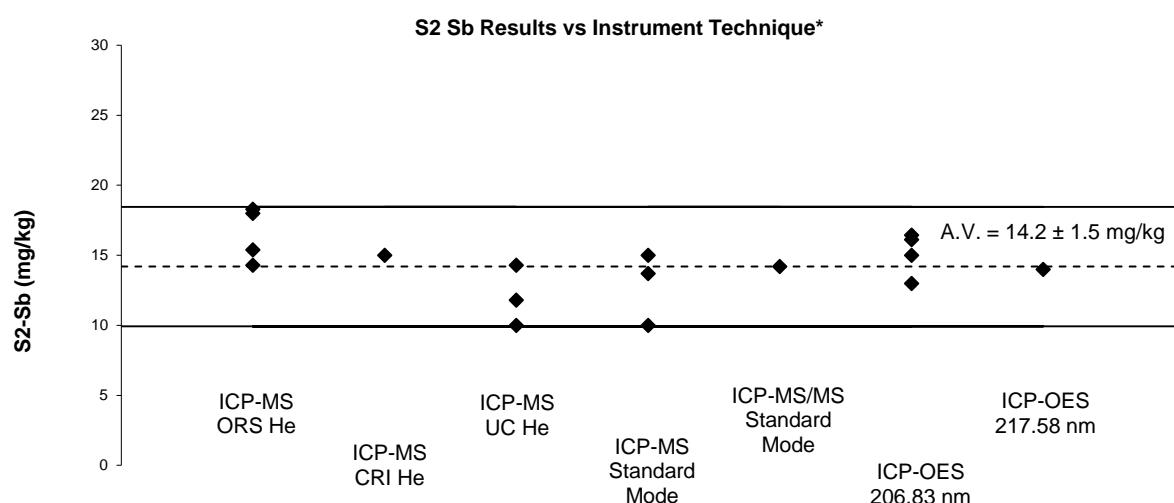
Plots of results for Al versus instrumental technique used are presented in Figure 64.



\*Results from laboratories 3 and 16 were excluded. ICP-MS DRC He, plotted as ICP-MS UC He

Figure 64 Al Results vs. Instrumental Technique

**Antimony** The between-laboratory CV for Sb in S2 was 18%, which is higher than the Thomson/Horwitz CV of 11%. There was no evident relationship between participants' results for Sb and the instrumental technique they used (Figure 65).

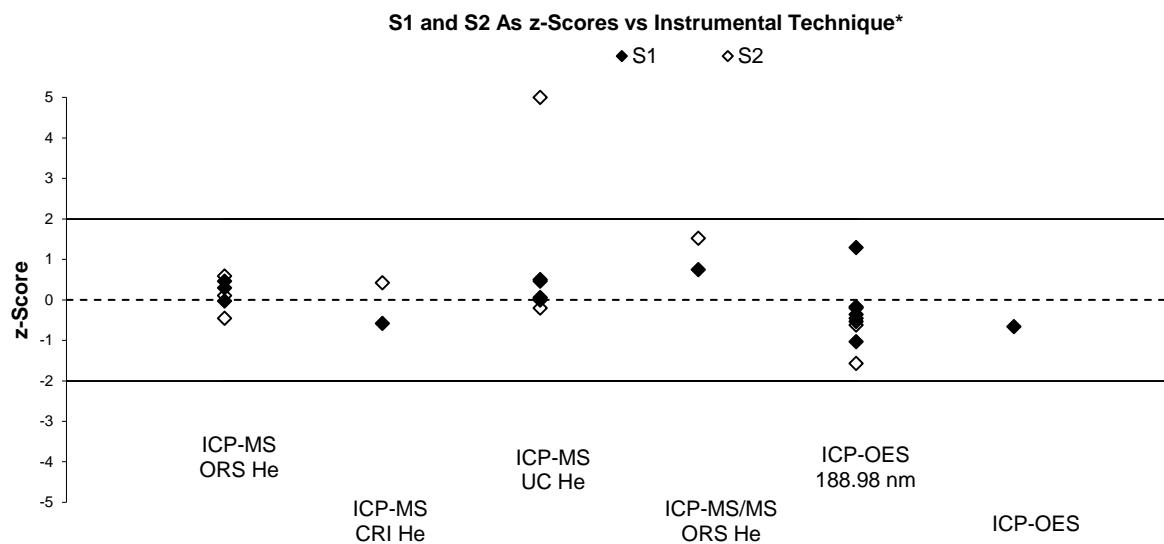


\*ICP-MS DRC He, plotted as ICP-MS UC He

Figure 65 Sb Performance in S1 vs. Instrumental Technique

**Arsenic** measurements in the two study samples did not present difficulty to participating laboratories, with the between-laboratory CV being half of that predicted by Horwitz.

Figure 66 presents plots of participants' z-scores versus instrumental technique used.



\*z-Score >5 has been plotted as 5. ICP-MS DRC He, plotted as ICP-MS UC He

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

**Boron** level in Sample S1 was low, which may have presented difficulty to some laboratories. The between-laboratory CV was high (26%).

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

Plots of participants' results versus instrumental technique used are presented in Figure 67.

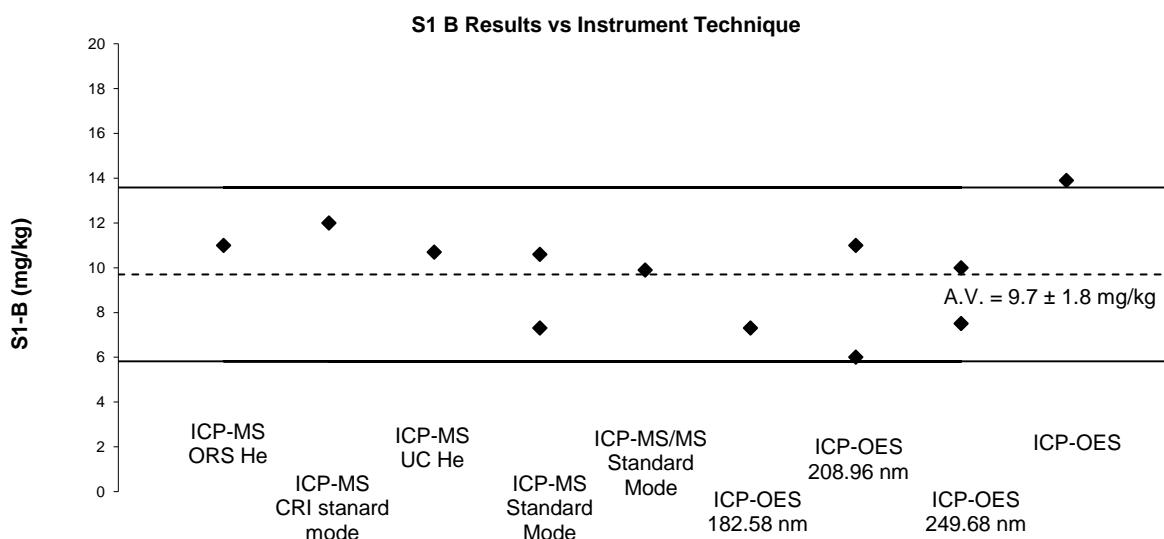
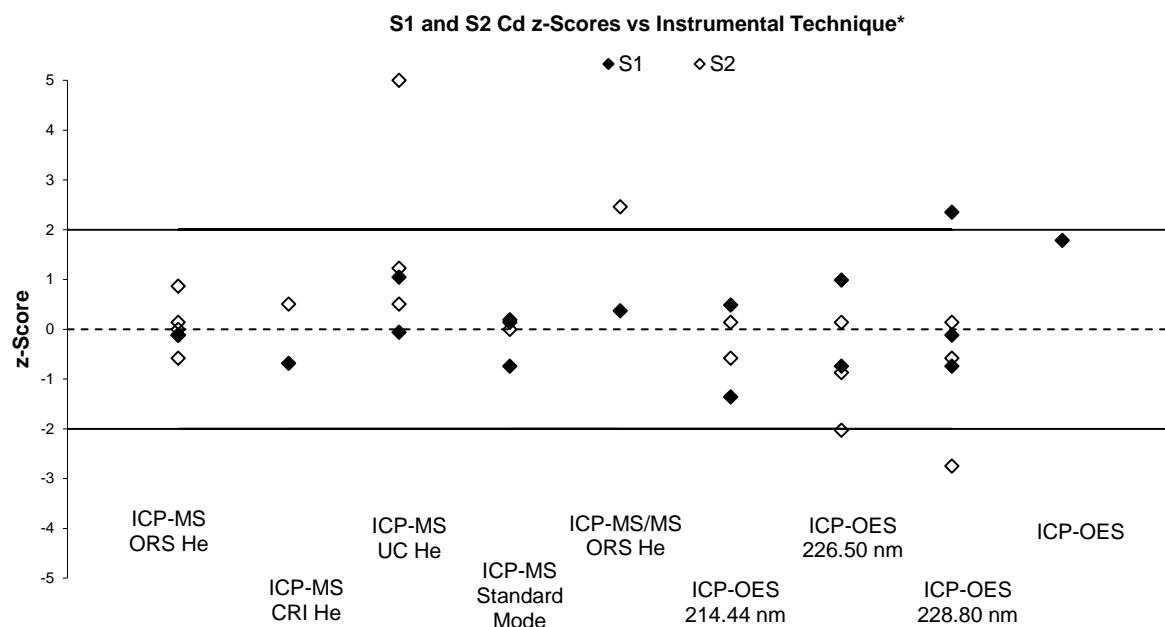


Figure 67 B Results in S1 vs. Instrumental Technique

**Cadmium** Figure 68 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

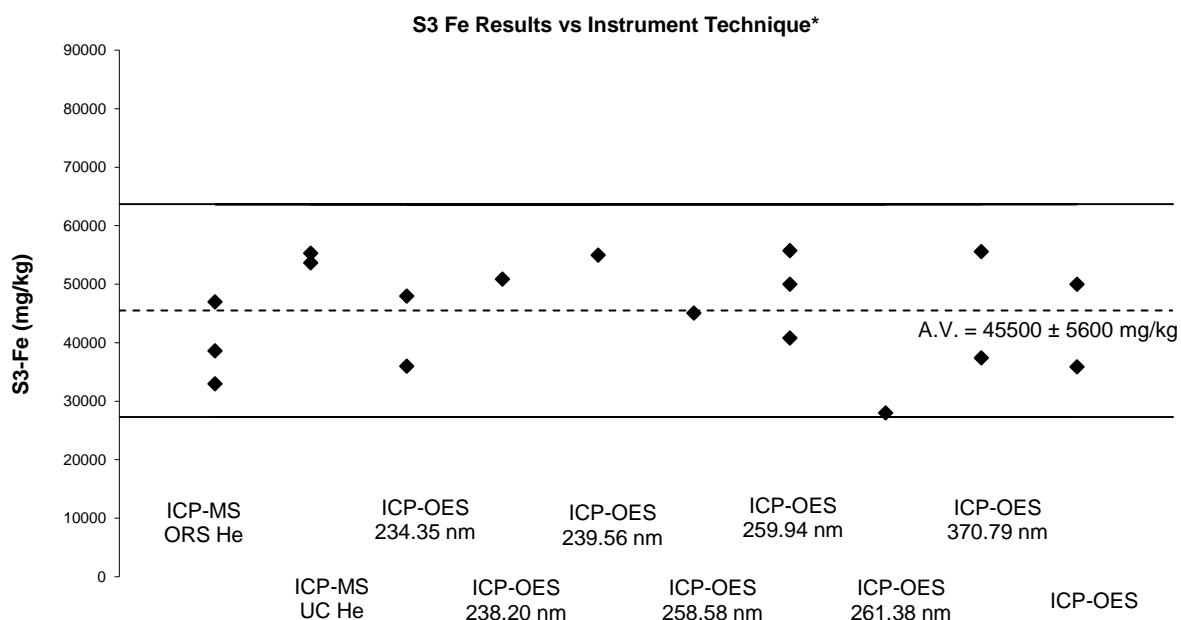
228.804 nm and Fe 214.445 nm respectively. If ICP-OES is used with one of these two wavelengths then online inter-element correction may be required as soil is usually rich in Fe.



\*z-Score >5 has been plotted as 5. ICP-MS DRC He, plotted as ICP-MS UC He

Figure 68 Cd Performance in S1 and S2 vs. Instrumental Technique

**Iron** level in the study sample S3 was high (45,500 mg/kg). Plots of participants' results versus instrumental technique used are presented in Figure 69. ICP-OES with various wavelengths was the instrumental technique chosen by the majority of participating laboratories. Five laboratories used ICP-MS and some reported using dilutions factors of up to 25,000. No relationship between Fe results and instrumental technique used was evident.



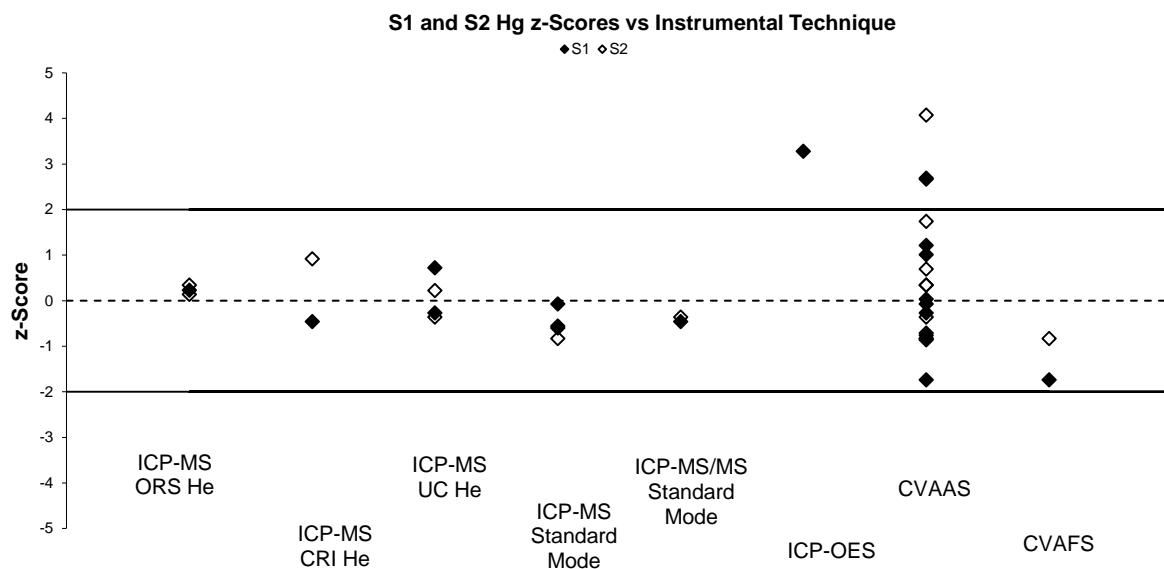
\*ICP-MS DRC He, plotted as ICP-MS UC He

Figure 69 Fe Results vs. Instrumental Technique

**Lithium** The results reported for Li in S2 were too variable, with a between-laboratory CV of 36%, and hence no assigned value could be set for this test. ICP-MS has low sensitivity for light elements due to space-charge effects. An internal standard with similar behaviour may overcome this problem.

**Mercury** ICP-MS in standard mode and CVAAS were the preferred measurement techniques (Figure 70). The results reported by Laboratory 19 were high and returned unsatisfactory z-scores in both samples S1 and S2. Expired calibration standards, when not prepared fresh from stock solutions before measurement, are the most common cause of high Hg results. Laboratory 19 may need to check their procedure for Hg analysis.

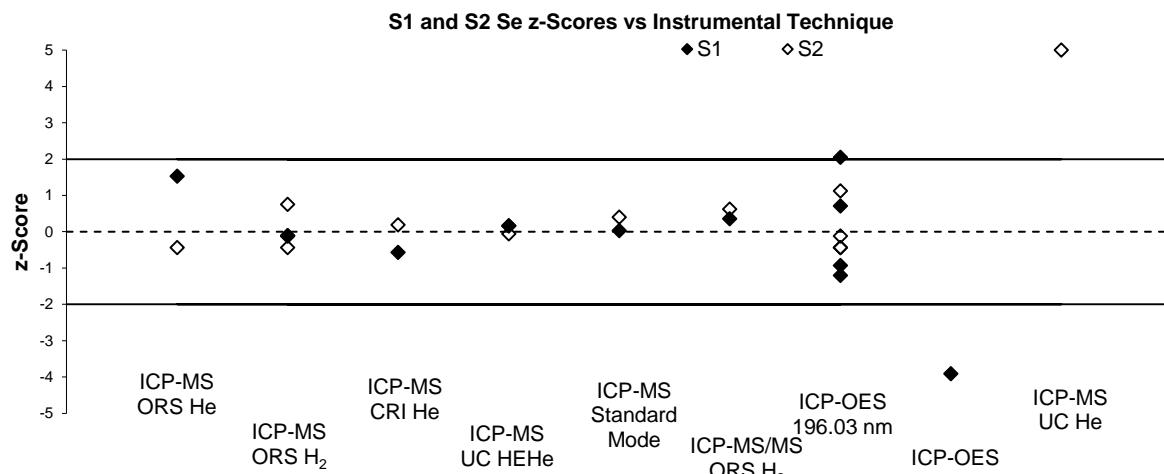
ICP-OES may not be the right technique for measurements of low level Hg in the sediment samples as it is not sensitive enough.



ICP-MS DRC He, plotted as ICP-MS UC He

Figure 70 Hg Performance in S1 and S2 vs. Instrumental Technique

**Selenium** Although Se was at similar levels in both S1 and S2, the between-laboratory CV for Se in S2 was 8.9%, and almost double in S1 at 16%. A large number of results were produced by ICP-OES measurements and all but two retuned satisfactory z-scores (Figure 71).



\*z-Score >5 has been plotted as 5. ICP-MS DRC HEHe, plotted as ICP-MS UC HEHe

Figure 71 Se Performance in S1 and S2 vs. Instrumental Technique

**Potassium and Sodium** emission signals are significantly enhanced in the presence of other easily ionised elements. Concentrations much higher than the true values are frequently obtained for these two tests when measurements are made by ICP-OES with axial view plasma (ICP-OES-AV) without an ionisation buffer or correction equation. Plots of participants' results for Na and K versus instrumental technique used are presented in Figures 72 and 73.

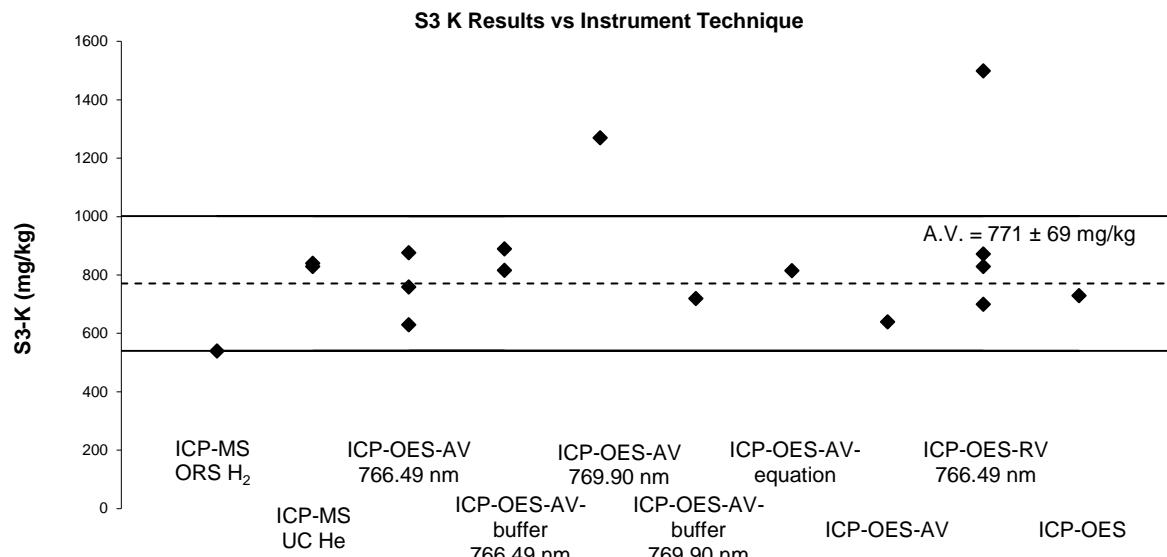


Figure 72 K Results vs. Instrumental Technique

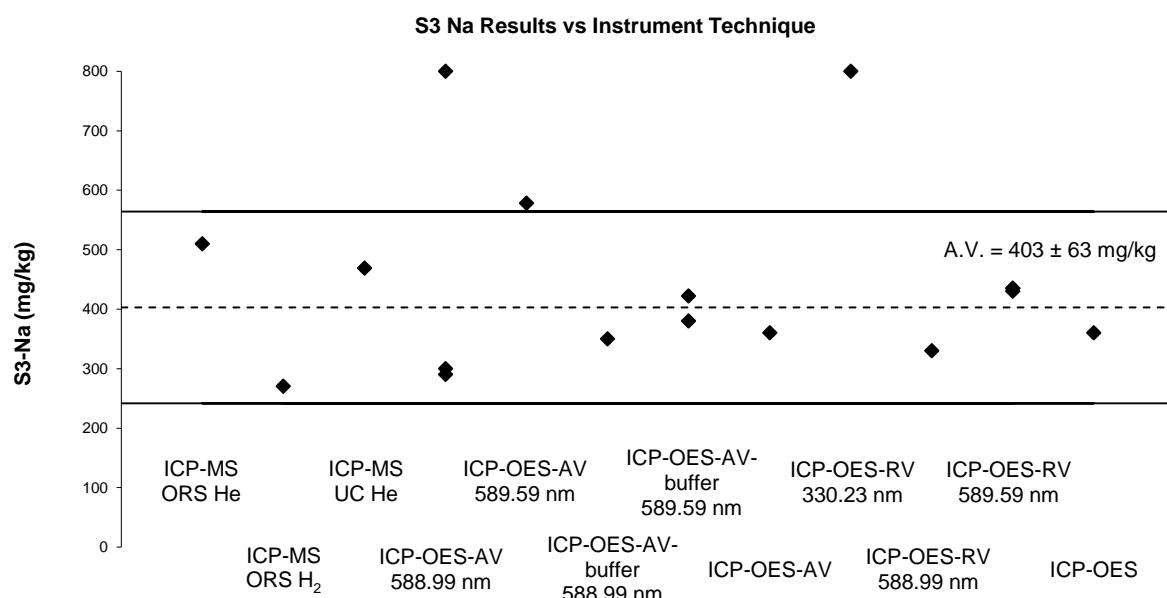


Figure 73 Na Results vs. Instrumental Technique

**Moisture content** Measurement of moisture content in the test sample S2 did not present significant difficulty to participating laboratories. All results reported for moisture content returned satisfactory z-scores except for one.

**Thorium** No assigned value was set for Th because the results reported for this element were not compatible with each other. No relationship between Th results, extraction method and instrumental technique used was evident.

## 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”.<sup>21</sup>

The method descriptions provided by participants are presented in Table 10. With two exceptions, all participants used a ratio sample mass/extraction solution of 1 to 20 and shook the sample for 1-2 min. Laboratory **14** used a ratio of 2:20 for sample mass/extraction solution and Laboratory **19** used a ratio of 2:10. Plots of participants' results versus the analytical methods used for the exchangeable bases measurement are presented in Figures 74 to 77.

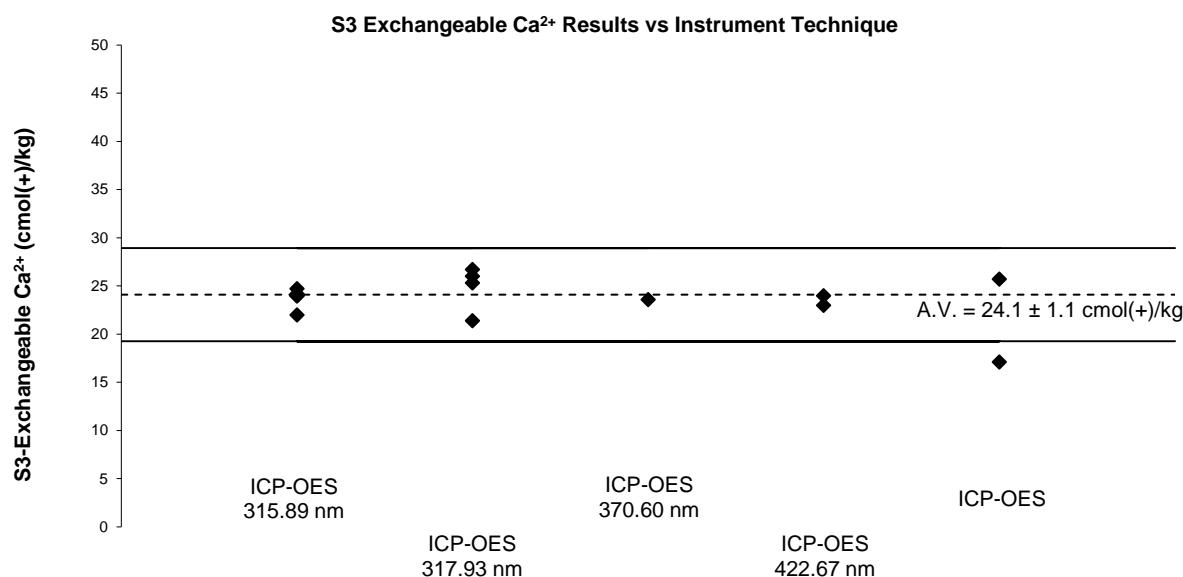


Figure 74 Exchangeable Ca<sup>2+</sup> Results vs. Analytical Methods

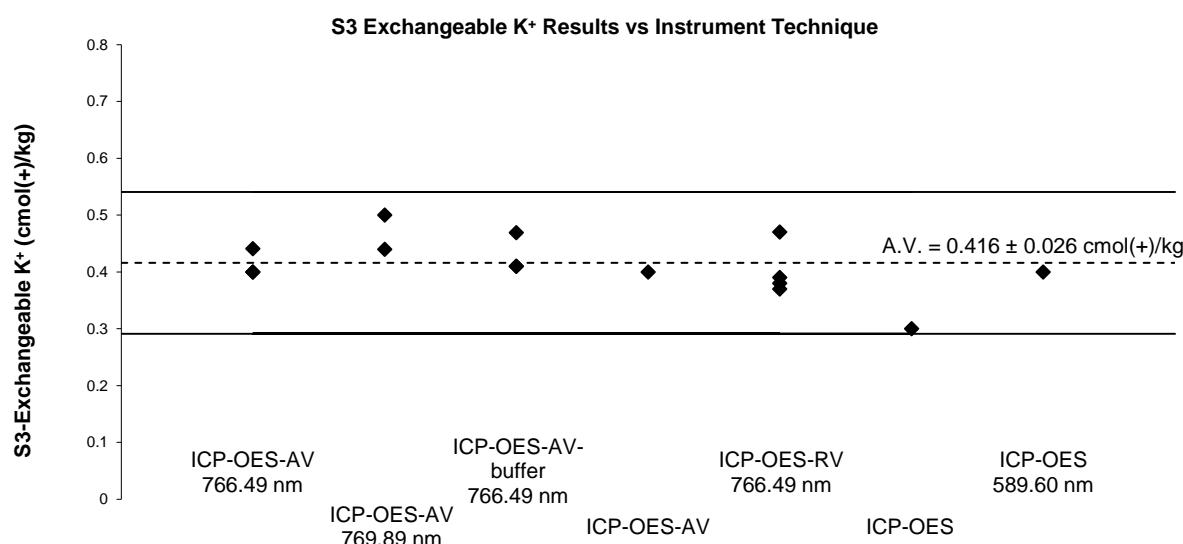


Figure 75 Exchangeable K<sup>+</sup> Results vs. Analytical Methods

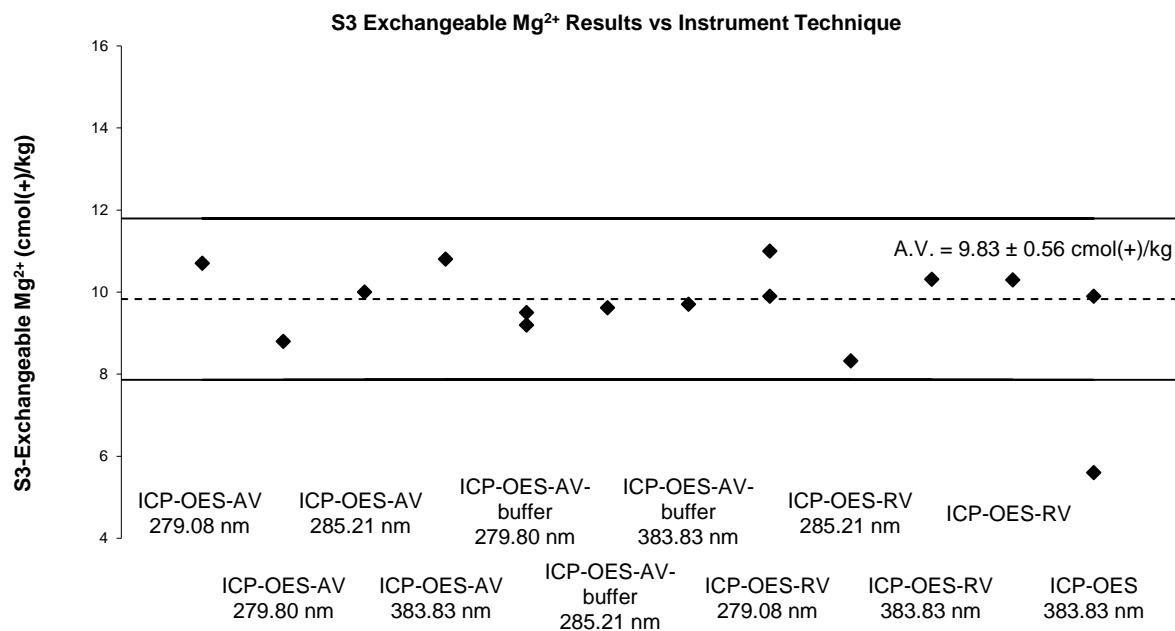


Figure 76 Exchangeable Mg<sup>2+</sup> Results vs. Analytical Methods

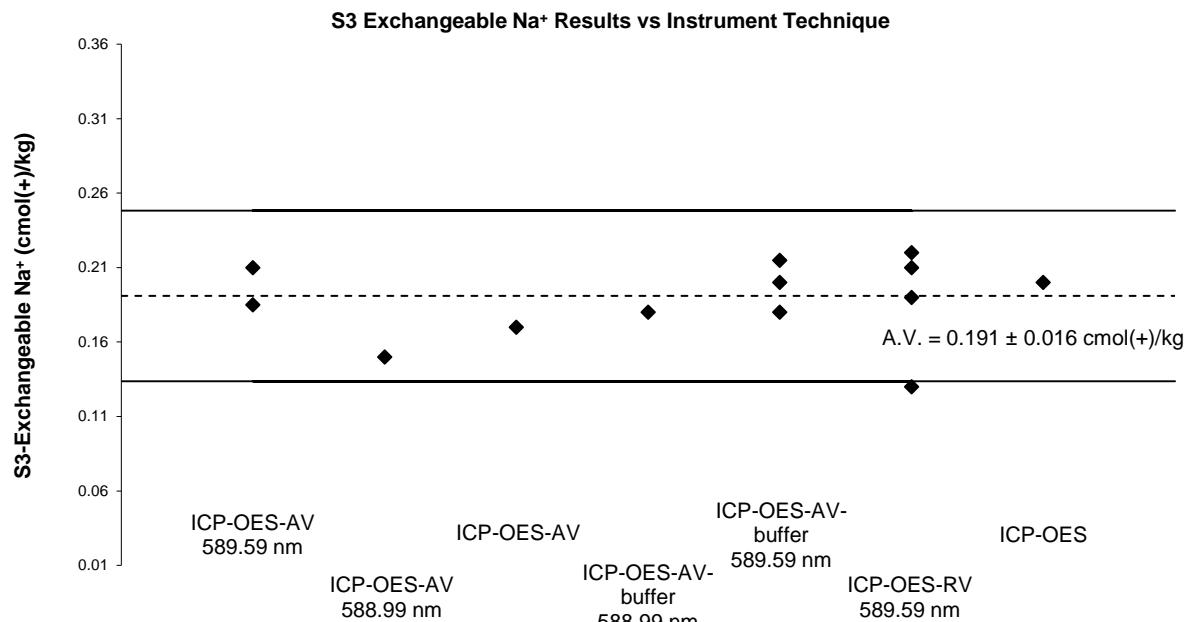


Figure 77 Exchangeable Na<sup>+</sup> Results vs. Analytical Methods

## 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”.<sup>21</sup> All participants shook the sample for 16 hours and used a ratio of 1 : 100 sample mass/extraction solution (Table 5).

**Colwell K** Three participants extracted K in S3 using 0.5 M NaHCO<sub>3</sub> and reported results for this test. Two laboratories used ICP-OES to measure Colwell K and one used ICP-MS. The three results were in a relative agreement with each other, centred on 220 mg/kg (Figure 78).

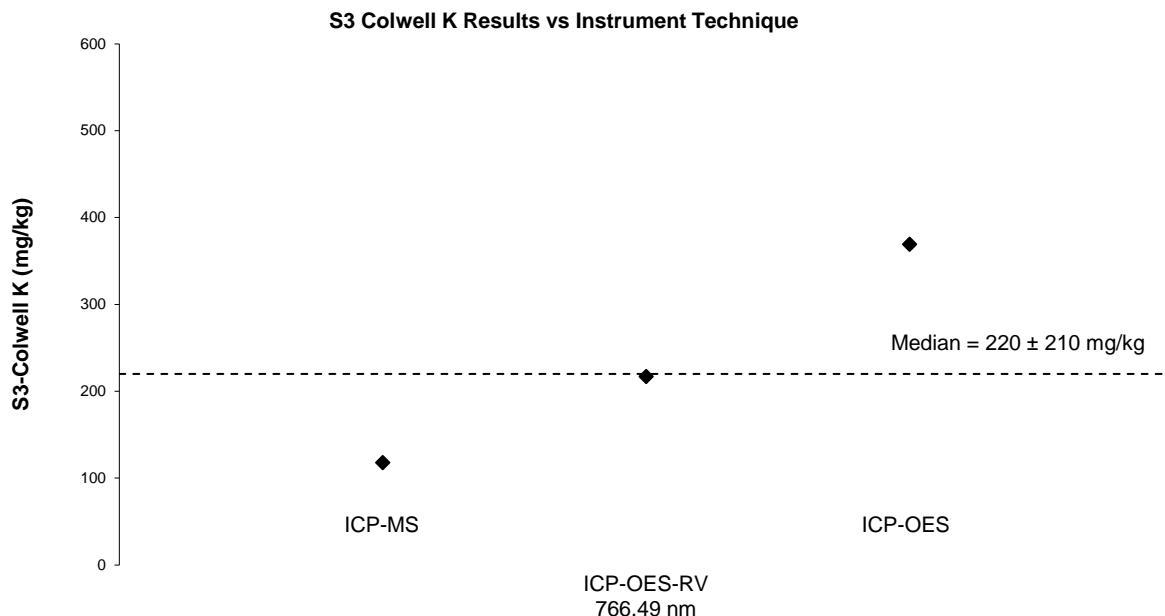


Figure 78 Colwell K Results vs. Instrumental Technique

**Colwell P** Seven results were reported for Colwell P in S3 and all were compatible with each other and with the assigned value of 44.8 mg/kg. Plots of participants' results versus the instrumental technique used are presented in Figure 79.

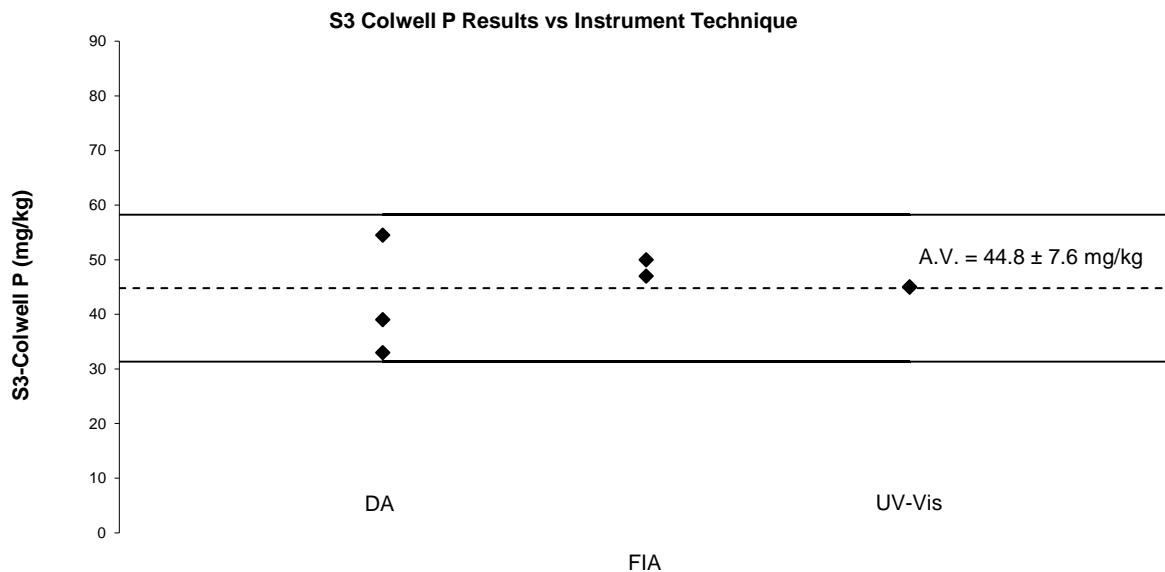


Figure 79 Colwell P Results vs. Instrumental Technique

## 6.8 Participants' Results and Analytical Methods for Phosphorus Buffer Index-PBI<sub>+</sub>ColP

P Buffer Index-PBI<sub>+</sub>ColP gives an indication of soil ability to fix P and make it unavailable to plant uptake. Four laboratories reported results for this test. The results were in relatively good agreement with each other (CV13%), centred on the value of 115 mg/kg (Figure 80).

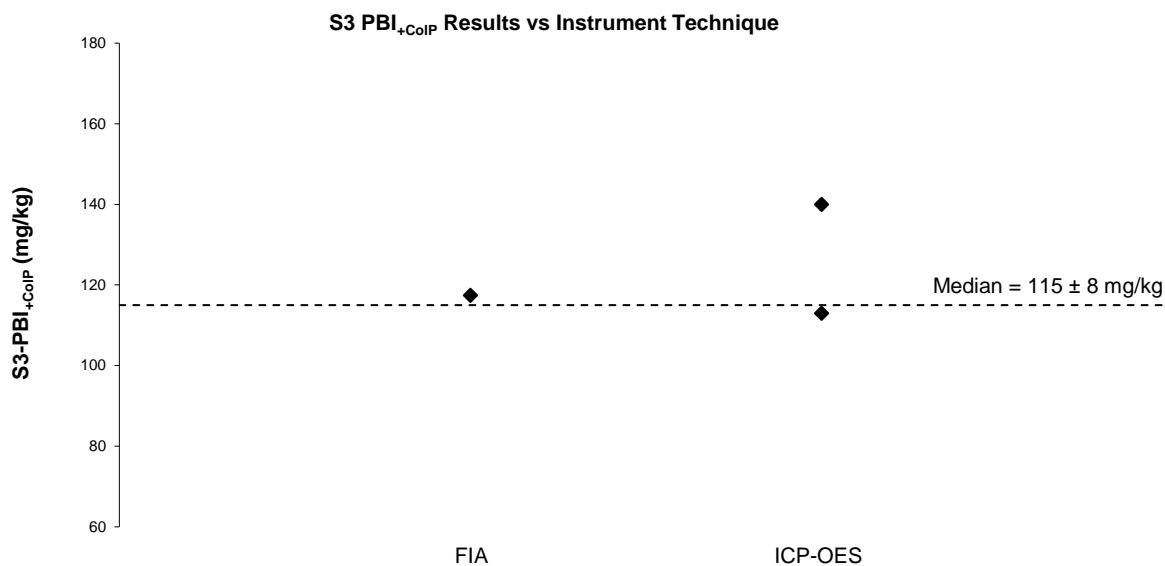


Figure 80 PBI Results vs. Instrumental Technique

## 6.9 Participants' Results and Analytical Methods for Total P

**Total P** assigned value was 850 mg/kg. Eleven participants reported results for total P and all performed satisfactorily but one (Figure 81).

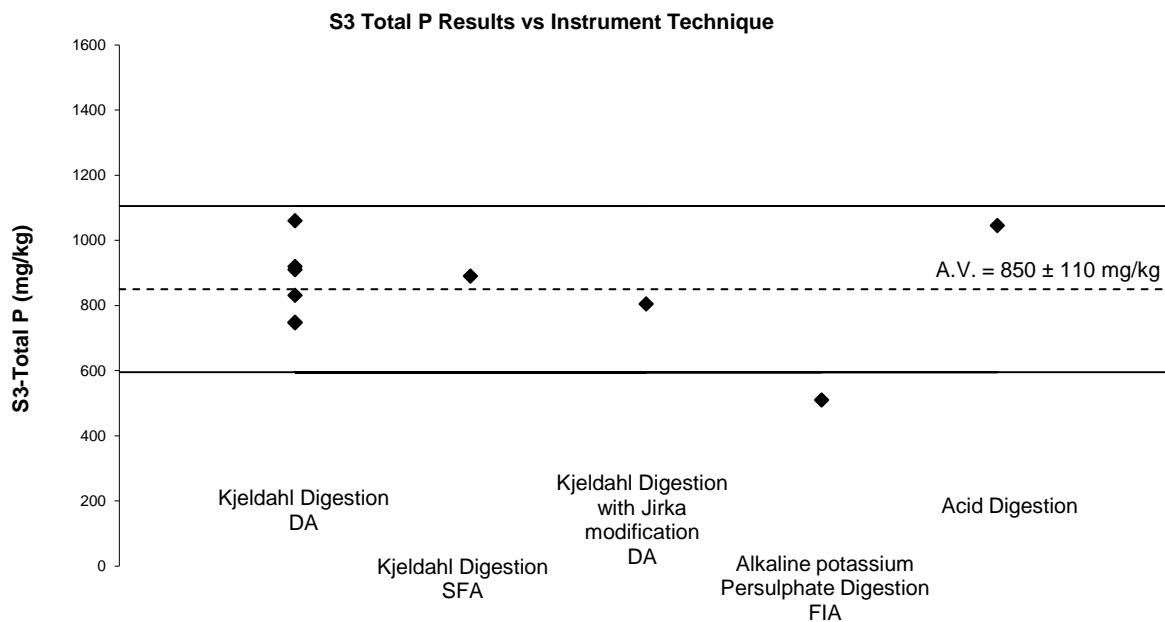


Figure 81 TP Results vs. Analytical Method

## 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<sub>x</sub>. The method descriptions provided by participants are presented in Table 9. A plot of participants' results versus analytical method and measurement technique used for TN analysis in S3 is presented in Figure 82.

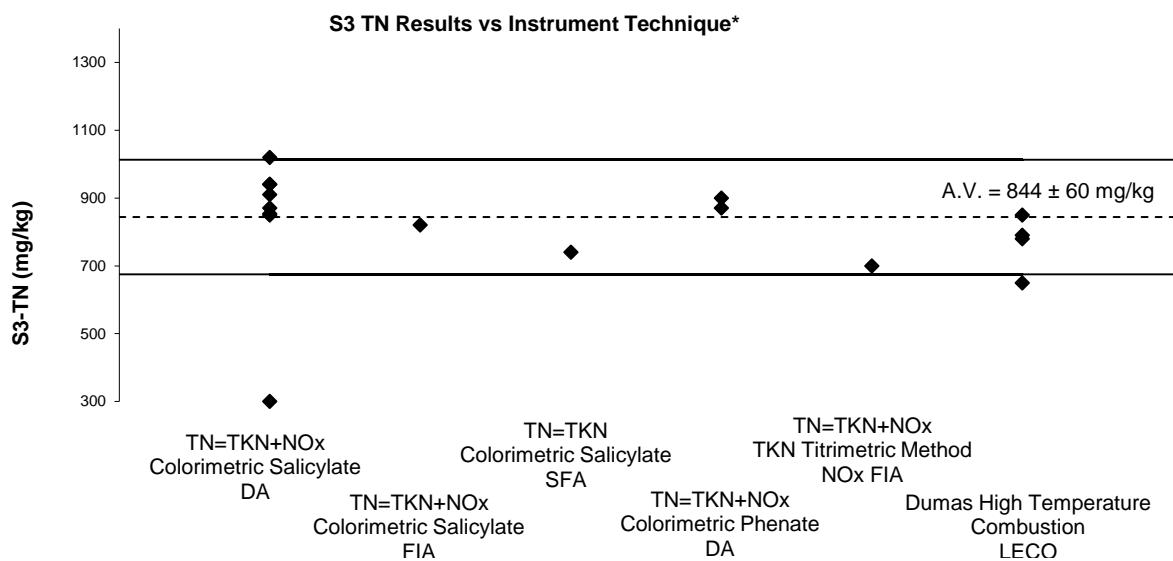


Figure 82 TN Results vs. Analytical Method

## 6.11 Participants' Results and Analytical Methods for Total Carbon and Total Organic Carbon

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

**Total Carbon** All reported results for TC in S3 returned satisfactory z-scores.

**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.<sup>21-24</sup>

Eight participants used a high temperature oxidation method and seven used a chemical oxidation method based on the “Walkley-Black” method (Figure 83).

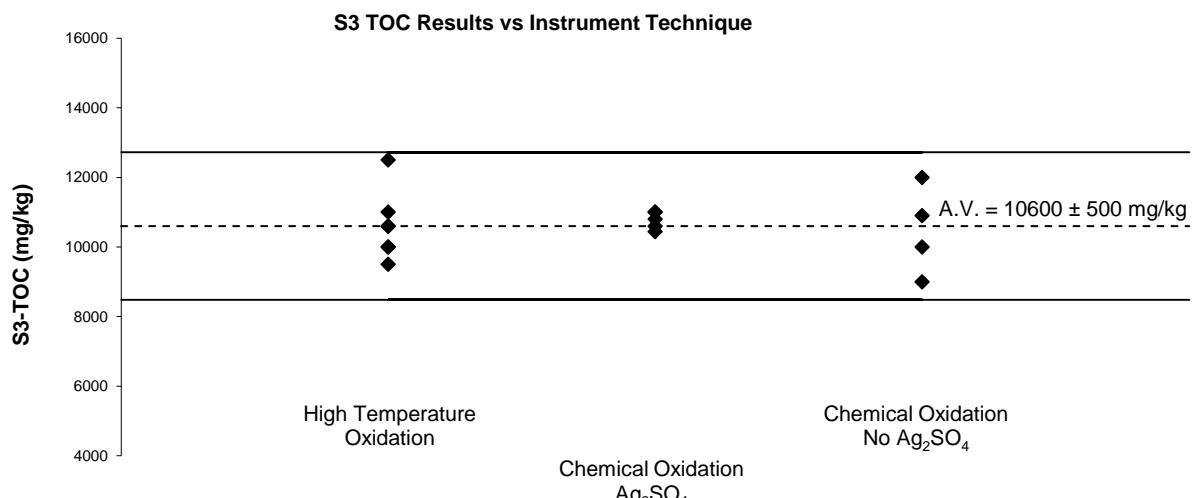


Figure 83 TOC Result vs. Analytical Method

The high temperature oxidation method for organic carbon determination can be rapid and reliable when inorganic carbon is removed prior to combustion. The separation of organic carbon from inorganic carbon can be achieved by ashing or acid treatment. One participant reported: “Sample was Fizz test with 4 M HCl and no Fizzing observed. Therefore no acid treatment was carried for TOC”.

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.<sup>24, 25</sup>

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.<sup>24, 25</sup>

## 6.12 Comparison with Previous NMI Proficiency Tests of Metals in Soil

AQA 22-02 is the thirtieth NMI proficiency test of metals in soil.

Participants' performance in measurement of metals in soil over the last ten years is presented in Figure 84. 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 75).

Table 75 Control Samples Used by Participants

Lab. Code	Description of Control Samples
1	PACS3
4	CRM
5	CRM – NMI RM- AGAL -12
6	Spiked Sample
7	CRM - ASPAC 7098-C1 ASPAC 7118
8	CRM
9	RM – Previous AQA PT scheme samples
10	CRM – agal10 & agal12
11	CRM – ICV 1, CRM540
12	RM – AGAL 12
13	Spiked Sample

14	RM
15	Spiked Sample
17	CRM
19	CRM – AGAL 12 & LOAM B
21	CRM – Agal-12 Biosoil
22	CRM
24	CRM
25	AGAL 10 & AGAL 12
26	CRM – CRM036
27	Spiked Sample
28	RM

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

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

Surplus test samples from this study are available from NMI.

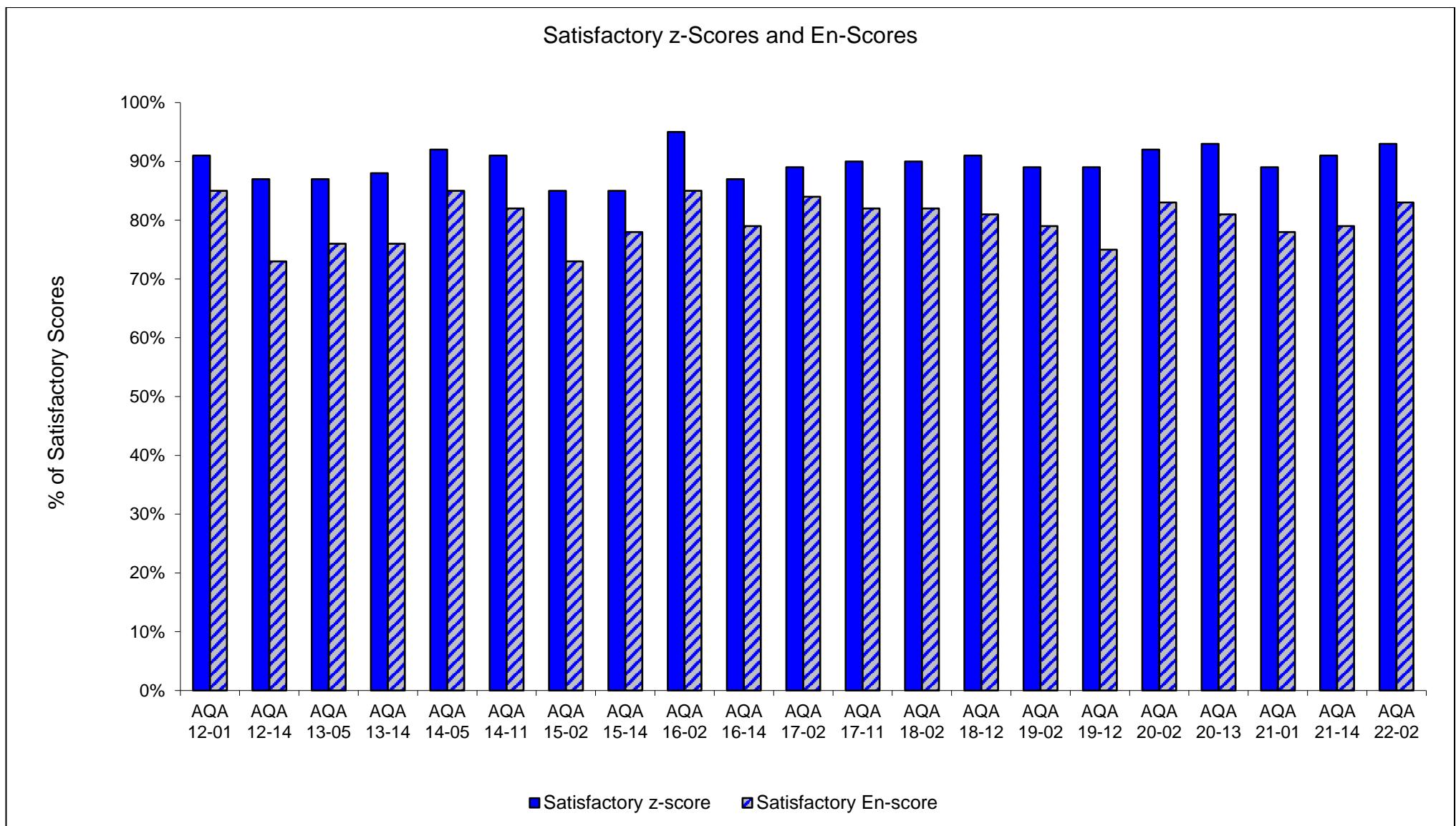


Figure 84 Participants' Performance over Time (2012-2022)

## 7 REFERENCES

- [1] ISO/IEC 17043:2010, Conformity assessment – *General requirements for proficiency testing*.
- [2] NMI 2016, *NMI Chemical Proficiency Testing Study Protocol*, viewed 9 May 2022, <<http://www.industry.gov.au>>.
- [3] NMI 2016, *NMI Chemical Proficiency Testing Statistical Manual*, viewed 9 May 2022, <<http://www.industry.gov.au>>.
- [4] Thompson, M, Ellison, S & Wood, R 2006, ‘The international harmonized protocol for proficiency testing of (chemical) analytical laboratories’, *Pure Appl. Chem*, vol 78, pp 145-196.
- [5] National Environmental Protection Council, *NEPM Schedule B (1) Guidelines on the Investigation Levels for Soil and Groundwater*, viewed 20 February 2020, <[https://www.legislation.gov.au/Details/F2013C00288/Html/Volume\\_2](https://www.legislation.gov.au/Details/F2013C00288/Html/Volume_2)>.
- [6] ISO 13528:2015(E), *Statistical methods for use in proficiency testing by interlaboratory comparisons*.
- [7] Thompson, M, Ellison 2000, ‘Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing’, *Analyst*, vol 125, pp 385-386.
- [8] ISO/IEC 17025:2018, *General requirements for the competence of testing and calibration laboratories*.
- [9] Eurachem/CITAC Guide, *Quantifying uncertainty in analytical measurement* 3<sup>nd</sup> edition, viewed 9 May 2022, <<http://www.eurachem.org>>.
- [10] Bertil, M, Naykki, T, Hovind, H & Krysell, M 2012, *Nordtest Report Handbook for Calculation of Measurement Uncertainty in Environmental Laboratories TR 537* 3<sup>rd</sup> Edition Nordest Tekniikantie, Finland, Esopo,.
- [11] Hibbert, B 2007, *Quality Assurance for the Analytical Chemistry Laboratory*, Oxford University Press.
- [12] ISO (2008), *Guide to the Expression of Uncertainty in Measurement (GUM)*, Geneva, Switzerland.
- [13] Eurolab 2002, Technical Report No 1/2002 - *Measurement Uncertainty in Testing*.
- [14] NMI, *Estimating Measurement Uncertainty for Chemists* – viewed 9 May 2022, <[www.industry.gov.au/client-services/training-and-assessment](http://www.industry.gov.au/client-services/training-and-assessment)>.
- [15] ASTM E2554-13, *Standard Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques*.
- [16] Roje, V.,(2010) *Multi-elemental analysis of marine sediment reference material MESS-3: one-step microwave digestion and determination by high resolution inductively coupled plasma-mass spectrometry (HR-ICP-MS)*, Chemical papers 64 (4) 409-414
- [17] Cotton, F. A., Wilkinson, G., (1998) *Advanced Inorganic Chemistry*, ( 4<sup>th</sup> ed, p394-401). NY, USA

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

## **APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING**

### **Sample Preparation**

**Sample S1** was a sediment material fortified for 24 elements, dried, ground and passed through a 212 µm sieve prior to being divided into portions of approximately 30 g each.

**Sample S2** was the dried sediment sample S1 to which a known amount of water was added. The moist sediment was mixed and divided into portions of 35 g each.

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

### **Sample Analysis and Homogeneity Testing**

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

No homogeneity testing was carried out for Sb, calcium chloride-extractable B, Colwell K and PBI.

### **Sample Analysis for Acid Extractable Elements**

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

Test portions of approximately 0.5 g for the dried sediment sample and 1.5 g for the moist soil sample were weighed into a 50 mL graduated polypropylene centrifuge tube. The samples were digested using 3 mL of concentrated nitric acid and 3 mL of concentrated hydrochloric acid on a hot block at 95°C ± 5°C. After digestion, each sample was diluted to 40 mL with Milli-Q water and then further diluted as necessary for ICP-MS determination.

The measurement instrument was calibrated using external standards for targeted analytes. A set of quality control samples consisting of blanks, blank matrix spike, matrix matched reference materials, duplicates and sample matrix spikes, was carried through the same set of procedures and analysed at the same time as the samples. A summary of the instrument condition used and the ion/wavelength monitored for each analyte is given in Table 76.

Table 76 Instrumental Technique used for Acid Extractable Elements

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

Ca	ICP-OES	Y	NA	NA	800	NA	422.673 m,
Cd	ICP-MS	Rh	NA	He	800	800	111 m/z
Co	ICP-MS	Rh	ORS	He	NA	800	59 m/z
Cr	ICP-MS	Rh	ORS	He	800	NA	52 m/z
Cu	ICP-MS	Rh	ORS	He	800	800	63 m/z
Fe	ICP-OES	Y	NA	NA	800	NA	238.204 nm
Hg	ICP-MS	Rh	NA	He	800	800	201 m/z
K	ICP-MS	Rh	ORS	He	800	NA	39 m/z
Li	ICP-MS	Rh	ORS	He	NA	800	7 m/z
Mg	ICP-OES	Y	NA	NA	800	NA	285.213 nm
Mn	ICP-MS	Rh	ORS	He	800	800	55 m/z
Mo	ICP-MS	Rh	ORS	He	800	NA	95 m/z
Na	ICP-MS	Rh	ORS	He	800	NA	23 m/z
Ni	ICP-MS	Rh	ORS	He	800	800	60 m/z
P	ICP-MS	Rh	ORS	HEHe	800	NA	31 m/z
Pb	ICP-MS	Ir	NA	He	800	NA	Average of 206, 207 m/z
Rb	ICP-MS	Rh	ORS	He	800	NA	85 m/z
S	ICP-OES	Y	NA	NA	800	NA	181.972 nm
Se	ICP-MS	Rh	ORS	HEHe	800	800	78 m/z
Sn	ICP-MS	Rh	NA	He	800	NA	118 m/z
Sr	ICP-MS	Rh	ORS	He	NA	800	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	NA	He	NA	800	238 m/z
V	ICP-MS	Rh	ORS	He	800	NA	51 m/z
Zn	ICP-MS	Rh	ORS	He	800	800	64 m/z

### Sample Analysis for Exchangeable Bases

A test portion of 5 g was weighed into a 100 mL polypropylene container. The container was then filled with 100 mL 1M NH<sub>4</sub>Cl. The suspension was shaken, at room temperature for 1 h, centrifuged, and filtered through 0.45 µm filter. A summary of the measurement techniques used is presented in Table 77.

Table 77 Instrumental Technique used for Exchangeable Bases

Analyte	Instrument	Internal Standard	Final Dilution Factor	Wavelength nm
Exchangeable Ca <sup>2+</sup>	ICP-OES	Y	40	315.887
Exchangeable Mg <sup>2+</sup>	ICP-OES	Y	40	279.8
Exchangeable Na <sup>+</sup>	ICP-OES	Y	40	588.995
Exchangeable K <sup>+</sup>	ICP-OES	Y	40	766.491

### Sample Analysis for Total Carbon and Total Organic Carbon

The measurements for TC and TOC were made using NMI Method NWS15.<sup>32</sup> For TOC measurements a portion of sample weighing 0.25 g was reacted for 12 hours with 20 mL diluted hydrochloric acid to remove inorganic carbon. The sample was further purged with nitrogen gas to remove the inorganic carbon in solution and further diluted with 20 mL Milli-Q water. The insoluble part was then filtered and collected on a filter, dried and

analysed as total carbon (TC). The TOC was calculated as the sum of the TOC from the insoluble part and the dissolved organic carbon (DOC) from liquid solution.

### **Sample Analysis for Total Nitrogen**

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

Organic nitrogen from a test portion of 1 g was converted to ammonia with 50 mL digestion reagent (potassium sulfate, sulfuric acid and cupric sulfate) on a block digester at 400 °C ± 5 °C for 4 hours. The digested solution was then made alkaline with sodium hydroxide solution, distilled into a steam distillation analyser unit and automatically titrated with standard hydrochloric acid to the end point. The amount of ammonia nitrogen was then calculated.

For NOx measurements a test portion of 10 g was weighed into a 100 mL polypropylene container. The container was then filled with 95 mL Milli-Q water. The suspension was shaken, at room temperature for 1 h, centrifuged, and filtered through 0.45 µm filter. NO<sub>3</sub><sup>-</sup>-N was further measured by cadmium reduction to NO<sub>2</sub><sup>-</sup>-N followed by NO<sub>x</sub> (the reduced NO<sub>2</sub><sup>-</sup>-N plus original NO<sub>2</sub><sup>-</sup>-N) measurements by FIA.

## APPENDIX 2 - ASSIGNED VALUE, Z-SCORE AND E<sub>n</sub> SCORE CALCULATION

The assigned value was calculated as the robust average using the procedure described in 'ISO 13258:2015(E)<sup>8</sup>; the uncertainty was estimated as:

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

where:

$u_{rob\ av}$  robust average standard uncertainty

$S_{rob\ mean}$  robust average standard deviation

$p$  number of results

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

A worked example is set out below in Table 78.

Table 78 Uncertainty of Assigned Value for As in Sample S1

No. results (p)	15
Robust Average	4.02 mg/kg
$S_{rob\ av}$	0.38
$u_{rob\ av}$	0.12 mg/kg
$k$	2
$U_{rob\ av}$	0.25 mg/kg

The assigned value for As in Sample S1 is **4.02 ± 0.25 mg/kg**

### z-Score and E<sub>n</sub>-score

For each participant's result z-score and E<sub>n</sub>-score are calculated according to Equation 2 and Equation 3 respectively (see page 18).

A worked example is set out below in Table 79.

Table 79 z-Score and E<sub>n</sub>-score for As result reported by Laboratory 2 in S1

As Result mg/kg	Assigned Value mg/kg	Set Target Standard Deviation	z-Score	E <sub>n</sub> -Score
4.47±1.23	4.02±0.25	15% as PCV or 0.15x4.02= =0.60 mg/kg	$z = \frac{(4.47 - 4.02)}{0.60}$ $z = 0.75$	$E_n = \frac{(4.47 - 4.02)}{\sqrt{1.23^2 + 0.25^2}}$ $E_n = 0.36$

### APPENDIX 3 - USING PT DATA FOR UNCERTAINTY ESTIMATION

When a laboratory has successfully participated in at least 6 proficiency testing studies, the standard deviation from proficiency testing studies can also be used to estimate the uncertainty of their measurement results.<sup>10, 12</sup> An example is given. Between 2009 and 2022 NMI carried out 25 proficiency tests of metals in soil. These studies involved analyses of acid-extractable elements at low and high levels in dried soil, moist soil, biosoil, sediment and sludge.

**Laboratory X** submitted results for As in all of these PTs. All reported results returned satisfactory z-scores. This data can usefully be separated into two ranges of results 0.5 to 10 mg/kg and 10 to 100 mg/kg (Tables 80 and 81).

Taking the average of the robust CV over these PT samples for each concentration range gives estimates of the relative standard uncertainty of 12% and 9.4% respectively. Using a coverage factor of two gives relative expanded uncertainties of 25% and 19% respectively, at a level of confidence of approximately 95%.

Table 80 Laboratory X Reported Results for As at 0.5 to 10 mg/kg Level.

Study No.	Sample	Laboratory result mg/kg	Assigned value* mg/kg	Robust CV of all results (%)	Number of Results
AQA 09-13	S1 - Biosoil	4.091	3.64	16	11
	S2 - Soil	4.29	4.57	15	12
AQA 11-01	S1 - Biosoil	3.54	3.57	19.7	18
AQA 13-05	S1 - Soil	9.22	9.21	14	22
AQA 14-11	S1 - Sediment	7.91	7.37	11.8	21
AQA 15-02	S1 - Moist Sludge	8.29	7.02	13	22
	S2 - Moist Sludge	7.42	7.02	11.3	17
AQA 15-14	S1 - Sediment	10	9.95	6.7	17
	S2 - Soil	4.53	4.47	6.4	14
AQA 16-02	S2 - Clay	2.67	2.11	14	20
AQA 16-14	S1 - Soil	6.03	5.61	20	17
AQA 17-02	S2 - Soil	3.71	3.76	10	13
AQA 18-02	S1 - Compost	2.22	2.73	11	17
AQA 19-02	S1 - Soil	2.83	2.65	11	24
AQA 19-12	S1 - Soil	2.32	2.12	16	16
AQA 20-13	S1 - Biosoil	2.85	3.29	11	17
AQA 21-01	S1 - Sediment	7.02	6.26	6.9	18
AQA 21-01	S2 - Moist Sludge	3.99	3.58	12.6	13
AQA 22-02	S1 - Sediment	3.57	4.02	9.5	15
AQA 22-02	S2 - moist Soil	3.57	3.56	6.2	13
Average				11.7**	

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

Table 81 Laboratory X Reported Results for As at 10 to 100 mg/kg Level.

Study No.	Sample	Laboratory result mg/kg	Assigned value* mg/kg	Robust CV of all results (%)	Number of Results
AQA 10-12	S1 - Soil	16.6	14.4	8.5	19
AQA 11-12	S1 - Moist Sludge	25	21.6	15	13
AQA 12-01	S1 - Sediment	18.4	17.3	8.1	21
AQA 12-14	S2 - Soil	16.6	14.8	11	20
AQA 13-14	S1 - Sandy Soil	16.6	15.1	10.4	21
AQA 14-05	S1 - Soil	13.2	12.3	7.8	25
AQA 17-11	S1 - Sediment	18.1	17.4	11	22
AQA 18-12	S2 - Soil	10.4	9.6	8	20
AQA 19-12	S2 - Sediment	21	19.9	9	19
AQA 20-02	S1 - Soil	18.8	21.6	8.8	23
AQA 20-02	S2 - Moist Soil	16.5	17.8	6.7	24
AQA 21-14	S1 - Sediment	19.5	2.5	8.9	21
Average				9.4**	

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

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

Table 82 Uncertainty of As Results Estimated Using PT Data.

Results mg/kg	Uncertainty mg/kg
1.00	0.25
5.0	1.3
20.0	3.8
75	15

The estimates of 25% and 20% relative passes the test of being reasonable, and the analysis of the 33 different PT samples over twelve years can be assumed to include all the relevant uncertainty components (different matrices, operators, reagents, calibrators etc.), and so complies with ISO 17025:2018.

## APPENDIX 4 - ACRONYMS AND ABBREVIATIONS

APHA	American Public Health Association
A.V.	Assigned Value
CRI	Collision Reaction Interface
CRM	Certified Reference Material
CV	Coefficient of Variation
CV <sub>rob</sub>	Robust Coefficient of Variation
CV-AAS	Cold Vapour-Atomic Absorption Spectrometry
CV-AFS	Cold Vapour-Atomic Fluorescence Spectrometry
DA	Discrete Analyser
FIA	Flow Injection Analyser
GUM	Guide to the Expression of Uncertainty in Measurement
HEHe	High energy He mode
H.V.	Homogeneity Value
ICP-MS	Quadrupole - Inductively Coupled Plasma - Mass Spectrometry
ICP-OES-AV	Inductively Coupled Plasma - Optical Emission Spectrometry- axial view
ICP-OES-AV-buffer	Inductively Coupled Plasma - Optical Emission Spectrometry- axial view with buffer
ICP-OES-AV-equation	Inductively Coupled Plasma - Optical Emission Spectrometry- axial view with correction equation
ICP-OES-RV	Inductively Coupled Plasma - Optical Emission Spectrometry- radial view
IC	Ion chromatograph
IR	Infrared Detector
ISO/IEC	International Organisation for Standardisation / International Electrotechnical Commission
Max	Maximum value in a set of results
Md	Median
Min	Minimum value in a set of results
MU	Measurement Uncertainty
M.V.	Median Value
N	Number of Participants
NATA	National Association of Testing Authorities
NMI	National Measurement Institute (of Australia)
NR	Not Reported
NT	Not Tested
ORS	Octopole Reaction System
PCV	Performance Coefficient of Variation
PFAS	Polyfluoroalkyl Substances
PT	Proficiency Test
R.A.	Robust Average
RM	Reference Material
Robust CV	Robust Coefficient of Variation
Robust SD	Robust Standard Deviation
S.V.	Spiked value or formulated concentration of a PT sample
SS	Spiked sample

SI	The International System of Units
$s_{\text{sam}}^2$	Sampling variance
$s_a/\sigma$	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
$\sigma$	Target standard deviation
UC	Universal Cell
UV-Vis	Ultraviolet and Visible Spectroscopy

## APPENDIX 5 - INSTRUMENT DETAILS

Table 83 Instrument Conditions Ag

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None			NA		107
3	ICP-MS	Rh			N/A	25-250	107
4	ICP-OES-RV					NA	328.068
5	ICP-OES-RV				NA	100	328.068
6	ICP-MS	Rh	CRI	He	NA	500	107
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Y	NA	NA	NA	20	328.068
10	ICP-MS	Sc,Rh,Ir,In	KED	He	NA	800	107
11	ICP-MS	Rh	ORS	He	NA	500	107
12	ICP-MS	Rh	NA	NA	NA	625	109
13	ICP-OES-AV	Lu	NA	NA	NA	25	328.289
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	103		He	NA	50	107
16	ICP-OES-AV	Lutetium	NA	NA	NA	10-500	328.068nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	328.069nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	NA	NA			NA	NA	NA
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-MS	In	KED	He	NA	1000	
24	ICP-MS	Sc, Ir, Rh	ORS	He	NA		107 m/z
25	ICP-MS	Rh	ORS	He	NA	500	107
26	ICP-OES-AV-buffer	Y			NA	1	328.068
27	ICP-MS	103 Rh	DRC	He	NA	20	107 Ag
28	ICP-MS	103 Rh	ORS	He	N/A	50	107
29					NA		
30	NA	NA	NA	NA	NA	NA	NA

**Table 84 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-OES-AV	None			NA		176.502
3	ICP-OES-AV	Lu			N/A	25-250	396.152
4	ICP-OES-RV					NA	308.215
5	ICP-OES-RV				NA	100	308.215
6	ICP-MS	Sc	CRI	He	NA	500	27
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-OES-RV	Y			NA	800	396.152
11	ICP-MS	Sc	ORS	He	NA	500	27
12	ICP-MS	Sc	NA	NA	NA	625	27
13	ICP-OES-AV	Lu	NA	NA	NA	2500	236.705
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	72		standard mode	NA	20	27
16	ICP-OES-AV	Lutetium	NA	NA	NA	10-500	236.705nm
17	ICP-OES	Eu & Cs	NA	NA	NA	50	236.707, 308.215, 396.15nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-RV	Yttrium			NA	250	396.153
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc			NA	100	
24	ICP-OES-AV-buffer	Cs, Eu			NA		308.215 nm
25	ICP-MS	Sc	ORS	He	NA	5000	27
26	ICP-OES-AV-buffer	Y			NA	1	396.153
27	ICP-MS	45 Sc	DRC	He	NA	20	27 Al
28	ICP-MS	45 Sc	ORS	He	N/A	50	27
29					NA		
30	NA	NA	NA	NA	NA	NA	NA

**Table 85 Instrument Conditions As**

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None	ORS	He			75
3	ICP-MS	Ge	ORS	He	25-250	N/A	75
4	ICP-OES-RV						188.98
5	ICP-OES-RV				100	100	188.98
6	ICP-MS	Rh	CRI	He	500	500	75
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Y	NA	NA	20	20	188.979
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	800	75
11	ICP-MS	Y	ORS	He	NA	500	75
12	ICP-MS	Rh	KED	He	625	625	75
13	ICP-OES-AV	Lu	NA	NA	20	25	188.98
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	72		He	50	50	75
16	ICP-OES-AV	Lutetium	NA	NA	10-500	10-500	188.98nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	50	188.89nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			250	250	188.979
21	ICP-MS	Rh	KED	He	1000	NA	75
22	ICP-MS	In	KED	He	NA	1000	
24	ICP-MS	Sc, Ir, Rh	ORS	He			75 m/z
25	ICP-MS	Rh	ORS	He	500	500	75
26	ICP-OES-AV-buffer	Bi			1	1	193.696
27	ICP-MS	72 Ge	DRC	He	NA	20	75 As
28	ICP-MS	103 Rh	ORS	He	50	N/A	75
29							
30	ICP-OES-AV					NA	

**Table 86 Instrument Conditions B**

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None				NA	11
3	ICP-MS	Li6			N/A	25-250	11
4	ICP-OES-RV				NA		249.772
5	ICP-OES-RV				100	NA	249.678
6	ICP-MS	Sc	CRI	NA	500	NA	11
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	NA	11
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	NA	NA	625	NA	10
13	ICP-OES-AV	Lu	NA	NA	20	NA	182.577
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	89		standard mode	50	NA	11
16	ICP-OES-AV	Lutetium	NA	NA	10-500	NA	208.956nm
17	ICP-OES	Eu & Cs	NA	NA	50	NA	249.773nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			250	NA	249.677
21	ICP-MS	Sc	KED	He	1000	NA	11
22	NA	NA	NA	NA	NA	NA	NA
24	ICP-OES-AV-buffer	Cs, Eu				NA	249.772 nm
25	ICP-MS	Sc	ORS	He	500	NA	11
26	ICP-OES-AV-buffer	Bi			1	NA	208.957
27	NA	NA	NA	NA	NA	NA	NA
28	ICP-MS	45 Sc	NA		N/A	50	11
29						NA	
30	ICP-OES-AV					NA	

**Table 87 Instrument Conditions Ba**

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA	NA
2	ICP-OES-AV	None			NA		455.403
3	ICP-MS	Rh			25-250	25-250	138
4	ICP-OES-RV					NA	585.367
5	ICP-OES-RV				NA	100	455.403
6	ICP-MS	Rh	CRI	He	NA	500	135
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	NA	800	134
11	ICP-MS	Rh	ORS	He	NA	500	135
12	ICP-MS	Rh	NA	NA	NA	625	138
13	ICP-OES-AV	Lu	NA	NA	NA	25	230.424
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	159		He	NA	50	137
16	ICP-OES-AV	Lutetium	NA	NA	NA	10-500	230.424nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	585.369nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			NA	250	233.527
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc			NA	100	
24	ICP-MS	Sc, Ir, Rh	ORS	He	NA		137 m/z
25	ICP-MS	Sc	ORS	He	NA	500	135
26	ICP-OES-AV-buffer	Y			NA	1	233.527
27	ICP-MS	103 Rh	DRC	He	NA	20	137 Ba
28	ICP-MS	103 Rh	ORS	He	50	50	137
29					NA		
30	NA	NA	NA	NA	NA	NA	NA

**Table 88 Instrument Conditions Be**

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None				NA	9
3	ICP-MS	Li6			25-250	N/A	9
4	ICP-OES-RV					NA	585.367
5	ICP-OES-RV				100	NA	313.042
6	ICP-MS	Sc	CRI	NA	500	NA	9
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Y	NA	NA	20	NA	313.107
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	NA	9
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	NA	NA	625	NA	9
13	ICP-OES-AV	Lu	NA	NA	20	NA	313.107
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	72		standard mode	50	NA	9
16	ICP-OES-AV	Lutetium	NA	NA	10-500	NA	313.042nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	313.042nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	NA	NA			NA	NA	NA
21	ICP-MS	Sc	NA	He	2000	NA	9
22	NA	NA	NA	NA	NA	NA	NA
24	ICP-MS	Sc, Ir, Rh	ORS	He		NA	9 m/z
25	ICP-MS	Sc[No Gas]	ORS	standard mode	500	NA	9
26	ICP-OES-AV-buffer	Y			1	NA	313.107
27	NA	NA	NA	NA	NA	NA	NA
28	ICP-MS	45 Sc	NA		50	N/A	9
29						NA	
30	NA					NA	

**Table 89 Instrument Conditions Bi**

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None				NA	209
3	ICP-MS	Lu			N/A	25-250	209
4	ICP-MS	Rh, Sc, Ir	ORS			NA	209
5						NA	
6	ICP-MS	Lu	CRI	He	500	NA	209
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	NA	209
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Ir	NA	NA	625	NA	209
13	ICP-MS	Lu	ORS	standard mode	200	NA	209
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	159		standard mode	50	NA	209
16	ICP-MS	Lutetium	ORS	No Gas	200	NA	209m/z
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	209 m/z
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	NA	NA			NA	NA	NA
21	ICP-MS	Tb	NA	He	2000	NA	209
22	NA	NA	NA	NA	NA	NA	NA
24	ICP-MS	Sc, Ir, Rh	ORS	He		NA	209 m/z
25	ICP-MS	Lu	ORS	He	500	NA	209
26			1	1	193.696		
27	NA	NA	NA	NA	NA	NA	NA
28	ICP-MS	193 Y	ORS	He	N/A	50	209
29						NA	
30	NA					NA	

**Table 90 Instrument Conditions Ca**

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						NA	
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-RV	Lu			N/A	25-250	316
4	ICP-OES-RV						315.887
5	ICP-OES-RV				100	NA	315.887
6	NA	NA	NA	NA	NA	NA	NA
7						NA	
8	ICP-OES-AV	Y 371.029				NA	430.253
9							
10	ICP-OES-RV	Y			800	NA	422.673
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	KED	He	625	NA	44
13	ICP-OES-AV	Lu	NA	NA	400	NA	315.887
14	ICP-OES-AV-buffer	Yttrium	NA	NA	50	NA	317.933
15	ICP-MS	89		H2	50	NA	40
16	ICP-OES-AV	Lutetium	NA	NA	10-500	NA	315.887nm
17	ICP-OES	Eu & Cs	NA	NA	50	NA	315.887, 370.602nm
18						NA	
19	ICP-OES-RV	Yttrium			250	NA	315.887
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc			100	NA	
24	ICP-OES-AV-buffer	Cs, Eu				NA	370.602 nm
25	NA	NA	NA	NA	NA	NA	NA
26	ICP-OES-AV-buffer	Y			1	NA	315.887
27	ICP-OES-AV	175 Lu	NA	NA	200	NA	317.933 nm
28	ICP-MS	45 Sc	ORS	He	N/A	50	44
29	NA	NA	NA	NA	NA	NA	NA
30	NA	NA	NA	NA	NA	NA	NA

**Table 91 Instrument Conditions Cd**

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None	ORS	He			111
3	ICP-MS	Rh			25-250	N/A	111
4	ICP-OES-RV				NA		226.502
5	ICP-OES-RV				100	100	214.439
6	ICP-MS	Rh	CRI	He	500	500	111
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Y	NA	NA	20	20	228.802
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	800	111
11	ICP-MS	Rh	ORS	He	NA	500	114
12	ICP-MS	Rh	NA	NA	625	625	111
13	ICP-OES-AV	Lu	NA	NA	20	25	228.802
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	103		He	50	50	111
16	ICP-OES-AV	Lutetium	NA	NA	10-500	10-500	214.439nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	50	226.502nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			250	250	226.502
21	ICP-MS	Rh	KED	He	1000	NA	111
22	ICP-MS	In	KED	He	NA	1000	
24	ICP-MS	Sc, Ir, Rh	ORS	He			111 m/z
25	ICP-MS	Rh	ORS	He	500	500	111
26	ICP-OES-AV-buffer	Bi			1	1	228.802
27	ICP-MS	103 Rh	DRC	He	NA	20	111 Cd
28	ICP-MS	103 Rh	ORS	He	50	N/A	111
29							
30	ICP-OES-AV					NA	

**Table 92 Instrument Conditions Co**

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None	ORS	He	NA		59
3	ICP-MS	Ge	ORS	He	N/A	25-250	59
4	ICP-OES-RV				NA		228.615
5	ICP-OES-RV				NA	100	228.615
6	ICP-MS	Sc	CRI	He	NA	500	59
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	NA	800	59
11	ICP-MS	Sc	ORS	He	NA	500	59
12	ICP-MS	Ge	KED	He	NA	625	59
13	ICP-OES-AV	Lu	NA	NA	NA	25	228.615
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	103		He	NA	50	59
16	ICP-OES-AV	Lutetium	NA	NA	NA	10-500	231.160nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	228.616nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			NA	250	228.616
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc			NA	100	
24	ICP-MS	Sc, Ir, Rh	ORS	He	NA		59 m/z
25	ICP-MS	Sc	ORS	He	NA	500	59
26	ICP-OES-AV-buffer	Bi			NA	1	228.616
27	ICP-MS	45 Sc	DRC	He	NA	20	59 Co
28	ICP-MS	103 Rh	ORS	He	N/A	50	59
29					NA		
30	NA	NA	NA	NA	NA	NA	NA

**Table 93 Instrument Conditions Cr**

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None	ORS	He		NA	52
3	ICP-MS	Ge	ORS	He	25-250	N/A	52
4	ICP-OES-RV						267.716
5	ICP-OES-RV				100	NA	267.716
6	ICP-MS	Sc	CRI	He	500	NA	52
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Y	NA	NA	20	NA	267.716
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	NA	52
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	KED	He	625	NA	52
13	ICP-OES-AV	Lu	NA	NA	20	NA	205.56
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	72		He	50	NA	52
16	ICP-OES-AV	Lutetium	NA	NA	10-500	NA	205.560nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	267.716nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			250	NA	267.716
21	ICP-MS	Sc	KED	He	1000	NA	52
22	NA	NA	NA	NA	NA	NA	NA
24	ICP-MS	Sc, Ir, Rh	ORS	He		NA	52 m/z
25	ICP-MS	Sc	ORS	He	500	NA	63
26	ICP-OES-AV-buffer	Bi			1	NA	205.560
27	NA	NA	NA	NA	NA	NA	NA
28	ICP-MS	45 Sc	ORS	He	50	N/A	52
29						NA	
30	ICP-OES-AV					NA	

**Table 94 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None	ORS	He			63
3	ICP-MS	Ge	ORS	He	25-250	N/A	63
4	ICP-OES-RV						327.395
5	ICP-OES-RV				100	100	324.754
6	ICP-MS	Sc	CRI	He	500	500	63
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Y	NA	NA	20	20	324.752
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	800	63
11	ICP-MS	Sc	ORS	He	NA	500	63
12	ICP-MS	Ge	KED	He	625	625	63
13	ICP-OES-AV	Lu	NA	NA	20	25	324.754
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	103		He	50	50	63
16	ICP-OES-AV	Lutetium	NA	NA	10-500	10-500	324.754nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	50	327.395nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			250	250	324.752
21	ICP-MS	Ga	KED	He	1000	NA	63
22	ICP-OES-AV	Sc			NA	100	
24	ICP-MS	Sc, Ir, Rh	ORS	He			63 m/z
25	ICP-MS	Sc	ORS	He	500	500	52
26	ICP-OES-AV-buffer	Y			1	1	324.752
27	ICP-MS	72 Ge	DRC	He	NA	20	63 Cu
28	ICP-MS	103 Rh	ORS	He	50	N/A	63
29							
30	ICP-OES-AV					NA	

**Table 95 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						NA	
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-RV	Ge	ORS	He	N/A	25-250	259.94
4	ICP-OES-RV						258.588
5	ICP-OES-RV				100	NA	234.35
6	NA	NA	NA	NA	NA	NA	NA
7						NA	
8	ICP-OES-AV	Y 371.029				NA	370.792
9							
10	ICP-OES-RV	Y			800	NA	238.204
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	KED	He	625	NA	56
13	ICP-OES-AV	Lu	NA	NA	2000	NA	261.382
14	ICP-OES-AV-buffer	Yttrium	NA	NA	500	NA	259.94
15	ICP-MS	103		He	50	NA	56
16	ICP-OES-AV	Lutetium	NA	NA	10-500	NA	234.350nm
17	ICP-OES	Eu & Cs	NA	NA	50	NA	238.204, 258.588, 259.940nm
18						NA	
19	ICP-OES-RV	Yttrium			250	NA	259.939
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc			100	NA	
24	ICP-OES-AV-buffer	Cs, Eu				NA	370.792 nm
25	ICP-MS	Sc	ORS	He	25000	NA	56
26	ICP-OES-AV-buffer	Y			1/100	NA	239.562
27	ICP-MS	45 Sc	DRC	He	200	NA	56 Fe
28	ICP-MS	103 Rh	ORS	He	N/A	50	133
29	NA	NA	NA	NA	NA	NA	NA
30	NA	NA	NA	NA	NA	NA	NA

**Table 96 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None					202
3	CVAAS	--			25-250	N/A	253.7
4	AAS						253.7
5	CVAFS						
6	ICP-MS	Lu	CRI	He	500	500	201
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	Hydride	NA	NA	NA	120	120	253.7
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	800	202
11	ICP-MS	Lu	ORS	He	NA	500	202
12	ICP-MS	Ir	NA	NA	625	625	201
13	CVAAS	NA	NA	NA	200	250	253.7
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	193		standard mode	50	50	202
16	CVAAS	NA	NA	NA	100	100	253.7nm
17	FIMS-AAS	NA	NA	NA	50	50	253.7nm
18	CVAAS	NA	NA	NA	NA	NA	NA
19	CVAAS				250	250	253.7
21	ICP-MS	Tb	KED	He	1000	NA	201
22	CVAAS				NA	100	
24	CVAAS						253.7 nm
25	ICP-MS	Lu	ORS	He	500	500	202
26	CVAAS				10	1	253.700
27	ICP-MS	175 Lu	DRC	He	NA	20	201 Hg
28	CVAAS	Eu	NA		N/A	50	258.3
29							
30	ICP-OES-AV					NA	

**Table 97 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						NA	
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-RV	Lu			N/A	25-250	766.491
4	ICP-OES-RV						766.491
5	ICP-OES-RV				100	NA	766.491
6	NA	NA	NA	NA	NA	NA	NA
7						NA	
8	ICP-OES-AV	Y 371.029				NA	769.897
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	NA	39
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	KED	He	625	NA	39
13	ICP-OES-AV	Lu	NA	NA	20	NA	766.491
14	ICP-OES-AV-buffer	Yttrium	NA	NA	50	NA	766.491
15	ICP-MS	89		H2	20	NA	39
16	ICP-OES-AV	Caesium	NA	NA	10-500	NA	766.491nm
17	ICP-OES	Eu & Cs	NA	NA	50	NA	404.721nm, 766.491nm
18						NA	
19	ICP-OES-RV	Yttrium			250	NA	766.49
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc			100	NA	
24	ICP-OES-AV-buffer	Cs, Eu				NA	769.897 nm
25	NA	NA	NA	NA	NA	NA	NA
26	ICP-OES-AV-buffer	Y			1	NA	766.490
27	ICP-OES-AV	175 Lu	NA	He	20	NA	766.490 nm
28	ICP-OES-AV-equation	103 Rh	NA		50	N/A	253.7
29	NA	NA	NA	NA	NA	NA	NA
30	NA	NA	NA	NA	NA	NA	NA

**Table 98 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None			NA		7
3	ICP-OES-RV	Li6			N/A	25-250	670.3
4	ICP-MS	Rh, Sc, Ir	ORS				7
5					NA		
6	ICP-MS	Sc	CRI	NA	NA	500	7
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	NA	800	7
11	ICP-MS	Sc	ORS	standard mode	NA	500	7
12	ICP-MS	Sc	NA	NA	NA	625	7
13	ICP-OES-AV	Lu	NA	NA	NA	25	670.783
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	72		H2	NA	20	7
16	ICP-OES-AV	Lutetium	NA	NA	NA	10-500	670.783nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	7 m/z
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			NA	250	670.784
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-MS	In	KED	He	NA	1000	
24	ICP-MS	Sc, Ir, Rh	ORS	He	NA		7 m/z
25	ICP-MS	Sc[No Gas]	ORS	standard mode	NA	500	7
26	ICP-OES-AV-buffer	Y			NA	1	670.784
27	ICP-MS	45 Sc	DRC	NA	NA	20	7 Li
28	ICP-MS	45 Sc	ORS	He	N/A	50	39
29					NA		
30	NA	NA	NA	NA	NA	NA	NA

Table 99 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						NA	
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-RV	Lu			N/A	25-250	279.8
4	ICP-OES-RV				NA		383.829
5	ICP-OES-RV				100	NA	383.23
6	NA	NA	NA	NA	NA	NA	NA
7						NA	
8	ICP-OES-AV	Y 371.029				NA	277.983
9							
10	ICP-OES-RV	Y			800	NA	279.078
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	KED	He	625	NA	25
13	ICP-OES-AV	Lu	NA	NA	20	NA	279.8
14	ICP-OES-AV-buffer	Yttrium	NA	NA	500	NA	285.213
15	ICP-MS	89		H2	50	NA	24
16	ICP-OES-AV	Lutetium	NA	NA	10-500	NA	279.800nm
17	ICP-OES	Eu & Cs	NA	NA	50	NA	383.829nm
18						NA	
19	ICP-OES-RV	Yttrium			250	NA	279.077
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc			100	NA	
24	ICP-OES-AV-buffer	Cs, Eu				NA	383.829 nm
25	NA	NA	NA	NA	NA	NA	NA
26	ICP-OES-AV-buffer	Bi			1	NA	285.213
27	ICP-OES-AV	175 Lu	NA	NA	200	NA	279.077 nm
28	ICP-MS	103 Rh	ORS	He	N/A	50	139
29	NA	NA	NA	NA	NA	NA	NA
30	NA	NA	NA	NA	NA	NA	NA

**Table 100 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-OES-AV	None					257.61
3	ICP-MS	Ge	ORS	He	25-250	N/A	55
4	ICP-OES-RV						261.02
5	ICP-OES-RV				100		257.61
6	ICP-MS	Sc	CRI	He	500	500	55
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	800	55
11	ICP-MS	Sc	ORS	He	NA	500	55
12	ICP-MS	Sc	KED	He	625	625	55
13	ICP-OES-AV	Lu	NA	NA	20	25	257.61
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	72		standard mode	50	50	55
16	ICP-OES-AV	Lutetium	NA	NA	10-500	10-500	257.610nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	50	261.021nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			250	250	257.61
21	ICP-MS	Sc	KED	He	2000	NA	55
22	ICP-OES-AV	Sc			NA	100	
24	ICP-MS	Sc, Ir, Rh	ORS	He			55 m/z
25	ICP-MS	Sc	ORS	He	500	500	55
26	ICP-OES-AV-buffer	Y			1	1	257.610
27	ICP-MS	45 Sc	DRC	He	NA	20	55 Mn
28	ICP-MS	45 Sc	NA		N/A	50	7
29							
30	ICP-OES-AV					NA	

Table 101 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None		He		NA	98
3	ICP-MS	Rh			25-250	N/A	95
4	ICP-OES-RV					NA	202.032
5	ICP-OES-RV				100	NA	202.032
6	ICP-MS	Rh	CRI	He	500	NA	95
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Y	NA	NA	20	NA	202.031
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	NA	95
11	NA	NA	NA	NA	NA	NA	95
12	ICP-MS	Rh	NA	NA	625	NA	95
13	ICP-OES-AV	Lu	NA	NA	20	NA	202.032
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	89		He	50	NA	95
16	ICP-OES-AV	Lutetium	NA	NA	10-500	NA	202.032nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	202.032nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			250	NA	202.031
21	ICP-MS	Rh	KED	He	2000	NA	98
22	NA	NA	NA	NA	NA	NA	NA
24	ICP-MS	Sc, Ir, Rh	ORS	He		NA	95 m/z
25	ICP-MS	Rh	ORS	He	500	NA	95
26	ICP-OES-AV-buffer	Bi				NA	202.031
27	NA	NA	NA	NA	NA	NA	NA
28	ICP-MS	45 Sc	ORS	He	N/A	50	24
29						NA	
30	ICP-OES-AV					NA	

**Table 102 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						NA	
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-RV	Lu			N/A	25-250	588.995
4	ICP-OES-RV				NA		589.592
5	ICP-OES-RV				100	NA	330.237
6	NA	NA	NA	NA	NA	NA	NA
7						NA	
8	ICP-OES-AV	Y 371.029				NA	588.995
9							
10	ICP-OES-RV	Y			800	NA	589.592
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	KED	He	625	NA	23
13	ICP-OES-AV	Lu	NA	NA	400	NA	588.995
14	ICP-OES-AV-buffer	Yttrium	NA	NA	50	NA	589.592
15	ICP-MS	89		H2	10	NA	23
16	ICP-OES-AV	Lutetium	NA	NA	10-500	NA	588.995nm
17	ICP-OES	Eu & Cs	NA	NA	50	NA	330.237, 589.592nm
18						NA	
19	ICP-OES-RV	Yttrium			250	NA	589.592
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc			100	NA	
24	ICP-OES-AV-buffer	Cs, Eu				NA	589.592 nm
25	NA	NA	NA	NA	NA	NA	NA
26	ICP-OES-AV-buffer	Y			1/100	NA	588.995
27	ICP-OES-AV	175 Lu	NA	NA	20	NA	589.592 nm
28	ICP-MS	45 Sc	ORS	He	50	N/A	55
29	NA	NA	NA	NA	NA	NA	NA
30	NA	NA	NA	NA	NA	NA	NA

Table 103 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None		He			60
3	ICP-MS	Ge	ORS	He	25-250	N/A	60
4	ICP-OES-RV						231.604
5	ICP-OES-RV				100	100	216.555
6	ICP-MS	Sc	CRI	He	500	500	60
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Y	NA	NA	20	20	231.604
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	800	60
11	ICP-MS	Sc	ORS	He	NA	500	60
12	ICP-MS	Ge	KED	He	625	625	60
13	ICP-OES-AV	Lu	NA	NA	20	25	231.604
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	103		He	50	50	60
16	ICP-OES-AV	Lutetium	NA	NA	10-500	10-500	231.604nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	50	231.604nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			250	250	231.604
21	ICP-MS	Ga	KED	He	1000	NA	60
22	ICP-OES-AV	Sc			NA	100	
24	ICP-MS	Sc, Ir, Rh	ORS	He			60 m/z
25	ICP-MS	Sc	ORS	He	500	500	60
26	ICP-OES-AV-buffer	Bi			1	1	231.604
27	ICP-MS	72 Ge	DRC	He	NA	20	60 Ni
28	ICP-MS	103 Rh	ORS	He	50	N/A	95
29							
30	ICP-OES-AV					NA	

**Table 104 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						NA	
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-RV	Lu			N/A	25-250	213.618
4	ICP-OES-RV				NA		185.827
5						NA	
6	NA	NA	NA	NA	NA	NA	NA
7						NA	
8	ICP-OES-AV	Y 371.029				NA	213.618
9							
10	ICP-OES-AV	Y			800	NA	213.618
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	KED	He	625	NA	31
13	ICP-OES-AV	Lu	NA	NA	2000	NA	182.143
14	ICP-OES-AV-buffer	Yttrium	NA	NA	50	NA	213.618
15	ICP-MS	72		He	50	NA	31
16	ICP-OES-AV	Lutetium	NA	NA	10-500	NA	213.618nm
17	ICP-OES	Eu & Cs	NA	NA	50	NA	185.827nm
18						NA	
19	ICP-OES-AV	Yttrium			250	NA	213.617
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc			100	NA	
24	ICP-OES-AV-buffer	Cs, Eu				NA	185.827 nm
25	ICP-MS	Sc	ORS	He	500	NA	31
26	ICP-OES-AV-buffer	Bi			1	NA	178.221
27	ICP-OES-AV	175 Lu	NA	NA	20	NA	178.219 nm
28	ICP-MS	45 Sc	ORS	He	N/A	50	23
29	NA	NA	NA	NA	NA	NA	NA
30	NA	NA	NA	NA	NA	NA	NA

**Table 105 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None				NA	206+207+208
3	ICP-MS	Lu			25-250	N/A	206+207+208
4	ICP-OES-RV						220.353
5	ICP-OES-RV				100	NA	220
6	ICP-MS	Lu	CRI	He	500	NA	208
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Y	NA	NA	20	NA	220.353
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	NA	208
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Ir	NA	NA	625	NA	206+207+208
13	ICP-OES-AV	Lu	NA	NA	20	NA	220.353
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	159		standard mode	50	NA	208
16	ICP-OES-AV	Lutetium	NA	NA	10-500	NA	220.353nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	220.353nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			250	NA	220.353
21	ICP-MS	Tb	KED	He	1000	NA	206+207+208
22	NA	NA	NA	NA	NA	NA	NA
24	ICP-MS	Sc, Ir, Rh	ORS	He		NA	208 m/z
25	ICP-MS	Lu	ORS	He	500	NA	208
26	ICP-OES-AV-buffer	Bi			1	NA	220.353
27	NA	NA	NA	NA	NA	NA	NA
28	ICP-MS	103 Rh	ORS	He	50	N/A	60
29						NA	
30	ICP-OES-AV					NA	

**Table 106 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None				NA	85
3	ICP-MS	Lu			N/A	25-250	
4	ICP-MS	Rh, Sc, Ir	ORS				85
5						NA	220.353
6	NA	NA	NA	NA	NA	NA	NA
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	NA	85
11	NA	NA	NA	NA	NA	NA	NA
12						NA	
13	ICP-MS	Ge	ORS	standard mode	200	NA	85
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	89		He	50	NA	85
16	ICP-MS	Germanium	ORS	He	200	NA	85m/z
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	85 m/z
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	NA	NA			NA	NA	NA
21	ICP-MS	Rh	NA	He	2000	NA	85
22	NA	NA	NA	NA	NA	NA	NA
24	ICP-MS	Sc, Ir, Rh	ORS	He		NA	85 m/z
25	NA	NA	NA	NA	NA	NA	NA
26	NA					NA	
27	NA	NA	NA	NA	NA	NA	NA
28							
29						NA	
30	NA					NA	

**Table 107 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						NA	
2	NA	NA	NA	NA	NA	NA	NA
3	ICP-OES-RV	Lu			N/A	25-250	181.972
4	ICP-OES-RV						178.165
5	ICP-OES-RV				100	NA	181.972
6	NA	NA	NA	NA	NA	NA	NA
7						NA	
8	ICP-OES-AV	Y 371.029				NA	181.972
9							
10	ICP-OES-AV	Y			800	NA	181.972
11	NA	NA	NA	NA	NA	NA	NA
12						NA	
13	ICP-OES-AV	Lu	NA	NA	20	NA	181.972
14	ICP-OES-AV-buffer	Yttrium	NA	NA	50	NA	181.972
15	ICP-MS/MS	89-105		O2	50	NA	32
16	ICP-OES-AV	Lutetium	NA	NA	10-500	NA	181.972nm
17	ICP-OES	Eu & Cs	NA	NA	50	NA	178.165,181.972nm
18						NA	
19	ICP-OES-AV	Yttrium			250	NA	181.975
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc			100	NA	
24	ICP-OES-AV-buffer	Cs, Eu				NA	181.972 nm
25	NA	NA	NA	NA	NA	NA	NA
26	ICP-OES-AV-buffer	Y			1	NA	180.669
27	ICP-OES-AV	175 Lu	NA	NA	20	NA	181.975 nm
28	ICP-OES-AV-equation	Eu	NA		N/A	50	185.8
29	NA	NA	NA	NA	NA	NA	NA
30	NA	NA	NA	NA	NA	NA	NA

**Table 108 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None	ORS		NA		121
3	ICP-MS	Ge			25-250	N/A	121
4	ICP-OES-RV				NA		206.834
5	ICP-OES-RV				NA	100	217.582
6	ICP-MS	Rh	CRI	He	NA	500	123
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	NA	800	121
11	ICP-MS	Rh	ORS	He	NA	500	121
12	ICP-MS	Rh	NA	NA	NA	625	121
13	ICP-OES-AV	Lu	NA	NA	NA	25	206.834
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	193		standard mode	NA	50	121
16	ICP-OES-AV	Lutetium	NA	NA	NA	10-500	206.834nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	206.834nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			NA	250	206.836
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-MS	In	KED	He	NA	1000	
24	ICP-MS	Sc, Ir, Rh	ORS	He	NA		121 m/z
25	ICP-MS	Rh	ORS	He	NA	500	123
26	NA				NA	1	
27	ICP-MS	103 Rh	DRC	He	NA	20	121 Sb
28	ICP-MS	193 Y	ORS	He	50	N/A	208
29					NA		
30	NA	NA	NA	NA	NA	NA	NA

Table 109 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None	ORS	H2			78
3	ICP-MS	Ge	ORS	H2	25-250	25-250	78
4	ICP-OES-RV						196.026
5	ICP-OES-RV				100	100	196.026
6	ICP-MS	Sc	CRI	He	500	500	78
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Y	NA	NA	20	20	196.026
10	ICP-MS	Sc,Rh,Ir,In	KED	HEHe	800	800	78
11	ICP-MS	Y	ORS	H2	NA	500	78
12	ICP-MS	Rh	NA	NA	625	625	82
13	ICP-OES-AV	Lu	NA	NA	20	25	196.026
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	72		H2	50	50	78
16	ICP-OES-AV	Lutetium	NA	NA	10-500	10-500	196.026nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	50	196.026nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			250	250	196.026
21	ICP-MS	Te	NA	He	2000	NA	82
22	ICP-MS	In	KED	He	NA	1000	
24	ICP-MS	Sc, Ir, Rh	ORS	He			78 m/z
25	ICP-MS	Rh	ORS	H2	500	500	78
26	ICP-OES-AV-buffer	Bi					196.026
27	ICP-MS	72 Ge	DRC	HEHe	NA	20	78 Se
28	ICP-MS	103 Rh	ORS	He	N/A	50	85
29							
30	ICP-OES-AV					NA	

Table 110 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None				NA	118
3	ICP-MS	Rh			25-250	25-250	118
4	ICP-OES-RV					NA	189.925
5	ICP-OES-RV				100	NA	189.925
6	ICP-MS	Rh	CRI	He	500	NA	118
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Y	NA	NA	20	NA	189.927
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	NA	118
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Rh	NA	NA	625	NA	118
13	ICP-OES-AV	Lu	NA	NA	20	NA	189.925
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	103		He	50	NA	120
16	ICP-OES-AV	Lutetium	NA	NA	10-500	NA	189.925nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	189.926nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			250	NA	189.927
21	ICP-MS	Rh	NA	He	2000	NA	120
22	NA	NA	NA	NA	NA	NA	NA
24	ICP-MS	Sc, Ir, Rh	ORS	He		NA	118 m/z
25	ICP-MS	Rh	ORS	He	500	NA	118
26	ICP-OES-AV-buffer	Bi			1	NA	189.927
27	NA	NA	NA	NA	NA	NA	NA
28	ICP-OES-AV-equation	Eu	NA		N/A	50	178.2
29						NA	
30	NA					NA	

Table 111 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None			NA		88
3	ICP-OES-RV	Lu			N/A	25-250	407.771
4	ICP-OES-RV						430.544
5	ICP-OES-RV				NA	100	407.771
6	ICP-MS	Rh	CRI	He	NA	500	88
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	NA	800	88
11	ICP-MS	Rh	ORS	He	NA	500	88
12	ICP-MS	Rh	NA	NA	NA	625	88
13	ICP-OES-AV	Lu	NA	NA	NA	25	421.552
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	89		He	NA	50	88
16	ICP-OES-AV	Lutetium	NA	NA	NA	10-500	407.771nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	430.545nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			NA	250	407.771
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc			NA	100	
24	ICP-MS	Sc, Ir, Rh	ORS	He	NA		88 m/z
25	ICP-MS	Rh	ORS	He	NA	500	88
26	ICP-OES-AV-buffer	Y			NA	1	421.552
27	ICP-MS	72 Ge	DRC	He	NA	20	88 Sr
28	ICP-MS	103 Rh	ORS	He	50	50	78
29					NA		
30	NA	NA	NA	NA	NA	NA	NA

Table 112 Instrument Conditions Th

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S3 Final Dilution Factor	S2 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None				NA	232
3	ICP-MS	Lu			N/A	25-250	
4	ICP-MS	Rh, Sc, Ir	ORS			NA	232
5					100	NA	
6	NA	NA	NA	NA	NA	NA	NA
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	NA	232
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Ir	NA	NA	625	NA	232
13	ICP-MS	Lu	ORS	standard mode	200	NA	232
14	NA	NA	NA	NA	NA	NA	NA
15	NT					NA	
16	ICP-MS	Lutetium	ORS	No Gas	200	NA	232m/z
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	232 m/z
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	NA	NA			NA	NA	NA
21	NA	NA	NA	NA	NA	NA	NA
22	NA	NA	NA	NA	NA	NA	NA
24	ICP-MS	Sc, Ir, Rh	ORS	He		NA	232 m/z
25	NA	NA	NA	NA	500	NA	NA
26	NA				1	NA	
27	NA	NA	NA	NA	NA	NA	NA
28							
29						NA	
30	NA					NA	

Table 113 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None			NA		205
3	ICP-MS	Lu			N/A	25-250	205
4	ICP-MS	Rh, Sc, Ir	ORS			NA	205
5	ICP-OES-RV				NA	100	190.794
6	ICP-MS	Lu	CRI	He	NA	500	205
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	NA	800	205
11	ICP-MS	Lu	ORS	He	NA	500	205
12	ICP-MS	Ir	NA	NA	NA	625	205
13	ICP-OES-AV	Lu	NA	NA	NA	25	190.794
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	159		standard mode	NA	20	205
16	ICP-OES-AV	Lutetium	NA	NA	NA	10-500	190.764nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	203 m/z
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	NA	NA			NA	NA	NA
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-MS	In	KED	He	NA	1000	
24	ICP-MS	Sc, Ir, Rh	ORS	He	NA		205 m/z
25	ICP-MS	Lu	ORS	He	NA	500	205
26	ICP-OES-AV-buffer	Bi			NA	1	190.801
27	ICP-MS	175 Lu	DRC	He	NA	20	205 Tl
28	ICP-MS	103 Rh	ORS	He	50	50	118
29					NA		
30	NA	NA	NA	NA	NA	NA	NA

**Table 114 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None			NA		238
3	ICP-MS	Lu			N/A	25-250	238
4	ICP-MS	Rh, Sc, Ir	ORS	He		NA	238
5					NA	100	
6	ICP-MS	Lu	CRI	He	NA	500	238
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	NA	800	238
11	ICP-MS	Lu	ORS	He	NA	500	238
12	ICP-MS	Ir	NA	NA	NA	625	238
13	ICP-MS	Lu	ORS	standard mode	NA	250	238
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	159		standard mode	NA	50	238
16	ICP-MS	Lutetium	ORS	No Gas	NA	200	238m/z
17	ICP-MS	Ir, Rh & Sc	NA	NA	NA	50	238 m/z
18		NA	NA	NA	NA	NA	NA
19	NA	NA			NA	NA	NA
21	NA	NA	NA	NA	NA	NA	NA
22	ICP-MS	In	KED	He	NA	1000	
24	ICP-MS	Sc, Ir, Rh	ORS	He	NA		238 m/z
25	ICP-MS	Lu	ORS	He	NA	500	238
26	NA				NA		
27	ICP-MS	175 Lu	DRC	He	NA	20	238 U
28	ICP-MS	103 Rh	ORS	He	N/A	50	88
29					NA		
30	NA	NA	NA	NA	NA	NA	NA

**Table 115 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-OES-AV	None				NA	292.402
3	ICP-MS	Ge			25-250	N/A	51
4	ICP-OES-RV			He			311.837
5	ICP-OES-RV				100	NA	292.401
6	ICP-MS	Sc	CRI	He	500	NA	51
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9							
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	NA	51
11	NA	NA	NA	NA	NA	NA	NA
12	ICP-MS	Sc	KED	He	625	NA	51
13	ICP-OES-AV	Lu	NA	NA	20	NA	292.401
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS	72		He	50	NA	51
16	ICP-OES-AV	Lutetium	NA	NA	10-500	NA	292.401nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	311.837nm
18		NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			250	NA	292.402
21	ICP-MS	Sc	KED	He	2000	NA	51
22	NA	NA	NA	NA	NA	NA	NA
24	ICP-MS	Sc, Ir, Rh	ORS	He		NA	51 m/z
25	ICP-MS	Sc	ORS	He	500	NA	51
26	ICP-OES-AV-buffer	Y			1	NA	290.880
27	NA	NA	NA	NA	NA	NA	NA
28	ICP-MS	193 Y	ORS	He	N/A	50	232
29						NA	
30	NA					NA	

Table 116 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	NA	NA	NA	NA	NA	NA	NA
2	ICP-MS/MS	None					206.2
3	ICP-MS	Ge			25-250	25-250	66
4	ICP-OES-RV			He			206.2
5	ICP-OES-RV				100	100	206.2
6	ICP-MS	Sc	CRI	He	500	500	66
7	NA	NA	NA	NA	NA	NA	NA
8	NA	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	Y	NA	NA	20	20	213.857
10	ICP-MS	Sc,Rh,Ir,In	KED	He	800	800	64
11	ICP-MS	Sc	ORS	He	NA	500	66
12	ICP-MS	Ge	KED	He	625	625	66
13	ICP-OES-AV	Lu	NA	NA	20	25	206.2
14	NA	NA	NA	NA	NA	NA	NA
15	ICP-MS			He	50	50	66
16	ICP-OES-AV	Lutetium	NA	NA	10-500	10-500	206.200nm
17	ICP-MS	Ir, Rh & Sc	NA	NA	50	50	206.2, 334.502nm
18	ICP-OES-AV	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV	Yttrium			250	250	206.200
21	ICP-MS	Ga	KED	He	1000	NA	66
22	ICP-OES-AV	Sc			NA	100	
24	ICP-MS	Sc, Ir, Rh	ORS	He			68 m/z
25	ICP-MS	Sc	ORS	He	500	500	66
26	ICP-OES-AV-buffer	Bi			1	1	206.200
27	ICP-MS	72 Ge	DRC	He	NA	20	66 Zn
28	ICP-MS	193 Y	ORS	He	N/A	50	205
29							
30	ICP-OES-AV					NA	

Table 117 Instrument Conditions Exchangeable Ca<sup>2+</sup>

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm) /Ion(m/z)/ Absorbance(nm)
1						
2	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV-buffer				100	316
4	ICP-OES-RV					315.887
5	ICP-OES-AV				20	422.673
6	NA	NA	NA	NA	NA	NA
7	ICP-OES-RV					
8						
9	NA	NA	NA	NA	NA	NA
10	ICP-OES-RV	Y	NA	NA	40	422.673
11	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV	Y	NA		20	317.933
13						
14	ICP-OES-AV-buffer	Yttrium	No	No	10	317.933
15	NT					
16	ICP-OES-AV	Lutetium	NA	NA	100	315.887
17	ICP-OES	Eu & Cs	NA	NA	500	315.887, 370.602nm
18	ICP-OES-AV					317.933
19	ICP-OES-RV	Yttrium			50	315.887
21	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc				
24	ICP-OES-AV-buffer	Cs, Eu			25	370.602 nm
25						
26	NA					
27	ICP-OES-AV	175 Lu	NA	NA	20	317.933 nm
28	ICP-OES	Eu	NA		1	315.9
29	NA	NA	NA	NA	NA	NA
30	NA	NA	NA	NA	NA	NA

**Table 11718 Instrument Conditions Exchangeable Mg<sup>2+</sup>**

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						
2	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV-buffer				100	279.8
4	ICP-OES-RV					383.829
5	ICP-OES-AV				20	285.213
6	NA	NA	NA	NA	NA	NA
7	ICP-OES-RV					
8						
9	NA	NA	NA	NA	NA	NA
10	ICP-OES-RV	Y	NA	NA	40	279.078
11	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV	Y	NA		20	285.213
13						
14	ICP-OES-AV-buffer	Yttrium	No	No	10	285.213
15	NT					
16	ICP-OES-AV	Lutetium	NA	NA	100	279.8
17	ICP-OES	Eu & Cs	NA	NA	500	383.829nm
18	ICP-OES-AV					383.829
19	ICP-OES-RV	Yttrium			50	279.077
21	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc				
24	ICP-OES-AV-buffer	Cs, Eu			25	383.829 nm
25						
26	NA					
27	ICP-OES-AV	175 Lu	NA	NA	20	279.077 nm
28	ICP-OES	Eu	NA		1	383.8
29	NA	NA	NA	NA	NA	NA
30	NA	NA	NA	NA	NA	NA

Table 1189 Instrument Conditions Exchangeable Na<sup>+</sup>

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/Ion(m/z)/Absorbance(nm)
1						
2	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV-buffer				100	588.995
4	ICP-OES-RV					589.592
5	ICP-OES-AV				20	589.592
6	NA	NA	NA	NA	NA	NA
7	ICP-OES-AV					
8						
9	NA	NA	NA	NA	NA	NA
10	ICP-OES-RV	Y	NA	NA	40	589.592
11	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV	Y	NA		20	589.592
13						
14	ICP-OES-AV-buffer	Yttrium	No	No	10	589.592
15	NT					
16	ICP-OES-AV	Lutetium	NA	NA	100	588.995
17	ICP-OES	Eu & Cs	NA	NA	500	330.237, 589.592nm
18	ICP-OES-AV					589.592
19	ICP-OES-RV	Yttrium			50	589.592
21	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc				
24	ICP-OES-AV-buffer	Cs, Eu			25	589.592 nm
25						
26	NA					
27	ICP-OES-AV	175 Lu	NA	NA	20	589.592 nm
28	ICP-OES	Eu	NA		1	766.5
29	NA	NA	NA	NA	NA	NA
30	NA	NA	NA	NA	NA	NA

**Table 120 Instrument Conditions Exchangeable K<sup>+</sup>**

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/Ion(m/z)/Absorbance(nm)
1						
2	NA	NA	NA	NA	NA	NA
3	ICP-OES-AV-buffer				100	766.491
4	ICP-OES-RV					253.7
5	ICP-OES-AV				20	766.491
6	NA	NA	NA	NA	NA	NA
7	ICP-OES-AV					
8						
9	NA	NA	NA	NA	NA	NA
10	ICP-OES-RV	Y	NA	NA	40	766.491
11	NA	NA	NA	NA	NA	NA
12	ICP-OES-RV	Y	NA		20	766.49
13						
14	ICP-OES-AV-buffer	Yttrium	No	No	10	766.491
15	NT					
16	ICP-OES-AV	Caesium	NA	NA	100	766.491
17	ICP-OES	Eu & Cs	NA	NA	500	404.721nm, 766.491nm
18	ICP-OES-AV					769.897
19	ICP-OES-RV	Yttrium			50	766.49
21	NA	NA	NA	NA	NA	NA
22	ICP-OES-AV	Sc				
24	ICP-OES-AV	Cs, Eu			25	769.897 nm
25						
26	NA					
27	ICP-OES-AV	175 Lu	NA	NA	20	766.490 nm
28	ICP-OES	Eu	NA		1	589.6
29	NA	NA	NA	NA	NA	NA
30	NA	NA	NA	NA	NA	NA

**END OF REPORT**