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Institute

Proficiency Test Final Report AQA 24-15 Metals, Nutrients and Anions in Soil

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SUMMARY

This report presents the results of the proficiency test AQA 24-15 Metals, Nutrients and Anions in Soil. The study covers the measurement of acid extractable elements: Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cs, Cu, Fe, Hg, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Rb, S, Sb, Se, Sn, Sr, U, V and Zn. Measurement of pH, electrical conductivity (EC), water soluble bromide (Br^-), chloride (Cl^-), fluoride (F^-), iodide (I^-), orthophosphate-P ($\text{PO}_4^{3-}\text{-P}$), and sulphate (SO_4^{2-}) and of total Kjeldahl nitrogen (TKN), 2M KCl extractable ammonium nitrogen ($\text{NH}_4^+\text{-N}$) and 2M KCl extractable nitrate nitrogen ($\text{NO}_3^-\text{-N}$) were also included in the program.

The sample set consisted of one dried soil sample, one dried biosolid sample, and one dried agricultural soil sample.

Thirty laboratories registered to participate, and twenty-nine submitted results.

The assigned values were the robust average of participants' results. The associated uncertainties were estimated from the robust standard deviation of the participants' results.

The outcomes of the study were assessed against the aims as follows, to:

- i. *compare the performance of participant laboratories and assess their accuracy;*

Laboratory performance was assessed using both z-scores and E_n -scores.

Of 886 z-scores, 827 (93%) were acceptable with $|z| \leq 2.0$.

Of 886 E_n -scores, 723 (82%) were acceptable with $|E_n| < 1.0$.

Laboratory 23 returned the highest number of acceptable z scores (49 out of 49 reported).

Laboratory 10 returned the highest number of acceptable E_n -scores (46 out of 46 reported).

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

Sample S1 challenged some participants' methods. This was a low-level natural sandy soil sample often encountered by laboratories in their routine operation and aims to support laboratories' method performance with this type of matrix. The precision of some participants' methods hampered their attempts to report results for this sample.

Some laboratories extracted their samples using dilute acids. Most of the results reported by the aforementioned laboratories, for acid extractable elements were lower than the assigned value. Dilute acids might extract only a fraction of some elements from the contaminated soil.

Cs and Sb in S2 followed by low level Ni, Sr, Cd and Rb in S1 and B in S1 and S2 presented analytical difficulty to participating laboratories. A limited number of laboratories reported results for these tests or/and the reported results were variable.

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

Over the last 26 studies of inorganic analytes in soil, the average proportion of acceptable scores was 90% for z-scores and 80% for E_n -scores.

- 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 930 numerical results, 913 (98%) were reported with an expanded measurement uncertainty. The magnitude of the reported expanded uncertainties was within the range 0.24% to 1633% of the reported value. An example of estimating measurement uncertainty using proficiency testing data only is given in Appendix 3.

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

The test samples of this study were checked for homogeneity and are well characterised, both by in-house testing and from the results of the proficiency round. Surplus test samples are 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 inter-laboratory comparison.”¹ NMI PT studies target chemical testing in areas of high public significance such as trade, environment, and food safety. NMI offers studies in:

- inorganic analytes in soil, water, food and pharmaceuticals;
- pesticide residues in fruit and vegetables, soil and water;
- petroleum hydrocarbons in soil and water;
- PFAS in water, soil, biosolid, biota and food;
- chlorophyll a in water; and
- controlled drug assay, drugs in wipes and clandestine laboratory.

AQA 24-15 is the 35th 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;
- compare the performance of participant laboratories with their past performance;
- develop the practical application of traceability and measurement uncertainty; and
- produce materials that can be used in method validation and as control samples.

1.3 Study Conduct

The conduct of NMI proficiency tests is described in the NMI Chemical Proficiency Testing Study Protocol.² The statistical methods used are described in the NMI Chemical Proficiency Statistical Manual.³ These documents have been prepared with reference to ISO Standard 17043¹ and The International Harmonized Protocol for Proficiency Testing of (Chemical) Analytical Laboratories.⁴

NMI is accredited by National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043:2023 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 55 tests (in both matrices) were selected from those for which an investigation level is published in the Guideline on the Investigation Levels for Soil and Groundwater, promulgated by the National Environmental Protection Council (NEPC)⁵ and from analytes commonly measured in soil.

2.2 Participation

Thirty laboratories participated and twenty-nine submitted results.

The timetable of the study was:

Invitation issued:	26 August 2024
Samples dispatched:	16 September 2024
Results due:	10 October 2024
Interim report issued:	22 October 2024
Preliminary report issued:	24 October 2024

The due date for results was extended to 18 October 2024. This is a large and complex study, and we received multiple requests for the results deadline to be extended due to staff shortages.

2.3 Test Material Specification

Three samples were provided for analysis:

Sample S1 was 30 g of dried soil;

Sample S2 was 30 g of dried biosolid, a reference material previously prepared by NMI; and

Sample S3 was 75 g of dried agricultural soil.

2.4 Laboratory Code

All participant laboratories were assigned a confidential code number.

2.5 Sample Preparation, Analysis and Homogeneity Testing

A full homogeneity test was conducted for acid extractable elements in S1, except for Na, Rb and Sn. Participants' results gave no reason to question the homogeneity of these tests; all reported results, except one, were in good agreement with each other. No homogeneity test was conducted for acid extractable elements in S2. This was a reference material prepared by NMI and the homogeneity of this material has been previously established.

Sample S3 was prepared and packaged using a process that has been demonstrated to produce homogeneous samples in previous studies. Therefore, only a partial homogeneity test was conducted, excepting fluoride, iodide, orthophosphate-P and S. The results from the partial homogeneity tests 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 in the present study. Stability studies conducted for the previous proficiency tests of inorganic analytes in soil found no significant changes in any of the analytes' concentration.

2.7 Sample Storage, Dispatch and Receipt

The samples were dispatched by courier on 16 September 2024.

A description of the test samples and instructions for participants, and a form for participants to confirm the receipt of the test samples, were sent with 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. For *Sample S3* for 2M KCl Extractable (NO_3^- -N) and (NH_4^+ -N), participants are asked to follow the extraction procedure described below:
 “Prepare a 1:10 w/v soil/2M KCl extracting solution. For example, weigh 5 g of soil into a suitable bottle or jar and add 50 mL of 2M KCl extracting solution. Mechanically shake (end-over-end preferred), at room temperature for 1 h. Allow around 20-30 min for soil to settle and clarify and then take a known aliquot for the measurement technique employed. Further dilution of the aliquot may be required.”
 Measure the analytes using a colorimetric method; and report results of 1:10 soil/2M KCl extracting solution on as received basis in units of mg/kg for: 2M KCl extractable ammonium-N (NH_4^+ -N) and 2M KCl extractable nitrate-N (NO_3^- -N).
- Report on as received basis in units of mg/kg except for EC and pH. EC results are to be reported in units of $\mu\text{S}/\text{cm}$.

SAMPLE S1 Dried Soil		SAMPLE S2 Dried Biosolid		SAMPLE S3 Dried Agricultural Soil	
Test Acid Extractable	Estimated Values* [mg/kg]	Test Acid Extractable	Estimated Values* [mg/kg]	Test	Estimated Values* [mg/kg]
Al	200-8000	Ag	0.5-20	Ca (acid extractable)	250-10000
As	0.5-20	As	0.5-20	Fe (acid extractable)	>10000
B	0.5-20	B	0.5-20	K (acid extractable)	1500-60000
Ba	5-200	Be	0.05-2	Mg (acid extractable)	250-10000
Cd	<5	Bi	0.05-2	Na (acid extractable)	250-10000
Cr	0.5-20	Cd	0.05-2	P (acid extractable)	30-1200
Co	0.5-20	Cs	0.5-20	S (acid extractable)	NA
Cu	5-200	Cr	5-200	Water Soluble Bromide (Br^-) - 1:5 soil/water extract	<20
Li	<5	Fe	1000-40000	Water Soluble Chloride (Cl^-) - 1:5 soil/water extract	<2000
Mn	5-200	Hg	0.05-2	Water Soluble Fluoride (F^-) - 1:5 soil/water extract	<10
Na	30-1200	Mn	30-1200	Water Soluble Iodide (I^-) - 1:5 soil/water extract	<20
Ni	0.5-20	Mo	0.5-20	Water Soluble Orthophosphate-P (PO_4^{3-} -P) - 1:5 soil/water extract	<5
Pb	5-200	Ni	5-200	Water Soluble Sulphate (SO_4^{2-}) 1:5 soil/water extract	5-200
Rb	0.5-20	Pb	5-200	pH of 1:5 soil/water suspension	2-12
Sn	0.5-20	Rb	0.5-20	EC of 1:5 soil/water extract	30-1200
Sr	0.5-20	Se	0.5-20	Kjeldahl nitrogen, total (TKN)	30-1200
V	0.5-20	Sb	0.05-2	2M KCl Extractable (Nitrate-N)	5-200
Zn	5-200	U	0.5-20	2M KCl Extractable (Ammonium-N)	5-200
		Zn	30-1200		

2.9 Interim and Preliminary Reports

An interim report was emailed participants on 22 October 2024. A preliminary report was issued on 24 October 2024. This report included: a summary of the results reported by laboratories, assigned values, performance coefficient of variations, z-scores and E_n -scores for each analyte tested by participants. There was a change in the uncertainty of the assigned values for As in S1 and S2, from 0.6 mg/kg to 0.7 mg/kg for both samples. There was no significant change in participants' E_n -scores.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Method Summaries

Summaries of test methods are transcribed in Tables 1 to 10.

Table 1 Methodology for Acid Extractable Elements

Lab. Code	Method Reference	Staggered Digestion	Sample Mass (g)	Temp. (°C)	Time (min)	Vol. HNO ₃ (mL)	Vol. HCl (mL)	Vol. HNO ₃ (1:1) (mL)	Vol. HCl (1:1) (mL)	Vol. H ₂ O ₂ (mL)	Other (mL)
1*	USEPA 3050		1	95	60	10				6	
2	USEPA method 200.02 Revision 2.8	NA	1	95	60			2	10	2	
3*	In-house method		0.25	100-120-140	180	2.5				2.5	
4	Acid Digestion of sediment, sludges and soil-USEPA 3050		1	95	90	3	3				
5	US EPA 3050B	Yes	0.5	95	120	7.5	5			1.5	
6	US-EPA 200.2		1	95	50	2	2				10 (H ₂ O)
7	In-House Method		2	100	60	4	8			4	
8	200.2 Revision 2.8	Yes	1	95 ± 5	60	2	10			2	
9	AOAC 990.08	NA	0.5	85	240	3	2				
10	USEPA Method 6010c, USEPA Methods 7471B, 7470A, 7471B	No	1	90 - 98	90	3	3				
11	USEPA Method 200.8 Determination of trace elements in waters and wastes by ICP-MS		1	95	120	2	1				
12	EPA Method 200.7	No	0.5	95	120	2	2				10 (H ₂ O)
13	In House - referencing APHA 3125	No	0.4	120	60	2.5	7.5				
14*	USEPA Method 3050.	Yes	3	95	120	10	5	10		6	
15	USEPA 3010		2	95-105	60	4	12				
17*	APHA 3125 (Modified)	No	1	110	65	3.5	1.5				
18	US EPA METHOD 3010	No	2	95-105	60	4	12				4 (H ₂ O)
19	USEPA Method 200.2, Revision 2.8	Yes	1	95	60			2		2	10 (HCl 1:4)
20	USEPA Method 6010c, USEPA Methods 7471B, 7470A, 7471B		1	90-98	90	3	3				
21*	200.2		1	98	90	3	3				
22	Inhouse Method	No	1.5	95	90	1	1.5			1	
23	AS4479; USEPA 3050; 200.8; 200.7, 6010, 6020		1	100	120	3	3				
24	USEPA 200.2 Rev 2.8	Yes	1	95	60			2		2	10 (HCl 1:4)
25	EPA (Environmental Protection Agency) 1994 Method 200.8		2	109	60	800	400				1200
26			2.5	95	90	4	12			2	
29	In House, US EPA 6020B		2	95-105	60	4	12				4 (H ₂ O)
30	In house method	No	0.5	95	60	9	3				

*Additional information in Table 10.

Table 2 Methodology for Total Kjeldahl Nitrogen

Lab. Code	Method Reference	Digestion	Distillation	Other Test Method	Measurement Method	Instrument
1*	APHA 4500B	Yes	Yes		Titrimetric Method	FOSS Digester
2	APHA 4500 - Norg. A & D.	Yes	No		Colorimetric - salicylate method	DA
3*	According to EN 13342 and DIN ISO 11261, modified by BUCHI (BUCHI 2013)	Yes	Yes		Titrimetric method	Manual Analysis
4	APHA 4500	Yes			Colorimetric - salicylate method	DA
7	In-House Method	Yes			Colorimetric - salicylate method	FIA
8	APHA 22nd edition 4500 Norg A & D with Jirka Modification-Jirka et al. (1976)	Yes	No		Colorimetric - phenate method	DA
9	ISO 11261	Yes	Yes		Titrimetric method	Kjeldal
10	APHA latest Edition. Analytical Methods for Waters and Wastewaters 4500-Inorg-D	Yes	No		Colorimetric - salicylate method	DA
11	APHA, Standard Methods for the Examination of Water and Wastewater, Method No. 4500-Norg-D (TKN Block Digestion and FIA) and 4130 (Inorganic Non-metals by FIA)	Yes	Yes		Colorimetric - salicylate method	FIA
13*					TKN = TN-NO _x (Dumas)	LECO
14*	APHA Method 4500-N Org D	Yes	No	No	Colorimetric - salicylate method	DA
15	APHA 4500-Norg-B,C,D. (4-123)(TKN)	Yes	No		Colorimetric - phenate method	DA
17	APHA: 4500-N(org) B	Yes	Yes		Titrimetric method	NA
19	APHA 4500-Norg D	Yes	No		Colorimetric - salicylate method	DA
20	APHA 4500-Norg-D	Yes	Yes		Colorimetric - salicylate method	DA
22	Inhouse	Yes	No		Colorimetric - salicylate method	FIA
23		Yes	Yes		Titrimetric method	Foss
24	APHA 4500	Yes			Colorimetric - phenate method	DA

*Additional information in Table 10.

Table 3 Methodology for 2M KCl Extractable Ammonium-N and Nitrate-N

Lab. Code	Method Reference		Sample Mass (g)	Extraction Solution 2M KCl Volume (mL)	Shake time (hours)	Measurement Method		Measurement Instrument	
	NH4+-N	NO3--N				NH4+-N	NO3--N	NH4+-N	NO3--N
1	APHA 4500G	APHA 4500F	10	100	1	Colorimetric - Phenate method	Colorimetric - Sulfanilamide - NEDD Cd Reduction	FIA	FIA
3	Method 10205 TNT Plus 831-HACH	Method 8171 Cadmium Reduction-HACH	5	50	1	Colorimetric - Salicylate method	Colorimetric-Sulfanilamide-NEDD Cd reduction	Manual Analysis DR 1900 HACH.	Manual Analysis DR 1900 HACH
4	APHA	APHA	5	50	2	Colorimetric - Phenate method	Colorimetric - Vanadium III method	DA	DA
7		In-House Method	10	50	1		Colorimetric - Vanadium III method		DA
10	APHA latest Edition. Analytical Methods for Waters and Wastewaters 4500-Inorgs-D	APHA latest Edition. Analytical Methods for Waters and Wastewaters 4500-NO2-B, 4500-NO3 E and 4500-N C	5	25	1.5	Colorimetric - Phenate method	Colorimetric - Vanadium III method	DA	DA
13	In House Method	In House Method	2	20	1	Colorimetric - Salicylate method	Colorimetric - Vanadium III method	FIA	FIA
14	Rayment and Lyons Method 7C1	NA	5	50	1 hr	OPA method	NA	FIA	NA
16	7C2b	7C2b	10	100	1	Colorimetric - Salicylate method	Colorimetric-Sulfanilamide-NEDD Cd reduction	FIA	FIA
20			5	25	1	Colorimetric - Phenate method	Colorimetric - Vanadium III method	DA	DA
22	Inhouse	Inhouse	3	30	1	Colorimetric - Phenate method	Colorimetric-Sulfanilamide-NEDD Cd reduction	FIA	FIA
23			8	80	1 hour	SFA with fluorescence detector	Colorimetric-Sulfanilamide-NEDD Cd reduction	SFA	SFA
24	1:5 KCl by FIA								

*Additional information in Table 10.

Table 4 Methodology for Water Soluble Bromide

Lab. Code	Method Reference	Sample Mass (g)	Water Volume (mL)	Shake Time (hours)	Measurement Method	Measurement Instrument
4	APHA	5	25	2	Ion Chromatographic Method	IC
7	In-House Method	10	50	1	Ion Chromatographic Method	IC
8	APHA 4110	10	50	1 hour	Ion Chromatographic Method	IC
10	APHA latest Edition. Analytical Methods for Waters and Wastewaters, 4110b. Ion Chromatography with Chemical Suppression of Eluent Conductivity	10	50	1.5	Ion Chromatographic Method	IC
11		25	125	1		
14	APHA 4110 D	10	50	1	Ion Chromatographic Method	IC
18	USEPA method 9056A	8	40	1	Ion Chromatographic Method	IC
20	APHA 4110 b	16	80	1	Ion Chromatographic Method	IC
21	APHA 4110B and C	5	25	3	Ion Chromatographic Method	IC
23		8	40	1 hour	Ion Chromatographic Method	IC
24	APHA 4110	10	50	1	Ion Chromatographic Method	IC

Table 5 Methodology for Water Soluble Chloride

Lab. Code	Method Reference	Sample Mass (g)	Water Volume (mL)	Shake Time (hours)	Measurement Method	Measurement Instrument
1	APHA 4110B	10	50	1	Ion Chromatographic Method	Dionex
2	In House	10	50	1	Ferricyanide Colorimetric Method	DA
3	USEPA Silver Nitrate Buret Titration Method 8225-HACH	20	100	1	Argentometric Titration	Titration
4	APHA	5	25	2	Ion Chromatographic Method	IC
5	APHA 4110 B	12	60	1	Ion Chromatographic Method	IC
7	In-House Method	10	50	1	Ferricyanide Colorimetric Method	DA
8	APHA 4500-Cl-	10	50	1 hour	Ferricyanide Colorimetric Method	DA
10	APHA latest Edition. Analytical Methods for Waters and Wastewaters, 4110b. Ion Chromatography with Chemical Suppression of Eluent Conductivity.	10	50	1.5	Ion Chromatographic Method	IC
11	EPA Method 325.2 and APHA Standard Method 4500-Cl--E	25	125	1	Mercuric Thiocyanate	DA
13		2	10	1	Mercuric Nitrate Titration	Segmented Flow Analyser
14	APHA Method 4500-Cl- G	10	50	1	Mercuric Thiocyanate	DA
15	APHA-4500-Cl E	5	50	1	Ferricyanide Colorimetric Method	DA
16	5A1				Mercuric Thiocyanate	FIA
18	USEPA method 9056A	8	40	1	Ion Chromatographic Method	IC
19	APHA 4500 Cl - G	10	50	1	Mercuric Thiocyanate	DA

Lab. Code	Method Reference	Sample Mass (g)	Water Volume (mL)	Shake Time (hours)	Measurement Method	Measurement Instrument
20	APHA 4110 b	16	80	1	Ion Chromatographic Method	IC
21	APHA 4110B and C	5	25	3	Ion Chromatographic Method	IC
23		8	40	1 hour	Ion Chromatographic Method	IC
24	APHA 4500 CL E	10	50	1	Mercuric Thiocyanate	DA

Table 6 Methodology for Water Soluble Fluoride

Lab. Code	Method Reference	Sample Mass (g)	Water Volume (mL)	Shake Time (hours)	Measurement Method	Measurement Instrument
1	APHA 4500C	10	50	1	Ion Selective Electrode Method	Orion
2	In House	10	50	1	Ion Selective Electrode Method	Selective Electrode
3	USEPA SPADNS Method 8029- HACH	20	100	1	Other	DR 1900 HACH
4	APHA	5	25	2	Ion Selective Electrode Method	Selective Electrode
7	In-House Method	10	50	1	Ion Chromatographic Method	IC
8	APHA 4500-F-	10	50	1 hour	Ion Selective Electrode Method	Selective Electrode
10	APHA latest Edition. Standard Methods for the Examination of Water and Wastewater, 4500 F-C	10	50	1.5	Ion Selective Electrode Method	Selective Electrode
11		25	125	1	Ion Selective Electrode Method	Selective Electrode
13		2	10	1	Ion Selective Electrode Method	Selective Electrode
14	APHA Method 4500-F- C	10	50	1	Ion Selective Electrode Method	Selective Electrode
19	APHA 4500-F C	10	50	1	Ion Selective Electrode Method	Selective Electrode
20	APHA 4500 F C	16	80	1	Ion Selective Electrode Method	Selective Electrode
21		5	25	3		
23		8	40	1 hour	Ion Chromatographic Method	IC
24	APHA 4500-F- A & C	10	50	1	Other	Selective Electrode

Table 7 Methodology for Water Soluble Iodide

Lab. Code	Method Reference	Sample Mass (g)	Water Volume (mL)	Shake Time (hours)	Measurement Method	Measurement Instrument
4	APHA	5	25	2	Ion Chromatographic Method	IC
7	In-House Method	10	50	1	Ion Chromatographic Method	IC
8	APHA 4110	10	50	1 hour	Ion Chromatographic Method	IC
11	Water Analysis with Instrumentation from Radiometer Analytical. Iodide In Water. 918-502-9007A	25	125	1	Ion Selective Electrode Method	Iodide Selective Electrode
14	APHA Method 4110 D	10	50	1	Ion Chromatographic Method	IC
18	USEPA method 9056A	8	40	1	Ion Chromatographic Method	IC
20	APHA 4110 b	16	80	1	Ion Chromatographic Method	IC
21		5	25	3		
23		8	40	1 hour	Ion Chromatographic Method	IC
24	APHA 4110	10	50	1	Ion Chromatographic Method	IC

Table 8 Methodology for Water Soluble Orthophosphate-P

Lab. Code	Method Reference	Sample Mass (g)	Water Volume (mL)	Shake Time (hours)	Measurement Method	Measurement Instrument
1	APHA 4500F	10	50	1	Ascorbic Acid Colorimetric Method	FIA
2	In House	10	50	1	Vanadomolybdophosphoric Colorimetric Method	DA
3	USEPA PhosVer 3 (Ascorbic Acid) Method 8048-HACH	20	100	1	Ascorbic Acid Colorimetric Method	Manual Analysis DR 1900 HACH
4	APHA	5	25	2	Ascorbic Acid Colorimetric Method	DA
7	In-House Method	10	50	1	Ascorbic Acid Colorimetric Method	DA
8	APHA 4500-P	10	50	1 hour	Ascorbic Acid Colorimetric Method	DA
10	APHA latest Edition. Standard Methods for the Examination of Water and Wastewater, 4500-P E&J	10	50	1.5	Ascorbic Acid Colorimetric Method	DA
11	USEPA method 365.2 Phosphorus colorimetric	25	125	1	Ascorbic Acid Colorimetric Method	DA
13		2	10	1	Vanadomolybdophosphoric Colorimetric Method	FIA
14	APHA Method 4500-P F	10	50	1	Ascorbic Acid Colorimetric Method	DA
15	APHA 4500-P-B-D	5	50	1	Ascorbic Acid Colorimetric Method	DA
18	APHA 21st Edition 4500-P E	8	40	1	Ascorbic Acid Colorimetric Method	DA
19	APHA 4500-P F	10	50	1	Ascorbic Acid Colorimetric Method	DA
20	APHA 4500-P E&J	16	80	1	Vanadomolybdophosphoric Colorimetric Method	DA

Lab. Code	Method Reference	Sample Mass (g)	Water Volume (mL)	Shake Time (hours)	Measurement Method	Measurement Instrument
21	APHA 4110B and C	5	25	3	Ion Chromatographic Method	IC
23		8	40	1 hour	Ion Chromatographic Method	IC
24	APHA 4500-P A,B & F	10	50	1	Ascorbic Acid Colorimetric Method	DA

*Additional information in Table 10.

Table 9 Methodology for Water Soluble Sulphate

Lab. Code	Method Reference	Sample Mass (g)	Water Volume (mL)	Shake Time (hours)	Measurement Method	Measurement Instrument
1	APHA 4110B	10	50	1	Ion Chromatographic Method	Dionex
2	In House	10	50	1	ICP-Method	ICP-OES
3	USEPA SulfaVer 4 Method 8051-HACH	20	100	1	Turbidimetric Method	Manual Analysis DR 1900 HACH
4	APHA	5	25	2	Ion Chromatographic Method	IC
5	APHA 4110 B	12	60	1	Ion Chromatographic Method	IC
7	In-House Method	10	50	1	Colorimetric Method	DA
8	APHA 3120	10	50	1 hour	ICP-Method	ICP-OES
10	APHA latest Edition. Analytical Methods for Waters and Wastewaters, 4110b. Ion Chromatography with Chemical Suppression of Eluent Conductivity.	10	50	1.5	Ion Chromatographic Method	IC
11	USEPA method 375.4 Sulfate Turbimetric	25	125	1	Turbidimetric Method	DA
13		2	10	1	ICP-Method	ICP-OES
14*	USEPA Method 6010D (2018) and APHA Method 3120	10	50	1	ICP-Method	ICP-OES
15	APHA 4500 - SO ₄ E	5	50	1	Turbidimetric Method	DA
18	USEPA method 9056A	8	40	1	Ion Chromatographic Method	IC
19	APHA 3120	10	50	1	ICP-Method	ICP-OES
20	APHA 4110 b	16	80	1	Ion Chromatographic Method	IC
21	APHA 4110B and C	5	25	3	Ion Chromatographic Method	IC
23		8	40	1 hour	Ion Chromatographic Method	IC
24	1:5 Soluble Sulphate	10	50	1	ICP-Method	ICP-OES

*Additional information in Table 10.

3.2 Instruments Used for Measurements

The instruments and settings used by participants for acid extractable elements are presented in Appendix 5.

3.3 Additional Information

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

Table 10 Additional information

Lab Code	Additional Information
1	Acid Extractable Elements: - Instrumental Technique: Air-C ₂ H ₂ and N ₂ O used for AAS elements, Li and Sr by Emission AAS - Methodology: Leached in 10% HCl after digestion before making to volume Total Kjeldahl Nitrogen: ammonia distilled by FOSS distillation system.
3	Acid Extractable Elements: - Instrument Technique: All measurements were done using AAS-Atomic Absorption Spectrometer (Thermo Scientific) except Phosphorus which was analysed using the Olsen method and measured using Specord 50 UV-VIS Spectrophotometer. - Methodology: Final digest is filtered into a 50ml tube. Digestion tube is rinsed a few times with distilled water and filtered into the same tube. The 50ml tube is then filled to the mark before a properly mixed aliquot is taken for analysis. Further dilution might be required. Total Kjeldahl Nitrogen: pH endpoint titration.
13	Total Kjeldahl Nitrogen: TKN = TN by LECO and NO _x by FIA
14	Acid Extractable Elements: - Instrument Technique: Instrument for Hg: Cetac Hg Analyser Total Kjeldahl Nitrogen: Block Digestion and DA Sulphate: Sulphur results from OES was converted to SO ₄ since the DA results were too negative indicating possible matrix interference.
17	Acid Extractable Elements: Methodology: Reverse Aqua Regia S1: Results reported are the average of replicates S3: Metals: Lower sample mass digested (0.25g)
21	Acid extractable elements: Methods- ISO 17025 accreditation only applies to lead results. Instrumental technique- See previous submissions

3.4 Basis of Participants' Measurement Uncertainty Estimates

Participants were requested to provide information about the basis of their uncertainty estimates (Tables 11 and 12).

Table 11 Basis of Uncertainty Estimate

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation ^a		Guide Document for Estimating MU
		Precision	Method Bias	
1	Top Down - precision and estimates of the method and laboratory bias k = 2	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration Recoveries of SS	Eurochem Guide 2007
2	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - CRM Duplicate Analysis	CRM Instrument Calibration	Eurachem/CITAC Guide
3	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration	

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation ^a		Guide Document for Estimating MU
		Precision	Method Bias	
4	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples	Recoveries of SS	ISO/GUM
5	Top Down - precision and estimates of the method and laboratory bias k = 2	Control Samples - CRM Duplicate Analysis	CRM Recoveries of SS	
6	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control Samples - CRM Duplicate Analysis	CRM Instrument Calibration Laboratory Bias from PT Studies	Eurachem/CITAC Guide
7	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Control Samples - CRM	CRM Instrument Calibration Recoveries of SS	
8	Top Down - precision and estimates of the method and laboratory bias k = 2	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration	Eurachem/CITAC Guide
9	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - CRM Duplicate Analysis		
10	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Duplicate Analysis	Recoveries of SS	
11	Top Down - precision and estimates of the method and laboratory bias k = 2	Control Samples Duplicate Analysis	CRM	Eurachem/CITAC Guide
12	Top Down - precision and estimates of the method and laboratory bias k = 2	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration Recoveries of SS	ISO/GUM
13	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - RM Duplicate Analysis	Instrument Calibration Standard Purity	Nordtest Report TR537
14	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - SS Duplicate Analysis Instrument Calibration	Instrument Calibration Laboratory Bias from PT Studies Recoveries of SS	Calculated from in-house QA/QC plan
15	Bottom Up (ISO/GUM, fish bone/ cause and effect diagram) Coverage factor not reported	Control Samples - CRM Instrument Calibration	CRM Instrument Calibration	ISO/GUM
16	Top Down - reproducibility (standard deviation) from PT studies used directly Coverage factor not reported	Control Samples - CRM Duplicate Analysis Instrument Calibration		Nordtest Report TR537

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation ^a		Guide Document for Estimating MU
		Precision	Method Bias	
17	Other: Internal Quality Guidelines Coverage factor not reported	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration	
18*	Coverage factor not reported	Control Samples - CRM Instrument Calibration	CRM	
19	Bottom Up (ISO/GUM, fish bone/ cause and effect diagram) Coverage factor not reported		CRM Instrument Calibration Variation in Sample Moisture Content Laboratory Bias from PT Studies Recoveries of SS	Eurachem/CITAC Guide
20	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - SS		Eurachem/CITAC Guide
21*	Professional judgment k = 2	Control Samples Duplicate Analysis Instrument Calibration		
22	Bottom Up (ISO/GUM, fish bone/ cause and effect diagram) Coverage factor not reported	Duplicate Analysis Instrument Calibration	Instrument Calibration Recoveries of SS	Eurachem/CITAC Guide
23	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - CRM Duplicate Analysis		
24	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - RM Duplicate Analysis		Eurachem/CITAC Guide
25	Standard deviation of replicate analyses multiplied by 2 or 3 Coverage factor not reported	Duplicate Analysis	Recoveries of SS	
26	Top Down - precision and estimates of the method and laboratory bias k = 2	Control Samples - CRM Duplicate Analysis Instrument Calibration	CRM	ISO/GUM
28	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples Duplicate Analysis	CRM	NMI Uncertainty Course
29	Top Down - precision and estimates of the method and laboratory bias Coverage factor not reported	Control Samples - SS	Recoveries of SS	ISO/GUM
30	Professional judgment Coverage factor not reported			

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

Table 12 Additional Information for Basis of Uncertainty Estimate

Lab Code	Additional Information
18	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
21	Method uncertainty data is based on acceptance criterion of +/- 20% or 30% for control samples set by method

3.5 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.

Table 13 Participant Comments

Lab. Code	Participants' Comments	Study Coordinator's Response
1	Very good study. S3 helpful with water soluble anions. The standard deviations from this study will prove helpful with laboratory MU in future.	Thank you for your feedback.
13	Sample 1 results not reported due to sample homogeneity issues. Multiple digests were performed with poor RSD's and inconsistent results.	Thank you for your feedback. This was a natural soil sample, aimed at reproducing the samples encountered by laboratories in their routine operation. A full homogeneity assessment was conducted for this sample. No significant differences were noticed in the concentration of any of the analytes of interest (see Appendix 1).
17	S1 showed non-homogeneity.	A poor RSD is not necessarily an indication of sample inhomogeneity but also of a poor method precision. For example, a sample size of only 0.4 g might not be representative of the entire sample. Laboratories should consider adjusting the sample size taken for analyses based on the matrix type (sandy soil, compost, sludge, moist soil. etc). Alternatively, they should reassess their LOR for that matrix or adjust their estimate of uncertainty.

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participant results are listed in Tables 14 to 68 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 56. An example chart with interpretation guide is shown in Figure 1.

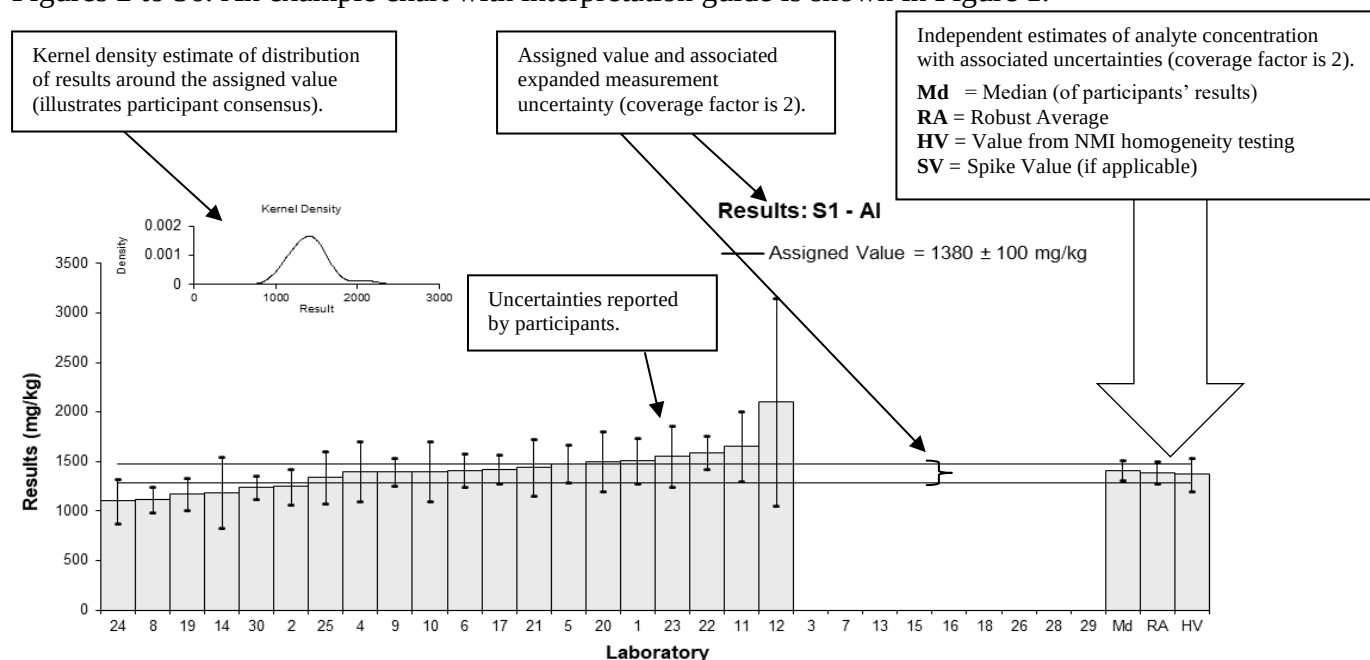


Figure 1 Guide to Presentation of Results

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.^{3, 4, 6}

4.3 Assigned Value

An example of the assigned value calculation using data from the present study is given in Appendix 2. The assigned value is defined as: 'the value attributed to a particular property of a proficiency test item.'¹ In this study the property is the mass fraction of analyte. Assigned values were the robust average of participants' results, outliers removed; the expanded uncertainties were estimated from the associated robust standard deviations.^{4, 6}

4.4 Robust Average and Robust Between-Laboratory Coefficient of Variation

The robust averages and associated expanded measurement uncertainties were calculated using the procedure described in 'Statistical methods for use in proficiency testing by interlaboratory comparisons, ISO13528.'⁶ The robust between-laboratory coefficient of variation (robust CV) is a measure of the variability of participants' results and was calculated using the procedure described in ISO13528.⁶

4.5 Target Standard Deviation for Proficiency Assessment

The target standard deviation for proficiency assessment (σ) is the product of the assigned value (X) and the performance coefficient of variation (PCV). This value is used for

calculation of participant z-score and provides scaling for laboratory deviation from the assigned value.

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

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

4.6 z-Score

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

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

Where:

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

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

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

4.7 E_n-Score

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

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

where:

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

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

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

4.8 Traceability and Measurement Uncertainty

Laboratories accredited to AS ISO/IEC Standard 17025⁸ must establish and demonstrate the traceability and measurement uncertainty associated with their test results. Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.⁹

5 TABLES AND FIGURES

Table 14

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	1510	230	0.63	0.52
2	1245.1	175	-0.65	-0.67
3	NT	NT		
4	1400	300	0.10	0.06
5	1480	190	0.48	0.47
6	1410	170	0.14	0.15
7	NT	NT		
8	1120	130	-1.26	-1.59
9	1400	140	0.10	0.12
10	1400	300	0.10	0.06
11	1650	353	1.30	0.74
12*	2100	1050	3.48	0.68
13	NR	NR		
14	1186.7	356.01	-0.93	-0.52
15	NT	NT		
16	NT	NT		
17	1424.231	150	0.21	0.25
18	NT	NT		
19	1170	162	-1.01	-1.10
20	1500	300	0.58	0.38
21	1440	288	0.29	0.20
22	1590	170	1.01	1.06
23	1550	310	0.82	0.52
24	1100	219.82	-1.35	-1.16
25	1340	268	-0.19	-0.14
26	NT	NT		
28	NT	NT		
29	NT	NT		
30	1240	120	-0.68	-0.90

* Outlier, see Section 4.2

Statistics

Assigned Value	1380	100
Homogeneity Value	1370	170
Robust Average	1390	110
Median	1410	100
Mean	1410	
N	20	
Max	2100	
Min	1100	
Robust SD	190	
Robust CV	14%	

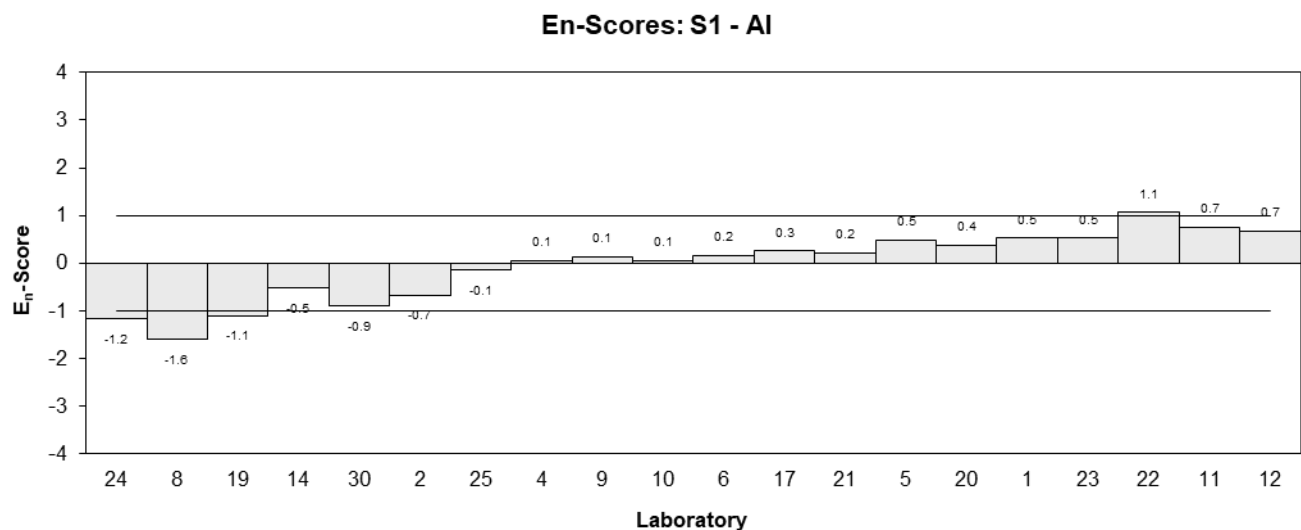
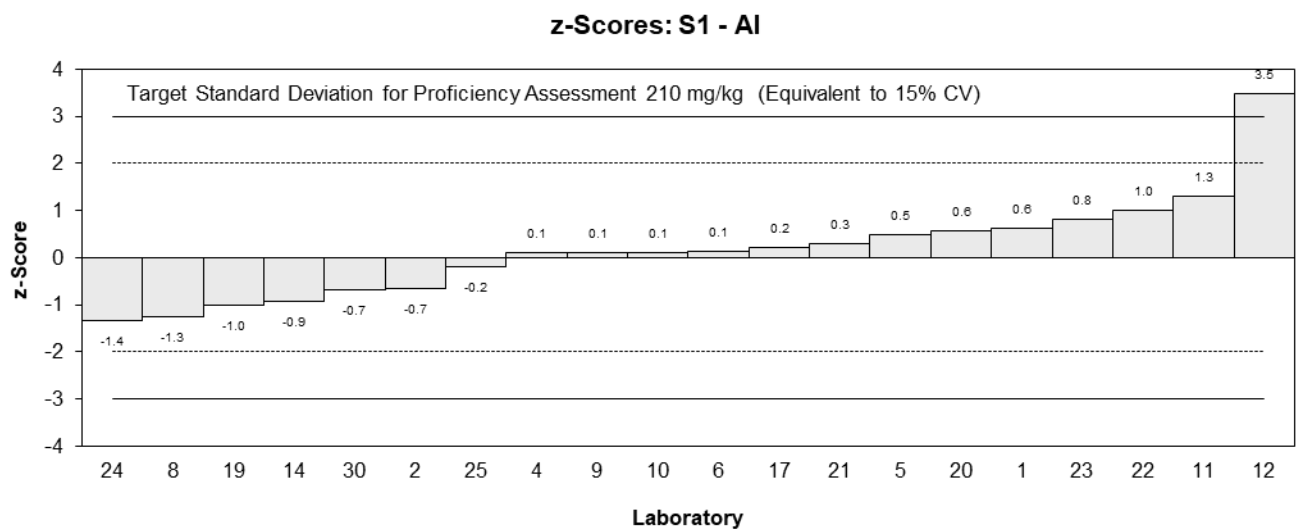
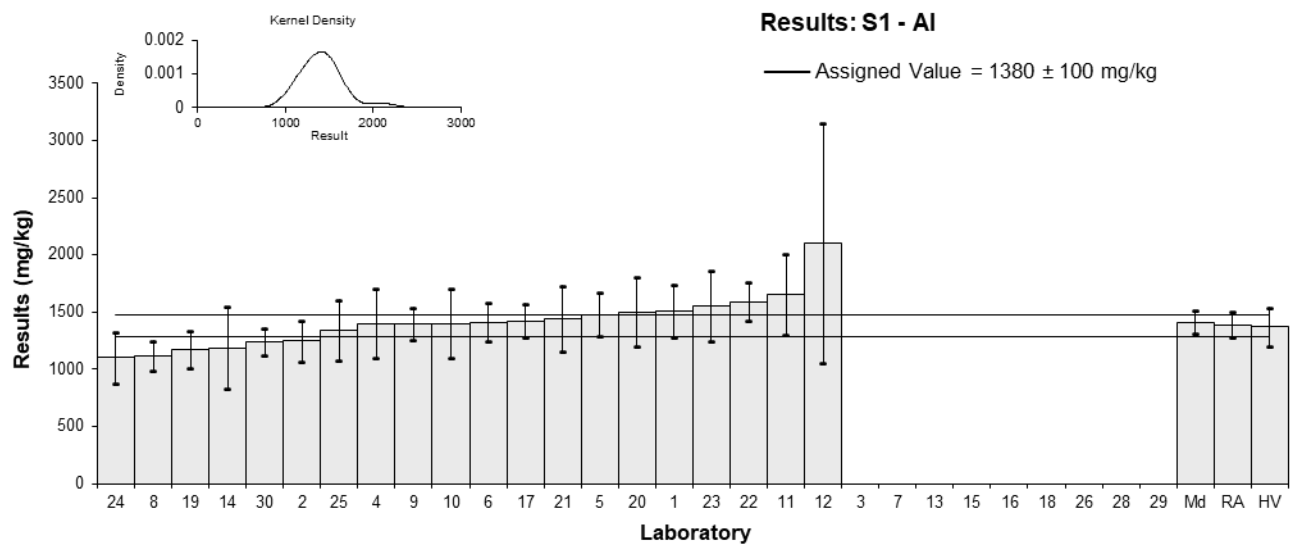


Figure 2

Table 15

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	1.5	0.50	1.94	0.80
2	1.013	0.148	-0.31	-0.32
3	NT	NT		
4	<4	4		
5	<3	NR		
6	0.98	0.17	-0.46	-0.44
7	NT	NT		
8	<5	NR		
9	1.2	0.3	0.56	0.36
10	1	1	-0.37	-0.08
11	<5	NR		
12	<5	NR		
13	NR	NR		
14	1.28	0.384	0.93	0.49
15	NT	NT		
16	NT	NT		
17	1.379	0.14	1.38	1.46
18	NT	NT		
19	<5	1		
20	1	1	-0.37	-0.08
21	0.68	0.20	-1.85	-1.60
22	0.94	0.32	-0.65	-0.40
23	0.964	0.193	-0.54	-0.47
24	0.9	0.15	-0.83	-0.85
25	1.35	0.27	1.25	0.87
26	0.88	0.30	-0.93	-0.60
28	NT	NT		
29	NT	NT		
30	1.13	0.11	0.23	0.27

Statistics

Assigned Value	1.08	0.15
Homogeneity Value	0.96	0.12
Robust Average	1.08	0.15
Median	1.00	0.11
Mean	1.08	
N	15	
Max	1.5	
Min	0.68	
Robust SD	0.23	
Robust CV	22%	

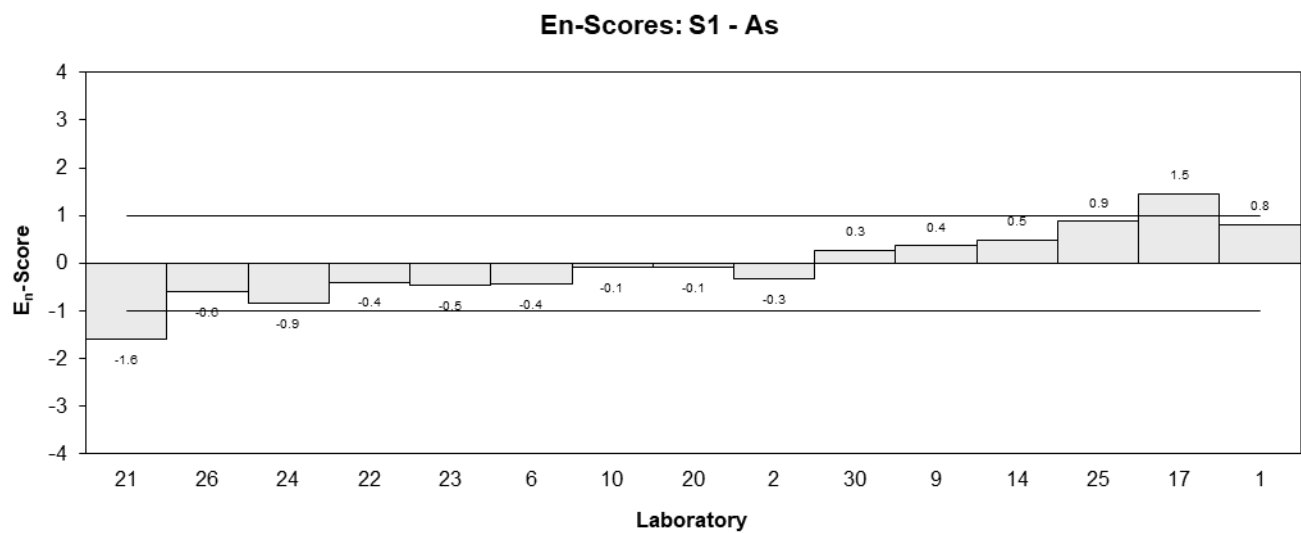
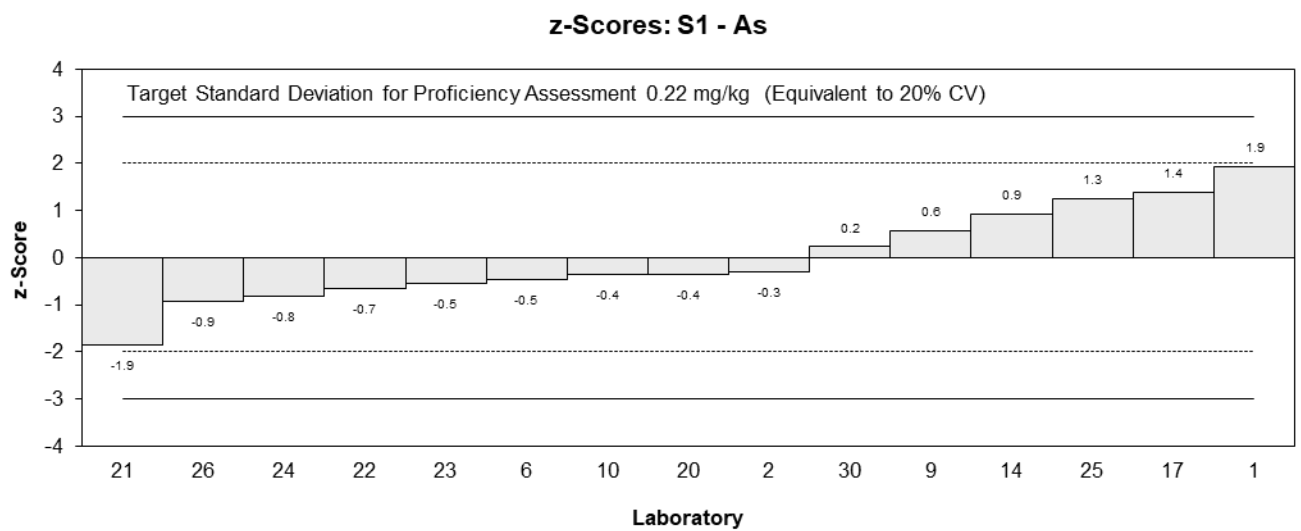
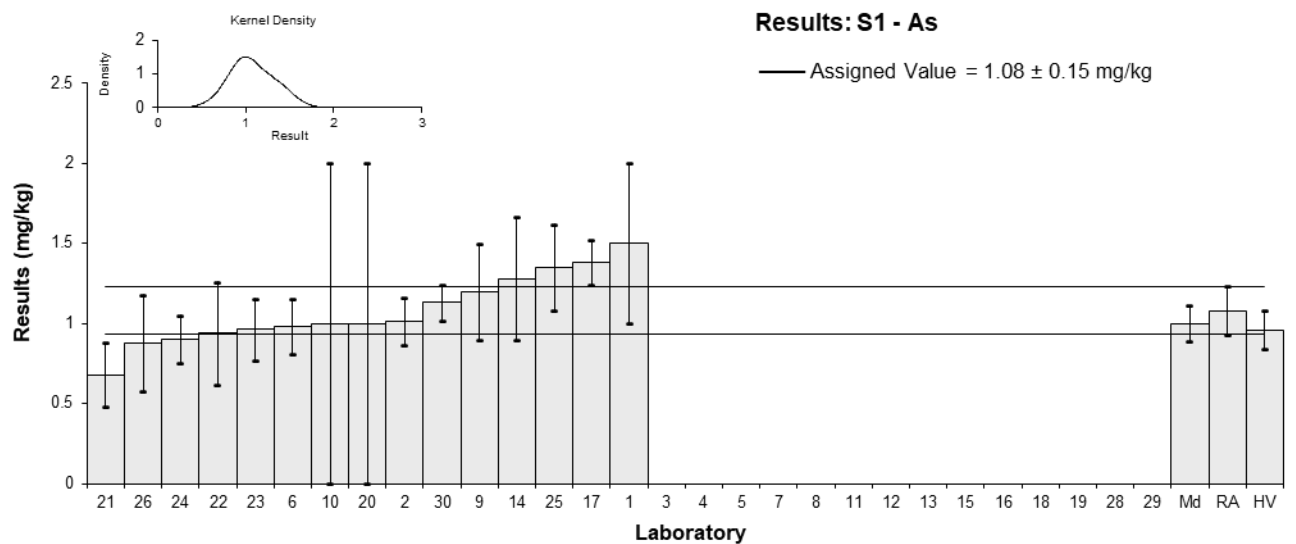


Figure 3

Table 16

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	9.5	3.3
2	<50	NR
3	NT	NT
4	<10	10
5	<2	NR
6	<20	3.6
7	NT	NT
8	<50	NR
9	<5	0.5
10	<10	10
11	<5	NR
12	1.7	0.1
13	NR	NR
14	0	5
15	NT	NT
16	NT	NT
17	<3	NR
18	NT	NT
19	<50	10
20	<10	NR
21	0.61	0.122
22	0.93	0.27
23	1.03	0.21
24	<5	NR
25	<1.3	NR
26	NT	NT
28	NT	NT
29	NT	NT
30	<5	NR

Statistics

Assigned Value	Not Set	
Homogeneity Value	1.08	0.13
Robust Average	1.2	1.1
Median	0.98	0.82
Mean	2.30	
N	6	
Max	9.5	
Min	0	
Robust SD	1.1	
Robust CV	95%	

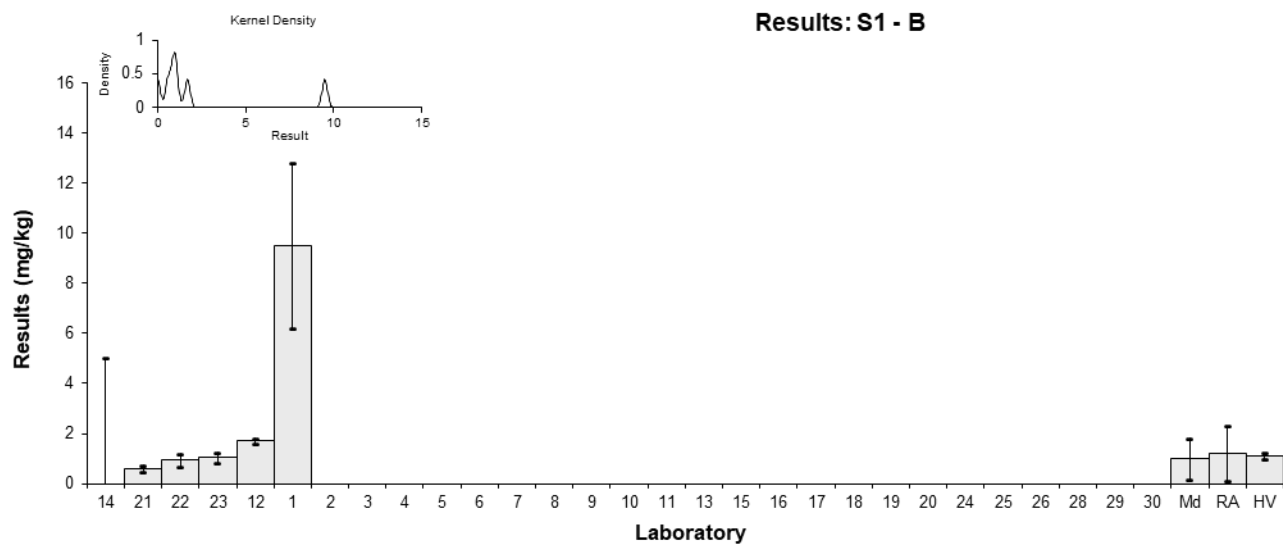


Figure 4

Table 17

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	16	2.3	0.59	0.58
2	12.95	1.532	-0.47	-0.57
3	NT	NT		
4	16	4	0.59	0.39
5	18.2	3.5	1.36	0.99
6	13.07	0.83	-0.43	-0.62
7	NT	NT		
8	10	1	-1.50	-2.09
9	12.6	1.3	-0.59	-0.77
10	15	4	0.24	0.16
11	16.3	2.72	0.70	0.61
12*	22	6.6	2.69	1.13
13	NR	NR		
14	13.53	4.059	-0.27	-0.17
15	NT	NT		
16	NT	NT		
17	NR	NR		
18	NT	NT		
19	10	2	-1.50	-1.60
20	17	4	0.94	0.62
21	NR	NR		
22	17.5	1.9	1.12	1.22
23	13.9	2.78	-0.14	-0.12
24	10.6	1.88	-1.29	-1.42
25	17.3	3.46	1.05	0.77
26	NT	NT		
28	NT	NT		
29	NT	NT		
30	12.3	1.2	-0.70	-0.92

* Outlier, see Section 4.2

Statistics

Assigned Value	14.3	1.8
Homogeneity Value	15.1	1.8
Robust Average	14.5	1.9
Median	14.5	1.7
Mean	14.7	
N	18	
Max	22	
Min	10	
Robust SD	3.2	
Robust CV	22%	

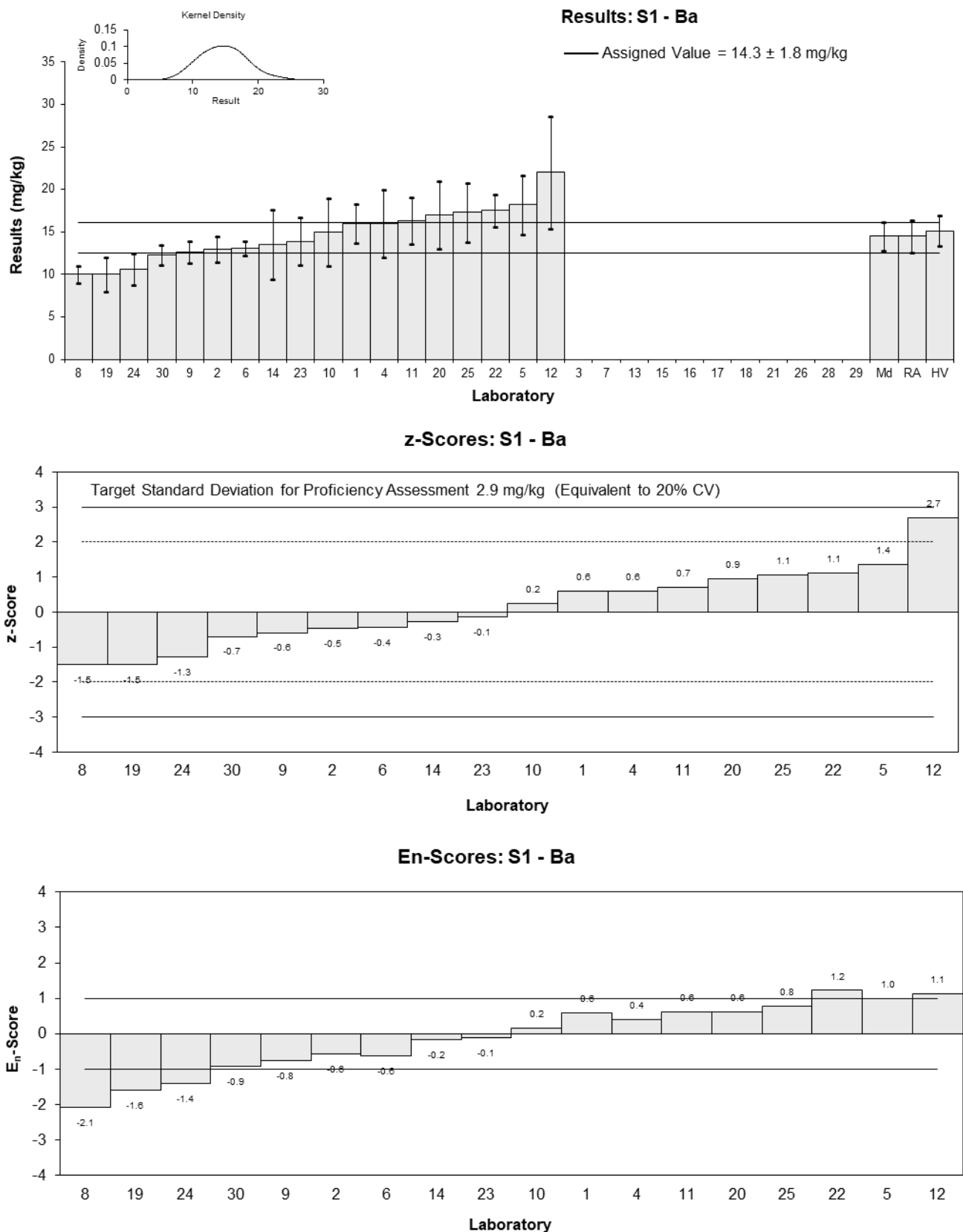


Figure 5

Table 18

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1*	0.2	0.10	24.07	1.65
2	<1	NR		
3	NT	NT		
4	<0.4	0.4		
5	<0.1	NR		
6	0.0338	0.0073	-0.09	-0.05
7	NT	NT		
8	<1	NR		
9	<0.1	0.1		
10	0.03	0.03	-0.64	-0.14
11	<5	NR		
12	<1	NR		
13	NR	NR		
14**	0	0.3	-5.00	-0.11
15	NT	NT		
16	NT	NT		
17	NR	NR		
18	NT	NT		
19	<1	0.4		
20	0.03	0.03	-0.64	-0.14
21	0.023	0.01	-1.66	-0.87
22	0.04	0.01	0.81	0.43
23	0.0362	0.007	0.26	0.16
24	<0.1	NR		
25	0.0475	0.0095	1.90	1.03
26*	0.07	0.01	5.17	2.71
28	NT	NT		
29	NT	NT		
30	<0.1	NR		

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	0.0344	0.0085
Homogeneity Value	0.0353	0.0042
Robust Average	0.043	0.017
Median	0.0362	0.0077
Mean	0.0567	
N	9	
Max	0.2	
Min	0.023	
Robust SD	0.020	
Robust CV	47%	

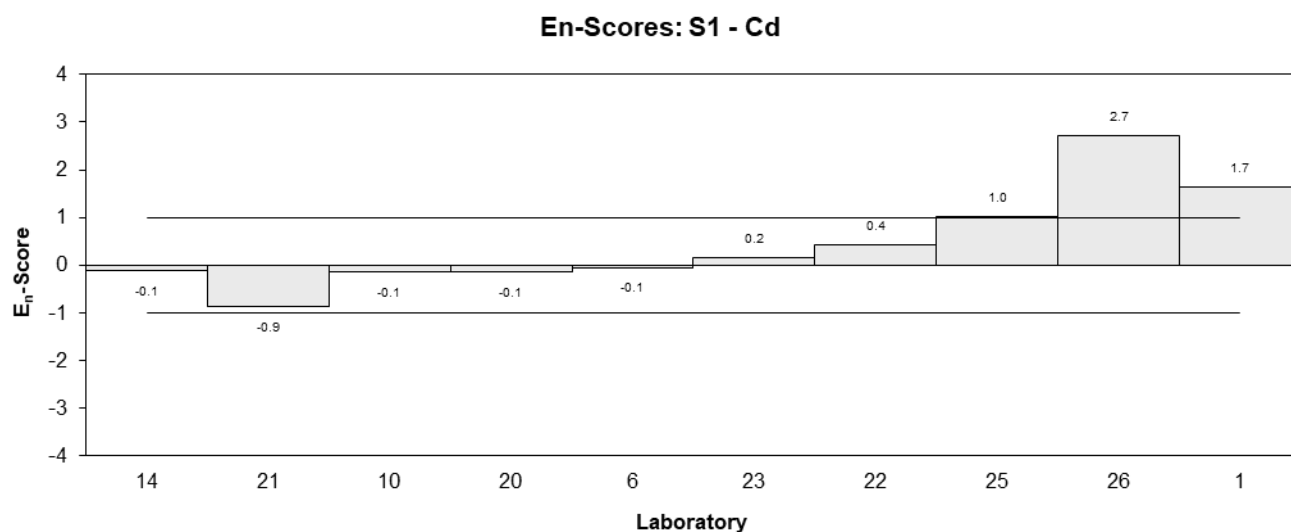
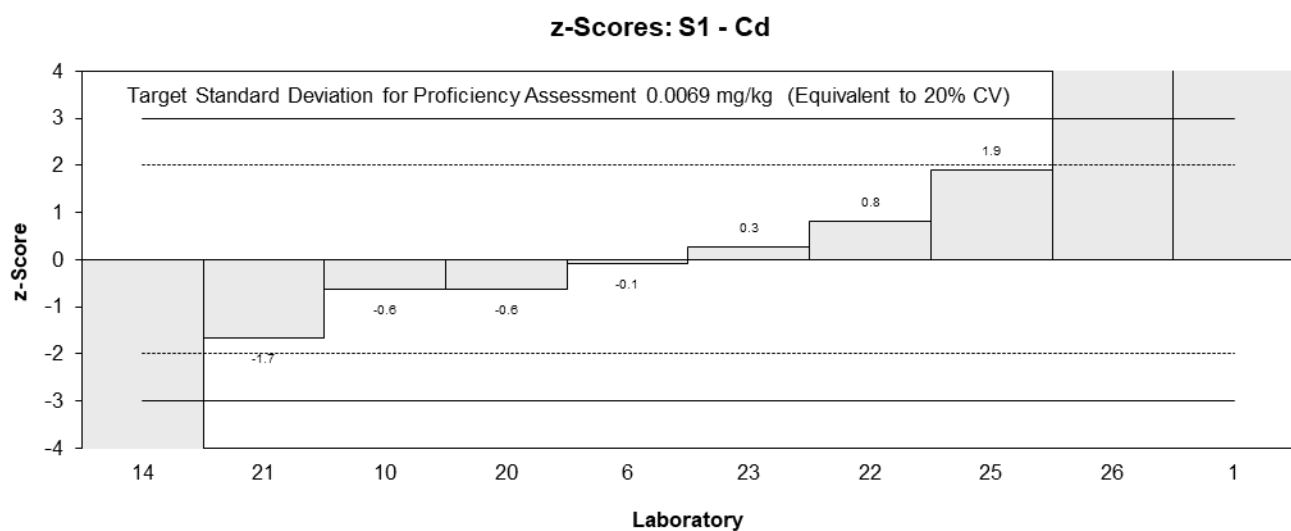
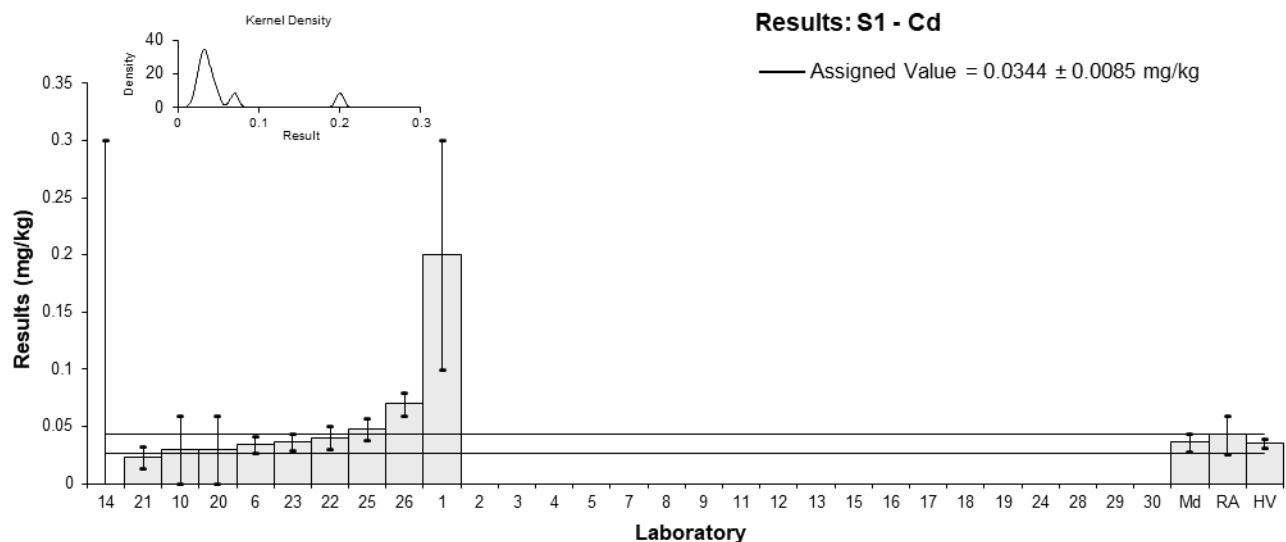


Figure 6

Table 19

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	1.2	0.30	0.50	0.34
2	0.932	0.097	-0.72	-1.08
3	NT	NT		
4	1	1	-0.41	-0.09
5	1.25	0.29	0.73	0.52
6	1.02	0.30	-0.32	-0.22
7	NT	NT		
8	<2	NR		
9	1.1	0.3	0.05	0.03
10	1	0.6	-0.41	-0.15
11	<5	NR		
12	1.5	0.3	1.88	1.28
13	NR	NR		
14	1.01	0.303	-0.37	-0.25
15	NT	NT		
16	NT	NT		
17	NR	NR		
18	NT	NT		
19	<2	1		
20	1.3	1	0.96	0.21
21	<4	NR		
22	1.00	0.42	-0.41	-0.21
23	1.02	0.20	-0.32	-0.31
24	0.8	0.13	-1.33	-1.70
25	1.3	0.26	0.96	0.74
26	NT	NT		
28	NT	NT		
29	NT	NT		
30	1.05	0.11	-0.18	-0.26

Statistics

Assigned Value	1.09	0.11
Homogeneity Value	1.02	0.12
Robust Average	1.09	0.11
Median	1.02	0.08
Mean	1.10	
N	15	
Max	1.5	
Min	0.8	
Robust SD	0.17	
Robust CV	16%	

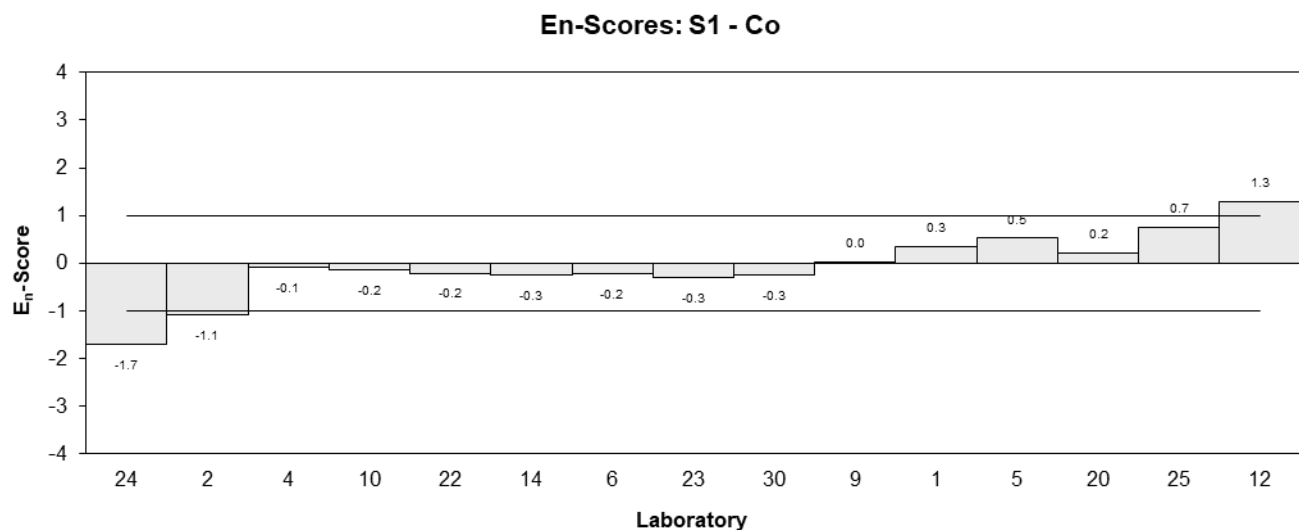
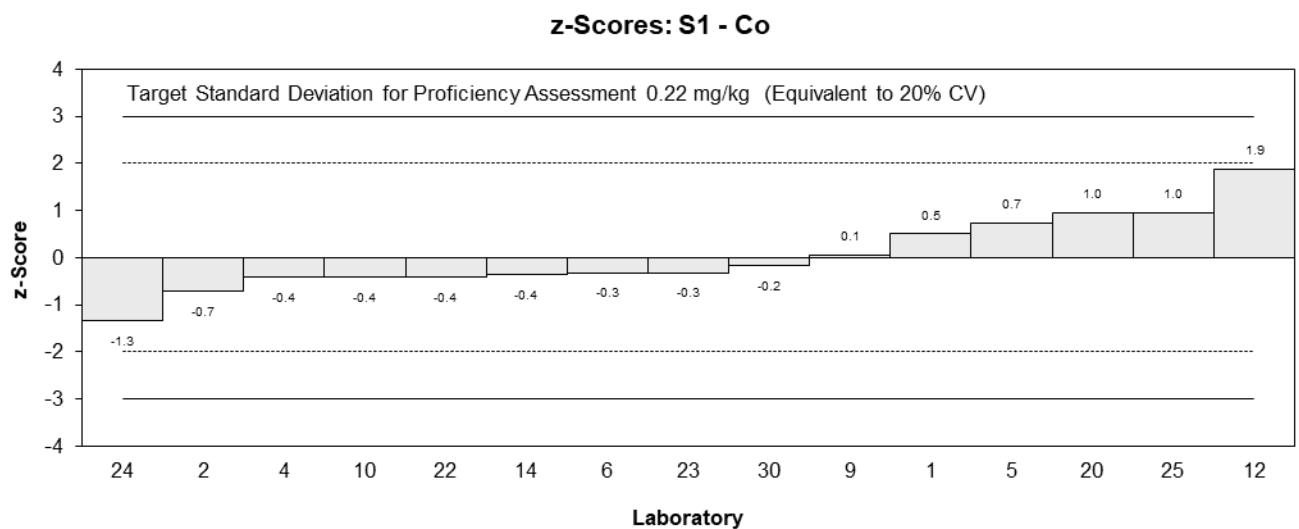
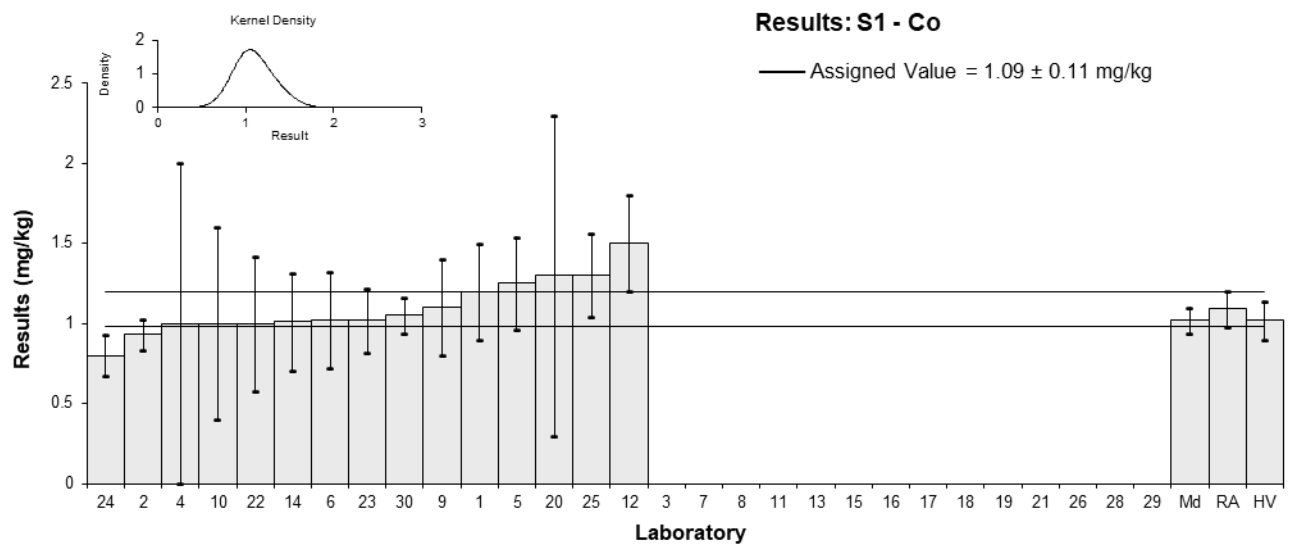


Figure 7

Table 20

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	4.5	1.0	0.61	0.45
2	3.248	0.433	-0.95	-1.23
3	NT	NT		
4	5	1	1.23	0.91
5	4.69	1.08	0.85	0.58
6	3.38	0.43	-0.79	-1.02
7	NT	NT		
8	4	1	-0.01	-0.01
9	4.1	0.4	0.11	0.15
10	4	1	-0.01	-0.01
11	<5	NR		
12	5.4	1.1	1.73	1.17
13	NR	NR		
14	3.77	1.131	-0.30	-0.20
15	NT	NT		
16	NT	NT		
17	NR	NR		
18	NT	NT		
19	3	1	-1.26	-0.92
20	4.5	1	0.61	0.45
21	NR	NR		
22	3.41	1.10	-0.75	-0.51
23	3.87	0.77	-0.17	-0.16
24	3.4	0.95	-0.76	-0.58
25	5	1	1.23	0.91
26	3.46	1.1	-0.69	-0.46
28	NT	NT		
29	NT	NT		
30	3.66	0.37	-0.44	-0.61

Statistics

Assigned Value	4.01	0.44
Homogeneity Value	3.90	0.47
Robust Average	4.01	0.44
Median	3.94	0.48
Mean	4.02	
N	18	
Max	5.4	
Min	3	
Robust SD	0.75	
Robust CV	19%	

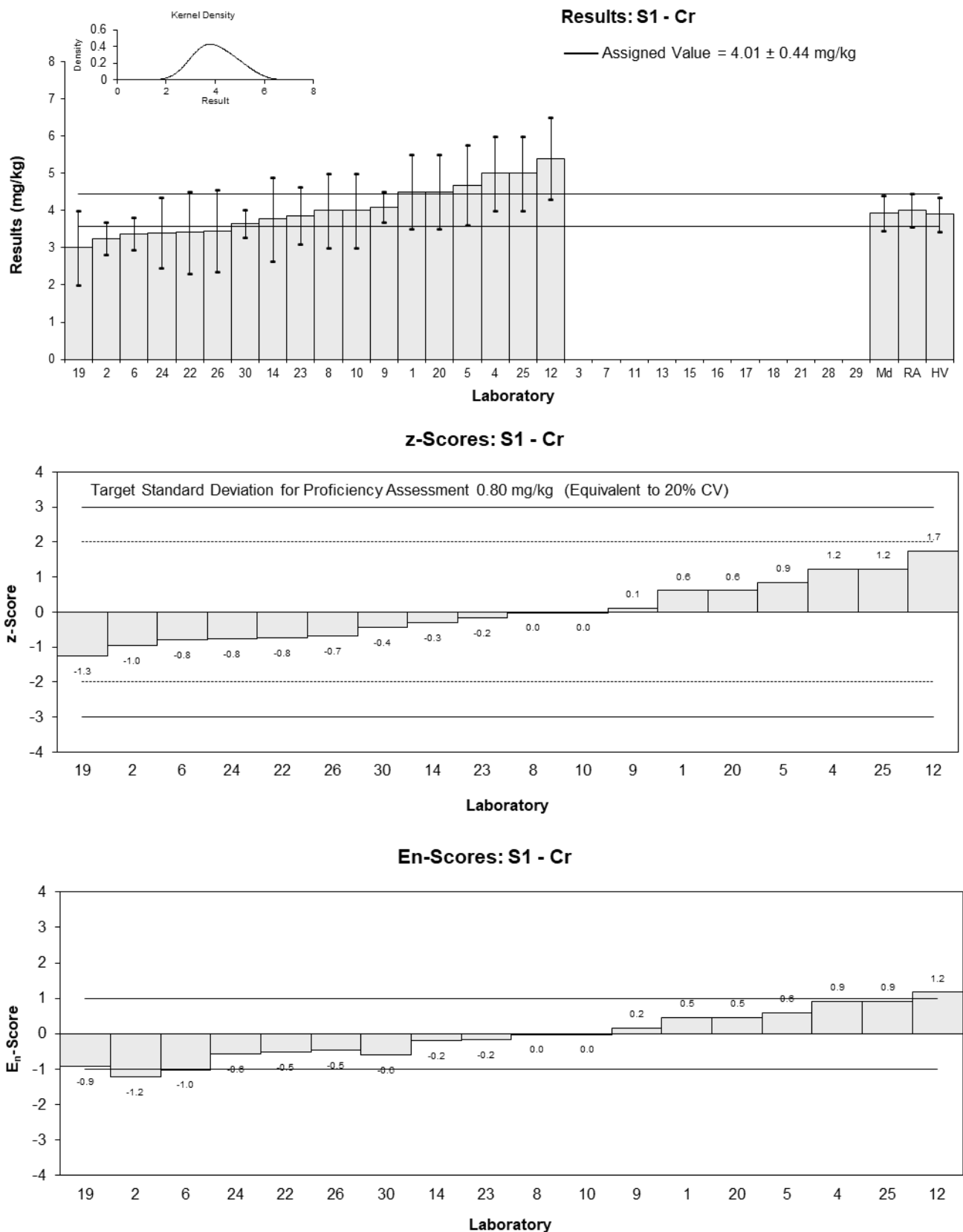


Figure 8

Table 21

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	14	2.8	0.57	0.37
2	10.899	1.135	-1.03	-1.32
3	NT	NT		
4	15	4	1.09	0.51
5	15.0	2.3	1.09	0.84
6	12.3	2.2	-0.31	-0.25
7	NT	NT		
8	13	2	0.05	0.04
9	12.6	1.3	-0.16	-0.18
10	13	3	0.05	0.03
11	11.5	1.81	-0.72	-0.68
12	16	3.2	1.60	0.92
13	NR	NR		
14	12.35	3.705	-0.28	-0.14
15	NT	NT		
16	NT	NT		
17	14.256	1.5	0.70	0.75
18	NT	NT		
19	12	3	-0.47	-0.28
20	14	3	0.57	0.35
21	NR	NR		
22	11.0	1.4	-0.98	-1.10
23	12.2	2.4	-0.36	-0.27
24	9.7	1.63	-1.65	-1.67
25	15	3	1.09	0.66
26	10.91	2.8	-1.03	-0.67
28	NT	NT		
29	NT	NT		
30	12.6	1.26	-0.16	-0.19

Statistics

Assigned Value	12.9	1.0
Homogeneity Value	12.5	1.5
Robust Average	12.9	1.0
Median	12.6	1.2
Mean	12.9	
N	20	
Max	16	
Min	9.7	
Robust SD	1.8	
Robust CV	14%	

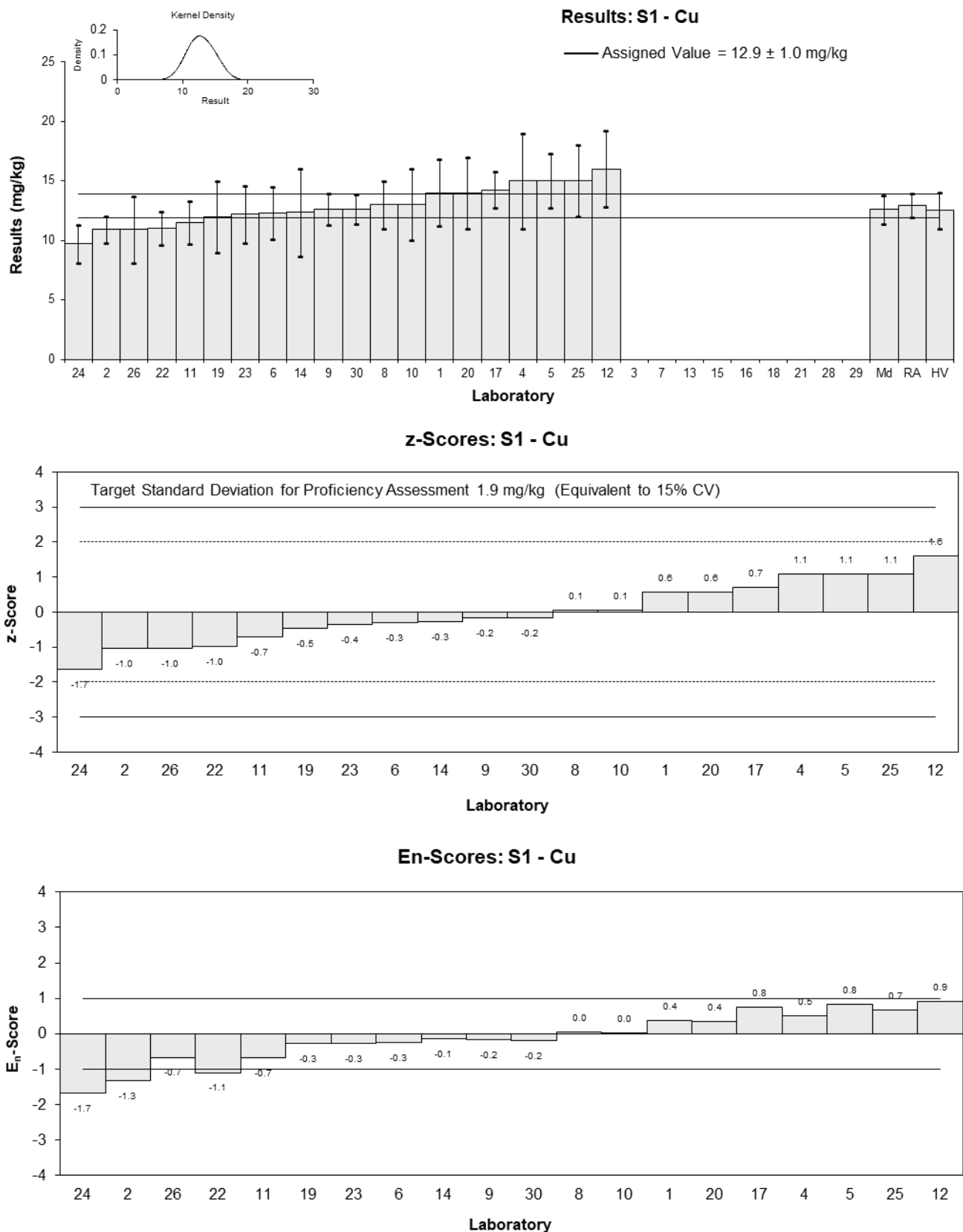


Figure 9

Table 22

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1*	1.0	0.35	3.50	1.15
2	0.564	0.133	-0.20	-0.16
3	NT	NT		
4	<1	1		
5	<1	NR		
6	0.56	0.28	-0.24	-0.10
7	NT	NT		
8*	0.3	0.1	-2.45	-2.34
9	0.51	0.2	-0.66	-0.37
10	0.6	0.6	0.10	0.02
11*	0.921	NR	2.83	4.63
12	NT	NT		
13	NR	NR		
14	0.55	0.165	-0.32	-0.21
15	NT	NT		
16	NT	NT		
17	<1	NR		
18	NT	NT		
19	0.5	0.072	-0.75	-0.86
20	<1	NR		
21	NR	NR		
22	0.70	0.14	0.95	0.71
23	0.805	0.161	1.85	1.23
24*	0.3	NR	-2.45	-4.00
25	0.59	0.118	0.02	0.01
26	NT	NT		
28	NT	NT		
29	NT	NT		
30	<1	NR		

* Outlier, see Section 4.2

Statistics

Assigned Value	0.588	0.072
Homogeneity Value	0.586	0.070
Robust Average	0.60	0.16
Median	0.564	0.066
Mean	0.608	
N	13	
Max	1	
Min	0.3	
Robust SD	0.23	
Robust CV	37%	

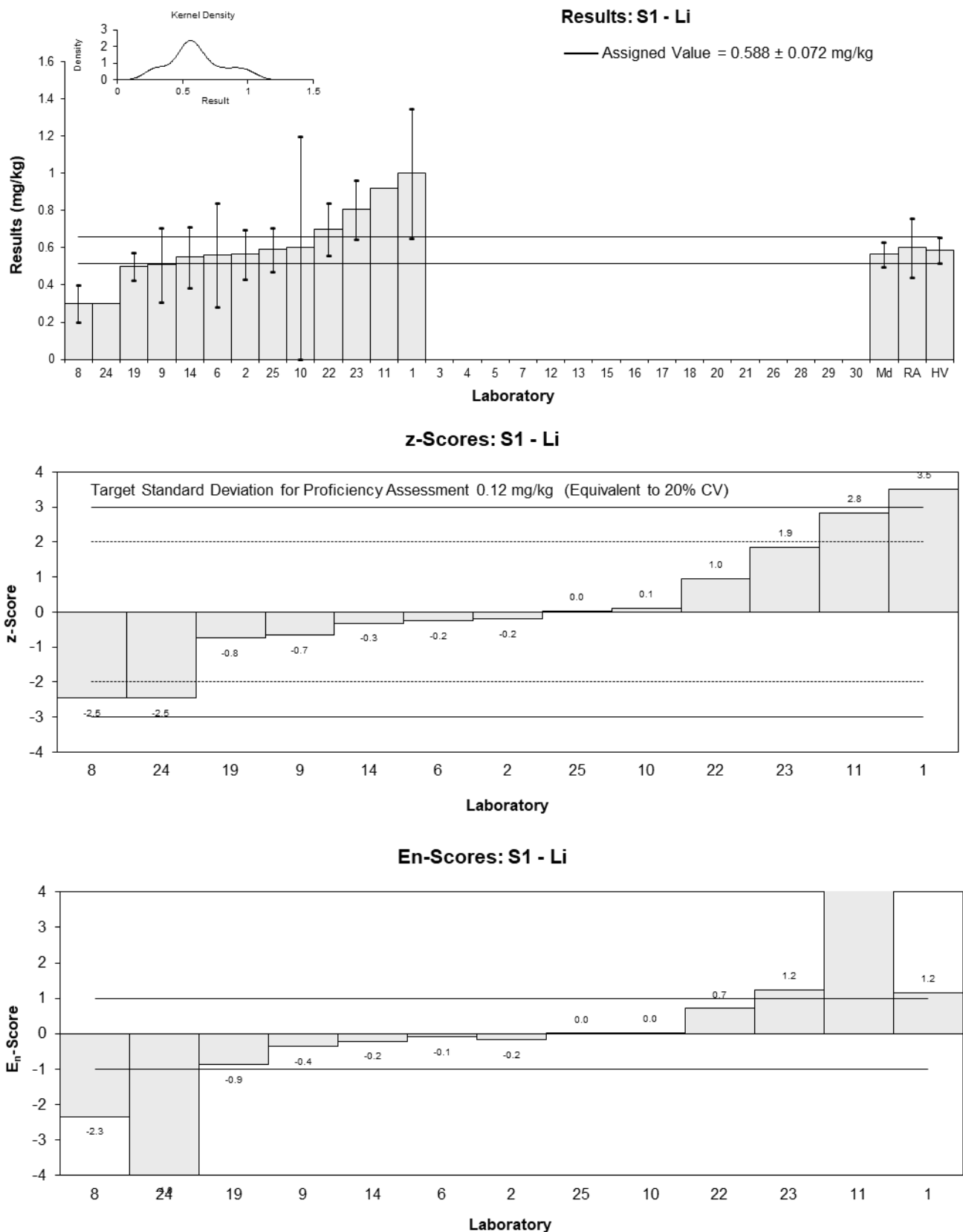


Figure 10

Table 23

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	21	4.8	0.61	0.44
2	14.494	1.424	-1.12	-1.66
3	NT	NT		
4	22	4	0.88	0.73
5	21.8	4.1	0.83	0.67
6	18.3	2.0	-0.11	-0.14
7	NT	NT		
8	16	2	-0.72	-0.93
9	17.6	1.8	-0.29	-0.40
10	24	5	1.42	0.98
11	17.1	3.20	-0.43	-0.42
12	24	4.8	1.42	1.01
13	NR	NR		
14	16.51	4.953	-0.59	-0.41
15	NT	NT		
16	NT	NT		
17	NR	NR		
18	NT	NT		
19	17	4	-0.45	-0.38
20	20	5	0.35	0.24
21	NR	NR		
22	17.1	3.4	-0.43	-0.40
23	16.7	3.3	-0.53	-0.51
24	13	2.19	-1.52	-1.88
25	22.25	4.45	0.95	0.72
26	NT	NT		
28	NT	NT		
29	NT	NT		
30	16.9	1.7	-0.48	-0.67

Statistics

Assigned Value	18.7	2.1
Homogeneity Value	18.6	2.2
Robust Average	18.7	2.1
Median	17.4	1.7
Mean	18.7	
N	18	
Max	24	
Min	13	
Robust SD	3.6	
Robust CV	19%	

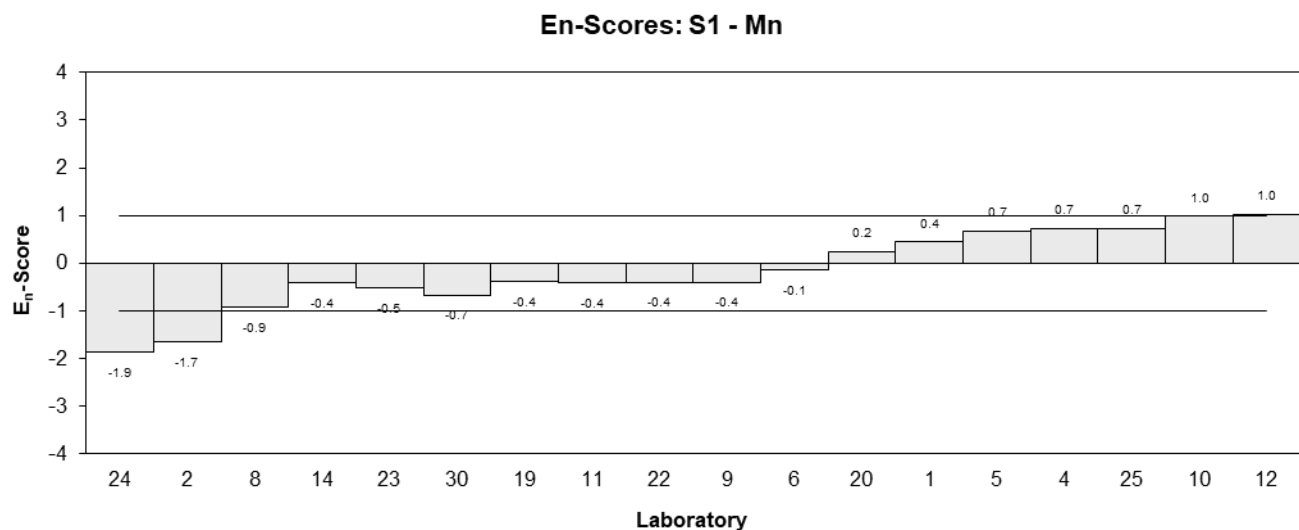
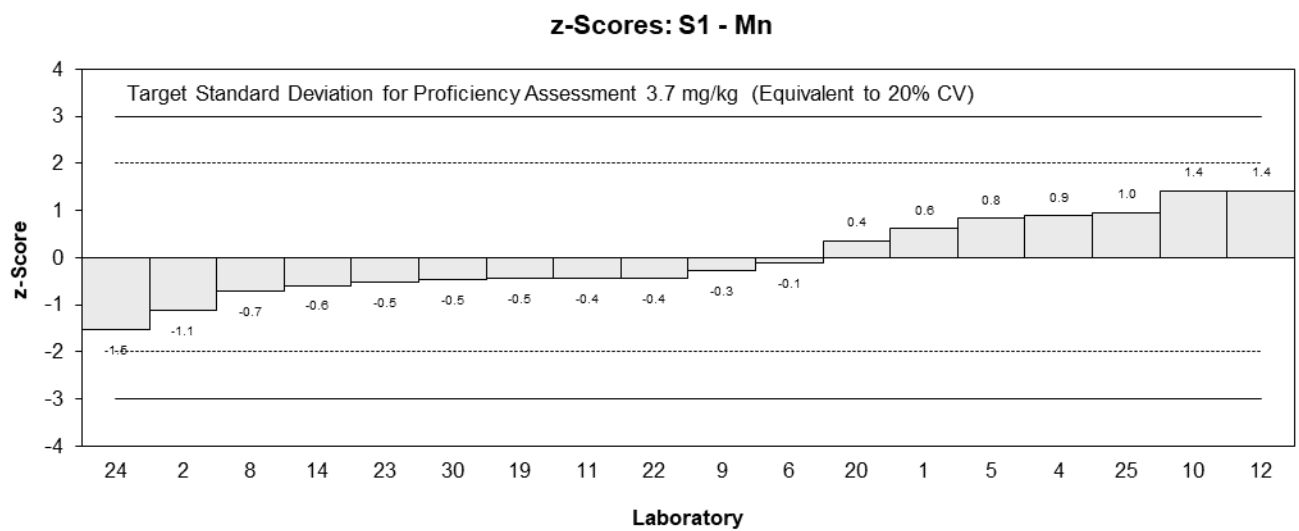
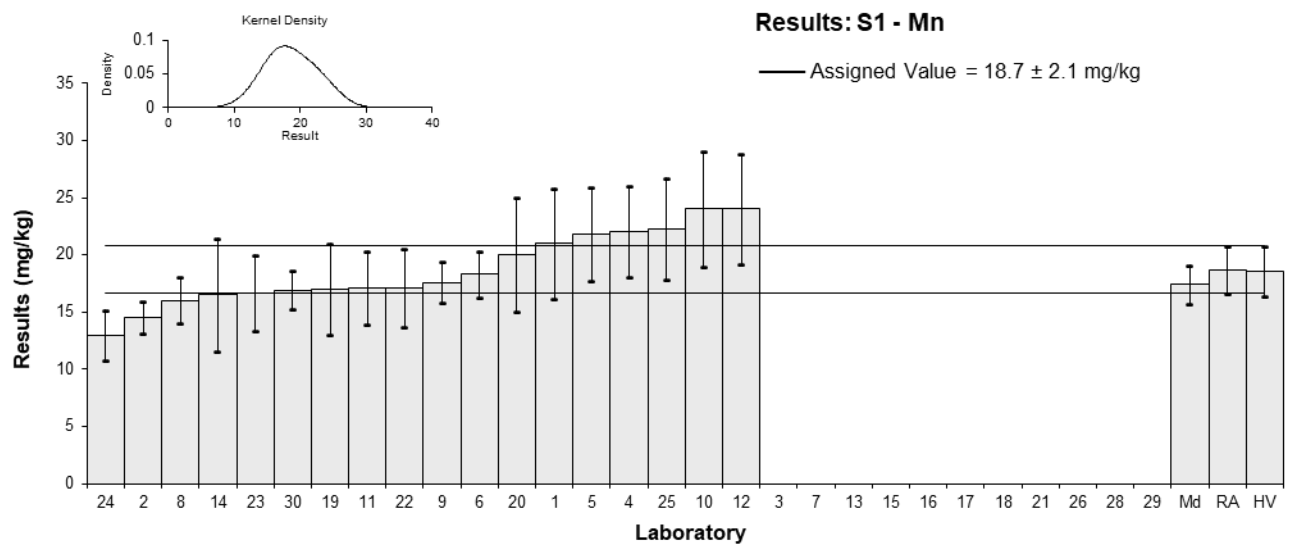


Figure 11

Table 24

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	34	5.5	0.57	0.44
2	<50	NR		
3	NT	NT		
4	40	10	1.56	0.83
5	24.2	8.0	-1.03	-0.64
6	<40	27		
7	NT	NT		
8	<50	NR		
9	<50	5		
10	30	10	-0.08	-0.04
11	42.6	5.20	1.98	1.57
12	<200	NR		
13	NR	NR		
14	23.17	6.951	-1.20	-0.82
15	NT	NT		
16	NT	NT		
17	NR	NR		
18	NT	NT		
19	<50	40		
20	30	10	-0.08	-0.04
21	<40	NR		
22	30.9	6.2	0.07	0.05
23	24.2	4.8	-1.03	-0.85
24	<50	NR		
25	<130	NR		
26	NT	NT		
28	NT	NT		
29	NT	NT		
30	27.7	2.8	-0.46	-0.44

Statistics

Assigned Value	30.5	5.7
Robust Average	30.5	5.7
Median	30.0	5.7
Mean	30.7	
N	10	
Max	42.6	
Min	23.17	
Robust SD	7.2	
Robust CV	24%	

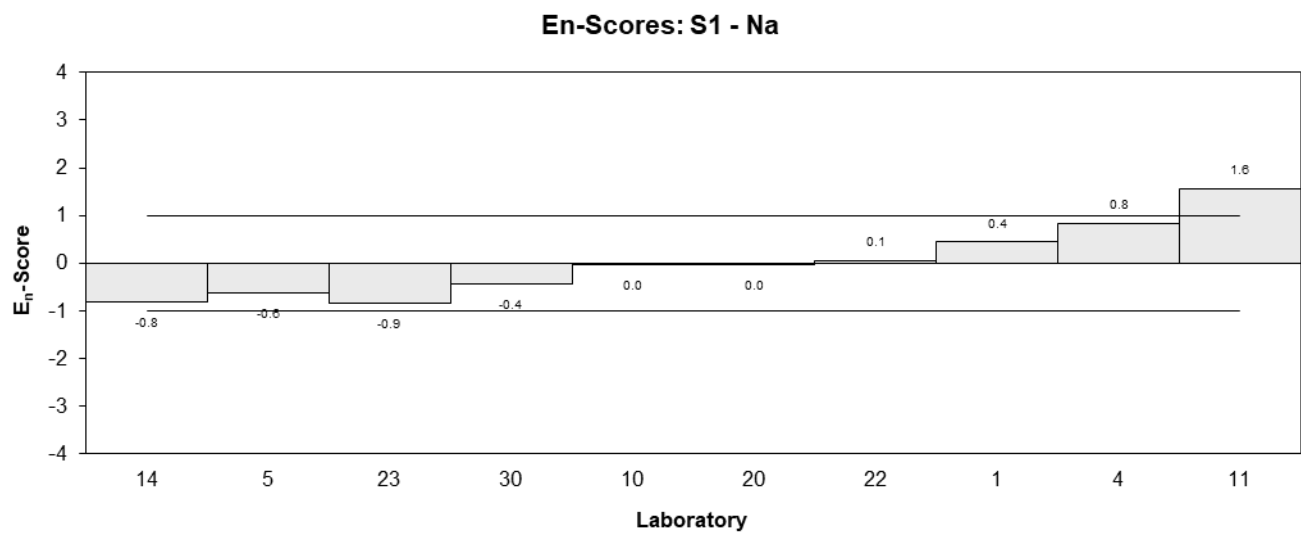
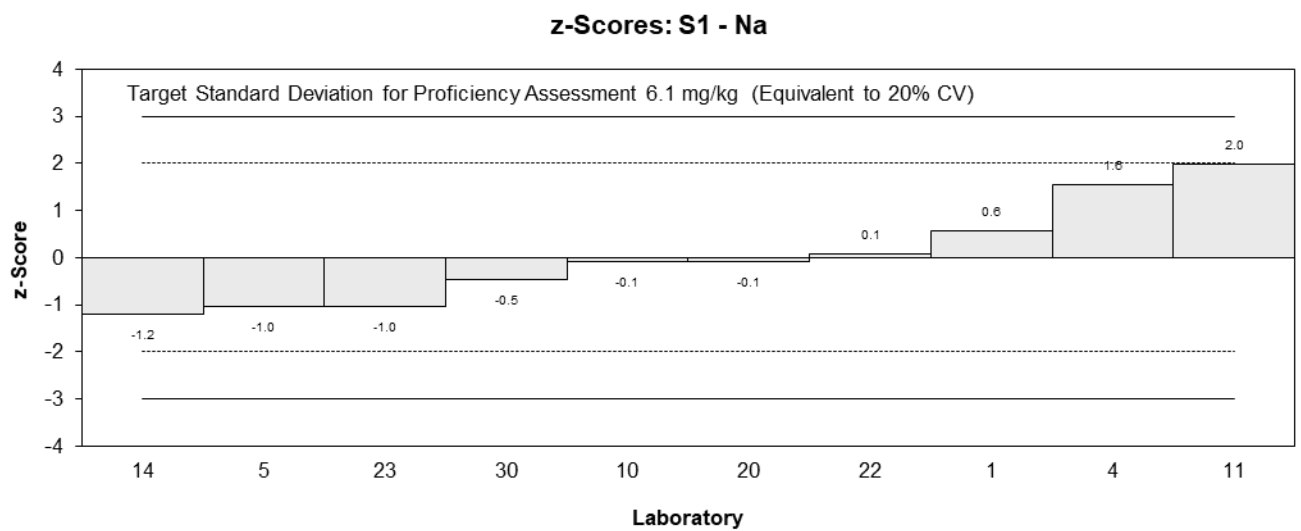
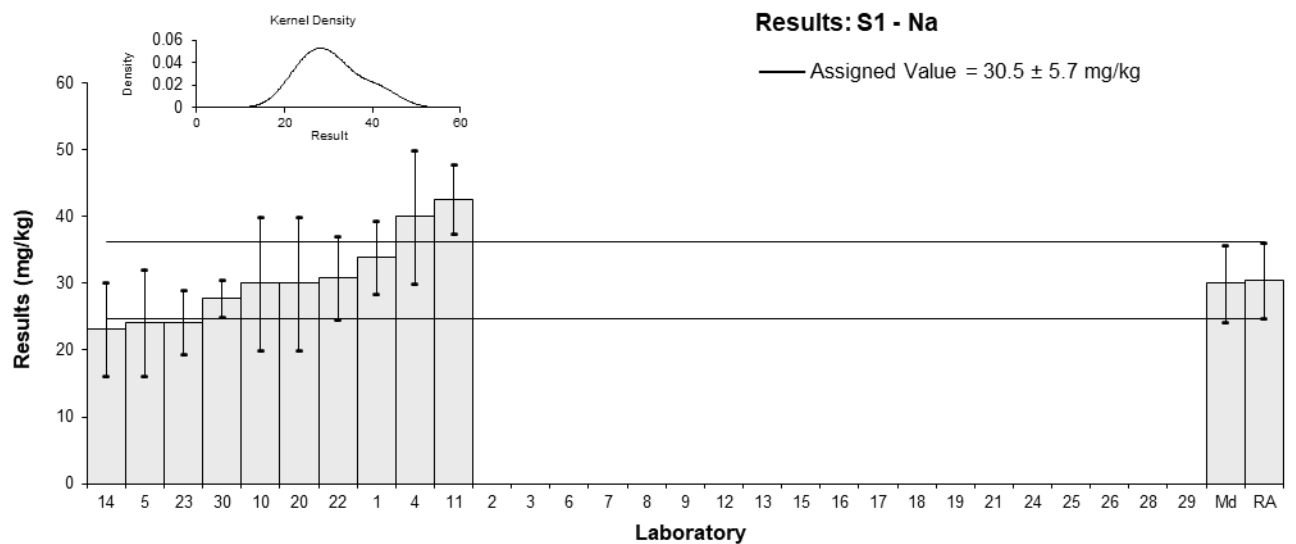


Figure 12

Table 25

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	2.3	0.46	1.85	1.13
2	1.368	0.266	-0.93	-0.78
3	NT	NT		
4	2	1	0.95	0.31
5	<2	NR		
6	1.26	0.19	-1.25	-1.18
7	NT	NT		
8	<2	NR		
9	1.5	0.2	-0.54	-0.50
10	2	1	0.95	0.31
11	<5	NR		
12	2.4	0.7	2.14	0.95
13	NR	NR		
14	1.24	0.372	-1.31	-0.92
15	NT	NT		
16	NT	NT		
17	2.321	0.2	1.91	1.78
18	NT	NT		
19	<2	0.5		
20	1.7	1	0.06	0.02
21	NR	NR		
22	1.54	0.31	-0.42	-0.32
23	1.58	0.32	-0.30	-0.23
24	1.2	0.24	-1.43	-1.25
25	1.9	0.38	0.65	0.45
26	1.25	0.4	-1.28	-0.86
28	NT	NT		
29	NT	NT		
30	1.34	0.13	-1.01	-1.04

Statistics

Assigned Value	1.68	0.30
Homogeneity Value	1.50	0.18
Robust Average	1.68	0.30
Median	1.56	0.29
Mean	1.68	
N	16	
Max	2.4	
Min	1.2	
Robust SD	0.47	
Robust CV	28%	

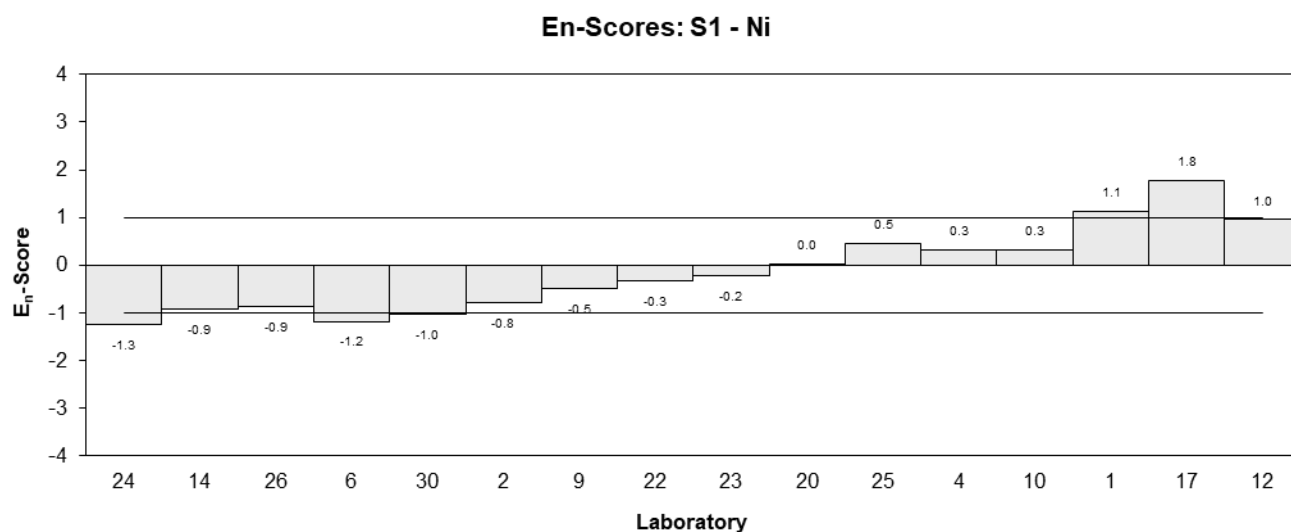
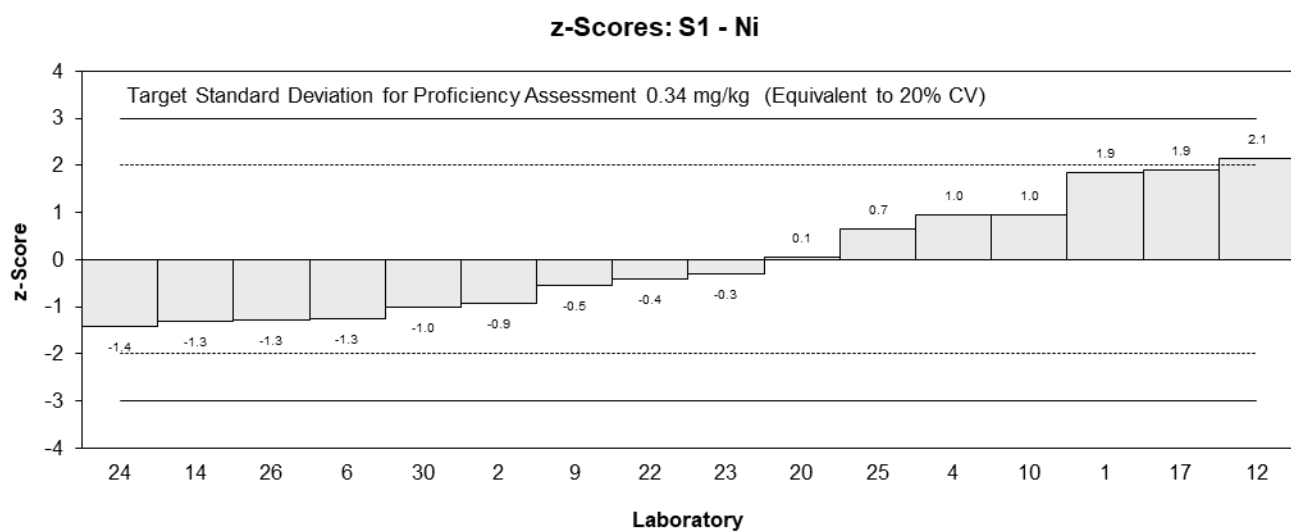
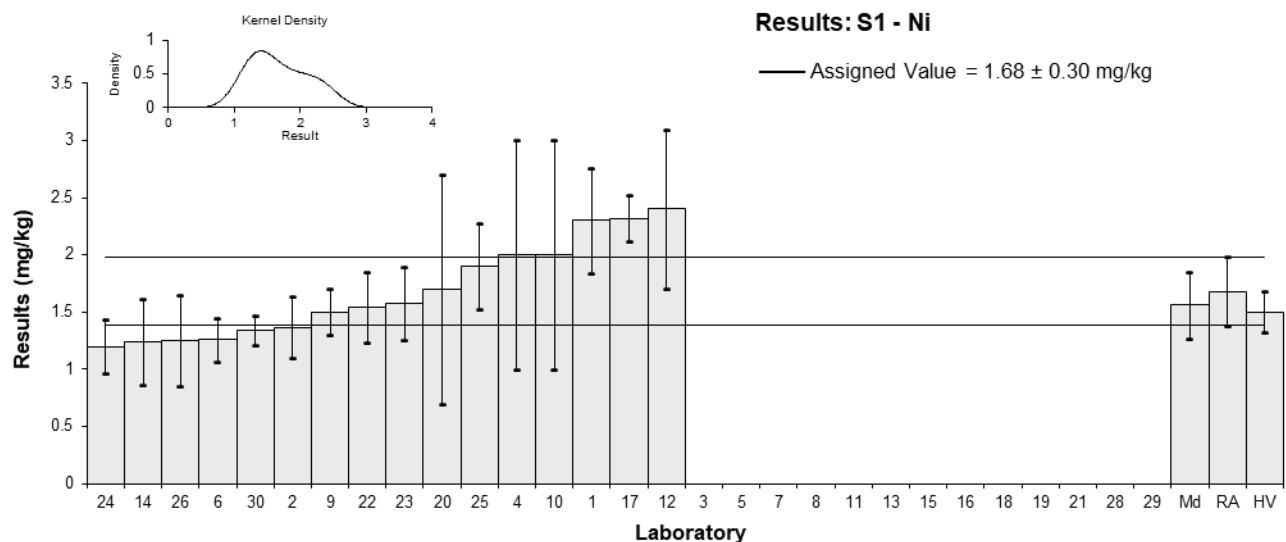


Figure 13

Table 26

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	42	9.3	0.51	0.31
2	33.391	5.388	-0.96	-0.93
3	NT	NT		
4	45	9	1.03	0.64
5	41.0	5.5	0.34	0.33
6	36.1	5.5	-0.50	-0.47
7	NT	NT		
8	39	5	0.00	0.00
9	36.4	3.6	-0.44	-0.58
10	40	8	0.17	0.12
11	38.1	4.50	-0.15	-0.17
12	46	9.2	1.20	0.73
13	NR	NR		
14	41.9	12.57	0.50	0.23
15	NT	NT		
16	NT	NT		
17	NR	NR		
18	NT	NT		
19	35	11	-0.68	-0.35
20	44	10	0.85	0.48
21	33.8	6.76	-0.89	-0.71
22	37.5	7.5	-0.26	-0.19
23	39.6	7.9	0.10	0.07
24	35	5.82	-0.68	-0.62
25	47.75	9.55	1.50	0.88
26	29.46	9.0	-1.63	-1.02
28	NT	NT		
29	NT	NT		
30	38.2	3.8	-0.14	-0.17

Statistics

Assigned Value	39.0	2.7
Homogeneity Value	36.0	4.3
Robust Average	39.0	2.7
Median	38.6	2.8
Mean	39.0	
N	20	
Max	47.75	
Min	29.46	
Robust SD	4.9	
Robust CV	12%	

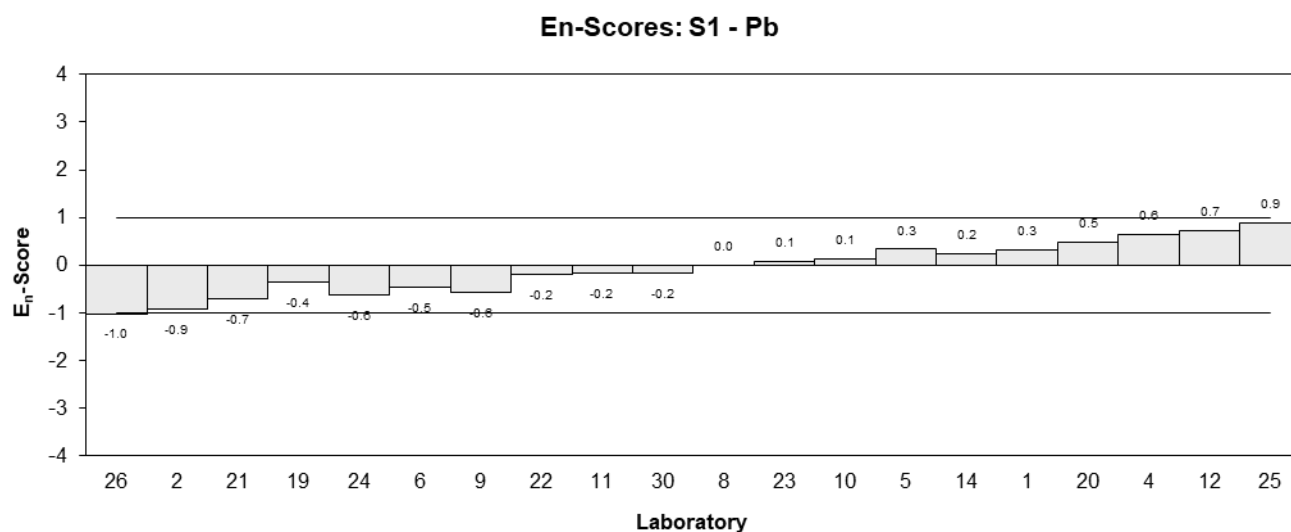
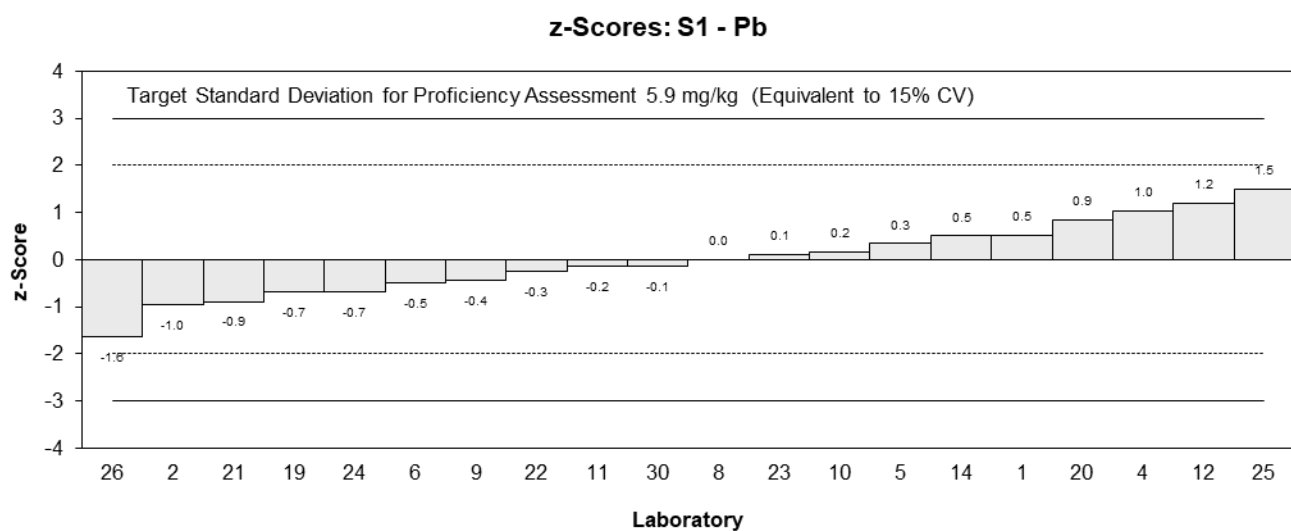
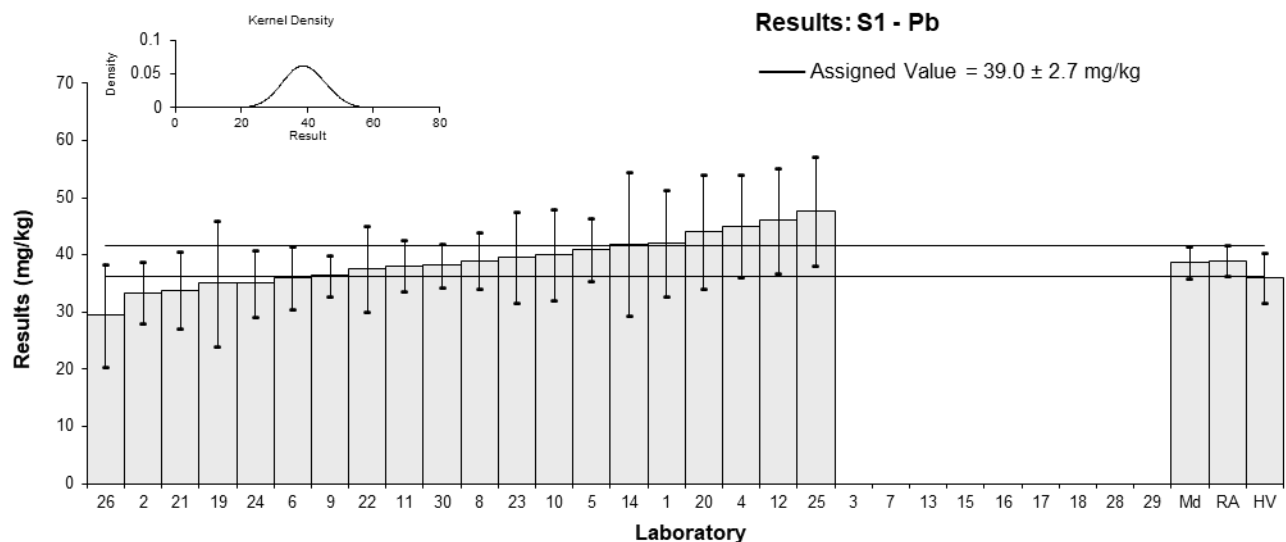


Figure 14

Table 27

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	1.720	NR	-0.63	-0.64
3	NT	NT		
4	2	1	0.08	0.03
5	NT	NT		
6	2.16	0.35	0.48	0.36
7	NT	NT		
8	1.3	0.2	-1.70	-1.53
9	NT	NT		
10	2	0.5	0.08	0.05
11	NR	NR		
12	NT	NT		
13	NR	NR		
14	NT	NT		
15	NT	NT		
16	NT	NT		
17	NT	NT		
18	NT	NT		
19	1.5	0.13	-1.19	-1.14
20	2.3	1	0.84	0.31
21	NR	NR		
22	NR	NR		
23	2.53	0.51	1.42	0.87
24	1.6	NR	-0.94	-0.95
25	2.595	0.519	1.59	0.96
26	NT	NT		
28	NT	NT		
29	NT	NT		
30	NT	NT		

Statistics

Assigned Value	1.97	0.39
Robust Average	1.97	0.39
Median	2.00	0.41
Mean	1.97	
N	10	
Max	2.595	
Min	1.3	
Robust SD	0.50	
Robust CV	25%	

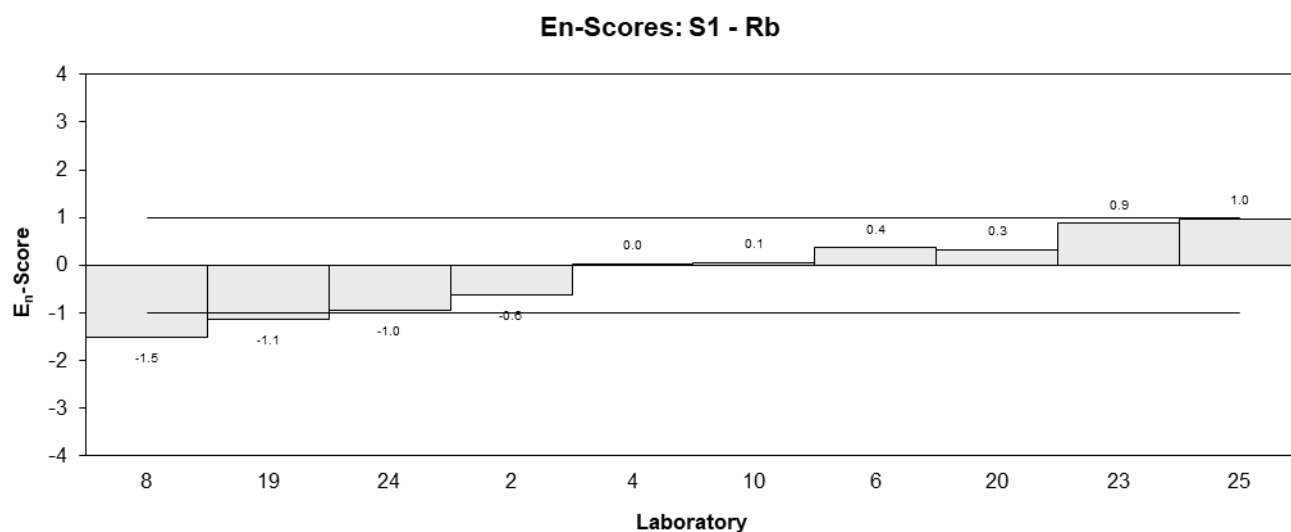
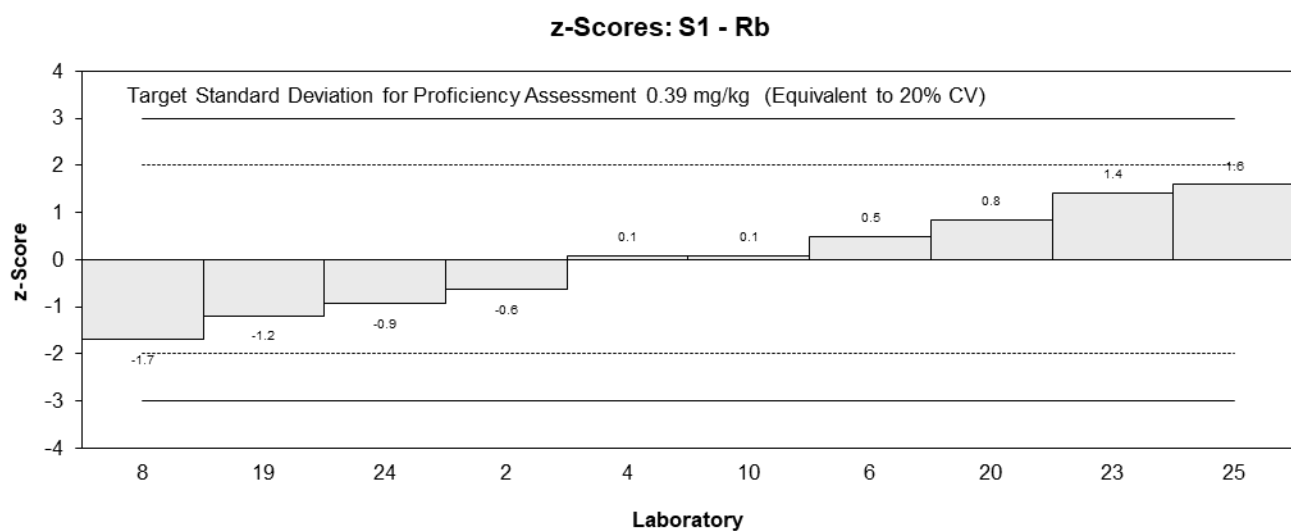
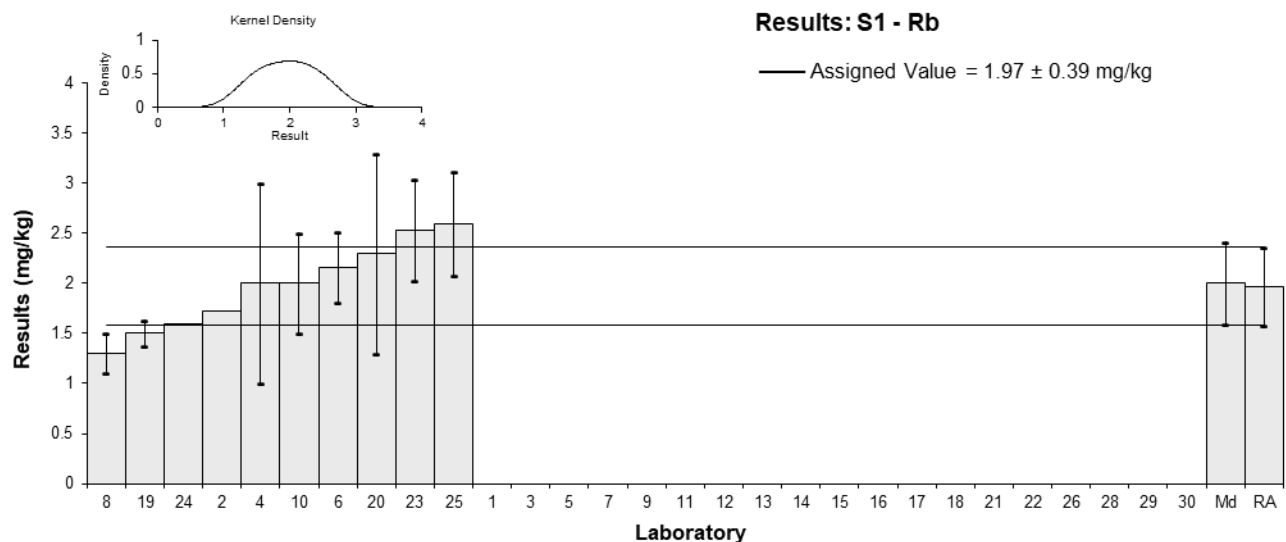


Figure 15

Table 28

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	9.2	2.4	0.38	0.20
2	7.927	1.669	-0.59	-0.44
3	NT	NT		
4	9	3	0.23	0.10
5	9.29	1.83	0.45	0.31
6	7.7	1.1	-0.77	-0.80
7	NT	NT		
8	9	1	0.23	0.26
9	8.2	0.8	-0.38	-0.51
10	9	3	0.23	0.10
11	7.55	0.793	-0.88	-1.17
12	9.7	0.8	0.77	1.01
13	NR	NR		
14	7.36	2.208	-1.03	-0.59
15	NT	NT		
16	NT	NT		
17	9.405	1	0.54	0.61
18	NT	NT		
19	8	3	-0.54	-0.23
20	12	3	2.53	1.08
21	NR	NR		
22	8.02	1.60	-0.52	-0.40
23	8.10	1.62	-0.46	-0.35
24	8	1.30	-0.54	-0.49
25	11	2.2	1.76	1.01
26	NT	NT		
28	NT	NT		
29	NT	NT		
30	9.45	0.95	0.57	0.67

Statistics

Assigned Value	8.70	0.58
Robust Average	8.70	0.58
Median	9.00	0.77
Mean	8.84	
N	19	
Max	12	
Min	7.36	
Robust SD	1.0	
Robust CV	12%	

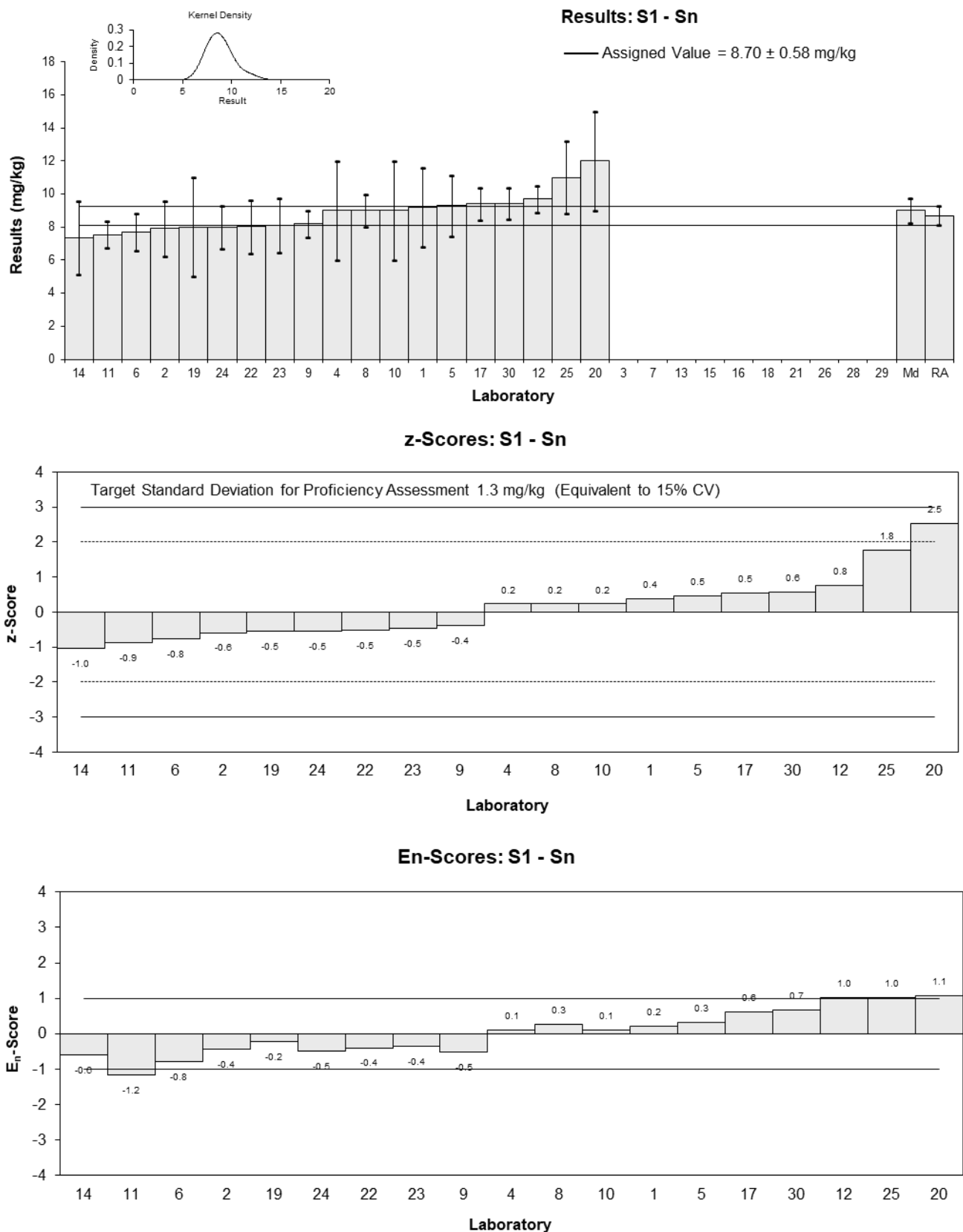


Figure 16

Table 29

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	2.9	0.71	2.00	1.05
2	1.588	0.247	-1.16	-1.13
3	NT	NT		
4	2	1	-0.17	-0.07
5	2.18	0.50	0.27	0.18
6	1.54	0.26	-1.28	-1.22
7	NT	NT		
8	3	1	2.25	0.88
9	1.8	0.2	-0.65	-0.67
10	2	1	-0.17	-0.07
11	<5	NR		
12*	4.6	0.4	6.11	4.76
13	NR	NR		
14	1.53	0.459	-1.30	-0.94
15	NT	NT		
16	NT	NT		
17	NR	NR		
18	NT	NT		
19	2	0.5	-0.17	-0.11
20	2.4	1	0.80	0.31
21	<4	NR		
22	2.08	0.42	0.02	0.02
23	2.81	0.56	1.79	1.12
24	1.6	0.3	-1.14	-1.02
25	2.3	0.46	0.56	0.40
26	NT	NT		
28	NT	NT		
29	NT	NT		
30	1.45	0.14	-1.50	-1.64

* Outlier, see Section 4.2

Statistics

Assigned Value	2.07	0.35
Homogeneity Value	2.30	0.28
Robust Average	2.13	0.37
Median	2.00	0.36
Mean	2.22	
N	17	
Max	4.6	
Min	1.45	
Robust SD	0.61	
Robust CV	29%	

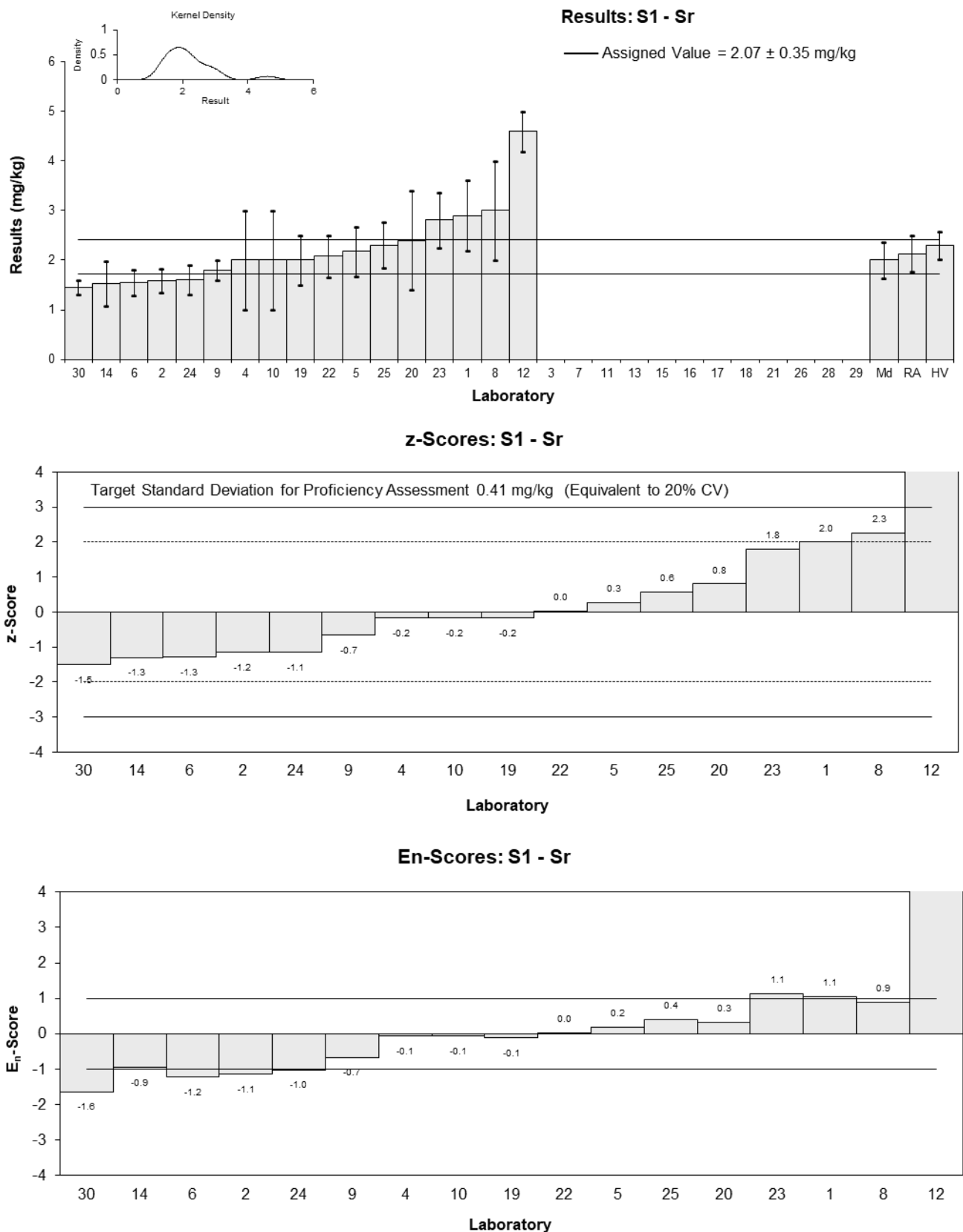


Figure 17

Table 30

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	5.8	1.5	0.40	0.26
2	4.280	0.829	-1.01	-1.05
3	NT	NT		
4	6	2	0.59	0.30
5	6.30	1.39	0.87	0.61
6	<10	6.7		
7	NT	NT		
8	<5	NR		
9	4.8	0.5	-0.53	-0.71
10	6	2	0.59	0.30
11	<5	NR		
12	6.5	0.8	1.05	1.11
13	NR	NR		
14	4.5	1.35	-0.81	-0.58
15	NT	NT		
16	NT	NT		
17	NR	NR		
18	NT	NT		
19	<5	1		
20	6.1	2	0.68	0.35
21	NR	NR		
22	5.01	1.00	-0.34	-0.30
23	5.13	1.03	-0.22	-0.20
24	4	0.75	-1.28	-1.40
25	6.05	1.21	0.63	0.50
26	NT	NT		
28	NT	NT		
29	NT	NT		
30	4.66	0.47	-0.66	-0.90

Statistics

Assigned Value	5.37	0.63
Homogeneity Value	5.14	0.62
Robust Average	5.37	0.63
Median	5.47	0.64
Mean	5.37	
N	14	
Max	6.5	
Min	4	
Robust SD	0.94	
Robust CV	18%	

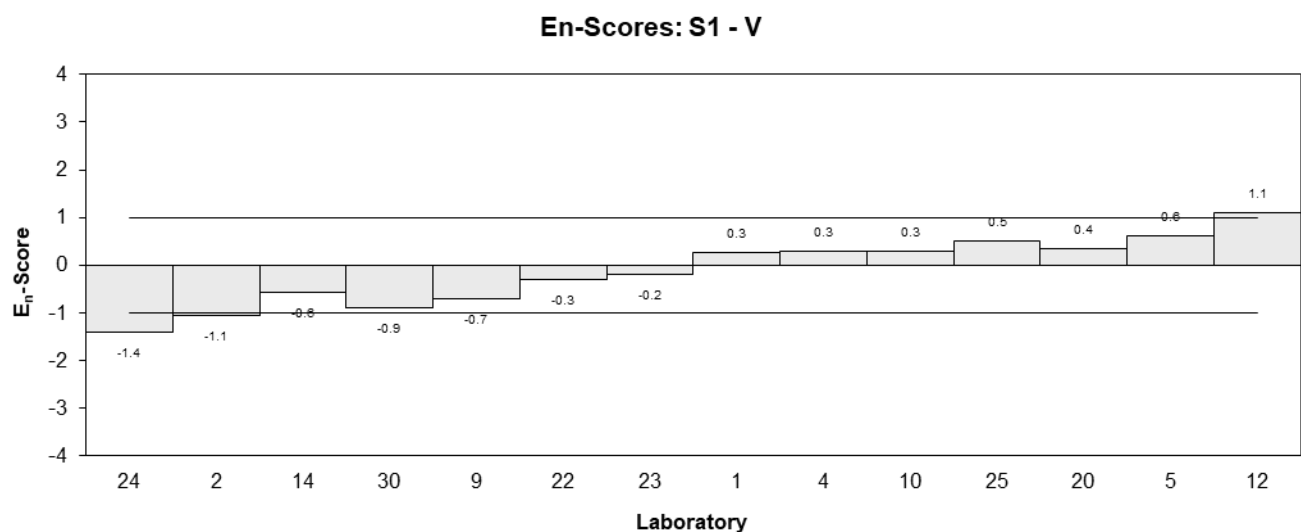
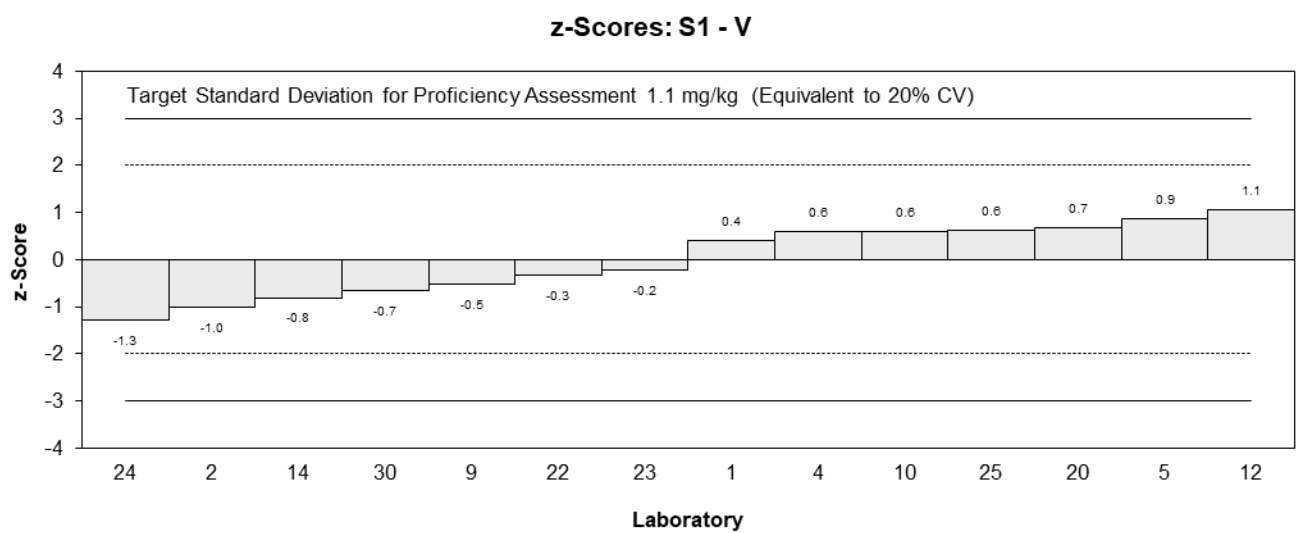
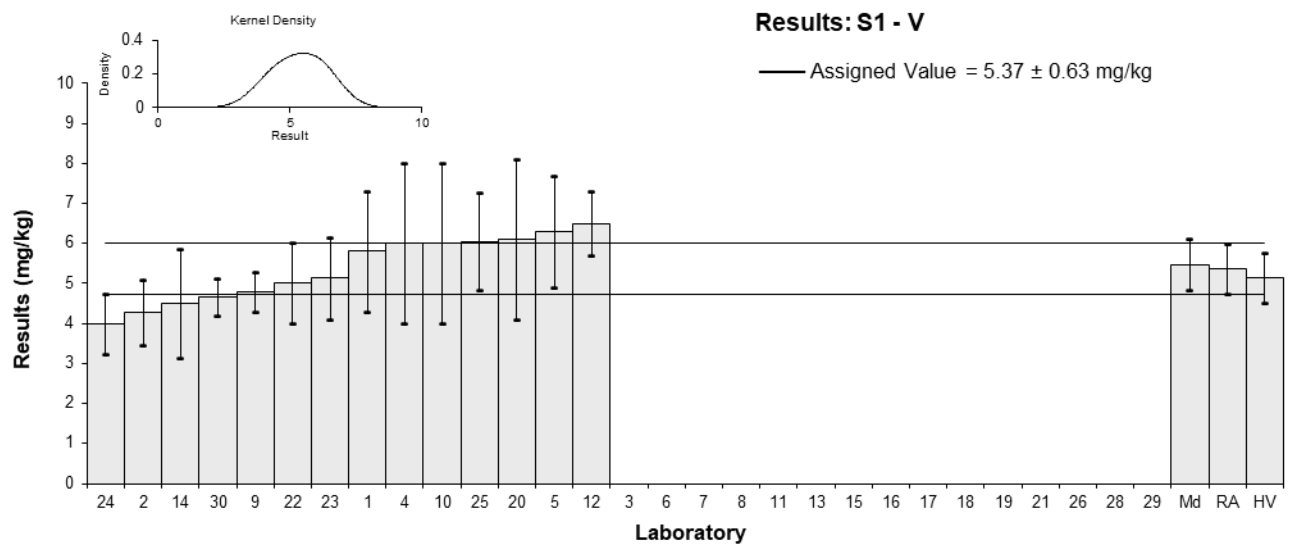


Figure 18

Table 31

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	15	3.2	0.68	0.52
2	11.122	1.368	-0.79	-1.10
3	NT	NT		
4	13	3	-0.08	-0.06
5	12.9	3.6	-0.11	-0.08
6	12.7	2.8	-0.19	-0.16
7	NT	NT		
8	11	1	-0.83	-1.34
9	14.6	1.5	0.53	0.71
10	13	3	-0.08	-0.06
11	12.5	2.80	-0.27	-0.23
12	18	3.6	1.82	1.25
13	NR	NR		
14	11.68	3.504	-0.58	-0.41
15	NT	NT		
16	NT	NT		
17	NR	NR		
18	NT	NT		
19	11	2.5	-0.83	-0.78
20	16	3	1.06	0.86
21	18.2	3.64	1.89	1.29
22	13.2	2.6	0.00	0.00
23	11.7	2.3	-0.57	-0.57
24	11.5	1.79	-0.64	-0.77
25	14	2.8	0.30	0.26
26	10.24	3	-1.12	-0.91
28	NT	NT		
29	NT	NT		
30	15.8	1.6	0.98	1.26

Statistics

Assigned Value	13.2	1.3
Homogeneity Value	11.7	1.4
Robust Average	13.2	1.3
Median	13.0	1.3
Mean	13.4	
N	20	
Max	18.2	
Min	10.24	
Robust SD	2.2	
Robust CV	17%	

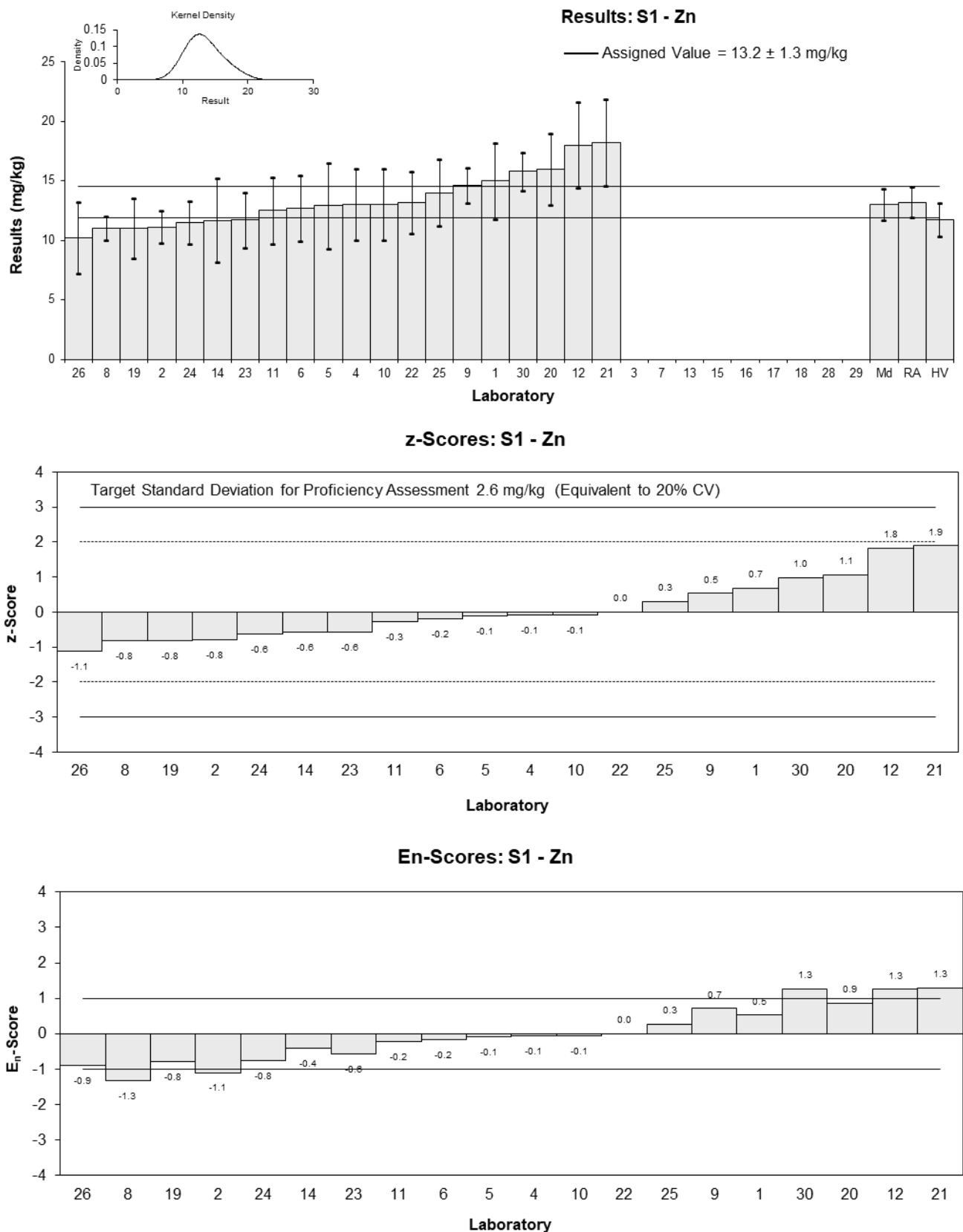


Figure 19

Table 32

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	6.247	1.063	0.12	0.10
3	NT	NT		
4	6	2	-0.15	-0.07
5	5.97	0.84	-0.18	-0.20
6	7.7	1.8	1.69	0.86
7	6.10	1.53	-0.04	-0.03
8	8	2	2.02	0.93
9	5.8	0.6	-0.37	-0.54
10	7	2	0.93	0.43
11	5.83	1.11	-0.34	-0.28
12	4.5	0.9	-1.78	-1.78
13	6.17	0.6	0.03	0.05
14	6.28	1.884	0.15	0.07
15	6.5	0.98	0.39	0.36
16	NT	NT		
17	6.348	0.6	0.23	0.33
18	6.25	1.04	0.12	0.10
19	6	1	-0.15	-0.14
20	6.1	2	-0.04	-0.02
21	4.5	1.35	-1.78	-1.20
22	6.00	1.2	-0.15	-0.12
23	5.64	1.13	-0.54	-0.44
24	5.9	1.33	-0.26	-0.18
25	6.35	1.27	0.23	0.16
26	NT	NT		
28	NT	NT		
29	6.34	1.27	0.22	0.16
30	6.35	0.64	0.23	0.31

Statistics

Assigned Value	6.14	0.19
Robust Average	6.14	0.19
Median	6.14	0.16
Mean	6.16	
N	24	
Max	8	
Min	4.5	
Robust SD	0.38	
Robust CV	6.2%	

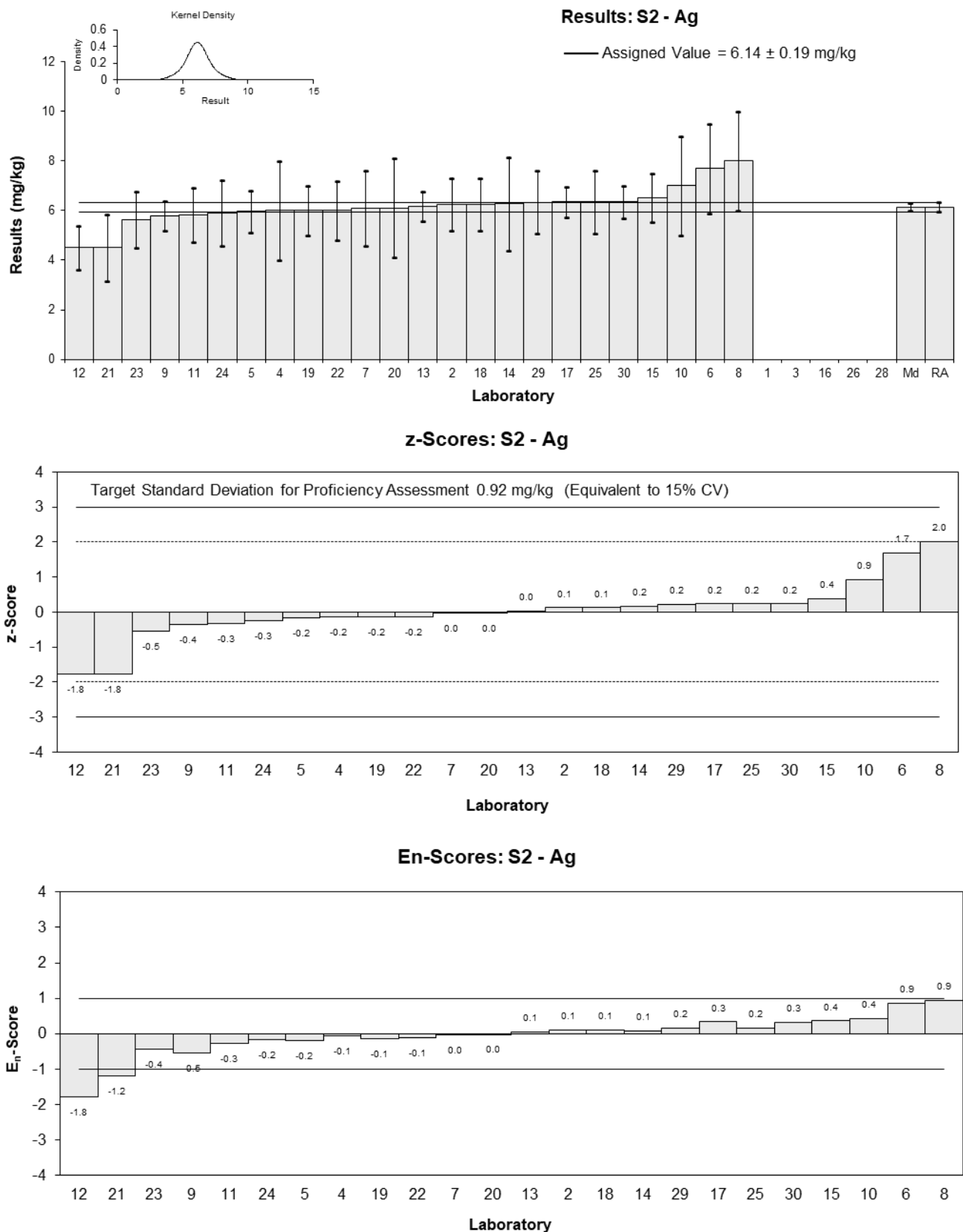


Figure 20

Table 33

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	3.514	0.442	-0.55	-0.64
3	NT	NT		
4	4	4	0.30	0.04
5	4.4	1.0	0.99	0.56
6	3.36	0.37	-0.82	-1.09
7	4.00	1	0.30	0.17
8*	7	1	5.52	3.10
9	3.7	0.4	-0.23	-0.28
10	3	3	-1.44	-0.28
11	<5	NR		
12	<5	NR		
13	3.70	0.5	-0.23	-0.24
14	4.16	1.248	0.57	0.26
15	3.9	0.6	0.12	0.11
16	NT	NT		
17	3.925	0.4	0.17	0.21
18	4.04	0.62	0.37	0.32
19	<5	1		
20	4.4	4	0.99	0.14
21	<10	NR		
22	2.90	0.58	-1.62	-1.50
23	3.49	0.70	-0.59	-0.46
24	3.8	0.62	-0.05	-0.05
25	3.95	0.79	0.21	0.15
26	3.59	1.21	-0.42	-0.20
28	NT	NT		
29	4.28	0.856	0.78	0.51
30	4.00	0.40	0.30	0.37

* Outlier, see Section 4.2

Statistics

Assigned Value	3.83	0.22
Robust Average	3.87	0.23
Median	3.93	0.19
Mean	3.96	
N	21	
Max	7	
Min	2.9	
Robust SD	0.42	
Robust CV	11%	

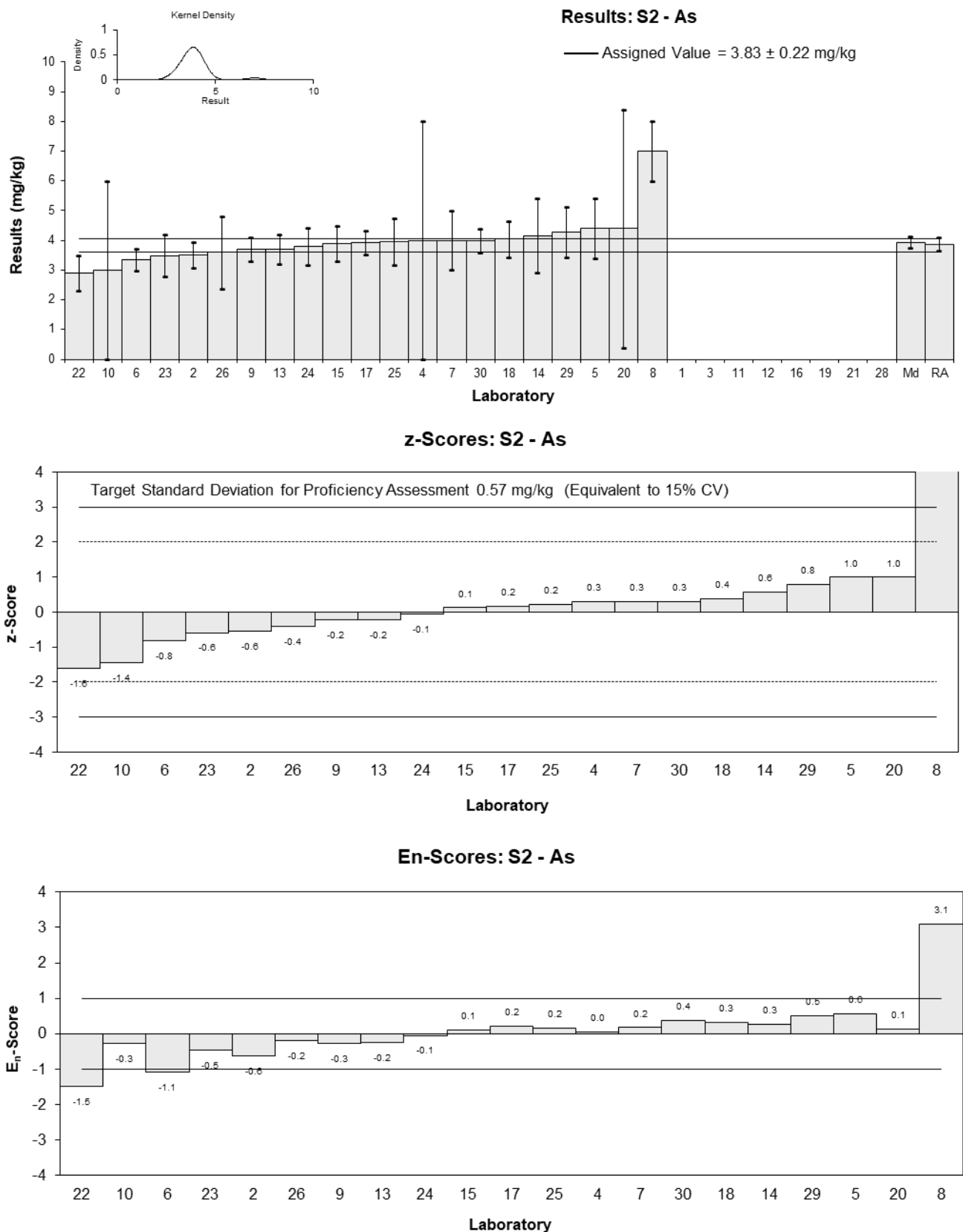


Figure 21

Table 34

Sample Details

Sample No.	S2
Matrix	Biosolid
Analyte	B
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	<50	NR		
3	NT	NT		
4	<10	10		
5	<10	NR		
6	2.8	1.4	-0.09	-0.03
7	<10	NR		
8	<50	NR		
9	<5	0.5		
10	3	3	0.26	0.05
11	<5	NR		
12	3.5	0.2	1.14	1.09
13	2.62	0.3	-0.40	-0.36
14**	0	5	-5.00	-0.57
15	< 5	NR		
16	NT	NT		
17	<3	NR		
18	< 10	NR		
19	<50	10		
20	<10	NR		
21	2.4	0.72	-0.79	-0.49
22	2.17	0.43	-1.19	-0.96
23	3.97	0.79	1.96	1.16
24	<5	NR		
25	2.5	0.5	-0.61	-0.47
26	NT	NT		
28	NT	NT		
29	<10	<2		
30	<5	NR		

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	2.85	0.56
Robust Average	2.85	0.56
Median	2.71	0.39
Mean	2.87	
N	8	
Max	3.97	
Min	2.17	
Robust SD	0.63	
Robust CV	22%	

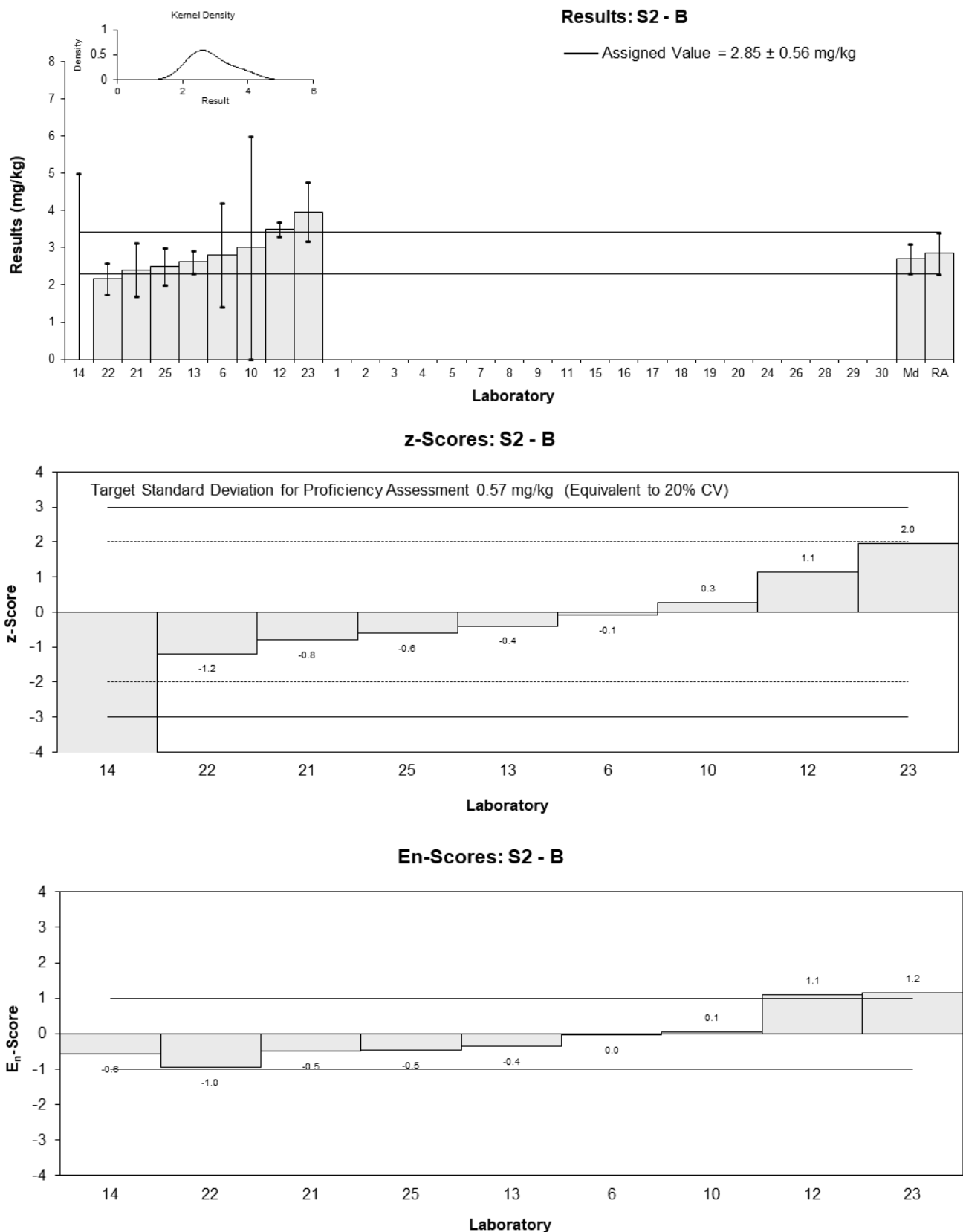


Figure 22

Table 35

Sample Details

Sample No.	S2
Matrix	Biosolid
Analyte	Be
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	0.509	0.109	-1.15	-0.83
3	NT	NT		
4	<1	1		
5	<1	NR		
6	0.61	0.11	-0.05	-0.04
7	< 2	NR		
8	<1	NR		
9	0.60	0.2	-0.16	-0.07
10	<1	1		
11	<5	NR		
12	<0.6	NR		
13	0.65	0.1	0.38	0.29
14	0.63	0.189	0.16	0.07
15	< 2	NR		
16	NT	NT		
17	0.7822	0.1	1.81	1.40
18	< 2	NR		
19	<1	0.3		
20	<1	NR		
21	NR	NR		
22	0.46	0.09	-1.68	-1.39
23	0.668	0.134	0.57	0.35
24	0.6	0.12	-0.16	-0.11
25	0.655	0.131	0.43	0.27
26	NT	NT		
28	NT	NT		
29	<2	<0.4		
30	<1	NR		

Statistics

Assigned Value	0.615	0.066
Robust Average	0.615	0.066
Median	0.620	0.038
Mean	0.616	
N	10	
Max	0.7822	
Min	0.46	
Robust SD	0.084	
Robust CV	14%	

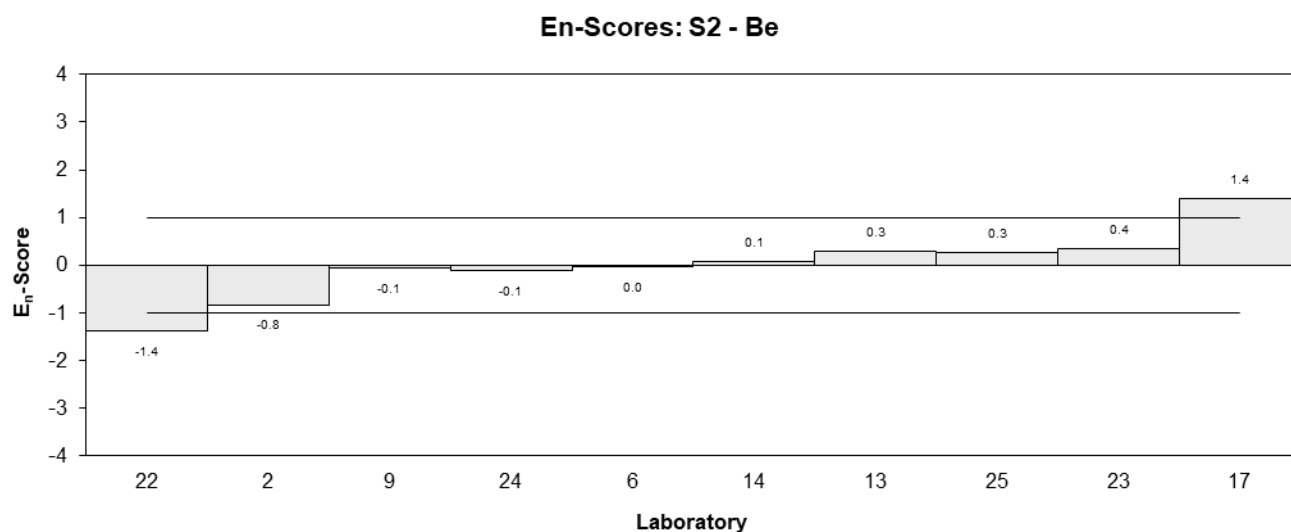
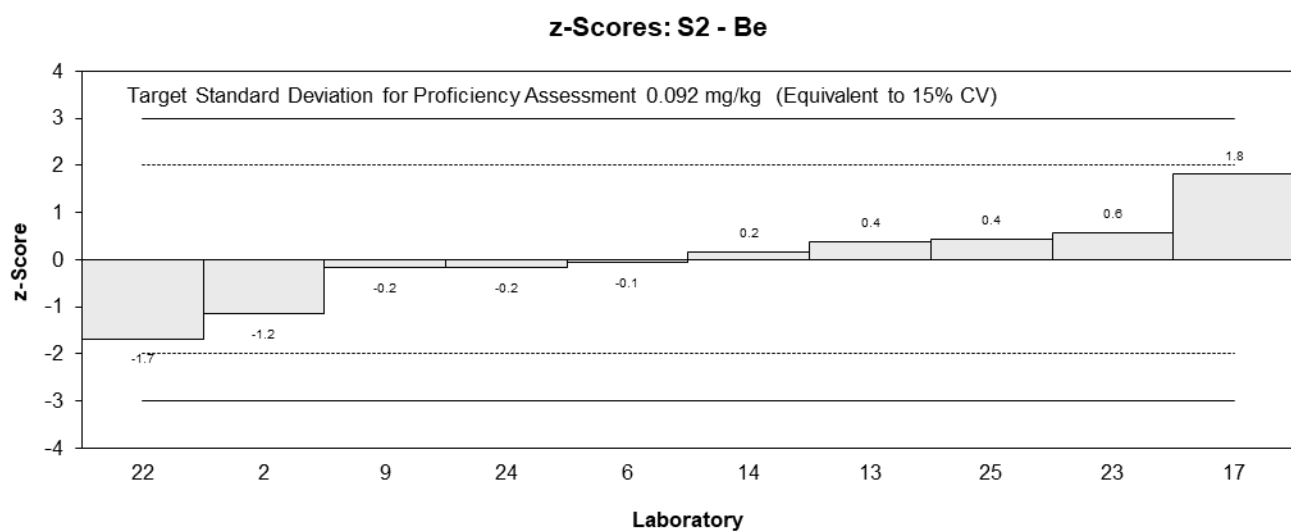
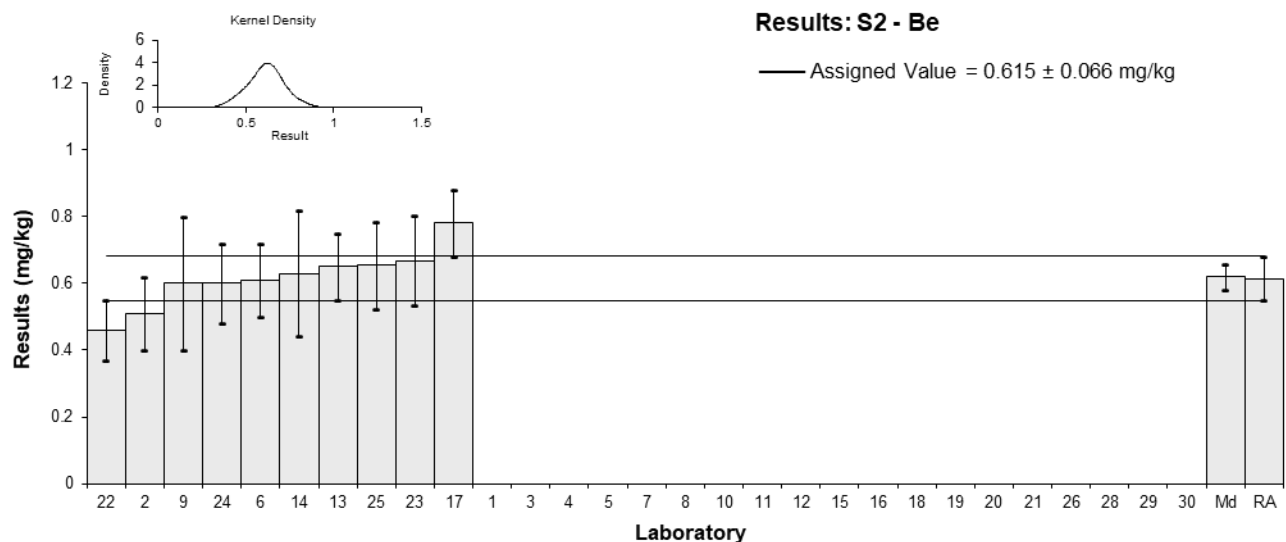


Figure 23

Table 36

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	0.727	0.2492	-0.11	-0.05
3	NT	NT		
4	<1	1		
5	NT	NT		
6	0.700	0.099	-0.35	-0.31
7	<10	NR		
8	0.7	0.2	-0.35	-0.18
9	0.80	0.2	0.55	0.28
10	<1	1		
11	NR	NR		
12	NT	NT		
13	0.54	0.1	-1.80	-1.56
14	NT	NT		
15	< 10	NR		
16	NT	NT		
17	0.902	0.3	1.47	0.53
18	< 10	NR		
19	0.7	0.11	-0.35	-0.29
20	<1	NR		
21	NR	NR		
22	0.62	0.12	-1.07	-0.83
23	0.773	0.155	0.31	0.20
24	0.8	NR	0.55	0.77
25	0.83	0.166	0.82	0.49
26	NT	NT		
28	NT	NT		
29	<10	<2		
30	<1	NR		

Statistics

Assigned Value	0.739	0.079
Robust Average	0.739	0.079
Median	0.727	0.082
Mean	0.736	
N	11	
Max	0.902	
Min	0.54	
Robust SD	0.10	
Robust CV	14%	

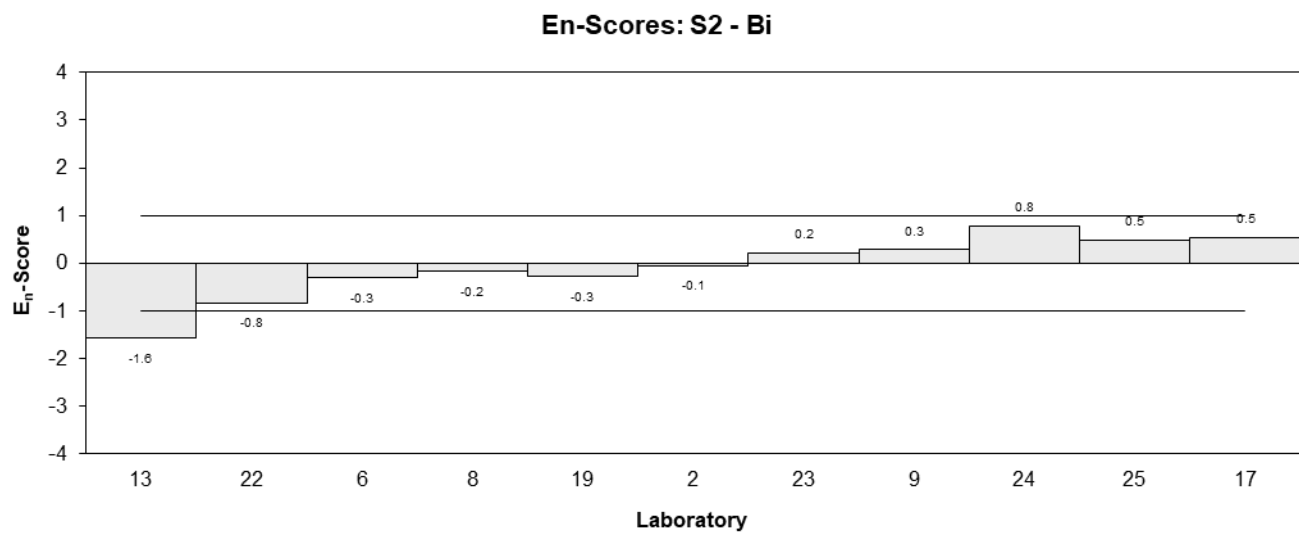
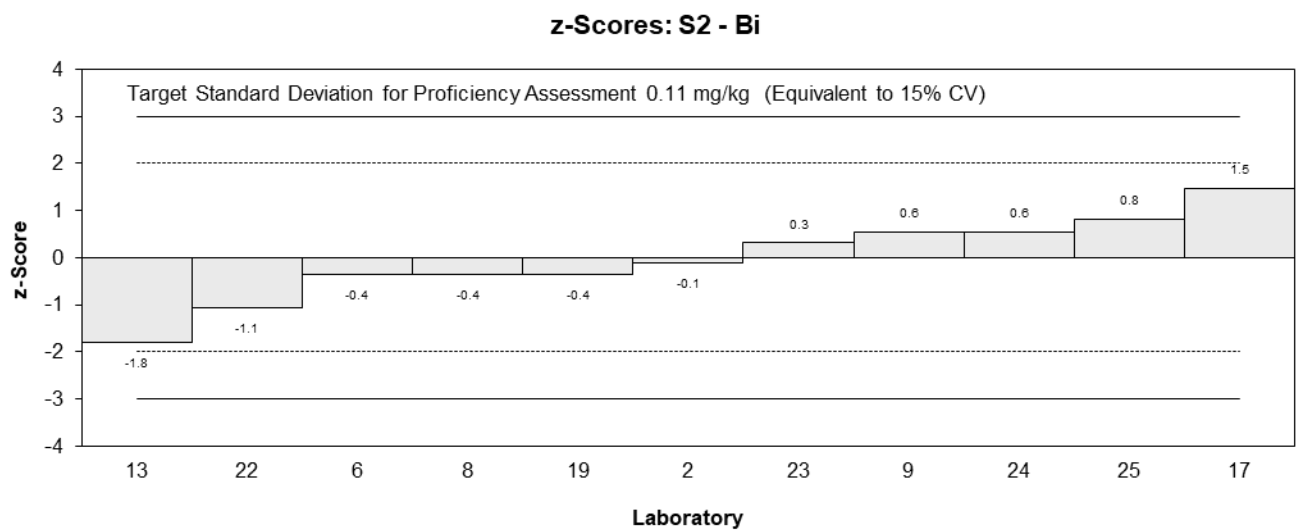
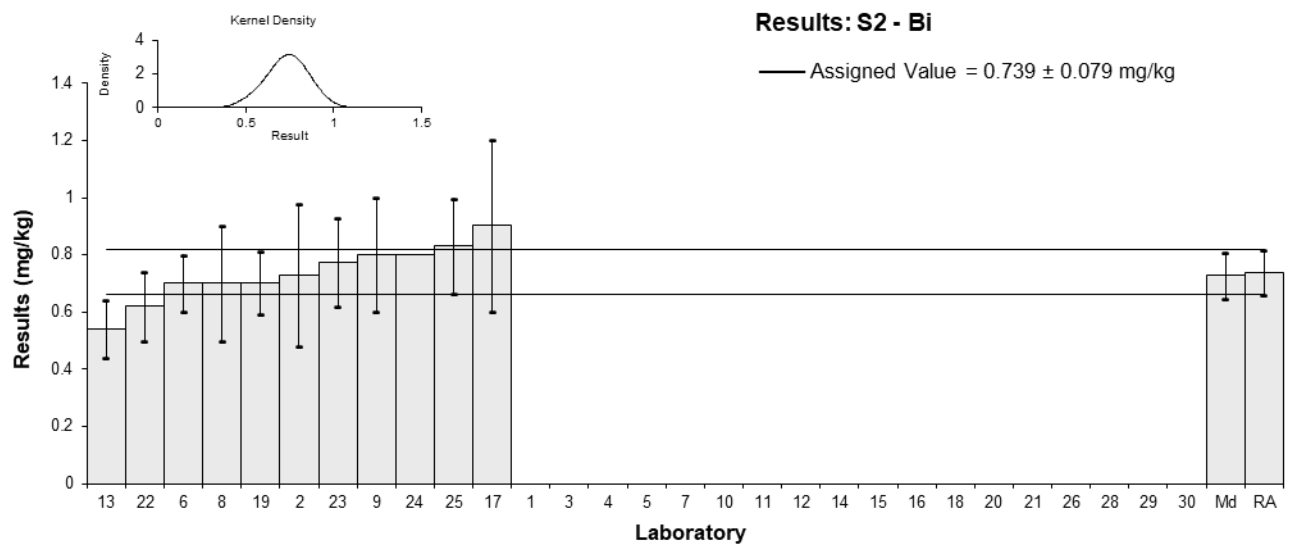


Figure 24

Table 37

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	0.673	0.14	-0.50	-0.38
3	NT	NT		
4	1	0.4	2.50	0.68
5	0.77	0.10	0.39	0.42
6	0.731	0.088	0.04	0.04
7	0.68	0.17	-0.43	-0.27
8	<1	NR		
9	0.74	0.2	0.12	0.06
10	0.7	0.4	-0.25	-0.07
11	<5	NR		
12	<1	NR		
13	0.72	0.1	-0.06	-0.07
14*	0.3	0.09	-3.92	-4.54
15	0.74	0.12	0.12	0.11
16	NT	NT		
17	0.784	0.1	0.52	0.55
18	0.75	0.11	0.21	0.20
19	<1	0.4		
20	0.7	0.5	-0.25	-0.05
21	0.65	0.20	-0.71	-0.38
22	0.64	0.13	-0.80	-0.66
23	0.713	0.143	-0.13	-0.10
24	0.7	0.1	-0.25	-0.26
25	0.762	0.1524	0.32	0.23
26	0.74	0.15	0.12	0.09
28	NT	NT		
29	0.742	0.148	0.14	0.10
30	0.786	0.079	0.54	0.71

* Outlier, see Section 4.2

Statistics

Assigned Value	0.727	0.027
Robust Average	0.722	0.029
Median	0.731	0.025
Mean	0.715	
N	21	
Max	1	
Min	0.3	
Robust SD	0.053	
Robust CV	7.4%	

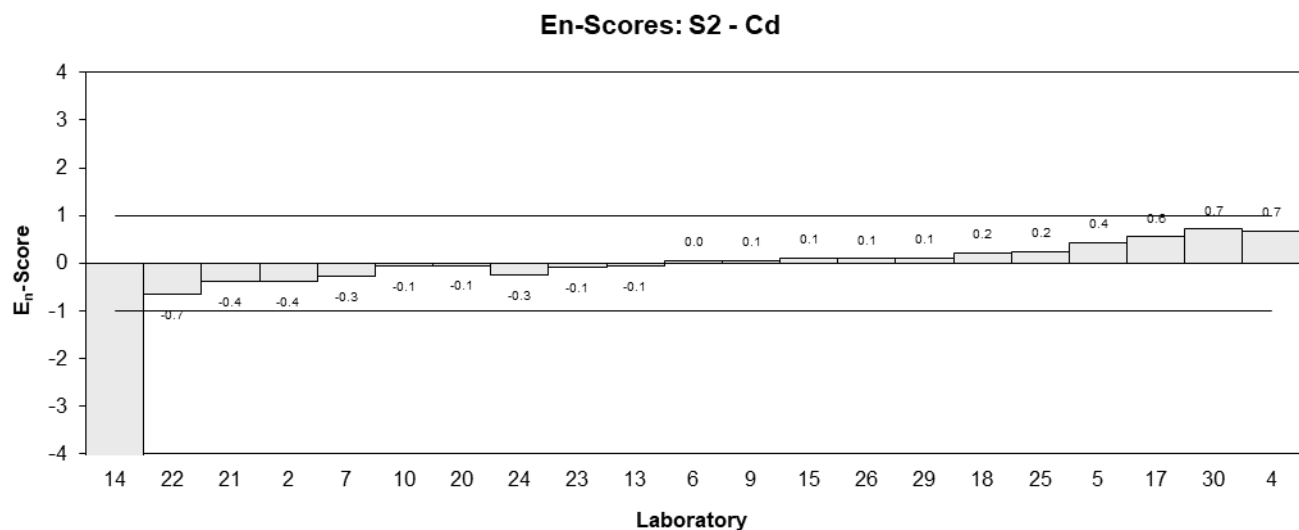
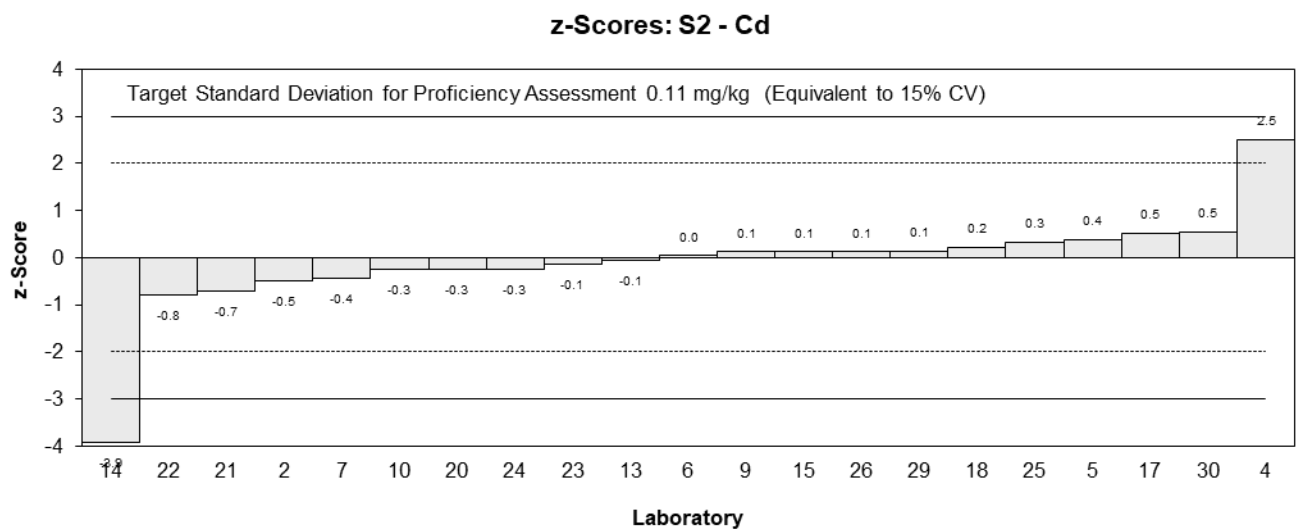
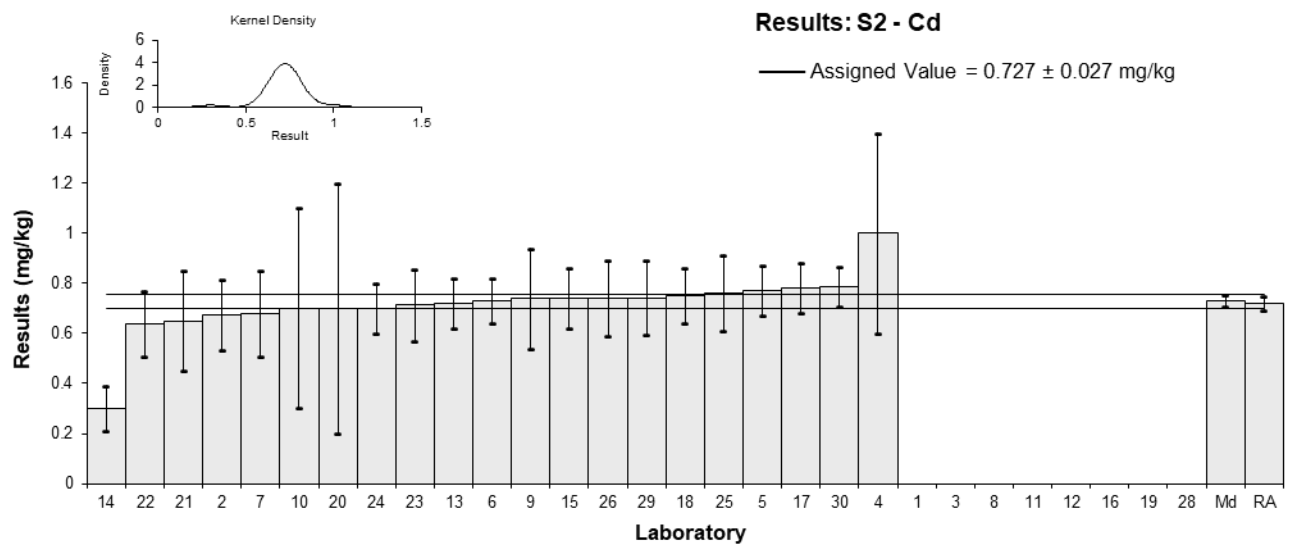


Figure 25

Table 38

Sample Details

Sample No.	S2
Matrix	Biosolid
Analyte	Cr
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	31.156	3.805	-0.50	-0.41
3	NT	NT		
4	31	7	-0.55	-0.25
5	37.3	4.8	1.37	0.90
6	39.0	6.2	1.89	0.98
7	34.83	8.71	0.62	0.23
8	30	5	-0.85	-0.54
9	33.2	3.3	0.12	0.11
10	33	7	0.06	0.03
11	29.1	5.18	-1.13	-0.69
12	34	6.8	0.37	0.17
13	32.3	4.0	-0.15	-0.12
14	31.59	9.477	-0.37	-0.13
15	31.9	4.8	-0.27	-0.18
16	NT	NT		
17	35.683	4	0.88	0.68
18	32.2	5.1	-0.18	-0.11
19	31	10	-0.55	-0.18
20	34	7	0.37	0.17
21	NR	NR		
22	26.6	5.3	-1.89	-1.13
23	34.3	6.9	0.46	0.21
24	32.7	9.16	-0.03	-0.01
25	33.75	6.75	0.29	0.14
26	28.88	8.8	-1.20	-0.44
28	NT	NT		
29	37.8	7.56	1.52	0.65
30	33.1	3.3	0.09	0.08

Statistics

Assigned Value	32.8	1.4
Robust Average	32.8	1.4
Median	32.9	1.2
Mean	32.8	
N	24	
Max	39	
Min	26.6	
Robust SD	2.8	
Robust CV	8.4%	

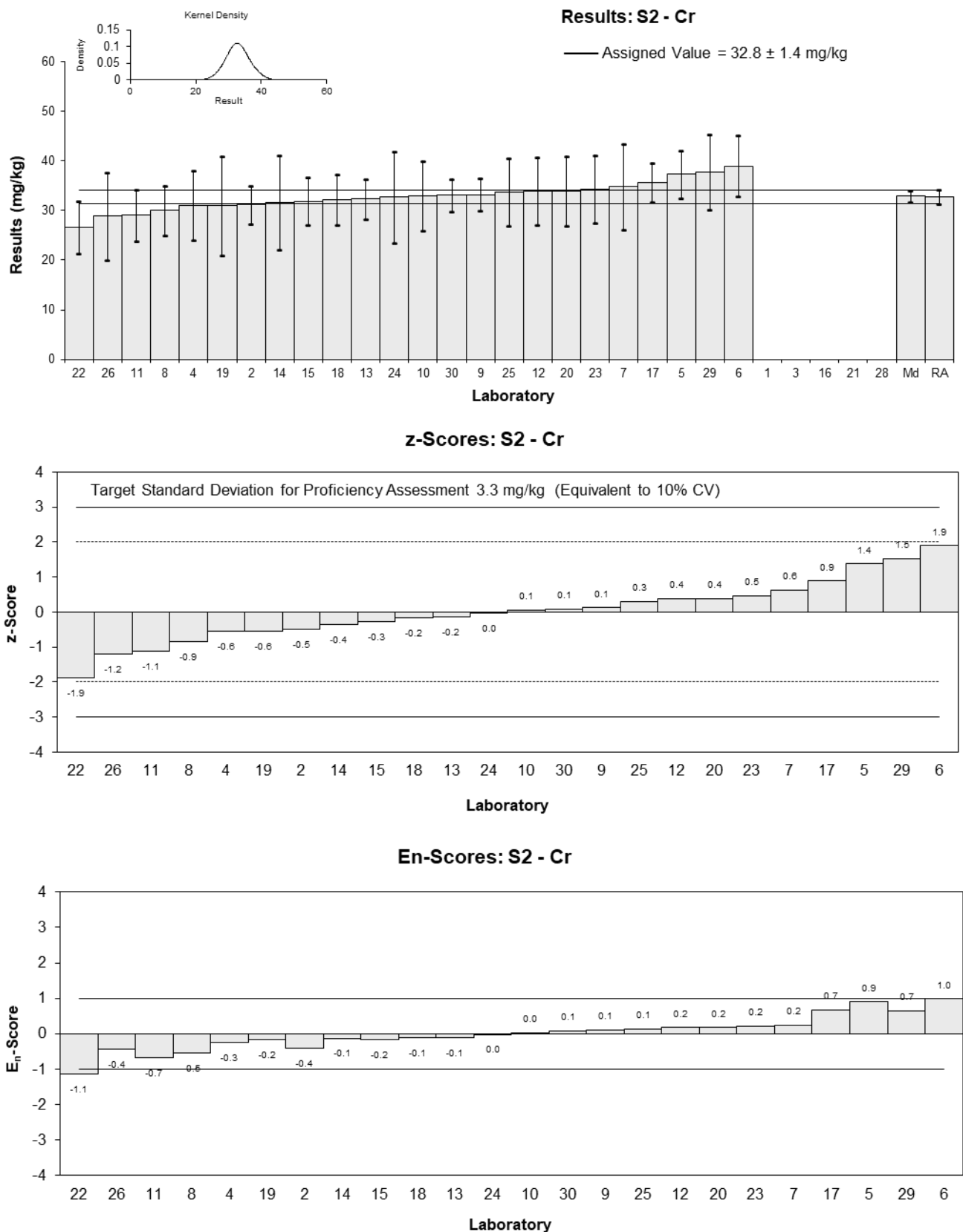


Figure 26

Table 39

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	NT	NT
2	0.717	NR
3	NT	NT
4	<1	1
5	NT	NT
6	0.85	0.16
7	NR	NR
8	0.5	0.1
9	NT	NT
10	<1	1
11	NR	NR
12	NT	NT
13	NT	NT
14	NT	NT
15	NT	NT
16	NT	NT
17	1.216	0.1
18	NT	NT
19	0.5	0.041
20	<1	NR
21	NR	NR
22	NR	NR
23	1.16	0.23
24	0.8	NR
25	0.98	0.196
26	NT	NT
28	NT	NT
29	NT	NT
30	NT	NT

Statistics

Assigned Value	Not Set	
Robust Average	0.84	0.27
Median	0.83	0.31
Mean	0.840	
N	8	
Max	1.216	
Min	0.5	
Robust SD	0.31	
Robust CV	36%	

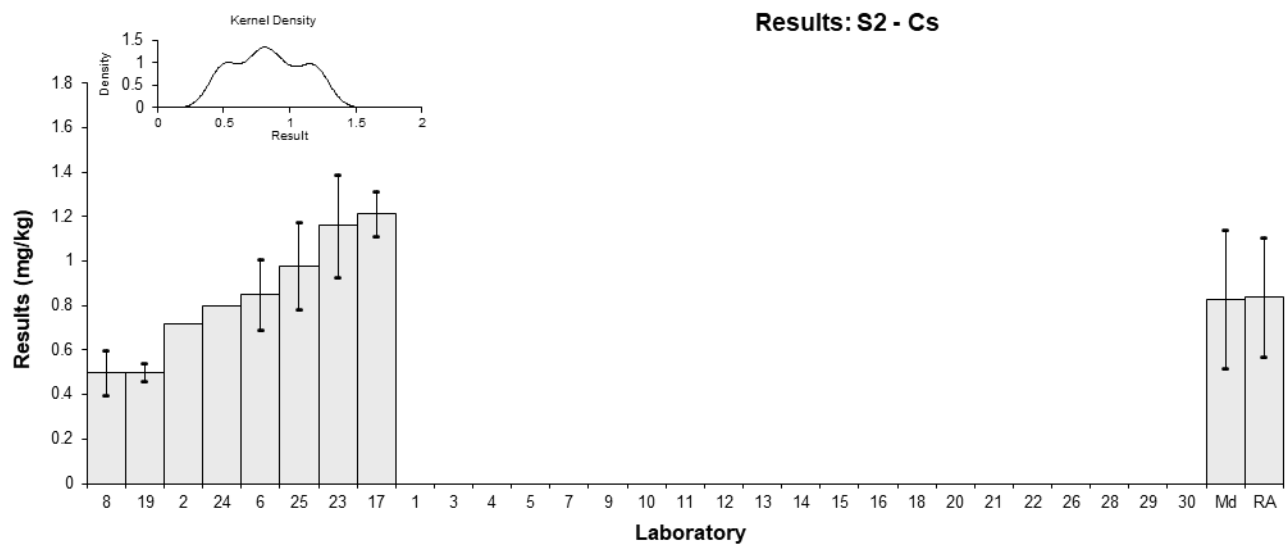


Figure 27

Table 40

Sample Details

Sample No.	S2
Matrix	Biosolid
Analyte	Fe
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	28787	2671	1.90	1.57
3	NT	NT		
4	23000	5000	-0.50	-0.23
5	24000	1100	-0.08	-0.12
6	28300	2900	1.69	1.31
7	24400.70	6100.18	0.08	0.03
8	27500	3140	1.36	0.98
9	23900	2400	-0.12	-0.11
10	22000	5000	-0.91	-0.43
11	20300	3960	-1.61	-0.94
12	24000	7200	-0.08	-0.03
13	23547	2355	-0.27	-0.25
14	22748.89	6824.667	-0.60	-0.21
15	23000	3500	-0.50	-0.32
16	NT	NT		
17	24760.8	2500	0.23	0.20
18	23333	4334	-0.36	-0.19
19	25800	10800	0.66	0.15
20	24000	5000	-0.08	-0.04
21	19400	3880	-1.98	-1.18
22	26773	5350	1.06	0.47
23	23300	4660	-0.37	-0.19
24	26800	9326	1.07	0.28
25	21550	4310	-1.10	-0.59
26	NT	NT		
28	NT	NT		
29	25900	5180	0.70	0.32
30	24600	2500	0.17	0.14

Statistics

Assigned Value	24200	1200
Robust Average	24200	1200
Median	24000	900
Mean	24200	
N	24	
Max	28787	
Min	19400	
Robust SD	2400	
Robust CV	9.7%	

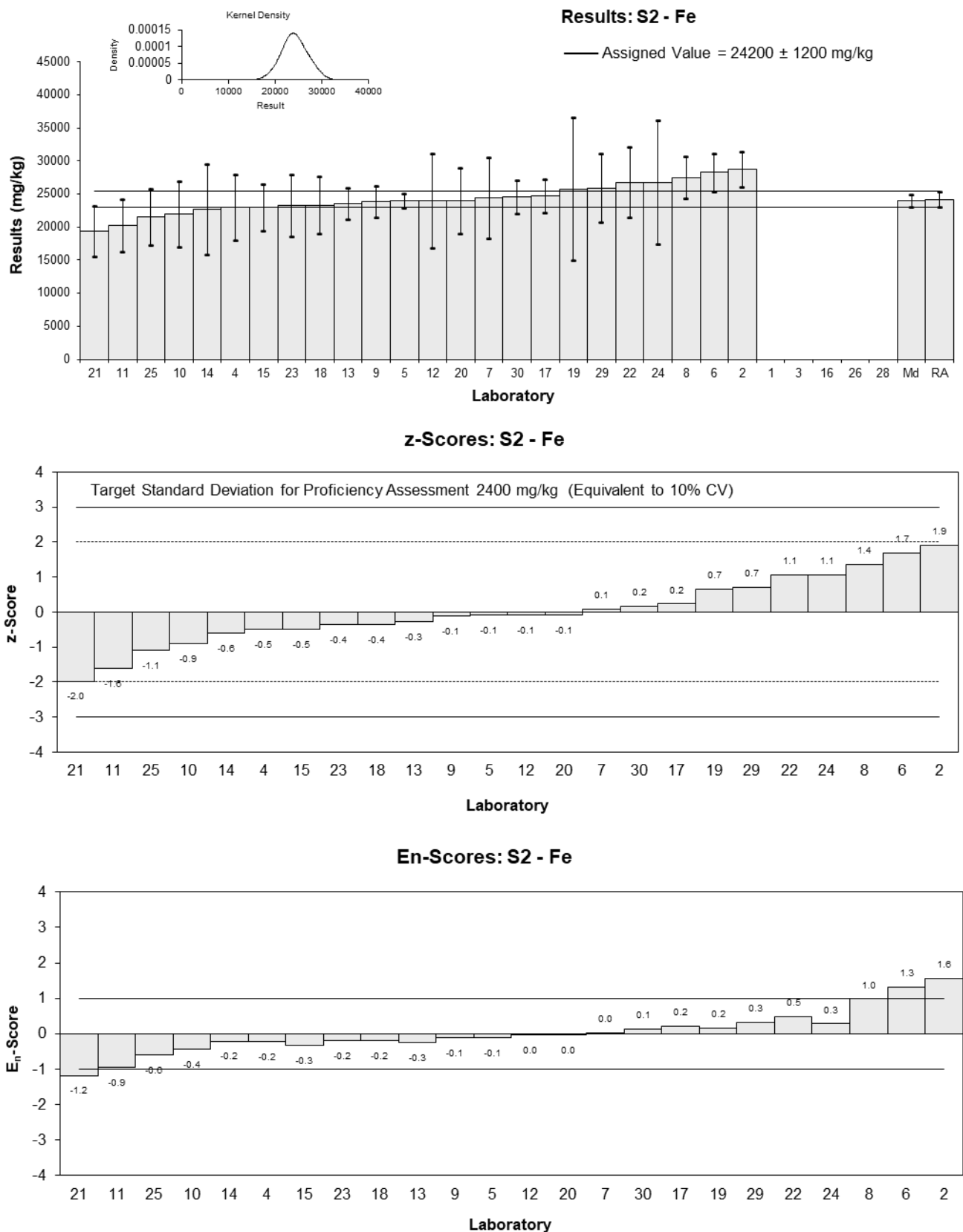


Figure 28

Table 41

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	0.56	0.095	0.60	0.61
3	NT	NT		
4	0.5	0.2	0.00	0.00
5	0.500	0.031	0.00	0.00
6	0.506	0.062	0.06	0.09
7	0.46	0.12	-0.40	-0.33
8	0.5	0.1	0.00	0.00
9	0.50	0.2	0.00	0.00
10	0.6	0.2	1.00	0.50
11	0.485	0.110	-0.15	-0.13
12	0.3	0.08	-2.00	-2.38
13	0.57	0.1	0.70	0.68
14	0.514	0.1542	0.14	0.09
15	0.5	0.08	0.00	0.00
16	NT	NT		
17	0.554	0.05	0.54	0.96
18	0.46	0.07	-0.40	-0.54
19	0.4	0.055	-1.00	-1.64
20	0.74	0.3	2.40	0.80
21	0.51	0.15	0.10	0.07
22	0.44	0.09	-0.60	-0.64
23	0.481	0.096	-0.19	-0.19
24	0.5	0.07	0.00	0.00
25	0.525	0.105	0.25	0.23
26	0.44	0.2	-0.60	-0.30
28	NT	NT		
29	0.470	0.094	-0.30	-0.31
30	0.532	0.053	0.32	0.54

Statistics

Assigned Value	0.500	0.026
Robust Average	0.500	0.026
Median	0.500	0.022
Mean	0.502	
N	25	
Max	0.74	
Min	0.3	
Robust SD	0.051	
Robust CV	10%	

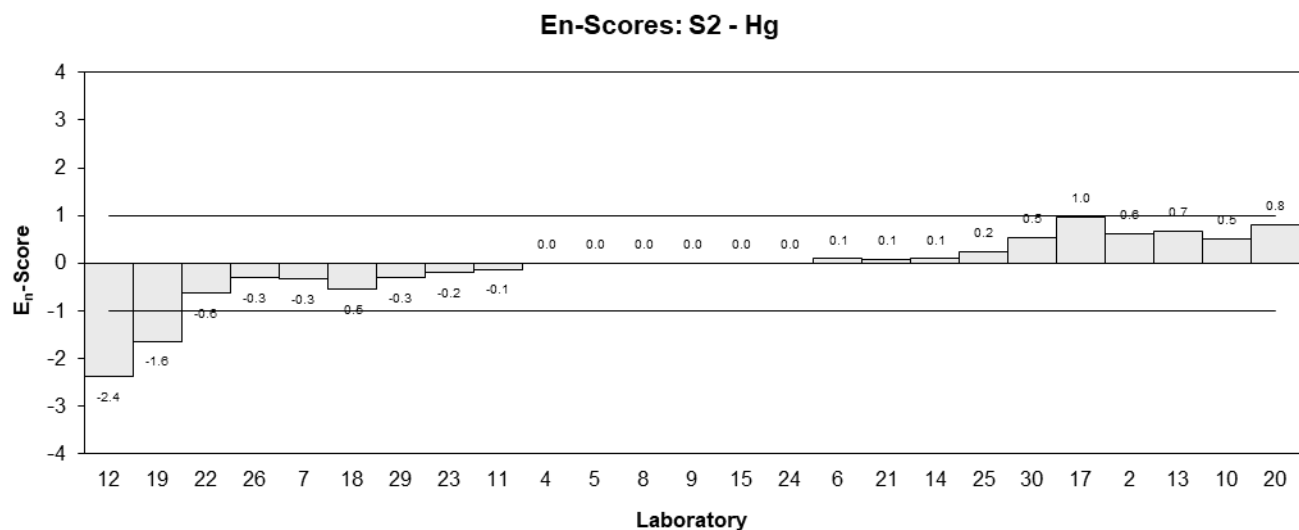
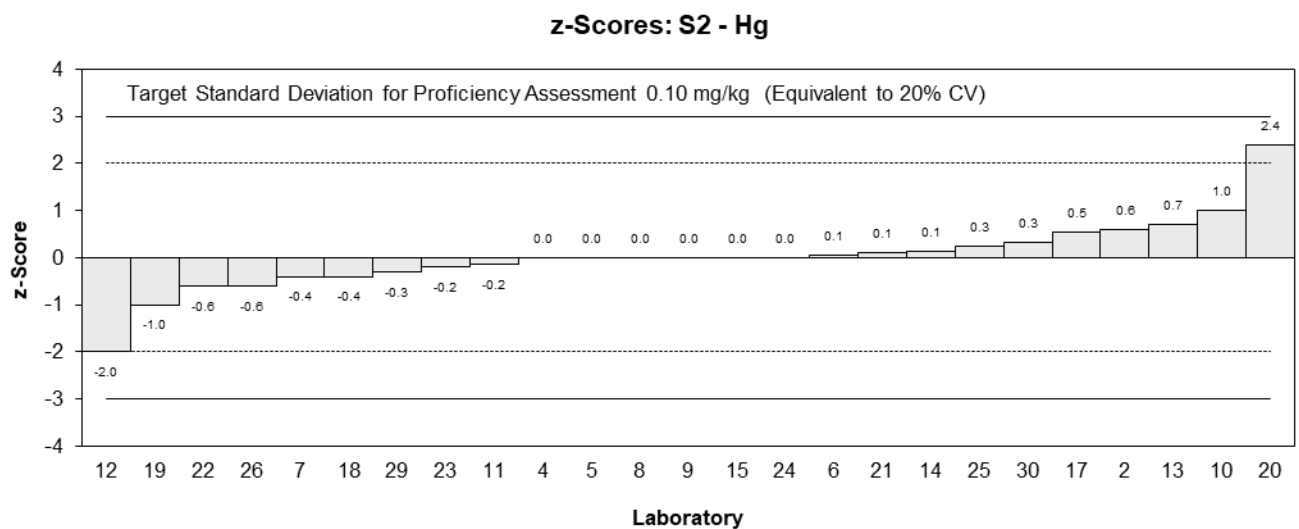
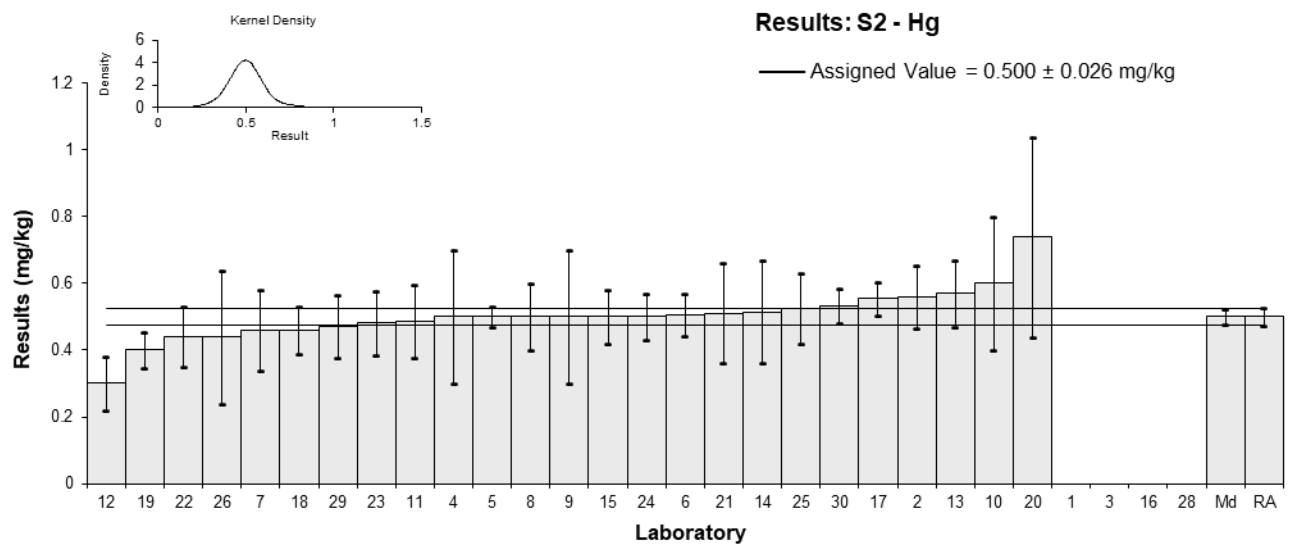


Figure 29

Table 42

Sample Details

Sample No.	S2
Matrix	Biosolid
Analyte	Mn
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	497.224	46.967	0.27	0.27
3	NT	NT		
4	460	100	-0.50	-0.24
5	490	30	0.12	0.19
6	550	56	1.36	1.16
7	483.56	120.89	-0.01	0.00
8	492	50	0.17	0.16
9	470	50	-0.29	-0.27
10	490	100	0.12	0.06
11	392	73.3	-1.90	-1.24
12	460	92	-0.50	-0.26
13	469	50	-0.31	-0.29
14	497.17	149.151	0.27	0.09
15	453	68	-0.64	-0.45
16	NT	NT		
17	484.01	50	0.00	0.00
18	470	66	-0.29	-0.21
19	505	109	0.43	0.19
20	490	100	0.12	0.06
21	NR	NR		
22	555	110	1.47	0.64
23	461	92	-0.48	-0.25
24	484	81.6	0.00	0.00
25	488.5	97.7	0.09	0.05
26	NT	NT		
28	NT	NT		
29	498	100	0.29	0.14
30	498	50	0.29	0.27

Statistics

Assigned Value	484	11
Robust Average	484	11
Median	489	7
Mean	484	
N	23	
Max	555	
Min	392	
Robust SD	21	
Robust CV	4.4%	

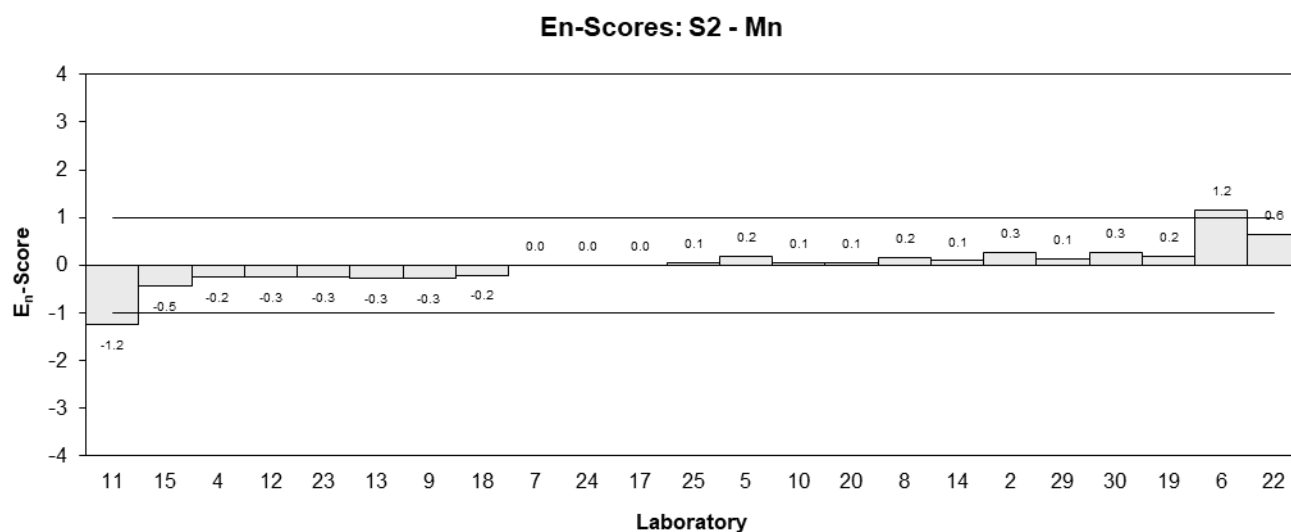
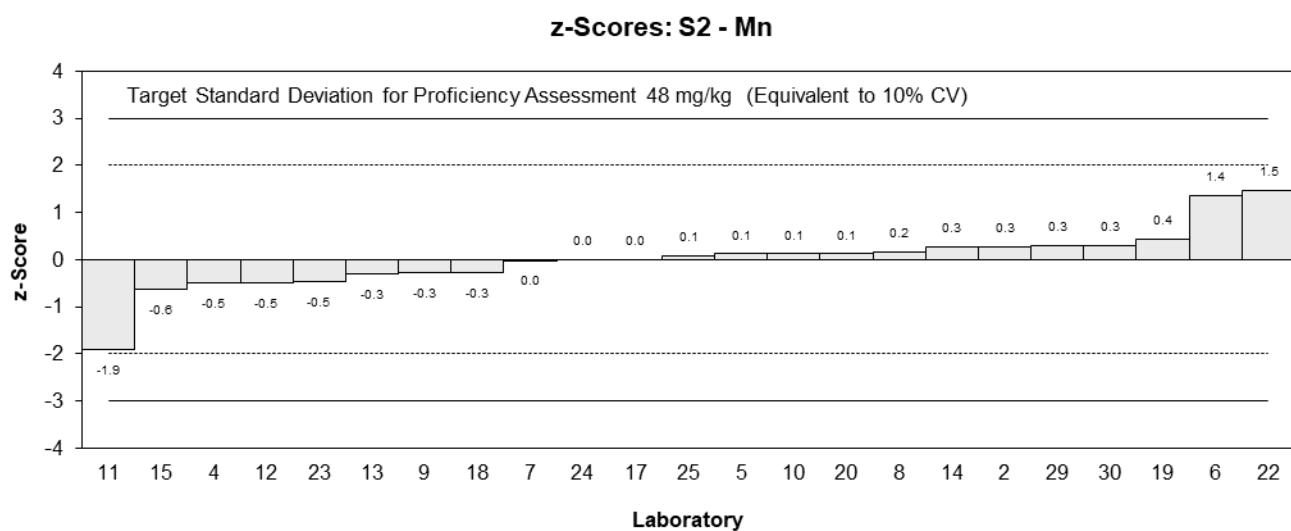
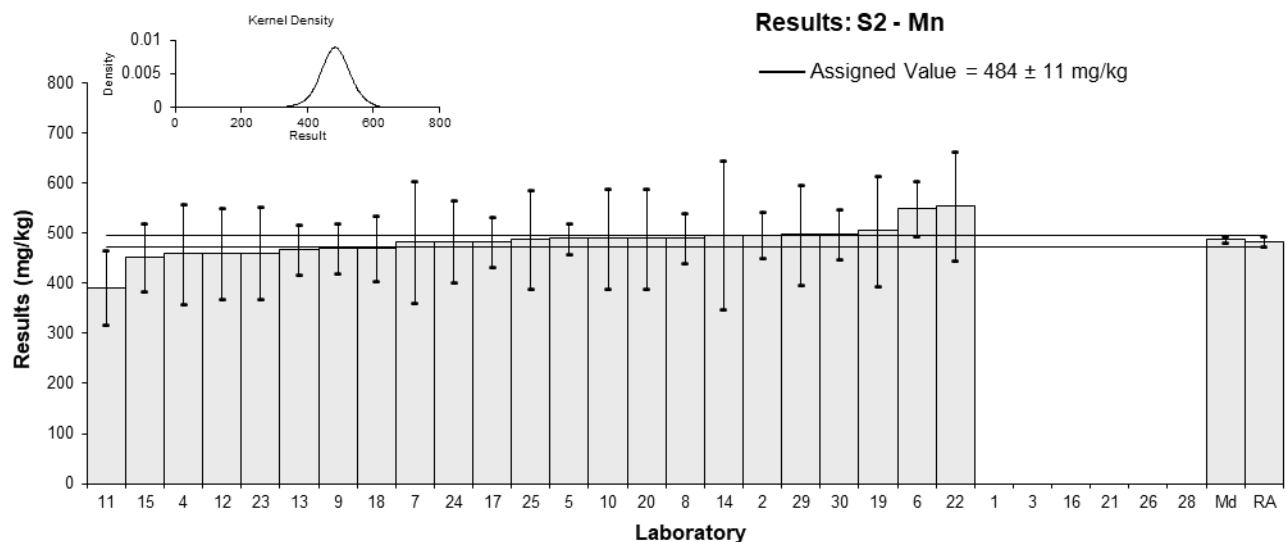


Figure 30

Table 43

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	1.346	0.246	-0.66	-0.67
3	NT	NT		
4	1	1	-1.77	-0.54
5	1.62	0.40	0.23	0.16
6	1.61	0.39	0.19	0.14
7	< 5	NR		
8	<2	NR		
9	1.8	0.2	0.81	0.93
10	1	1	-1.77	-0.54
11	<5	NR		
12	<5	NR		
13	1.56	0.2	0.03	0.04
14**	0	1	-5.00	-1.53
15	2	0.3	1.45	1.29
16	NT	NT		
17	1.674	0.2	0.40	0.46
18	< 5	NR		
19	<2	1		
20	1.4	1	-0.48	-0.15
21	NR	NR		
22*	0.56	0.11	-3.19	-4.69
23	1.44	0.29	-0.35	-0.32
24	1.4	0.3	-0.48	-0.43
25	1.65	0.33	0.32	0.27
26	NT	NT		
28	NT	NT		
29	<5	<0.75		
30	1.84	0.18	0.94	1.14

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	1.55	0.18
Robust Average	1.49	0.23
Median	1.56	0.15
Mean	1.46	
N	15	
Max	2	
Min	0.56	
Robust SD	0.36	
Robust CV	24%	

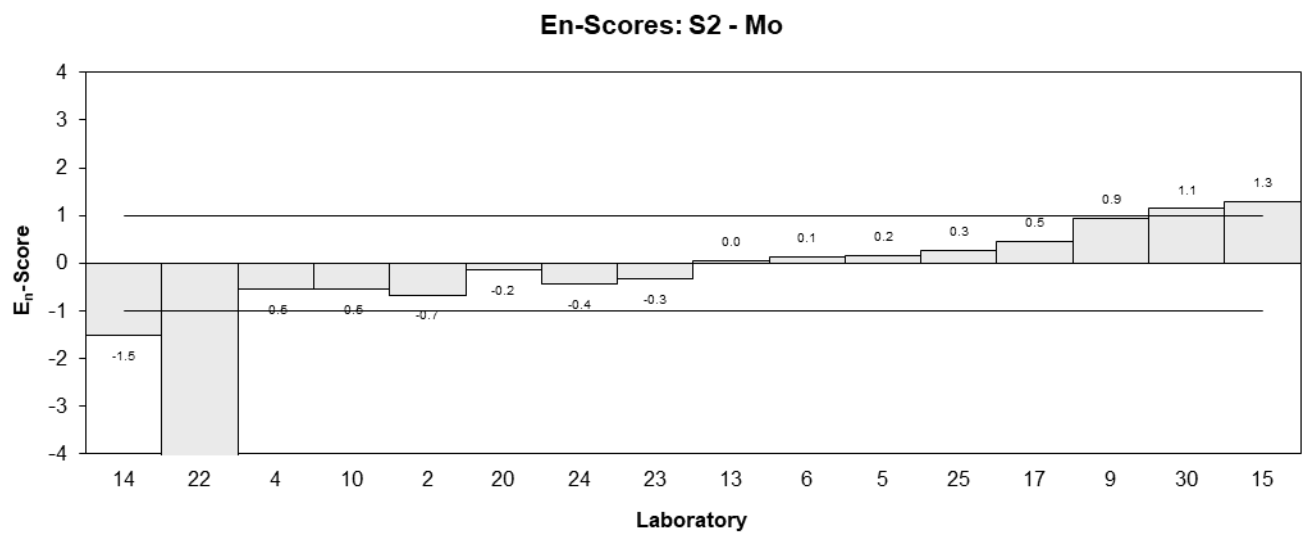
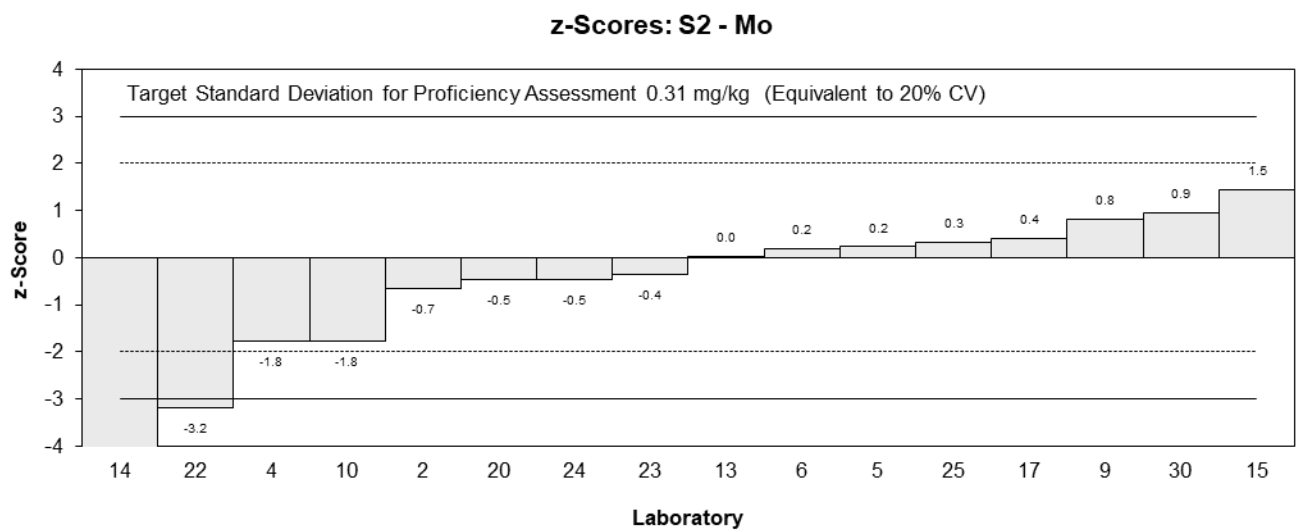
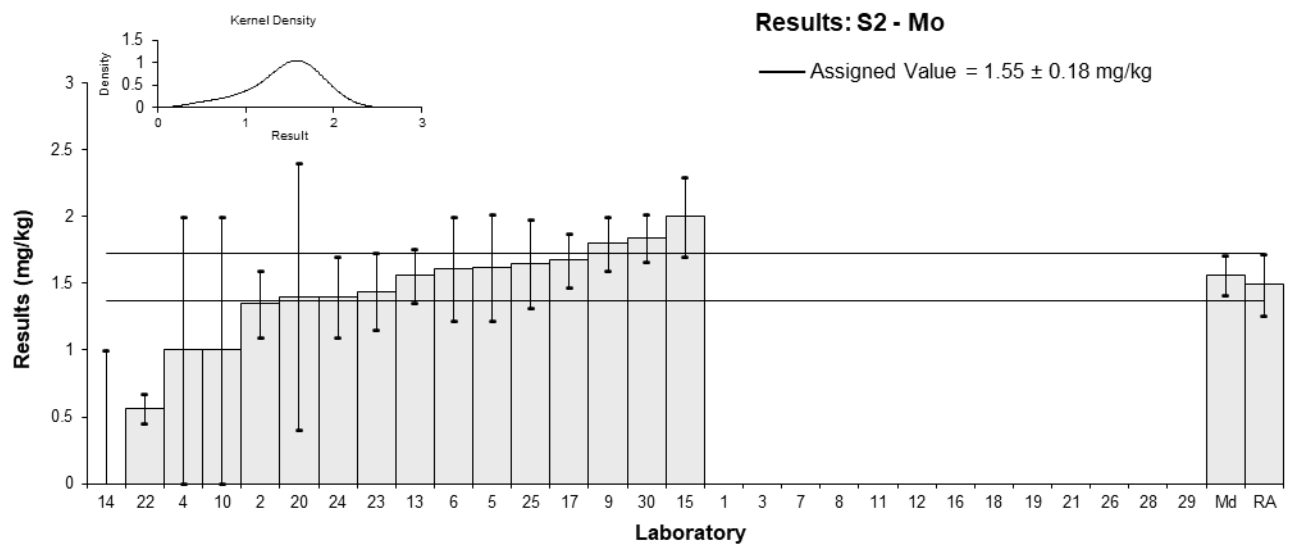


Figure 31

Table 44

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	15.494	1.794	-0.13	-0.16
3	NT	NT		
4	15	4	-0.34	-0.20
5	15.8	3.0	0.00	0.00
6	18.3	2.7	1.05	0.90
7	15.59	3.90	-0.09	-0.05
8	14	2	-0.76	-0.85
9	15.9	1.6	0.04	0.06
10	17	3	0.51	0.39
11	14.4	2.29	-0.59	-0.58
12	17	5	0.51	0.24
13	15.7	1.8	-0.04	-0.05
14	14.94	4.482	-0.36	-0.19
15	15.5	2.3	-0.13	-0.12
16	NT	NT		
17	18.842	2	1.28	1.44
18	15.7	2.5	-0.04	-0.04
19	15	2.4	-0.34	-0.32
20	17	4	0.51	0.30
21	NR	NR		
22	13.2	2.6	-1.10	-0.97
23	17.6	3.5	0.76	0.50
24	15.4	3.02	-0.17	-0.13
25	16.65	3.33	0.36	0.25
26	12.72	3.8	-1.30	-0.80
28	NT	NT		
29	15.7	3.14	-0.04	-0.03
30	15.7	1.6	-0.04	-0.06

Statistics

Assigned Value	15.8	0.7
Robust Average	15.8	0.7
Median	15.7	0.6
Mean	15.8	
N	24	
Max	18.842	
Min	12.72	
Robust SD	1.3	
Robust CV	8.5%	

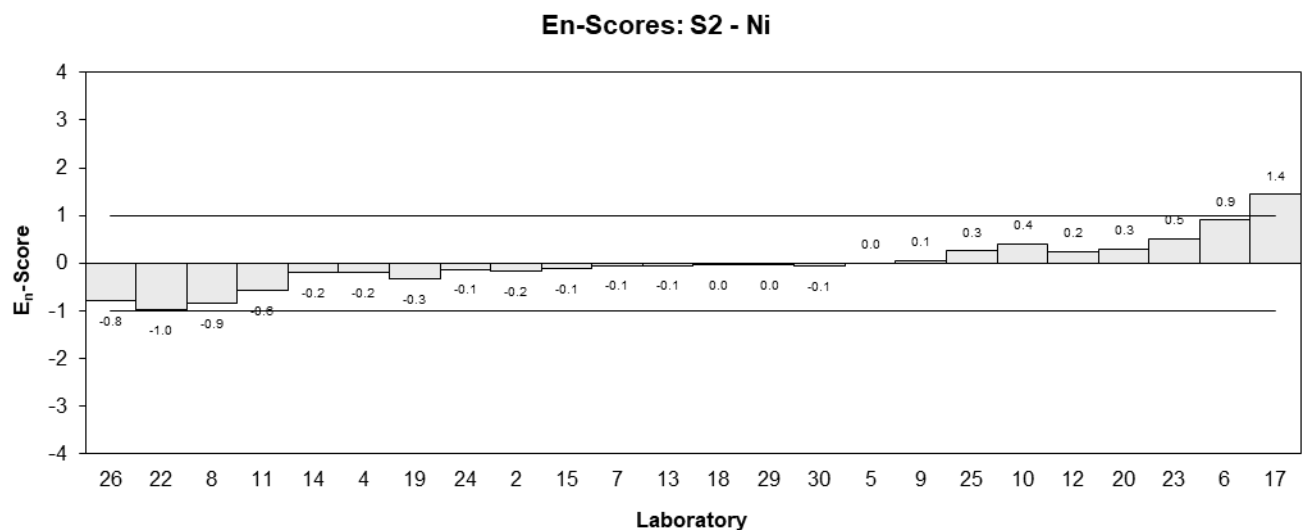
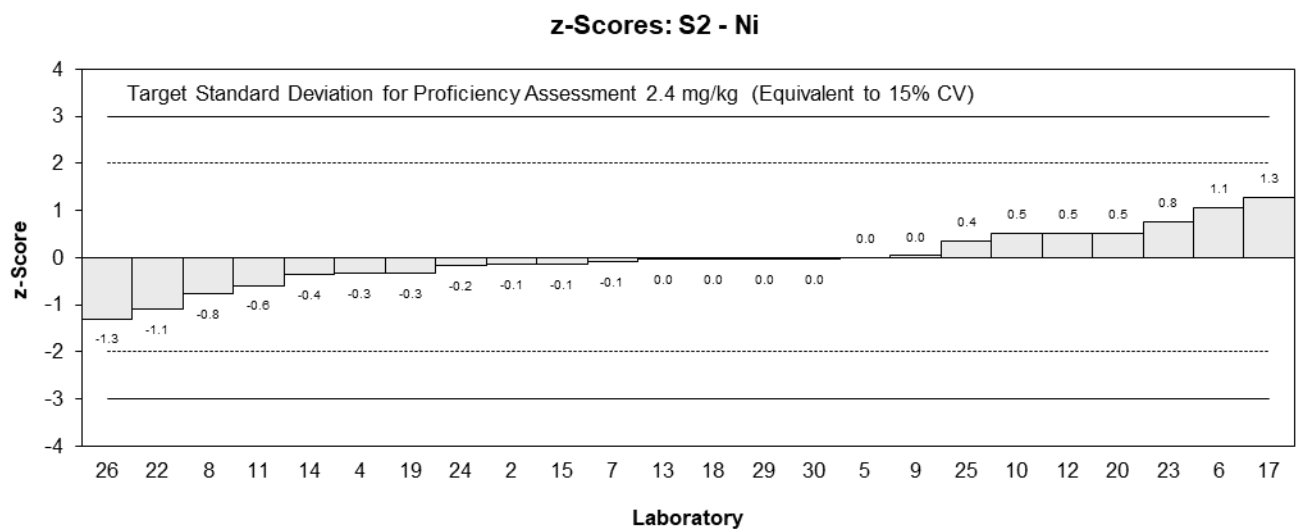
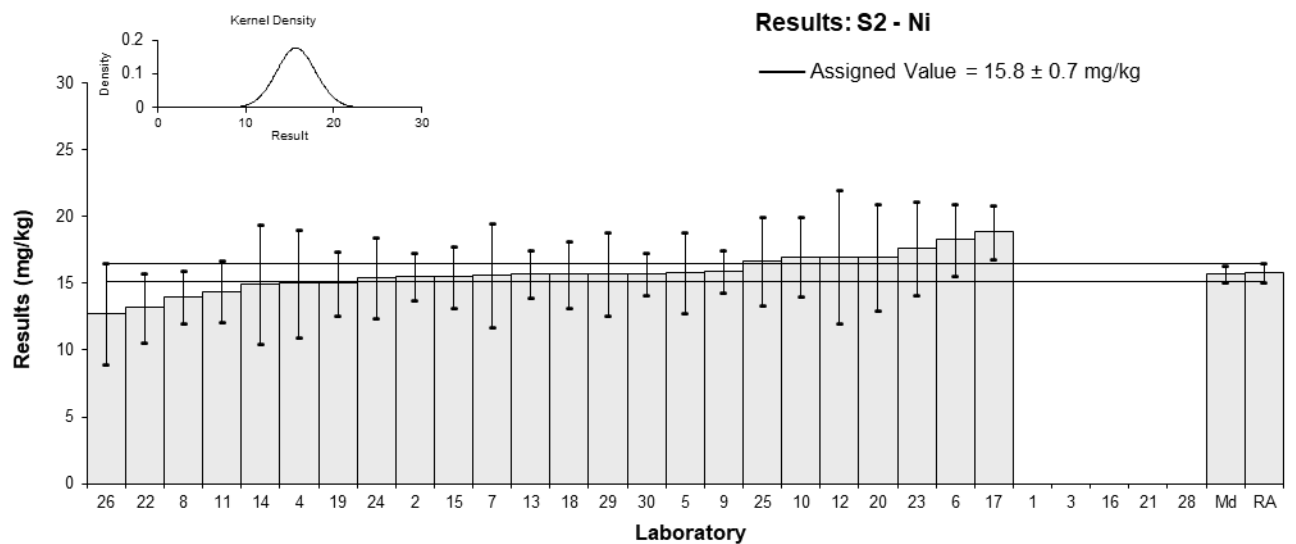


Figure 32

Table 45

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	31.855	5.144	-0.11	-0.07
3	NT	NT		
4	30	7	-0.68	-0.31
5	32.0	4.7	-0.06	-0.04
6	38.5	5.8	1.96	1.07
7	33.53	8.39	0.41	0.16
8	33	4	0.25	0.19
9	32.3	3.3	0.03	0.03
10	32	7	-0.06	-0.03
11	32.4	3.82	0.06	0.05
12	28	5.6	-1.30	-0.74
13	28.7	3.3	-1.09	-1.02
14	34	10.2	0.56	0.18
15	32.7	4.9	0.16	0.10
16	NT	NT		
17	33.466	0.3	0.39	1.21
18	32.7	5.5	0.16	0.09
19	30	9	-0.68	-0.24
20	31	7	-0.37	-0.17
21	34	NR	0.56	1.80
22	27.5	5.5	-1.46	-0.84
23	33.8	6.8	0.50	0.23
24	33.8	5.62	0.50	0.28
25	33.1	6.62	0.28	0.13
26	25.74	7.9	-2.01	-0.81
28	NT	NT		
29	33.3	6.66	0.34	0.16
30	33.9	3.4	0.53	0.48

Statistics

Assigned Value	32.2	1.0
Robust Average	32.2	1.0
Median	32.7	0.8
Mean	32.1	
N	25	
Max	38.5	
Min	25.74	
Robust SD	2.1	
Robust CV	6.5%	

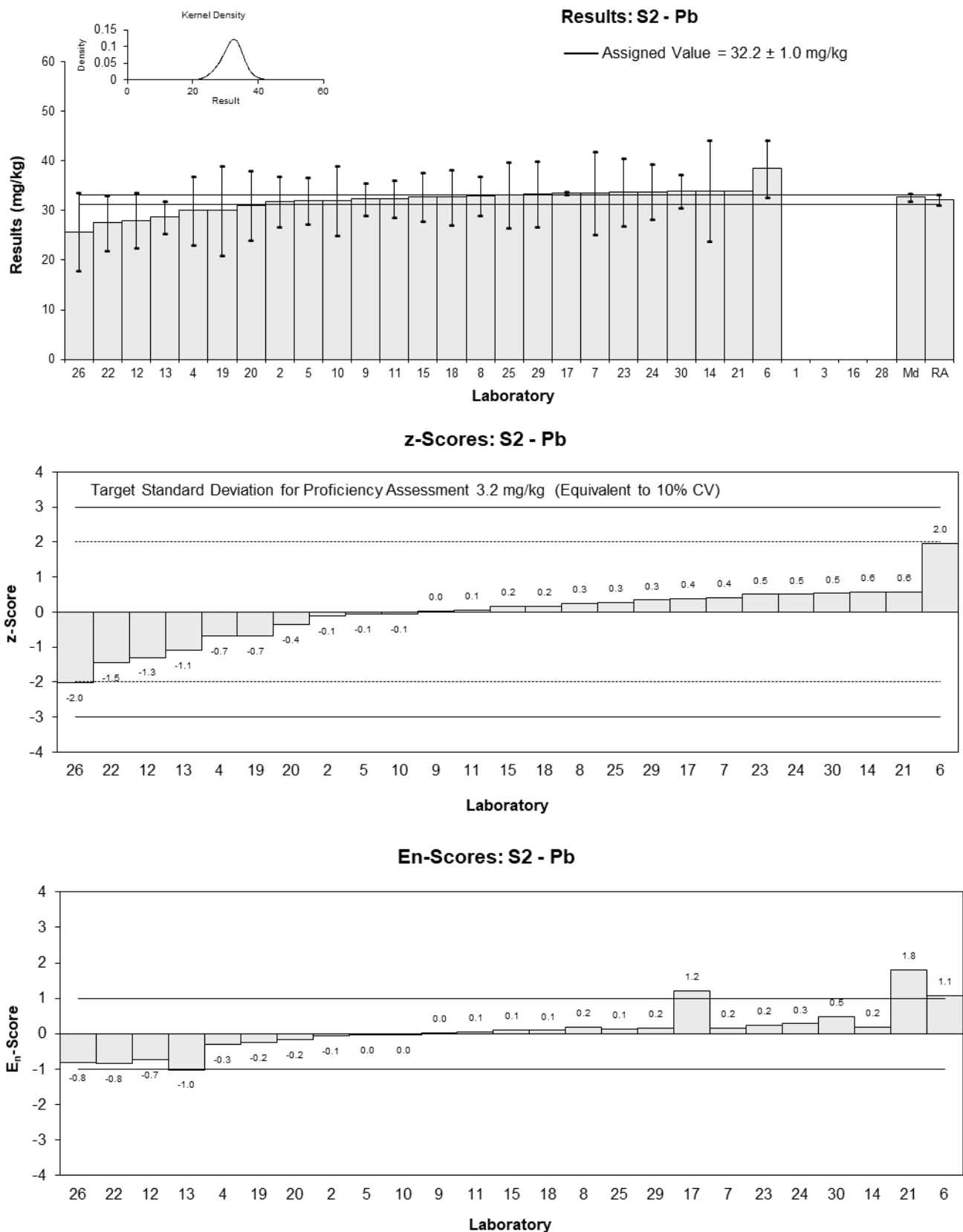


Figure 33

Table 46

Sample Details

Sample No.	S2
Matrix	Biosolid
Analyte	Rb
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	11.126	NR	-0.25	-0.25
3	NT	NT		
4	10	3	-0.73	-0.45
5	NT	NT		
6	13.5	1.4	0.77	0.67
7	NR	NR		
8	7.8	0.8	-1.67	-1.60
9	NT	NT		
10	9.4	3	-0.98	-0.61
11	NR	NR		
12	NT	NT		
13	NT	NT		
14	NT	NT		
15	NT	NT		
16	NT	NT		
17	NT	NT		
18	NT	NT		
19	9.5	0.81	-0.94	-0.90
20	14	3	0.98	0.61
21	NR	NR		
22	NR	NR		
23	15.9	3.2	1.79	1.07
24	11.3	NR	-0.17	-0.17
25	14.25	2.85	1.09	0.70
26	NT	NT		
28	NT	NT		
29	NT	NT		
30	NT	NT		

Statistics

Assigned Value	11.7	2.3
Robust Average	11.7	2.3
Median	11.2	2.4
Mean	11.7	
N	10	
Max	15.9	
Min	7.8	
Robust SD	3.0	
Robust CV	25%	

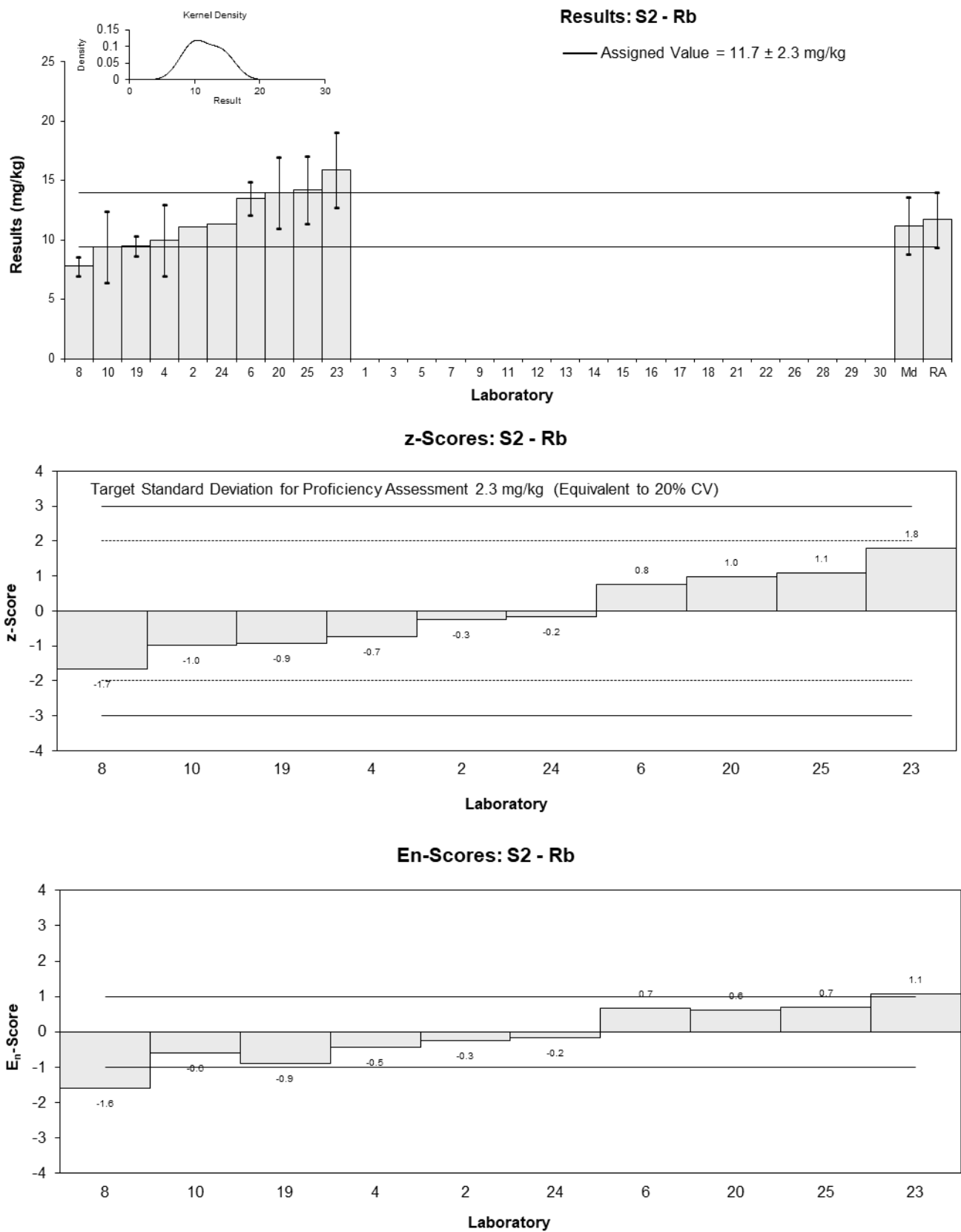


Figure 34

Table 47

Sample Details

Sample No.	S2
Matrix	Biosolid
Analyte	Sb
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty
1	NT	NT
2	0.555	0.218
3	NT	NT
4	<10	10
5	NT	NT
6	0.89	0.18
7	< 10	NR
8	<5	NR
9	1.1	0.2
10	<10	10
11	<5	NR
12	<0.5	NR
13	1.19	0.2
14**	0	3
15	1.3	0.2
16	NT	NT
17	1.292	0.1
18	< 10	NR
19	<5	2
20	<10	NR
21	NR	NR
22**	0.07	0.01
23	0.58	0.12
24	0.7	0.25
25	1.1	0.22
26	NT	NT
28	NT	NT
29	<10	<1.5
30	1.38	0.14

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	Not Set	
Robust Average	1.01	0.28
Median	1.10	0.24
Mean	1.01	
N	10	
Max	1.38	
Min	0.555	
Robust SD	0.35	
Robust CV	35%	

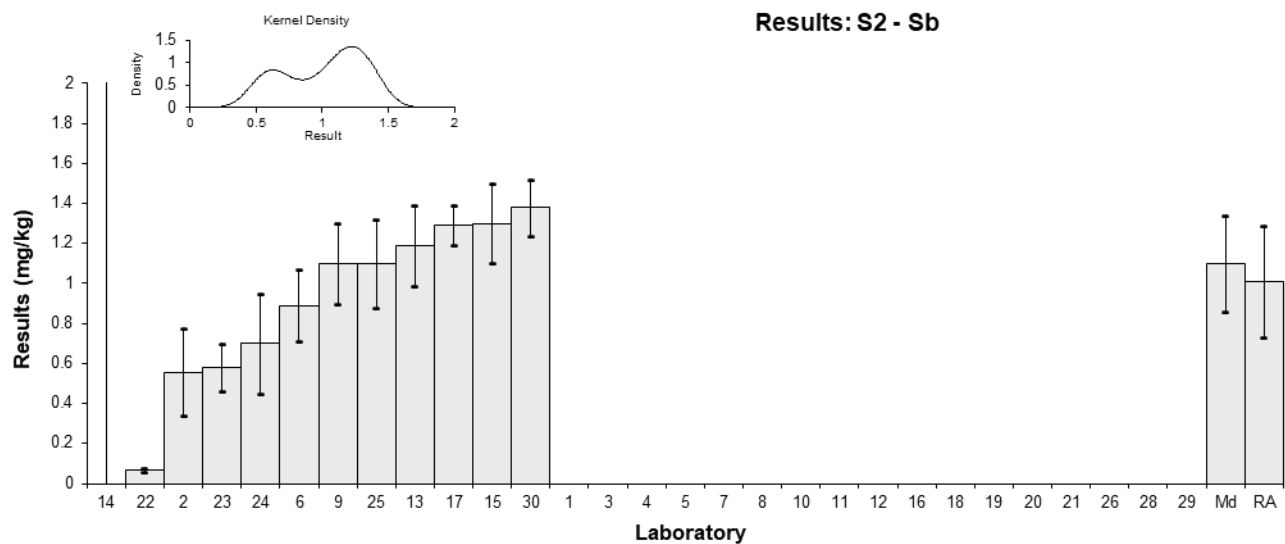


Figure 35

Table 48

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	1.341	NR	-0.38	-0.42
3	NT	NT		
4	<2	2		
5	<4	NR		
6	1.3	1.4	-0.52	-0.11
7	< 2	NR		
8	<5	NR		
9	1.8	0.2	1.21	1.07
10	<2	2		
11	<5	NR		
12	<5	NR		
13	1.46	0.2	0.03	0.03
14*	5.23	1.569	13.03	2.38
15	< 2	NR		
16	NT	NT		
17	1.406	0.15	-0.15	-0.15
18	< 2	NR		
19	<5	1		
20	<2	NR		
21	NR	NR		
22	0.87	0.17	-2.00	-1.87
23	1.23	0.25	-0.76	-0.61
24	2	0.55	1.90	0.90
25	1.7	0.34	0.86	0.58
26	NT	NT		
28	NT	NT		
29	<2	<0.4		
30	1.38	0.14	-0.24	-0.24

* Outlier, see Section 4.2

Statistics

Assigned Value	1.45	0.26
Robust Average	1.51	0.31
Median	1.41	0.20
Mean	1.79	
N	11	
Max	5.23	
Min	0.87	
Robust SD	0.41	
Robust CV	27%	

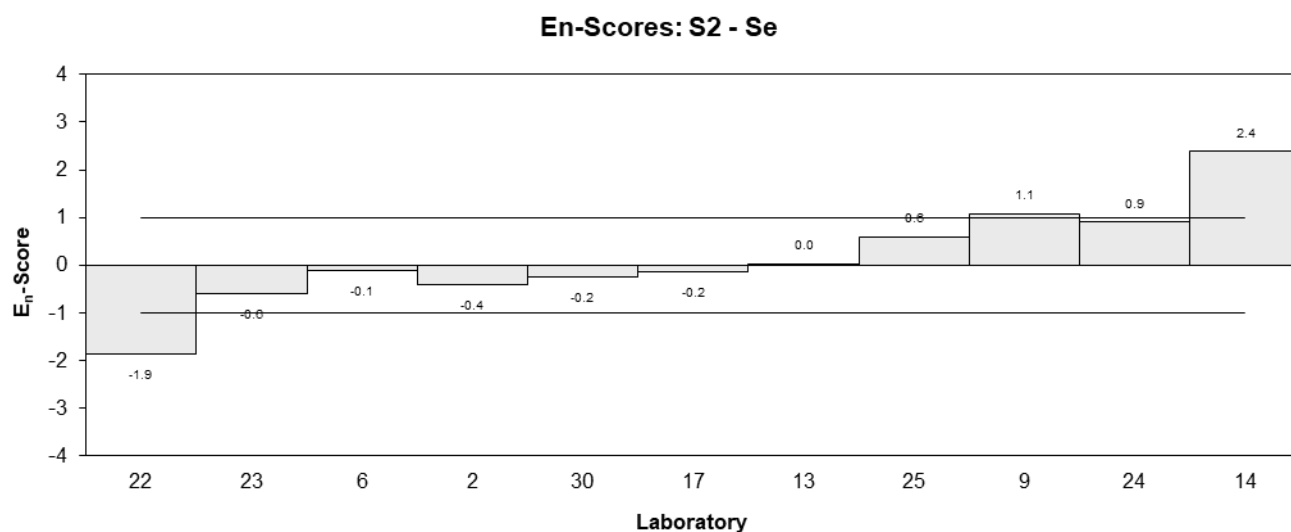
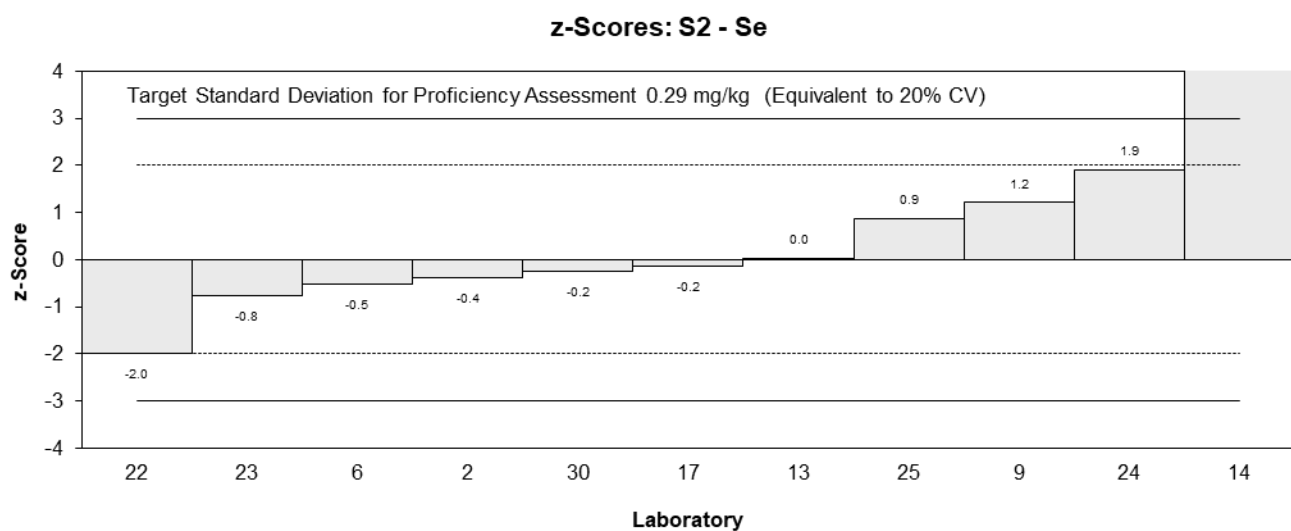
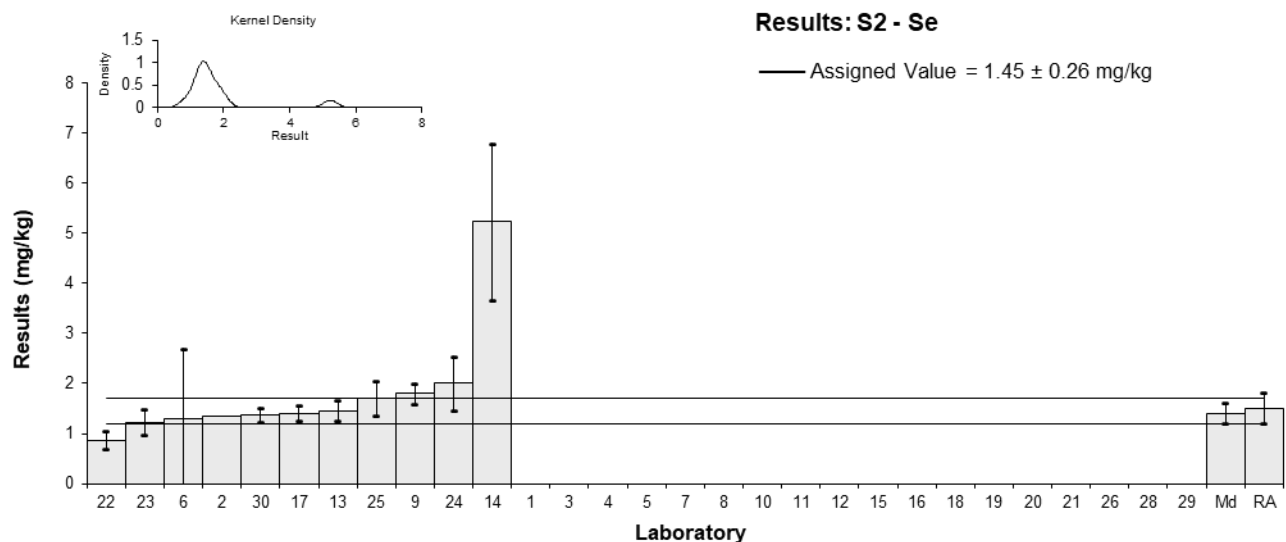


Figure 36

Table 49

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	1.018	0.121	-0.20	-0.22
3	NT	NT		
4	1	1	-0.32	-0.05
5	1.15	0.14	0.63	0.62
6	1.117	0.095	0.43	0.54
7	< 10	NR		
8	1.0	0.2	-0.32	-0.23
9	1.2	0.2	0.95	0.70
10	0.7	0.7	-2.22	-0.50
11	<5	NR		
12	NT	NT		
13	1.04	0.2	-0.06	-0.05
14	NT	NT		
15	< 10	NR		
16	NT	NT		
17	1.184	0.1	0.85	1.05
18	< 10	NR		
19	0.9	14.7	-0.95	-0.01
20	0.9	0.5	-0.95	-0.30
21**	0.100	0.03	-6.03	-11.12
22	0.92	0.18	-0.83	-0.66
23	1.11	0.22	0.38	0.26
24	1.1	NR	0.32	0.63
25	1.1	0.22	0.32	0.21
26	NT	NT		
28	NT	NT		
29	<10	<2		
30	1.17	0.11	0.76	0.88

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	1.05	0.08
Robust Average	1.05	0.08
Median	1.07	0.07
Mean	1.04	
N	16	
Max	1.2	
Min	0.7	
Robust SD	0.12	
Robust CV	12%	

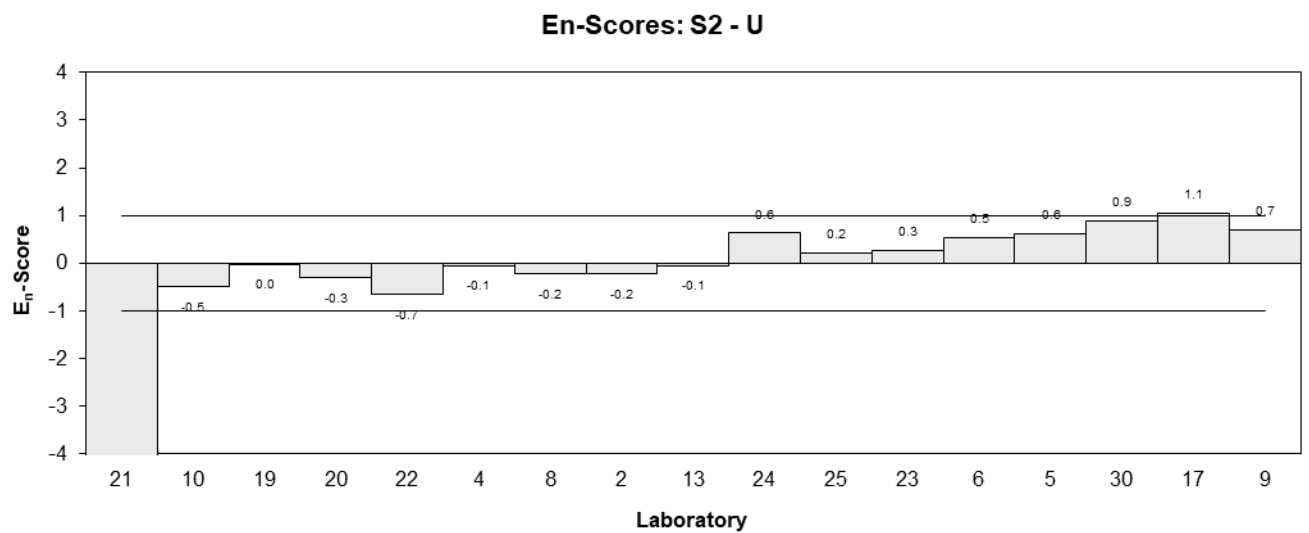
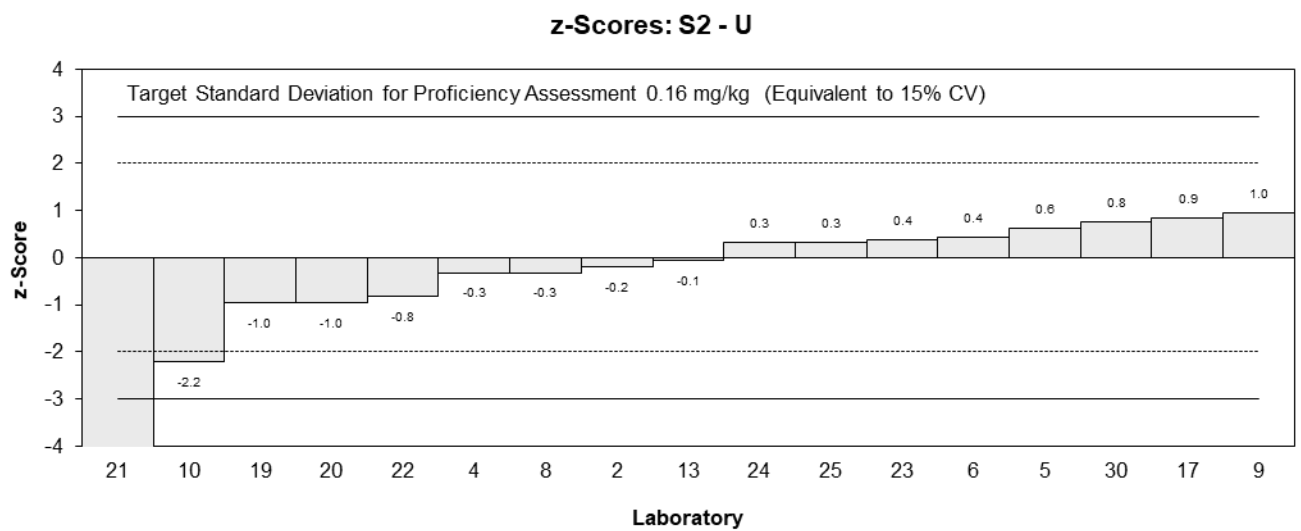
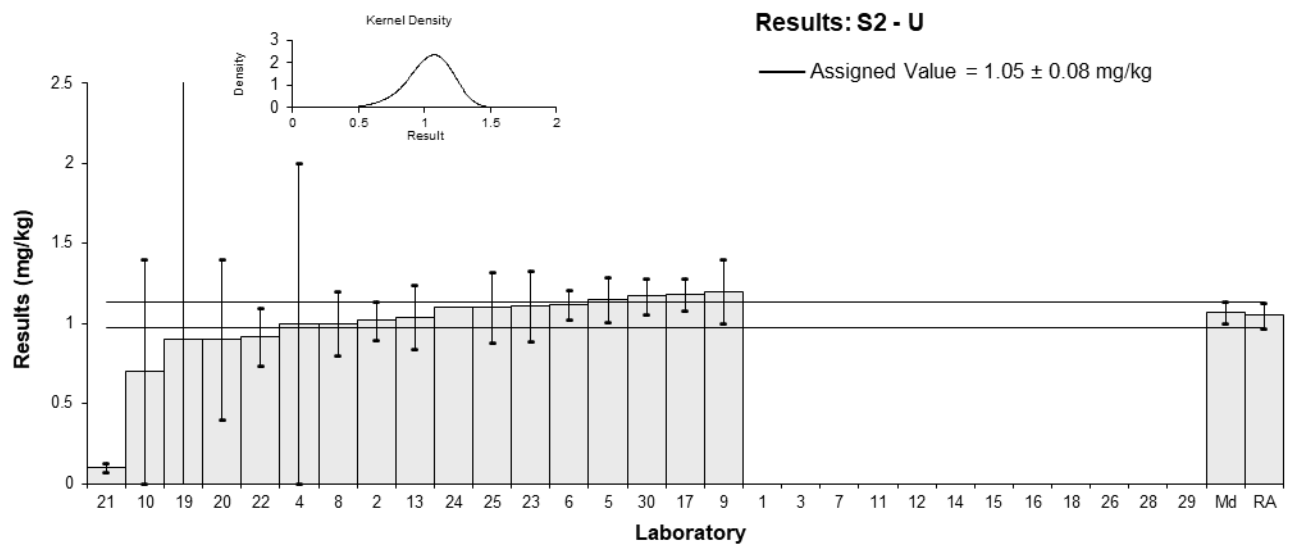


Figure 37

Table 50

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	NT	NT		
2	171.418	19.971	-0.42	-0.36
3	NT	NT		
4	160	40	-1.06	-0.47
5	190	15	0.61	0.66
6	223	16	2.46	2.52
7	178.70	44.68	-0.02	-0.01
8	194	20	0.84	0.71
9	180	18	0.06	0.05
10	170	40	-0.50	-0.22
11	146	32.7	-1.84	-0.99
12	160	32	-1.06	-0.58
13	171	19	-0.45	-0.40
14	179.98	53.994	0.05	0.02
15	170	26	-0.50	-0.33
16	NT	NT		
17	193.20	20	0.79	0.67
18	172	26	-0.39	-0.26
19	183	39	0.22	0.10
20	180	40	0.06	0.02
21	198	39.6	1.06	0.47
22	165	30	-0.78	-0.45
23	176	35	-0.17	-0.08
24	180	27.94	0.06	0.03
25	183	36.6	0.22	0.11
26	175.40	52	-0.20	-0.07
28	NT	NT		
29	186	37.2	0.39	0.18
30	200	20	1.17	0.99

Statistics

Assigned Value	179	7
Robust Average	179	7
Median	180	7
Mean	179	
N	25	
Max	223	
Min	146	
Robust SD	14	
Robust CV	7.7%	

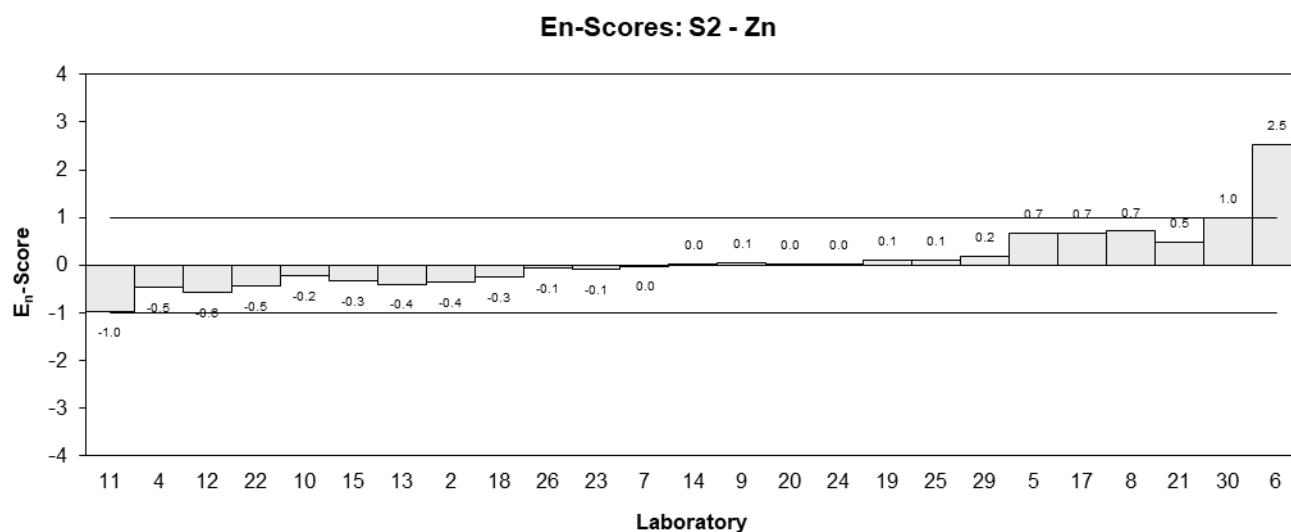
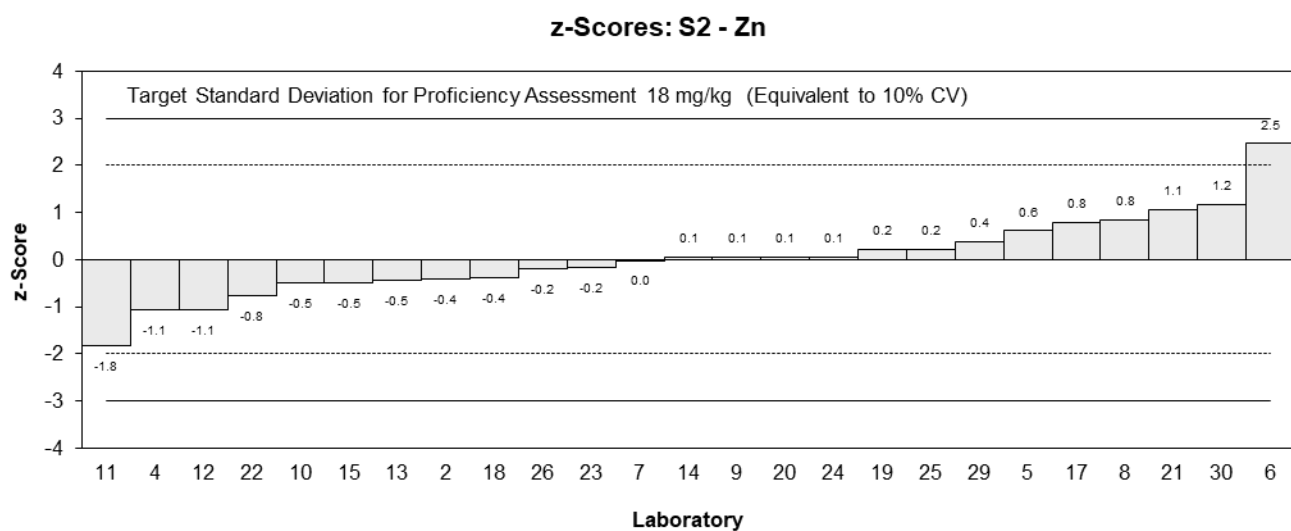
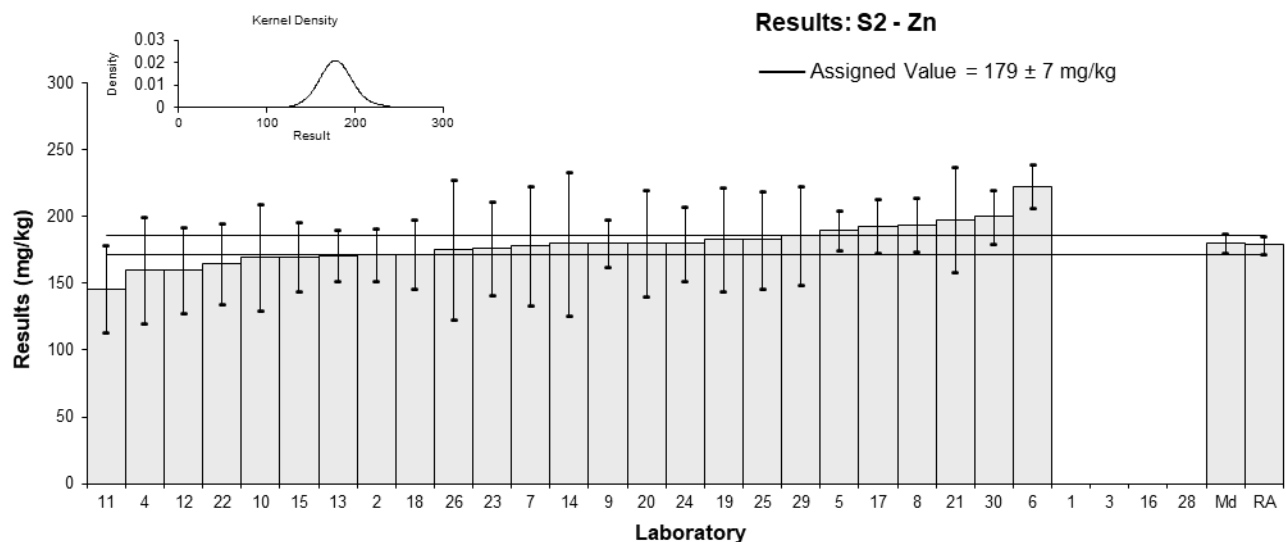


Figure 38

Table 51

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	5450	1060	-0.59	-0.32
2	5889.2	1071	0.17	0.09
3*	750.0	5.0	-8.70	-29.63
4	5400	1000	-0.67	-0.38
5	5700	500	-0.16	-0.17
6	NT	NT		
7	5785.22	1468.81	-0.01	0.00
8	6020	1900	0.40	0.12
9	5700	570	-0.16	-0.15
10	5800	1000	0.02	0.01
11	5160	1630	-1.09	-0.38
12	5900	1180	0.19	0.09
13	6809	680	1.76	1.45
14**	100.003	30.0009	-9.83	-32.96
15	6000	900	0.36	0.23
16	NR	NR		
17	5806	600	0.03	0.03
18	NT	NT		
19	6150	1427	0.62	0.25
20	5300	1200	-0.85	-0.40
21	6190	1238	0.69	0.32
22	5770	1150	-0.03	-0.02
23	5440	1090	-0.60	-0.32
24	6080	1160	0.50	0.25
25	NT	NT		
26	NT	NT		
28	NT	NT		
29	5770	1154	-0.03	-0.02
30	5820	580	0.05	0.05

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	5790	170
Homogeneity Value	6290	750
Robust Average	5760	190
Median	5790	170
Mean	5580	
N	22	
Max	6809	
Min	750	
Robust SD	350	
Robust CV	6%	

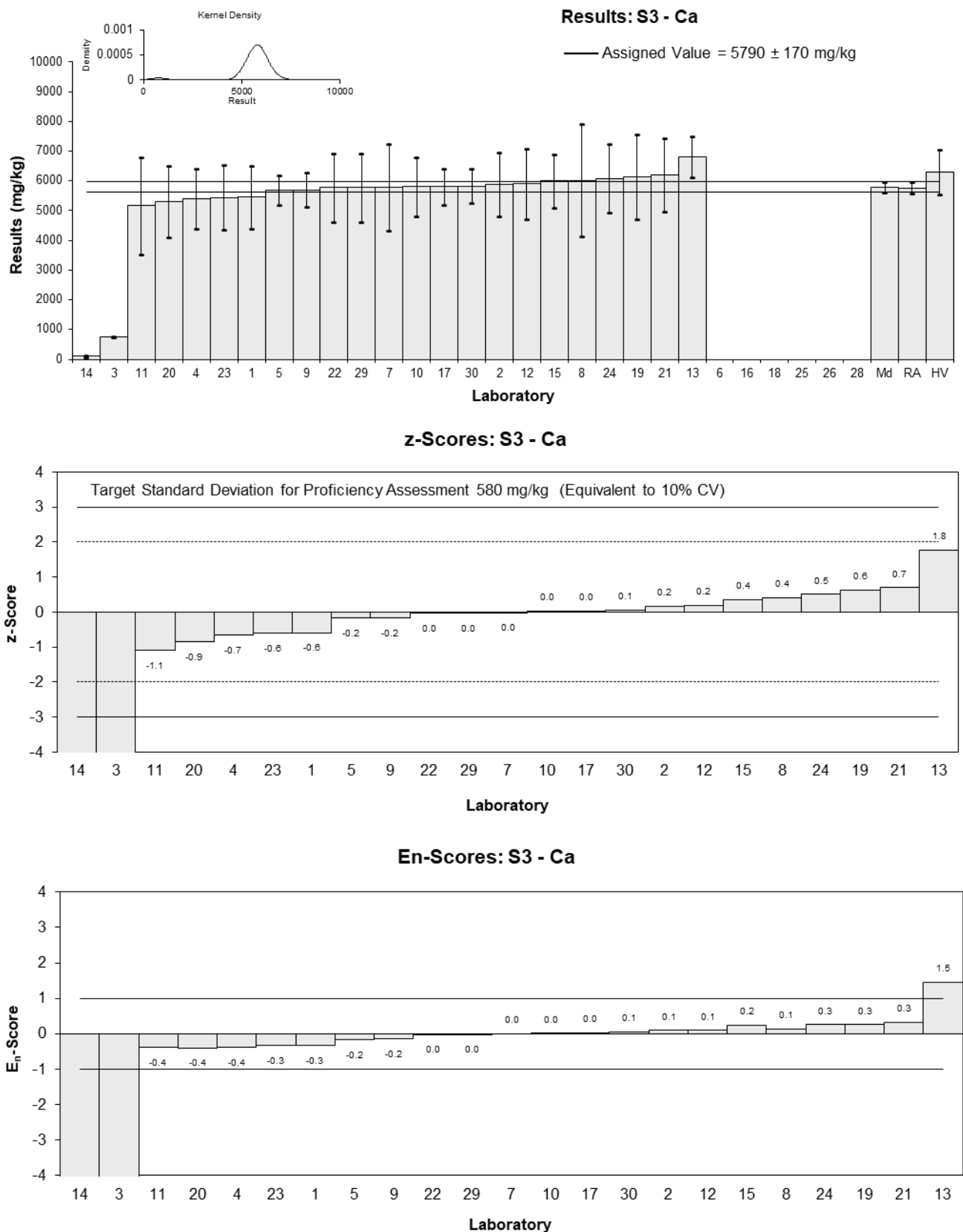


Figure 39

Table 52

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	32500	5200	0.17	0.13
2	38351.6	3554	1.40	1.45
3	34388.49	1410.14	0.57	0.83
4	24000	5000	-1.62	-1.33
5	27900	1300	-0.80	-1.20
6	NT	NT		
7	35767.00	8941.75	0.86	0.43
8	21800	2490	-2.08	-2.59
9	34400	3400	0.57	0.60
10	26000	5000	-1.20	-0.99
11**	250000	48800	45.91	4.47
12	37000	11100	1.11	0.46
13	31134	3100	-0.12	-0.13
14**	4.489	1.3467	-6.67	-10.93
15	29000	4400	-0.57	-0.51
16	NR	NR		
17	34941	3000	0.68	0.78
18	32054	6030	0.07	0.05
19	26600	11200	-1.07	-0.44
20	34000	6000	0.48	0.35
21	34400	6880	0.57	0.36
22	36440	7300	1.00	0.60
23	32500	6500	0.17	0.11
24	29500	10266	-0.46	-0.21
25	NT	NT		
26	NT	NT		
28	NT	NT		
29	39900	7980	1.72	0.97
30	23500	2400	-1.72	-2.18

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	31700	2900
Homogeneity Value	36800	4400
Robust Average	31700	2900
Median	32500	2700
Mean	31600	
N	22	
Max	39900	
Min	21800	
Robust SD	5500	
Robust CV	17%	

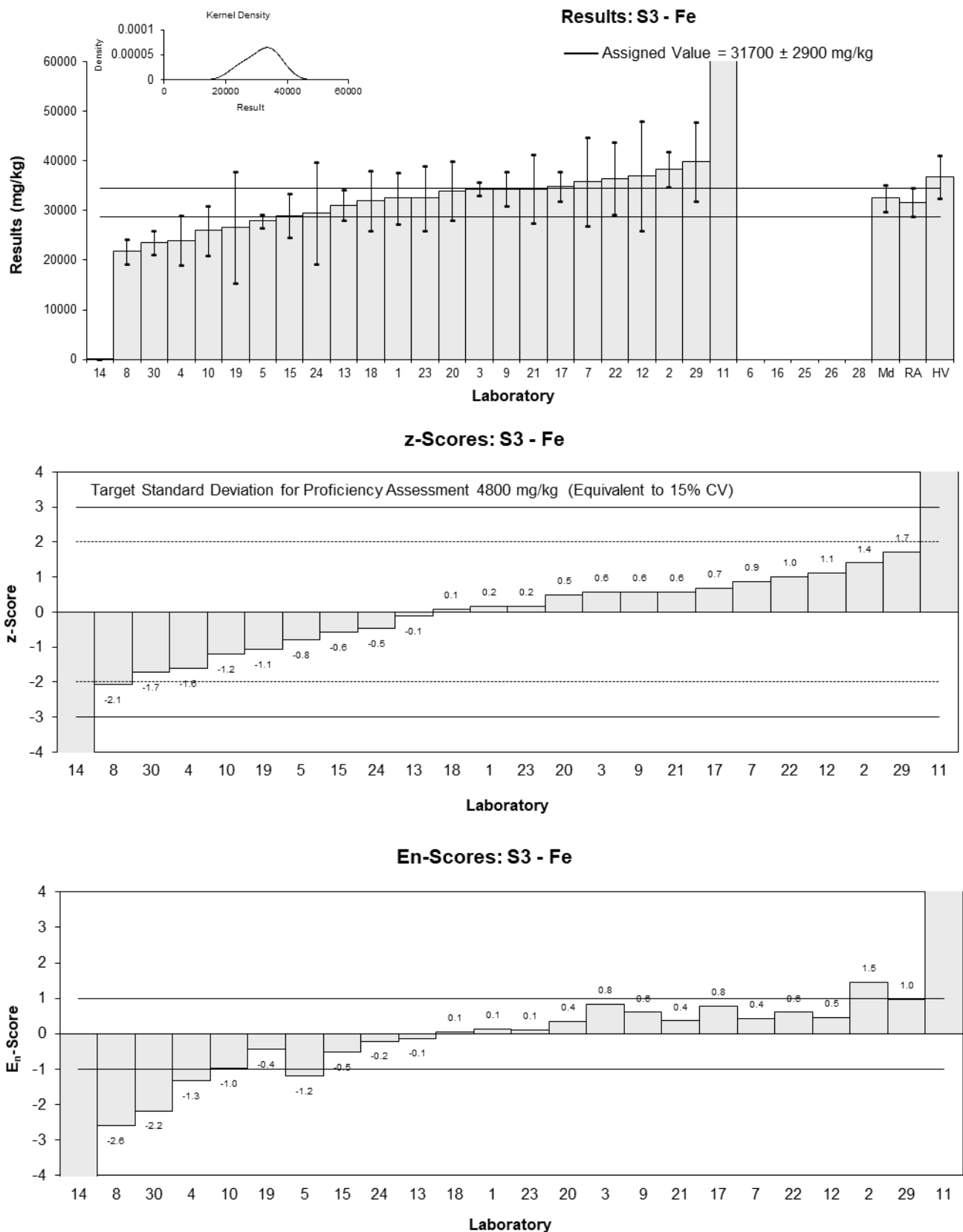


Figure 40

Table 53

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	1870	470	-0.11	-0.06
2	2048.8	277	0.52	0.49
3	2156.65	39.46	0.90	2.03
4	1400	300	-1.75	-1.55
5	1770	300	-0.46	-0.40
6	NT	NT		
7	2062.79	515.70	0.57	0.31
8	1440	262	-1.61	-1.60
9	2040	200	0.49	0.60
10	1900	300	0.00	0.00
11	1940	198	0.14	0.17
12	2400	960	1.75	0.52
13	1732	170	-0.59	-0.81
14**	18.248	5.4744	-6.60	-15.66
15	2100	315	0.70	0.59
16	NR	NR		
17	1917	200	0.06	0.07
18	NT	NT		
19	1670	300	-0.81	-0.71
20	2000	500	0.35	0.19
21	1900	380	0.00	0.00
22	1980	390	0.28	0.20
23	1860	370	-0.14	-0.10
24	1800	294	-0.35	-0.31
25	NT	NT		
26	NT	NT		
28	NT	NT		
29	2160	432	0.91	0.58
30	1490	150	-1.44	-2.13

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	1900	120
Homogeneity Value	2240	270
Robust Average	1900	120
Median	1910	110
Mean	1890	
N	22	
Max	2400	
Min	1400	
Robust SD	220	
Robust CV	12%	

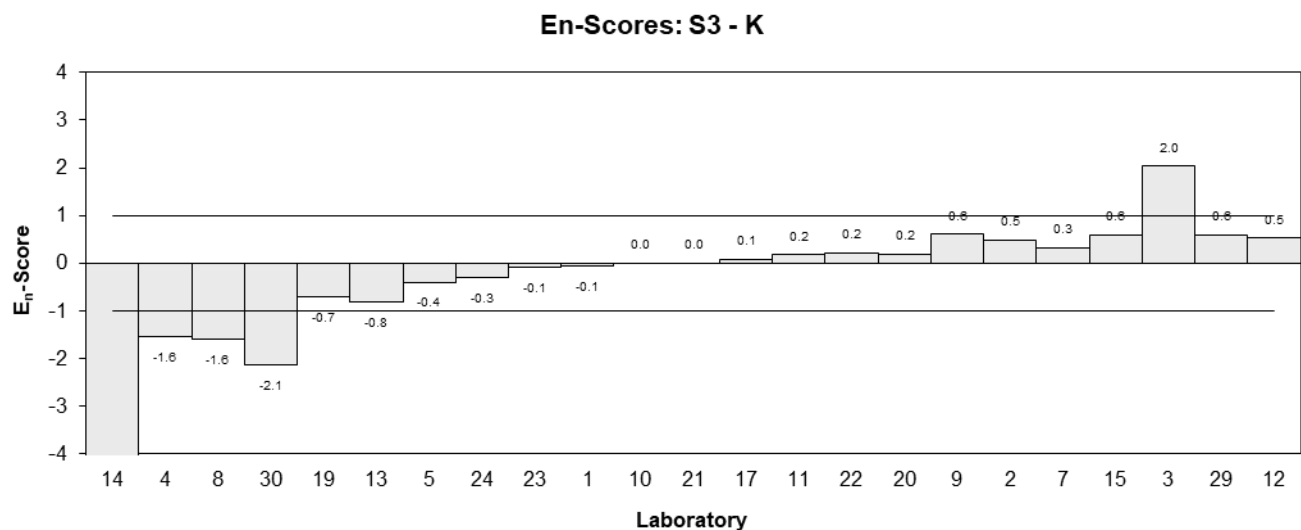
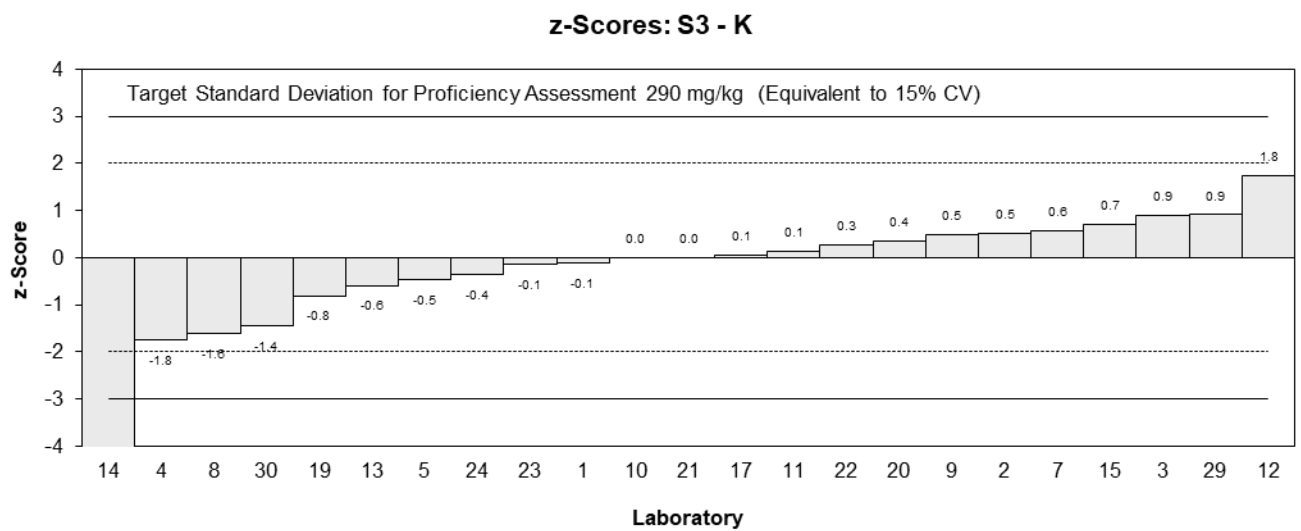
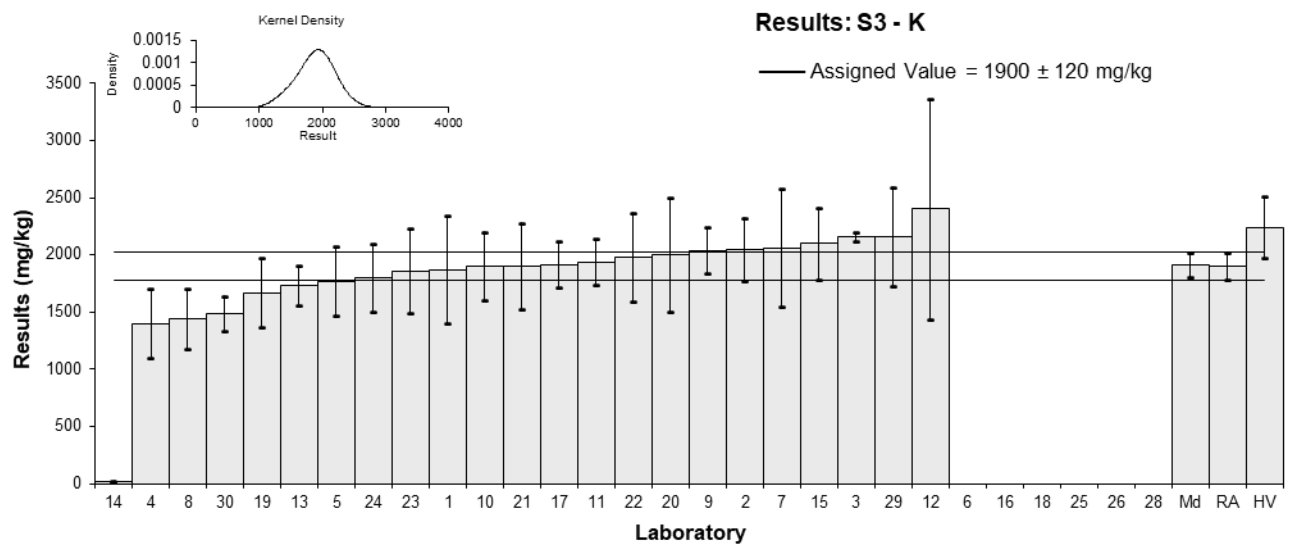


Figure 41

Table 54

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	5520	980	-0.10	-0.08
2	6331.2	1056	0.87	0.65
3	3719.86	63.36	-2.24	-5.01
4	4700	1000	-1.07	-0.84
5	5200	1500	-0.48	-0.26
6	NT	NT		
7	6359.43	1589.86	0.90	0.47
8	4680	1090	-1.10	-0.80
9	5800	580	0.24	0.29
10	5600	1000	0.00	0.00
11	5640	1770	0.05	0.02
12	6600	1320	1.19	0.73
13	5775	580	0.21	0.25
14**	50.368	15.1104	-6.61	-14.99
15	5700	855	0.12	0.11
16	NR	NR		
17	6039	600	0.52	0.62
18	NT	NT		
19	4950	960	-0.77	-0.63
20	5600	1200	0.00	0.00
21	6170	1234	0.68	0.44
22	5650	1130	0.06	0.04
23	5540	1110	-0.07	-0.05
24	5590	1026	-0.01	-0.01
25	NT	NT		
26	NT	NT		
28	NT	NT		
29	6530	1306	1.11	0.69
30	4650	470	-1.13	-1.59

** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	5600	370
Homogeneity Value	6150	740
Robust Average	5600	370
Median	5620	330
Mean	5560	
N	22	
Max	6600	
Min	3719.86	
Robust SD	690	
Robust CV	12%	

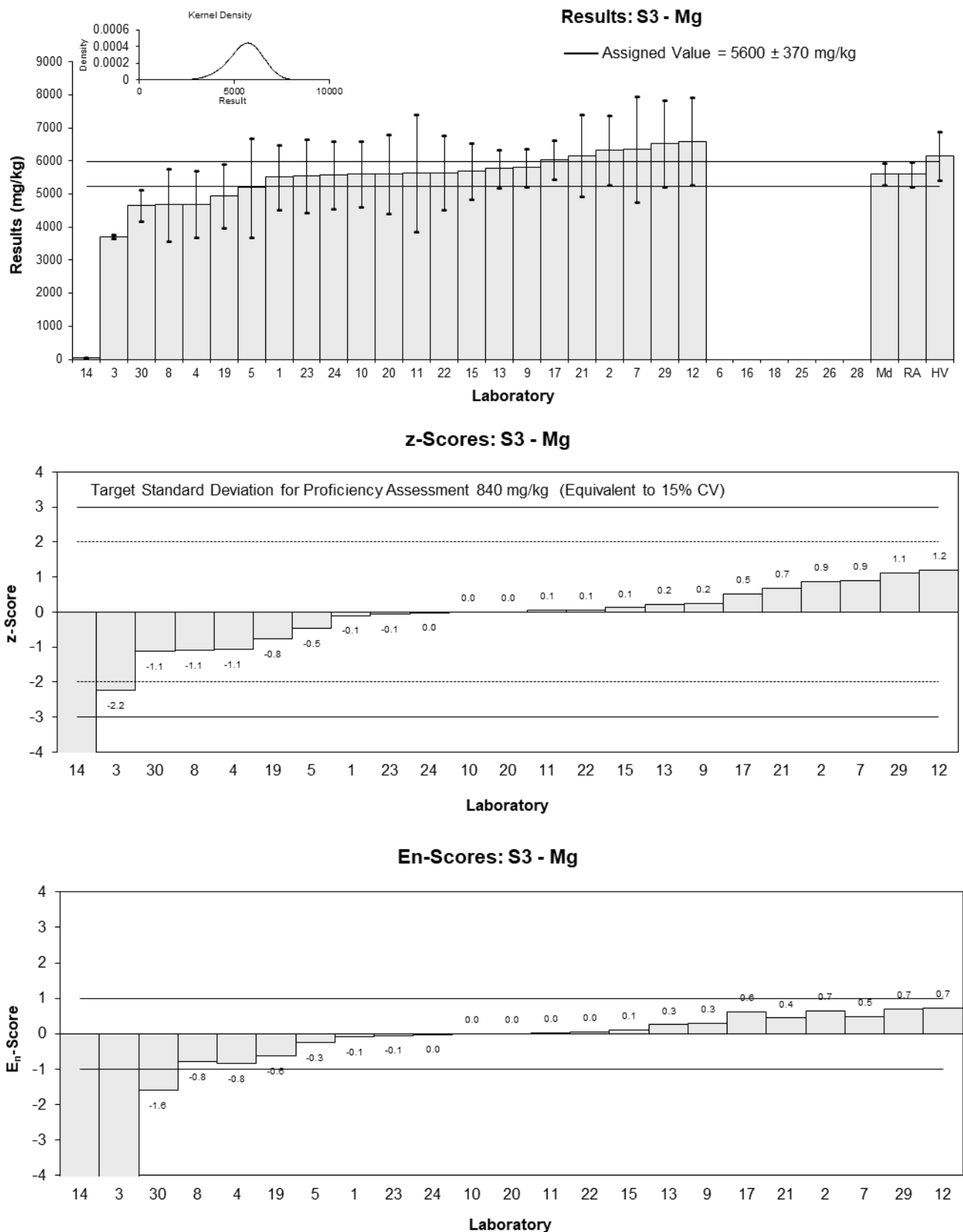


Figure 42

Table 55

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	1130	170	0.37	0.23
2	1130.6	189	0.37	0.21
3*	3019.17	266.86	17.70	7.15
4	1100	300	0.09	0.03
5	1000	160	-0.83	-0.55
6	NT	NT		
7	1064	266	-0.24	-0.10
8	1170	234	0.73	0.34
9	1070	110	-0.18	-0.17
10	1200	200	1.01	0.54
11	1090	133	0.00	0.00
12	1100	660	0.09	0.02
13	1038	100	-0.48	-0.48
14*	493.208	147.9624	-5.48	-3.89
15	1000	150	-0.83	-0.58
16	NR	NR		
17	1074	100	-0.15	-0.15
18	NT	NT		
19	1170	53	0.73	1.20
20	1200	300	1.01	0.36
21	1100	220	0.09	0.04
22	1010	200	-0.73	-0.39
23	1160	230	0.64	0.30
24	1050	156	-0.37	-0.25
25	NT	NT		
26	NT	NT		
28	NT	NT		
29	991	198	-0.91	-0.49
30	1090	110	0.00	0.00

* Outlier, see Section 4.2

Statistics

Assigned Value	1090	40
Homogeneity Value	1180	140
Robust Average	1090	40
Median	1090	40
Mean	1150	
N	23	
Max	3019.17	
Min	493.208	
Robust SD	81	
Robust CV	7.4%	

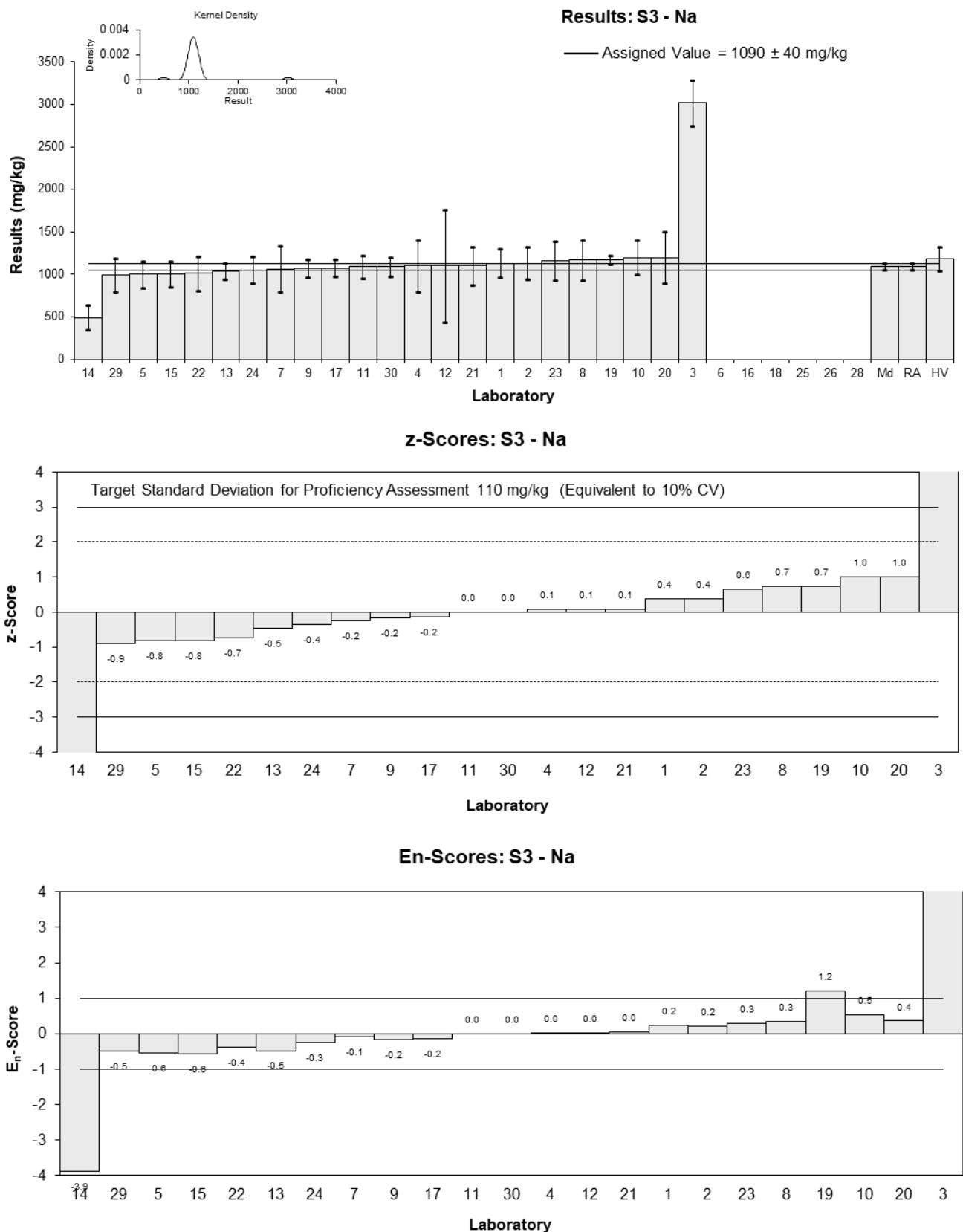


Figure 43

Table 56

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	210	34	-0.14	-0.08
2	217.5	25.08	0.21	0.16
3*	460.05	22.38	11.60	9.73
4	190	50	-1.08	-0.45
5	230	40	0.80	0.41
6	NT	NT		
7	230.41	57.60	0.82	0.30
8	150	38	-2.96	-1.58
9	210	20	-0.14	-0.13
10	210	60	-0.14	-0.05
11	205	26	-0.38	-0.28
12	NT	NT		
13	252	25	1.83	1.41
14**	2.363	0.7089	-9.89	-17.52
15	140	21	-3.43	-3.02
16	NR	NR		
17	225.6	20	0.59	0.54
18	214	41	0.05	0.02
19	200	40	-0.61	-0.31
20	220	50	0.33	0.14
21	250	50	1.74	0.72
22	226	45	0.61	0.28
23	202	40	-0.52	-0.26
24	200	NR	-0.61	-1.08
25	NT	NT		
26	NT	NT		
28	NT	NT		
29	235	470	1.03	0.05
30	189	19	-1.13	-1.07

* Outlier, ** Extreme Outlier, see Section 4.2

Statistics

Assigned Value	213	12
Homogeneity Value	221	27
Robust Average	215	13
Median	212	11
Mean	221	
N	22	
Max	460.05	
Min	140	
Robust SD	25	
Robust CV	11%	

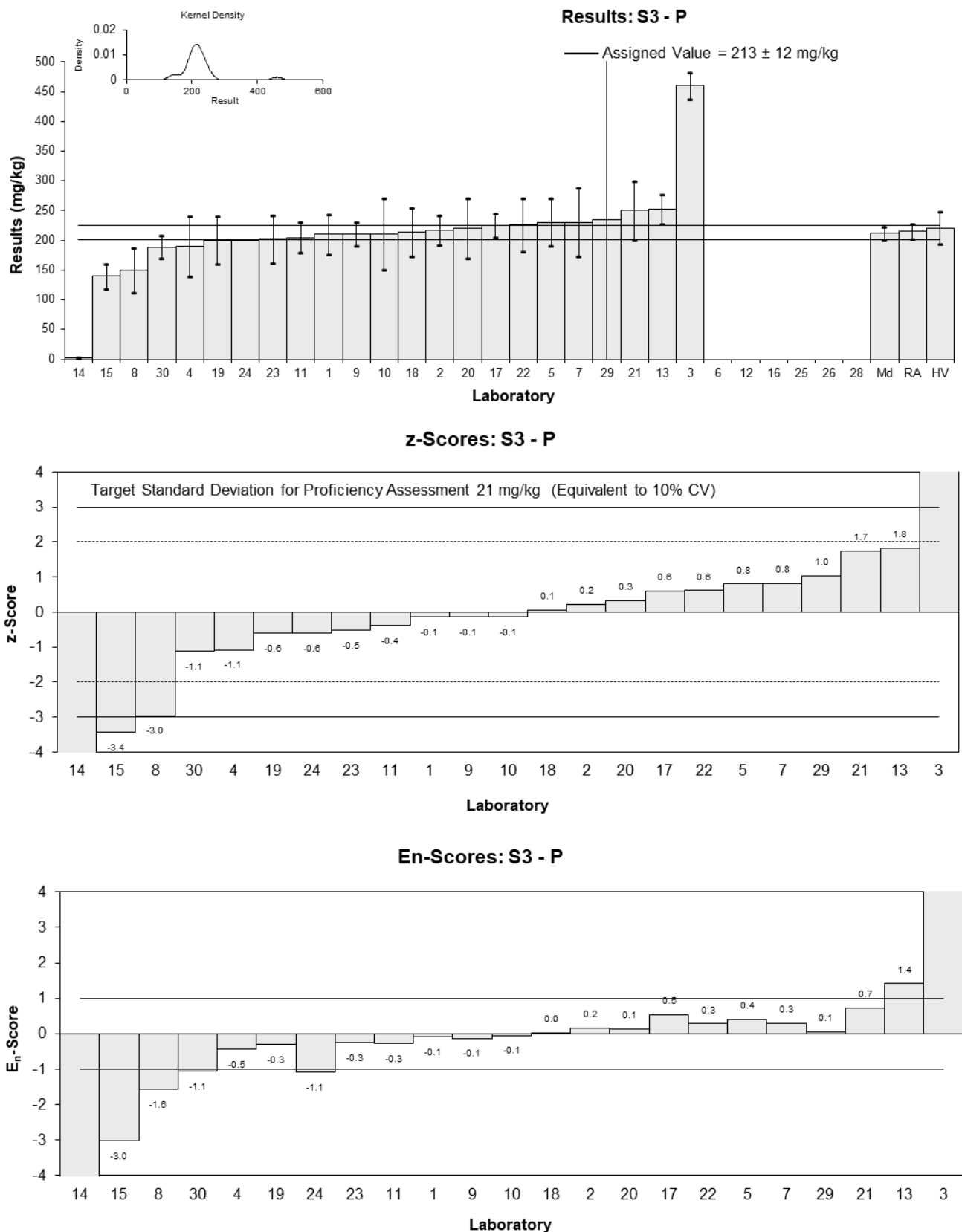


Figure 44

Table 57

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	155	30	0.32	0.21
2	147.86	19.66	-0.01	-0.01
3	NT	NT		
4	140	40	-0.36	-0.19
5	140	8	-0.36	-0.47
6	NT	NT		
7	< 200	NR		
8	120	38	-1.26	-0.69
9	220	20	3.24	2.88
10	150	60	0.09	0.03
11	119	28	-1.31	-0.91
12*	470	94	14.50	3.38
13	123	15	-1.13	-1.18
14*	33.961	10.1883	-5.14	-6.29
15*	240	36	4.14	2.36
16	NR	NR		
17	NT	NT		
18	NT	NT		
19	140	17	-0.36	-0.35
20	150	40	0.09	0.05
21	NR	NR		
22	155	31	0.32	0.20
23	175	35	1.22	0.71
24	140	NR	-0.36	-0.53
25	NT	NT		
26	NT	NT		
28	NT	NT		
29	NT	NT		
30	222	22	3.33	2.78

* Outlier, see Section 4.2

Statistics

Assigned Value	148	15
Robust Average	158	27
Median	149	15
Mean	169	
N	18	
Max	470	
Min	33.961	
Robust SD	46	
Robust CV	29%	

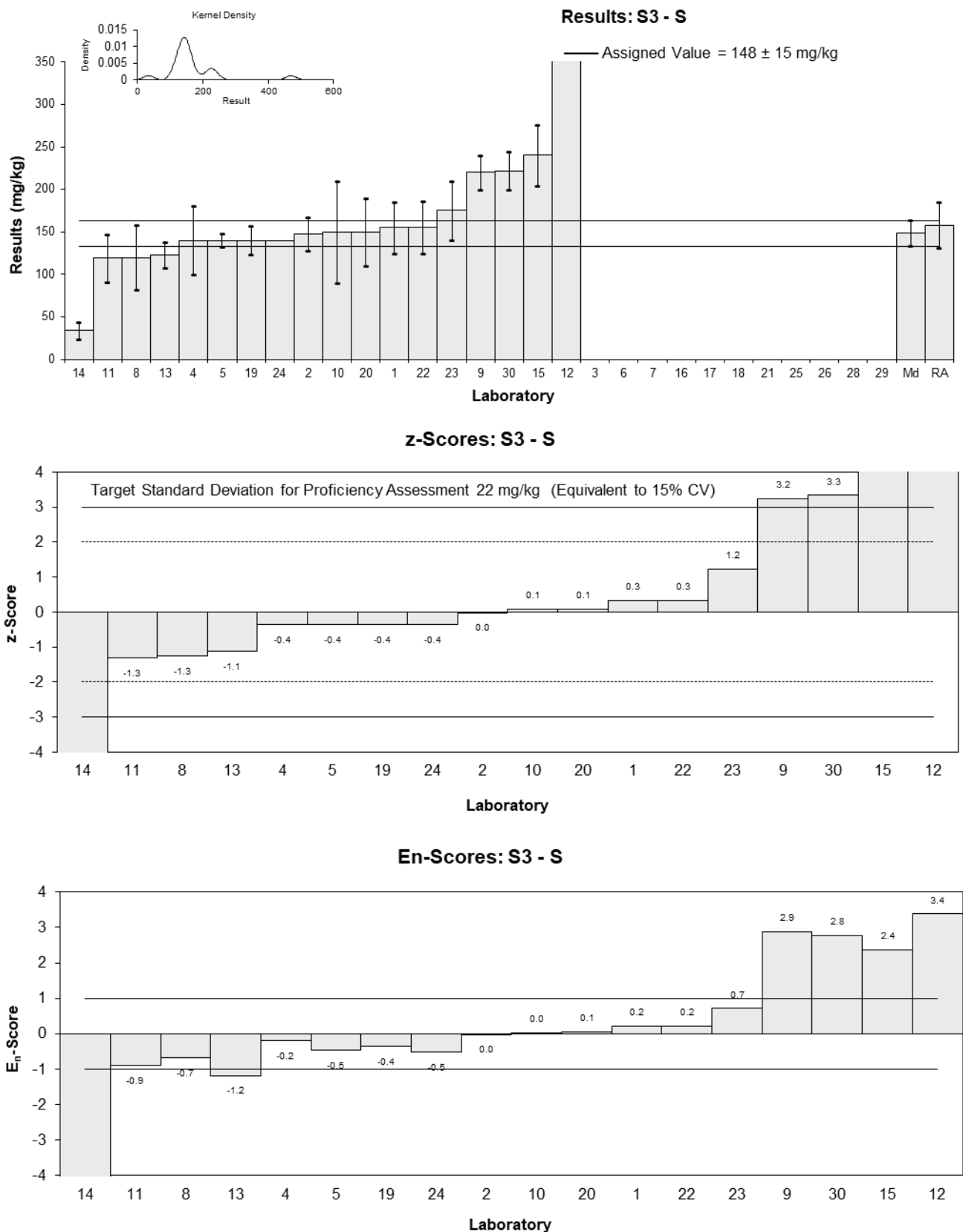


Figure 45

Table 58

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	NT	NT
2	NT	NT
3	NT	NT
4	<2.5	3
5	NT	NT
6	NT	NT
7	< 5	NR
8	2.08	0.21
9	NT	NT
10	<2.5	2.5
11	NR	NR
12	NT	NT
13	NT	NT
14	2.44	0.732
15	NT	NT
16	NR	NR
17	NT	NT
18	< 5	NR
19	NT	NT
20	<2.5	NR
21	2.6	0.52
22	NR	NR
23	2.7	0.54
24	2.07	0.19
25	NT	NT
26	NT	NT
28	<5	5
29	NT	NT
30	NT	NT

Statistics

Assigned Value	Not Set	
Homogeneity Value	3.00	0.45
Robust Average	NA (N<6)	
Median	2.44	0.43
Mean	2.38	
N	5	
Max	2.7	
Min	2.07	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

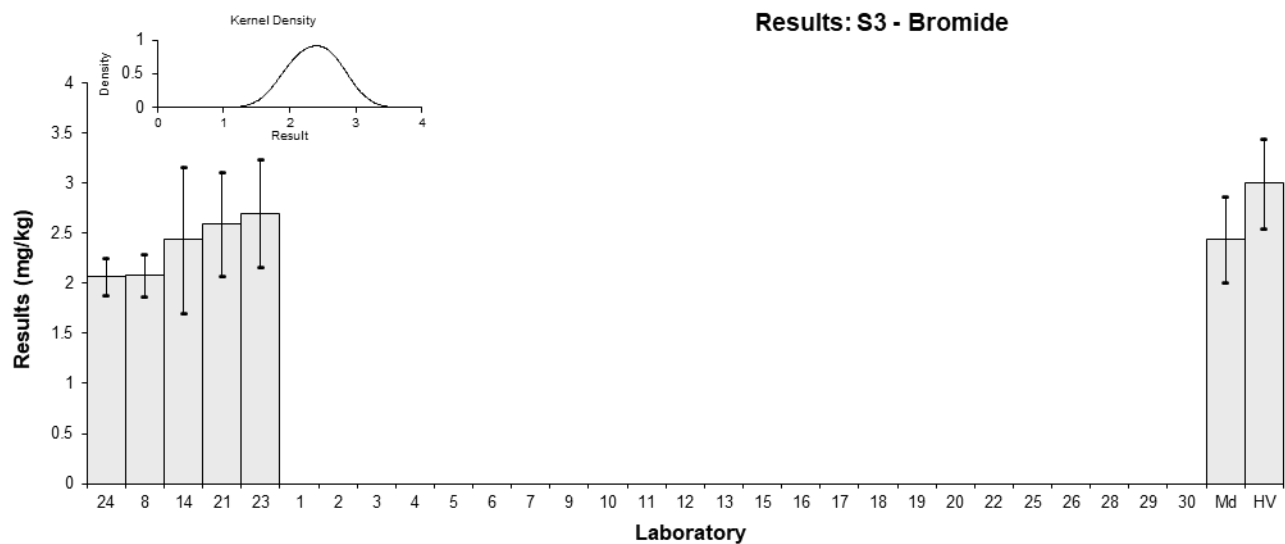


Figure 46

Table 59

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	500	63	0.14	0.11
2	480.2	58.2	-0.26	-0.21
3*	743.8	1.8	5.09	13.14
4	510	100	0.34	0.17
5	486	46	-0.14	-0.14
6	NT	NT		
7	498	149	0.10	0.03
8	510	47	0.34	0.34
9	NT	NT		
10	490	150	-0.06	-0.02
11	480	96	-0.26	-0.13
12*	120	7.2	-7.57	-18.36
13	345	35	-3.00	-3.72
14	530.9	159.27	0.77	0.24
15	440	88	-1.08	-0.59
16	469.9	NR	-0.47	-1.22
17	NT	NT		
18	483	71.5	-0.20	-0.14
19	530	70	0.75	0.51
20	510	100	0.34	0.17
21	460	92	-0.67	-0.35
22	NR	NR		
23	530	80	0.75	0.45
24	556	55.6	1.28	1.07
25	NT	NT		
26	NT	NT		
28	471	47	-0.45	-0.43
29	NT	NT		
30	NT	NT		

* Outlier, see Section 4.2

Statistics

Assigned Value	493	19
Homogeneity Value	427	64
Robust Average	493	22
Median	490	16
Mean	483	
N	21	
Max	743.8	
Min	120	
Robust SD	40	
Robust CV	8.1%	

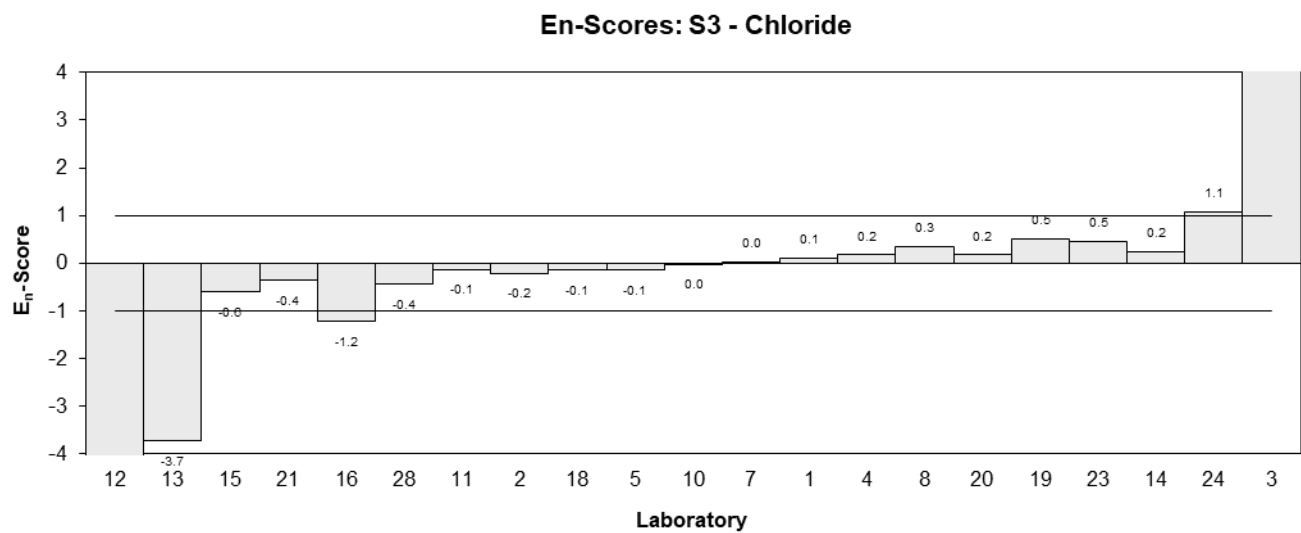
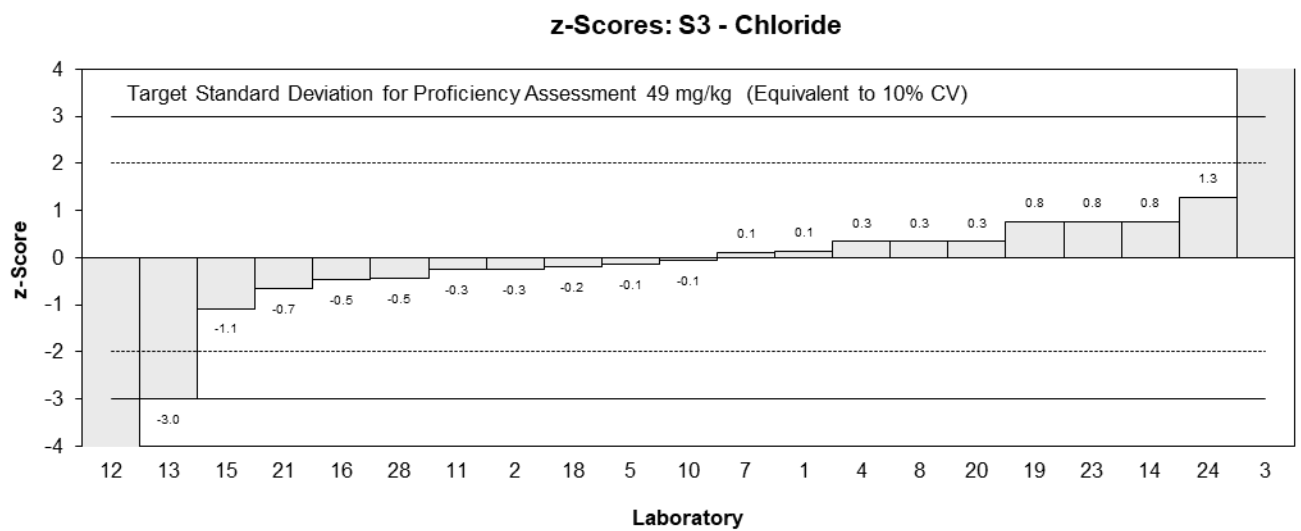
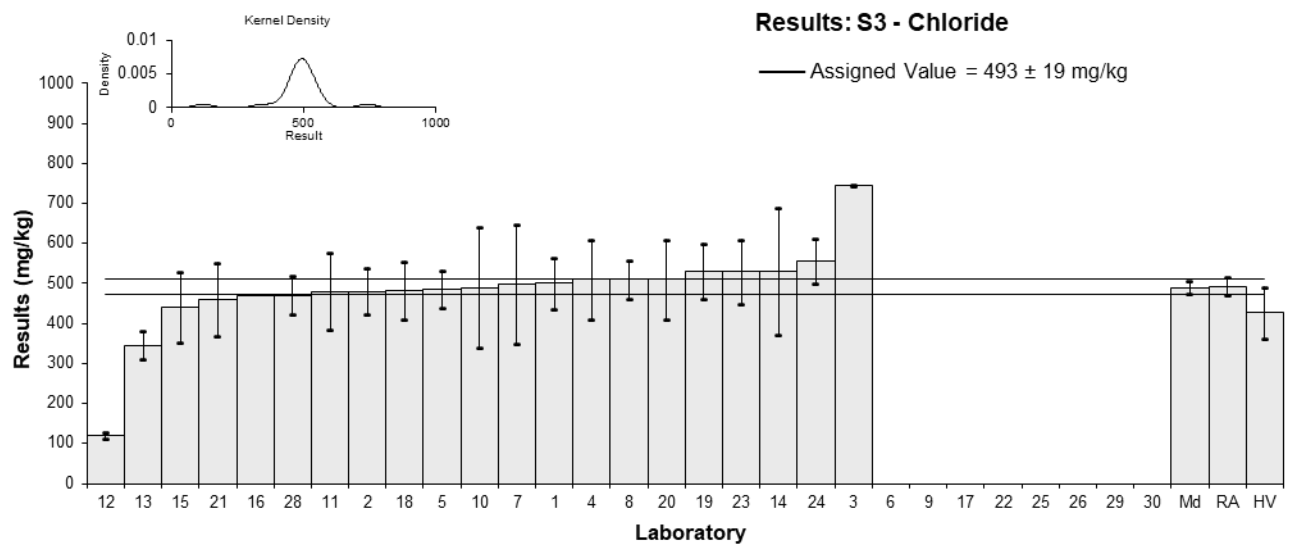


Figure 47

Table 60

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	1.6	0.65	-0.35	-0.17
2	1.225	0.071	-1.44	-1.77
3*	4.98	0.03	9.48	12.00
4	2	0.4	0.81	0.58
5	NT	NT		
6	NT	NT		
7	< 5	NR		
8	2	0.3	0.81	0.69
9	NT	NT		
10	2	1	0.81	0.27
11	<5	NR		
12	NT	NT		
13	1.15	0.2	-1.66	-1.70
14	1.84	0.552	0.35	0.20
15	NT	NT		
16	NR	NR		
17	NT	NT		
18	NT	NT		
19	2	0.34	0.81	0.64
20	1.6	1	-0.35	-0.12
21	NR	NR		
22	NR	NR		
23	1.3	0.26	-1.22	-1.12
24	2	0.43	0.81	0.55
25	NT	NT		
26	NT	NT		
28	1.91	1	0.55	0.18
29	NT	NT		
30	NT	NT		

* Outlier, see Section 4.2

Statistics

Assigned Value	1.72	0.27
Robust Average	1.77	0.29
Median	1.91	0.09
Mean	1.97	
N	13	
Max	4.98	
Min	1.15	
Robust SD	0.42	
Robust CV	24%	

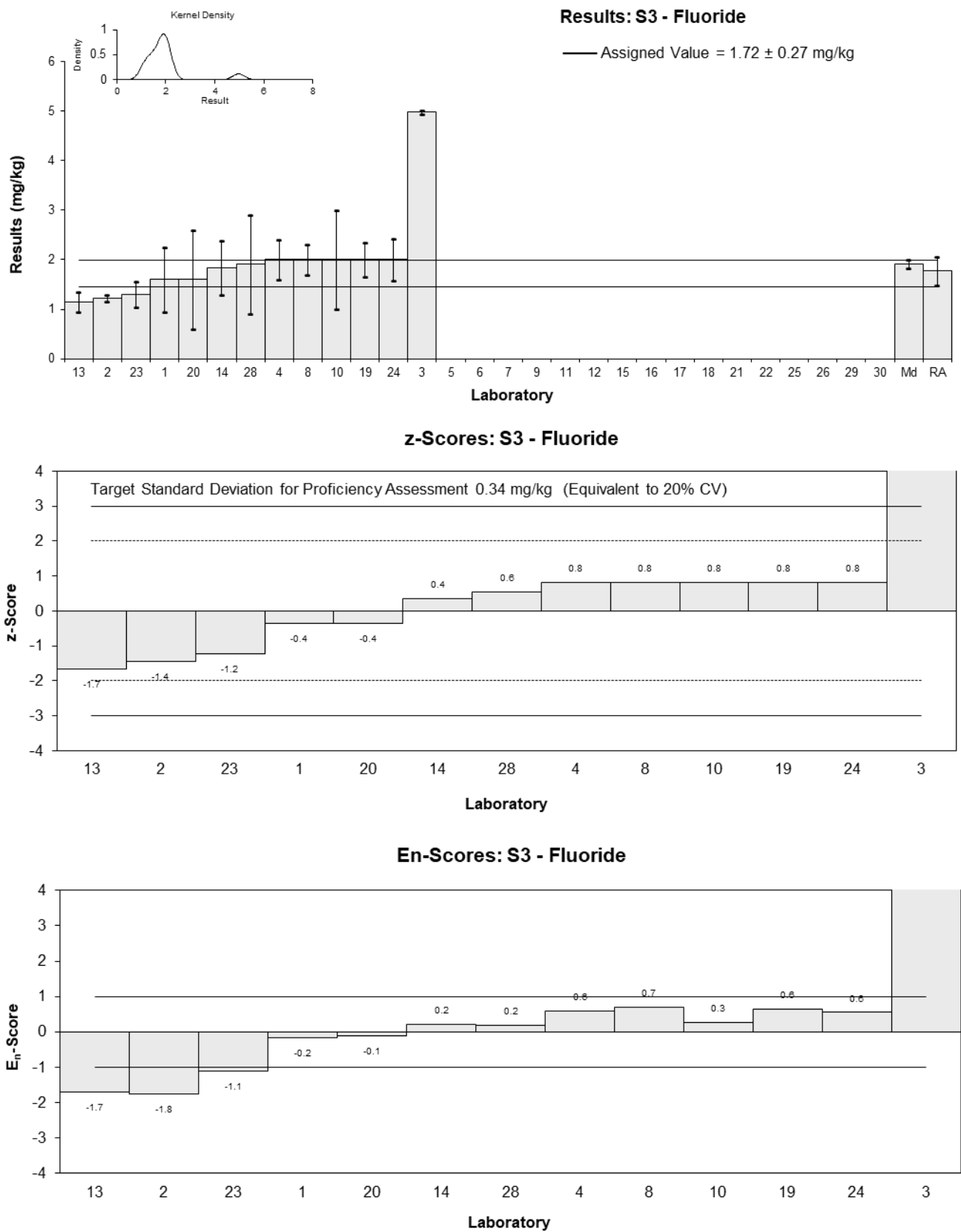


Figure 48

Table 61

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	NT	NT
2	NT	NT
3	NT	NT
4	<1	1
5	NT	NT
6	NT	NT
7	< 5	NR
8	1.52	0.15
9	NT	NT
10	NT	NT
11	1.1	0.2
12	NT	NT
13	NT	NT
14	1.32	0.396
15	NT	NT
16	NR	NR
17	NT	NT
18	< 5	NR
19	NT	NT
20	1	1
21	NR	NR
22	NR	NR
23	<1	0.2
24	1.32	0.214
25	NT	NT
26	NT	NT
28	<5	5
29	NT	NT
30	NT	NT

Statistics

Assigned Value	Not Set	0.33
Robust Average	NA (N<6)	
Median	1.32	
Mean	1.25	
N	5	
Max	1.52	
Min	1	
Robust SD	NA (N<6)	
Robust CV	NA (N<6)	

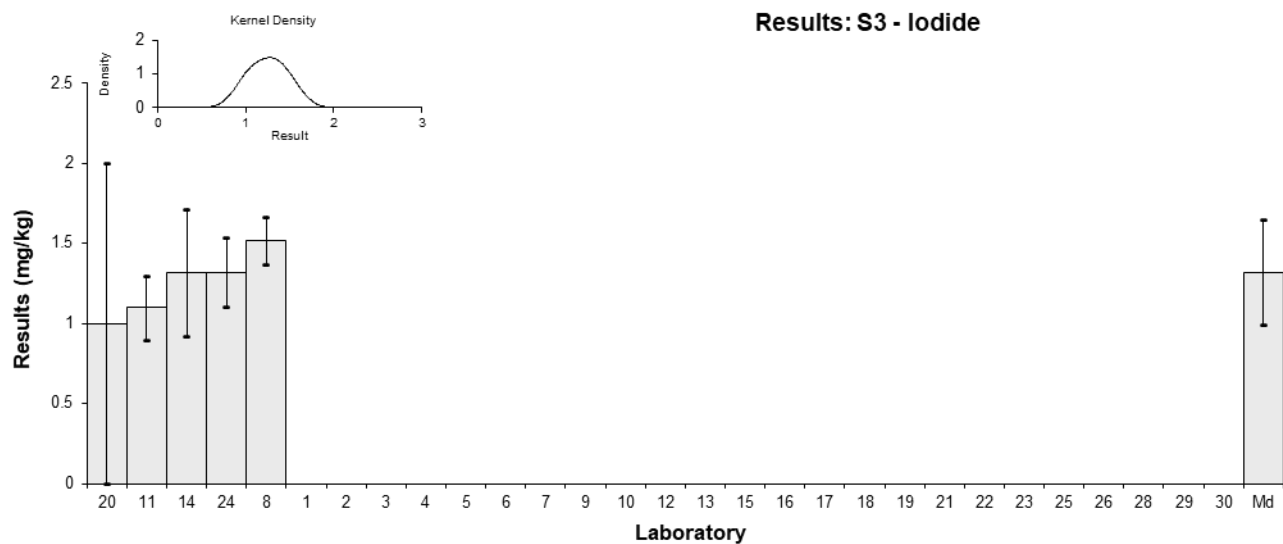


Figure 49

Table 62

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty
1	0.30	0.15
2	0.12	0.0147
3	0.33	0.02
4	<0.5	0.5
5	NT	NT
6	NT	NT
7	< 10	NR
8	0.1	0.02
9	NT	NT
10	<0.5	0.5
11	<0.2	NR
12	<5	NR
13	1.5	0.2
14	0.472	0.1416
15	< 1	NR
16	NR	NR
17	NT	NT
18	< 10	NR
19	0.1	0.05
20	<0.5	NR
21	<1	NR
22	NR	NR
23	<1	0.2
24	0.2	0.022
25	NT	NT
26	NT	NT
28	NT	NT
29	NT	NT
30	NT	NT

Statistics

Assigned Value	Not Set	
Robust Average	0.28	0.18
Median	0.25	0.18
Mean	0.390	
N	8	
Max	1.5	
Min	0.1	
Robust SD	0.20	
Robust CV	74%	

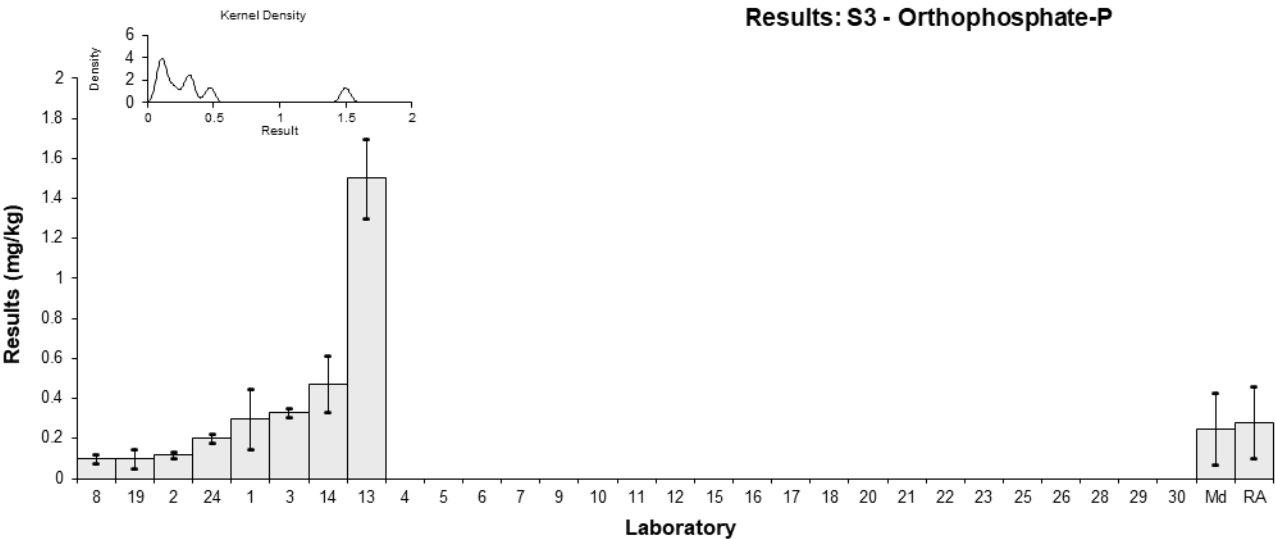


Figure 50

Table 63

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	96	18	0.22	0.18
2	97.896	NR	0.32	0.49
3	117.5	2.5	1.39	2.08
4	90	20	-0.11	-0.09
5	76	6	-0.87	-1.19
6	NT	NT		
7	< 30	NR		
8	110	12	0.98	1.06
9	NT	NT		
10	80	30	-0.65	-0.37
11	<50	NR		
12	NT	NT		
13*	35	3.5	-3.10	-4.56
14*	34	10.2	-3.15	-3.68
15	< 10	NR		
16	NR	NR		
17	NT	NT		
18	52.6	7.87	-2.14	-2.75
19	120	48	1.52	0.57
20	90	30	-0.11	-0.06
21	97	19.4	0.27	0.22
22	NR	NR		
23	88	18	-0.22	-0.18
24	90	13.7	-0.11	-0.11
25	NT	NT		
26	NT	NT		
28	76.3	7.6	-0.85	-1.11
29	NT	NT		
30	NT	NT		

* Outlier, see Section 4.2

Statistics

Assigned Value	92	12
Homogeneity Value	76	11
Robust Average	86	16
Median	90	11
Mean	84.4	
N	16	
Max	120	
Min	34	
Robust SD	25	
Robust CV	29%	

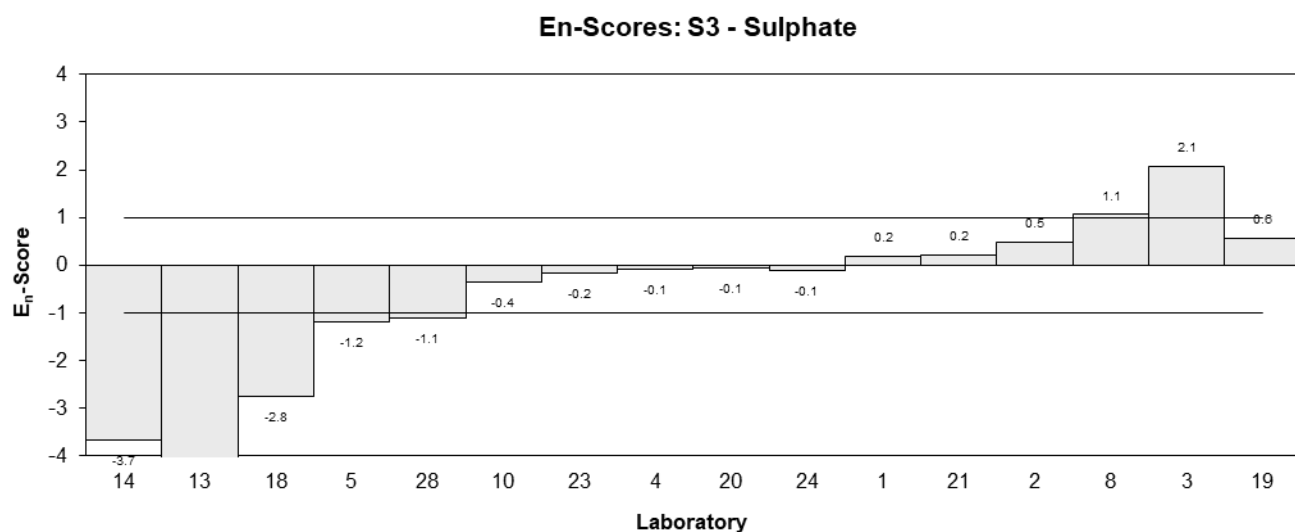
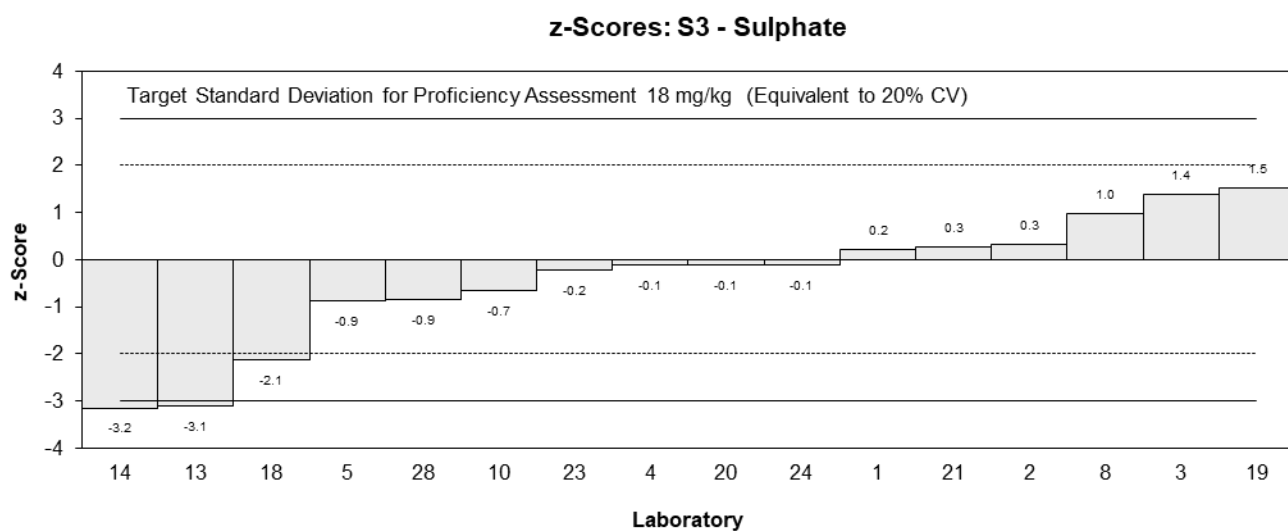
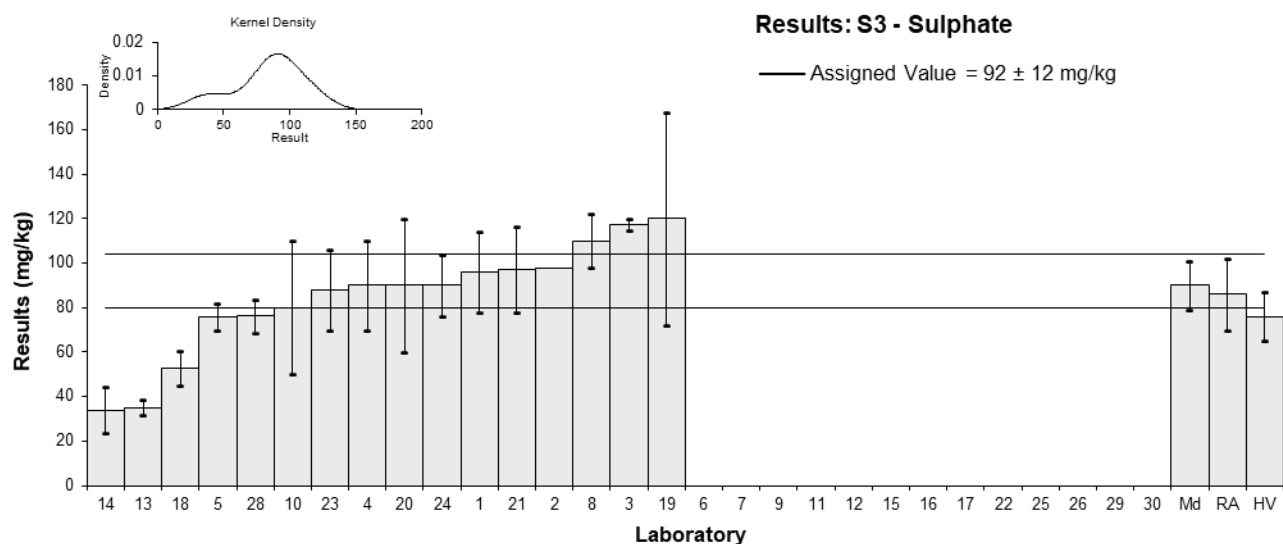


Figure 51

Table 64

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E_n
1	790	69	1.70	1.43
2	794	15.8	1.76	2.71
3	994	8	4.73	7.64
4	690	100	0.22	0.14
5	638	19	-0.55	-0.82
6	NT	NT		
7	595	179	-1.19	-0.44
8	659	15	-0.24	-0.37
9	676	60	0.01	0.01
10	680	200	0.07	0.02
11	540	54	-2.00	-1.99
12	740	7.4	0.96	1.56
13	876	87	2.98	2.09
14	625	187.5	-0.74	-0.26
15	609	122	-0.98	-0.51
16	595	22	-1.19	-1.72
17	610.30	60	-0.96	-0.89
18	683	68.3	0.12	0.10
19	714	17.5	0.58	0.87
20	650	150	-0.37	-0.16
21	695	139	0.30	0.14
22	NR	NR		
23	650	98	-0.37	-0.24
24	664	60.76	-0.16	-0.15
25	NT	NT		
26	NT	NT		
28	NT	NT		
29	622.8	34.88	-0.77	-0.97
30	NT	NT		

Statistics

Assigned Value	675	41
Homogeneity Value	658	33
Robust Average	675	41
Median	664	32
Mean	687	
N	23	
Max	994	
Min	540	
Robust SD	78	
Robust CV	12%	

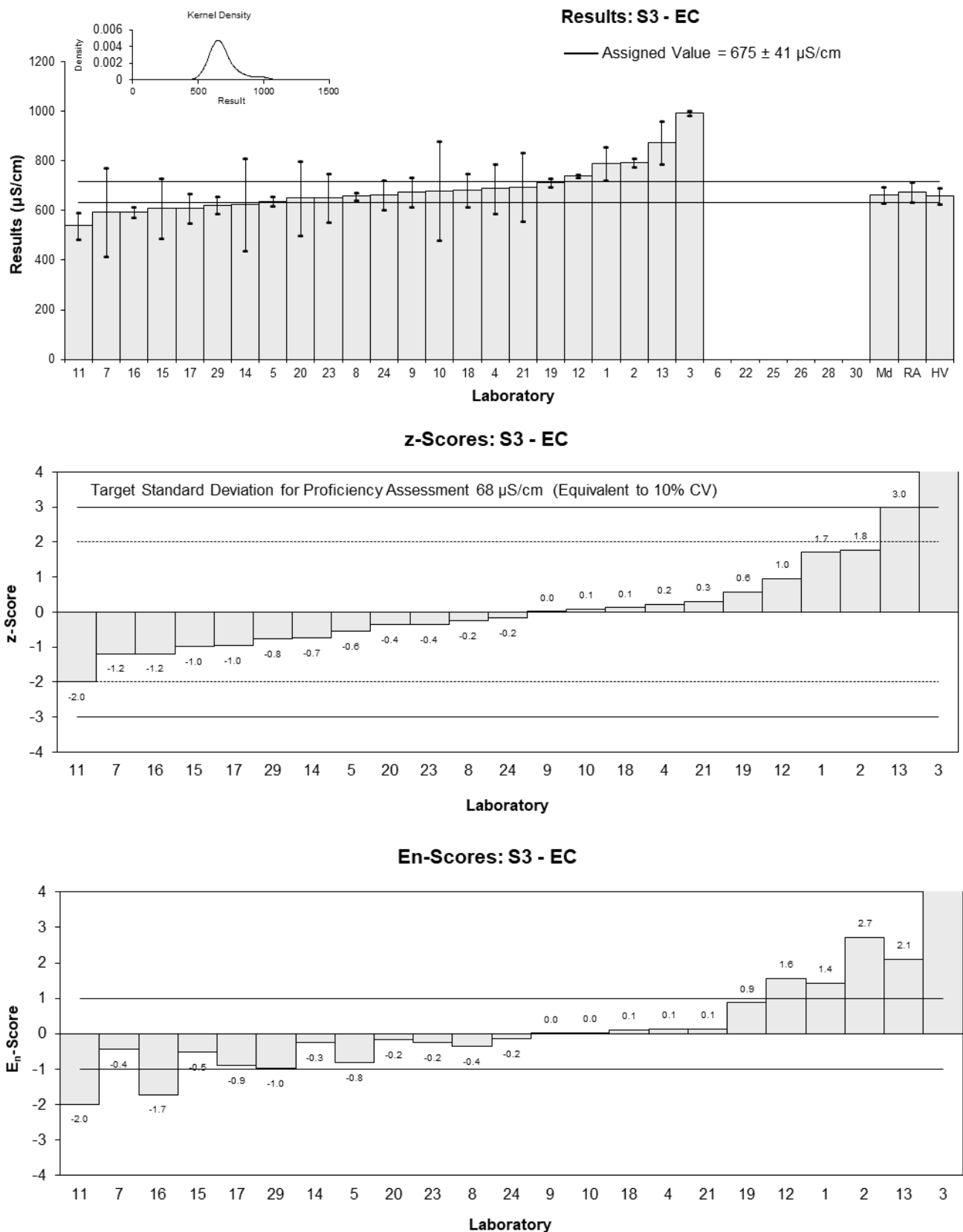


Figure 52

Table 65

Sample Details

Sample No.	S3
Matrix	Soil
Analyte	pH

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	7.4	0.2	-0.08	-0.09
2	7.14	0.06	-1.08	-2.23
3	7.48	0.27	0.23	0.21
4	7.3	0.2	-0.46	-0.53
5	7.47	0.15	0.19	0.27
6	NT	NT		
7	7.32	0.2	-0.39	-0.44
8	7.5	0.07	0.31	0.61
9	7.4	0.2	-0.08	-0.09
10	7.3	0.2	-0.46	-0.53
11	7.5	0.3	0.31	0.25
12	7.5	NR	0.31	0.73
13	4.28	0.2	-12.09	-13.76
14	NT	NT		
15	7.5	0.3	0.31	0.25
16	7.44	0.15	0.08	0.11
17	7.80	0.8	1.46	0.47
18	7.8	1.08	1.46	0.35
19	7.2	0.077	-0.85	-1.64
20	7.5	0.3	0.31	0.25
21	7.7	1.54	1.08	0.18
22	NR	NR		
23	7.4	1.5	-0.08	-0.01
24	7.5	0.209	0.31	0.34
25	NT	NT		
26	NT	NT		
28	NT	NT		
29	7.11	0.98	-1.19	-0.31
30	NT	NT		

Statistics

Assigned Value	7.42	0.11
Homogeneity Value	7.46	0.37
Robust Average	7.42	0.11
Median	7.46	0.04
Mean	7.30	
N	22	
Max	7.8	
Min	4.28	
Robust SD	0.20	
Robust CV	2.7%	

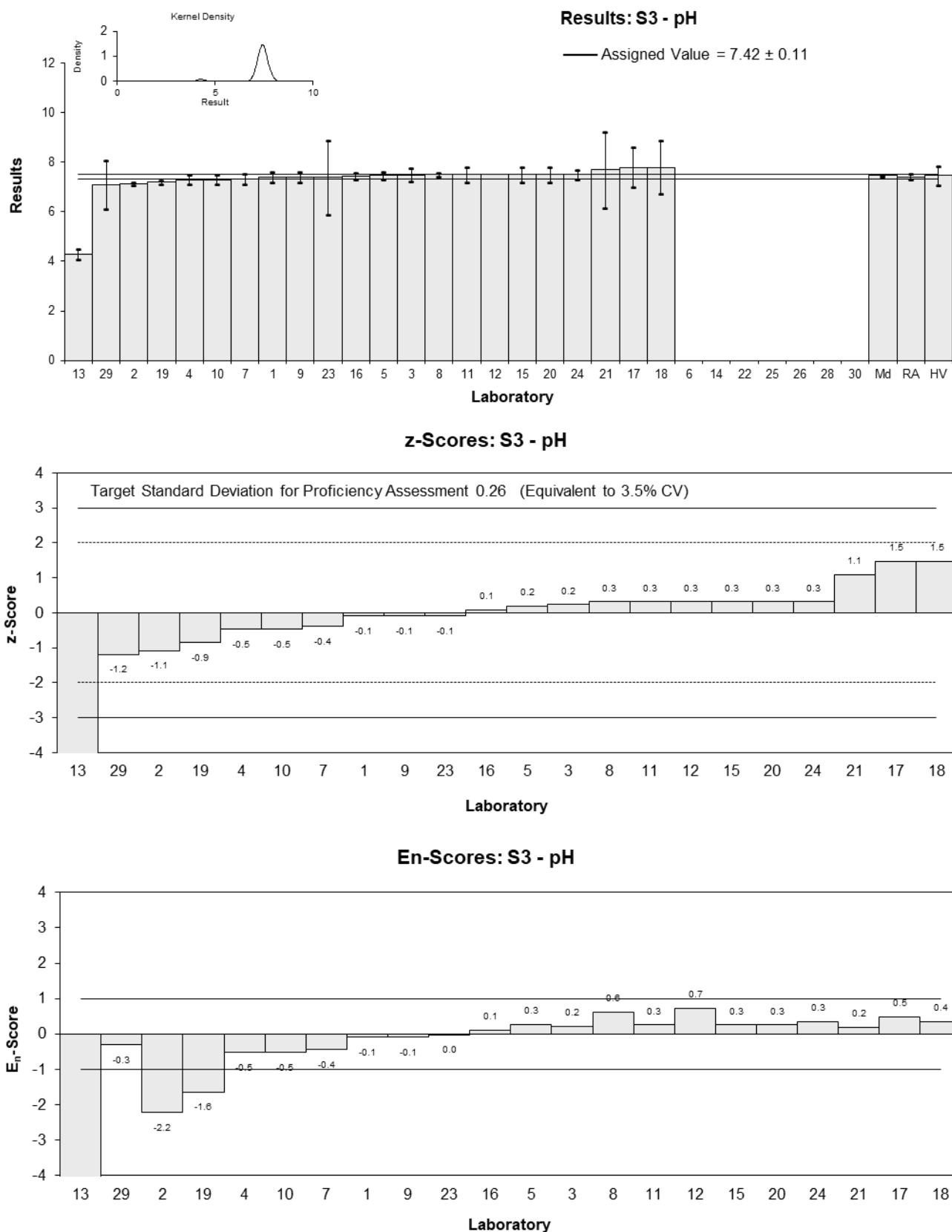


Figure 53

Table 66

Sample Details

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

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	1030	185	0.20	0.10
2	937.3	236.7	-0.72	-0.30
3	738.3	76.4	-2.69	-2.80
4	1000	300	-0.10	-0.03
5	NT	NT		
6	NT	NT		
7	1170	351	1.58	0.45
8	1080	192	0.69	0.35
9	860	90	-1.49	-1.39
10	1000	400	-0.10	-0.02
11	1000	200	-0.10	-0.05
12	NT	NT		
13	989	100	-0.21	-0.18
14	1138.2	341.46	1.27	0.37
15	1000	200	-0.10	-0.05
16	NR	NR		
17	1242.83	120	2.31	1.74
18	NT	NT		
19	950	314	-0.59	-0.19
20	1100	300	0.89	0.29
21	NR	NR		
22	1093	164	0.82	0.48
23	960	140	-0.50	-0.33
24	920	248	-0.89	-0.35
25	NT	NT		
26	NT	NT		
28	NT	NT		
29	NT	NT		
30	NT	NT		

Statistics

Assigned Value	1010	60
Homogeneity Value	960	140
Robust Average	1010	60
Median	1000	62
Mean	1010	
N	18	
Max	1242.83	
Min	738.3	
Robust SD	110	
Robust CV	11%	

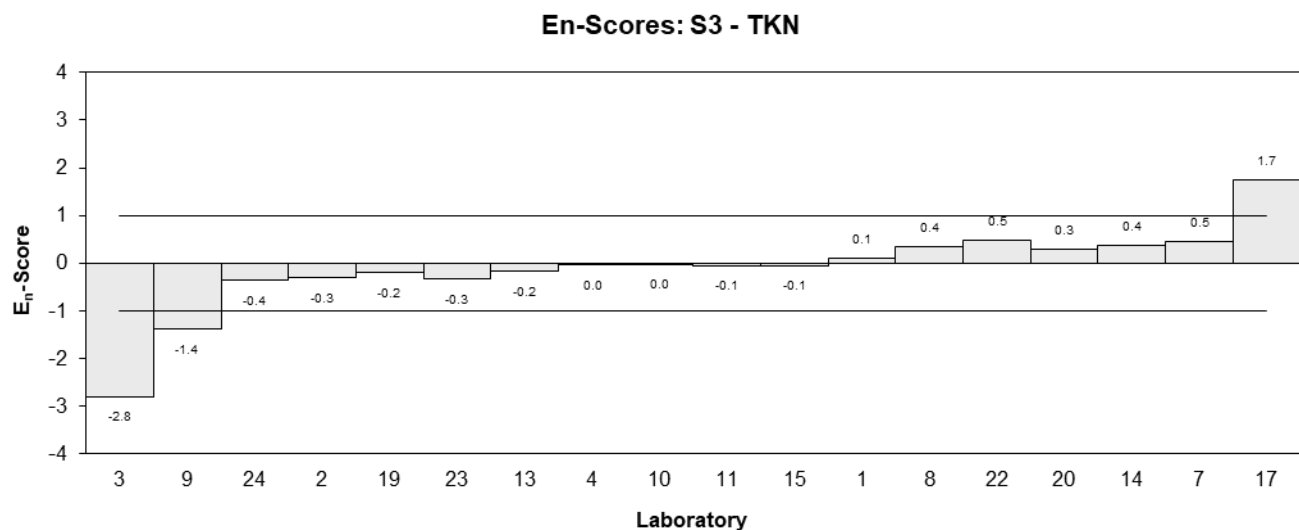
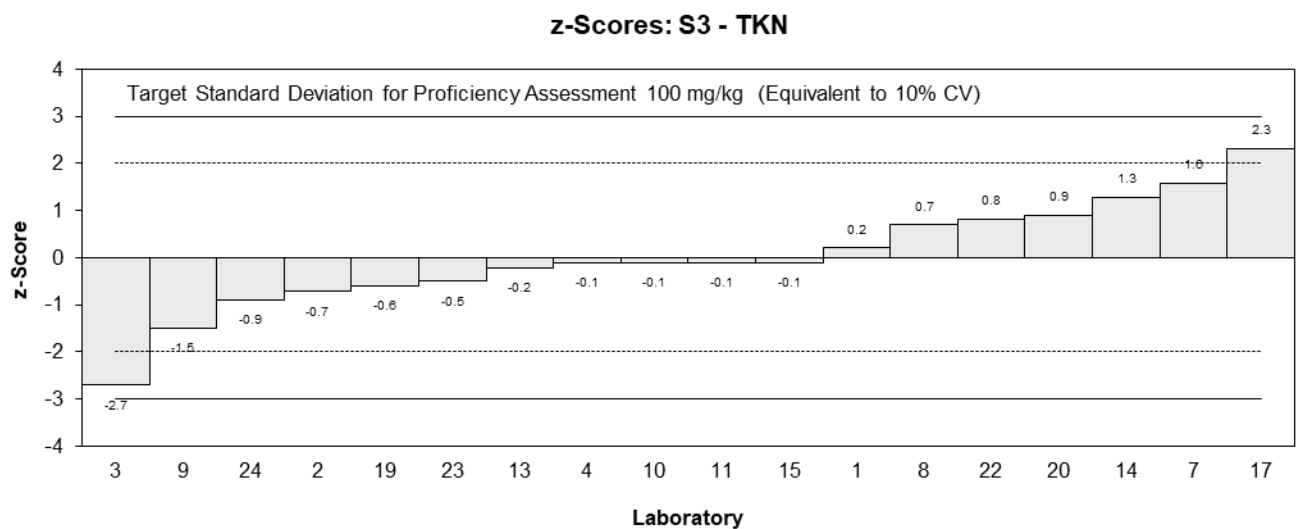
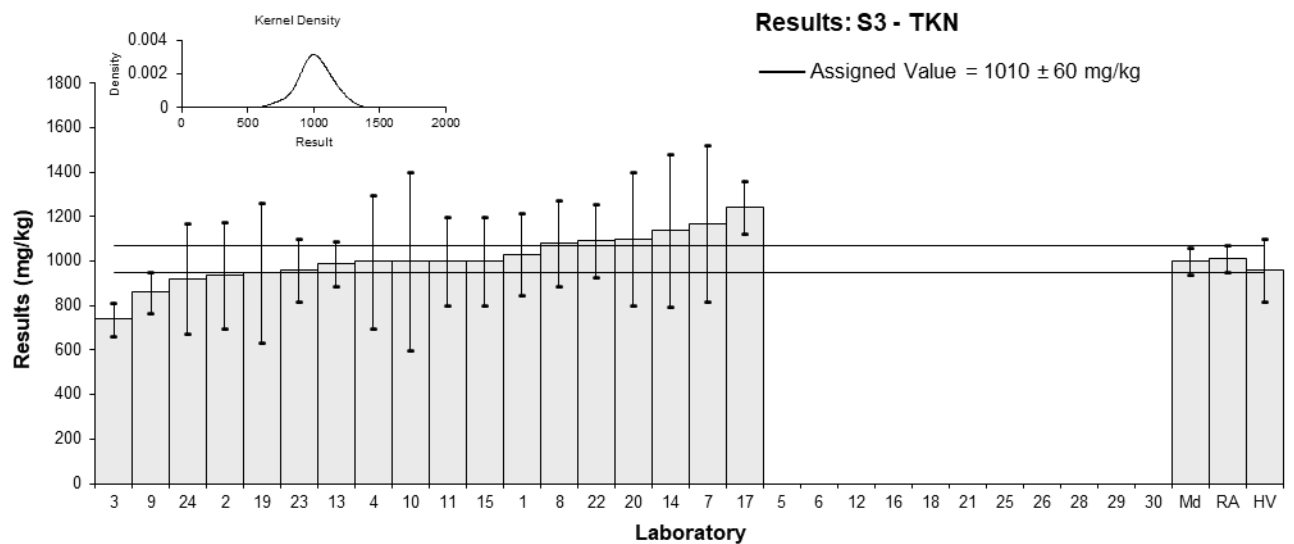


Figure 54

Table 67

Sample Details

Sample No.	S3
Matrix	Soil
Analyte	Ammonium-N
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	12.1	2.5	0.00	0.00
2	NT	NT		
3*	27.5	0.4	8.48	10.58
4	11	2	-0.61	-0.45
5	NT	NT		
6	NT	NT		
7	NT	NT		
8	NT	NT		
9	NT	NT		
10	12	5	-0.06	-0.02
11	NR	NR		
12	NT	NT		
13	12.9	1.3	0.44	0.42
14	15.27	4.581	1.75	0.66
15	NT	NT		
16	11.7	2.36	-0.22	-0.15
17	NT	NT		
18	NT	NT		
19	NR	NR		
20	9.4	4	-1.49	-0.64
21	NR	NR		
22	12.8	3.0	0.39	0.21
23	12	2.4	-0.06	-0.04
24	14.4	1.48	1.27	1.13
25	NT	NT		
26	NT	NT		
28	10.3	1.0	-0.99	-1.05
29	NT	NT		
30	NT	NT		

* Outlier, see Section 4.2

Statistics

Assigned Value	12.1	1.4
Homogeneity Value	11.8	1.8
Robust Average	12.5	1.6
Median	12.1	1.0
Mean	13.4	
N	12	
Max	27.5	
Min	9.4	
Robust SD	2.2	
Robust CV	17%	

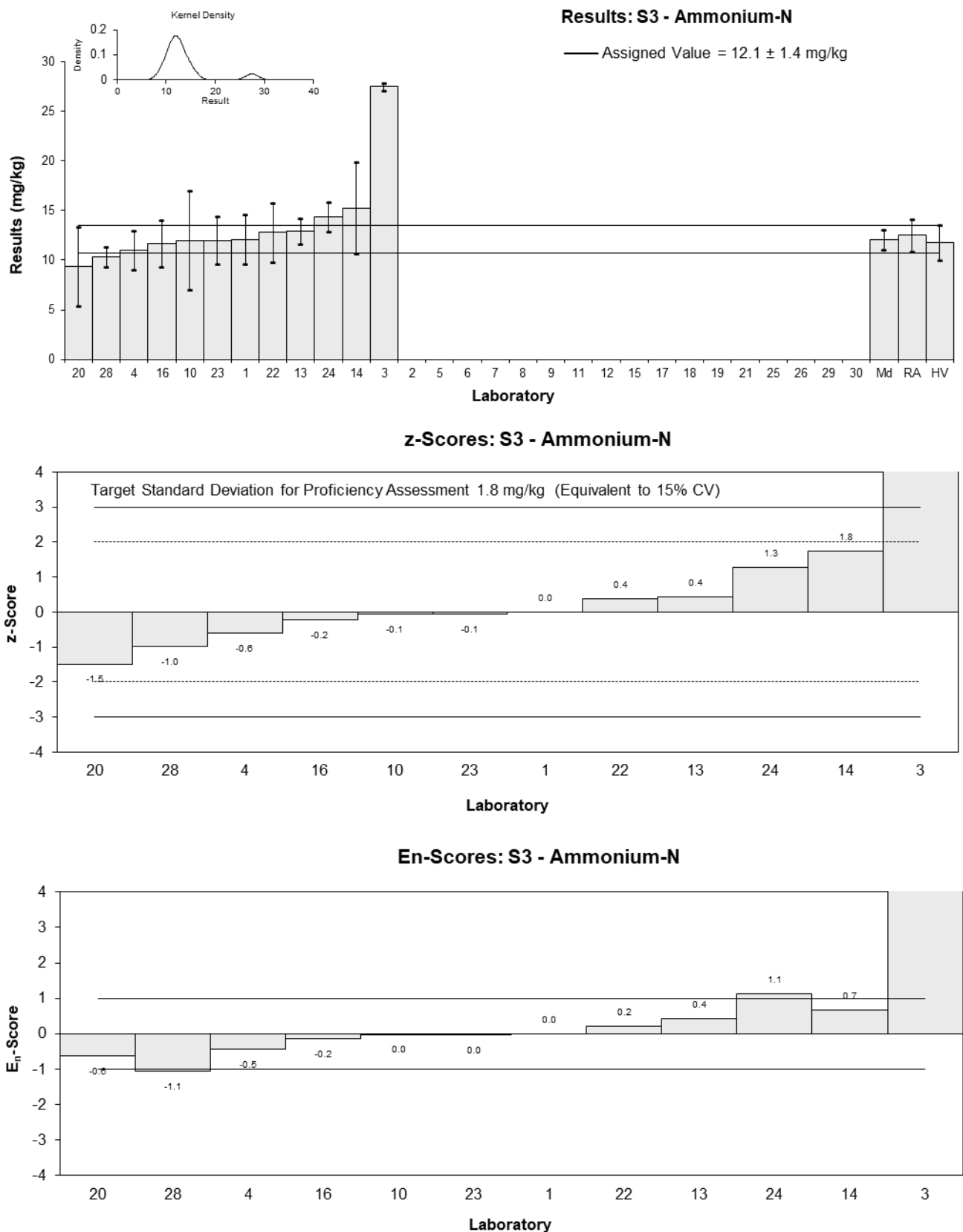


Figure 55

Table 68

Sample Details

Sample No.	S3
Matrix	Soil
Analyte	Nitrate-N
Unit	mg/kg

Participant Results

Lab. Code	Result	Uncertainty	z	E _n
1	17.1	3.3	0.64	0.44
2	NT	NT		
3	21	3	2.31	1.71
4	17	3	0.60	0.44
5	NT	NT		
6	NT	NT		
7	15.2	4.56	-0.17	-0.09
8	NT	NT		
9	NT	NT		
10	16	6	0.17	0.07
11	NR	NR		
12	NT	NT		
13	11.0	1.1	-1.97	-3.09
14	NT	NT		
15	NT	NT		
16	14.9	1.11	-0.30	-0.47
17	NT	NT		
18	NT	NT		
19	NR	NR		
20	15	4	-0.26	-0.15
21	NR	NR		
22	14.9	2.6	-0.30	-0.25
23	15	3	-0.26	-0.19
24	NR	NR		
25	NT	NT		
26	NT	NT		
28	15.2	1.5	-0.17	-0.22
29	NT	NT		
30	NT	NT		

Statistics

Assigned Value	15.6	1.0
Homogeneity Value	15.1	2.3
Robust Average	15.6	1.0
Median	15.2	0.3
Mean	15.7	
N	11	
Max	21	
Min	11	
Robust SD	1.4	
Robust CV	8.9%	

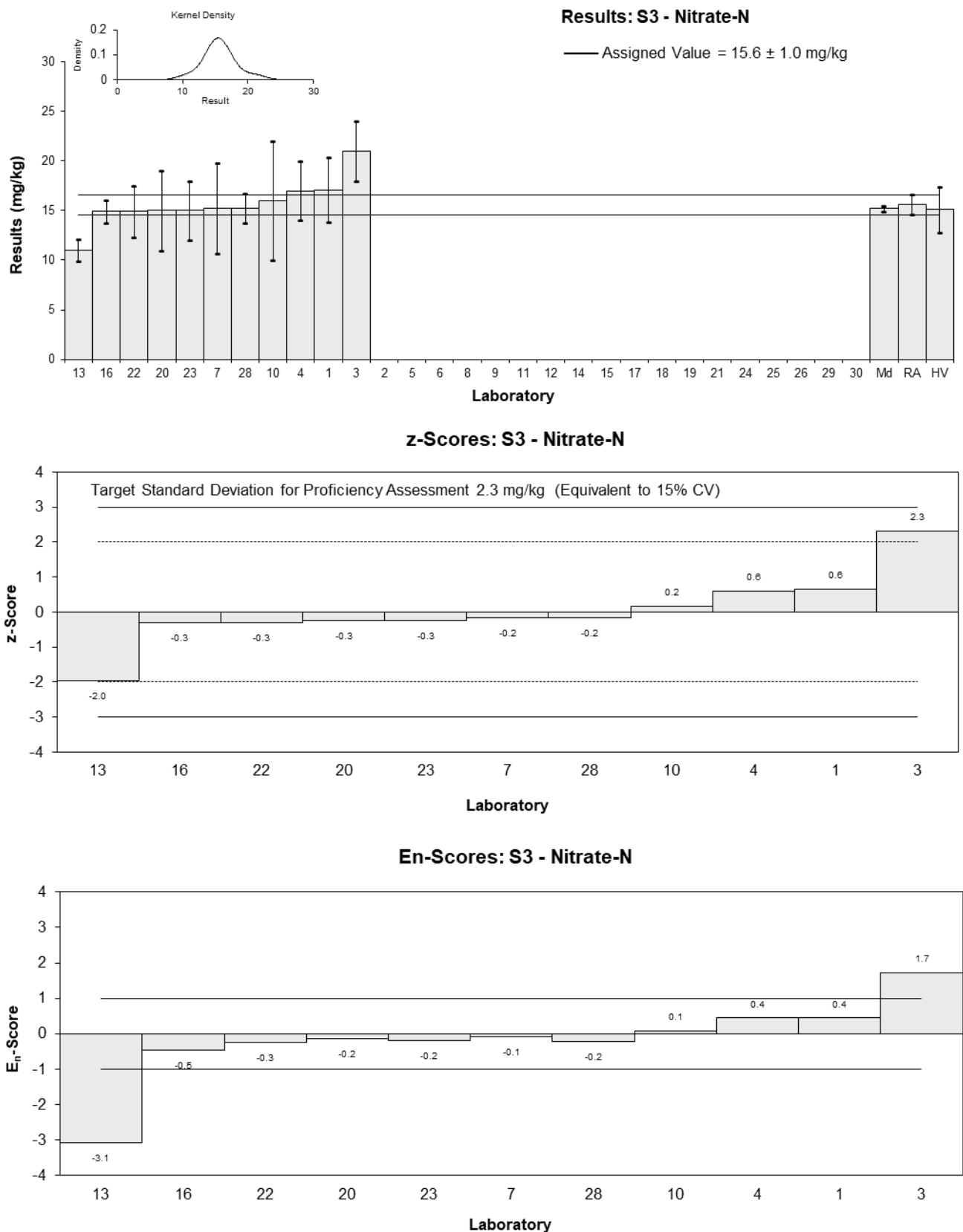


Figure 56

6 DISCUSSION OF RESULTS

6.1 Assigned Value and Traceability

Sample S1 was unspiked sandy soil while **Sample S2** was a biosolid reference material previously prepared by NMI.

Sample S3 was a composite of unspiked agricultural soil samples sent to the NMI for testing.

Assigned Values were the robust average of participants' results. The robust averages used as assigned values and their associated expanded uncertainties were calculated using the procedure described in ISO13528 'Statistical methods for use in proficiency testing by interlaboratory comparisons'. Results less than 50% and more than 150% of the robust average were investigated and then excluded prior to calculation of each assigned value (see subchapters 4.2 and 4.3).⁶ 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 B in S1, Cs and Sb in S2, and bromide, iodide, and orthophosphate-P in S3 because the reported results were either too few or too variable. However, participants may still compare their reported results for some of these tests with the median of participants' results and/or the homogeneity value. Descriptive statistics for these elements are presented in Section 5.

Traceability The assigned value is not traceable to any external reference; it is traceable to the consensus of participants' results deriving 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 930 numerical results, 913 (98%) were reported with an expanded measurement uncertainty. The magnitude of these expanded uncertainties was within the range 0.24% to 1633% of the reported value. The participants used a wide variety of procedures to estimate the expanded measurement uncertainty. These are presented in Table 11.

Approaches to estimating measurement uncertainty include: standard deviation of replicate analysis, Horwitz formula, long term reproducibility, professional judgement, bottom up approach, top down approach using precision and estimates of method and laboratory bias, and top down approach using only the reproducibility from inter-laboratory comparison studies.^{9 – 14}

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 56). 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, 25 laboratories reported results for Pb in S2. The uncertainty of the assigned value estimated from the robust standard deviation of the 25 laboratories' results is 1.0 mg/kg (3.1% of the assigned value). Therefore, Laboratory 17 might have under-estimated their expanded measurement uncertainty (0.3 mg/kg or 0.90% of their reported value) as an uncertainty estimated from one measurement cannot be smaller than the uncertainty estimated from 25 measurements. Alternatively, estimates of uncertainties for Na in S3 larger than 260 mg/kg (the uncertainty of the assigned value, 40 mg/kg plus the allowable variation from the assigned value, the target standard deviation of 110 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 uncertainty reported by Laboratory 12 for Na in S3 (660 mg/kg) might have been over-estimated.

Laboratory 3 should review their calculation procedure for estimating measurement uncertainty as some of their uncertainties were very low.

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

Laboratories 4, 6, 9, 10, 19, 23 and 28 attached estimates of the expanded measurement uncertainty to results reported as a range (“less than”). An estimate of uncertainty expressed as a value cannot be attached to a result expressed as a range.⁹

Laboratories 4, 6, 10, 19, 20 and 29 reported estimates of expanded uncertainty for some of their measurement results which were equal to larger than the results themselves.

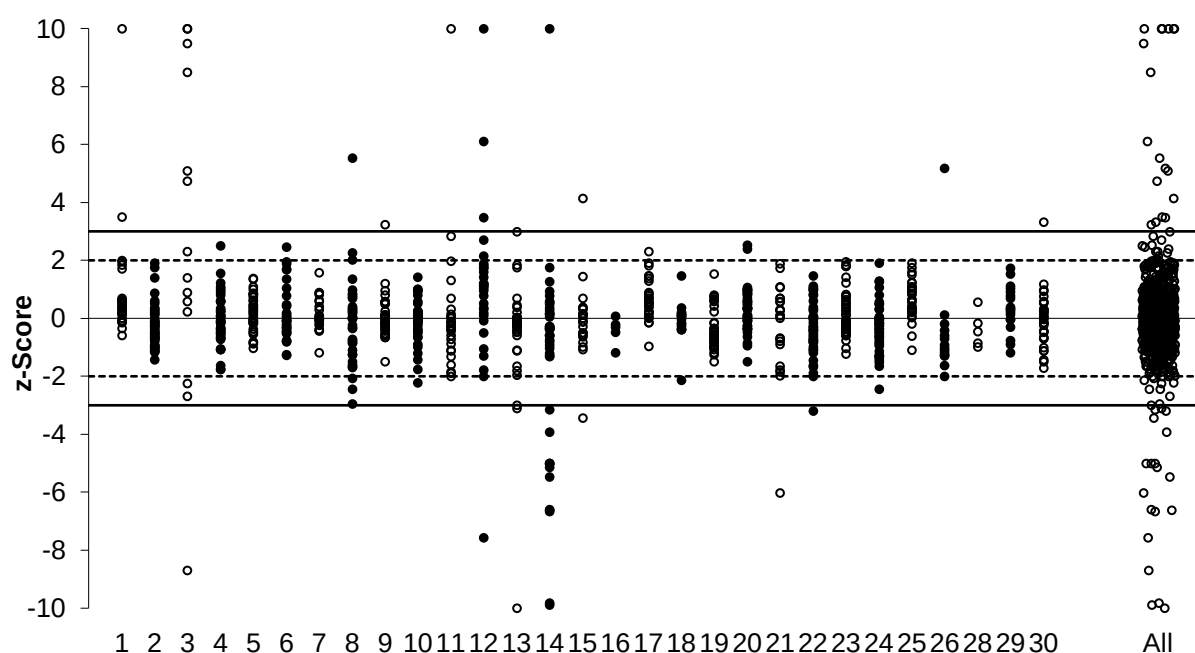
In some cases, the results were reported with an inappropriate number of significant figures. The recommended format is to write uncertainty to no more than two significant figures and then to write the result with the corresponding number of decimal places. For example, instead of 0.727 ± 0.2492 mg/kg, it is better to report 0.73 ± 0.25 mg/kg or instead of 22748.89 ± 6824.667 mg/kg, it is better to report 22749 ± 6800 mg/kg.⁹

6.3 z-Score

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

The target standard deviation defines satisfactory performance in a proficiency test. Target standard deviations equivalent to 3.5% to 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 fixed reference value points for assessment of laboratory performance, independent of group performance.

The between laboratory coefficient of variation predicted by the Thompson equation⁷ and the participants’ coefficient of variation resulted in this study are presented for comparison in Table 69.



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

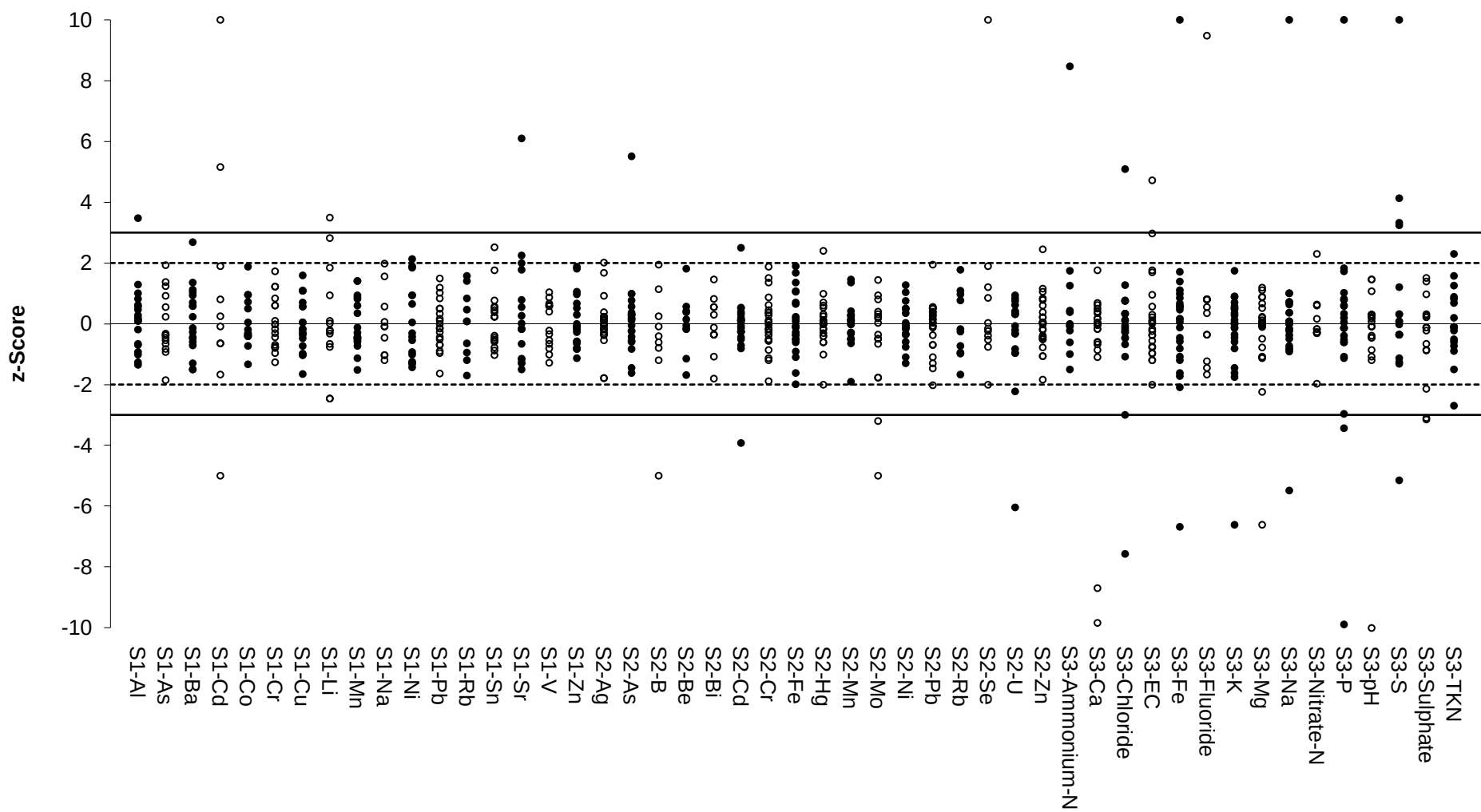
Figure 57 z-Score Dispersal by Laboratory

The dispersal of participants’ z-scores is presented in Figure 57 (by laboratory code) and in Figure 59 (by test). Of 886 results for which z-scores were calculated, 827 (93%) returned an

Table 69 Between Laboratory CV of this Study, Thompson CV and Set Target SD

Sample	Test	Assigned value (mg/kg)	Between Laboratories CV*	Thompson/ Horwitz CV	Target SD (as PCV)
S1	Al	1380	13%	5.4%	15%
S1	As	1.08	22%	16%	20%
S1	Ba	14.3	21%	11%	20%
S1	Cd	0.0344	26%	22%	20%
S1	Co	1.09	16%	16%	20%
S1	Cr	4.01	19%	13%	20%
S1	Cu	12.9	14%	11%	15%
S1	Li	0.588	15%	17%	20%
S1	Mn	18.7	19%	10%	20%
S1	Na	30.5	24%	9.6%	20%
S1	Ni	1.68	28%	15%	20%
S1	Pb	39.0	13%	9.2%	15%
S1	Rb	1.97	25%	14%	20%
S1	Sn	8.70	12%	12%	15%
S1	Sr	2.07	27%	14%	20%
S1	V	5.37	18%	12%	20%
S1	Zn	13.2	17%	11%	20%
S2	Ag	6.14	6.2%	12%	15%
S2	As	3.83	10%	13%	15%
S2	B	2.85	22%	14%	20%
S2	Be	0.615	14%	17%	15%
S2	Bi	0.739	14%	17%	15%
S2	Cd	0.727	6.6%	17%	15%
S2	Cr	32.8	8.4%	9.5%	10%
S2	Cs	Not Set	37%	NA	Not Set
S2	Fe	24200	9.7%	3.5%	10%
S2	Hg	0.500	10%	18%	20%
S2	Mn	484	4.4%	6.3%	10%
S2	Mo	1.55	18%	15%	20%
S2	Ni	15.8	8.5%	11%	15%
S2	Pb	32.2	6.5%	9.5%	10%
S2	Rb	11.7	25%	11%	20%
S2	Sb	Not Set	35%	NA	Not Set
S2	Se	1.45	23%	15%	20%
S2	U	1.05	12%	16%	15%
S2	Zn	179	7.7%	7.3%	10%
S3	Ca	5790	5.5%	4.3%	10%
S3	Fe	31700	17%	3.4%	15%
S3	K	1900	12%	5.1%	15%
S3	Mg	5600	12%	4.4%	15%
S3	Na	1090	6.7%	5.6%	10%
S3	P	213	10%	7.1%	10%
S3	S	148	15%	7.5%	15%
S3	Chloride	493	6.6%	6.3%	10%
S3	Fluoride	1.72	22%	15%	20%
S3	Sulphate	92	19%	8.1%	20%
S3	EC	675 μ S/cm	12%	6%	10%
S3	pH	7.42	2.7%	12%	3.5%
S3	TKN	1010	11%	5.6%	10%
S3	Ammonium-N	12.1	15%	11%	15%
S3	Nitrate-N	15.6	8.9%	11%	15%

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



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

Figure 59 z-Score Dispersal by Test

Summary of Participant's Performance in AQA 24-15 Samples S1, S2 and S3

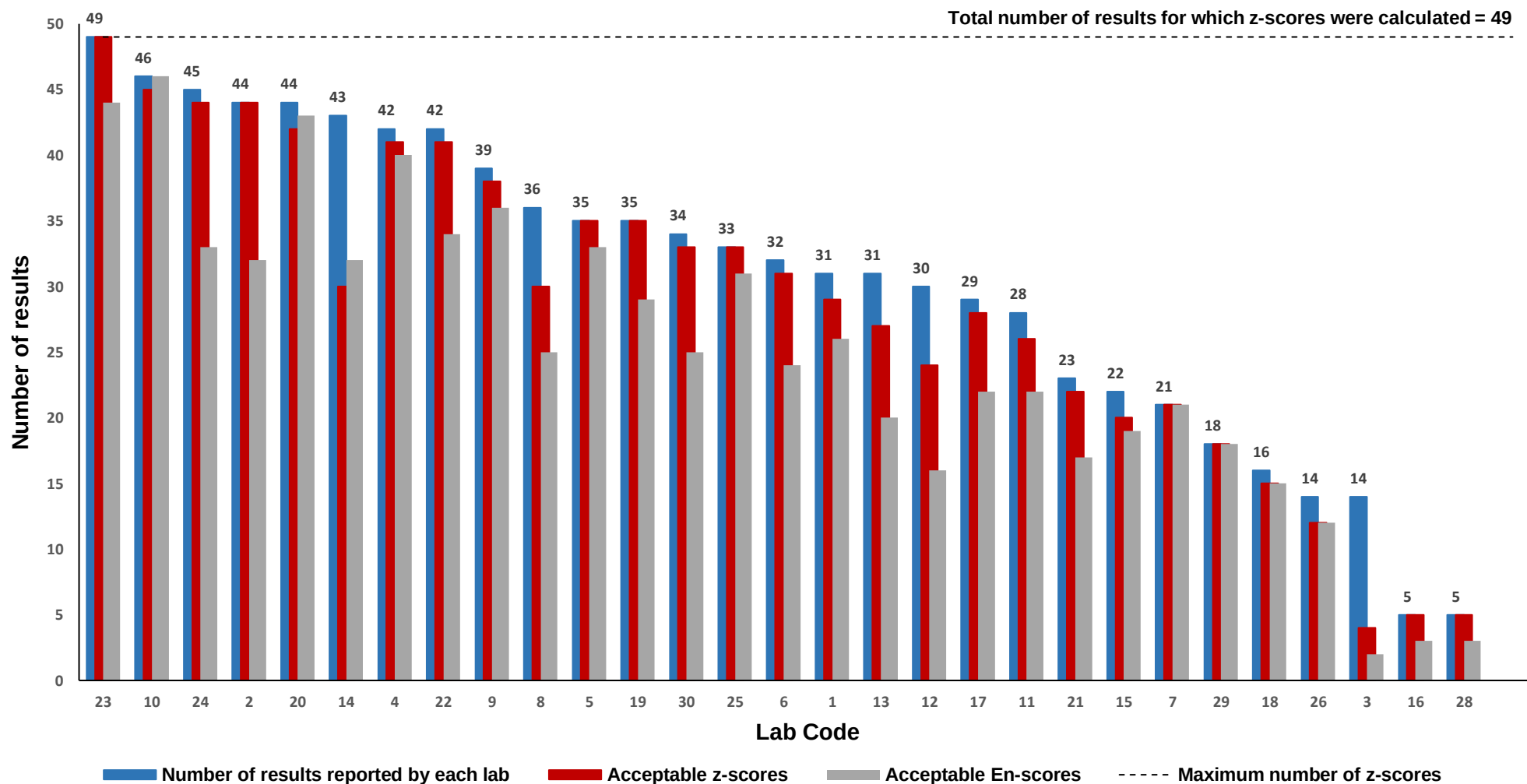


Figure 60 Summary of Participants' Performance

Table 70 Summary of Participants' Results and Performance in Sample S1

Lab Code	Al (mg/kg)	As (mg/kg)	B (mg/kg)	Ba (mg/kg)	Cd (mg/kg)	Co (mg/kg)	Cr (mg/kg)	Cu (mg/kg)	Li (mg/kg)	Mn (mg/kg)	Na (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Rb (mg/kg)	Sn (mg/kg)	Sr (mg/kg)	V (mg/kg)	Zn (mg/kg)
AV	1380	1.08	Not Set	14.3	0.0344	1.09	4.01	12.9	0.588	18.7	30.5	1.68	39.0	1.97	8.70	2.07	5.37	13.2
HV	1370	0.96	1.08	15.1	0.0353	1.02	3.90	12.5	0.586	18.6	NA	1.50	36.0	NA	NA	2.30	5.14	11.7
1	1510	1.5	9.5	16	0.2	1.2	4.5	14	1.0	21	34	2.3	42	NT	9.2	2.9	5.8	15
2	1245.1	1.013	<50	12.95	<1	0.932	3.248	10.899	0.564	14.494	<50	1.368	33.391	1.720	7.927	1.588	4.280	11.122
3	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
4	1400	<4	<10	16	<0.4	1	5	15	<1	22	40	2	45	2	9	2	6	13
5	1480	<3	<2	18.2	<0.1	1.25	4.69	15.0	<1	21.8	24.2	<2	41.0	NT	9.29	2.18	6.30	12.9
6	1410	0.98	<20	13.07	0.0338	1.02	3.38	12.3	0.56	18.3	<40	1.26	36.1	2.16	7.7	1.54	<10	12.7
7	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
8	1120	<5	<50	10	<1	<2	4	13	0.3	16	<50	<2	39	1.3	9	3	<5	11
9	1400	1.2	<5	12.6	<0.1	1.1	4.1	12.6	0.51	17.6	<50	1.5	36.4	NT	8.2	1.8	4.8	14.6
10	1400	1	<10	15	0.03	1	4	13	0.6	24	30	2	40	2	9	2	6	13
11	1650	<5	<5	16.3	<5	<5	<5	11.5	0.921	17.1	42.6	<5	38.1	NR	7.55	<5	<5	12.5
12	2100	<5	1.7	22	<1	1.5	5.4	16	NT	24	<200	2.4	46	NT	9.7	4.6	6.5	18
13	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
14	1186.7	1.28	0	13.53	0	1.01	3.77	12.35	0.55	16.51	23.17	1.24	41.9	NT	7.36	1.53	4.5	11.68
15	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
16	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
17	1424.231	1.379	<3	NR	NR	NR	NR	14.256	<1	NR	NR	2.321	NR	NT	9.405	NR	NR	NR
18	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
19	1170	<5	<50	10	<1	<2	3	12	0.5	17	<50	<2	35	1.5	8	2	<5	11
20	1500	1	<10	17	0.03	1.3	4.5	14	<1	20	30	1.7	44	2.3	12	2.4	6.1	16
21	1440	0.68	0.61	NR	0.023	<4	NR	NR	NR	NR	<40	NR	33.8	NR	NR	<4	NR	18.2
22	1590	0.94	0.93	17.5	0.04	1.00	3.41	11.0	0.70	17.1	30.9	1.54	37.5	NR	8.02	2.08	5.01	13.2
23	1550	0.964	1.03	13.9	0.0362	1.02	3.87	12.2	0.805	16.7	24.2	1.58	39.6	2.53	8.10	2.81	5.13	11.7
24	1100	0.9	<5	10.6	<0.1	0.8	3.4	9.7	0.3	13	<50	1.2	35	1.6	8	1.6	4	11.5
25	1340	1.35	<1.3	17.3	0.0475	1.3	5	15	0.59	22.25	<130	1.9	47.75	2.595	11	2.3	6.05	14
26	NT	0.88	NT	NT	0.07	NT	3.46	10.91	NT	NT	NT	1.25	29.46	NT	NT	NT	NT	10.24
28	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
29	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
30	1240	1.13	<5	12.3	<0.1	1.05	3.66	12.6	<1	16.9	27.7	1.34	38.2	NT	9.45	1.45	4.66	15.8

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

Table 71 Summary of Participants' Results and Performance in Sample S2

Lab Code	Ag (mg/kg)	As (mg/kg)	B (mg/kg)	Be (mg/kg)	Bi (mg/kg)	Cd (mg/kg)	Cr (mg/kg)	Cs (mg/kg)	Fe (mg/kg)	Hg (mg/kg)	Mn (mg/kg)	Mo (mg/kg)	Ni (mg/kg)	Pb (mg/kg)	Rb (mg/kg)	Sb (mg/kg)	Se (mg/kg)	U (mg/kg)	Zn (mg/kg)
AV	6.14	3.83	2.85	0.615	0.739	0.727	32.8	Not Set	24200	0.500	484	1.55	15.8	32.2	11.7	Not Set	1.45	1.05	179
1	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
2	6.247	3.514	<50	0.509	0.727	0.673	31.156	0.717	28787	0.56	497.224	1.346	15.494	31.855	11.126	0.555	1.341	1.018	171.418
3	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
4	6	4	<10	<1	<1	1	31	<1	23000	0.5	460	1	15	30	10	<10	<2	1	160
5	5.97	4.4	<10	<1	NT	0.77	37.3	NT	24000	0.500	490	1.62	15.8	32.0	NT	NT	<4	1.15	190
6	7.7	3.36	2.8	0.61	0.700	0.731	39.0	0.85	28300	0.506	550	1.61	18.3	38.5	13.5	0.89	1.3	1.117	223
7	6.10	4.00	<10	< 2	<10	0.68	34.83	NR	24400.70	0.46	483.56	< 5	15.59	33.53	NR	< 10	< 2	< 10	178.70
8	8	7	<50	<1	0.7	<1	30	0.5	27500	0.5	492	<2	14	33	7.8	<5	<5	1.0	194
9	5.8	3.7	<5	0.60	0.80	0.74	33.2	NT	23900	0.50	470	1.8	15.9	32.3	NT	1.1	1.8	1.2	180
10	7	3	3	<1	<1	0.7	33	<1	22000	0.6	490	1	17	32	9.4	<10	<2	0.7	170
11	5.83	<5	<5	<5	NR	<5	29.1	NR	20300	0.485	392	<5	14.4	32.4	NR	<5	<5	<5	146
12	4.5	<5	3.5	<0.6	NT	<1	34	NT	24000	0.3	460	<5	17	28	NT	<0.5	<5	NT	160
13	6.17	3.70	2.62	0.65	0.54	0.72	32.3	NT	23547	0.57	469	1.56	15.7	28.7	NT	1.19	1.46	1.04	171
14	6.28	4.16	0	0.63	NT	0.3	31.59	NT	22748.89	0.514	497.17	0	14.94	34	NT	0	5.23	NT	179.98
15	6.5	3.9	< 5	< 2	< 10	0.74	31.9	NT	23000	0.5	453	2	15.5	32.7	NT	1.3	< 2	< 10	170
16	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
17	6.348	3.925	<3	0.7822	0.902	0.784	35.683	1.216	24760.8	0.554	484.01	1.674	18.842	33.466	NT	1.292	1.406	1.184	193.20
18	6.25	4.04	< 10	< 2	< 10	0.75	32.2	NT	23333	0.46	470	< 5	15.7	32.7	NT	< 10	< 2	< 10	172
19	6	<5	<50	<1	0.7	<1	31	0.5	25800	0.4	505	<2	15	30	9.5	<5	<5	0.9	183
20	6.1	4.4	<10	<1	<1	0.7	34	<1	24000	0.74	490	1.4	17	31	14	<10	<2	0.9	180
21	4.5	<10	2.4	NR	NR	0.65	NR	NR	19400	0.51	NR	NR	NR	34	NR	NR	NR	0.100	198
22	6.00	2.90	2.17	0.46	0.62	0.64	26.6	NR	26773	0.44	555	0.56	13.2	27.5	NR	0.07	0.87	0.92	165
23	5.64	3.49	3.97	0.668	0.773	0.713	34.3	1.16	23300	0.481	461	1.44	17.6	33.8	15.9	0.58	1.23	1.11	176
24	5.9	3.8	<5	0.6	0.8	0.7	32.7	0.8	26800	0.5	484	1.4	15.4	33.8	11.3	0.7	2	1.1	180
25	6.35	3.95	2.5	0.655	0.83	0.762	33.75	0.98	21550	0.525	488.5	1.65	16.65	33.1	14.25	1.1	1.7	1.1	183
26	NT	3.59	NT	NT	NT	0.74	28.88	NT	NT	0.44	NT	NT	12.72	25.74	NT	NT	NT	NT	175.40
28	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
29	6.34	4.28	<10	<2	<10	0.742	37.8	NT	25900	0.470	498	<5	15.7	33.3	NT	<10	<2	<10	186
30	6.35	4.00	<5	<1	<1	0.786	33.1	NT	24600	0.532	498	1.84	15.7	33.9	NT	1.38	1.38	1.17	200

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

Table 72 Summary of Participants' Results and Performance for Sample S3

Lab Code	Ca (mg/kg)	Fe (mg/kg)	K (mg/kg)	Mg (mg/kg)	Na (mg/kg)	P (mg/kg)	S (mg/kg)	Bromide (mg/kg)	Chloride (mg/kg)
AV	5790	31700	1900	5600	1090	213	148	Not Set	493
HV	6290	36800	2240	6150	1180	221	NA	3.00	427
1	5450	32500	1870	5520	1130	210	155	NT	500
2	5889.2	38351.6	2048.8	6331.2	1130.6	217.5	147.86	NT	480.2
3	750.0	34388.49	2156.65	3719.86	3019.17	460.05	NT	NT	743.8
4	5400	24000	1400	4700	1100	190	140	<2.5	510
5	5700	27900	1770	5200	1000	230	140	NT	486
6	NT	NT	NT	NT	NT	NT	NT	NT	NT
7	5785.22	35767.00	2062.79	6359.43	1064	230.41	< 200	< 5	498
8	6020	21800	1440	4680	1170	150	120	2.08	510
9	5700	34400	2040	5800	1070	210	220	NT	NT
10	5800	26000	1900	5600	1200	210	150	<2.5	490
11	5160	250000	1940	5640	1090	205	119	NR	480
12	5900	37000	2400	6600	1100	NT	470	NT	120
13	6809	31134	1732	5775	1038	252	123	NT	345
14	100.003	4.489	18.248	50.368	493.208	2.363	33.961	2.44	530.9
15	6000	29000	2100	5700	1000	140	240	NT	440
16	NR	NR	NR	NR	NR	NR	NR	NR	469.9
17	5806	34941	1917	6039	1074	225.6	NT	NT	NT
18	NT	32054	NT	NT	NT	214	NT	< 5	483
19	6150	26600	1670	4950	1170	200	140	NT	530
20	5300	34000	2000	5600	1200	220	150	<2.5	510
21	6190	34400	1900	6170	1100	250	NR	2.6	460
22	5770	36440	1980	5650	1010	226	155	NR	NR
23	5440	32500	1860	5540	1160	202	175	2.7	530
24	6080	29500	1800	5590	1050	200	140	2.07	556
25	NT	NT	NT	NT	NT	NT	NT	NT	NT
26	NT	NT	NT	NT	NT	NT	NT	NT	NT
28	NT	NT	NT	NT	NT	NT	NT	<5	471
29	5770	39900	2160	6530	991	235	NT	NT	NT
30	5820	23500	1490	4650	1090	189	222	NT	NT

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

Table 72 Summary of Participants' Results and Performance for Sample S3 (continued)

Lab Code	Fluoride (mg/kg)	Iodide (mg/kg)	Orthophosphate-P (mg/kg)	Sulphate (mg/kg)	EC (μ S/cm)	pH	TKN (mg/kg)	Ammonium-N (mg/kg)	Nitrate-N (mg/kg)
AV	1.72	Not Set	Not Set	92	675	7.42	1010	12.1	15.6
HV	NA	NA	NA	76	658	7.46	960	11.8	15.1
1	1.6	NT	0.30	96	790	7.4	1030	12.1	17.1
2	1.225	NT	0.12	97.896	794	7.14	937.3	NT	NT
3	4.98	NT	0.33	117.5	994	7.48	738.3	27.5	21
4	2	<1	<0.5	90	690	7.3	1000	11	17
5	NT	NT	NT	76	638	7.47	NT	NT	NT
6	NT	NT	NT	NT	NT	NT	NT	NT	NT
7	< 5	< 5	< 10	< 30	595	7.32	1170	NT	15.2
8	2	1.52	0.1	110	659	7.5	1080	NT	NT
9	NT	NT	NT	NT	676	7.4	860	NT	NT
10	2	NT	<0.5	80	680	7.3	1000	12	16
11	<5	1.1	<0.2	<50	540	7.5	1000	NR	NR
12	NT	NT	<5	NT	740	7.5	NT	NT	NT
13	1.15	NT	1.5	35	876	4.28	989	12.9	11.0
14	1.84	1.32	0.472	34	625	NT	1138.2	15.27	NT
15	NT	NT	< 1	< 10	609	7.5	1000	NT	NT
16	NR	NR	NR	NR	595	7.44	NR	11.7	14.9
17	NT	NT	NT	NT	610.30	7.80	1242.83	NT	NT
18	NT	< 5	< 10	52.6	683	7.8	NT	NT	NT
19	2	NT	0.1	120	714	7.2	950	NR	NR
20	1.6	1	<0.5	90	650	7.5	1100	9.4	15
21	NR	NR	<1	97	695	7.7	NR	NR	NR
22	NR	NR	NR	NR	NR	NR	1093	12.8	14.9
23	1.3	<1	<1	88	650	7.4	960	12	15
24	2	1.32	0.2	90	664	7.5	920	14.4	NR
25	NT	NT	NT	NT	NT	NT	NT	NT	NT
26	NT	NT	NT	NT	NT	NT	NT	NT	NT
28	1.91	<5	NT	76.3	NT	NT	NT	10.3	15.2
29	NT	NT	NT	NT	622.8	7.11	NT	NT	NT
30	NT	NT	NT	NT	NT	NT	NT	NT	NT

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

6.5 Participants' Results and Analytical Methods for Acid Extractable Elements

A summary of participants' results, and performance is presented in Tables 70 to 72 and in Figures 57 to 60.

Sample S1 challenged some participants' analytical techniques. This was a low-level natural sandy soil sample often encountered by laboratories in their routine life. This sample aims to support laboratories to assess their level of reporting set for the analytes of interest in sandy soil, check their laboratory background contamination and/or assess their method performance in sandy soils. The precision of some participants' methods hampered their attempts to report results for this sample. The sample had a high silica content and the results for some elements were more prone to be method dependent (change with variation in temperature, time, etc).

Cs and Sb in S2 followed by low level Ni, Sr, Cd and Rb in S1 and B in S1 and S2 presented analytical difficulty to participating laboratories. A limited number of laboratories reported results for these tests or/and the reported results were variable.

The method descriptions provided by participants for acid extractable elements are presented in Tables 1 and 10 and 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.^{5, 15-18} While an aggressive digestion regime can produce high, misleading results, weak digestion regimes (low digestion temperature, reduced digestion time, diluted acids and/or a low ratio of acid to sample size) may extract just a fraction of the contaminants from the soil. There is no standardisation of methods for acid extractable elements. In general methods are conventionally defined by procedures involving extractions: with aqua regia or with various amounts of HNO₃, HCl, in combination or alone and most of these methods produce comparable results.¹⁹⁻²¹

In the present study most popular digestion methods involved: a sample size of between 0.5 g to 1 g, an extraction temperature between of 95°C to 120°C, an extraction time between 60 min to 120 min and a ratio HNO₃ to HCl of 1:1.

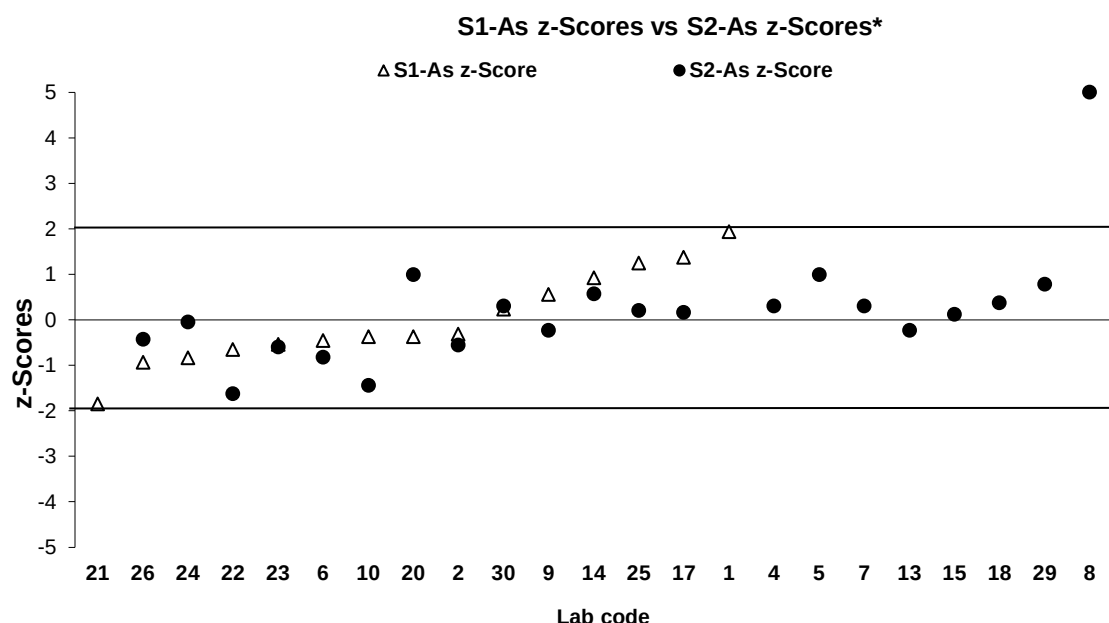
Laboratories 2, 19 and 24 extracted their sample at a temperature of 95°C for 60 min using dilute acids. Most of the results reported by them for acid extractable elements were lower than the assigned value. Dilute acids might extract only a fraction of some elements from the contaminated soil.

Laboratory 3 used for measurement of acid extractable elements in S3 a staggered digestion regime. They extracted their sample at 100°C, 120°C and 140°C temperatures for 180 min and used a sample size of only 0.25 g. They also reported using only HNO₃ and H₂O₂ as extraction agent. Most of the results reported by them returned unsatisfactory z-scores. Caution should be exercised when such a small sample size is taken for analysis as this might not be representative of the whole sample. Some acid extractable elements can be partially recovered from the soil when only HNO₃ is used for extraction.

Individual Element Commentary

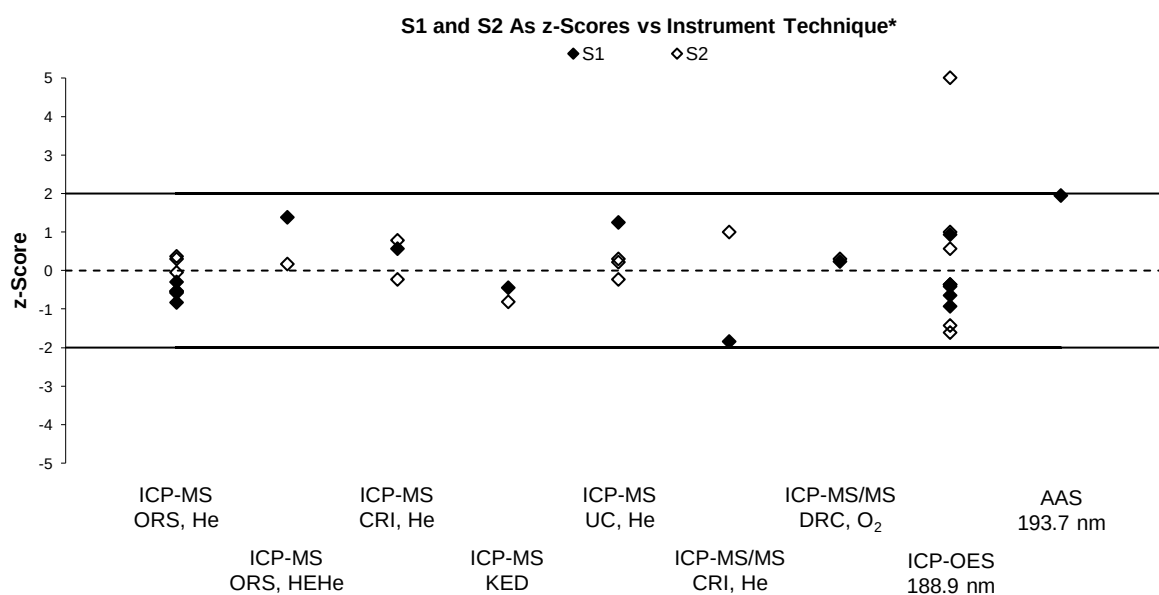
Arsenic level in S1 was 1.08 mg/kg and in S2 was 3.83 mg/kg. Laboratory 8 should review their procedure for measuring As in soil as they reported a result as less than 5 mg/kg in S1 but a result of 7 mg/kg in S2.

Participants can assess the precision and bias of their methods for As in soil in Figure 61. Plots of participants' performance versus instrumental techniques used for As measurements in S1 and S2 are presented in Figure 62. Most participants used ICP-MS in collision mode. Four laboratories measured the low level As in S1 by ICP-OES and wavelength 188.9. All performed satisfactorily.



Laboratory 8 z-score of 5.52 has been plotted as 5.

Figure 61 S1 and S2 As z-Scores vs. Laboratory Code



Laboratory 8 z-score of 5.52 has been plotted as 5.

Figure 62 S1 and S2 As z-Scores vs. Instrumental Technique

Antimony results in S2 were variable with a large between-laboratory CV of 35%. No assigned value was set for this test. Antimony is an element whose recovery strongly depends

on the acids employed for digestion. It is known that when only nitric acid is used, Sb is transformed into a mixture of insoluble oxides (Sb_2O_3 , Sb_2O_5 , $\text{Sb}_4\text{O}_4(\text{OH})_2(\text{NO}_3)_2$) but when hydrochloric acid is also involved it changes into chloro-complexes (SbCl_6^-). In an aqueous solution, sufficient hydrogen ion concentration must be maintained in order to prevent SbCl_6^- hydrolysis.²²⁻²⁴ Laboratories should consider using matrix matched control samples to assess their digestion regime and increase their estimates of uncertainty for Sb measurements in soil.

Plots of participants' performance versus instrumental techniques used for Sb measurements are presented in Figure 63

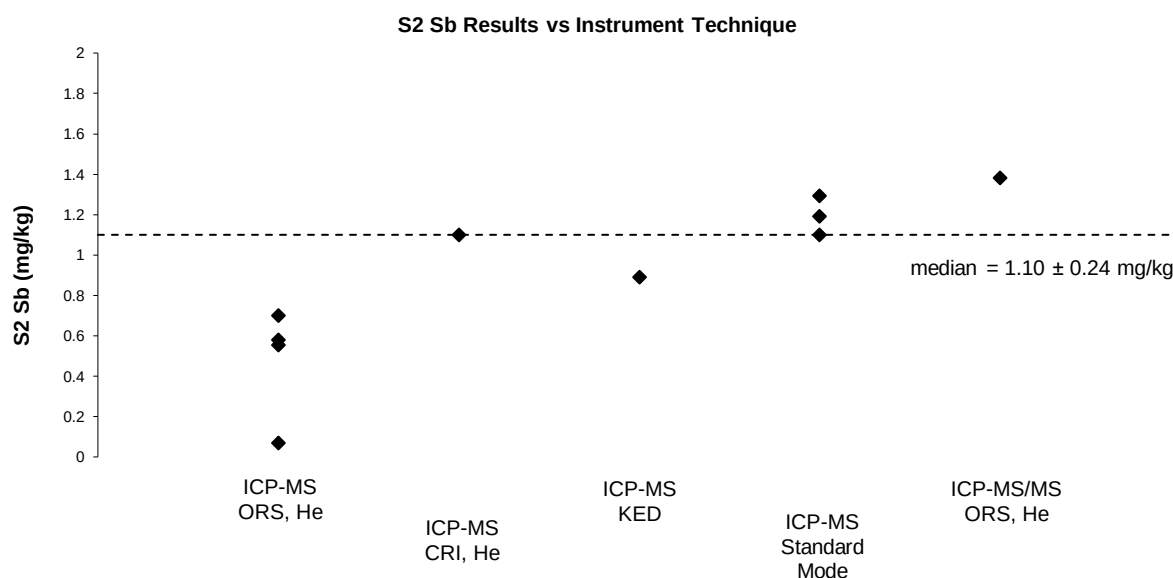
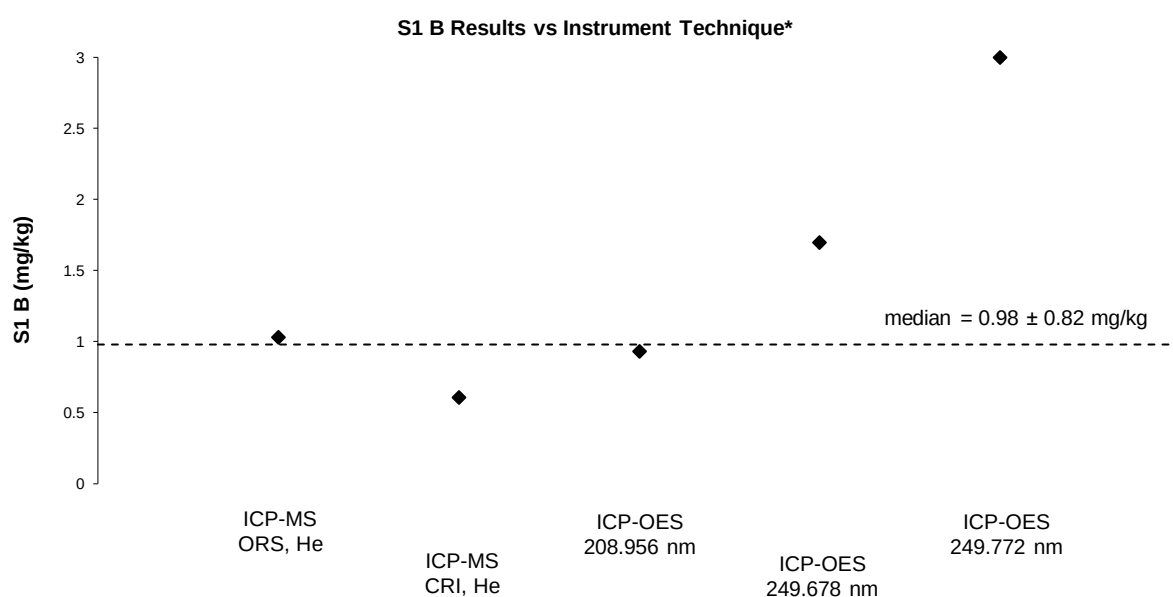


Figure 63 S2-Sb Results vs. Instrumental Technique



*The result reported by Laboratory 1 of 9.5 mg/kg has been plotted as 3.0 mg/kg.

Figure 64 S1-B Results vs. Instrumental Technique

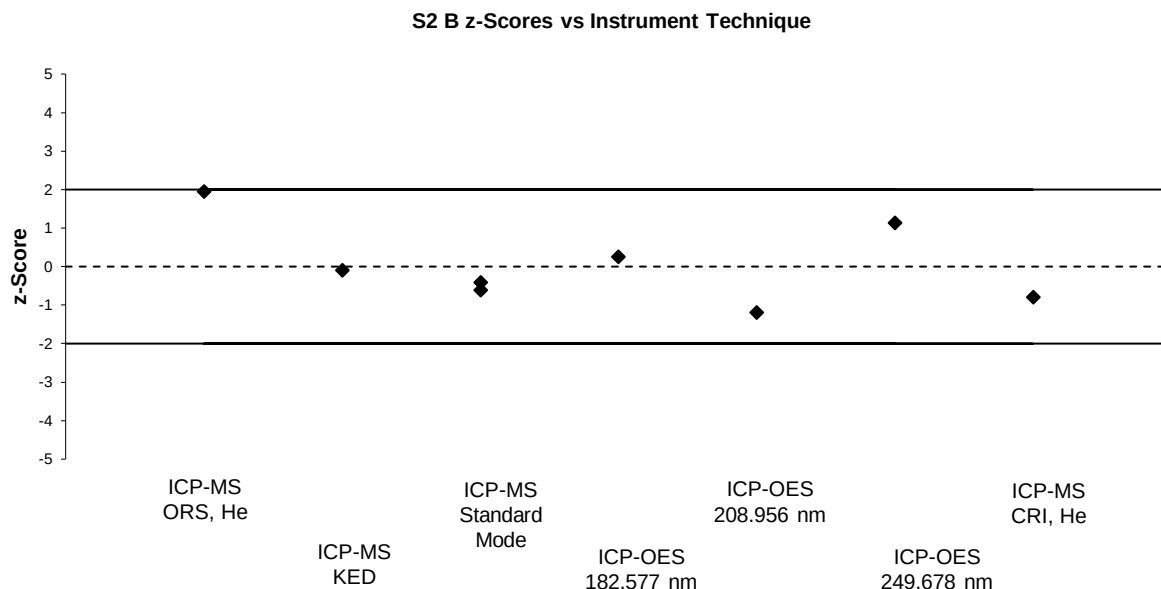


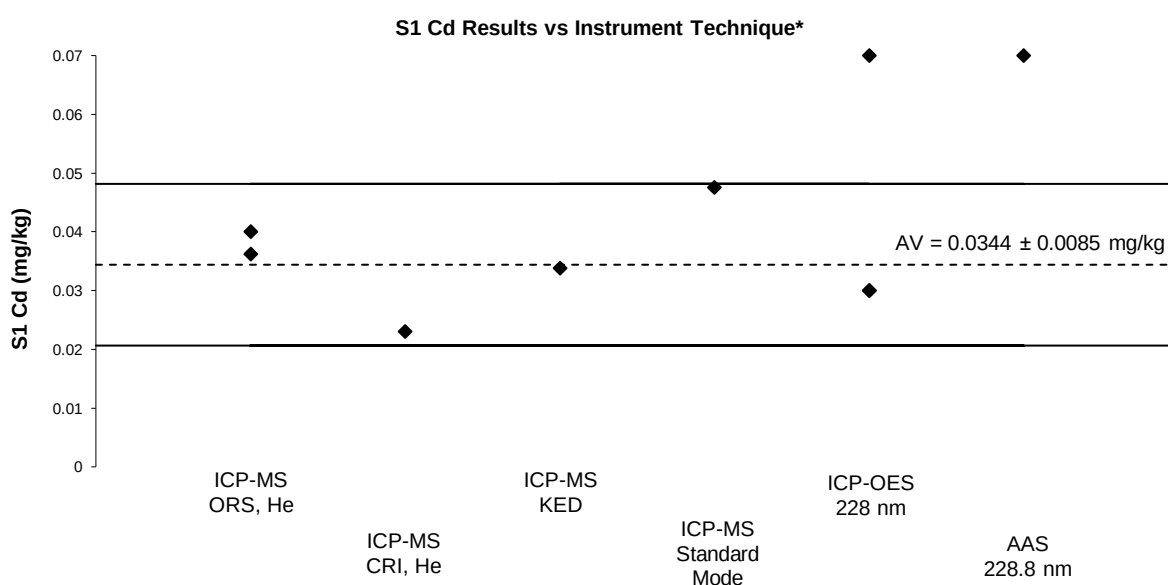
Figure 65 S2 B z-Scores vs. Instrumental Technique

Boron Measurements at low level in S1 and S2 challenged participants' analytical techniques. Only six laboratories reported results for this test in S1. The results reported by Laboratories 22 and 23 were in good agreement with the median value of 0.98 mg/kg and with the homogeneity value of 1.08 mg/kg. Boron level in S2 was 2.85 mg/kg. Of 25 laboratories who analysed sample S2, only 9 reported results for B. All performed acceptably but one.

Plots of B results in S1 versus the instrumental technique are presented in Figure 64 while plots of participants' performance for B in S2 are presented in Figure 65. Caution should be exercised when ICP-OES with wavelength 249.7 nm is used for B measurement without the correction equation. Fe line 249.771 nm has direct overlap interference on B line 249.7 nm.

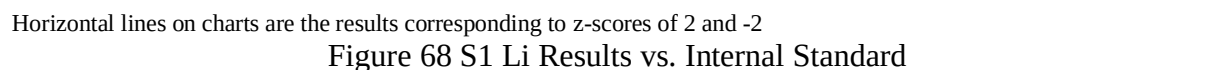
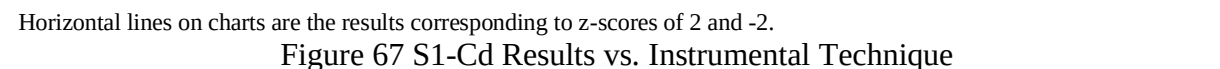
Cadmium level in S1 was low, at 0.0344 mg/kg.

Cd 228.802 nm can have significant spectral interferences from Fe 228.804 nm when measured, especially at low level, by ICP-OES, without correction equation (Figure 66).

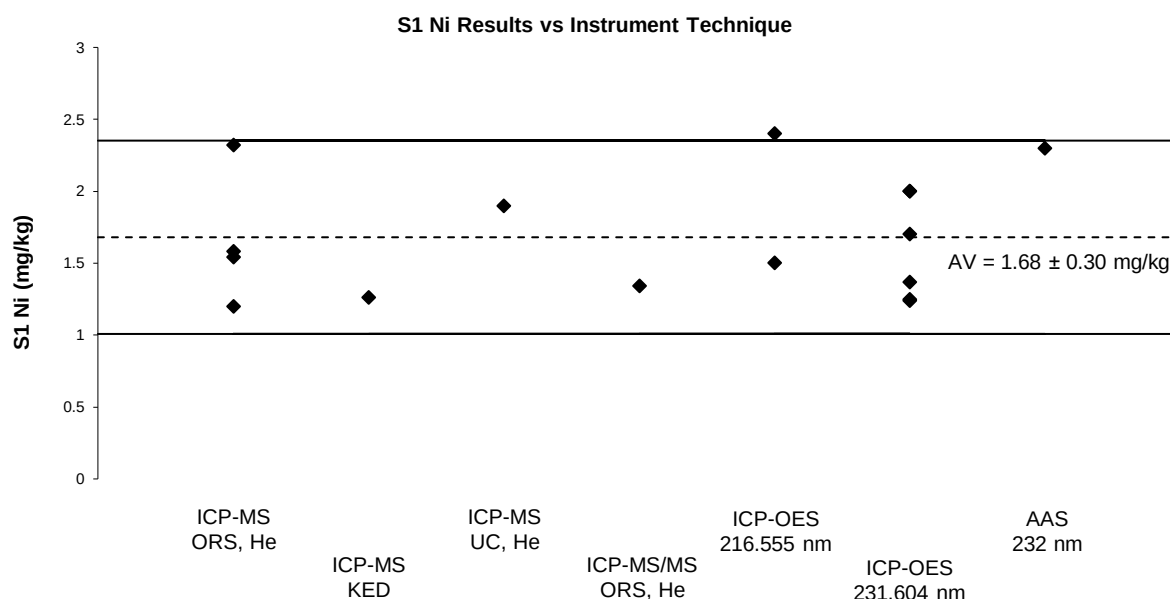


*Laboratory 1 result of 0.2 mg/kg has been plotted as 0.07 mg/kg. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2.

Figure 66 S1-Cd Results vs. Instrumental Technique



Nickel level in S1 was low, 1.68 mg/kg, and this might explain the high between laboratory CV returned for this element (28%). Figure 69 presents plots of participants' results versus instrumental technique. Eight laboratories reported using ICP-OES and seven used ICP-MS.



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

Figure 69 S1 Ni Results vs. Instrumental Technique

Rubidium Only 10 laboratories reported results for Rb in S1 and S2 and all used ICP-MS in standard mode or in collision mode (Figure 70).

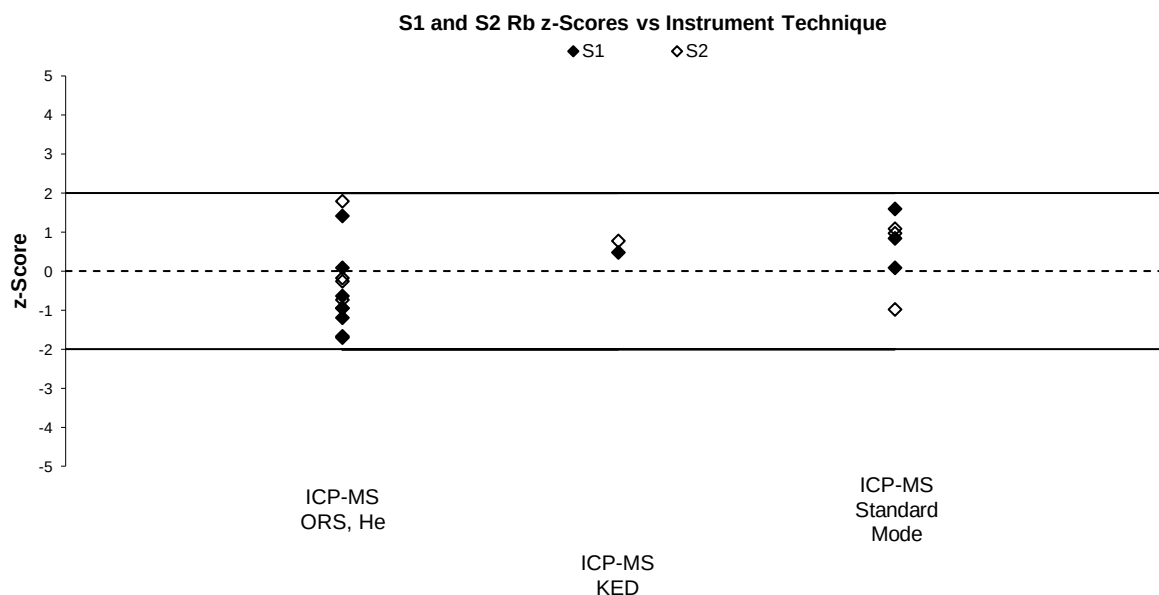
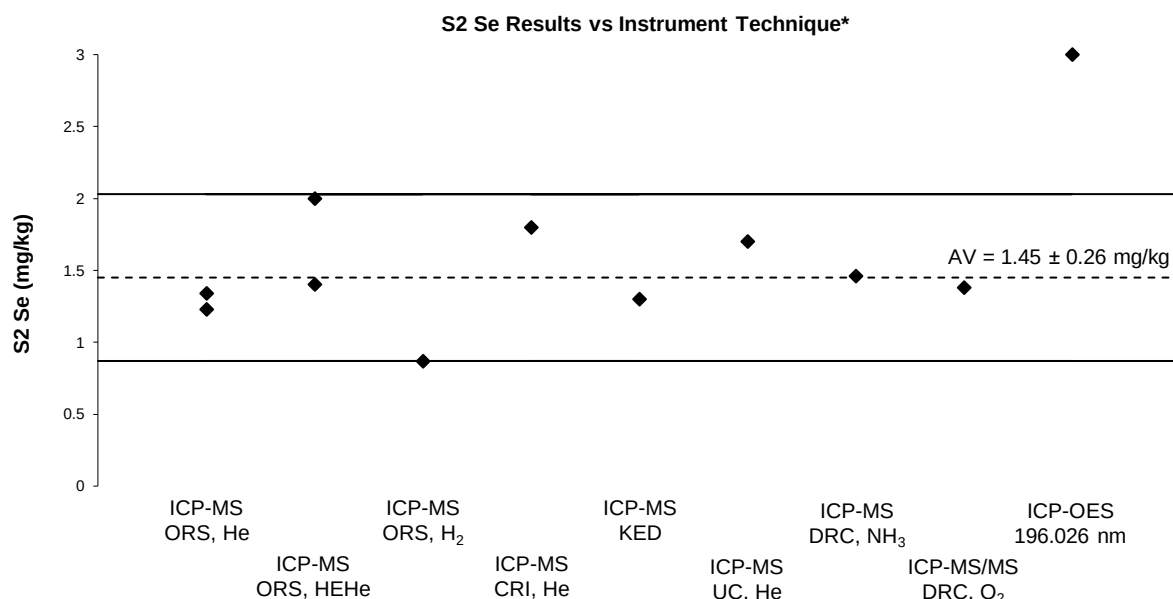


Figure 70 S1 and S2 Rb z-Scores vs. Instrumental Technique

Selenium Of 25 laboratories who reported results for acid extractable elements in S2 only 11 reported results for Se; 10 of these 11 laboratories performed acceptably. Selenium level in S2 was low at 1.45 mg/kg and challenged participants' analytical techniques. Laboratories used various instrumental techniques: ICP-MS in collision mode, ICP-MS in high energy mode, ICP-MS in reaction mode with H₂ or NH₃ as reaction gases or ICP-OES with wavelength

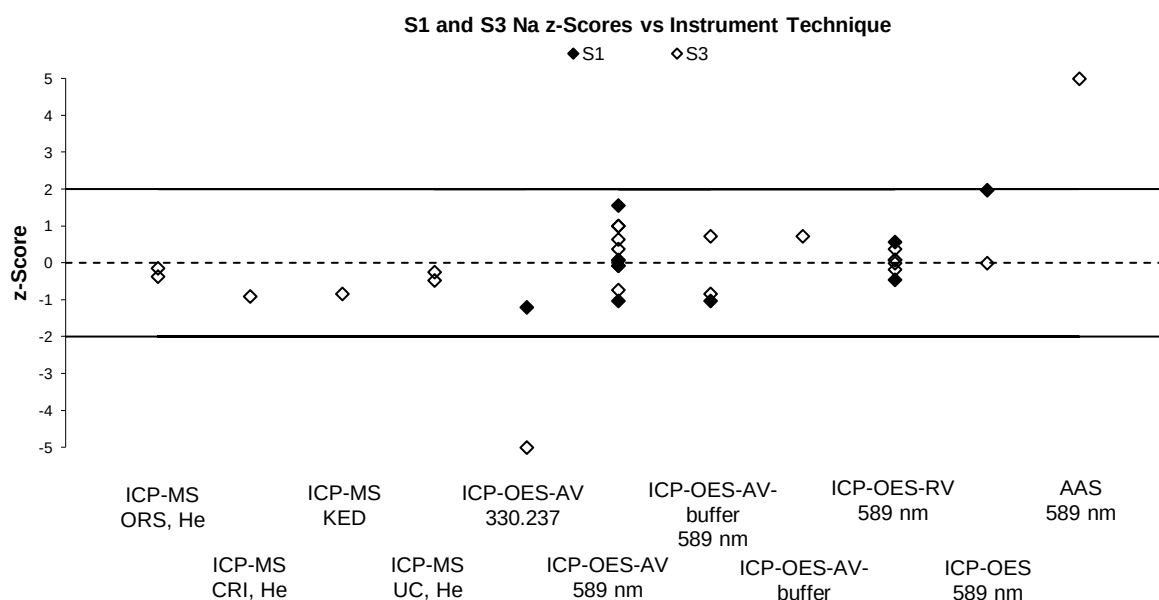
196.026 nm (Figure 71). The Se concentration might be too low for ICP-OES measurements with wavelength of 196.026 nm.



*Laboratory 14 result of 5.23 mg/kg has been plotted as 3 mg/kg. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2.

Figure 71 S2 Se Results vs. Instrumental Technique

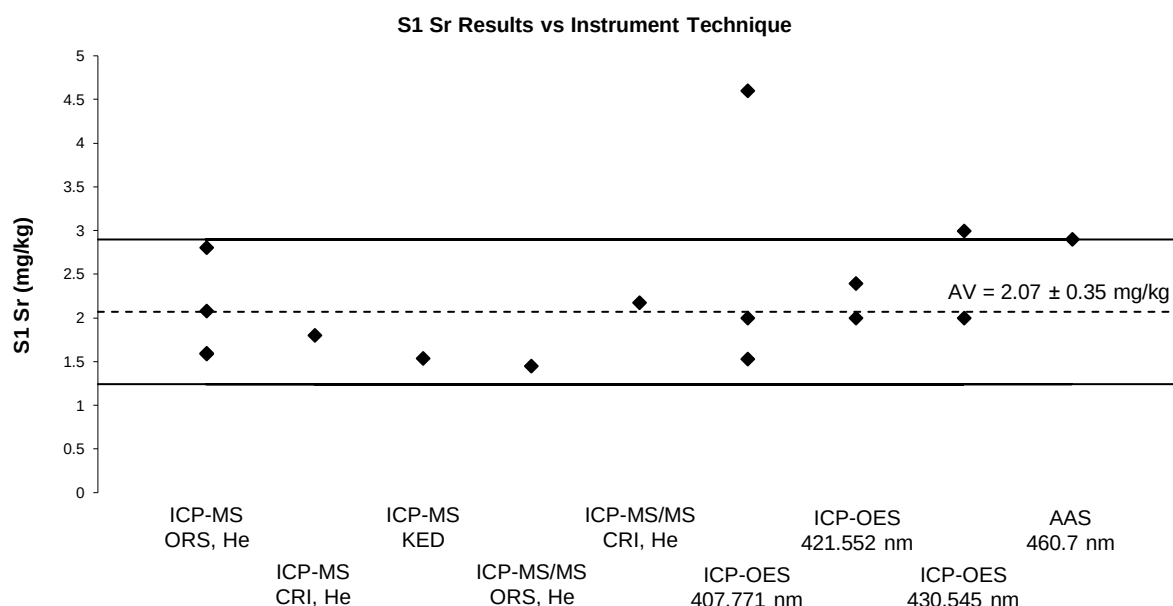
Sodium Participants' performance versus the instrumental techniques used for Na measurement in S1 and S3 are presented in Figure 72. ICP-OES with the wavelength 589 nm was the preferred measurement technique. ICP-OES with wavelength 330 nm has a low sensitivity and is not recommended for measurement of Na at low level.



*The z-score of 17.7 was plotted as 5 and the z-score of -5.48 was plotted as -5.

Figure 72 S1 and S3 Na z-Scores vs. Instrumental Technique

Strontium 17 laboratories reported results for this test and 16 performed acceptably. Figure 73 presents Sr results versus instrumental techniques used.



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

Figure 73 S1-Sr Results vs. Instrumental Technique

6.6 Participants' Results and Analytical Methods for 2M KCl Extractable Ammonium-N and Nitrate-N

Mineral nitrogen components, ammonium (NH_4^+), nitrite (NO_2^-) and nitrate (NO_3^-), are of particular interest when soil fertility is assessed. While water can extract NO_3^- -N and NO_2^- -N from a majority of soils, NH_4^+ -N has to be displaced by another cation when the surface soil colloids are negatively charged.²⁵

The participating laboratories were asked to analyse the sample using their normal measurement technique, but to follow the preparation procedure for the soil extract which involved a soil/2M KCl ratio of 1:10 and a mixing time of one hour. The method descriptions provided by participants are presented in Table 3. All but 2 participants used a soil/2M KCl ratio of 1:10.

2M KCl Extractable Ammonium-Nitrogen Plots of participants' results versus the analytical methods and instrumental technique used are presented in Figure 74. Compared to previous studies where all laboratories used the salicylate method or the phenate method, in the present study, two laboratories reported ammonium-nitrogen measurements in soil using the *o*-phthalaldehyde method with SFA or FIA determination. All results were in excellent agreement with each other but one.

2M KCl Extractable Nitrate-Nitrogen Many more laboratories are using the Vanadium (III) Chloride for NO_3^- -N reduction to NO_2^- -N (five) than in previous studies (Figure 75). All results were in excellent agreement with each other but one.

6.7 Participants' Results and Analytical Methods for Total Kjeldahl Nitrogen

TKN The two questionable TKN results were from Titrimetric measurements (Figure 76).

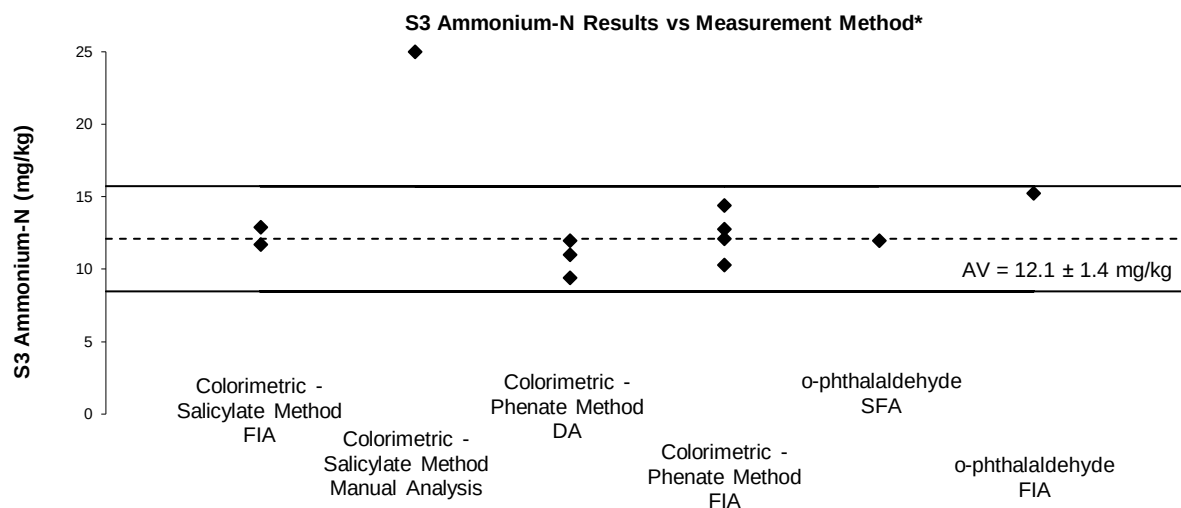
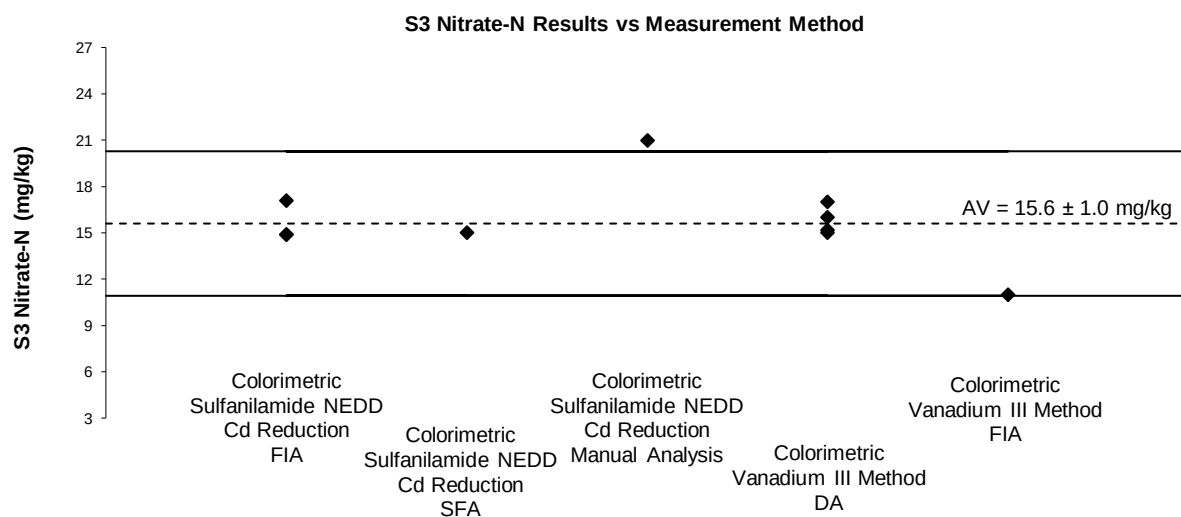
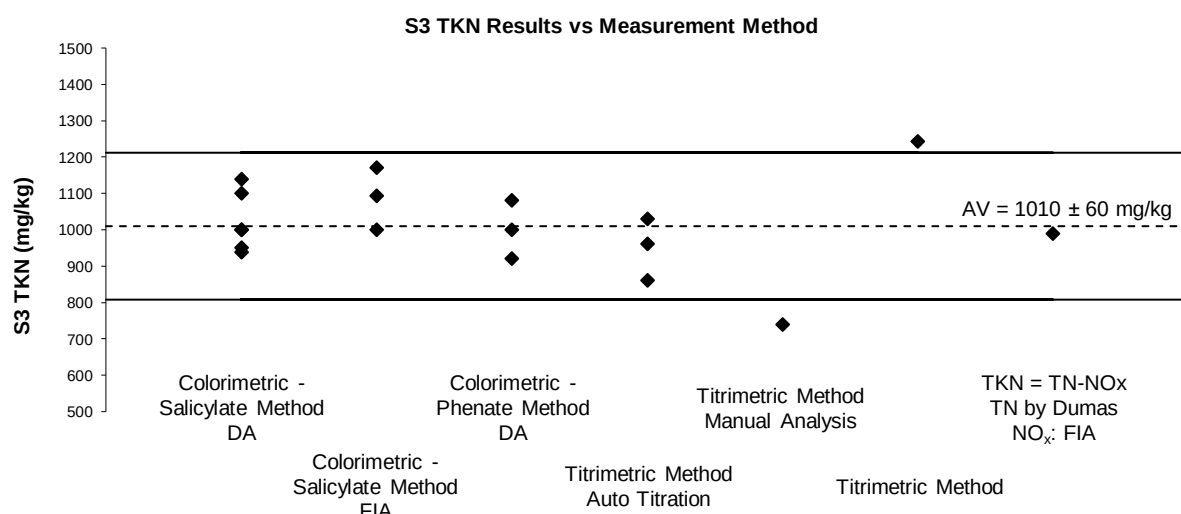


Figure 74 S3-NH₄⁺-N Results vs. Analytical Method and Measurement Technique



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

Figure 75: S3-NO₃⁻-N Results vs. Measurement Technique



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

Figure 76 S3-TKN Results vs. Measurement Technique

6.8 Participants' Results and Analytical Methods for Water Soluble Anions

Measurement of water-soluble anions in soil is an empirical procedure – where the method of extraction defines the measurand.^{24, 25} With testing laboratories using different methods, each could be considered to be measuring a different measurand that is their version of 'water soluble anions in soil'. This lack of uniformity in the procedures can make the comparison of participants' results difficult.

In a previous study of metals and anions in soil AQA 11-12, NMI conducted a study on water soluble anions content in soil using the same instrumental technique on two extraction procedures: one involved a soil/water ratio of 1: 5 and the other a soil/water ratio of 1:10. The fluoride, orthophosphate and sulphate results were found to change in direct proportion with the amount of water used in the extraction procedure.

In the present study participating laboratories were asked to analyse the sample using their normal measurement technique but to follow the same preparation procedure for the soil extract which involved: a soil/water ratio of 1:5 and a mixing time of one hour.

The method descriptions and instrumental techniques provided by participants are presented in Tables 5 to 7. All participating laboratories used a soil/water ratio of 1:5.

Individual Water-Soluble Anion Commentary

Bromide Only 5 laboratories reported results for bromide in S3. All reported results were in good agreement with each other and with the homogeneity value (H.V.) of 3 mg/kg. All laboratories used the Ion chromatographic Method (Figure 77).

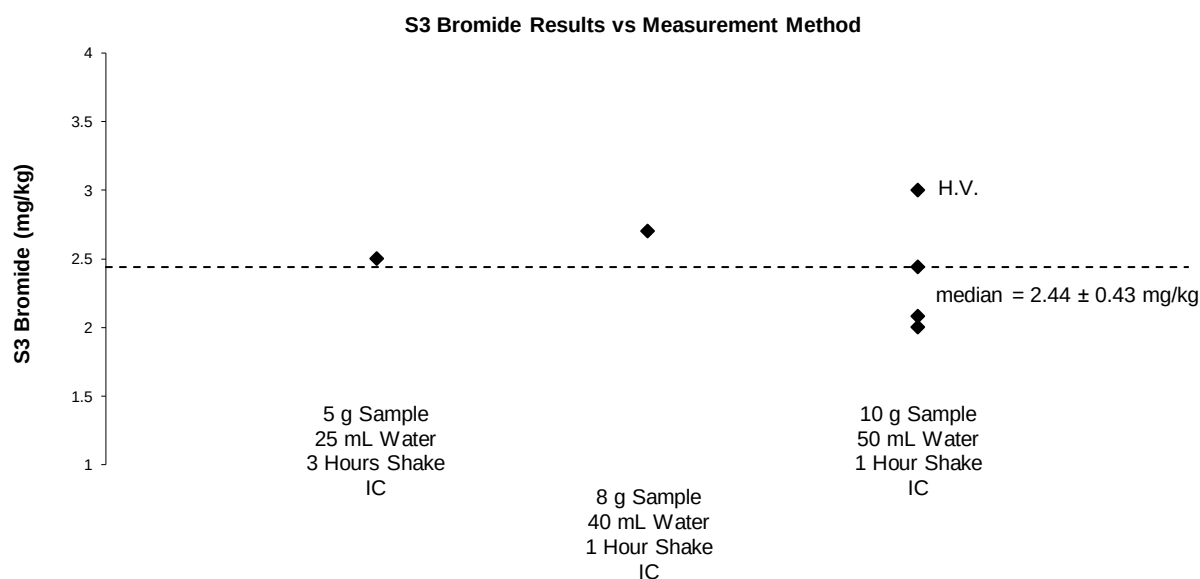
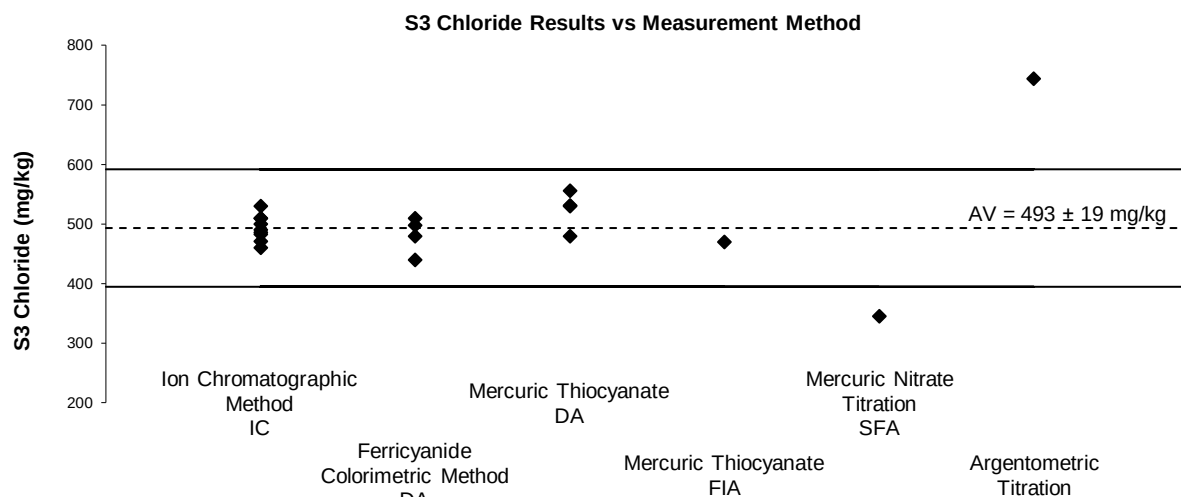


Figure 77 S3-Bromide Results vs. Method

Chloride Participants used various instrumental techniques for chloride measurement in S3, these are presented in Figure 78.

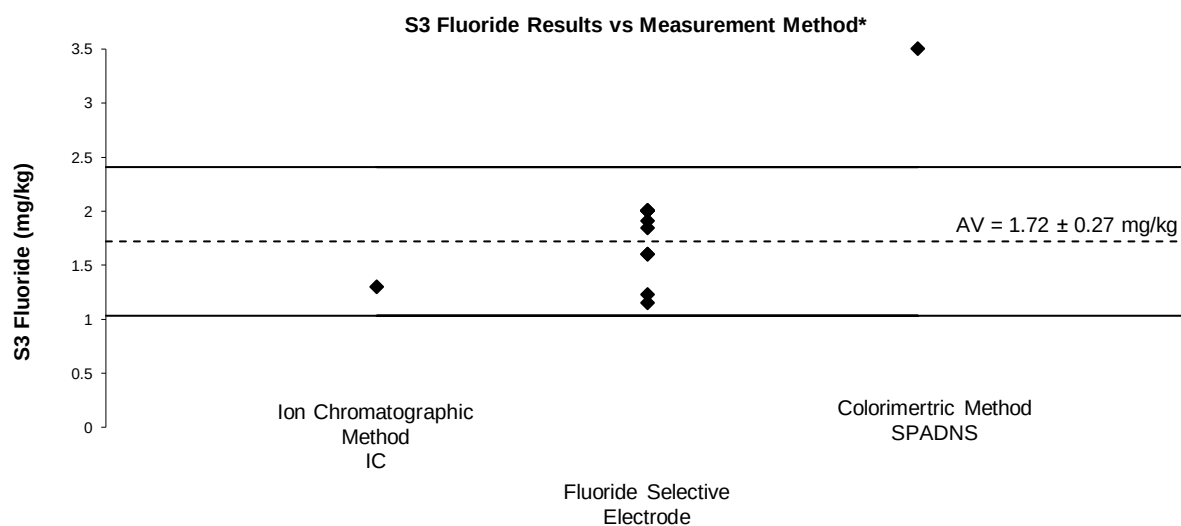
Fluoride Thirteen laboratories reported results for fluoride in S3 and performed acceptably except for one (Figure 79). Caution should be exercised when fluoride is measured by the colorimetric method as it suffers from interference from chlorides.²⁶

Iodide No assigned value could be set for iodide in S3 because only 5 laboratories reported results for this test. All 5 results were in good agreement with each other and with the median value of 1.32 mg/kg. (Figure 80).



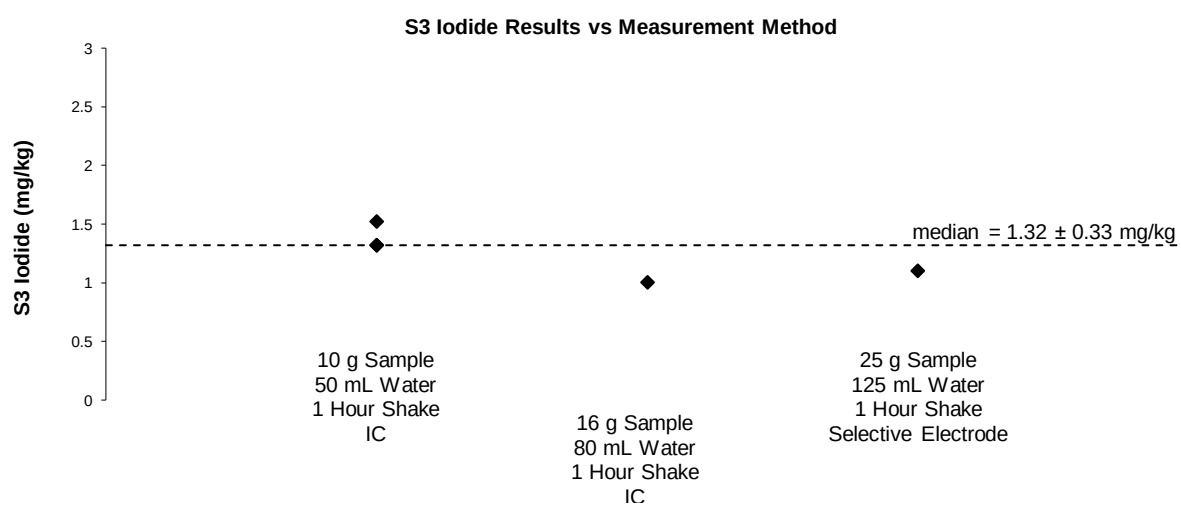
Horizontal lines on charts are the results corresponding to z-scores of 2 and -2.

Figure 78 S3-Chloride Results vs. Measurement Technique



*Laboratory 3 result of 4.98 mg/kg has been plotted as 3.5 mg/kg. Horizontal lines on charts are the results corresponding to z-scores of 2 and -2.

Figure 79 S3-Fluoride Results vs. Measurement Technique



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

Figure 80 S3-Iodide Results vs. Measurement Technique

Orthophosphate-P level in S3 was low, which may explain the variability in participants' results. Ascorbic acid colorimetric method was the most popular method used by participants for the measurement of orthophosphate-P (Figure 81).

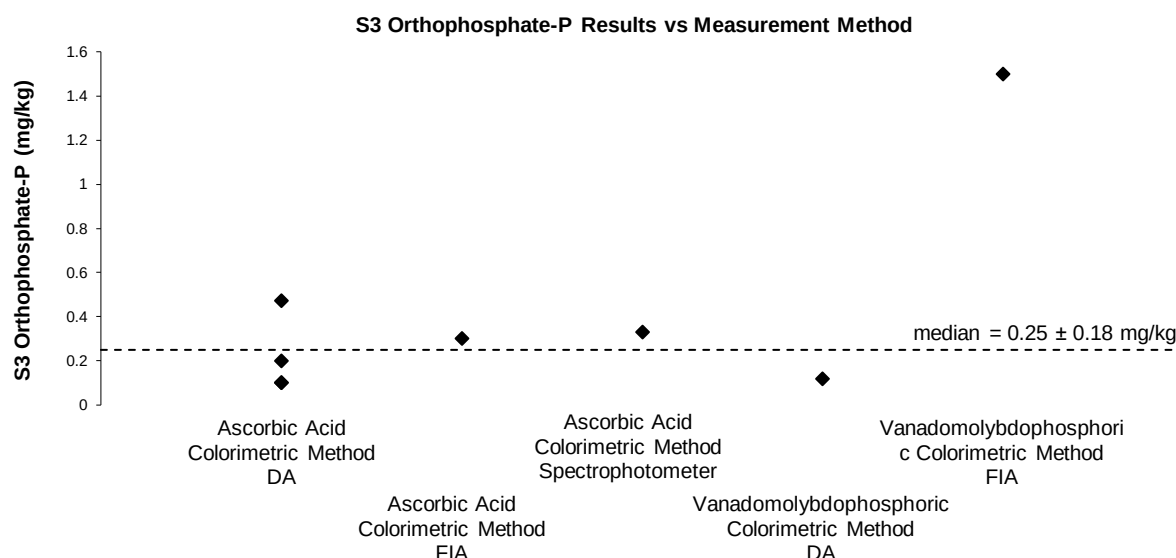
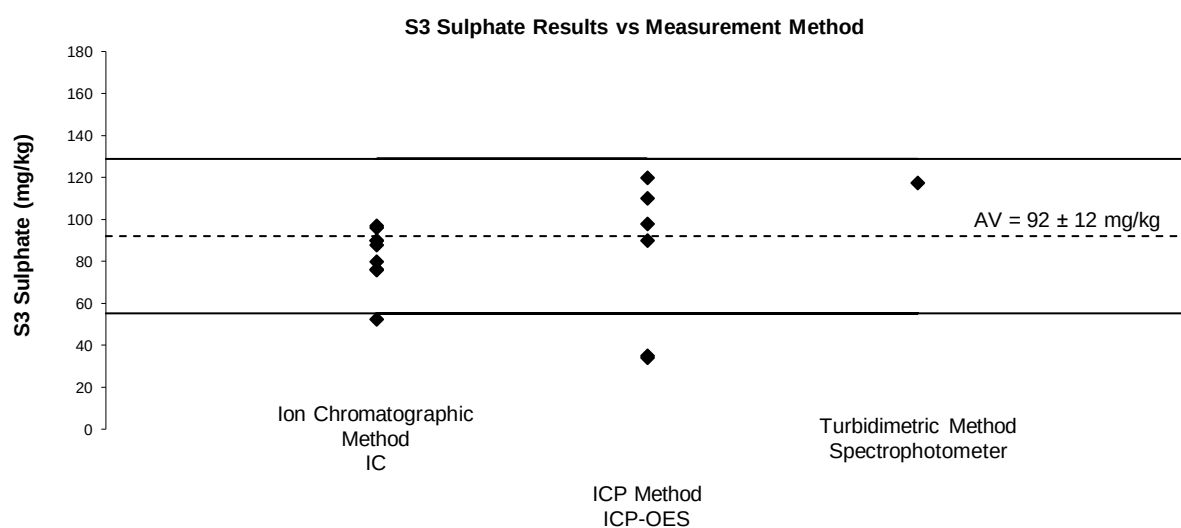


Figure 81 S3-Orthophosphate-P Results vs. Method

Sulphate A distribution of participants' results with the analytical method used is presented in Figure 82.



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

Figure 82 S3-Sulphate Results vs. Measurement Technique

Most of the results from ICP-OES measurements were biased high. False positive results can be produced when sulphate is measured by ICP-OES: this technique measures total S and not only S from sulphate compounds.

6.9 Comparison with Previous NMI Proficiency Tests Studies of Metals in Soil

AQA 24-15 is the 35th NMI proficiency study of inorganic analytes in soil. A summary of participants' performance over the last 12 years of studies is presented in Figure 83.

Over this period, the average proportion of acceptable scores was 90% for z-scores and 80% for E_n -scores.

Over time laboratories should expect at least 95% of its scores to lay within the range $|z| \leq 2.0$. Scores in the range $2.0 < |z| < 3.0$ occasionally can 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 are an indication of method or laboratory bias, even if all are within the acceptable range.

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.

6.10 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 73).

Table 73 Control Samples Used by Participants

Lab. Code	Description of Control Samples
1	CRM: AGAL-10/In-house SRMs
2	CRM
3	CRM: San Joaquin Soil, Clean Sandy Loam Soil
5	CRM: CRM036
6	CRM: SETOC-705 QC
7	CRM
8	CRM: Various
9	CRM: CRM016 Trace Metals – Fresh Water Sediment 3
12	CRM
13	RM: AGAL 12 (metals) In house AG reference
14	SS
15	CRM
16	CRM: ASPAC 10589-QC, ASPAC 7122-QC, ASPAC 9105, ASPAC 7468-QC, ASPAC 13959
17	CRM: NIST 2782 – Domestic Sludge
18	CRM: QCS-01-05 ICP Quality Control Standard #1; High Purity Standards CCV-1 Solution A; High Purity Standards CCV-1 Solution B; NMI AGAL-12 Biosoil; Australian Chemical Reagents Multi Element Standard; Australian Chemical Reagents Mixed Anion Standard; ERA Mercury WasteWatR
20	SS
23	CRM
24	RM
26	CRM: CRM ERA A Waters Company 540
29	SS

Matrix matched control samples taken through all steps of the analytical process, are most valuable quality control tools for assessing the 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 '*²⁷

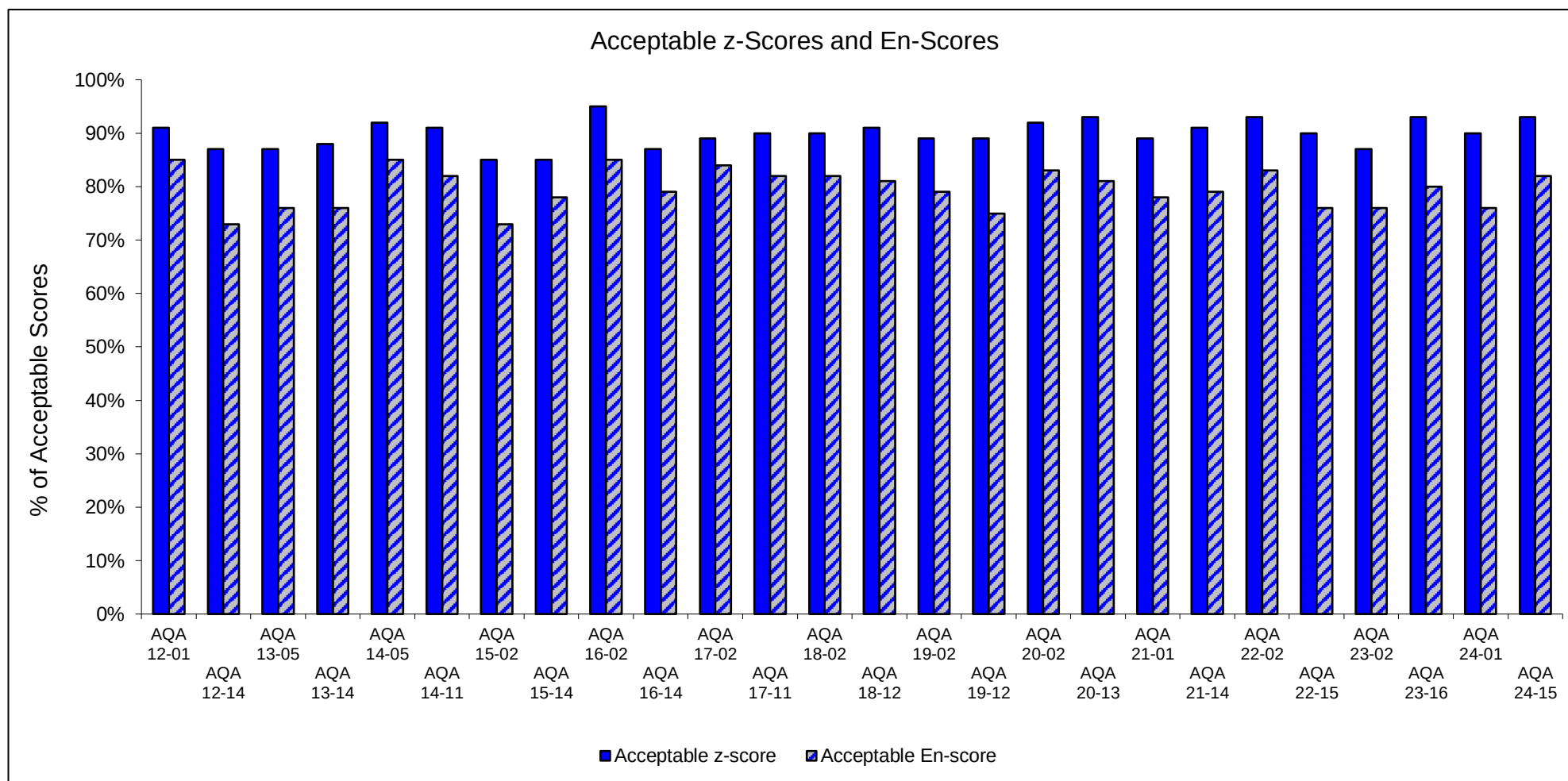


Figure 83 Participants' Performance over Time

7 REFERENCES

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

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APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING

Sample Preparation

Sample S1 was a sandy soil which was dried, ground and sieved prior to being thoroughly mixed and divided into portions of approximately 30 g each.

Sample S2 was a biosolid reference material previously prepared by NMI.

Sample S3 was a composite of agricultural soils given to the NMI for testing. The soil was dried, ground and sieved before mixing and dividing into portions of 75 g each.

Sample Analysis and Homogeneity Testing

The same preparation procedure as in previous NMI PT studies for inorganic analytes in soil was followed for Samples S1, S2 and S3. Partial homogeneity test was conducted for sample S3 except for fluoride, iodide, orthophosphate-P and sulphur. Three bottles were analysed in duplicate, and the average of these results was reported as the homogeneity value.

Measurements were made under repeatability conditions in random order.

No homogeneity test was conducted for acid extractable elements in S2. This was a reference material prepared by NMI and the homogeneity of this material has been previously established.

A full homogeneity test was conducted for all acid extractable elements in Sample S1 except for Na, Rb and Sn. Participants' results gave no reason to question the homogeneity of these tests; all were in good agreement with each other but one. Homogeneity testing for this sample was based on that described by Thompson and Fearn,²⁷ which is also the procedure as described in the International Harmonised Protocol for Proficiency Testing.⁴ A minimum of 6 bottles from S1 were selected at random. Duplicate test-portions were taken from each bottle and the concentration of all targeted analytes measured. Measurements were made under repeatability conditions in random order. The data for the full homogeneity testing of sample S1 can be found below in Tables 74 - 88.

Table 74 Sample S1 Al Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	1250	1360
10	1340	1230
16	1540	1330
22	1390	1340
32	1470	1430
35	1390	1510
40	1220	1400
Mean	1370	
CV	13%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.37	0.73	Pass
s_{an}/σ	0.44	0.50	Pass
s^2_{sam}	1060	20000	Pass

Table 75 Sample S1 As Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	0.95	1.00
10	0.90	0.82
16	1.11	0.94
22	1.05	0.92
32	1.02	0.98
35	0.92	1.04
40	0.85	0.96
Mean	0.96	
CV	16%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.35	0.73	Pass
s_{an}/σ	0.39	0.50	Pass
s^2_{sam}	0.0010	0.010	Pass

Table 76 Sample S1 B Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	0.94	1.00
10	1.05	1.00
16	1.38	1.06
22	1.06	1.13
32	1.09	1.04
35	1.21	1.05
40	1.09	1.08
Mean	1.08	
CV	18%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.73	0.73	Pass
s_{an}/σ	0.46	0.50	Pass
s^2_{sam}	0.0010	0.023	Pass

Table 77 Sample S1 Ba Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	13.7	14.5
10	14.5	16.6
16	16.7	13.9
22	14.4	14.7
32	15.4	17.0
40	13.3	16.2
Mean	15.1	
CV	19%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.34	0.78	Pass
s_{an}/σ	0.47	0.50	Pass
s^2_{sam}	0.00001	5.2	Pass

Table 78 Sample S1 Cd Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	0.0311	0.0326
16	0.0374	0.0328
22	0.0365	0.0347
32	0.0336	0.0384
35	0.0326	0.0338
40	0.0429	0.0375
Mean	0.0353	
CV	15%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.37	0.78	Pass
s_{an}/σ	0.37	0.50	Pass
s^2_{sam}	0.00001	0.00002	Pass

Table 79 Sample S1 Co Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	0.95	1.03
10	0.96	0.89
16	1.20	0.99
22	1.06	0.97
32	1.10	1.06
35	1.02	1.07
40	0.90	1.04
Mean	1.02	
CV	16%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.51	0.73	Pass
s_{an}/σ	0.39	0.50	Pass
s^2_{sam}	0.00063	0.017	Pass

Table 80 Sample S1 Cr Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	3.60	4.03
10	3.63	3.49
16	4.47	3.81
22	4.29	3.86
32	3.98	4.14
35	3.77	4.28
40	3.30	3.92
Mean	3.90	
CV	17%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.29	0.73	Pass
s_{an}/σ	0.42	0.50	Pass
s^2_{sam}	0.0032	0.27	Pass

Table 81 Sample S1 Cu Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	11.8	13.2
10	11.8	10.9
22	13.2	12.2
32	13.6	13.1
35	12.3	13.6
40	11.2	12.8
Mean	12.5	
CV	14%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.30	0.78	Pass
s_{an}/σ	0.45	0.50	Pass
s^2_{sam}	0.14	1.9	Pass

Table 82 Sample S1 Li Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	0.545	0.581
10	0.573	0.518
16	0.662	0.565
22	0.601	0.574
32	0.619	0.614
35	0.594	0.647
40	0.510	0.605
Mean	0.586	
CV	15%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.36	0.73	Pass
s_{an}/σ	0.37	0.50	Pass
s^2_{sam}	0.00004	0.0053	Pass

Table 83 Sample S1 Mn Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	18.2	18.7
10	17.6	16.7
16	21.6	18.0
22	19.7	17.6
32	19.1	19.0
35	18.3	20.6
40	16.1	18.9
Mean	18.6	
CV	16%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.41	0.73	Pass
s_{an}/σ	0.41	0.50	Pass
s^2_{sam}	0.00001	5.9	Pass

Table 84 Sample S1 Ni Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	1.41	1.56
10	1.44	1.29
16	1.75	1.44
22	1.50	1.48
32	1.55	1.56
35	1.45	1.57
40	1.29	1.45
Mean	1.50	
CV	16%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.53	0.73	Pass
s_{an}/σ	0.39	0.50	Pass
s^2_{sam}	0.00067	0.036	Pass

Table 85 Sample S1 Pb Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	32.1	35.2
10	33.4	31.0
16	40.1	37.5
22	38.2	34.4
32	38.9	38.5
35	33.5	38.8
Mean	36.0	
CV	13%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.43	0.78	Pass
s_{an}/σ	0.43	0.50	Pass
s^2_{sam}	4.4	15	Pass

Table 86 Sample S1 Sr Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	2.30	2.26
10	2.27	2.47
16	2.66	2.27
22	2.24	2.20
32	2.40	2.31
40	1.87	2.36
Mean	2.30	
CV	20%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.54	0.78	Pass
s_{an}/σ	0.50	0.50	Pass
s^2_{sam}	0.00001	0.13	Pass

Table 87 Sample S1 V Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	4.93	5.16
10	4.73	4.44
16	6.33	5.32
22	5.60	4.82
32	5.24	5.42
40	4.41	5.30
Mean	5.14	
CV	18%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.39	0.78	Pass
s_{an}/σ	0.45	0.50	Pass
s^2_{sam}	0.075	0.58	Pass

Table 88 Sample S1 Zn Homogeneity Testing

Container Number	Result (mg/kg)	
	Replicate 1	Replicate 2
5	12.3	11.4
10	10.5	9.8
16	13.5	11.0
22	12.3	11.1
32	12.2	12.4
40	11.7	12.0
Mean	11.7	
CV	15%	

Thompson and Fearn Homogeneity Tests²⁷

Test	Value	Critical	Result
Cochran	0.68	0.78	Pass
s_{an}/σ	0.49	0.50	Pass
s^2_{sam}	0.28	1.9	Pass

Sample Analysis for Acid Extractable Elements

The analysis for homogeneity were conducted by CRV section of NMI as per method NT2.49.²⁹ A test portion of approximately 0.7 g of soil for S1 and 0.5 g for S3 was weighed into a 50 mL graduated polypropylene centrifuge tube. The sample was 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.

The measurement instrument was calibrated using external standards for targeted analytes. A set of quality control samples consisting of blanks, blank matrix spike, and matrix matched reference materials, duplicates, and sample matrix spikes, was carried through the same set of procedures and analysed at the same time as the samples. A summary of the instrument conditions used, and the ion/wavelength monitored for each analyte is given in Table 89.

Table 89 Instrumental Technique used for Acid Extractable Elements

Analyte	Instrument	Internal Standard	Reaction/ Collision Cell	Cell Mode/Gas	S1 Final Dilution Factor	S3 Final Dilution Factor	Ion (m/z)/ Wavelength (nm)
Al	ICP-MS	Rh	NA	NA	800	NA	27 m/z
As	ICP-MS	Rh	ORS	He	800	NA	75 m/z
B	ICP-MS	Rh	NA	NA	800	NA	11 m/z
Ba	ICP-MS	Rh	ORS	He	800	NA	137 m/z
Ca	ICP-MS	Rh	ORS	He	NA	800	43 m/z
Cd	ICP-MS	Rh	NA	NA	800	NA	111 m/z
Co	ICP-MS	Rh	ORS	He	800	NA	59 m/z
Cr	ICP-MS	Rh	ORS	He	800	NA	52 m/z
Cu	ICP-MS	Rh	ORS	He	800	NA	65 m/z
Fe	ICP-MS	Rh	ORS	He	NA	800	56 m/z
K	ICP-MS	Rh	ORS	He	NA	800	39 m/z
Li	ICP-MS	Rh	ORS	He	800	NA	7 m/z
Mg	ICP-MS	Rh	ORS	He	NA	800	24 m/z
Mn	ICP-MS	Rh	ORS	He	800	NA	55 m/z
Na	ICP-MS	Rh	ORS	He	NA	800	23 m/z
Ni	ICP-MS	Rh	ORS	He	800	NA	60 m/z
P	ICP-MS	Rh	ORS	He	NA	800	31 m/z
Pb	ICP-MS	Ir	ORS	He	800	NA	208 m/z
Sr	ICP-MS	Rh	ORS	He	800	NA	88 m/z
V	ICP-MS	Rh	ORS	He	800	NA	51 m/z
Zn	ICP-MS	Rh	ORS	He	800	NA	66 m/z

NA= Not Applicable

Sample Analysis for Water Soluble Anions

Analyses for all the tests other than acid extractable elements were conducted by NMI Inorganics section.

A test portion of 10 g was weighed into a 50 mL polypropylene container. The container was then filled with deionised water. The suspension was shaken, at room temperature for 1 h, centrifuged, and filtered through 0.45 µm filter. A summary of the measurement methods and instrumental techniques is presented in Table 90.

Table 90 Summary of the Measurement Methods and Instrumental Techniques used by NMI

Anion	Measurement Method	Instrument
Ammonium-N	o-phthalaldehyde	SFA
Nitrate-N	Colorimetric Sulphanilamide NEDD Cd reduction	SFA
Total Kjeldahl Nitrogen	Titrimetric Method	Manual Analysis
Water Soluble Bromide	Ion Chromatographic Method	IC
Water Soluble Chloride	Ion Chromatographic Method	IC
Water Soluble Sulphate	Ion Chromatographic Method	IC

APPENDIX 2 - ASSIGNED VALUE, Z-SCORE AND E_N SCORE CALCULATION

The assigned value was calculated as the robust average using the procedure described in 'ISO13528(E), Statistical methods for use in proficiency testing by inter-laboratory comparisons – Annex C'.⁶ 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
 $S_{rob\ av}$ 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 91.

Table 91 Uncertainty of Assigned Value for As in Sample S1

No. results (p)	15
Robust Average	1.08 mg/kg
$S_{rob\ av}$	0.23 mg/kg
$u_{rob\ av}$	0.076 mg/kg
k	2
$U_{rob\ av}$	0.15 mg/kg

The assigned value for **As** in Sample S1 is **1.08 ± 0.15 mg/kg**.

z-Score and E_N-score

For each participant's result a z-score and E_N-score are calculated according to Equation 1 and Equation 2 respectively (see page 21). A worked example is set out below in Table 92.

Table 92 z-Score and E_N-score for As Result Reported by Laboratory 6 in S1

As Result mg/kg	Assigned Value mg/kg	Set Target Standard Deviation	z-Score	E _N -Score
0.98 ± 0.17	1.08 ± 0.15	20% as CV or 0.20 x 1.08 = =0.216 mg/kg	$z = \frac{(0.98 - 1.08)}{0.216}$ $z = -0.46$	$E_N = \frac{(0.98 - 1.08)}{\sqrt{0.17^2 + 0.15^2}}$ $E_N = -0.44$

APPENDIX 3 - USING PT DATA FOR UNCERTAINTY ESTIMATION

When a laboratory has successfully participated in at least 6 proficiency testing studies (e.g. is demonstrating control of bias and verification of repeatability), the standard deviation from proficiency testing studies (the reproducibility between laboratories variation) can also be used to estimate the uncertainty of their measurement results.^{10, 12} An example is given.

Between 2009 and 2024 NMI carried out 30 proficiency tests of metals in soil. These studies involved analyses of acid-extractable elements at low and high levels in dried soil, moist soil, biosolid, clay, compost, sediment and sludge.

Laboratory X submitted results for As in all of these studies. All reported results returned satisfactory z-scores. This data can usefully be separated into two ranges of results 1 to 10 mg/kg and 10 to 100 mg/kg (Tables 93 and 94). The pooled standard deviation of the robust CV over these PT samples for each concentration range gives estimates of the relative standard uncertainty of 13% and 9.6% respectively. Using a coverage factor of two gives relative expanded uncertainties of 26% and 20% respectively, at a level of confidence of approximately 95%.

Table 93 Laboratory X Reported Results for As at 1 to 10 mg/kg Level.

Study No.	Sample	Laboratory result mg/kg	Assigned value mg/kg	Number of laboratories	Robust CV of all results (%)
AQA 09-13	S1 – Biosolid	4.091	3.64	11	16
	S2 – Soil	4.29	4.57	12	15
AQA 11-01	S1 – Biosolid	3.54	3.57	18	20
AQA 13-05	S1 – Soil	9.22	9.21	22	14
AQA 14-11	S1 – Sediment	7.91	7.37	21	12
AQA 15-02	S1 – Moist Sludge	8.29	7.02	22	13
	S2 – Moist Sludge	7.42	7.02	17	11
AQA 15-14	S1 – Sediment	10	9.95	17	6.7
	S2 – Soil	4.53	4.47	14	6.4
AQA 16-02	S2 – Clay	2.67	2.11	20	14
AQA 16-14	S1 – Soil	6.03	5.61	17	20
AQA 17-02	S1 – Soil	3.71	3.76	13	10
	S2 – Soil	2.92	3.01	13	4
AQA 18-02	S1 – Compost	2.22	2.73	17	11
AQA 19-02	S1 – Soil	2.83	2.65	24	11
AQA 19-12	S1 – Soil	2.32	2.12	16	16
AQA 20-13	S1 – Biosolid	2.85	3.29	17	11
AQA 21-01	S1 – Sediment	7.02	6.26	18	6.9
	S2 – Moist Sludge	3.99	3.58	13	13
AQA 22-02	S1 – Sediment	4.32	4.02	15	9.5
	S2 – Moist Soil	3.57	3.56	13	6.2
AQA 22-15	S2 – Clay	4.29	3.63	19	17
AQA 23-02	S1 – Soil	4.41	4.12	16	5.9
	S2 – Sludge	4.43	4.8	8	24
AQA 24-15	S2 - Biosolid	3.70	3.83	20	10
Average					12%*
$pooled\ s\% = \sqrt{\frac{(11 - 1) \times 16^2 + (12 - 1) \times 15^2 + \dots + (20 - 1) \times 10^2}{413 - 25}}$					13%

* The pooled standard deviation was used.

Table 94 Laboratory X Reported Results for As at 10 to 100 mg/kg Level.

Study No.	Sample	Laboratory result mg/kg	Assigned value mg/kg	Number of Laboratories	Robust CV of all results (%)
AQA 10-12	S1 – Soil	16.6	14.4	19	8.5
AQA 11-12	S1 – Moist Sludge	25	21.6	13	15
AQA 12-01	S1 – Sediment	18.4	17.3	21	8.1
AQA 12-14	S2 – Soil	16.6	14.8	20	11
AQA 13-14	S1 – Sandy Soil	16.6	15.1	21	10
AQA 14-05	S1 – Soil	13.2	12.3	25	7.8
AQA 17-11	S1 – Sediment	18.1	17.4	22	11
AQA 18-12	S2 – Soil	10.4	9.6	20	8
AQA 19-12	S2 – Sediment	21	19.9	19	9
AQA 20-02	S1 – Soil	18.8	21.6	23	8.8
	S2 – Moist Soil	16.5	17.8	24	6.7
AQA 21-14	S1 – Sediment	19.5	20.9	21	8.9
AQA 22-15	S2 – Sediment	58.6	56.8	22	7.8
AQA 23-16	S1 – Soil	10.9	12.3	18	9.7
	S2 – Soil	12.4	12.3	17	9.4
AQA 24-01	S1 – Soil	35.9	35.2	20	10
	S2 – Moist Soil	11.8	12.5	16	13
Average					9.6%*
$pooled\ s\% = \sqrt{\frac{((19 - 1) \times 8.5^2 + (13 - 1) \times 15^2 + \cdots \cdots \cdots + (16 - 1) \times 13^2)}{341 - 17}}$					9.6%

* The pooled standard deviation was used

Table 95 sets out the expanded uncertainty for results of the measurement of As in soil, biosolid, clay, sediment, sludge, sandy soil, moist soil, compost and agricultural soil over the ranges 1 to 10 mg/kg and 10 to 100 mg/kg.

Table 95 Uncertainty of As Results Estimated Using PT Data.

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

The estimates of 26% and 20% relative passes the test of being reasonable, and the analysis of the 42 different PT samples over sixteen years can be assumed to include all the relevant uncertainty components (different matrices, operators, reagents, calibrators etc.), and so complies with AS ISO/IEC 17025.⁸

APPENDIX 4 - ACRONYMS AND ABBREVIATIONS

AAS	Atomic Absorption Spectroscopy
AOAC	Association of Official Agricultural Chemists
APHA	American Public Health Association
ASPAC	Australasian Soil and Plant Analysis Council
AV	Assigned Value
CITAC	Cooperation on International Traceability in Analytical Chemistry
CRI	Collision Reaction Interface
CRM	Certified Reference Material
CV	Coefficient of Variation
CVAAS	Cold Vapour Atomic Absorption Spectroscopy
CV _{rob}	Robust Coefficient of Variation
DA	Discreet Analyser
DRC	Dynamic Reaction Cell
EC	Electrical conductivity
FIA	Flow Injection Analyser
GUM	Guide to the Expression of Uncertainty in Measurement
HEHe	High energy He mode
HV	Homogeneity Value
ICP-MS	Quadrupole - Inductively Coupled Plasma - Mass Spectrometry
ICP-MS/MS	Inductively Coupled Plasma – Tandem Mass Spectrometry
ICP-OES	Inductively Coupled Plasma - Optical Emission 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-RV	Inductively Coupled Plasma - Optical Emission Spectrometry- radial view
IC	Ion chromatograph
IR	Infrared Detector
ISO/IEC	International Organisation for Standardisation / International Electrotechnical Commission
k	Coverage Factor
KED	Kinetic Energy Discrimination
Max	Maximum value in a set of results
Md	Median
Min	Minimum value in a set of results
MU	Measurement Uncertainty
M.V.	Median Value
N	Number of Participants
NATA	National Association of Testing Authorities
NIST	National Institute of Standards and Technology (of the United States of America)
NMI	National Measurement Institute (of Australia)
NR	Not Reported
NT	Not Tested
OPA	Orthophthaldialdehyde
ORS	Octopole Reaction System
PCV	Performance Coefficient of Variation
PFAS	Polyfluoroalkyl Substances

PT	Proficiency Test
RA	Robust Average
RM	Reference Material
CV_{rob}	Robust Coefficient of Variation
SD_{rob}	Robust Standard Deviation
SV	Spiked value or formulated concentration of a PT sample
SS	Spiked sample
SI	The International System of Units
s^2_{sam}	Sampling variance
s_a/σ	Analytical standard deviation divided by the target standard deviation
SFA	Segment Flow Analyser
SRM	Standard Reference Material (Trademark of NIST)
Target SD	Target standard deviation
TKN	Total Kjeldahl nitrogen
σ	Target standard deviation
UC	Universal Cell
USEPA	United States Environmental Protection Agency
UV-Vis	Ultraviolet and Visible Spectroscopy

APPENDIX 5 - INSTRUMENT DETAILS

Table 96 Instrument Conditions Ag

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
2	ICP-OES-AV	Eu				NA	328.069
4	ICP-MS	Indium	ORS	He	1000	NA	m/z 107
5	ICP-MS/MS	In		Standard Mode	10	NA	107
6	ICP-MS	Rh	KED	He	2000	NA	109
7	ICP-MS	103 Rh	DRC	He	NA	20	107
8	ICP-OES	Eu & Cs	NA	NA	50	NA	328.069nm
9	ICP-MS	Rh	CRI	He	100	NA	107
10	ICP-OES-AV	Lu	NA	NA	50	NA	328.289
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	107
12	ICP-OES-RV					NA	328.068
13	ICP-MS	Rh	NA	NA	625	NA	109
14	ICP-OES-AV	Lu			83	NA	328.068
15	ICP-MS	Rh		He		NA	
17	ICP-MS	Te-125	ORS	Standard Mode	40	NA	107
18	ICP-MS	Rh	ORS	He	500	NA	107 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	328.069nm
20	ICP-OES-AV	Lu	NA	NA	50	50	328.289
22	ICP-MS	103	ORS	He	0.036	NA	107
23	ICP-MS	Rh	ORS	He	800	NA	107
24	ICP-MS	Rh	ORS	He	50	NA	107
25	ICP-MS	Rh	NA		250	NA	
29	ICP-MS	Rh	CRI	He	500	NA	107
30	ICP-MS/MS	Rh	ORS	He	500	NA	107

Table 97 Instrument Conditions A1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			100	NA	309.3
2	ICP-OES-AV	Eu				NA	308.215
4	ICP-OES-AV	Lutetium	NA	NA	50-1000	NA	236.705nm
5	ICP-OES-AV-buffer	Y				NA	394.401
6	ICP-MS	Sc	KED	He	2000	NA	27
8	ICP-OES	Eu & Cs	NA	NA	50	NA	236.707, 308.215, 396.15nm
9	ICP-OES-AV	Te			100	NA	308.215
10	ICP-OES-AV	Lu	NA	NA	50	NA	236.705
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	27
12	ICP-OES-RV					NA	167.019
13	ICP-MS	Sc	UC	He	625	NA	27
14	ICP-OES-AV	Lu			83	NA	396.152
17	ICP-MS	Rh-103	ORS	Standard Mode	40	NA	27
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	308.215nm and 236.705nm
20	ICP-OES-AV	Lu	NA	NA	50	50	236.705
22	ICP-OES-AV	Lu			0.036	NA	237.312
23	ICP-MS	Rh	ORS	He	800	NA	27
24	ICP-MS	Sc	ORS	He	50	NA	27
25	ICP-MS	Sc	NA		250	NA	
30	ICP-MS/MS	Ge	ORS	He	5000	NA	27

Table 98 Instrument Conditions As

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	NA	193.7
2	ICP-MS	Sc,Ir,Rh				NA	75
4	ICP-MS	Germanium	ORS	He	1000	NA	m/z 75
5	ICP-MS/MS	Ge	CRI	He	10	NA	75
6	ICP-MS	Te	KED	He	400	NA	75
7	ICP-MS	72 Ge	DRC	He	20	20	75
8	ICP-OES	Eu & Cs	NA	NA	50	NA	188.89nm
9	ICP-MS	Ge	CRI	He	100	NA	75
10	ICP-OES-AV	Lu	NA	NA	50	NA	188.98
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	75
12	ICP-OES-RV					NA	188.98
13	ICP-MS	Rh	UC	He	625	NA	75
14	ICP-OES-AV	Lu			83	NA	188.98
15	ICP-MS	Rh		He		NA	
17	ICP-MS	Rh-103	ORS	HEHe	40	NA	75
18	ICP-MS	Rh	ORS	He	500	NA	75 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	188.980nm
20	ICP-OES-AV	Lu	NA	NA	50	50	188.98
22	ICP-OES-AV	Lu			0.036	NA	188.98
23	ICP-MS	Rh	ORS	He	800	NA	75
24	ICP-MS	Sc	ORS	He	50	NA	75
25	ICP-MS	Rh	UC	He	250	NA	
26	ICP-OES-AV	NA	NA			NA	
29	ICP-MS	Rh	CRI	He	500	NA	75
30	ICP-MS/MS	Ge	DRC	O2	500	NA	75-91

Table 99 Instrument Conditions B

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Lu			20	NA	249.772
2	ICP-OES-AV	Eu				NA	249.773
4	ICP-MS	Lithium	ORS	No Gas	1000	NA	m/z 11
5	ICP-OES-AV-buffer	Y				NA	208.957
6	ICP-MS	Sc	KED	He	1000/400	NA	11
7	ICP-MS	45 Sc	DRC	Other (No gas)	20	NA	11
8	ICP-OES	Eu & Cs	NA	NA	50	NA	249.773nm
9	ICP-OES-AV	Te			100	NA	249
10	ICP-OES-AV	Lu	NA	NA	50	NA	182.577
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	11
12	ICP-OES-RV					NA	249.678
13	ICP-MS	Sc	NA	NA	625	NA	10
14	ICP-OES-AV	Lu			83	NA	182.577
15	ICP-MS	Sc		He		NA	
17	ICP-MS	Rh-103	ORS	Standard Mode	40	NA	11
18	ICP-MS	Sc	ORS	He	500	NA	11 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	217.772nm
20	ICP-OES-AV	Lu	NA	NA	50	50	182.577
22	ICP-OES-AV	Lu			0.036	NA	208.956
23	ICP-MS	Rh	ORS	He	800	NA	11
24	ICP-MS	Sc	ORS	He	50	NA	11
25	ICP-MS	Sc	NA		250	NA	
29	ICP-MS	Sc	CRI	NA	500	NA	11
30	ICP-MS/MS	Ge	ORS	He	500	NA	11

Table 100 Instrument Conditions Ba

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	NA	553.6
2	ICP-OES-AV	Eu				NA	585.369
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	230.424nm
5	ICP-OES-AV-buffer	Y				NA	233.527
6	ICP-MS	Tb	KED	He	2000	NA	137
8	ICP-OES	Eu & Cs	NA	NA	50	NA	585.369nm
9	ICP-MS	Rh	CRI	He	100	NA	137
10	ICP-OES-AV	Lu	NA	NA	50	NA	230.424
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	137
12	ICP-OES-RV					NA	455.403
13	ICP-MS	Rh	NA	NA	625	NA	138
14	ICP-OES-AV	Lu			83	NA	493.408
17	ICP-MS	Te-125	ORS	Standard Mode	40	NA	137
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	585.369nm
20	ICP-OES-AV	Lu	NA	NA	50	50	230.424
22	ICP-OES-AV	Lu			0.036	NA	455.403
23	ICP-MS	Rh	ORS	He	800	NA	134Mini
24	ICP-MS	Ir	ORS	He	50	NA	137
25	ICP-MS	In	NA		250	NA	
30	ICP-MS/MS	Rh	ORS	He	500	NA	137

Table 101 Instrument Conditions Be

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
2	ICP-MS	Sc,Ir,Rh				NA	9
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	313.042nm
5	ICP-OES-AV-buffer	Y				NA	313.107
6	ICP-MS	Sc	KED	He	400	NA	9
7	ICP-MS	45 Sc	DRC	Other (No gas)	20	NA	9
8	ICP-OES	Eu & Cs	NA	NA	50	NA	313.042nm
9	ICP-MS	Ge	CRI	He	100	NA	9
10	ICP-OES-AV	Lu	NA	NA	50	NA	313.107
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	9
12	ICP-OES-RV					NA	
13	ICP-MS	Sc	NA	NA	625	NA	9
14	ICP-OES-AV	Lu			83	NA	313.042
15	ICP-MS	Sc		He		NA	
17	ICP-MS	Rh-103	ORS	Standard Mode	40	NA	9
18	ICP-MS	Sc	ORS	Standard Mode	500	NA	9 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	313.042
20	ICP-OES-AV	Lu	NA	NA	50	50	313.107
22	ICP-MS	6	ORS	He	0.036	NA	9
23	ICP-MS	Rh	ORS	He	800	NA	9
24	ICP-MS	Sc	ORS	He	50	NA	9
25	ICP-MS	Sc	NA		250	NA	
29	ICP-MS	Sc	CRI	NA	500	NA	9
30	ICP-MS/MS	Ge	ORS	He	500	NA	9

Table 102 Instrument Conditions Bi

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
2	ICP-MS	Sc,Ir,Rh	ORS	He		NA	209
4	ICP-MS	Lutetium	ORS	No Gas	1000	NA	m/z 209
6	ICP-MS	Tb	KED	He	400	NA	209
7	ICP-MS	175 Lu	DRC	He	NA	20	209
8	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	209 m/z
9	ICP-OES-AV	Te			100	NA	223.061
10	ICP-MS	Lu	ORS	standard mode	1000	NA	209
13	ICP-MS	Ir	NA	NA	625	NA	209
15	ICP-MS	Lu		He		NA	
17	ICP-MS	Ir-193	ORS	Standard Mode	40	NA	209
18	ICP-MS	Lu	ORS	He	500	NA	209 (m/z)
19	ICP-MS	Ir	ORS	He	NA	NA	209
20	ICP-OES-AV	Lu	NA	NA	50	50	315.887
22	ICP-MS	193	ORS	He	0.036	NA	209
23	ICP-MS	Rh	ORS	He	800	NA	209
24	ICP-MS	Ir	ORS	He	50	NA	209
25	ICP-MS	Ir	NA		250	NA	
29	ICP-MS	Lu	CRI	He	500	NA	209
30	ICP-MS/MS	Ir	ORS	He	500	NA	209

Table 103 Instrument Conditions Ca

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-RV	Lu			NA	50	317.933
2	ICP-OES-AV	Eu			NA		315.885
3	NA		NA	Other	NA	1	422.7
4	ICP-OES-AV	Lutetium	NA	NA	NA	50-500	315.887nm
5	ICP-OES-AV-buffer	Y			NA		315.887
7	ICP-MS	72 Ge	DRC	He	100	NA	40
8	ICP-OES	Eu & Cs	NA	NA	NA	50	315.887, 370.602nm
9	ICP-OES-AV	Y			NA	100	317.933
10	ICP-OES-AV	Lu	NA	NA	NA	1000	315.887
11	ICP-OES	Cs,Y	NA	NA	NA	1	317.993
12	ICP-OES-RV				NA		315.887
13	ICP-MS	Sc	UC	He	NA	625	44
14	ICP-OES-AV	Lu			NA	83	317.933
15	ICP-MS	Sc		H2	NA		
17	ICP-MS	Rh-103	ORS	Standard Mode	NA	160	44
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	315.887nm
20	ICP-OES-AV	Lu	NA	NA	50	50	228.802
22	ICP-OES-RV	Lu			NA	0.034	422.673
23	ICP-MS	Rh	ORS	He	NA	800	43Mini
24	ICP-MS	Sc	ORS	He	NA	50	44
29	ICP-MS	Sc	CRI	He	NA	500	40
30	ICP-OES-RV	Sc	NA		NA	500	317.933

Table 104 Instrument Conditions Cd

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	NA	228.8
2	ICP-OES-AV	Eu				NA	226.502
4	ICP-MS	Rhodium	ORS	He	1000	NA	m/z 111
5	ICP-MS/MS	In	CRI	He	10	NA	111
6	ICP-MS	Rh	KED	He	400	NA	111
7	ICP-MS	103 Rh	DRC	He	20	20	111&114
8	ICP-OES	Eu & Cs	NA	NA	50	NA	226.502nm
9	ICP-MS	Rh	CRI	He	100	NA	111
10	ICP-OES-AV	Lu	NA	NA	50	NA	228.802
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	111
12	ICP-OES-RV					NA	214.439
13	ICP-MS	Rh	NA	NA	625	NA	111
14	ICP-OES-AV	Lu			83	NA	214.439
15	ICP-MS	Rh		He		NA	
17	ICP-MS	Te-125	ORS	He	40	NA	111
18	ICP-MS	Rh	ORS	He	500	NA	111 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	226.502nm
20	ICP-OES-AV	Lu	NA	NA	50	50	228.615
22	ICP-MS	115	ORS	He	0.036	NA	111
23	ICP-MS	Rh	ORS	He	800	NA	111
24	ICP-MS	Rh	ORS	He	50	NA	111
25	ICP-MS	Rh	NA		250	NA	
26	ICP-OES-AV					NA	
29	ICP-MS	Rh	CRI	He	500	NA	111
30	ICP-MS/MS	Rh	ORS	He	500	NA	111

Table 105 Instrument Conditions Co

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	NA	240.7
2	ICP-MS	Sc,Ir,Rh				NA	59
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	231.160nm
5	ICP-MS/MS	Ga	CRI	He	10	NA	59
6	ICP-MS	Ga	KED	He	2000	NA	59
8	ICP-OES	Eu & Cs	NA	NA	50	NA	228.616nm
9	ICP-MS	Ge	CRI	He	100	NA	59
10	ICP-OES-AV	Lu	NA	NA	50	NA	228.615
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	59
12	ICP-OES-RV					NA	228.615
13	ICP-MS	Ge	UC	He	625	NA	59
14	ICP-OES-AV	Lu			83	NA	230.786
17	ICP-MS	Rh-103	ORS	He	40	NA	59
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	228.616nm
20	ICP-OES-AV	Lu	NA	NA	50	50	205.56
22	ICP-MS	72	ORS	He	0.036	NA	59
23	ICP-MS	Rh	ORS	He	800	NA	59
24	ICP-MS	Sc	ORS	He	50	NA	59
25	ICP-MS	Rh	UC	He	250	NA	
30	ICP-MS/MS	Ge	ORS	He	500	NA	59

Table 106 Instrument Conditions Cr

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	NA	357.9
2	ICP-OES-AV	Eu				NA	267.716
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	205.560nm
5	ICP-MS/MS	Ga	CRI	He	10	NA	52
6	ICP-MS	Sc	KED	He	400/1000	NA	52
7	ICP-MS	45 Sc	DRC	He	20	20	52
8	ICP-OES	Eu & Cs	NA	NA	50	NA	267.716nm
9	ICP-MS	Ge	CRI	He	100	NA	52
10	ICP-OES-AV	Lu	NA	NA	50	NA	205.56
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	52
12	ICP-OES-RV					NA	267.716
13	ICP-MS	Sc	UC	He	625	NA	52
14	ICP-OES-AV	Lu			83	NA	267.716
15	ICP-MS	Sc		He		NA	
17	ICP-MS	Rh-103	ORS	He	40	NA	52
18	ICP-MS	Sc	ORS	He	500	NA	52 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	267.716nm
20	ICP-OES-AV	Lu	NA	NA	50	50	324.754
22	ICP-MS	72	ORS	He	0.036	NA	52
23	ICP-MS	Rh	ORS	He	800	NA	52
24	ICP-MS	Sc	ORS	He	50	NA	52
25	ICP-MS	Sc	UC	He	250	NA	
26	ICP-OES-AV					NA	
29	ICP-MS	Sc	CRI	He	500	NA	52
30	ICP-MS/MS	Ge	ORS	He	500	NA	52

Table 107 Instrument Conditions Cs

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
2	ICP-MS	Sc,Ir,Rh	ORS	He		NA	133
4	ICP-MS	Lutetium	ORS	No Gas	1000	NA	m/z 133
6	ICP-MS	Tb	KED	He	2000	NA	133
8	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	133 m/z
10	ICP-MS	Ge	NA	NA	50	NA	107.846
17	ICP-MS	Te-125	ORS	Standard Mode	40	NA	133
19	ICP-MS	Rh	ORS	He	NA	NA	133
20	ICP-MS	Ge	ORS	standard mode	50	50	
22					0.036	NA	
23	ICP-MS	Rh	ORS	He	800	NA	133
24	ICP-MS	Rh	ORS	He	50	NA	133
25	ICP-MS	Rh	NA	He	250	NA	

Table 108 Instrument Conditions Cu

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	NA	324.7
2	ICP-OES-AV	Eu				NA	327.397
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	324.754nm
5	ICP-MS/MS	Ga	CRI	He	10	NA	63
6	ICP-MS	Ga	KED	He	1000	NA	63
8	ICP-OES	Eu & Cs	NA	NA	50	NA	327.395nm
9	ICP-OES-AV	Te			100	NA	217.895
10	ICP-OES-AV	Lu	NA	NA	50	NA	324.754
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	63
12	ICP-OES-RV					NA	324.754
13	ICP-MS	Ge	UC	He	625	NA	63
14	ICP-OES-AV	Lu			83	NA	327.395
17	ICP-MS	Rh-103	ORS	He	40	NA	63
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	327.397nm
20	ICP-OES-AV	Lu	NA	NA	50	50	324.754
22	ICP-MS	72	ORS	He	0.036	NA	63
23	ICP-MS	Rh	ORS	He	800	NA	63Mini
24	ICP-MS	Sc	ORS	He	50	NA	50
25	ICP-MS	Ga	UC	He	250	NA	
26	ICP-OES-AV					NA	
30	ICP-MS/MS	Ge	ORS	He	500	NA	63

Table 109 Instrument Conditions Fe

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			NA	50	372.1
2	ICP-OES-AV	Eu					259.939
4	ICP-OES-AV	Lutetium	NA	NA	50-1000	50-500	234.350nm
5	ICP-OES-AV-buffer	Y					273.955
6	ICP-MS	Sc	KED	He	2000	NA	56
7	ICP-MS	45 Sc	DRC	He	20	NA	56
8	ICP-OES	Eu & Cs	NA	NA	50	50	238.204, 258.588, 259.940nm
9	ICP-OES-AV	Y			1000	1000	240.489
10	ICP-OES-AV	Lu	NA	NA	5000	5000	261.382
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	1	56
12	ICP-OES-RV						234.35
13	ICP-MS	Sc	UC	He	625	625	56
14	ICP-OES-AV	Lu			83	83	238.204
15	ICP-MS	Sc		He			
17	ICP-MS	Rh-103	ORS	HEHe	40	160	56
18	ICP-MS	Sc	ORS	He	500	500	56 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	259.940nm and 258.588nm
20	CVAAS	NA	NA	NA	50	50	253.7
22	ICP-OES-AV	Lu			0.036	0.034	238.204
23	ICP-MS	Rh	ORS	He	800	800	56Mini
24	ICP-OES-AV	Eu			50	50	259.9
25	ICP-MS	Ga	UC	He	250	NA	
29	ICP-MS	Sc	CRI	He	500	500	56
30	ICP-MS/MS	Ge	ORS	He	5000	5000	56

Table 110 Instrument Conditions Hg

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
2	AAS					NA	253.7
4	CVAAS	NA	NA	NA	500	NA	253.7nm
5	CVAAS				10	NA	253.7
6	ICP-MS	Tb	KED	He	400	NA	201
7	ICP-MS	175 Lu	DRC	He	20	20	201&202
8	CETAC	NA	NA	NA	50	NA	253.7nm
9	ICP-MS	Ir	CRI	He	100	NA	202
10	CVAAS	NA	NA	NA	500	NA	253.7
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	201
12	ICP-OES-RV					NA	
13	ICP-MS	Ir	NA	NA	625	NA	201
14	CVAAS				83	NA	253.7
15	ICP-MS	Lu		He		NA	
17	ICP-MS	Ir-193	ORS	He	40	NA	201
18	ICP-MS	Lu	ORS	He	500	NA	202 (m/z)
19	CVAAS	NA	NA	NA	NA	NA	253.7nm
20	ICP-OES-AV	Lu	NA	NA	50	50	766.491
22	ICP-MS	193	ORS	He	0.036	NA	202
23	ICP-MS	Ir	ORS	He	800	NA	202
24	CVAAS	N\A			50	NA	253.7
25	ICP-MS	Ir	NA		250	NA	
26	CVAAS					NA	
29	ICP-MS	Lu	CRI	He	500	NA	202
30	ICP-MS/MS	Ir	ORS	He	500	NA	202

Table 111 Instrument Conditions K

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-RV	Lu			NA	50	766.49
2	ICP-OES-AV	Eu			NA		404.724
3	Other			Other	NA	1	766.5
4	ICP-OES-AV	Lutetium	NA	NA	NA	50-500	766.491nm
5	ICP-OES-AV-buffer	Y			NA		766.49
7	ICP-MS	72 Ge	DRC	He	100	NA	39
8	ICP-OES	Eu & Cs	NA	NA	NA	50	404.721nm, 766.491nm
9	ICP-OES-RV	Te			NA	100	766.491
10	ICP-OES-AV	Lu	NA	NA	NA	1000	766.491
11	ICP-OES	Cs,Y	NA	NA	NA	1	766.491
12	ICP-OES-RV				NA		766.491
13	ICP-MS	Sc	UC	He	NA	625	39
14	ICP-OES-AV	Lu			NA	83	769.897
15	ICP-MS	Sc		He	NA		
17	ICP-MS	Rh-103	ORS	He	NA	160	39
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	766.491nm
20	ICP-OES-AV	Lu	NA	NA	50	50	766.491
22	ICP-OES-RV	Lu			NA	0.034	766.491
23	ICP-MS	Rh	ORS	He	NA	800	39
24	ICP-MS	Sc	ORS	He	NA	50	39
29	ICP-MS	Sc	CRI	He	NA	500	39
30	ICP-OES-RV	Sc	NA		NA	500	766.491

Table 112 Instrument Conditions Li

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	NA	670.8
2	ICP-MS	Sc,Ir,Rh	ORS	He		NA	7
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	670.783nm
5	ICP-OES-AV-buffer	Y				NA	670.784
6	ICP-MS	Sc	KED	He	2000	NA	7
8	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	7 m/z
9	ICP-MS	Ge	CRI	He	100	NA	7
10	ICP-OES-AV	Lu	NA	NA	50	NA	670.783
11	ICP-OES	Cs,Y	NA	NA	1	NA	670.783
12	ICP-OES-RV					NA	670.783
13	ICP-MS	Sc	NA	NA	625	NA	7
14	NA	Lu			NT	NA	NT
17	ICP-MS	Rh-103	ORS	Standard Mode	40	NA	7
19	ICP-MS	Rh	NA	Standard Mode	NA	NA	7
20	ICP-OES-AV	Lu	NA	NA	50	50	670.783
22	ICP-MS	6	ORS	He	0.036	NA	7
23	ICP-MS	Rh	ORS	He	800	NA	7
24	ICP-MS	Sc	ORS	He	50	NA	7
25	ICP-MS	Sc	NA		250	NA	
30	ICP-MS/MS	Ge	ORS	He	500	NA	7

Table 113 Instrument Conditions Mg

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-RV	Lu			NA	50	279.077
2	ICP-OES-AV	Eu			NA		383.83
3	Other			Other	NA	20	285.2
4	ICP-OES-AV	Lutetium	NA	NA	NA	50-500	279.800nm
5	ICP-OES-AV-buffer	Y			NA		279.077
7	ICP-MS	72 Ge	DRC	He	100	NA	24
8	ICP-OES	Eu & Cs	NA	NA	NA	50	383.829nm
9	ICP-OES-AV	Y			NA	100	279.553
10	ICP-OES-AV	Lu	NA	NA	NA	1000	279.8
11	ICP-OES	Cs,Y	NA	NA	NA	1	285.213
12	ICP-OES-RV				NA		383.23
13	ICP-MS	Sc	UC	He	NA	625	25
14	ICP-OES-AV	Lu			NA	83	383.829
15	ICP-MS	Sc		He	NA		
17	ICP-MS	Rh-103	ORS	He	NA	160	24
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	383.829nm
20	ICP-OES-AV	Lu	NA	NA	50	50	279.8
22	ICP-OES-RV	Lu			NA	0.034	285.213
23	ICP-MS	Rh	ORS	He	NA	800	24
24	ICP-MS	Sc	ORS	He	NA	50	24
29	ICP-MS	Sc	CRI	He	NA	500	24
30	ICP-OES-RV	Sc	NA		NA	500	279.8

Table 114 Instrument Conditions Mn

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	40	279.5
2	ICP-OES-AV	Eu				NA	261.021
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	257.610nm
5	ICP-OES-AV-buffer	Y				NA	257.61
6	ICP-MS	Sc	KED	He	2000	NA	55
7	ICP-MS	45 Sc	DRC	He	20	20	55
8	ICP-OES	Eu & Cs	NA	NA	50	NA	261.021nm
9	ICP-OES-AV	Y			100	NA	257.61
10	ICP-OES-AV	Lu	NA	NA	50	NA	257.61
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	55
12	ICP-OES-RV					NA	257.61
13	ICP-MS	Sc	UC	He	625	NA	55
14	ICP-OES-AV	Lu			83	NA	260.568
15	ICP-MS	Sc		He		NA	
17	ICP-MS	Rh-103	ORS	He	40	NA	55
18	ICP-MS	Sc	ORS	He	500	NA	55 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	293.305nm
20	ICP-OES-AV	Lu	NA	NA	50	50	257.61
22	ICP-OES-AV	Lu			0.036	NA	257.61
23	ICP-MS	Rh	ORS	He	800	NA	55
24	ICP-MS	Sc	ORS	He	50	NA	55
25	ICP-MS	Rh	UC	He	250	NA	
29	ICP-MS	Sc	CRI	He	500	NA	55
30	ICP-MS/MS	Ge	ORS	He	5000	NA	55

Table 115 Instrument Conditions Mo

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
2	ICP-MS	Sc,Ir,Rh				NA	95
4	ICP-MS	Rhodium	ORS	He	1000	NA	m/z 95
5	ICP-MS/MS	Y		Standard Mode	10	NA	95
6	ICP-MS	Rh	KED	He	2000	NA	98
7	ICP-MS	103 Rh	DRC	He	20	20	95
8	ICP-OES	Eu & Cs	NA	NA	50	NA	202.032nm
9	ICP-MS	Rh	CRI	He	100	NA	95
10	ICP-OES-AV	Lu	NA	NA	50	NA	202.032
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	95
12	ICP-OES-RV					NA	202.032
13	ICP-MS	Rh	NA	NA	625	NA	95
14	ICP-OES-AV	Lu			83	NA	202.568
15	ICP-MS	Rh		He		NA	
17	ICP-MS	Te-125	ORS	Standard Mode	40	NA	95
18	ICP-MS	Rh	ORS	He	500	NA	95 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	202.032nm
20	ICP-OES-AV	Lu	NA	NA	50	50	202.032
22	ICP-MS	103	ORS	He	0.036	NA	95
23	ICP-MS	Rh	ORS	He	800	NA	95
24	ICP-MS	Rh	ORS	He	50	NA	95
25	ICP-MS	Rh	UC	He	250	NA	
29	ICP-MS	Rh	CRI	He	500	NA	95
30	ICP-MS/MS	Ge	ORS	He	500	NA	95

Table 116 Instrument Conditions Na

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-RV	Lu			20	50	589.592
2	ICP-OES-AV	Eu					589.593
3	Other			Other	NA	150	589
4	ICP-OES-AV	Lutetium	NA	NA	50	50-500	588.995nm
5	ICP-OES-AV-buffer	Y					589.592
6	ICP-MS	Sc	KED	He	2000	NA	23
7	ICP-MS	72 Ge	DRC	He	100	NA	23
8	ICP-OES	Eu & Cs	NA	NA	50	50	330.237, 589.592nm
9	ICP-OES-RV	Te			100	100	589.592
10	ICP-OES-AV	Lu	NA	NA	1000	1000	588.995
11	ICP-OES	Cs,Y	NA	NA	1	1	588.995
12	ICP-OES-RV						589.592
13	ICP-MS	Sc	UC	He	625	625	23
14	ICP-OES-AV	Lu			83	83	330.237
15	ICP-MS	Sc		He	NA		
17	ICP-MS	Rh-103	ORS	He	40	160	23
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	589.592nm
20	ICP-OES-AV	Lu	NA	NA	50	50	588.995
22	ICP-OES-AV	Lu			0.036	0.034	589.592
23	ICP-OES-AV	Y	NA	NA	800	800	588.995
24	ICP-MS	Sc	ORS	He	50	50	23
29	ICP-MS	Sc	CRI	He	NA	500	23
30	ICP-OES-RV	Sc	NA		500	500	588.995

Table 117 Instrument Conditions Ni

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	NA	232
2	ICP-OES-AV	Eu				NA	231.604
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	231.604nm
5	ICP-OES-AV-buffer	Y				NA	221.648
6	ICP-MS	Ga	KED	He	400/1000	NA	60
7	ICP-MS	72 Ge	DRC	He	20	20	60
8	ICP-OES	Eu & Cs	NA	NA	50	NA	231.604nm
9	ICP-OES-AV	Y			100	NA	216.555
10	ICP-OES-AV	Lu	NA	NA	50	NA	231.604
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	60
12	ICP-OES-RV					NA	216.555
13	ICP-MS	Ge	UC	He	625	NA	60
14	ICP-OES-AV	Lu			83	NA	231.604
15	ICP-MS	Sc		He		NA	
17	ICP-MS	Rh-103	ORS	He	40	NA	60
18	ICP-MS	Sc	ORS	He	500	NA	60 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	231.604nm
20	ICP-OES-AV	Lu	NA	NA	50	50	231.604
22	ICP-MS	72	ORS	He	0.036	NA	60
23	ICP-MS	Rh	ORS	He	800	NA	60
24	ICP-MS	Sc	ORS	He	50	NA	65
25	ICP-MS	Rh	UC	He	250	NA	
26	ICP-OES-AV					NA	
29	ICP-MS	Sc	CRI	He	500	NA	60
30	ICP-MS/MS	Ge	ORS	He	500	NA	60

Table 118 Instrument Conditions P

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Lu			NA	50	214.914
2	ICP-OES-AV	Eu			NA		185.827
3	Other		NA	Other	NA	1	880
4	ICP-OES-AV	Lutetium	NA	NA	NA	50-500	213.618nm
5	ICP-OES-AV-buffer	Y			NA		214.914
7	ICP-MS	72 Ge	DRC	He	100	NA	31
8	ICP-OES	Eu & Cs	NA	NA	NA	50	185.827nm
9	ICP-OES-RV	Te			NA	100	177.434
10	ICP-OES-AV	Lu	NA	NA	NA	1000	182.143
11	ICP-OES	Cs,Y	NA	NA	NA	1	213.618
13	ICP-MS	Sc	UC	He	NA	625	31
14	ICP-OES-AV	Lu			NA	83	213.618
15	ICP-OES-AV	Sc			NA		
17	ICP-MS	Rh-103	ORS	HEHe	NA	40	31
18	ICP-MS	Sc	ORS	He	NA	NA	31 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	185.827nm
20	ICP-OES-AV	Lu	NA	NA	50	50	182.143
22	ICP-OES-AV	Lu			NA	0.034	177.434
23	ICP-MS	Rh	ORS	He	NA	800	31
24	ICP-OES-AV	Eu			NA	50	185.8
29	ICP-MS	Sc	CRI	He	NA	500	31
30	ICP-OES-AV	Sc	NA		NA	500	213.618

Table 119 Instrument Conditions Pb

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	NA	283.3
2	ICP-OES-AV	Eu				NA	220.354
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	220.353nm
5	ICP-OES-AV-buffer	Y				NA	220.353
6	ICP-MS	Tb	KED	He	1000	NA	206+207+208
7	ICP-MS	175 Lu	DRC	He	20	NA	208
8	ICP-OES	Eu & Cs	NA	NA	50	NA	185.827nm
9	ICP-OES-AV	Te			100	NA	217
10	ICP-OES-AV	Lu	NA	NA	50	NA	220.353
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	208
12	ICP-OES-RV					NA	22.353
13	ICP-MS	Ir	NA	NA	625	NA	206+207+208
14	ICP-OES-AV	Lu			83	NA	220.353
15	ICP-MS	Lu		He		NA	
17	ICP-MS	Ir-193	ORS	Standard Mode	40	160	208
18	ICP-MS	Lu	ORS	He	500	NA	208 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	220.354nm
20	ICP-OES-AV	Lu	NA	NA	50	50	220.353
22	ICP-MS	193	ORS	He	0.036	NA	208
23	ICP-MS	Ir	ORS	He	800	NA	208
24	ICP-MS	Ir	ORS	He	50	NA	209
25	ICP-MS	Ir	NA		250	NA	
26	ICP-OES-AV					NA	
29	ICP-MS	Lu	CRI	He	500	NA	208
30	ICP-MS/MS	Ir	ORS	He	500	NA	206+207+208

Table 120 Instrument Conditions Rb

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
2	ICP-MS	Sc,Ir,Rh	ORS	He		NA	85
4	ICP-MS	Rhodium	ORS	He	1000	NA	m/z 85
6	ICP-MS	Rh	KED	He	2000	NA	85
8	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	220.353nm
10	ICP-MS	Ge	ORS	standard mode	1000	NA	85
19	ICP-MS	Rh	ORS	He	NA	NA	85
20	ICP-MS	Ge	ORS	standard mode	50	50	85
22					0.036	NA	
23	ICP-MS	Rh	ORS	He	800	NA	85
24	ICP-MS	Rh	ORS	He	50	NA	85
25	ICP-MS	Rh	NA		250	NA	

Table 121 Instrument Conditions S

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV	Lu			NA	50	181.975
2	ICP-OES-AV	Eu			NA		178.165
4	ICP-OES-AV	Lutetium	NA	NA	NA	50-500	181.972nm
5	ICP-OES-AV-buffer	Y			NA		181.975
7	ICP-MS	72 Ge	DRC	H2	100	NA	33&34
8	ICP-OES	Eu & Cs	NA	NA	NA	50	178.165,181.972nm
9	ICP-OES-AV	Te			NA	100	181.972
10	ICP-OES-AV	Lu	NA	NA	NA	1000	181.972
11	ICP-OES	Cs,Y	NA	NA	NA	1	181.972
13	ICP-OES-AV				NA	62.5	181.975
14	ICP-OES-AV	Lu			NA	83	181.972
15	ICP-OES-AV	Sc			NA		
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	191.972nm
20	ICP-OES-AV	Lu	NA	NA	50	50	181.972
22	ICP-OES-AV	Lu			NA	0.034	181.972
23	ICP-OES-AV	Y	NA	NA	NA	800	181.972
24	ICP-OES-AV	Eu	ORS	He	NA	50	182
30	ICP-OES-AV	Sc	NA	He	NA	500	180.669

Table 122 Instrument Conditions Sb

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
2	ICP-MS	Sc,Ir,Rh				NA	121
4	ICP-MS	Indium	ORS	No Gas	1000	NA	m/z 121
6	ICP-MS	Rh	KED	He	400	NA	121
7	ICP-MS	103 Rh	DRC	He	20	NA	121
8	ICP-OES	Eu & Cs	NA	NA	50	NA	178.165,181.972nm
9	ICP-MS	Rh	CRI	He	100	NA	121
10	ICP-OES-AV	Lu	NA	NA	50	NA	206.834
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	121
12	ICP-OES-RV					NA	217.582
13	ICP-MS	Rh	NA	NA	625	NA	121
14	ICP-OES-AV	Lu			83	NA	206.834
15	ICP-MS	Rh		He		NA	
17	ICP-MS	Te-125	ORS	Standard Mode	40	NA	121
18	ICP-MS	Rh	ORS	He	500	NA	123 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	206.834nm
20	ICP-OES-AV	Lu	NA	NA	50	50	206.834
22	ICP-MS	115	ORS	He	0.036	NA	121
23	ICP-MS	Rh	ORS	He	800	NA	121
24	ICP-MS	Rh	ORS	He	50	NA	121
25	ICP-MS	Rh	NA		250	NA	
29	ICP-MS	Rh	CRI	He	500	NA	123
30	ICP-MS/MS	Rh	ORS	He	500	NA	121

Table 123 Instrument Conditions Se

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
2	ICP-MS	Sc,Ir,Rh				NA	78
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	196.026nm
5	ICP-OES-AV-buffer	Y				NA	196.026
6	ICP-MS	Te	KED	He	400	NA	82
7	ICP-MS	72 Ge	DRC	HEHe	20	20	78
8	ICP-OES	Eu & Cs	NA	NA	50	NA	206.834nm
9	ICP-MS	Rh	CRI	He	100	NA	78
10	ICP-OES-AV	Lu	NA	NA	50	NA	196.026
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	78
12	ICP-OES-RV					NA	196.026
13	ICP-MS	Rh	DRC	NH3	625	NA	82
14	ICP-OES-AV	Lu			83	NA	196.026
15	ICP-MS	Rh		H2		NA	
17	ICP-MS	Te-125	ORS	HEHe	40	NA	78
18	ICP-MS	Rh	ORS	HEHe	500	NA	78 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	196.026nm
20	ICP-OES-AV	Lu	NA	NA	50	50	196.026
22	ICP-MS	72	ORS	H2	0.036	NA	78
23	ICP-MS	Rh	ORS	He	800	NA	78
24	ICP-MS	Sc	ORS	HEHe	50	NA	78
25	ICP-MS	Rh	UC	He	250	NA	
29	ICP-MS	Rh	CRI	H2	500	NA	78
30	ICP-MS/MS	Ge	DRC	O2	500	NA	80-96

Table 124 Instrument Conditions Sn

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	NA	286.3
2	ICP-OES-AV	Eu				NA	189.926
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	189.925nm
5	ICP-OES-AV-buffer	Y			10	NA	189.927
6	ICP-MS	Rh	KED	He	400	NA	120
8	ICP-OES	Eu & Cs	NA	NA	50	NA	189.926nm
9	ICP-MS	Rh	CRI	He	100	NA	118
10	ICP-OES-AV	Lu	NA	NA	50	NA	189.925
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	118
14	ICP-OES-AV	Lu			83	NA	189.925
17	ICP-MS	Te-125	ORS	He	40	NA	118
18	NA	NA	NA	NA	NA	NA	NA
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	189.926nm
20	ICP-OES-AV	Lu	NA	NA	50	50	189.925
22	ICP-OES-AV	Lu			0.036	NA	189.925
23	ICP-MS	Rh	ORS	He	800	NA	118
24	ICP-MS	Rh	ORS	He	50	NA	118
25	ICP-MS	Rh	NA		250	NA	
30	ICP-MS/MS	Rh	ORS	He	500	NA	118

Table 125 Instrument Conditions Sr

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	NA	460.7
2	ICP-MS	Sc,Ir,Rh				NA	88
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	407.771nm
5	ICP-MS/MS	Y	CRI	He	10	NA	88
6	ICP-MS	Rh	KED	He	400	NA	88
8	ICP-OES	Eu & Cs	NA	NA	50	NA	430.545nm
9	ICP-MS	Rh	CRI	He	100	NA	88
10	ICP-OES-AV	Lu	NA	NA	50	NA	421.552
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	88
13	ICP-MS	Rh	NA	NA	625	NA	88
14	ICP-OES-AV	Lu			83	NA	407.771
17	ICP-MS	Te-125	ORS	Standard Mode	40	NA	88
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	430.545nm
20	ICP-OES-AV	Lu	NA	NA	50	50	421.552
22	ICP-MS	72	ORS	He	0.036	NA	88
23	ICP-MS	Rh	ORS	He	800	NA	88
24	ICP-MS	Rh	ORS	He	50	NA	88
30	ICP-MS/MS	Ge	ORS	He	500	NA	88

Table 126 Instrument Conditions U

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
2	ICP-MS	Sc,Ir,Rh	ORS	He		NA	238
4	ICP-MS	Lutetium	ORS	No Gas	1000	NA	m/z 238
5	ICP-MS/MS	Ir		Standard Mode	10	NA	238
6	ICP-MS	Tb	KED	He	2000	NA	238
7	ICP-MS	175 Lu	DRC	He	NA	20	238
8	ICP-MS	Ir, Rh & Sc	NA	NA	50	NA	238 m/z
9	ICP-MS	Ir	CRI	He	100	NA	238
10	ICP-MS	Lu	ORS	standard mode	1000	NA	238
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	238
13	ICP-MS	Ir	NA	NA	625	NA	238
15	ICP-MS	Lu		He		NA	
17	ICP-MS	Ir-193	ORS	Standard Mode	40	NA	238
18	ICP-MS	Lu	ORS	He	500	NA	238 (m/z)
19	ICP-MS	Ir	ORS	He	NA	NA	238
20	ICP-MS	Lu	ORS	standard mode	50	50	
22	ICP-MS	193	ORS	He	0.036	NA	238
23	ICP-MS	Ir	ORS	He	800	NA	238
24	ICP-MS	Ir	ORS	He	50	NA	238
25	ICP-MS	Ir	NA		250	NA	
29	ICP-MS	Lu	CRI	He	500	NA	238
30	ICP-MS/MS	Ir	ORS	He	500	NA	238

Table 127 Instrument Conditions V

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	NA	318.4
2	ICP-MS	Sc,Ir,Rh				NA	51
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	292.401nm
5	ICP-MS/MS	Ga	CRI	He	10	NA	51
6	ICP-MS	Sc	KED	He	400	NA	51
8	ICP-OES	Eu & Cs	NA	NA	50	NA	311.837nm
9	ICP-MS	Ge	CRI	He	100	NA	51
10	ICP-OES-AV	Lu	NA	NA	50	NA	292.401
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	51
12	ICP-OES-RV					NA	292.401
13	ICP-MS	Sc	UC	He	625	NA	51
14	ICP-OES-AV	Lu			83	NA	292.401
17	ICP-MS	Rh-103	ORS	He	40	NA	51
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	311.839nm
20	ICP-OES-AV	Lu	NA	NA	50	50	292.401
22	ICP-OES-AV	Lu			0.036	NA	292.401
23	ICP-MS	Rh	ORS	He	800	NA	51
24	ICP-MS	Sc	ORS	He	50	NA	51
25	ICP-MS	Sc	UC		250	NA	
30	ICP-MS/MS	Ge	ORS	He	500	NA	51

Table 128 Instrument Conditions Zn

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	S1/S2 Final Dilution Factor	S3 Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	AAS	None			20	NA	213.9
2	ICP-OES-AV	Eu				NA	206.202
4	ICP-OES-AV	Lutetium	NA	NA	50	NA	206.200nm
5	ICP-OES-AV-buffer	Y				NA	213.857
6	ICP-MS	Ga	KED	He	1000	NA	66
7	ICP-MS	72 Ge	DRC	He	20	20	66
8	ICP-OES	Eu & Cs	NA	NA	50	NA	206.2, 334.502nm
9	ICP-OES-AV	Te			100	NA	334.502
10	ICP-OES-AV	Lu	NA	NA	50	NA	206.2
11	ICP-MS	Sc, Ga, Ge, Y, Rh, Ce, Ho, Ir	NA	He	1	NA	66
12	ICP-OES-RV					NA	206.2
13	ICP-MS	Ge	UC	He	625	NA	66
14	ICP-OES-AV	Lu			83	NA	213.857
15	ICP-MS	Sc		He		NA	
17	ICP-MS	Rh-103	ORS	He	40	NA	66
18	ICP-MS	Sc	ORS	He	500	NA	66 (m/z)
19	ICP-OES-AV-buffer	Eu	NA	NA	NA	NA	206.200nm
20	ICP-OES-AV	Lu	NA	NA	50	50	206.2
22	ICP-OES-AV	Lu			0.036	NA	206.2
23	ICP-MS	Rh	ORS	He	800	NA	64Mini
24	ICP-MS	Sc	ORS	He	50	NA	66
25	ICP-MS	Rh	UC		250	NA	
26	ICP-OES-AV					NA	
29	ICP-MS	Sc	CRI	He	500	NA	66
30	ICP-MS/MS	Ge	ORS	He	500	NA	66

END OF REPORT