



Australian Government
Department of Industry, Science,
Energy and Resources

National
Measurement
Institute

Proficiency Test Final Report AQA 20-09 Metals in Food

November 2020

ACKNOWLEDGMENTS

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

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

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

Luminita Antin

Jeffrey Merrick

Daniel Khaziran

Hamish Lenton

I would also like to thank Chad Jarolimek from CSIRO for reviewing this report.

Raluca Iavetz

Manager, Chemical Proficiency Testing

Phone: 61-2-9449 0111

proficiency@measurement.gov.au



Accredited for compliance with ISO/IEC 17043

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1 SUMMARY

This report presents the results of the proficiency test AQA 20-09 Inorganic Contaminants and Nutrients in Food. The study focused on the measurement of total: Ag, As, B, Ba, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, La, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Rb, S, Sb, Se, Sn, U, V and Zn in a freeze dried fish sample, and of total: Ag, Al, As, B, Ba, Ca, Cd, Co, Cr, Cs, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, S, Sb, Se, Sr, V, Zn, total kjeldahl nitrogen, total organic carbon and total ash in hemp flour.

Sixteen laboratories registered to participate and all submitted results.

The assigned values were the robust averages of participants' results, with the exception of As, Cd, Cr, Ni and Pb in the hemp Sample S2. The associated uncertainties were estimated from the robust standard deviation of the participants' results. However for As, Cd and Pb in S2 the assigned values were reference values measured using standard addition mass spectrometry. For Cr and Ni in S2 the assigned value was the reference values measured using isotope dilution mass spectrometry (IDMS). An information value by standard addition mass spectrometry (SA-ICP-MS) was also provided for both Al and V in S2 (Appendix 3).

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

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

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

Of 464 z-scores, 435 (94%) were satisfactory with |z| ≤ 2.0.

Of 464 E_n-scores, 404 (87%) were satisfactory with |E_n| ≤ 1.0.

- ii. *evaluate the laboratories' methods used in determination of total elements in food;*

Aluminium measurements are presenting difficulties to testing laboratories. Some participants may need to reassess their extraction method since they only recovered a fraction of Al from the hemp sample. According to Eurachem/CITAC Guide CG 4, laboratories should consider using matrix matched control samples to assess their extraction efficiency (the bias of their analytical methods). Bias can be expressed as recovery and should be corrected for or included in the uncertainty estimate.¹

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

Participants have improved their performance in the measurement of Cr and Ni in matrices with high silica content.

- iv. *develop the practical application of traceability and measurement uncertainty;*

Of 515 numerical results, 506 (98%) were reported with an expanded measurement uncertainty. The magnitude of these expanded uncertainties was within the range 1.5% to 146% of the reported value.

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

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

2 INTRODUCTION

2.1 NMI Proficiency Testing Program

The National Measurement Institute (NMI) is responsible for Australia's national measurement infrastructure providing a wide range of services, including a chemical proficiency testing program.

Proficiency testing (PT): "is evaluation of participant performance against pre-established criteria by means of interlaboratory comparison."² 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;
- PFOS/PFOA in water, soil, biota and food;
- allergens in food;
- controlled drug assay; and
- folic acid in flour.

AQA 20-09 is the fourteenth NMI proficiency test on inorganic contaminants and nutrients in food.

2.2 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 food;
- compare the performance of participant laboratories with their past performance; and
- develop the practical application of traceability and measurement uncertainty.
- produce materials that can be used in method validation and as control samples.

2.3 Study Conduct

The conduct of NMI proficiency tests is described in the NMI Chemical Proficiency Testing Study Protocol.³ The statistical methods used are described in the NMI Chemical Proficiency Statistical Manual.⁴ These documents have been prepared with reference to ISO Standard 17043² and The International Harmonised Protocol for Proficiency Testing of (Chemical) Analytical Laboratories.⁵

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

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

3 STUDY INFORMATION

3.1 Selection of Matrices and Inorganic Analytes

The fifty-eight tests in the study samples were representative of those commonly measured in food, and included toxic elements such as Cd and Pb and nutrient elements such as Na, P and Mg.

3.2 Participation

Sixteen laboratories participated and submitted results.

The timetable of the study was:

Invitation issued: 15 June 2020
Samples dispatched: 13 July 2020
Results due: 7 August 2020
Interim report issued: 11 August 2020

3.3 Test Material Specification

Two samples were provided for analysis:

- Sample S1 was 10 g of freeze dried fish; and
- Sample S2 was 30 g of hemp flour.

3.4 Laboratory Code

All participant laboratories were assigned a confidential code number.

3.5 Sample Preparation, Analysis and Homogeneity Testing

Test samples from previous studies have been demonstrated to be sufficiently homogeneous for the evaluation of participants' performance. Therefore, only a partial homogeneity test was conducted for all elements in S1 with the exception of S, as the same preparation procedure was followed in previous studies.² The results from the partial homogeneity testing for this sample are reported in the present study as the homogeneity value.

A full homogeneity test was conducted for all analytes in Sample S2 with the exception of Ag, S, TKN, TOC and total ash. Sample S2 was demonstrated to be sufficiently homogeneous for the evaluation of participants' performance.

The results returned by participants for S in S1 and S, TKN, TOC and total ash in S2 gave no reason to question the homogeneity of the test samples.

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

3.6 Stability of Analytes

No stability study was carried out during the period of the present study. Stability studies conducted for the previous proficiency tests of metals and nutrients in food found no significant changes in any of the analytes' concentration over the study period. Results of this study gave no reason to question the stability of the test samples.

3.7 Sample Storage, Dispatch and Receipt

Samples S1 and S2 were stored at room temperature before dispatch.

The samples were dispatched by courier on 13 July 2020.

A description of the test samples, instructions for participants, and a form for participants to confirm the receipt of the test samples, were included with the samples.

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

3.8 Instructions to Participants

Participants were instructed as follows:

- The samples should be stored during analysis at room temperature in a dry place e.g. desiccator with anhydrous calcium sulphate.
- Quantitatively analyse the samples using your normal test method.

- Participants are asked to report the results in units of **mg/kg** on as received basis for:

SAMPLE S1 (Fish)		SAMPLE S2 (Hemp)	
Test TOTAL	Approximate Conc. Range (as received basis) mg/kg	Test TOTAL	Approximate Conc. Range (as received basis) mg/kg
Ag	0.05-1	Ag	0.0001-0.002
As	1-20	Al	1-20
B	1-20	As	0.005-0.1
Ba	1-20	B	1-100
Ca	1000-20000	Ba	0.5-10
Cd	0.05-1	Ca	500-10000
Co	0.1-2	Cd	0.0005-0.01
Cr	0.5-10	Co	0.005-0.1
Cu	0.5-10	Cr	0.05-2
Fe	10-200	Cu	1-20
Hg	0.05-1	Cs	0.005-0.1
K	10000-200000	Fe	10-200
La	0.05-1	K	4000-80000
Li	0.05-1	Mg	1000-20000
Mg	1000-20000	Mn	10-200
Mn	0.5-10	Mo	0.05-1
Mo	0.01-0.2	Na	1-20
Na	2000-40000	Ni	0.1-2
Ni	0.05-1	P	4000-80000
P	8000-280000	Pb	0.005-0.1
Pb	0.05-1	S	NA
Rb	1-20	Sb	0.0005-0.01
S	NA	Se	0.005-0.1
Sb	0.05-1	Sr	5-100
Se	1-20	V	0.005-0.1
Sn	0.05-1	Zn	10-200
U	0.005-0.1	Total Kjeldahl Nitrogen	NA
V	0.05-1	Total Organic Carbon	NA
Zn	10-200	Total Ash at 600°C	NA

NA-Not Available

- Report results using the electronic results sheet emailed to you.
- Report results as you would report them to a client.
- Please send the requested details regarding the test method and the basis of your uncertainty estimate.

3.9 Interim Report

An interim report was e-mailed to participants on 11 August 2020.

4 PARTICIPANT LABORATORY INFORMATION

4.1 Test Method Summaries

Summaries of test methods are transcribed in Tables 1 to 4.

Table 1 Methodology for Total Elements in S1

Lab. Code	Method Reference	Sample Mass (g)	Temp. (°C)	Time (min)	Vol. HNO ₃ (mL)	Vol. HCl (mL)	Vol. HNO ₃ (1:1) (mL)	Vol. HCl (1:4) (mL)	Vol. H ₂ O ₂ (mL)	Other
1		0.2, 1.2	90	90	6	4				
2*	In house, hot block digestion	0.5 & 1	110	60	5	1.5				
3*	US EPA Method 6020A, Perkin Elmer Application Note 009036A_01, AOAC 2011.19 Cr,Se,Mo in infant formula, Perkin Elmer application Note - Determination of Mercury in waste water by ICP	0.5	180	30	6				1	
4	200.3 modified	0.2	120	240	6	1			2	
5	AOAC 990.08	0.25	85	240	3	2				
6	AOAC 984.27, USEPA 3050, USEPA 6010, USEPA 6020	0.5	99	60	4	12				4 (H ₂ O)
7		0.5	100	120	3	1				
9*		0.1	200	20	10					
11*	In-House	0.519	105	60	2.5	0.5				2 (H ₂ O)
12	In-house method. Microwave digestion for trace elements and Hot block digestion for major elements. Analysed by ICP-MS and ICP-OES	0.5	85-165	50	5					
13	AOAC 990.08	0.5	85	240	5	5				
14	USEPA method 200.2 Revision 2.8	1	95	60			2	10	2	
15	In House S6 (referencing APHA 3125)	0.4	120	60	2.5	7.5				
16*	Inhouse based on APHA 3125	0.5	95	120	8	1			2	

*Additional information in Table 5

Table 2 Methodology for Total Elements in S2

Lab. Code	Method Reference	Sample Mass (g)	Temp. (°C)	Time (min)	Vol. HNO ₃ (mL)	Vol. HCl (mL)	Vol. HNO ₃ (1:1) (mL)	Vol. H ₂ O ₂ (mL)	Other
2*	In house, hot block digestion	0.5 or 1	110	60	5	1.5			
3	US EPA Method 6020A, Perkin Elmer Application Note 009036A_01, AOAC 2011.19 Cr,Se,Mo in infant formula,	0.5	180	30	6			1	
5	AOAC 990.08	0.3	85	240	3	2			
6	AOAC 984.27, USEPA 3050, USEPA 6010, USEPA 6020	0.5	99		4	12			4 (H ₂ O)
7		0.5	100	120	3	1			

Table 2 Methodology for Total Elements in S2 (continued)

Lab. Code	Method Reference	Sample Mass (g)	Temp. (°C)	Time (min)	Vol. HNO ₃ (mL)	Vol. HCl (mL)	Vol. HNO ₃ (1:1) (mL)	Vol. H ₂ O ₂ (mL)	Other
8*	In house	0.7	90-98	60	3	3			
9*		0.1	200	18	10				
10		0.5				0.5	7	1	
12	In-house method. Microwave digestion for trace elements and Hot block digestion for major elements. Analysed by ICP-MS and ICP-OES	0.5	85-165	50	5				
13	AOAC 990.08	1	85	240	5	5			
15	In House S6 (referencing APHA 3125)	0.4	120	60	2.5	7.5			

*Additional information in Table 5

Table 3 Methodology for Total Kjeldahl Nitrogen in S2

Lab. Code	Method Reference	Measurement Method			Instrument
3	Australian Standard (AS) 2300.1.2.1 1991	Prot01			Tecator Kjeltec
5	AS 2300.1.2.1				Kjeldahl
7	APHA 4500-N B and C			Sulphuric acid digestion	Digestion Block and Auto-titration Unit
8	In house				DA
10	APHA Method 4500-N Org B and Method 4500-NH3 F			Colorimetric – phenate method	DA
15	Rayment & Lyons 7A5			Dumas combustion	LECO

Table 4 Methodology for Total Organic Carbon in S2

Lab. Code	Method Reference	Measurement Method		Instrument
7		High temp. combustion		TOC analyser
8	In house			Combustion
10	Rayment & Lyons, 2011, "Soil Chemical Methods-Australasia" 6B3	High Temperature Oxidation		LECO
15	Rayment & Lyons 6B2	Dumas combustion		LECO

4.2 Instruments Used for Measurements

The instruments and settings used by participants are presented in Appendix 5.

4.3 Additional Information

Participants had the option to report additional information for each sample analysed. These are transcribed in Table 5.

Table 5 Additional Information

Lab Code	Additional Information
2	Methodology for S1 and S2: 1g used for all elements except Se, 0.5g used for Se.
3	Methodology for S1: Mercury digestion performed using hot block at 95C, digested for 45 mins 3.5mL HNO ₃ , 3.5mL H ₂ O ₂ , 0.5g sample size.
8	Methodology for S2: Cold digest for 2 hr first.
9	Sample S1: Our laboratory noticed that microwave digestion of sample S1 at 200°C gave higher results than a block digestion at 95°C for the following elements: Cr, Mo, Na, Ni, Rb and Sn. Sample S2: We noticed that microwave digestion of sample S2 at 200°C gave higher results than a block digestion at 95°C for the following elements: Al, Cr, Cs, Mo, Na and Ni. Values for Sb did not appear homogeneous. Methodology for S1 and S2: We also did a block digest at 95°C to compare with our routine 200°C microwave digestion. Conditions of the block digest were 95°C for 30 minutes followed by 60 minutes at 60°C.
11	Sample S1: Storage: At room temperature, not stored in a desiccator. Se result analysed 27/07/2020 - re-digested on 24/07/20 through TMAH Digestion. Methodology for S1: Note: Selenium were prepared using a different extraction method: sample mass = 0.502 g, extraction with 5 mL of 12.5% Tetramethylammonium hydroxide, digestion temp 115°C for 60 mins.
16	Sample S1: Boron have been tested but did not obtain results that passed QC in time for submission. Methodology for S1: H ₂ O ₂ is added after the first hour. The second hour is only heated to 85 degrees C.

4.4 Basis of Participants' Measurement Uncertainty Estimates

Participants were requested to provide information about the basis of their uncertainty estimates (Table 6).

Table 6 Basis of Uncertainty Estimate

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

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation ^a		Guide Document for Estimating MU
		Precision	Method Bias	
			PT Studies Recoveries of SS	
14	Top Down – precision and estimates of the method and laboratory bias	Control Samples – RM Duplicate Analysis		Eurachem/CITAC Guide
15	Top Down – precision and estimates of the method and laboratory bias	Control Samples – RM Duplicate Analysis	Instrument Calibration Standard Purity	Nordtest Report TR537
16	Top Down – precision and estimates of the method and laboratory bias	Duplicate Analysis	CRM Laboratory Bias from PT Studies	IANZ Technical Guide

^aRM = Reference Material, CRM = Certified Reference Material, SS = Spiked Samples.

4.5 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. Participants' comments are reproduced in Table 7.

Table 7 Participants' Comments

Participants' Comments	Study Co-ordinator's Response
Values for Sb did not appear homogeneous.	A full homogeneity test was conducted for Sb in S2. This sample has been found to be sufficiently homogeneous for Sb for the evaluation of participants' results. Caution has to be exercised when a small sample size (0.1 g) is used for analysis as this may not be representative of the whole sample.

5 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

5.1 Results Summary

Participants' results are listed in Tables 8 to 63 with results' summary statistics: robust average, median, maximum, minimum, robust standard deviation (SD_{rob}) and robust coefficient of variation (CV_{rob}). Bar charts of the results and performance scores are presented in Figures 2 to 57. An example chart with interpretation guide is shown in Figure 1.

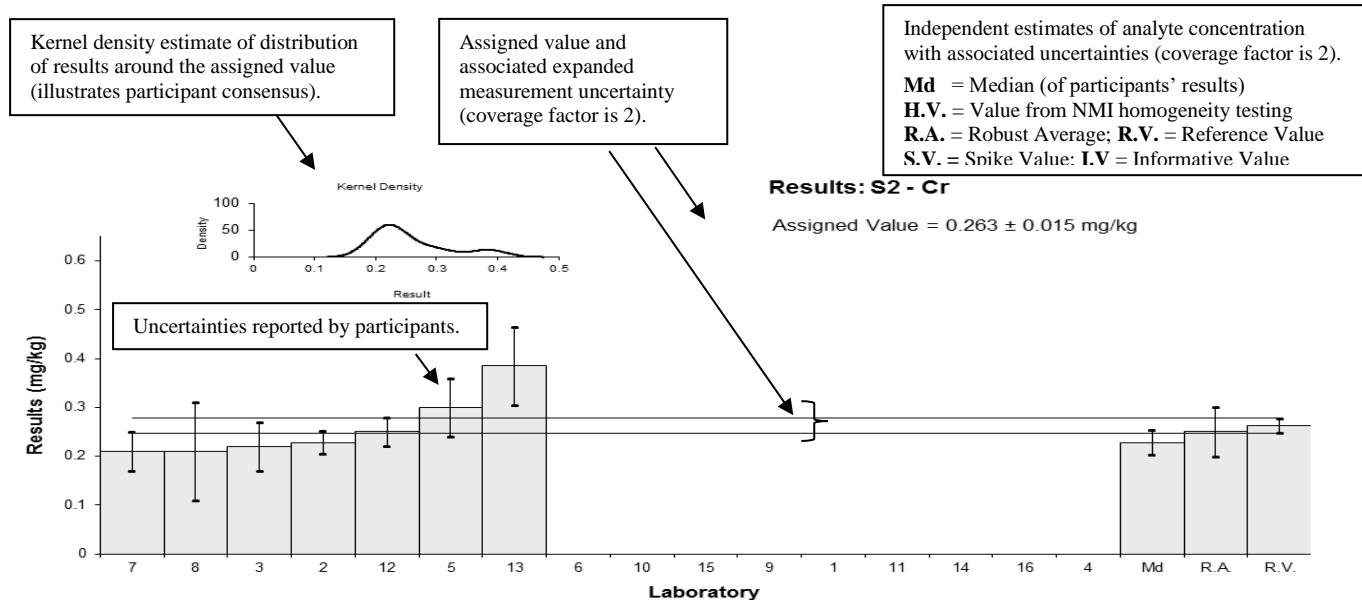


Figure 1 Guide to Presentation of Results

5.2 Assigned Value

An example of the assigned value calculation using data from the present study is given in Appendix 2. The assigned value is defined as: 'the value attributed to a particular property of a proficiency test item.'² In this study the property is the mass fraction of analyte. For As, Cd and Pb in S2, the assigned values were reference values measured using standard addition mass spectrometry. For Cr and Ni in S2, the assigned values were reference values measured using isotope dilution mass spectrometry. For all other analytes the assigned values were the robust average of participants' results; the expanded uncertainties were estimated from associated robust standard deviation.

5.3 Robust Average

The robust averages and associated expanded measurement uncertainties were calculated using the procedure described in 'Statistical methods for use in proficiency testing by inter-laboratory comparisons, ISO13528:2015(E)'.⁶

5.4 Robust Between-Laboratory Coefficient of Variation

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

5.5 Performance Coefficient of Variation (PCV)

The performance coefficient of variation (PCV) is a measure of the between laboratory variation that in the judgement of the study coordinator would be expected from participants. It is important to note that is not the coefficient of variation of participant results. The fixed value set for PCV is based on the existing regulation, the acceptance criteria indicated by the methods, the matrix, the concentration level of analyte and on experience from previous

studies. It is backed up by mathematical models such as Thompson Horwitz equation.⁷ By setting a fixed and realistic value for the PCV, the participant's performance does not depend on other participants' performance and can be compared from study to study and against achievable performance.

5.6 Target Standard Deviation

The target standard deviation (σ) is the product of the assigned value (X) and the performance coefficient of variation (PCV) as presented in Equation 1.

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

5.7 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 participants' 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 satisfactory;
- $2.0 < |z| < 3.0$ is questionable;
- $|z| \geq 3.0$ is unsatisfactory.

5.8 E_n-Score

An example of E_n-score calculation using data from the present study is given in Appendix 2. The E_n-score is complementary to the z-score in assessment of laboratory performance.

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

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

Where:

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

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

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

5.9 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025:2018⁸ must establish and demonstrate the traceability and measurement uncertainty associated with their test results. Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.¹

6 TABLES AND FIGURES

Table 8

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	Ag
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.15	0.03	-0.46	-0.34
2	0.161	0.016	0.00	0.00
3	0.16	0.01	-0.04	-0.07
4	0.16	0.04	-0.04	-0.02
5	0.17	0.03	0.37	0.28
6	< 10	NR		
7	NR	NR		
8	NT	NT		
9	0.18	0.03	0.79	0.59
10	NT	NT		
11	0.151	0.12	-0.41	-0.08
12	0.15	0.015	-0.46	-0.59
13	NT	NT		
14	0.2	NR	1.61	3.55
15	<1	NR		
16	0.15	0.14	-0.46	-0.08

Statistics

Assigned Value	0.161	0.011
Spike	0.175	0.011
Homogeneity Value	0.153	0.018
Robust Average	0.161	0.011
Median	0.160	0.011
Mean	0.163	
N	10	
Max.	0.2	
Min.	0.15	
Robust SD	0.014	
Robust CV	8.7%	

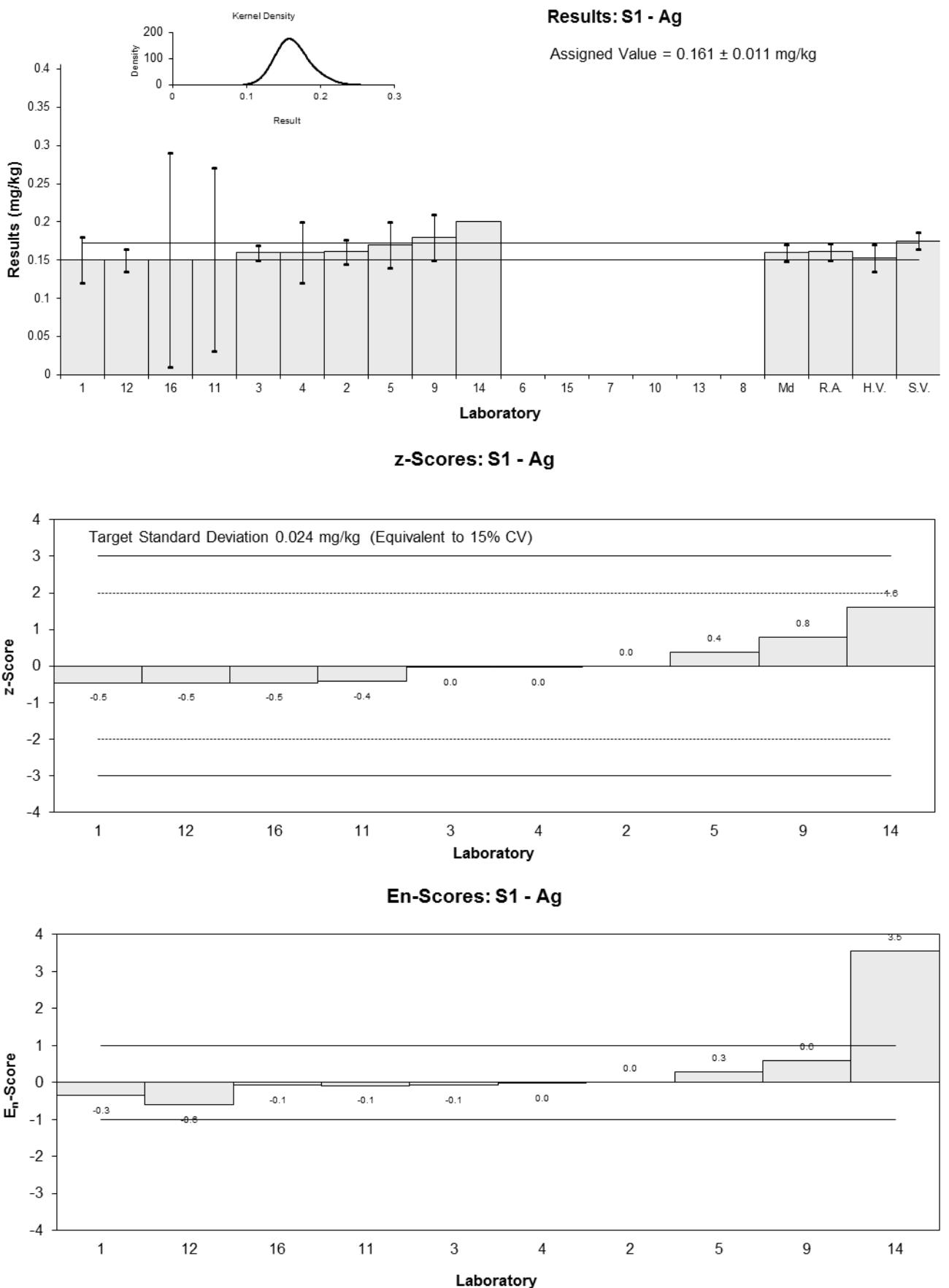


Figure 2

Table 9

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	2.2	0.44	-1.03	-0.80
2	2.51	0.251	-0.23	-0.26
3	2.6	0.04	0.00	0.00
4	3	1	1.03	0.39
5	2.14	0.43	-1.18	-0.93
6	< 5	NR		
7	2.3	0.5	-0.77	-0.54
8	NT	NT		
9	2.91	0.20	0.79	0.99
10	NT	NT		
11	2.75	2.0	0.38	0.07
12	2.5	0.25	-0.26	-0.29
13	2.458	0.49	-0.36	-0.26
14	2.88	0.699	0.72	0.38
15	2.47	0.5	-0.33	-0.23
16	3.3	1.2	1.79	0.57

Statistics

Assigned Value	2.60	0.24
Spike	2.49	0.07
Homogeneity Value	2.57	0.31
Robust Average	2.60	0.24
Median	2.51	0.22
Mean	2.62	
N	13	
Max.	3.3	
Min.	2.14	
Robust SD	0.35	
Robust CV	14%	

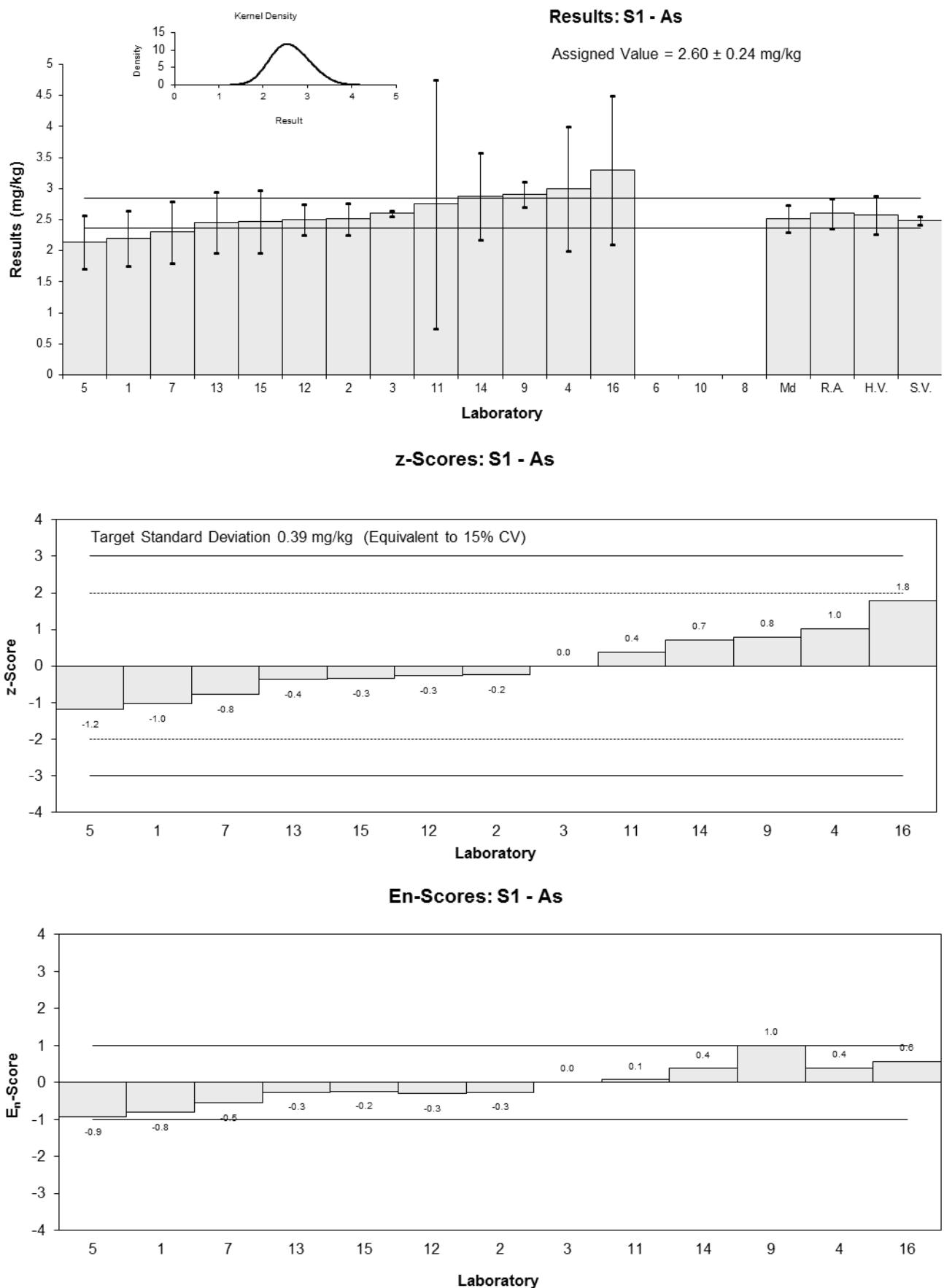


Figure 3

Table 10

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	NR	NR
3	NT	NT
4	1.9	0.7
5	2.24	0.45
6	< 50	NR
7	2.2	0.4
8	NT	NT
9	2.2	0.3
10	NT	NT
11	< 3	2.4
12	NT	NT
13	NT	NT
14	<5	NR
15	4.14	0.8
16	NT	NT

Statistics

Assigned Value	Not Set	
Spike	2.00	0.08
Homogeneity Value	1.77	0.21
Robust Average	2.32	0.55
Median	2.20	0.07
Mean	2.54	
N	5	
Max.	4.14	
Min.	1.9	
Robust SD	0.49	
Robust CV	21%	

Results: S1 - B

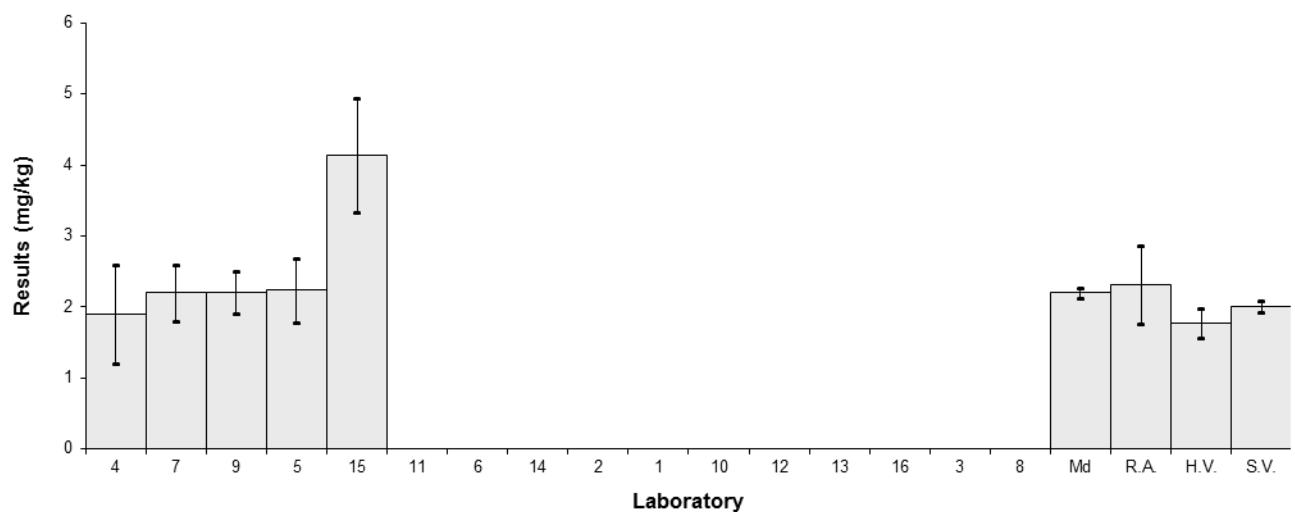


Figure 4

Table 11

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	3.4	0.68	-0.41	-0.32
2	NR	NR		
3	NT	NT		
4	3.8	0.8	0.33	0.22
5	3.42	0.68	-0.37	-0.29
6	< 10	NR		
7	3.7	0.7	0.15	0.11
8	NT	NT		
9	4.47	0.72	1.57	1.16
10	NT	NT		
11	3.67	2.6	0.09	0.02
12	3.6	0.36	-0.04	-0.05
13	3.490	0.70	-0.24	-0.18
14	3.6	NR	-0.04	-0.17
15	3.65	0.7	0.06	0.04
16	3.6	0.55	-0.04	-0.04

Statistics

Assigned Value	3.62	0.12
Spike	3.88	0.21
Homogeneity Value	3.53	0.42
Robust Average	3.62	0.12
Median	3.60	0.10
Mean	3.67	
N	11	
Max.	4.47	
Min.	3.4	
Robust SD	0.16	
Robust CV	4.4%	

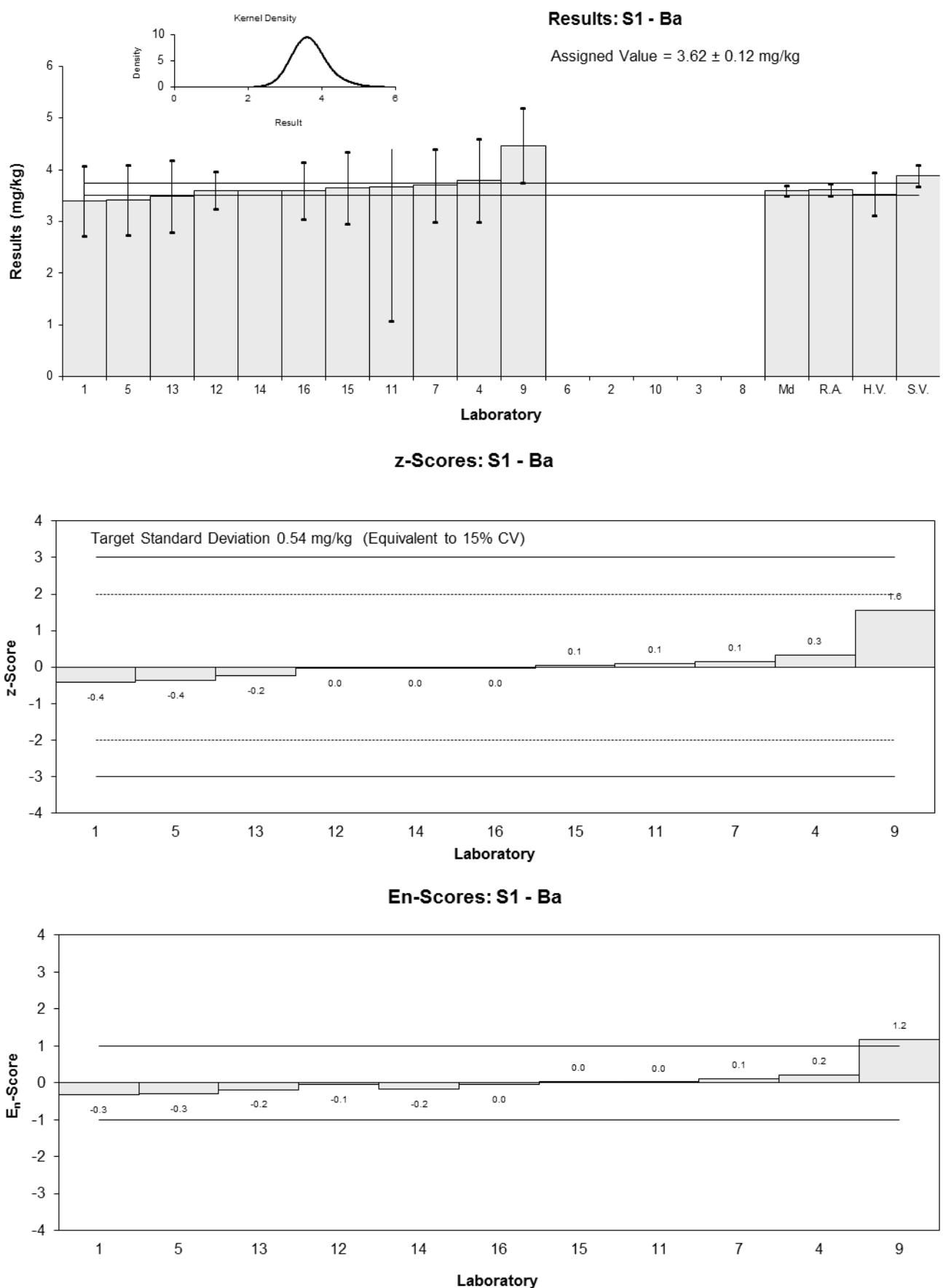


Figure 5

Table 12

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	Ca
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	2500	500	3.66	1.32
2	NR	NR		
3	NT	NT		
4	1700	200	-0.71	-0.59
5	1968	393.6	0.75	0.34
6	1880	280	0.27	0.17
7	1710	340	-0.66	-0.34
8	NT	NT		
9	171	12	-9.07	-18.27
10	NT	NT		
11	1840	190	0.05	0.05
12	1820	182	-0.05	-0.05
13	1733.14	346.63	-0.53	-0.27
14	1830	197.1	0.00	0.00
15	1780	200	-0.27	-0.23
16	69	21	-9.62	-19.05

Statistics

Assigned Value*	1830	90
Spike	Not Spiked	
Homogeneity Value	2100	250
Robust Average	1770	140
Median	1800	80
Mean	1583	
N	12	
Max.	2500	
Min.	69	
Robust SD	200	
Robust CV	11%	

*Robust Average excluding Laboratories 9 and 16.

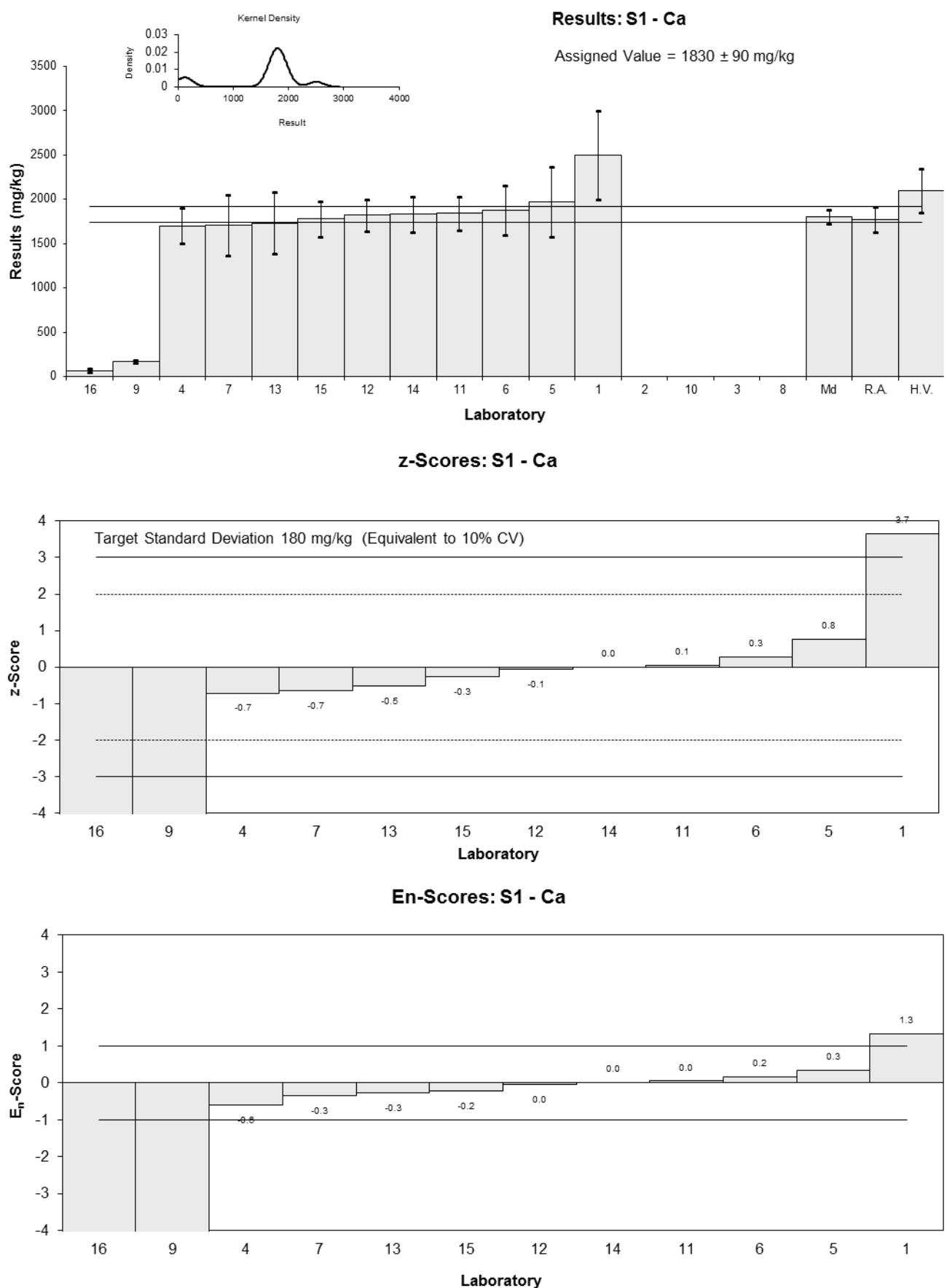


Figure 6

Table 13

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.12	0.024	-0.37	-0.28
2	0.131	0.013	0.21	0.27
3	0.12	0.01	-0.37	-0.57
4	0.13	0.04	0.16	0.07
5	0.12	0.02	-0.37	-0.33
6	< 1	NR		
7	0.12	0.02	-0.37	-0.33
8	NT	NT		
9	0.14	0.04	0.68	0.32
10	NT	NT		
11	0.144	0.11	0.89	0.15
12	0.12	0.12	-0.37	-0.06
13	0.123	0.02	-0.21	-0.19
14	0.12	0.025	-0.37	-0.27
15	0.14	0.02	0.68	0.61
16	0.13	0.19	0.16	0.02

Statistics

Assigned Value	0.127	0.007
Spike	0.138	0.004
Homogeneity Value	0.130	0.016
Robust Average	0.127	0.007
Median	0.123	0.003
Mean	0.128	
N	13	
Max.	0.144	
Min.	0.12	
Robust SD	0.0099	
Robust CV	7.8%	

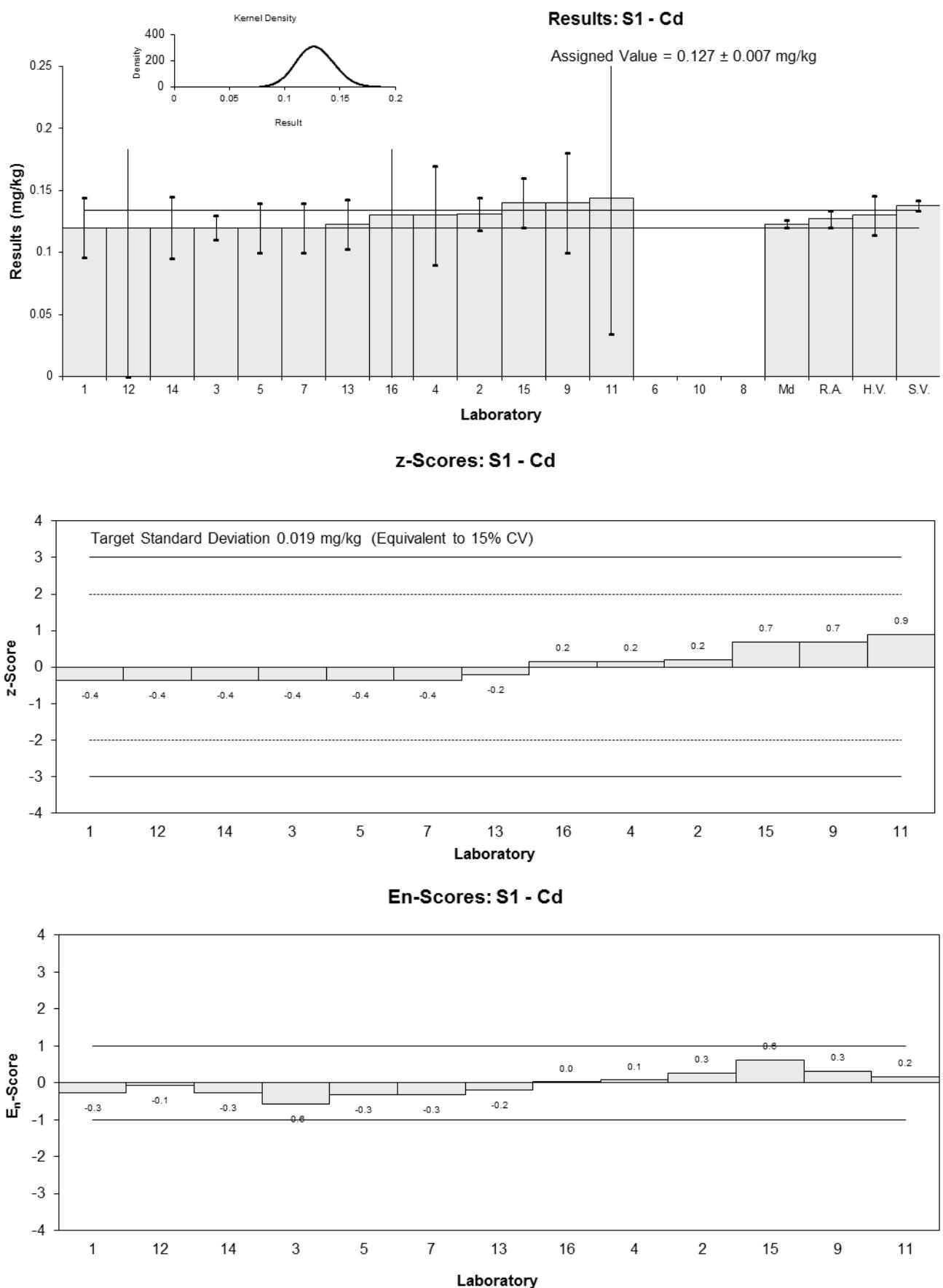


Figure 7

Table 14

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	Co
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.56	0.112	-1.07	-0.57
2	0.665	0.067	0.61	0.51
3	0.62	0.02	-0.11	-0.18
4	0.7	0.2	1.16	0.36
5	0.60	0.12	-0.43	-0.22
6	< 5	NR		
7	0.633	0.13	0.10	0.04
8	NT	NT		
9	0.79	0.11	2.60	1.42
10	NT	NT		
11	0.598	0.44	-0.46	-0.07
12	0.605	0.061	-0.35	-0.32
13	0.571	0.11	-0.89	-0.49
14	0.64	NR	0.21	0.38
15	0.63	0.1	0.05	0.03
16	0.63	0.094	0.05	0.03

Statistics

Assigned Value	0.627	0.034
Spike	Not Spiked	
Homogeneity Value	0.613	0.074
Robust Average	0.627	0.034
Median	0.630	0.027
Mean	0.634	
N	13	
Max.	0.79	
Min.	0.56	
Robust SD	0.049	
Robust CV	7.8%	

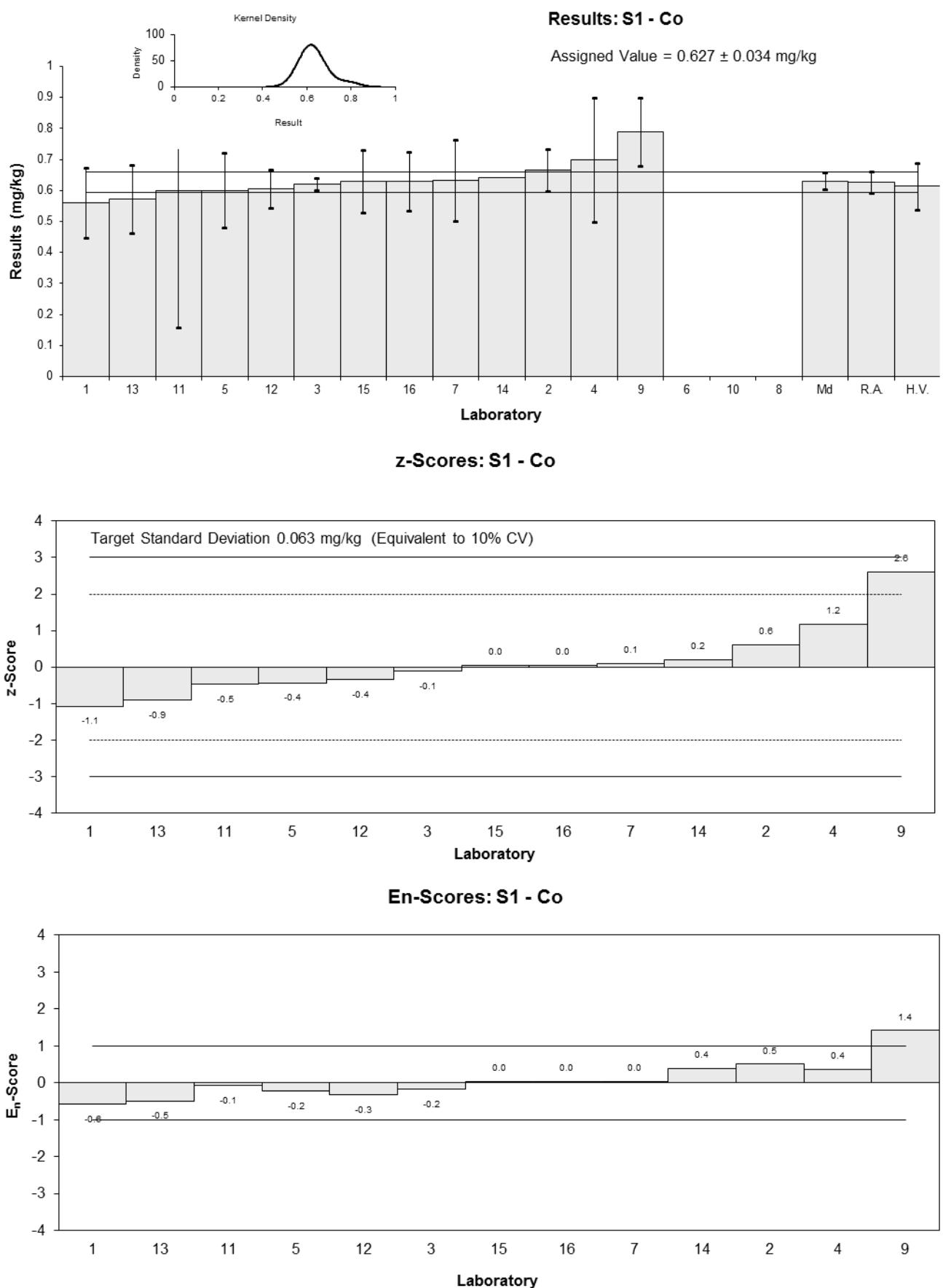


Figure 8

Table 15

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1.4	0.28	-0.24	-0.20
2	1.71	0.171	0.82	0.91
3	1.3	0.03	-0.58	-0.84
4	1.3	0.4	-0.58	-0.38
5	1.46	0.29	-0.03	-0.03
6	< 5	NR		
7	1.7	0.3	0.78	0.64
8	NT	NT		
9	NR	NR		
10	NT	NT		
11	1.27	0.95	-0.68	-0.21
12	1.7	0.2	0.78	0.81
13	1.558	0.31	0.30	0.24
14	1.23	0.51	-0.82	-0.44
15	2.20	0.5	2.48	1.36
16	1.1	0.25	-1.26	-1.16

Statistics

Assigned Value	1.47	0.20
Spike	1.50	0.10
Homogeneity Value	1.42	0.17
Robust Average	1.47	0.20
Median	1.43	0.17
Mean	1.49	
N	12	
Max.	2.2	
Min.	1.1	
Robust SD	0.27	
Robust CV	18%	

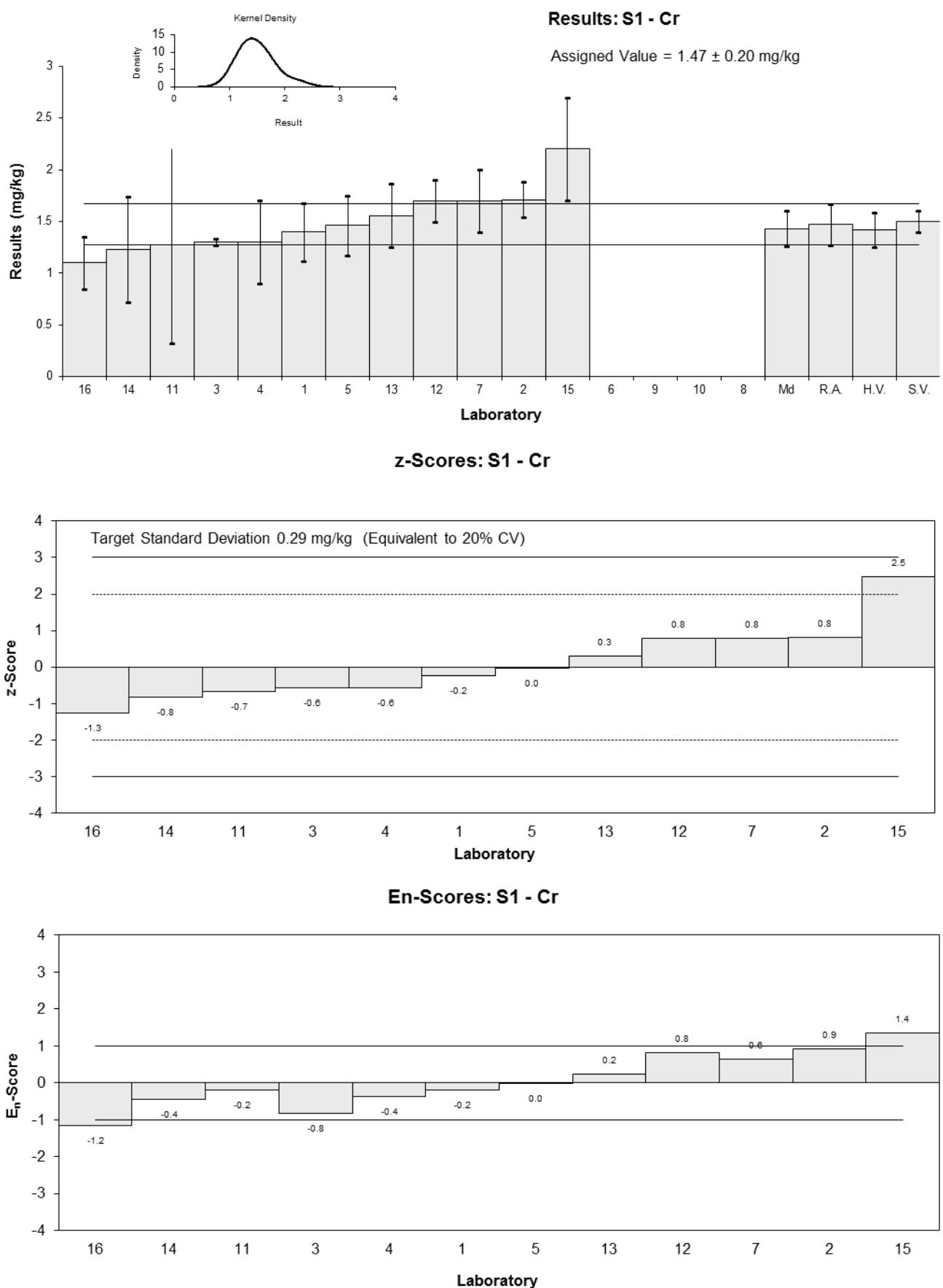


Figure 9

Table 16

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	1.39	0.139	-0.14	-0.12
3	1.4	0.06	-0.07	-0.10
4	1.4	0.3	-0.07	-0.03
5	1.34	0.27	-0.50	-0.25
6	< 20	NR		
7	1.5	0.3	0.64	0.29
8	NT	NT		
9	1.67	0.23	1.84	1.07
10	NT	NT		
11	1.27	0.95	-0.99	-0.15
12	1.3	0.13	-0.78	-0.72
13	1.292	0.26	-0.84	-0.43
14	1.4	0.52	-0.07	-0.02
15	1.53	0.3	0.85	0.39
16	1.5	0.4	0.64	0.22

Statistics

Assigned Value	1.41	0.08
Spike	Not Spiked	
Homogeneity Value	1.28	0.15
Robust Average	1.41	0.08
Median	1.40	0.09
Mean	1.42	
N	12	
Max.	1.67	
Min.	1.27	
Robust SD	0.11	
Robust CV	8.1%	

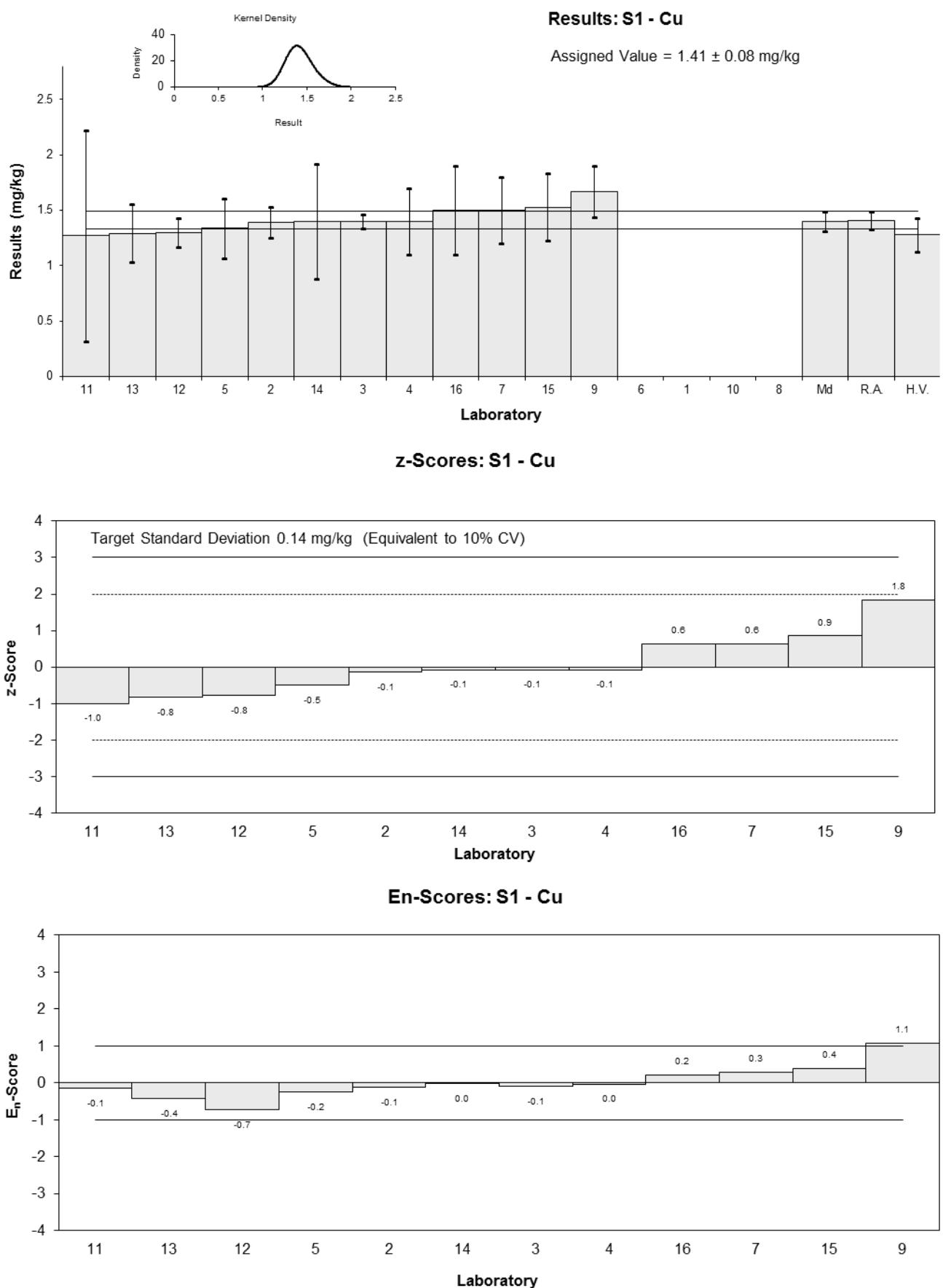


Figure 10

Table 17

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	Fe
Units	mg/kg

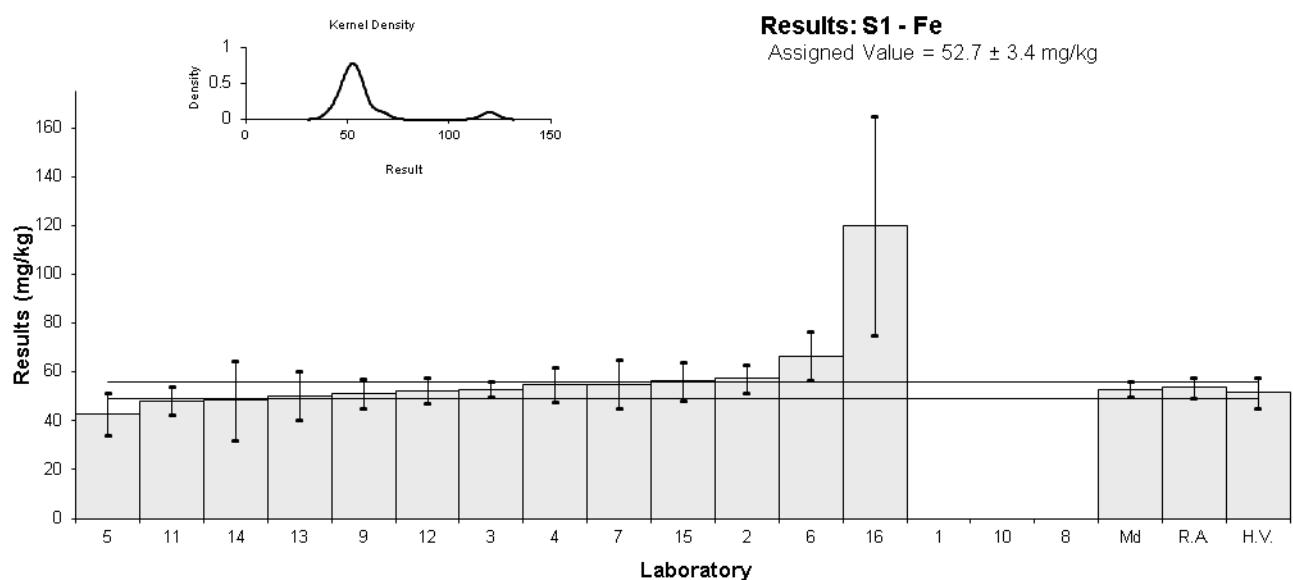
Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	57.3	5.73	0.87	0.69
3	53	3	0.06	0.07
4	55	7	0.44	0.30
5	42.74	8.55	-1.89	-1.08
6	66.6	10	2.64	1.32
7	55	10	0.44	0.22
8	NT	NT		
9	51.2	6.0	-0.28	-0.22
10	NT	NT		
11	48.3	5.9	-0.83	-0.65
12	52.5	5.3	-0.04	-0.03
13	50.293	10.06	-0.46	-0.23
14	48.4	16.2	-0.82	-0.26
15	56.3	8.0	0.68	0.41
16	120	45	12.77	1.49

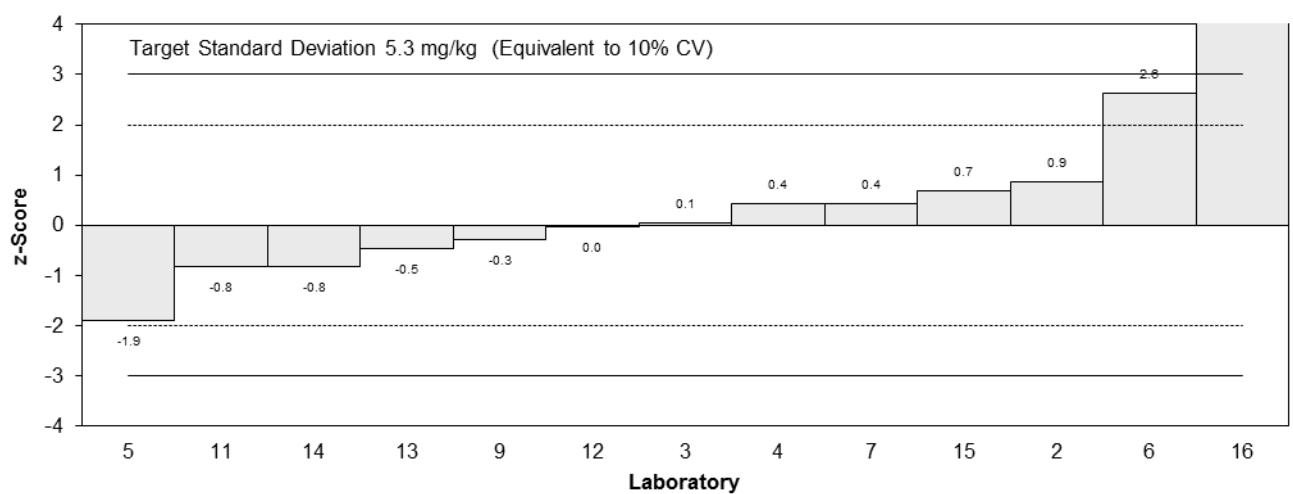
Statistics

Assigned Value*	52.7	3.4
Spike	Not Spiked	
Homogeneity Value	51.5	6.2
Robust Average	53.7	4.3
Median	53.0	3.0
Mean	58.2	
N	13	
Max.	120	
Min.	42.74	
Robust SD	6.2	
Robust CV	12%	

*Robust Average excluding Laboratory 16



z-Scores: S1 - Fe



En-Scores: S1 - Fe

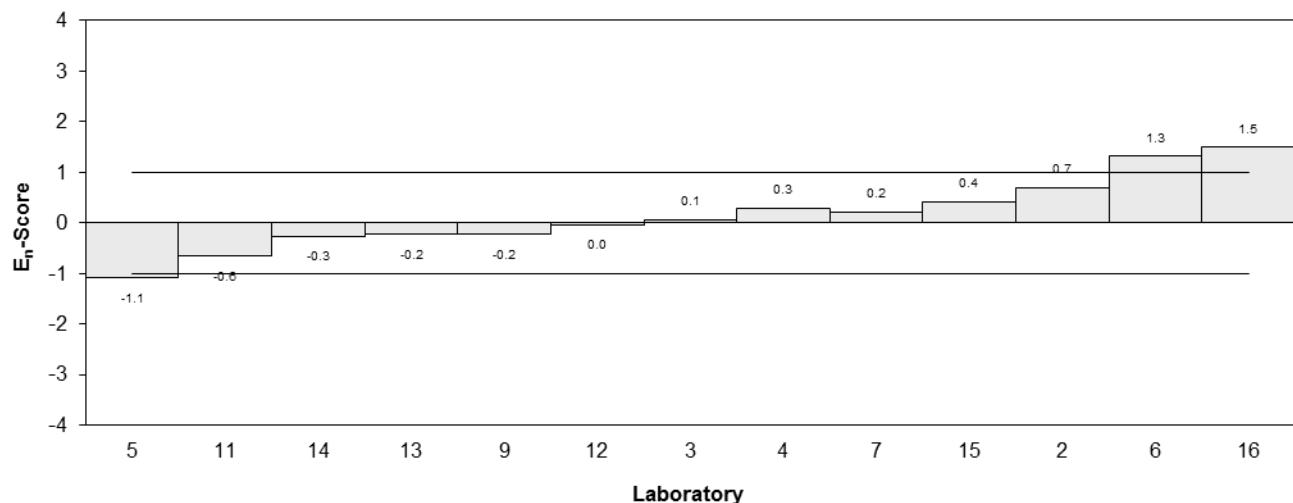


Figure 11

Table 18

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	0.437	0.044	0.53	0.44
3	0.462	0.04	1.13	1.01
4	0.40	0.06	-0.36	-0.23
5	0.40	0.08	-0.36	-0.18
6	0.436	0.065	0.51	0.30
7	0.4	0.08	-0.36	-0.18
8	NT	NT		
9	0.45	0.07	0.84	0.47
10	NT	NT		
11	0.386	0.28	-0.70	-0.10
12	0.4	0.04	-0.36	-0.32
13	0.278	0.06	-3.30	-2.12
14	0.2	0.057	-5.18	-3.48
15	0.44	0.07	0.60	0.34
16	0.4	0.12	-0.36	-0.12

Statistics

Assigned Value*	0.415	0.024
Spike	Not Spiked	
Homogeneity Value	0.428	0.051
Robust Average	0.408	0.028
Median	0.400	0.032
Mean	0.392	
N	13	
Max.	0.462	
Min.	0.2	
Robust SD	0.041	
Robust CV	10%	

*Robust Average excluding Laboratory 14

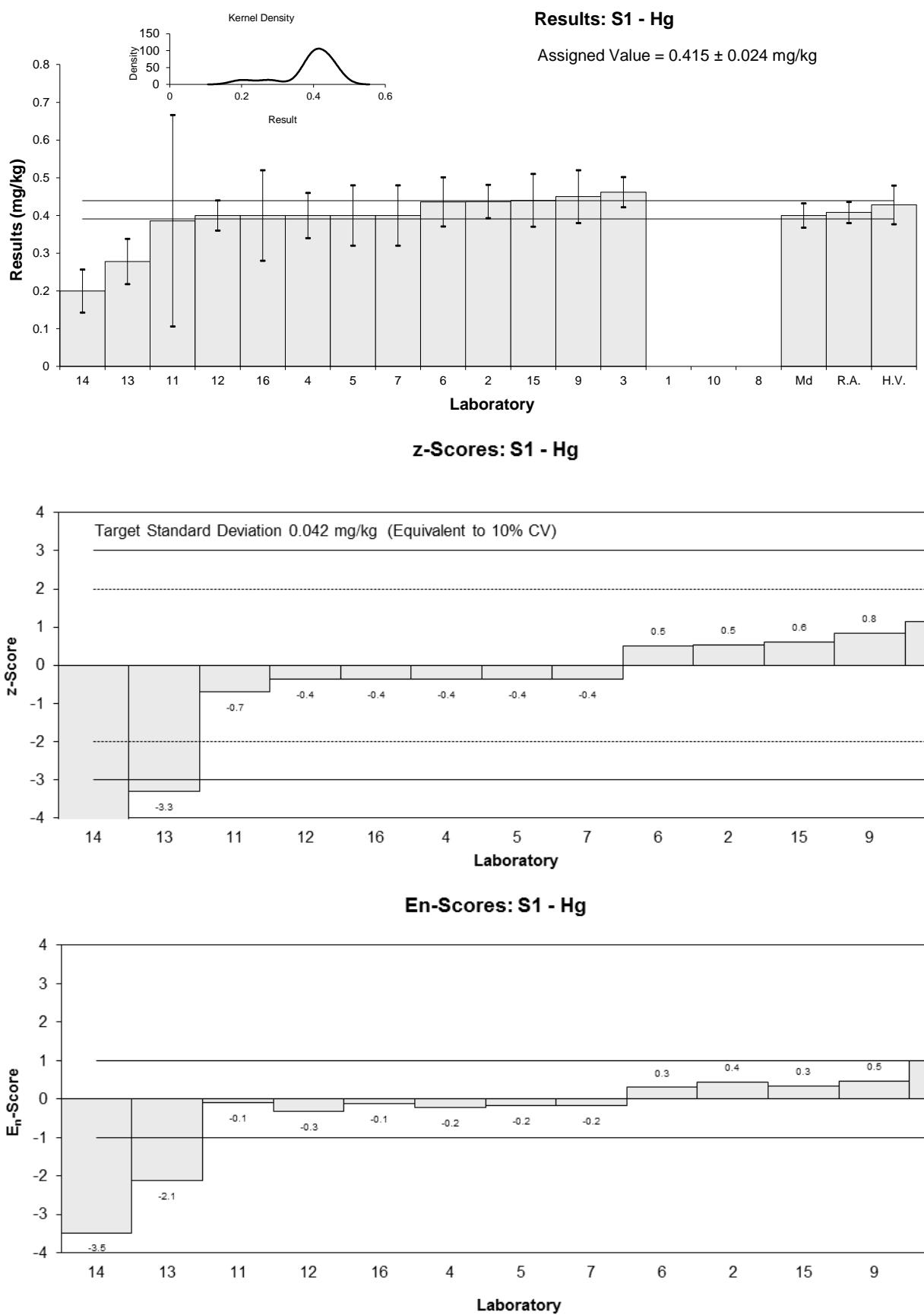


Figure 12

Table 19

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	K
Units	mg/kg

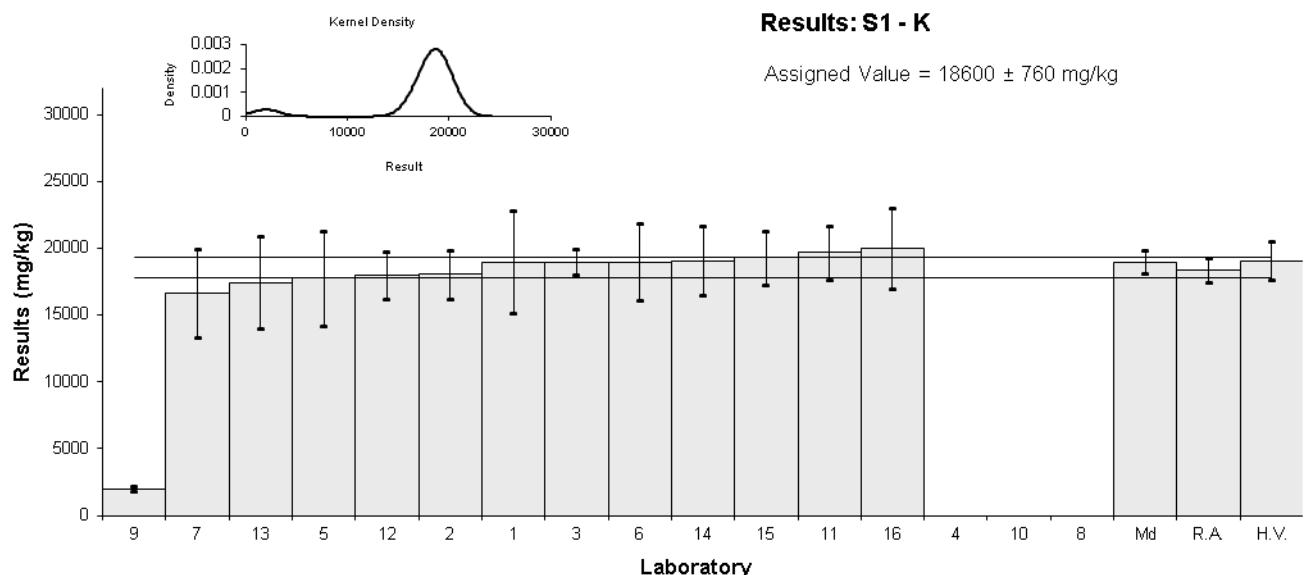
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	19000	3800	0.22	0.10
2	18050	1805	-0.30	-0.28
3	19000	1000	0.22	0.32
4	NR	NR		
5	17792	3559	-0.43	-0.22
6	19000	2900	0.22	0.13
7	16670	3330	-1.04	-0.57
8	NT	NT		
9	2018	200	-8.92	-21.10
10	NT	NT		
11	19700	2000	0.59	0.51
12	18014	1801	-0.32	-0.30
13	17462.70	3492.54	-0.61	-0.32
14	19100	2628.3	0.27	0.18
15	19300	2000	0.38	0.33
16	20000	3000	0.75	0.45

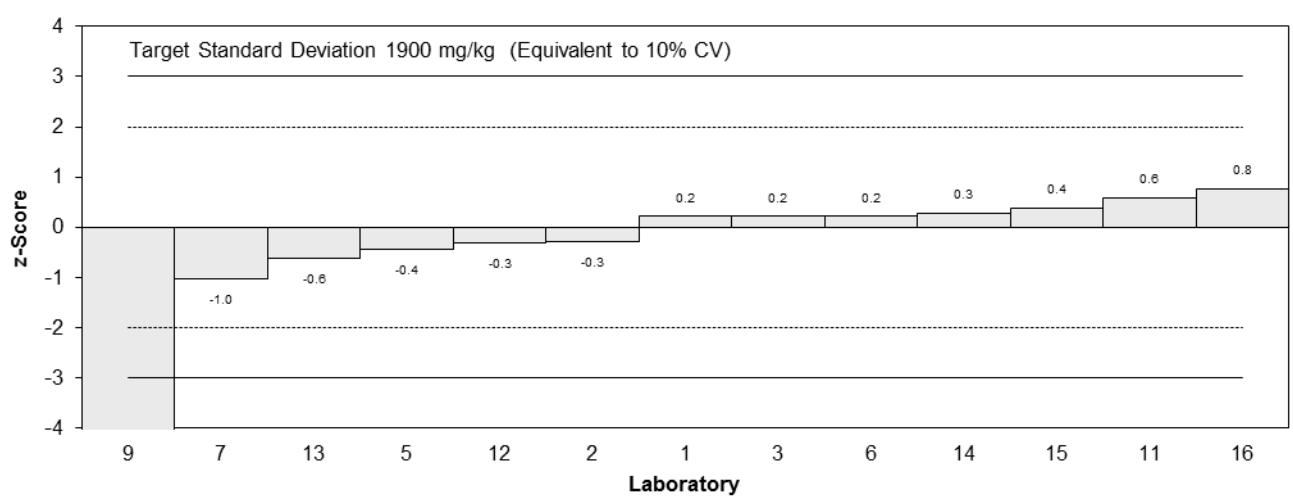
Statistics

Assigned Value*	18600	760
Spike	Not Spiked	
Homogeneity Value	19100	1400
Robust Average	18400	900
Median	19000	850
Mean	17316	
N	13	
Max.	20000	
Min.	2018	
Robust SD	1200	
Robust CV	6.5%	

*Robust Average excluding Laboratory 9



z-Scores: S1 - K



En-Scores: S1 - K

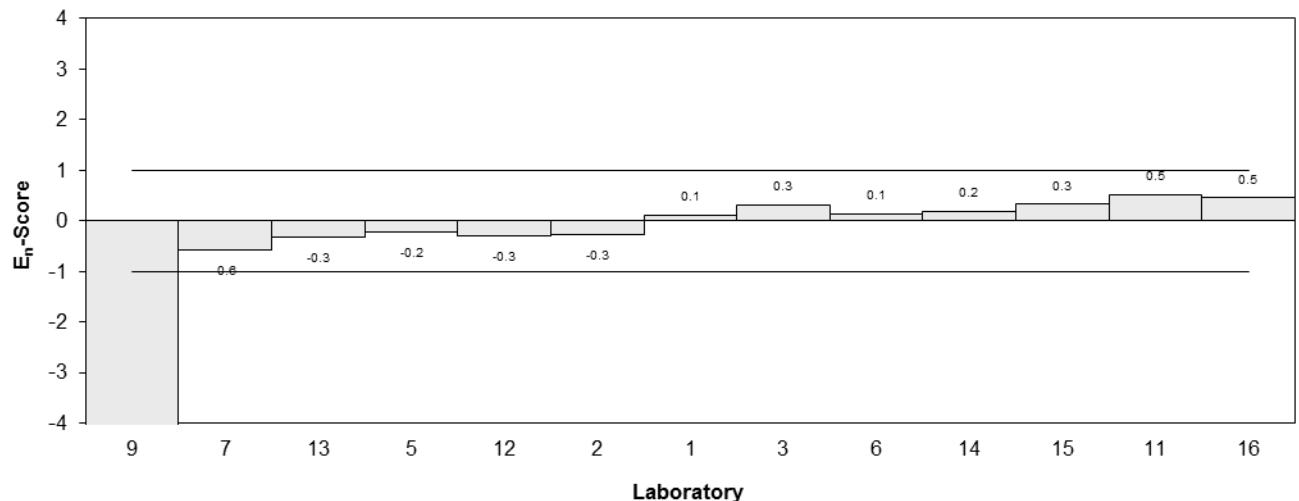


Figure 13

Table 20

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	La
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	NR	NR		
3	NT	NT		
4	NR	NR		
5	0.13	0.03	-0.08	-0.05
6	NT	NT		
7	0.13	0.03	-0.08	-0.05
8	NT	NT		
9	0.17	0.03	1.44	0.97
10	NT	NT		
11	0.132	0.10	0.00	0.00
12	NT	NT		
13	NT	NT		
14	0.1	NR	-1.21	-1.28
15	NT	NT		
16	0.13	0.068	-0.08	-0.03

Statistics

Assigned Value	0.132	0.025
Spike	0.148	0.004
Homogeneity Value	0.138	0.017
Robust Average	0.132	0.025
Median	0.130	0.002
Mean	0.132	
N	6	
Max.	0.17	
Min.	0.1	
Robust SD	0.025	
Robust CV	19%	

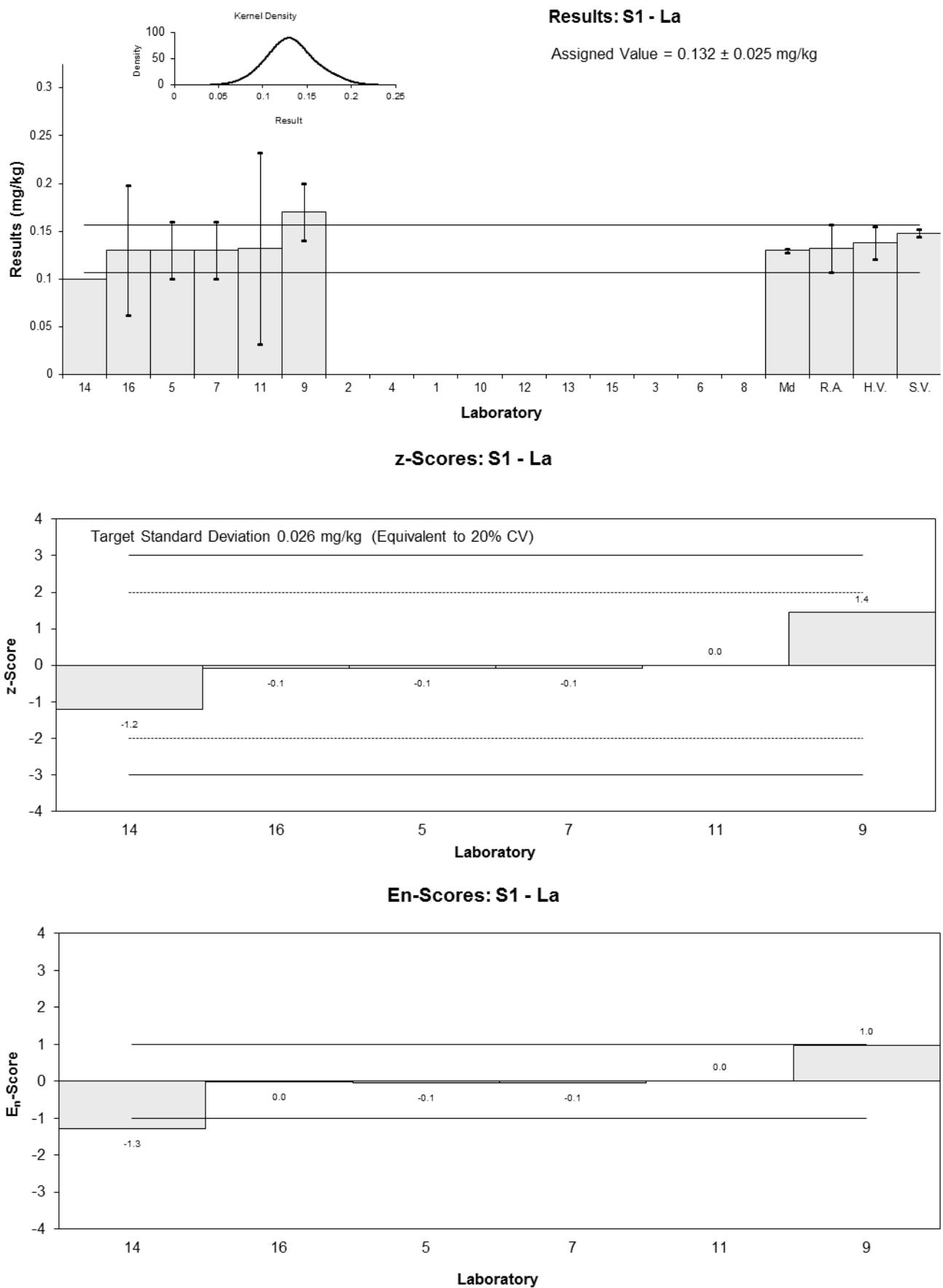


Figure 14

Table 21

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.24	0.048	0.22	0.18
2	NR	NR		
3	NT	NT		
4	<2	NR		
5	0.24	0.05	0.22	0.17
6	< 10	NR		
7	0.21	0.04	-0.43	-0.40
8	NT	NT		
9	0.24	0.04	0.22	0.20
10	NT	NT		
11	< 0.5	0.47		
12	NT	NT		
13	NT	NT		
14	0.2	NR	-0.65	-1.00
15	0.33	0.07	2.17	1.31
16	0.2	0.031	-0.65	-0.70

Statistics

Assigned Value	0.230	0.030
Spike	0.230	0.008
Homogeneity Value	0.207	0.025
Robust Average	0.230	0.030
Median	0.240	0.041
Mean	0.237	
N	7	
Max.	0.33	
Min.	0.2	
Robust SD	0.032	
Robust CV	14%	

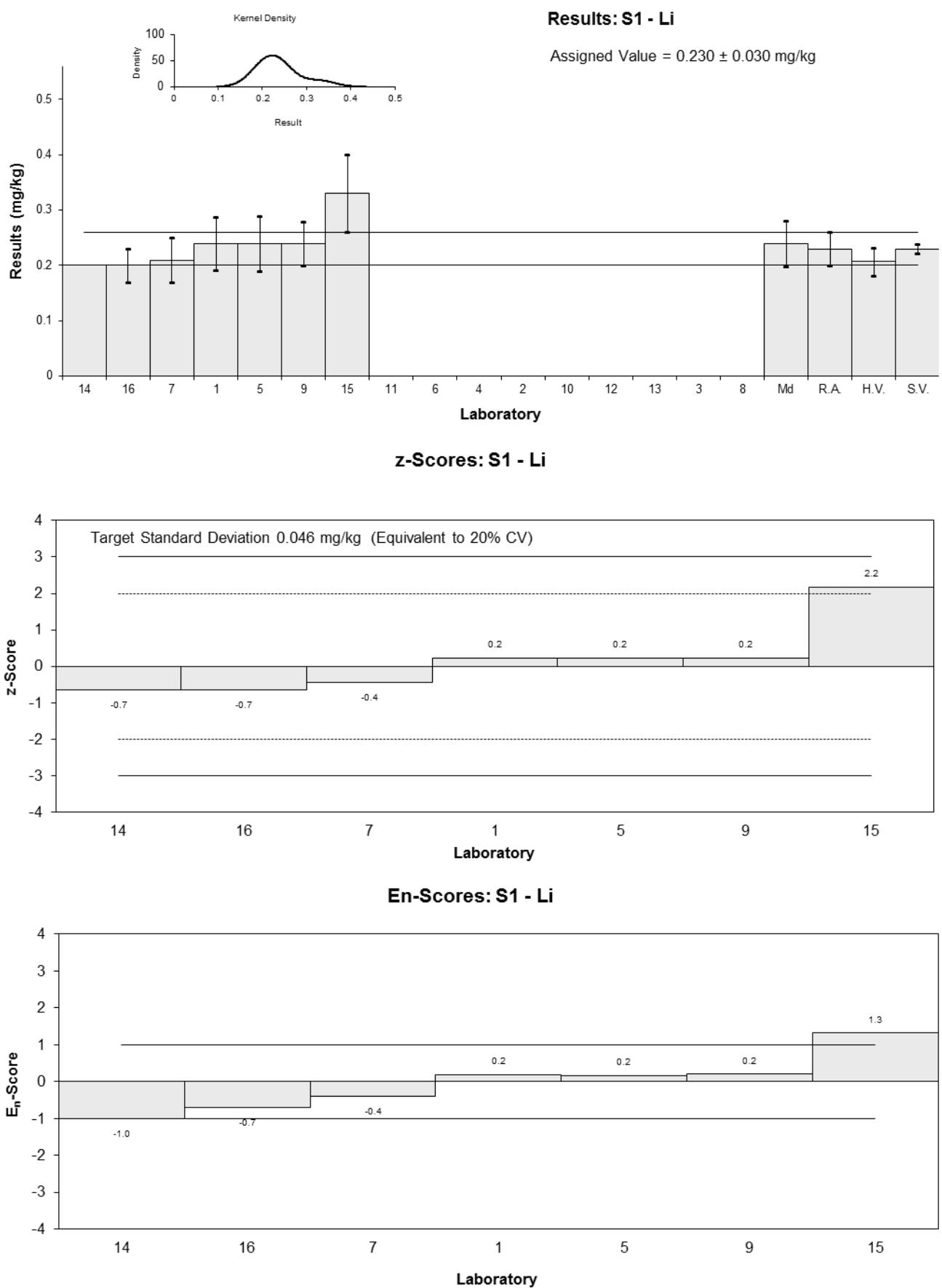


Figure 15

Table 22

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	Mg
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1800	360	2.95	1.13
2	1350	135	-0.29	-0.27
3	1430	100	0.29	0.35
4	1350	200	-0.29	-0.19
5	1446	289.3	0.40	0.19
6	1350	200	-0.29	-0.19
7	1250	250	-1.01	-0.55
8	NT	NT		
9	145	15	-8.96	-21.48
10	NT	NT		
11	1420	150	0.22	0.19
12	1365	137	-0.18	-0.17
13	1386.12	277.22	-0.03	-0.01
14	1270	135.5	-0.86	-0.82
15	1470	180	0.58	0.42
16	1400	210	0.07	0.05

Statistics

Assigned Value*	1390	60
Spike	Not Spiked	
Homogeneity Value	1400	170
Robust Average	1370	60
Median	1380	40
Mean	1317	
N	14	
Max.	1800	
Min.	145	
Robust SD	93	
Robust CV	6.8%	

*Robust Average excluding Laboratory 9

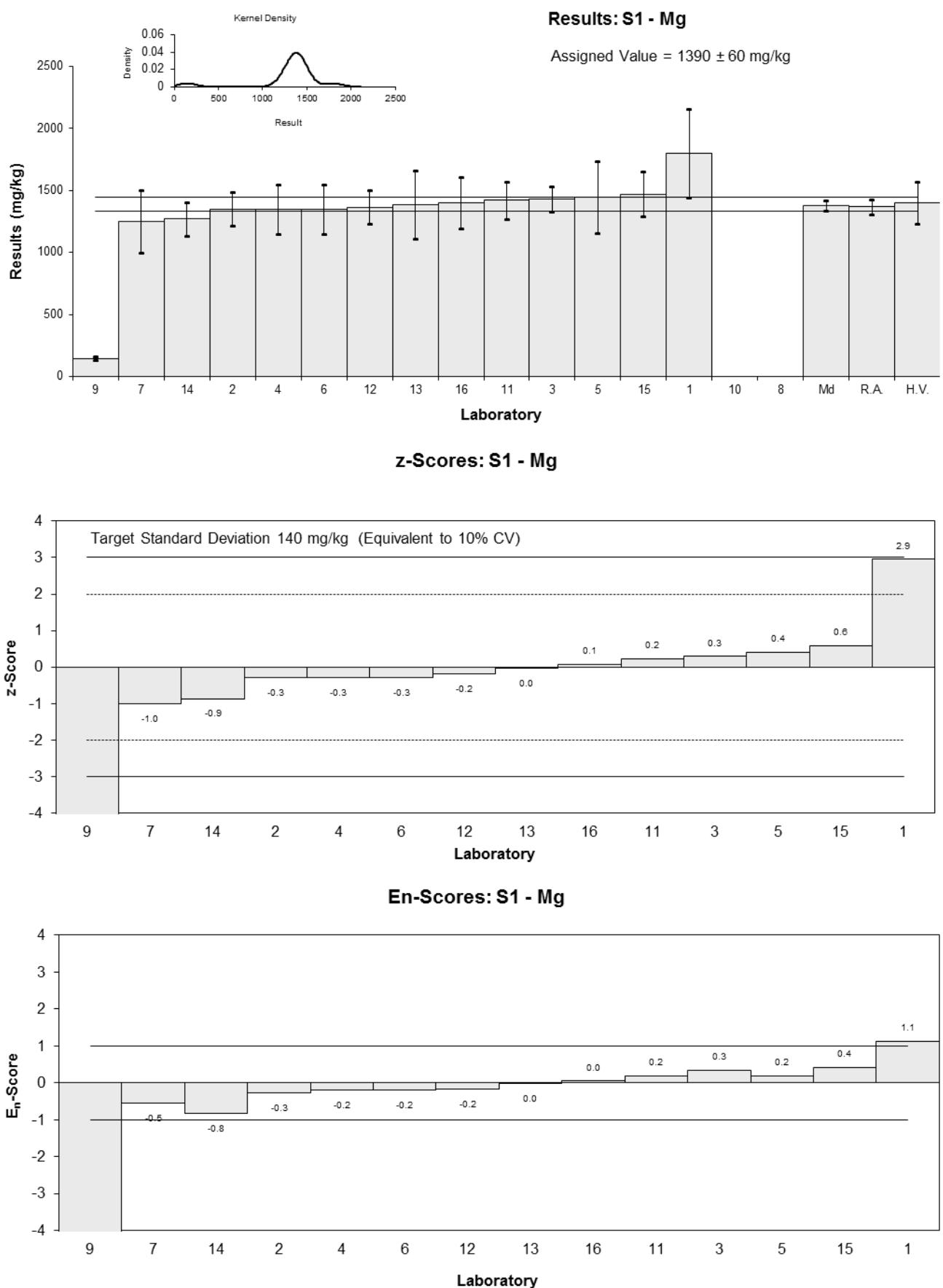


Figure 16

Table 23

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	0.913	0.091	-0.37	-0.33
3	0.95	0.05	0.02	0.03
4	0.91	0.14	-0.40	-0.25
5	1.02	0.20	0.76	0.35
6	< 10	NR		
7	0.96	0.19	0.13	0.06
8	NT	NT		
9	0.93	0.10	-0.19	-0.16
10	NT	NT		
11	0.86	0.65	-0.93	-0.13
12	0.9	0.09	-0.51	-0.46
13	0.910	0.18	-0.40	-0.20
14	0.91	0.17	-0.40	-0.21
15	1.28	0.3	3.50	1.09
16	1.1	0.16	1.60	0.90

Statistics

Assigned Value	0.948	0.052
Spike	Not Spiked	
Homogeneity Value	1.00	0.12
Robust Average	0.948	0.052
Median	0.922	0.024
Mean	0.970	
N	12	
Max.	1.28	
Min.	0.86	
Robust SD	0.072	
Robust CV	7.6%	

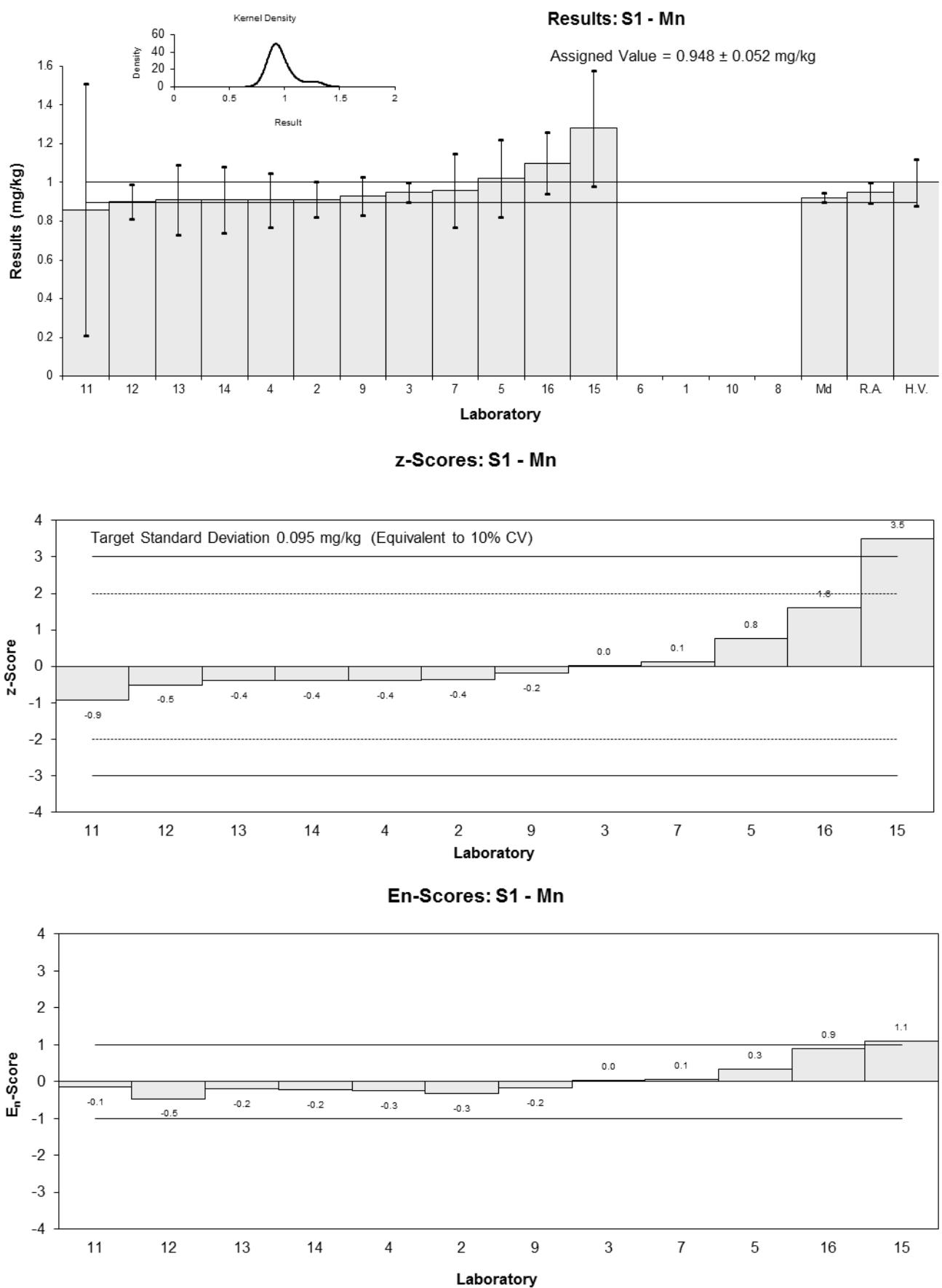


Figure 17

Table 24

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	Mo
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	NR	NR		
3	0.054	0.003	0.40	0.32
4	NR	NR		
5	0.06	0.01	1.00	0.64
6	< 10	NR		
7	0.06	0.01	1.00	0.64
8	NT	NT		
9	NR	NR		
10	NT	NT		
11	< 0.10	0.13		
12	0.044	0.01	-0.60	-0.38
13	0.050	0.01	0.00	0.00
14	<0.05	NR		
15	0.12	0.04	7.00	1.68
16	0.034	0.0075	-1.60	-1.13

Statistics

Assigned Value*	0.050	0.012
Spike	Not Spiked	
Homogeneity Value	0.042	0.005
Robust Average	0.054	0.015
Median	0.0540	0.0082
Mean	0.060	
N	7	
Max.	0.12	
Min.	0.034	
Robust SD	0.016	
Robust CV	30%	

*Robust Average excluding Laboratory 15

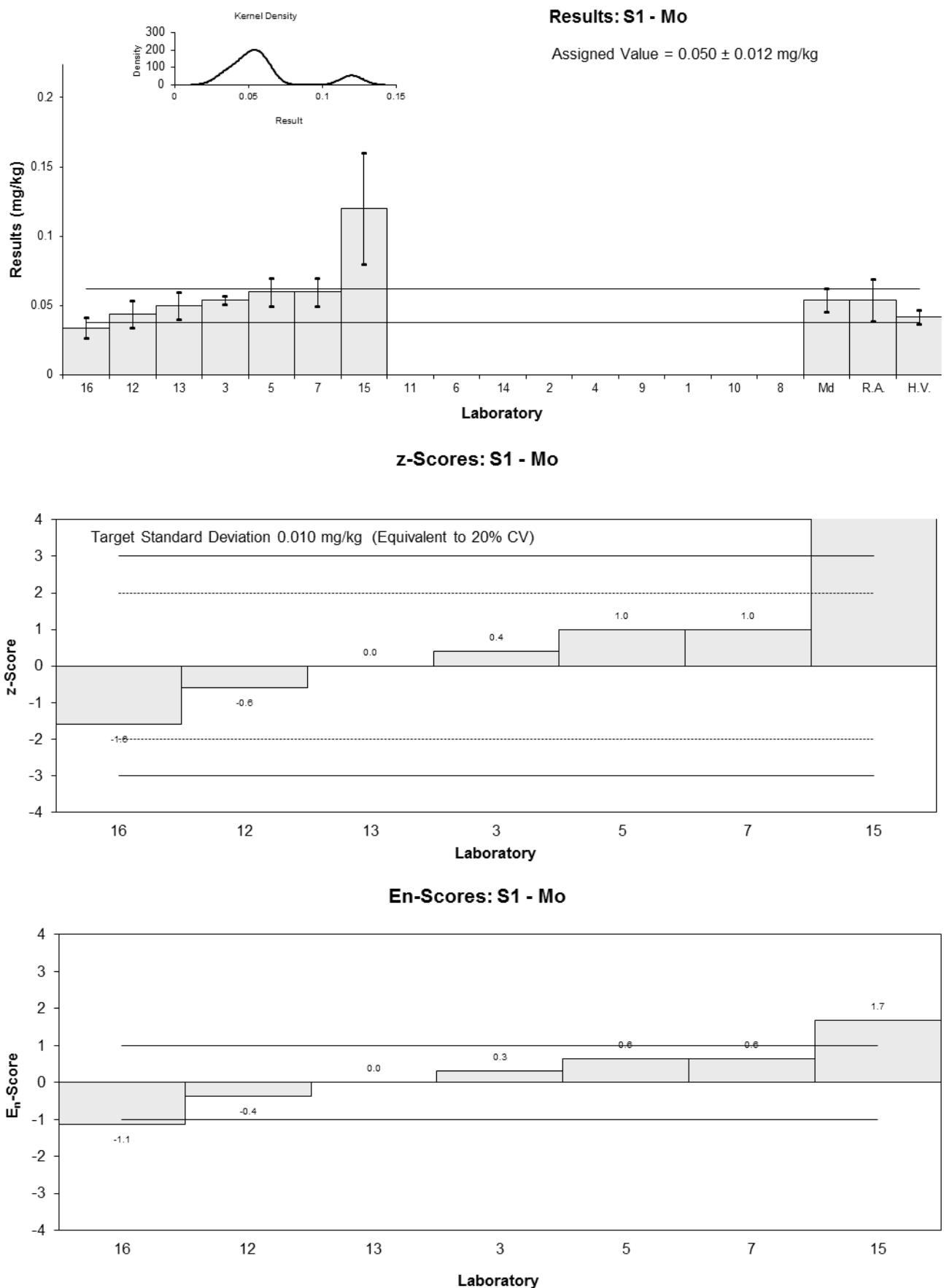


Figure 18

Table 25

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	Na
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	5500	1100	7.30	2.10
2	3360	336	0.57	0.51
3	3360	200	0.57	0.79
4	3150	500	-0.09	-0.06
5	3115	623.1	-0.20	-0.10
6	3190	480	0.03	0.02
7	2930	590	-0.79	-0.42
8	NT	NT		
9	NR	NR		
10	NT	NT		
11	3210	330	0.09	0.09
12	3150	315	-0.09	-0.09
13	3180.64	636.13	0.00	0.00
14	2890	429.1	-0.91	-0.65
15	3280	350	0.31	0.27
16	3300	490	0.38	0.24

Statistics

Assigned Value*	3180	110
Spike	Not Spiked	
Homogeneity Value	3180	380
Robust Average	3200	120
Median	3190	80
Mean	3355	
N	13	
Max.	5500	
Min.	2890	
Robust SD	170	
Robust CV	5.3%	

*Robust Average excluding Laboratory 1

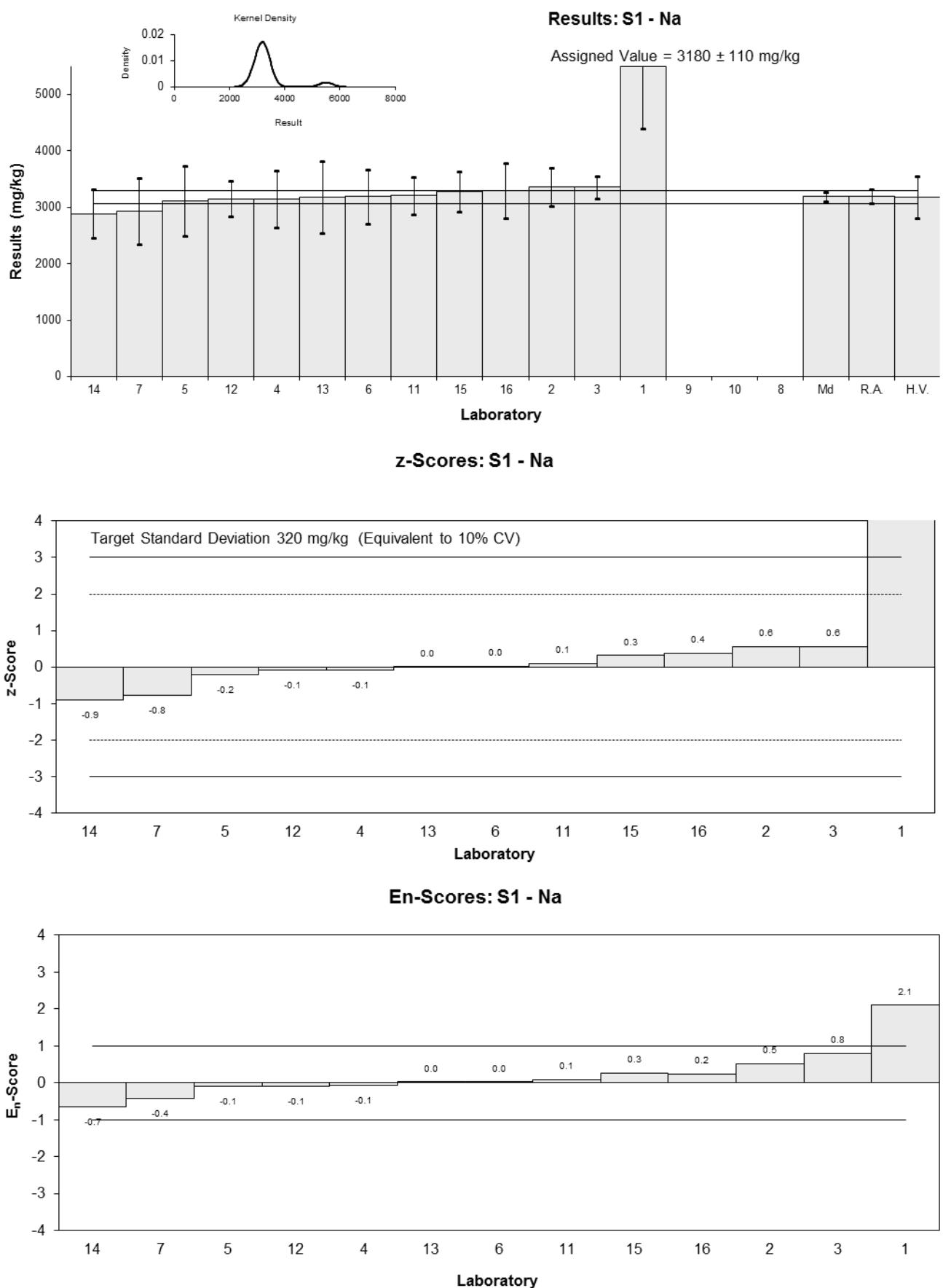


Figure 19

Table 26

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.57	0.114	-0.40	-0.32
2	0.701	0.070	0.65	0.62
3	NT	NT		
4	<0.7	NR		
5	0.87	0.17	2.02	1.23
6	< 5	NR		
7	0.74	0.15	0.97	0.65
8	NT	NT		
9	NR	NR		
10	NT	NT		
11	0.68	0.60	0.48	0.10
12	0.455	0.05	-1.33	-1.37
13	0.575	0.12	-0.36	-0.28
14	0.56	0.19	-0.48	-0.27
15	1.07	0.2	3.63	1.97
16	0.51	0.076	-0.89	-0.82

Statistics

Assigned Value*	0.62	0.11
Spike	Not Spiked	
Homogeneity Value	0.61	0.07
Robust Average	0.66	0.14
Median	0.628	0.099
Mean	0.67	
N	10	
Max.	1.07	
Min.	0.455	
Robust SD	0.17	
Robust CV	26%	

*Robust Average excluding Laboratory 15

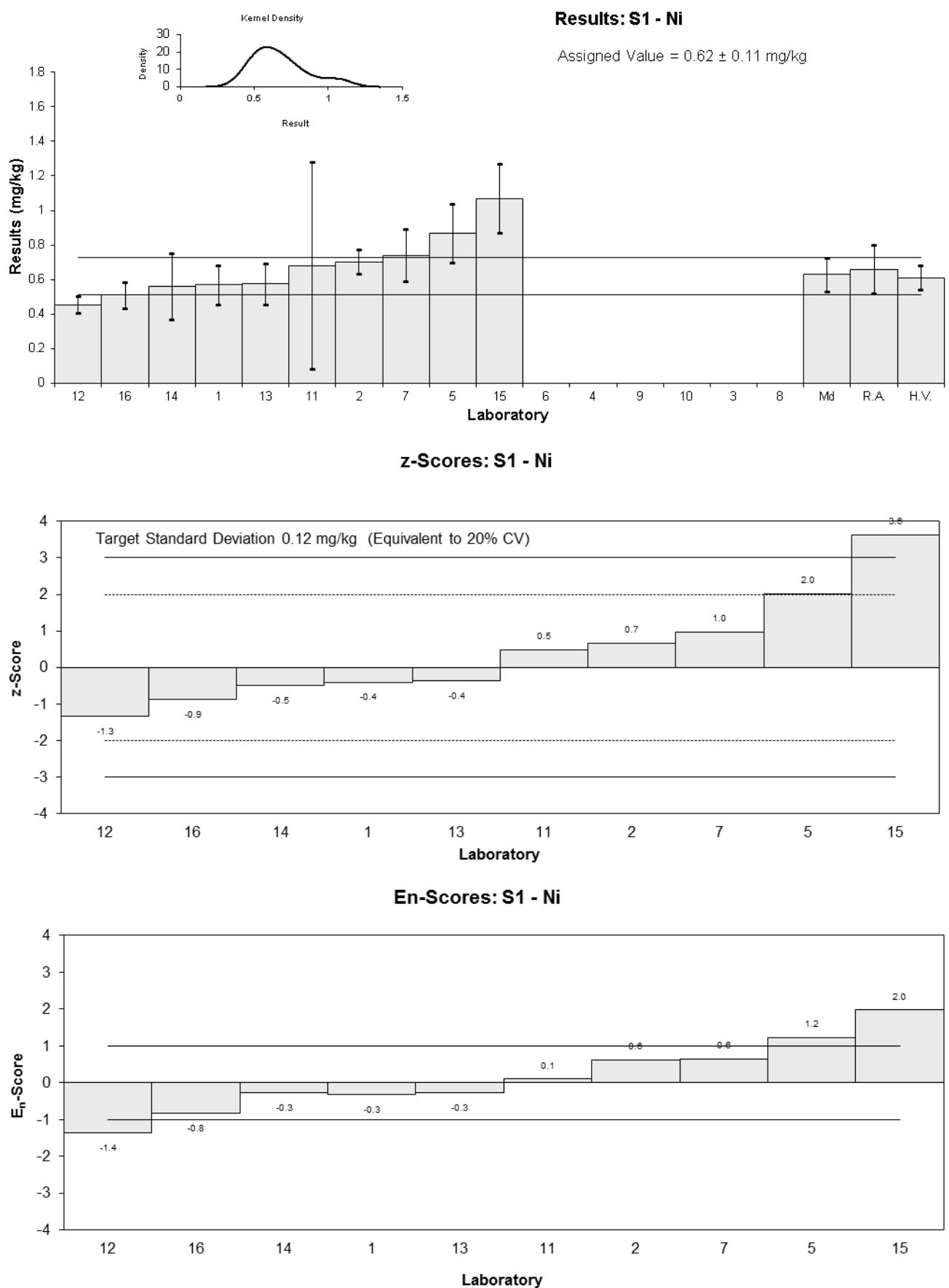


Figure 20

Table 27

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	P
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	NR	NR		
3	10900	870	1.51	1.37
4	10000	1200	0.56	0.40
5	8987	1809	-0.51	-0.25
6	8770	1320	-0.74	-0.49
7	8470	1690	-1.06	-0.56
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	9900	1000	0.45	0.37
12	9894	989	0.45	0.37
13	8715.005	3492.54	-0.80	-0.21
14	9390	NR	-0.08	-0.14
15	9390	1000	-0.08	-0.07
16	10000	1700	0.56	0.30

Statistics

Assigned Value	9470	580
Spike	Not Spiked	
Homogeneity Value	10000	1700
Robust Average	9470	580
Median	9390	610
Mean	9492	
N	11	
Max.	10900	
Min.	8470	
Robust SD	760	
Robust CV	8%	

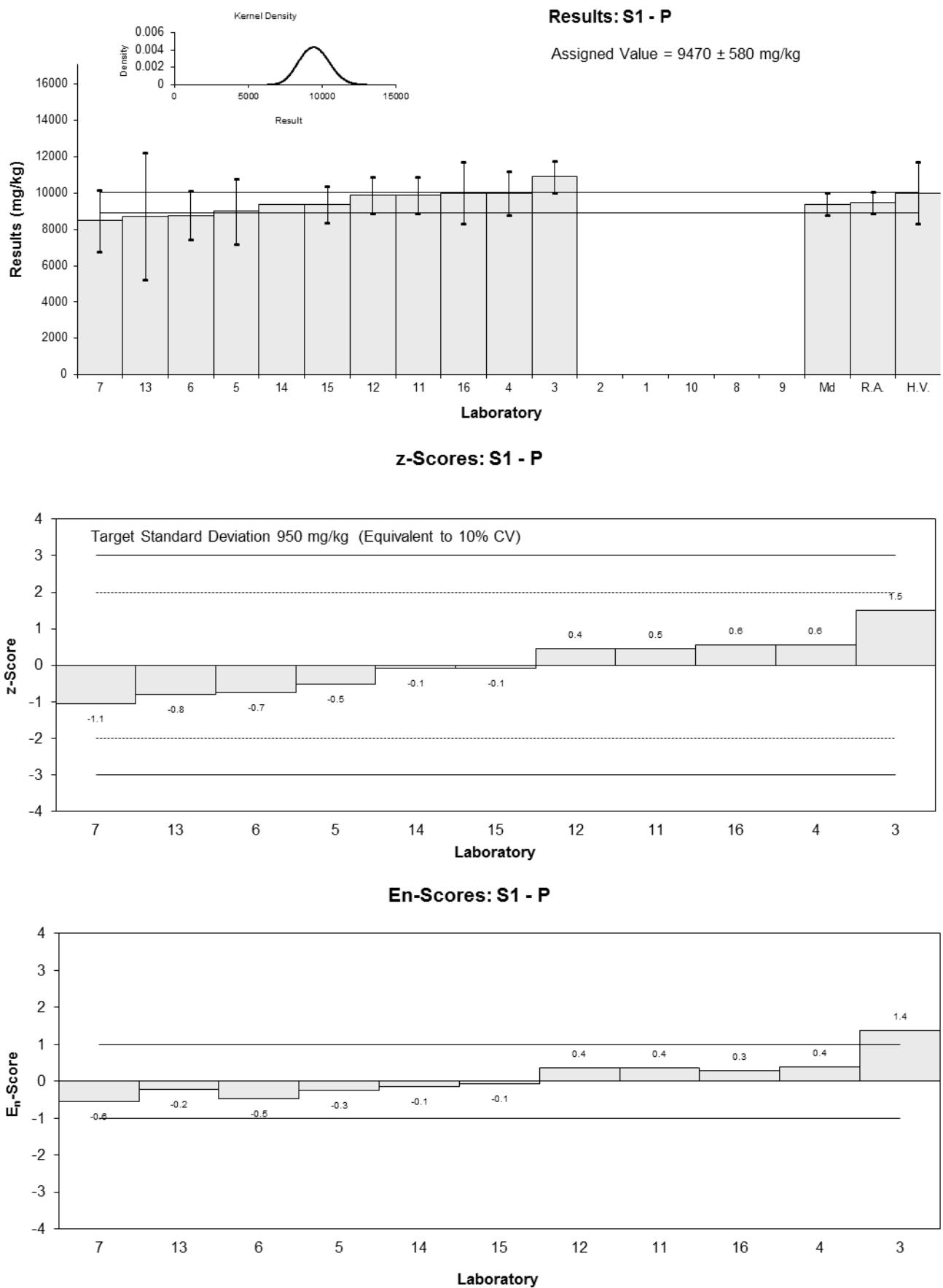


Figure 21

Table 28

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.39	0.078	7.31	2.56
2	0.187	0.019	0.04	0.04
3	0.19	0.02	0.14	0.15
4	0.21	0.05	0.86	0.45
5	0.18	0.04	-0.22	-0.14
6	< 5	NR		
7	0.17	0.03	-0.57	-0.46
8	NT	NT		
9	0.24	0.03	1.94	1.57
10	NT	NT		
11	0.166	0.13	-0.72	-0.15
12	0.17	0.02	-0.57	-0.61
13	0.189	0.04	0.11	0.07
14	0.16	0.012	-0.93	-1.25
15	0.22	0.05	1.22	0.64
16	0.17	0.078	-0.57	-0.20

Statistics

Assigned Value*	0.186	0.017
Spike	0.201	0.009
Homogeneity Value	0.185	0.022
Robust Average	0.191	0.021
Median	0.187	0.015
Mean	0.203	
N	13	
Max.	0.39	
Min.	0.16	
Robust SD	0.03	
Robust CV	16%	

*Robust Average excluding Laboratory 1

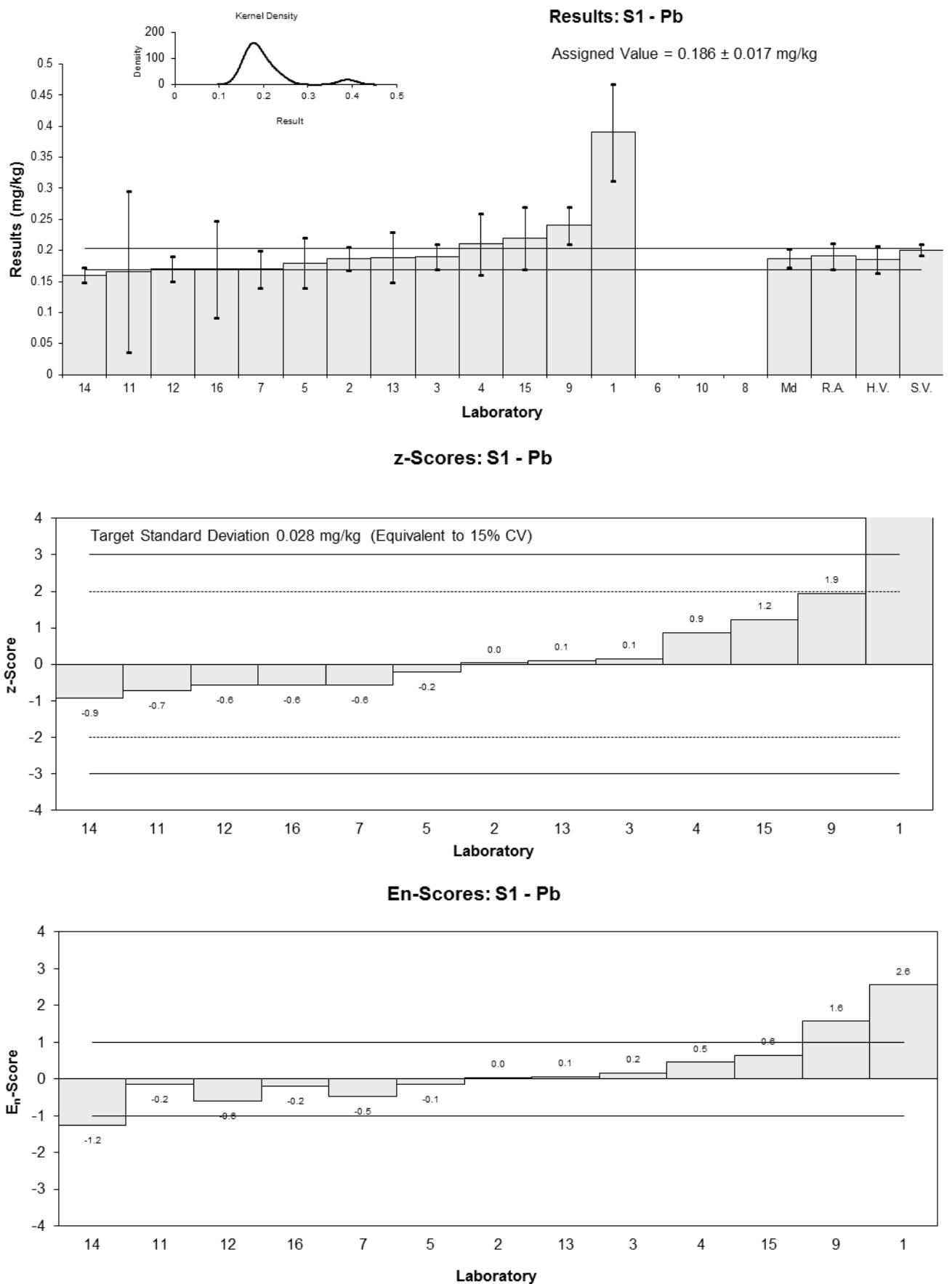


Figure 22

Table 29

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	NR	NR
3	NT	NT
4	NR	NR
5	6.37	1.27
6	NT	NT
7	7.0	1.4
8	NT	NT
9	NR	NR
10	NT	NT
11	6.67	4.7
12	NT	NT
13	NT	NT
14	7.0	NR
15	NT	NT
16	7.2	1.1

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	7.07	0.85
Robust Average	6.85	0.42
Median	7.00	0.37
Mean	6.85	
N	5	
Max.	7.2	
Min.	6.37	
Robust SD	0.37	
Robust CV	5.4%	

Results: S1 - Rb

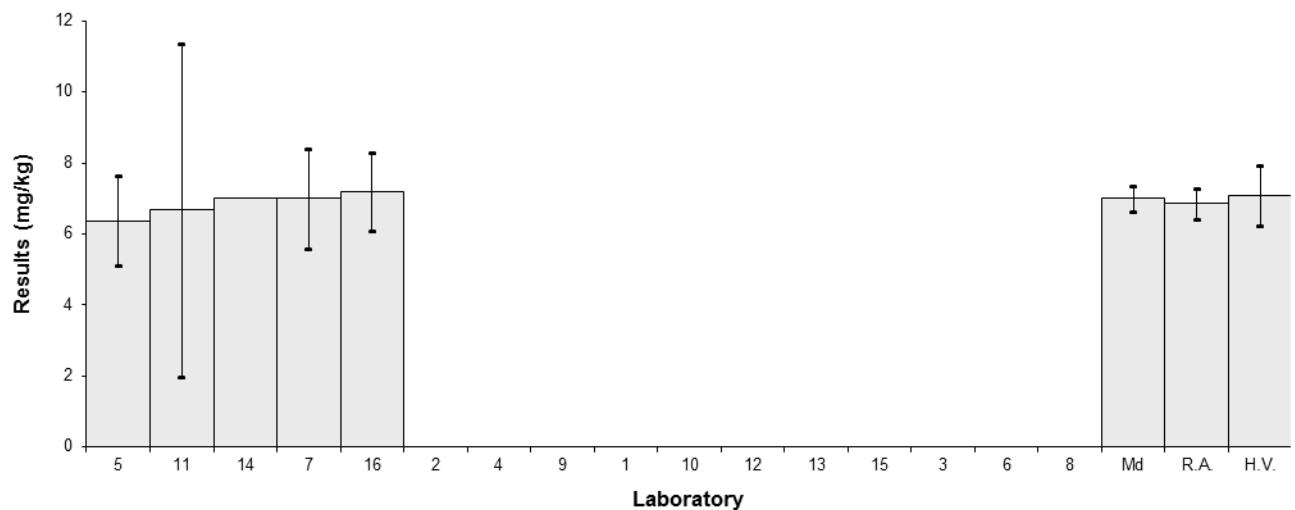


Figure 23

Table 30

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	S
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	10000	2000	-1.67	-0.88
2	NR	NR		
3	13800	1100	1.50	1.16
4	13000	1500	0.83	0.54
5	10371	2074	-1.36	-0.69
6	10800	1600	-1.00	-0.62
7	11400	2280	-0.50	-0.24
8	NT	NT		
9	12000	1200	0.00	0.00
10	NT	NT		
11	12800	1300	0.67	0.47
12	13004	1300	0.84	0.59
13	10915.50	2183.10	-0.90	-0.44
14	NT	NT		
15	11900	1300	-0.08	-0.06
16	13700	4200	1.42	0.39

Statistics

Assigned Value	12000	1100
Spike	Not Spiked	
Robust Average	12000	1100
Median	12000	990
Mean	11974	
N	12	
Max.	13800	
Min.	10000	
Robust SD	1500	
Robust CV	13%	

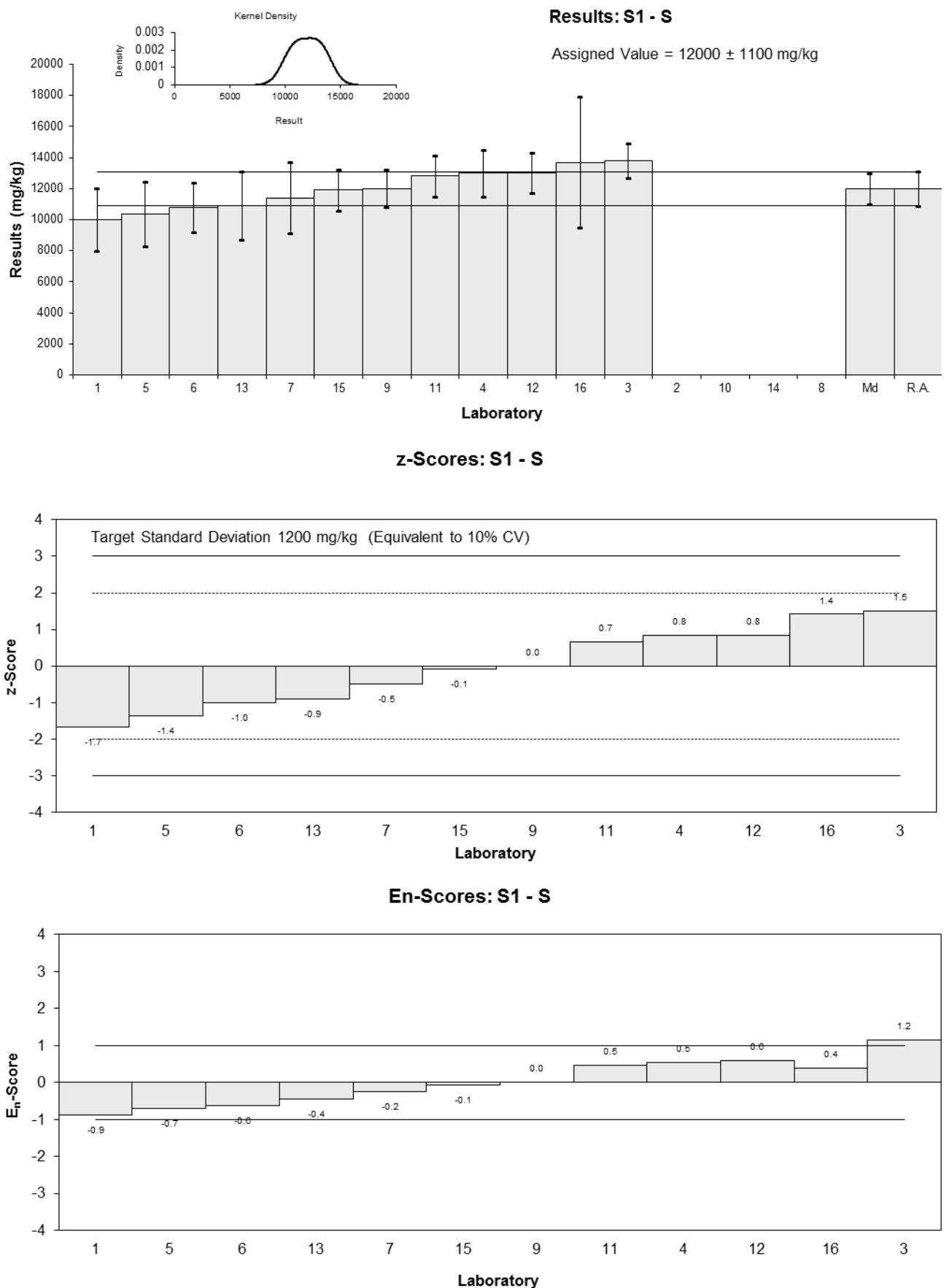


Figure 24

Table 31

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.09	0.018	-1.00	-0.51
2	0.109	0.011	0.90	0.66
3	0.11	0.01	1.00	0.78
4	<2	NR		
5	0.10	0.020	0.00	0.00
6	< 10	NR		
7	0.092	0.018	-0.80	-0.41
8	NT	NT		
9	NR	NR		
10	NT	NT		
11	< 0.5	0.48		
12	0.1	0.01	0.00	0.00
13	NT	NT		
14	0.1	NR	0.00	0.00
15	<1	NR		
16	0.16	0.032	6.00	1.82

Statistics

Assigned Value*	0.100	0.008
Spike	0.111	0.003
Homogeneity Value	0.108	0.013
Robust Average	0.102	0.010
Median	0.100	0.011
Mean	0.108	
N	8	
Max.	0.16	
Min.	0.09	
Robust SD	0.011	
Robust CV	11%	

*Robust Average excluding Laboratory 16

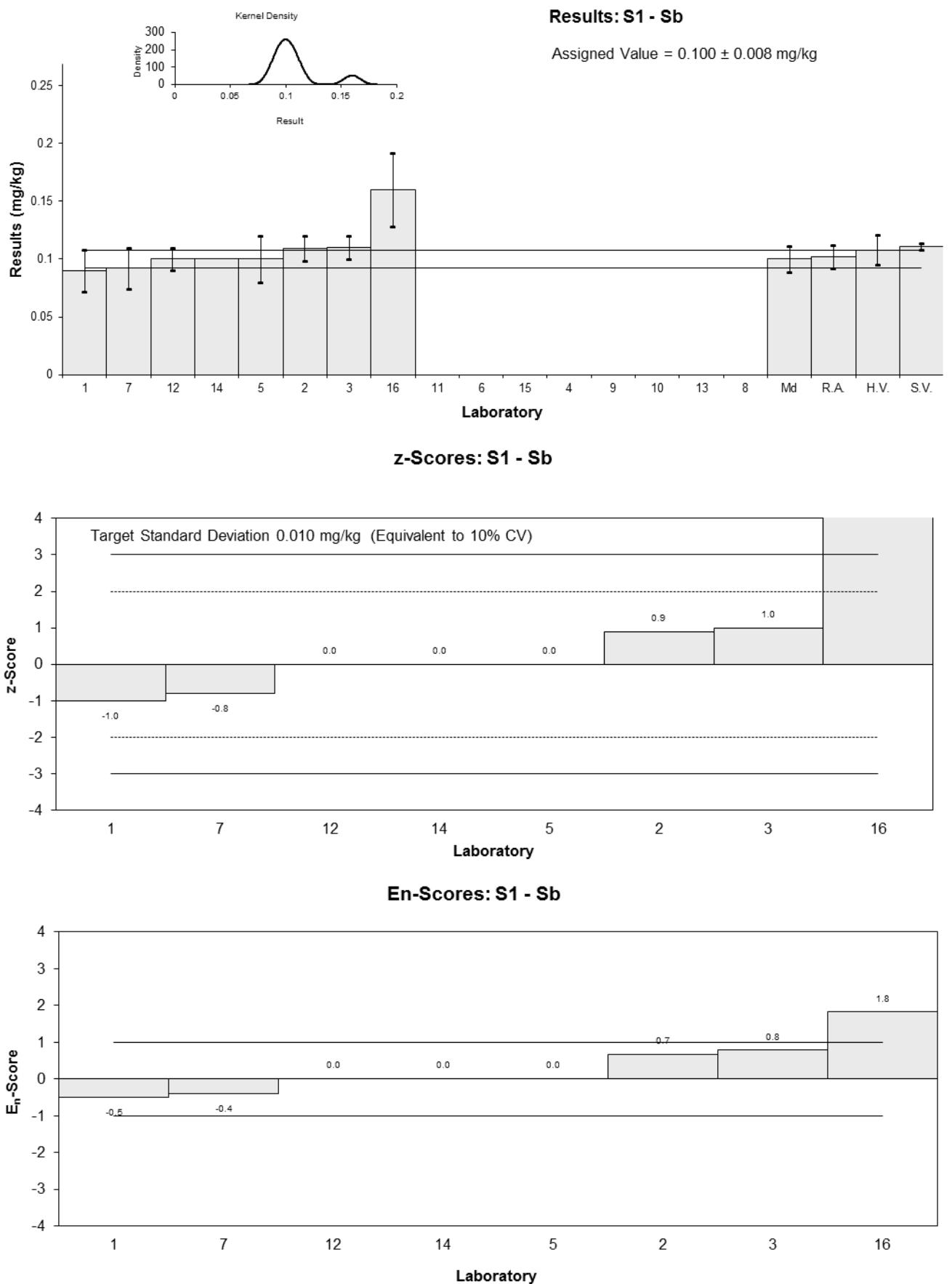


Figure 25

Table 32

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	2.5	0.5	-1.04	-0.77
2	3.41	0.341	1.01	0.95
3	3.0	0.4	0.09	0.08
4	2.7	0.7	-0.59	-0.34
5	2.39	0.48	-1.28	-0.98
6	< 5	NR		
7	2.6	0.52	-0.81	-0.58
8	NT	NT		
9	3.32	0.35	0.81	0.75
10	NT	NT		
11	2.62	0.37	-0.77	-0.69
12	3.85	0.385	2.00	1.76
13	3.560	0.71	1.35	0.77
14	3.01	0.91	0.11	0.05
15	2.85	0.5	-0.25	-0.18
16	2.8	1.2	-0.36	-0.13

Statistics

Assigned Value	2.96	0.33
Spike	2.69	0.19
Homogeneity Value	2.75	0.33
Robust Average	2.96	0.33
Median	2.85	0.22
Mean	2.97	
N	13	
Max.	3.85	
Min.	2.39	
Robust SD	0.47	
Robust CV	16%	

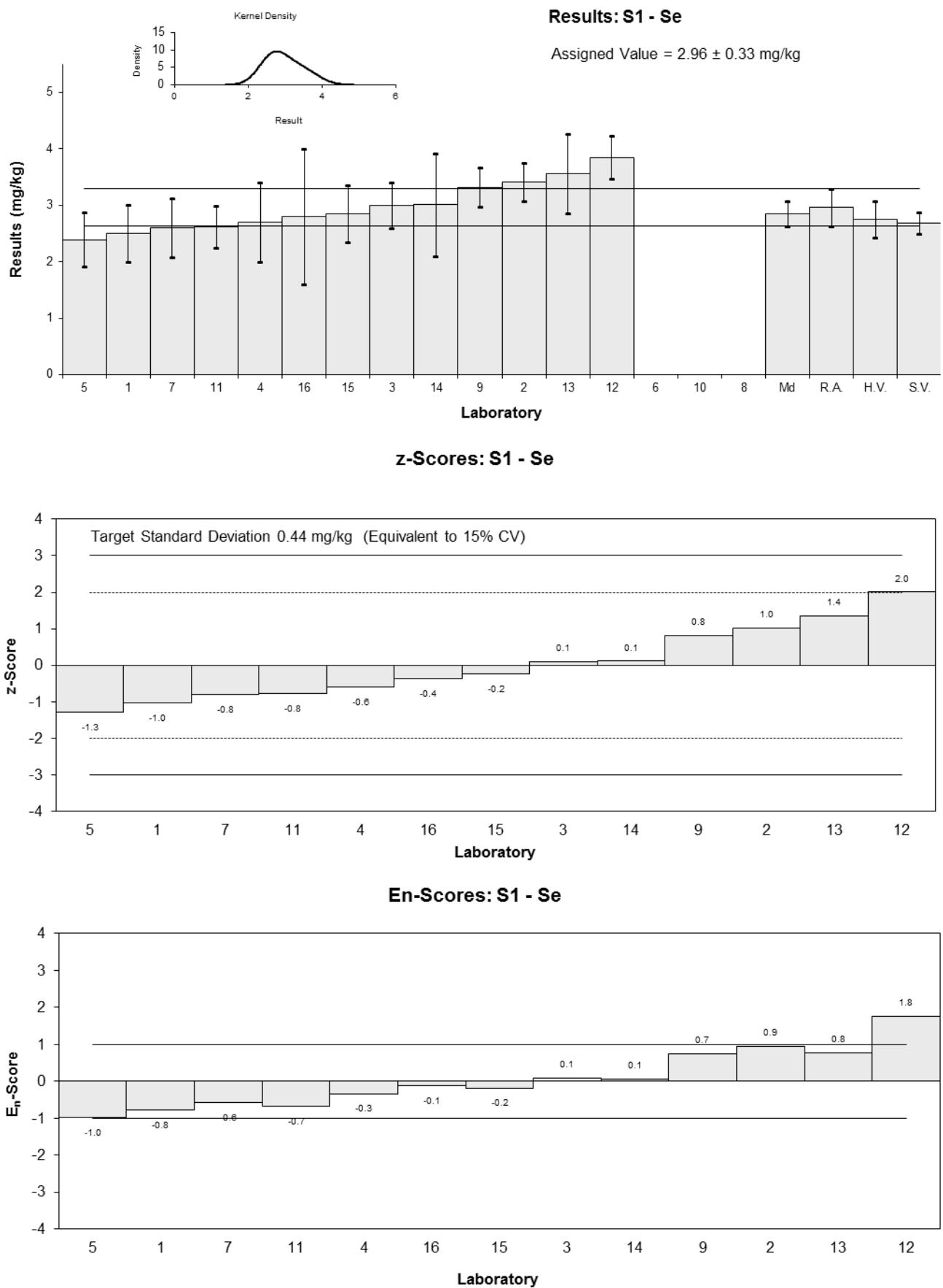


Figure 26

Table 33

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	0.146	0.015	-3.53	-3.39
3	0.31	0.01	0.00	0.00
4	<2	NR		
5	0.34	0.068	0.65	0.37
6	< 10	NR		
7	0.27	0.05	-0.86	-0.59
8	NT	NT		
9	NR	NR		
10	NT	NT		
11	< 0.5	0.47		
12	0.3	0.03	-0.22	-0.18
13	NT	NT		
14	<0.5	NR		
15	0.37	0.07	1.29	0.72
16	0.27	0.19	-0.86	-0.20

Statistics

Assigned Value*	0.310	0.046
Spike	0.320	0.009
Homogeneity Value	0.280	0.034
Robust Average	0.295	0.058
Median	0.300	0.041
Mean	0.287	
N	7	
Max.	0.37	
Min.	0.146	
Robust SD	0.062	
Robust CV	21%	

*Robust Average excluding Laboratory 2

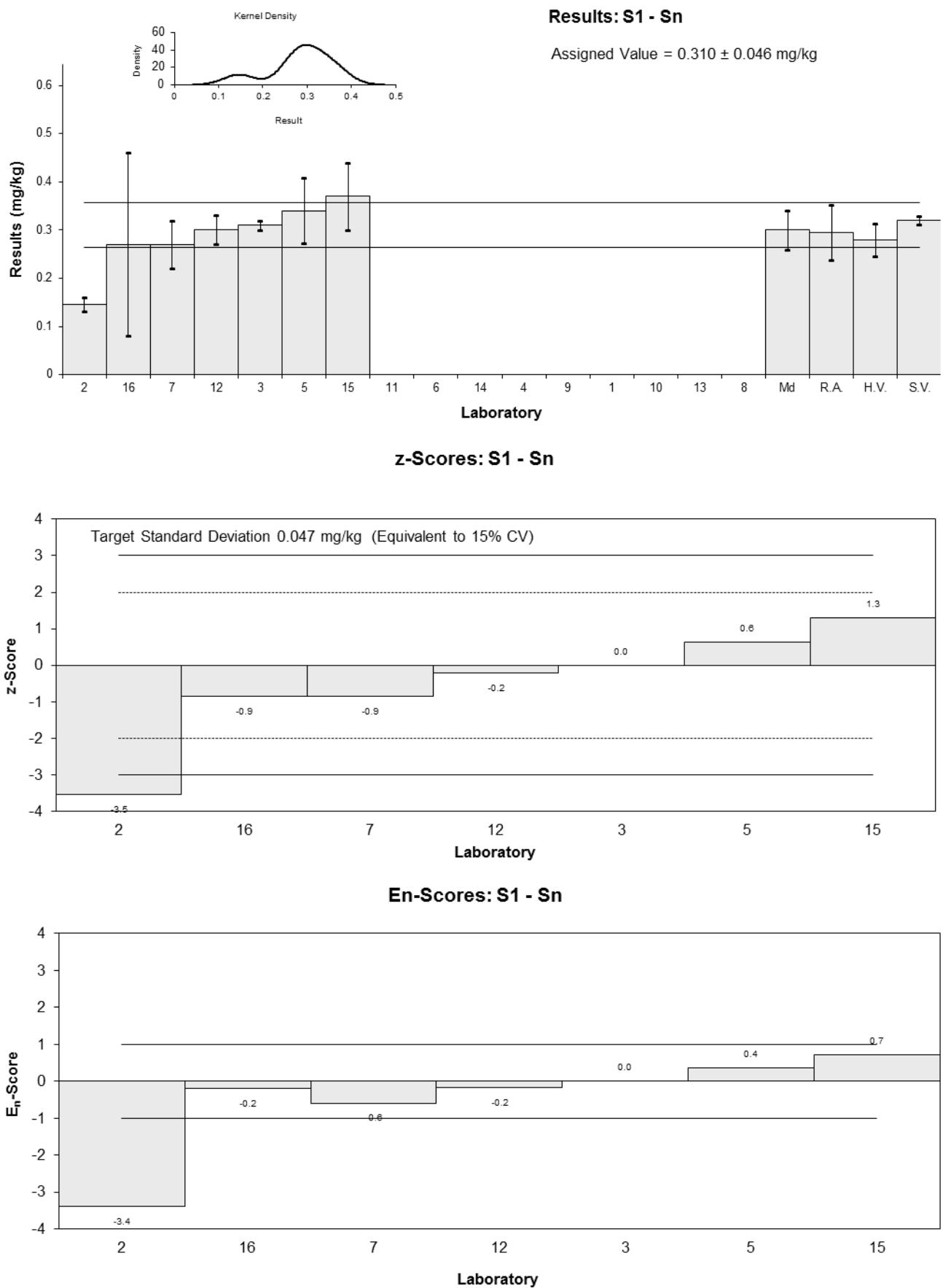


Figure 27

Table 34

Sample Details

Sample No.	S1
Matrix.	Fish
Analyte.	U
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.07	0.014	0.92	0.54
2	NR	NR		
3	NT	NT		
4	0.067	0.012	0.60	0.39
5	<0.05	0.009		
6	< 10	NR		
7	0.06	0.012	-0.16	-0.11
8	NT	NT		
9	0.074	0.01	1.36	1.01
10	NT	NT		
11	0.0617	0.044	0.02	0.00
12	0.055	0.01	-0.70	-0.53
13	NT	NT		
14	0.06	NR	-0.16	-0.21
15	0.05	0.02	-1.25	-0.54
16	0.056	0.02	-0.60	-0.26

Statistics

Assigned Value	0.0615	0.0072
Spike	0.0657	0.0019
Homogeneity Value	0.0603	0.0072
Robust Average	0.0615	0.0072
Median	0.0600	0.0057
Mean	0.0615	
N	9	
Max.	0.074	
Min.	0.05	
Robust SD	0.0087	
Robust CV	14%	

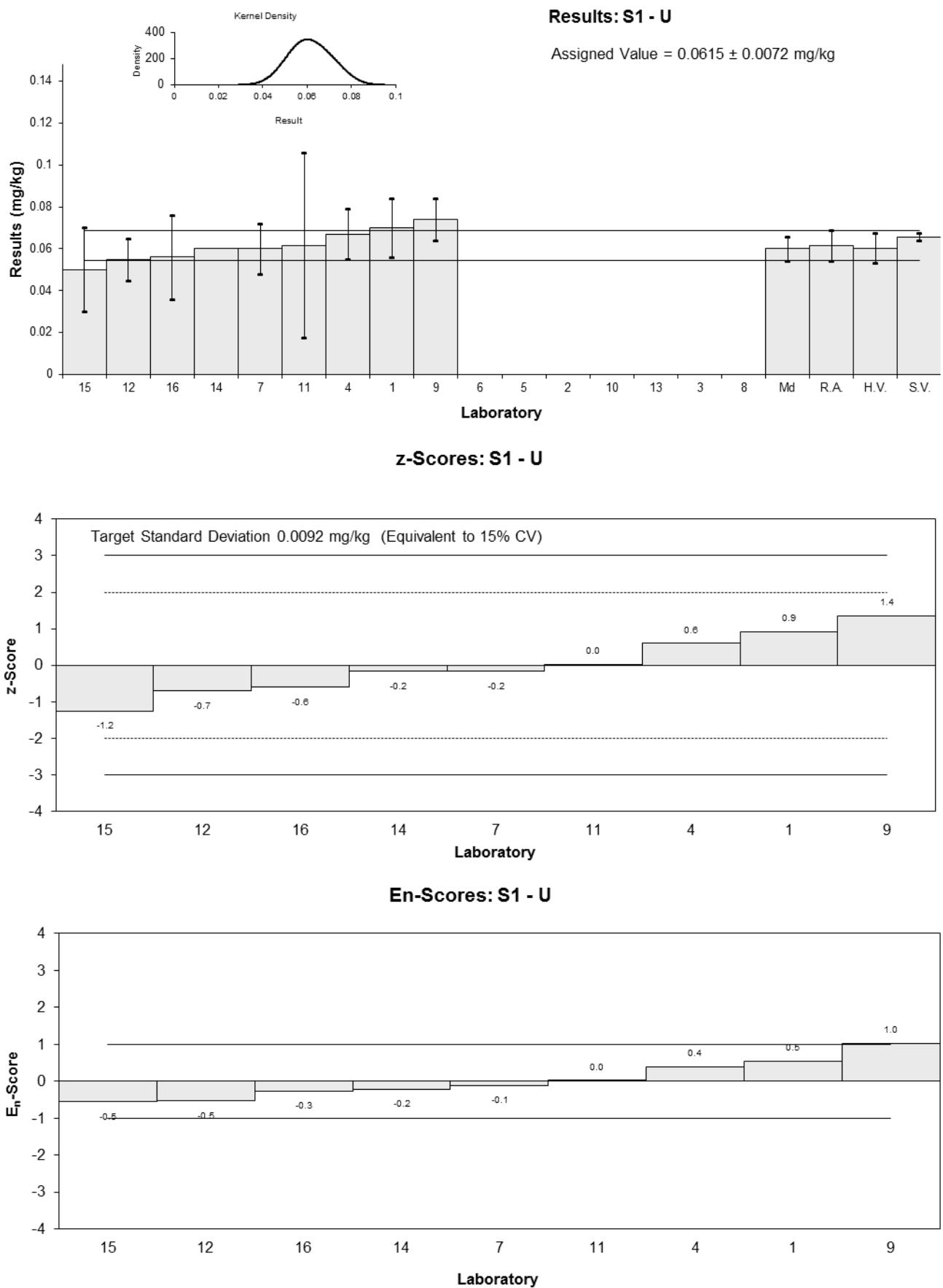


Figure 28

Table 35

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	NR	NR		
3	NT	NT		
4	0.32	0.1	-0.60	-0.31
5	0.44	0.087	1.04	0.58
6	< 5	NR		
7	0.31	0.06	-0.74	-0.47
8	NT	NT		
9	NT	NT		
10	NT	NT		
11	0.501	0.39	1.88	0.34
12	0.31	0.031	-0.74	-0.52
13	NT	NT		
14	<0.5	NR		
15	<1	NR		
16	0.3	0.083	-0.88	-0.50

Statistics

Assigned Value	0.364	0.099
Spike	0.385	0.013
Homogeneity Value	0.328	0.039
Robust Average	0.364	0.099
Median	0.315	0.016
Mean	0.364	
N	6	
Max.	0.501	
Min.	0.3	
Robust SD	0.097	
Robust CV	27%	

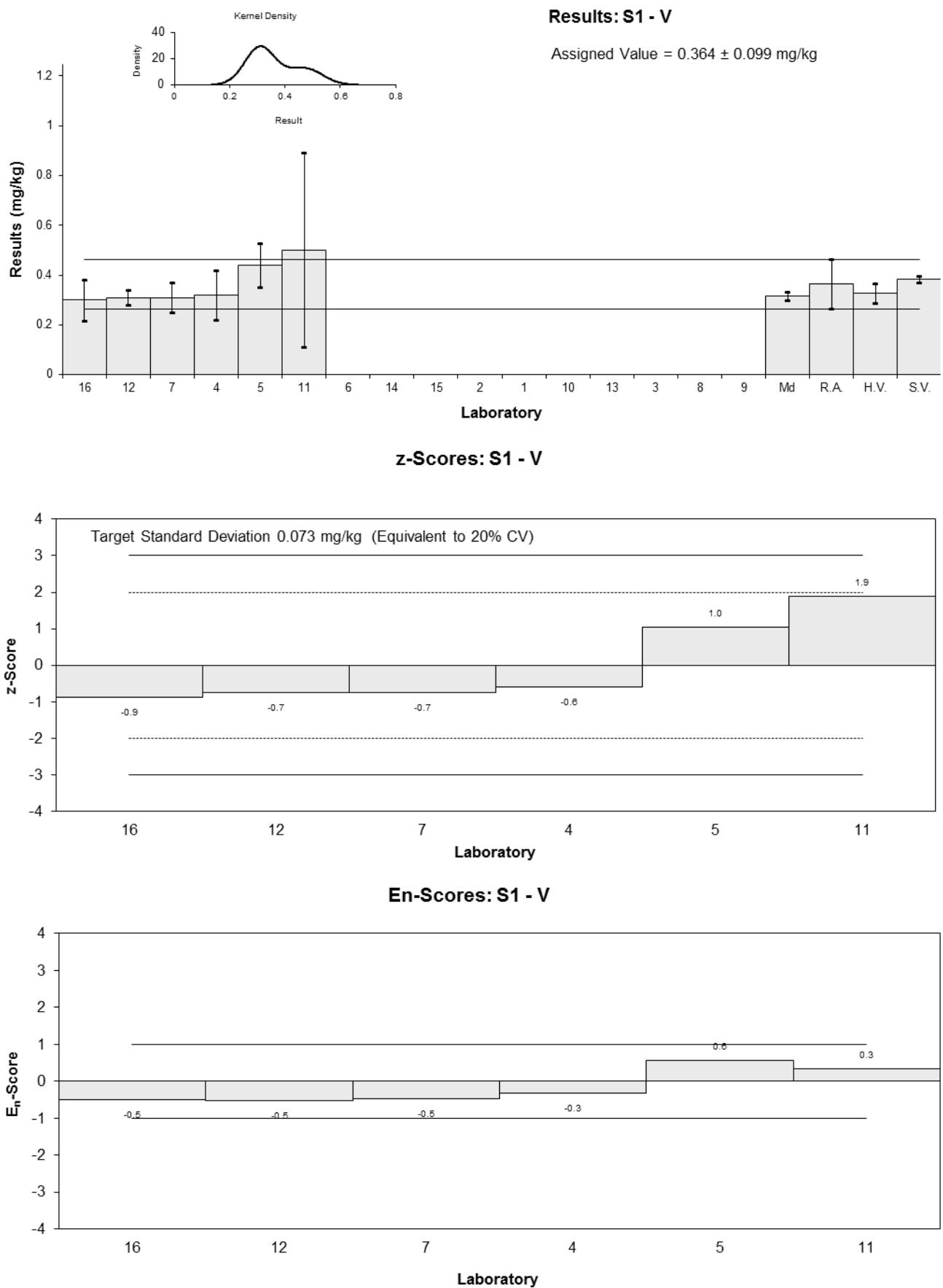


Figure 29

Table 36

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	37	7.4	0.63	0.29
2	34.1	3.41	-0.20	-0.19
3	37	5	0.63	0.43
4	34	5	-0.23	-0.15
5	29.90	5.98	-1.41	-0.80
6	35.2	5.3	0.11	0.07
7	35	7	0.06	0.03
8	NT	NT		
9	34.4	3.8	-0.11	-0.10
10	NT	NT		
11	33.1	24	-0.49	-0.07
12	36	3.6	0.34	0.31
13	30.562	6.11	-1.22	-0.68
14	36.6	10.5	0.52	0.17
15	35.8	6.0	0.29	0.16
16	35	6	0.06	0.03

Statistics

Assigned Value	34.8	1.3
Spike	Not Spiked	
Homogeneity Value	32.8	3.9
Robust Average	34.8	1.3
Median	35.0	0.9
Mean	34.5	
N	14	
Max.	37	
Min.	29.9	
Robust SD	1.9	
Robust CV	5.5%	

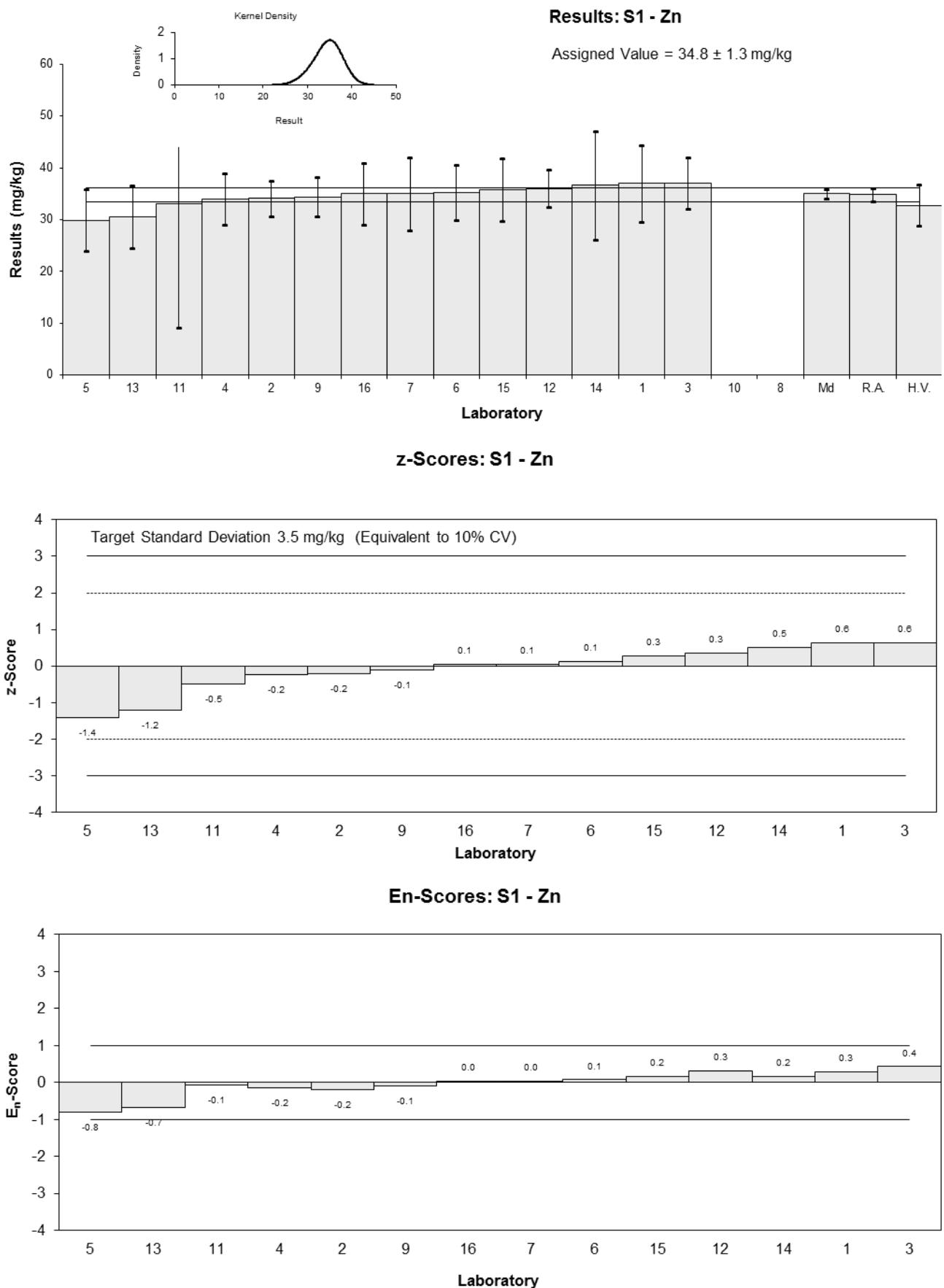


Figure 30

Table 37

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	7.01	0.701
3	10	3
4	NT	NT
5	4.30	0.86
6	< 20	NR
7	6.9	1.4
8	5.1	2
9	NR	NR
10	13.34	10
11	NT	NT
12	9.6	1
13	4.191	0.84
14	NT	NT
15	14.1	1.6
16	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Information Value*	12.8	0.51
Robust Average	8.3	3.5
Median	7.0	3.1
Mean	8.3	
N	9	
Max.	14.1	
Min.	4.191	
Robust SD	4.2	
Robust CV	51%	

*Information Value by SA-ICP-MS

Results: S2 - Al

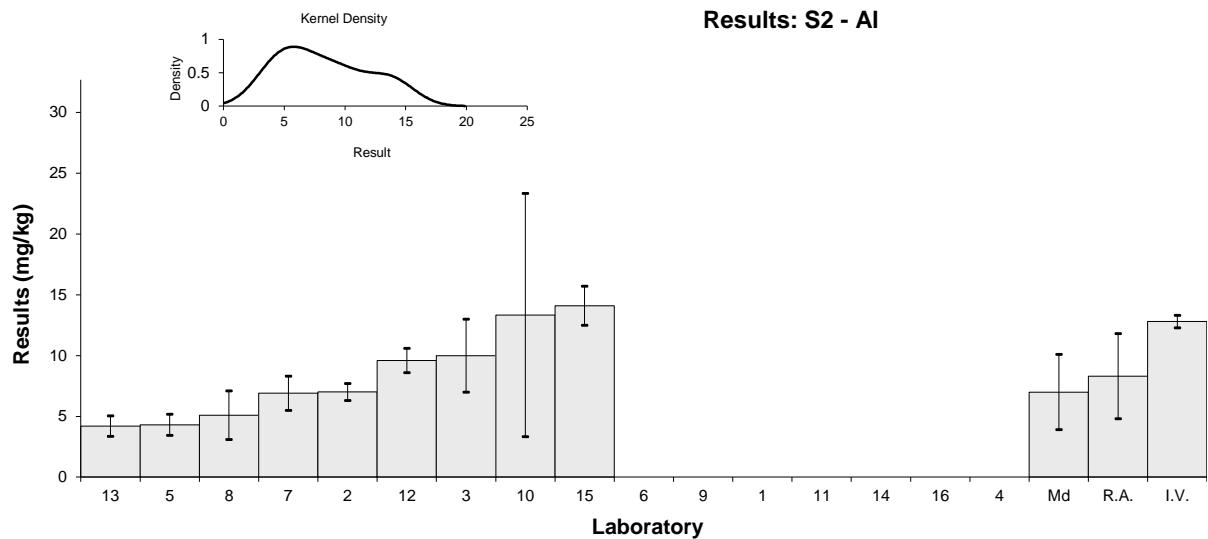


Figure 31

Table 38

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	0.014	0.0014	-0.05	-0.10
3	0.019	0.01	1.37	0.48
4	NT	NT		
5	0.02	0.00	1.65	8.59
6	< 5	NR		
7	<0.05	NR		
8	<0.1	NR		
9	NT	NT		
10	<0.2	0.2		
11	NT	NT		
12	0.016	0.002	0.52	0.87
13	< 0.5	NR		
14	NT	NT		
15	<1	NR		
16	NT	NT		

Statistics

Assigned Value*	0.01416	0.00068
Spike	Not Spiked	
Reference Value*	0.01416	0.00068
Robust Average	0.0173	0.0039
Median	0.0175	0.0047
Mean	0.0173	
N	4	
Max.	0.02	
Min.	0.014	
Robust SD	0.0031	
Robust CV	18%	

*Reference Value by SA-ICP-MS

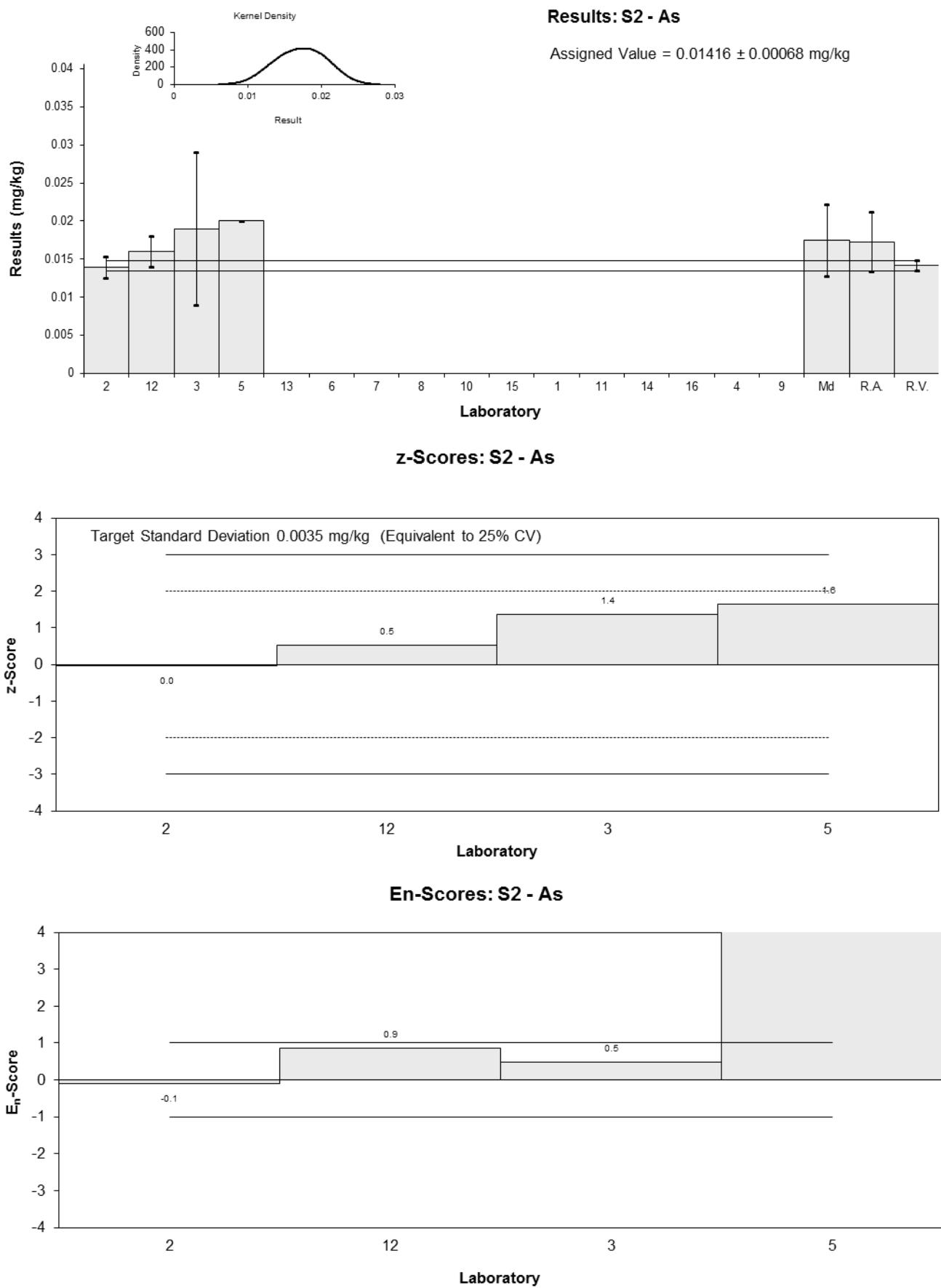


Figure 32

Table 39

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	B
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	NR	NR
3	NT	NT
4	NT	NT
5	11.81	2.36
6	< 50	NR
7	NR	NR
8	15	5
9	17.1	0.7
10	16	0.9
11	NT	NT
12	NT	NT
13	NT	NT
14	NT	NT
15	22.0	2.8
16	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	15.4	3.1
Robust Average	16.4	4.7
Median	16.0	2.0
Mean	16.4	
N	5	
Max.	22	
Min.	11.81	
Robust SD	4.2	
Robust CV	26%	

Results: S2 - B

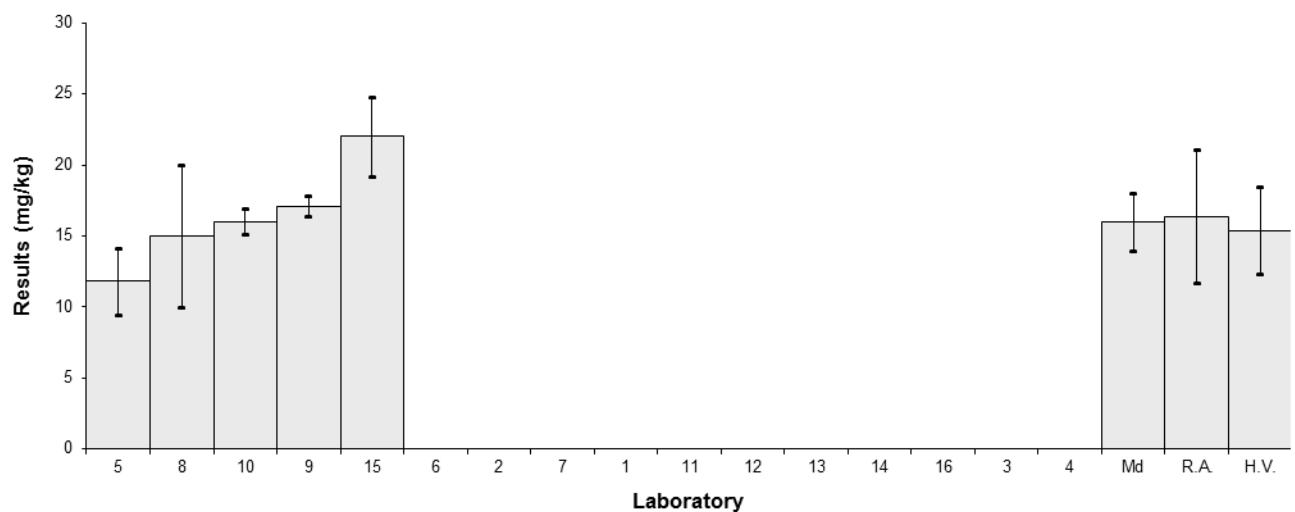


Figure 33

Table 40

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	NR	NR		
3	NT	NT		
4	NT	NT		
5	1.95	0.39	-0.05	-0.02
6	< 10	NR		
7	2.1	0.42	0.71	0.32
8	1.8	0.6	-0.82	-0.26
9	2.24	0.24	1.43	1.04
10	1.9	0.19	-0.31	-0.27
11	NT	NT		
12	1.9	0.19	-0.31	-0.27
13	1.951	0.39	-0.05	-0.02
14	NT	NT		
15	1.90	0.4	-0.31	-0.14
16	NT	NT		

Statistics

Assigned Value	1.96	0.12
Spike	Not Spiked	
Homogeneity Value	1.76	0.35
Robust Average	1.96	0.12
Median	1.93	0.03
Mean	1.97	
N	8	
Max.	2.24	
Min.	1.8	
Robust SD	0.13	
Robust CV	6.6%	

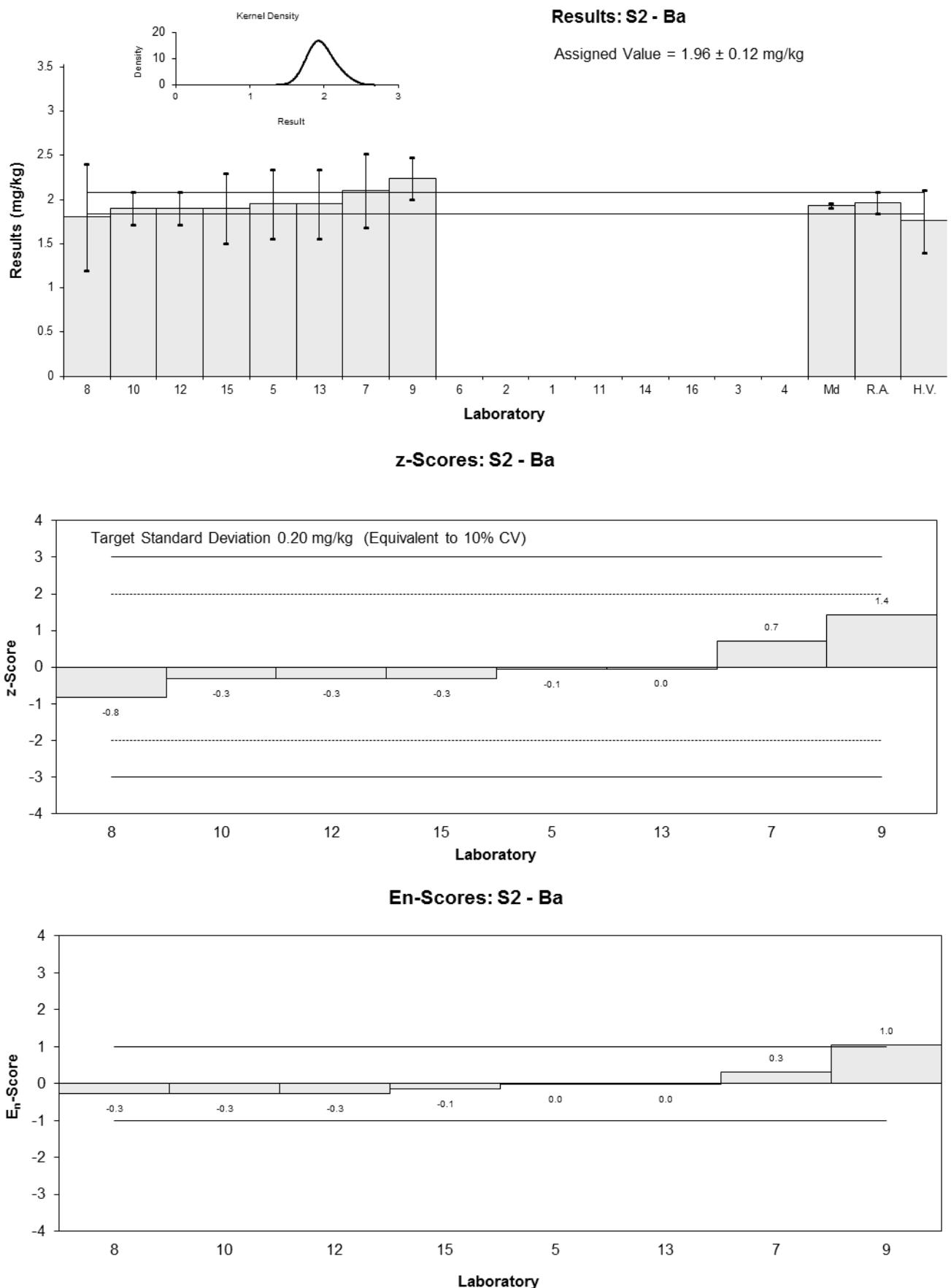


Figure 34

Table 41

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	Ca
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	1230	123	1.39	0.95
3	1100	60	0.19	0.17
4	NT	NT		
5	1136	227.20	0.52	0.23
6	1040	160	-0.37	-0.21
7	930	190	-1.39	-0.70
8	950	320	-1.20	-0.39
9	96.8	4.3	-9.10	-9.82
10	1031	64	-0.45	-0.41
11	NT	NT		
12	1029	103	-0.47	-0.36
13	1063.02	212.60	-0.16	-0.07
14	NT	NT		
15	1260	150	1.67	1.00
16	NT	NT		

Statistics

Assigned Value*	1080	100
Spike	Not Spiked	
Homogeneity Value	1100	130
Robust Average	1060	110
Median	1040	90
Mean	988	
N	11	
Max.	1260	
Min.	96.8	
Robust SD	140	
Robust CV	13%	

*Robust Average excluding Laboratory 9

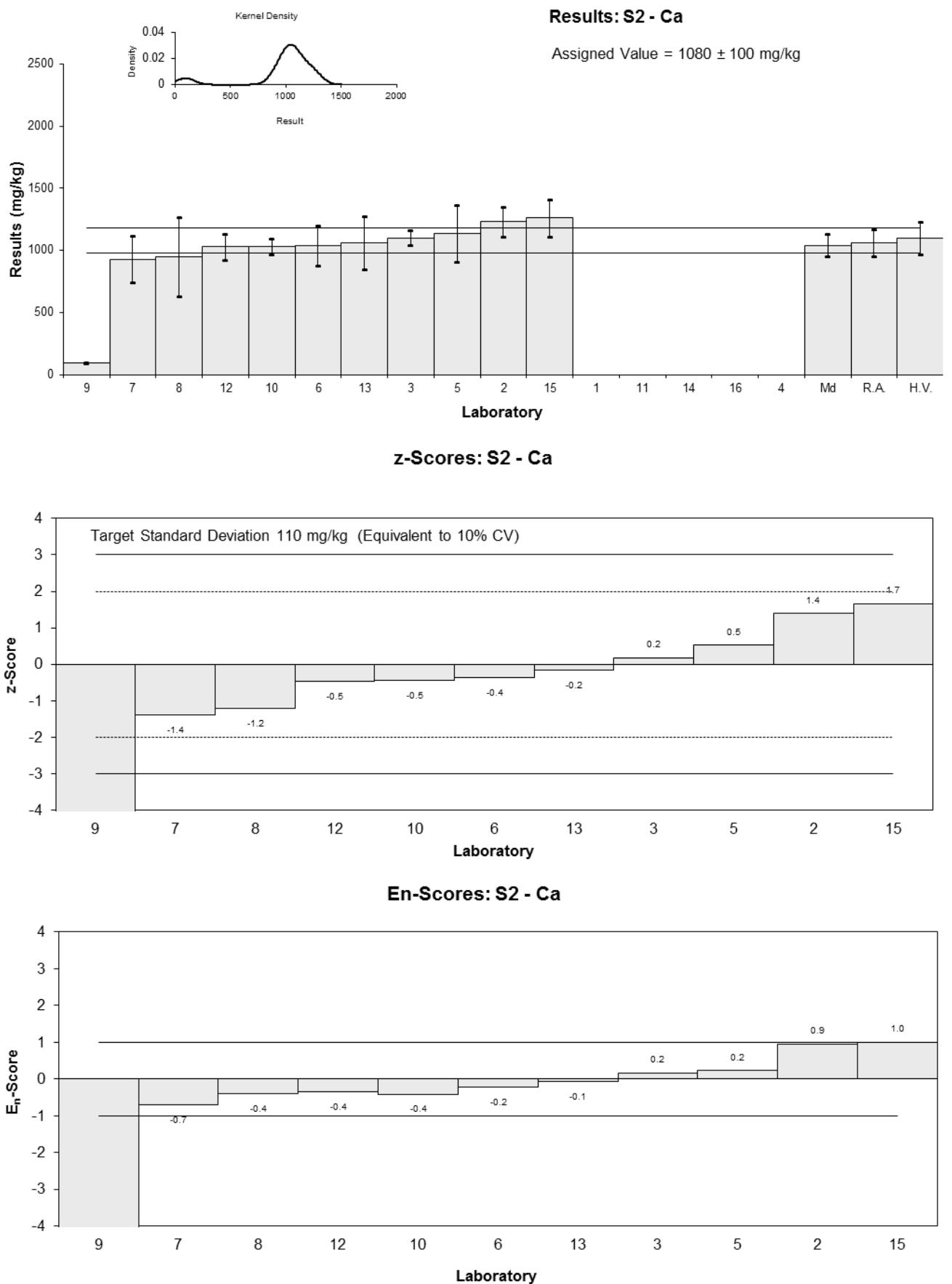


Figure 35

Table 42

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	0.005	0.0005	-0.04	-0.06
3	<0.01	0.001		
4	NT	NT		
5	<0.01	0.00		
6	< 1	NR		
7	<0.01	NR		
8	<0.1	NR		
9	0.006	0.004	0.75	0.23
10	<0.02	0.02		
11	NT	NT		
12	0.005	0.001	-0.04	-0.04
13	0.009	0.00	3.13	5.90
14	NT	NT		
15	<0.1	NR		
16	NT	NT		

Statistics

Assigned Value*	0.00505	0.00067
Spike	Not Spiked	
Reference Value*	0.00505	0.00067
Robust Average	0.0063	0.0027
Median	0.0055	0.0012
Mean	0.0063	
N	4	
Max.	0.009	
Min.	0.005	
Robust SD	0.0021	
Robust CV	34%	

*Reference Value by SA-ICP-MS

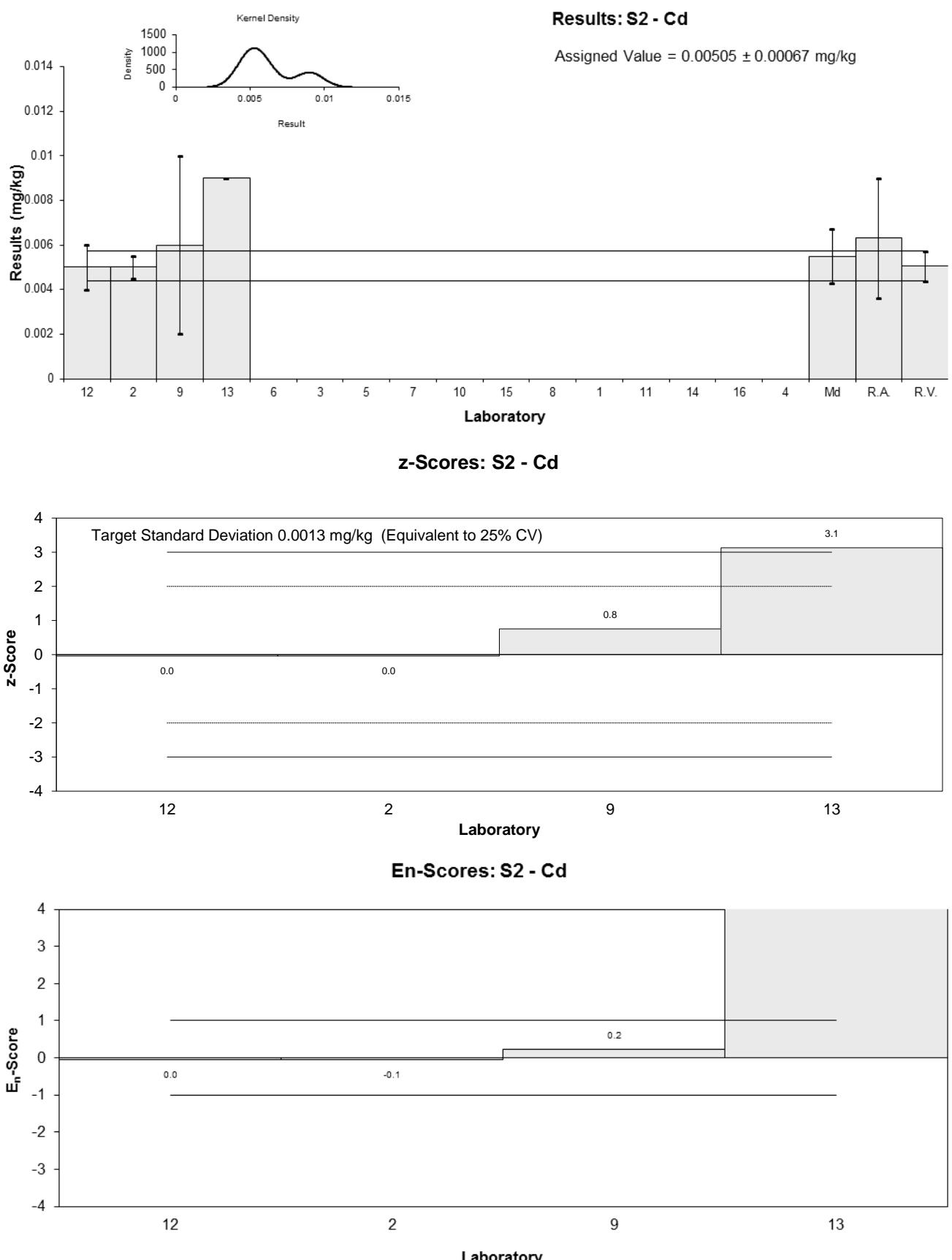


Figure 36

Table 43

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	0.058	0.006	-0.36	-0.35
3	0.062	0.015	0.08	0.04
4	NT	NT		
5	0.07	0.01	0.95	0.70
6	< 5	NR		
7	0.06	0.01	-0.14	-0.10
8	0.06	0.04	-0.14	-0.03
9	0.064	0.01	0.29	0.22
10	0.075	0.02	1.49	0.64
11	NT	NT		
12	0.041	0.004	-2.21	-2.41
13	0.055	0.01	-0.69	-0.51
14	NT	NT		
15	<0.1	NR		
16	NT	NT		

Statistics

Assigned Value	0.0613	0.0074
Spike	Not Spiked	
Homogeneity Value	0.0650	0.0130
Robust Average	0.0613	0.0074
Median	0.0600	0.0046
Mean	0.0606	
N	9	
Max.	0.075	
Min.	0.041	
Robust SD	0.0089	
Robust CV	15%	

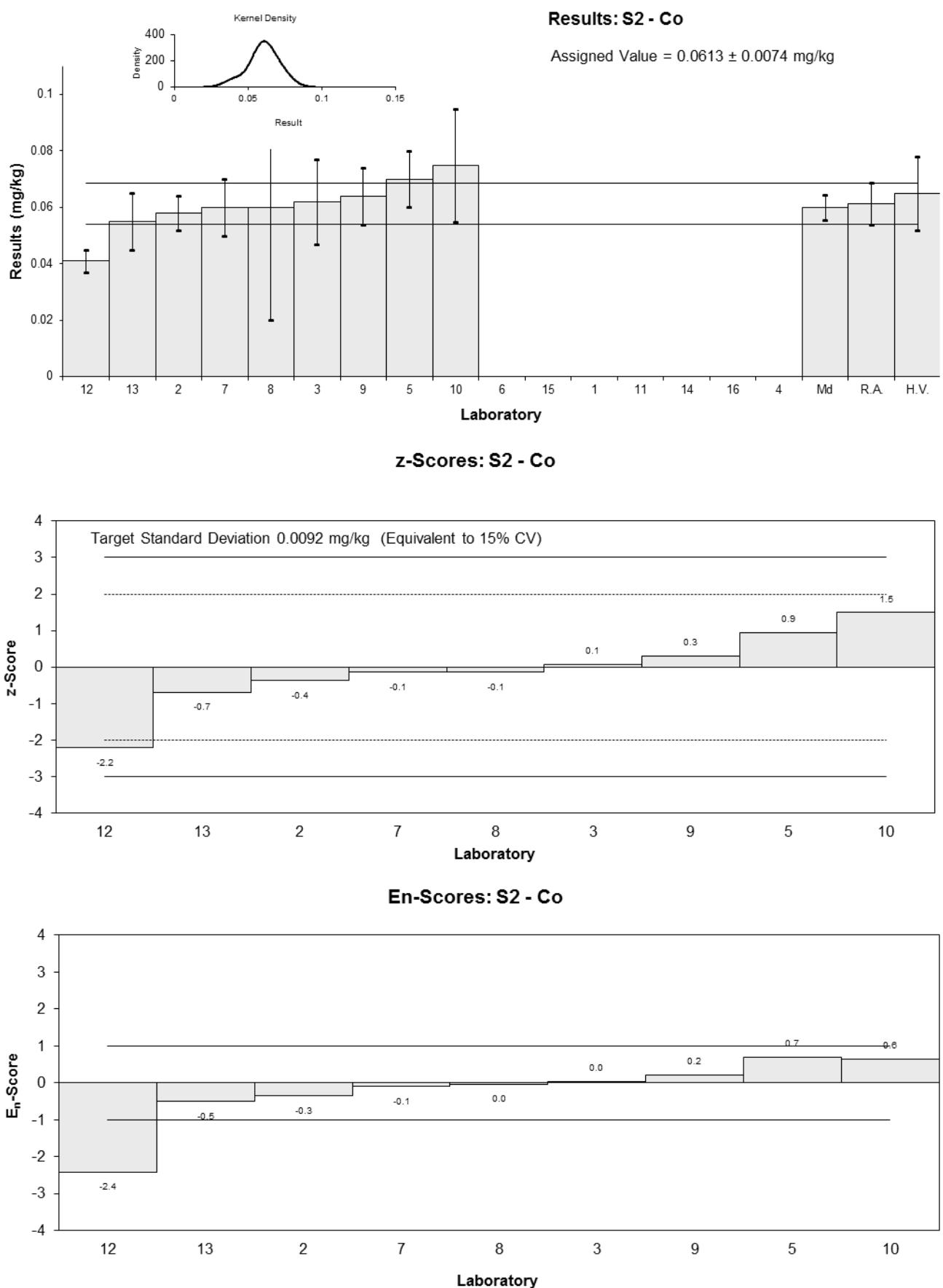


Figure 37

Table 44

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	0.228	0.023	-0.67	-1.27
3	0.22	0.05	-0.82	-0.82
4	NT	NT		
5	0.30	0.06	0.7	0.6
6	< 5	NR		
7	0.21	0.04	-1.01	-1.24
8	0.21	0.1	-1.01	-0.52
9	NR	NR		
10	<1	1		
11	NT	NT		
12	0.25	0.03	-0.25	-0.39
13	0.385	0.08	2.32	1.5
14	NT	NT		
15	<1	NR		
16	NT	NT		

Statistics

Assigned Value*	0.263	0.015
Spike	Not Spiked	
Reference Value*	0.263	0.015
Robust Average	0.250	0.051
Median	0.228	0.025
Mean	0.258	
N	7	
Max.	0.385	
Min.	0.21	
Robust SD	0.054	
Robust CV	22%	

*Reference Value by IDMS

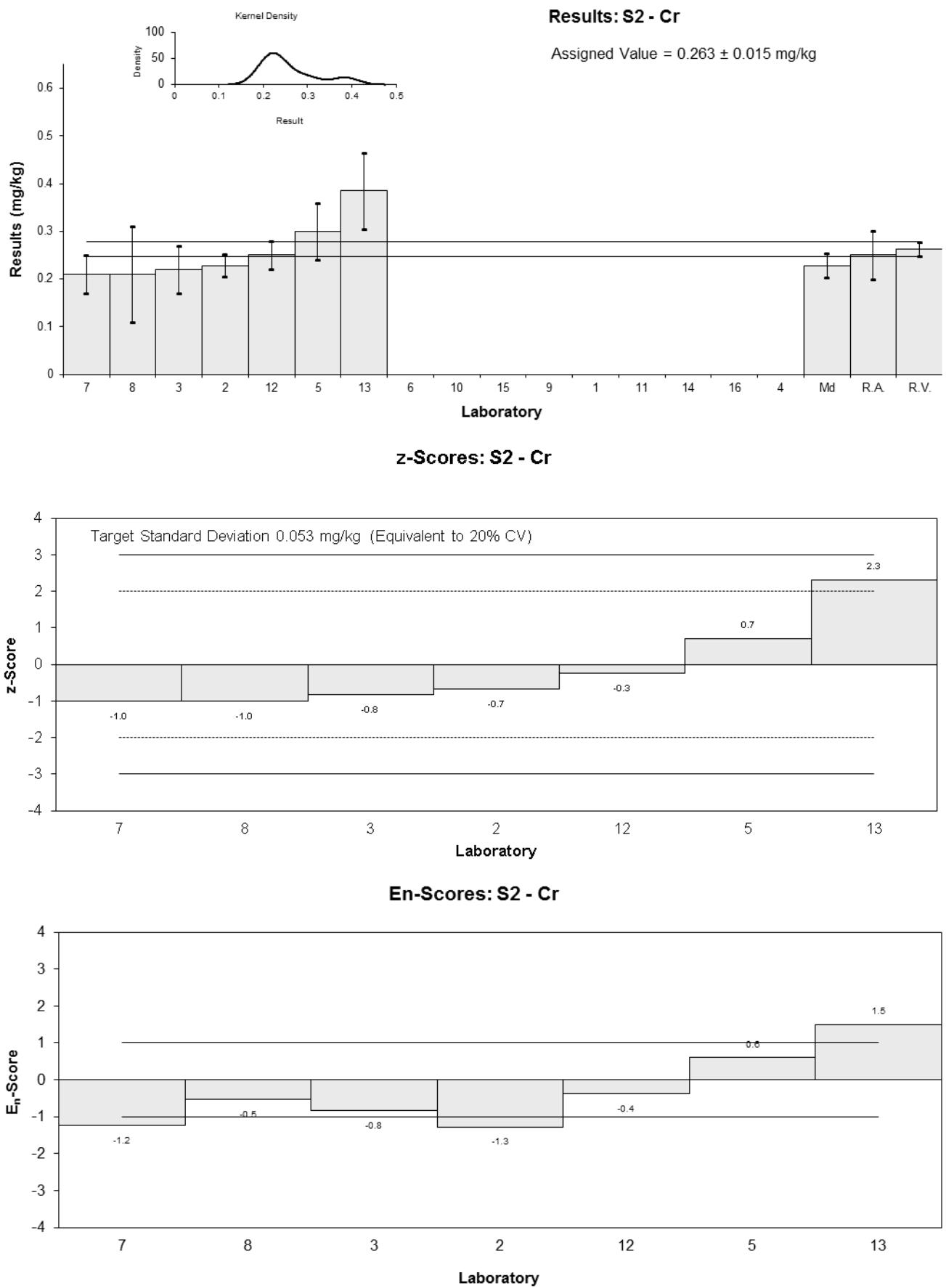


Figure 38

Table 45

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	14.2	1.42	0.84	0.69
3	13	3	-0.08	-0.03
4	NT	NT		
5	12.19	2.44	-0.69	-0.36
6	< 20	NR		
7	13	2.6	-0.08	-0.04
8	12	4	-0.84	-0.27
9	14.1	0.6	0.76	1.08
10	13	1	-0.08	-0.08
11	NT	NT		
12	14	1.4	0.69	0.57
13	12.886	2.58	-0.16	-0.08
14	NT	NT		
15	12.6	1.5	-0.38	-0.30
16	NT	NT		

Statistics

Assigned Value	13.1	0.7
Spike	Not Spiked	
Homogeneity Value	12.6	2.5
Robust Average	13.1	0.7
Median	13.0	0.6
Mean	13.1	
N	10	
Max.	14.2	
Min.	12	
Robust SD	0.88	
Robust CV	6.7%	

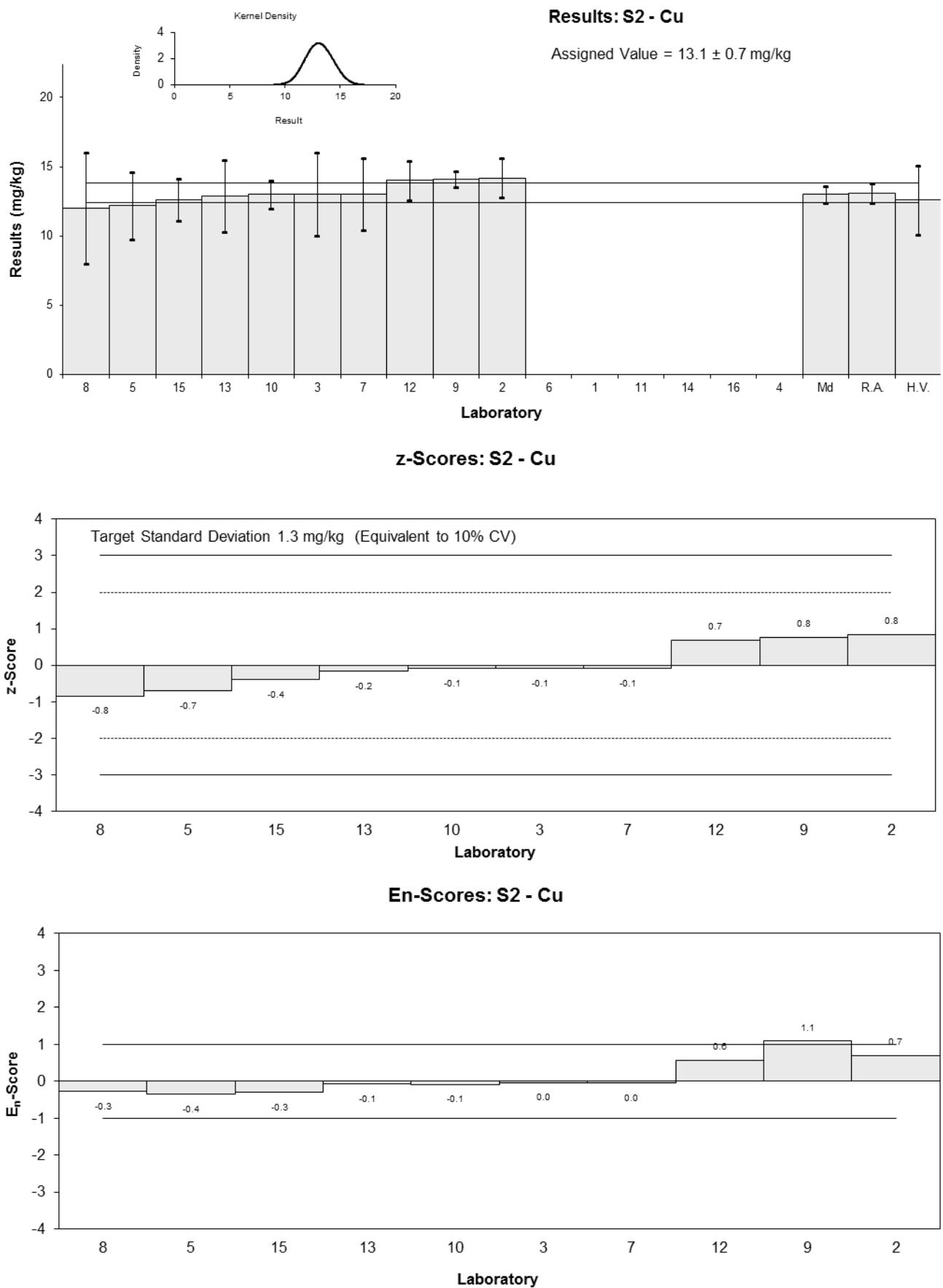


Figure 39

Table 46

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	Fe
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	129	12.9	0.66	0.49
3	130	20	0.74	0.40
4	NT	NT		
5	111.42	22.28	-0.79	-0.39
6	136	20	1.24	0.67
7	110	22	-0.91	-0.46
8	110	40	-0.91	-0.27
9	118	4	-0.25	-0.28
10	119	9.3	-0.17	-0.15
11	NT	NT		
12	145	14.5	1.98	1.36
13	113.181	22.64	-0.65	-0.32
14	NT	NT		
15	110	15	-0.91	-0.61
16	NT	NT		

Statistics

Assigned Value	121	10
Spike	Not Spiked	
Homogeneity Value	119	24
Robust Average	121	10
Median	118	8
Mean	121	
N	11	
Max.	145	
Min.	110	
Robust SD	13	
Robust CV	11%	

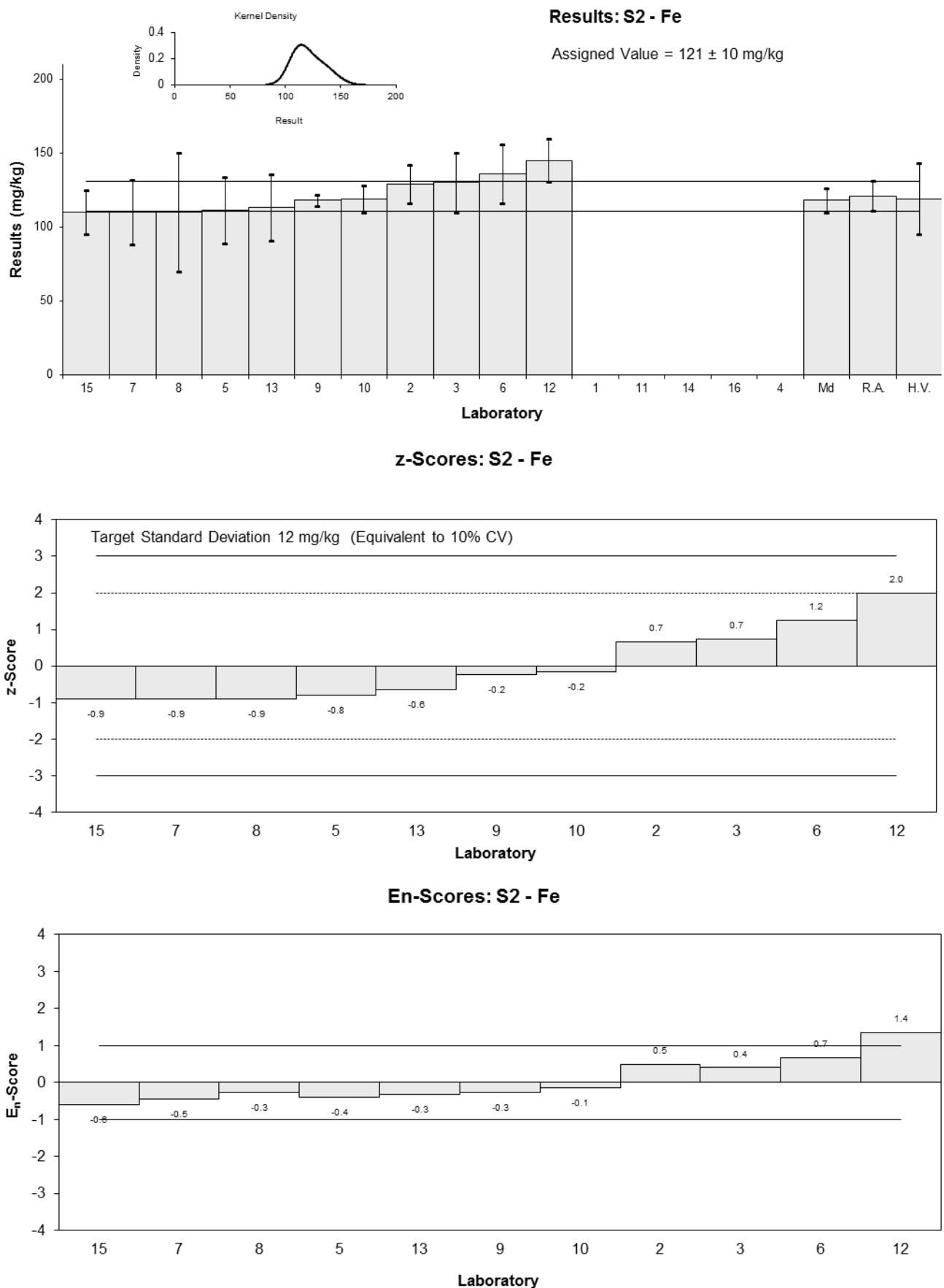


Figure 40

Table 47

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	K
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	8410	841	1.68	1.44
3	7000	480	0.28	0.29
4	NT	NT		
5	6683	1336.60	-0.04	-0.02
6	6380	960	-0.34	-0.27
7	6210	1240	-0.51	-0.34
8	5300	1800	-1.41	-0.72
9	736	16	-5.94	-7.30
10	5675	414	-1.04	-1.14
11	NT	NT		
12	6826	683	0.11	0.10
13	6859.00	1371.80	0.14	0.09
14	NT	NT		
15	7970	900	1.24	1.03
16	NT	NT		

Statistics

Assigned Value*	6720	820
Spike	Not Spiked	
Homogeneity Value	5990	900
Robust Average	6540	920
Median	6680	470
Mean	6186	
N	11	
Max.	8410	
Min.	736	
Robust SD	1200	
Robust CV	18%	

*Robust Average excluding Laboratory 9

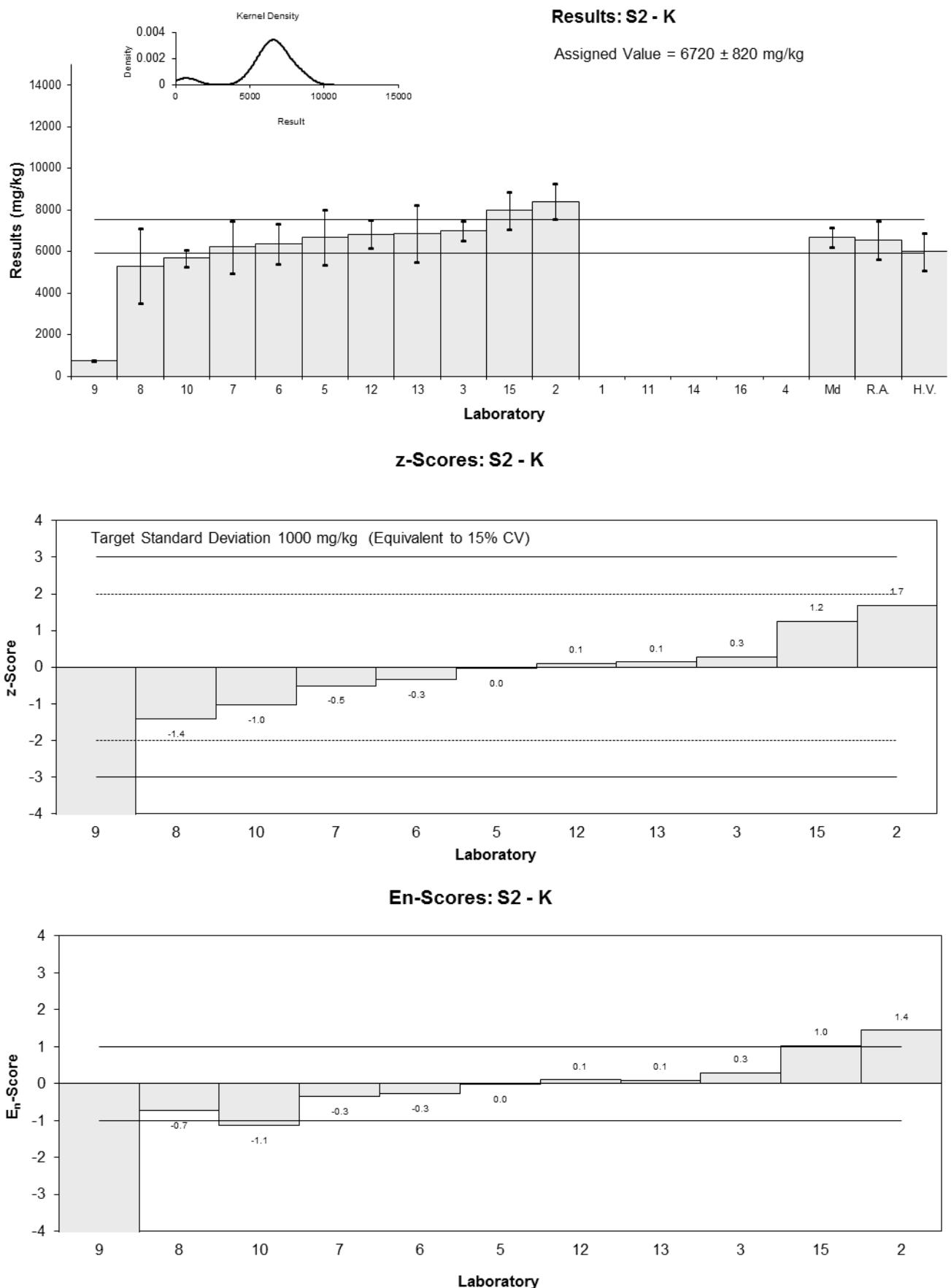


Figure 41

Table 48

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	Mg
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	3806	381	1.39	1.40
3	3190	80	0.08	0.14
4	NT	NT		
5	3176	635.20	0.06	0.04
6	3040	460	-0.23	-0.21
7	2730	550	-0.89	-0.69
8	2900	970	-0.53	-0.25
9	314	9	-6.00	-10.50
10	3091	370	-0.12	-0.13
11	NT	NT		
12	3025	303	-0.26	-0.31
13	2999.86	599.97	-0.32	-0.23
14	NT	NT		
15	3720	450	1.21	1.09
16	NT	NT		

Statistics

Assigned Value*	3150	270
Spike	Not Spiked	
Homogeneity Value	3700	700
Robust Average	3100	320
Median	3040	140
Mean	2908	
N	11	
Max.	3806	
Min.	314	
Robust SD	430	
Robust CV	14%	

*Robust Average excluding Laboratory 9

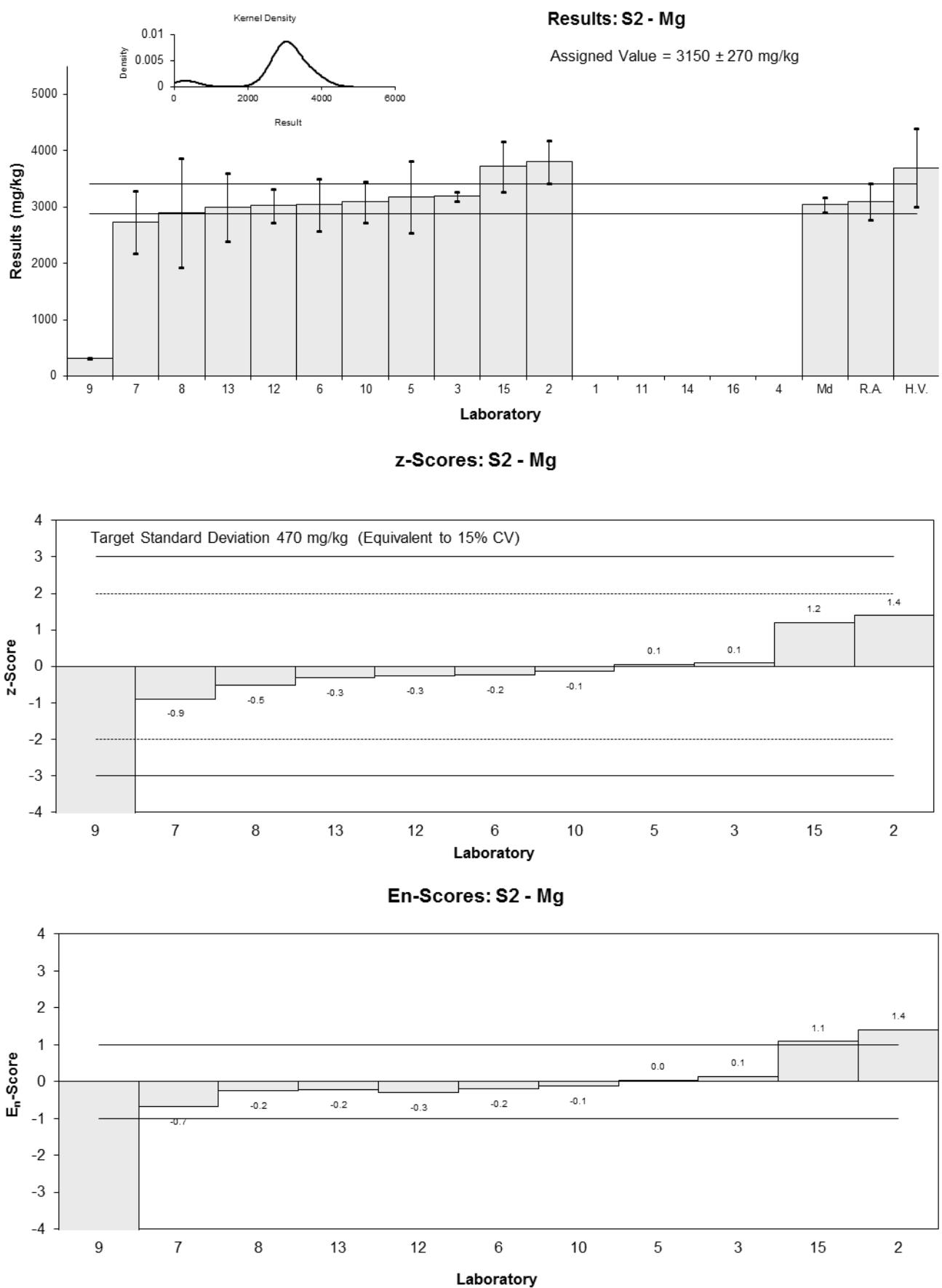


Figure 42

Table 49

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	Mn
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	96.2	9.62	0.87	0.70
3	88	11	-0.06	-0.04
4	NT	NT		
5	82.82	16.56	-0.64	-0.33
6	98.5	14.8	1.13	0.64
7	87	17	-0.17	-0.08
8	80	30	-0.96	-0.28
9	87.0	2.6	-0.17	-0.25
10	84	11	-0.51	-0.37
11	NT	NT		
12	92.5	9.25	0.45	0.38
13	82.925	16.59	-0.63	-0.32
14	NT	NT		
15	95.0	10	0.73	0.57
16	NT	NT		

Statistics

Assigned Value	88.5	5.3
Spike	Not Spiked	
Homogeneity Value	81	16
Robust Average	88.5	5.3
Median	87.0	4.2
Mean	88.5	
N	11	
Max.	98.5	
Min.	80	
Robust SD	7	
Robust CV	7.9%	

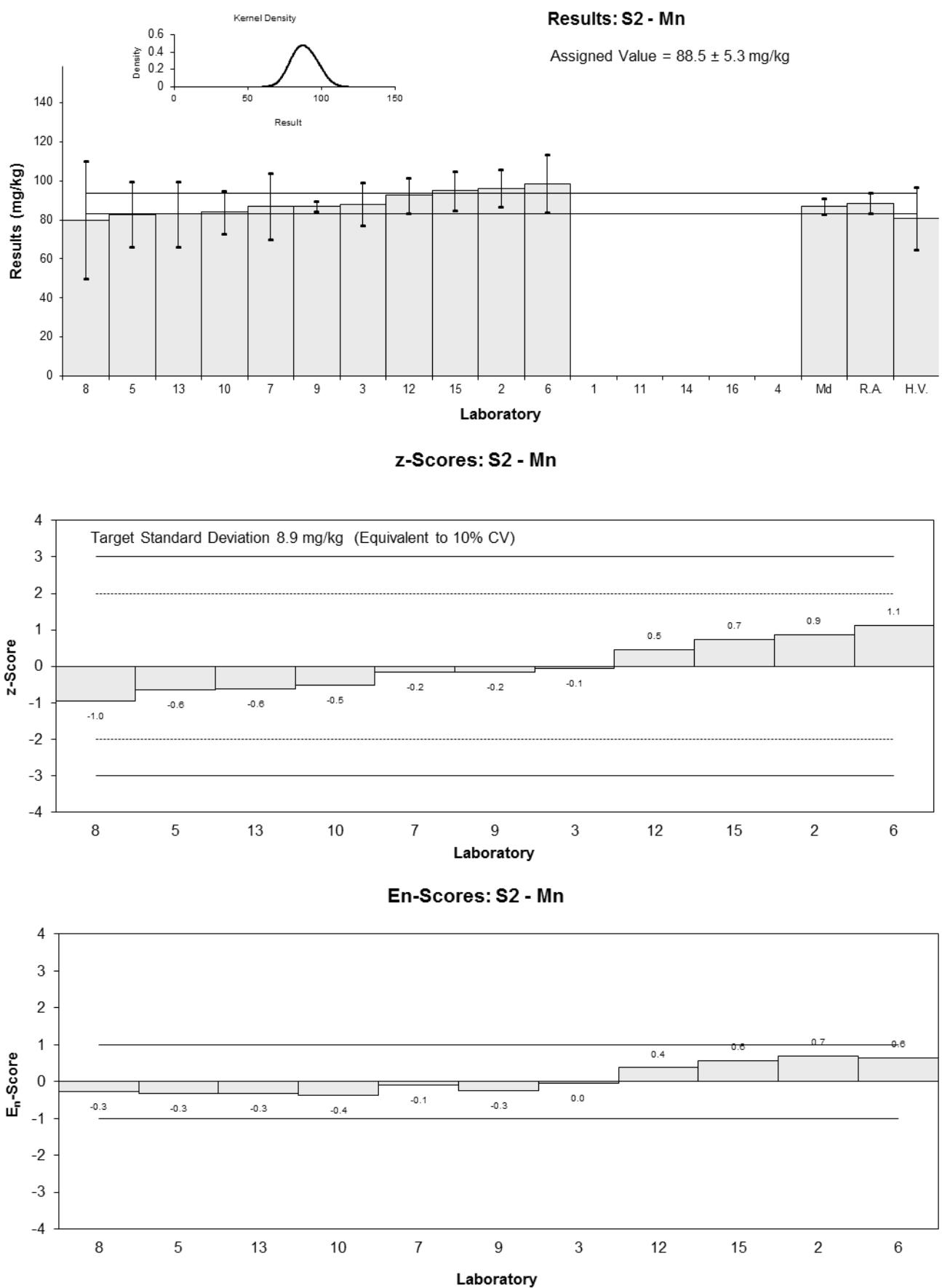


Figure 43

Table 50

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	NR	NR		
3	0.65	0.04	0.87	1.20
4	NT	NT		
5	0.54	0.11	-0.41	-0.29
6	< 10	NR		
7	0.59	0.12	0.17	0.12
8	0.52	0.3	-0.64	-0.18
9	NR	NR		
10	0.97	0.1	4.58	3.56
11	NT	NT		
12	0.61	0.061	0.41	0.45
13	0.554	0.11	-0.24	-0.17
14	NT	NT		
15	0.56	0.1	-0.17	-0.14
16	NT	NT		

Statistics

Assigned Value*	0.575	0.048
Spike	Not Spiked	
Homogeneity Value	0.62	0.12
Robust Average	0.589	0.057
Median	0.575	0.043
Mean	0.624	
N	8	
Max.	0.97	
Min.	0.52	
Robust SD	0.065	
Robust CV	11%	

*Robust Average excluding Laboratory 10

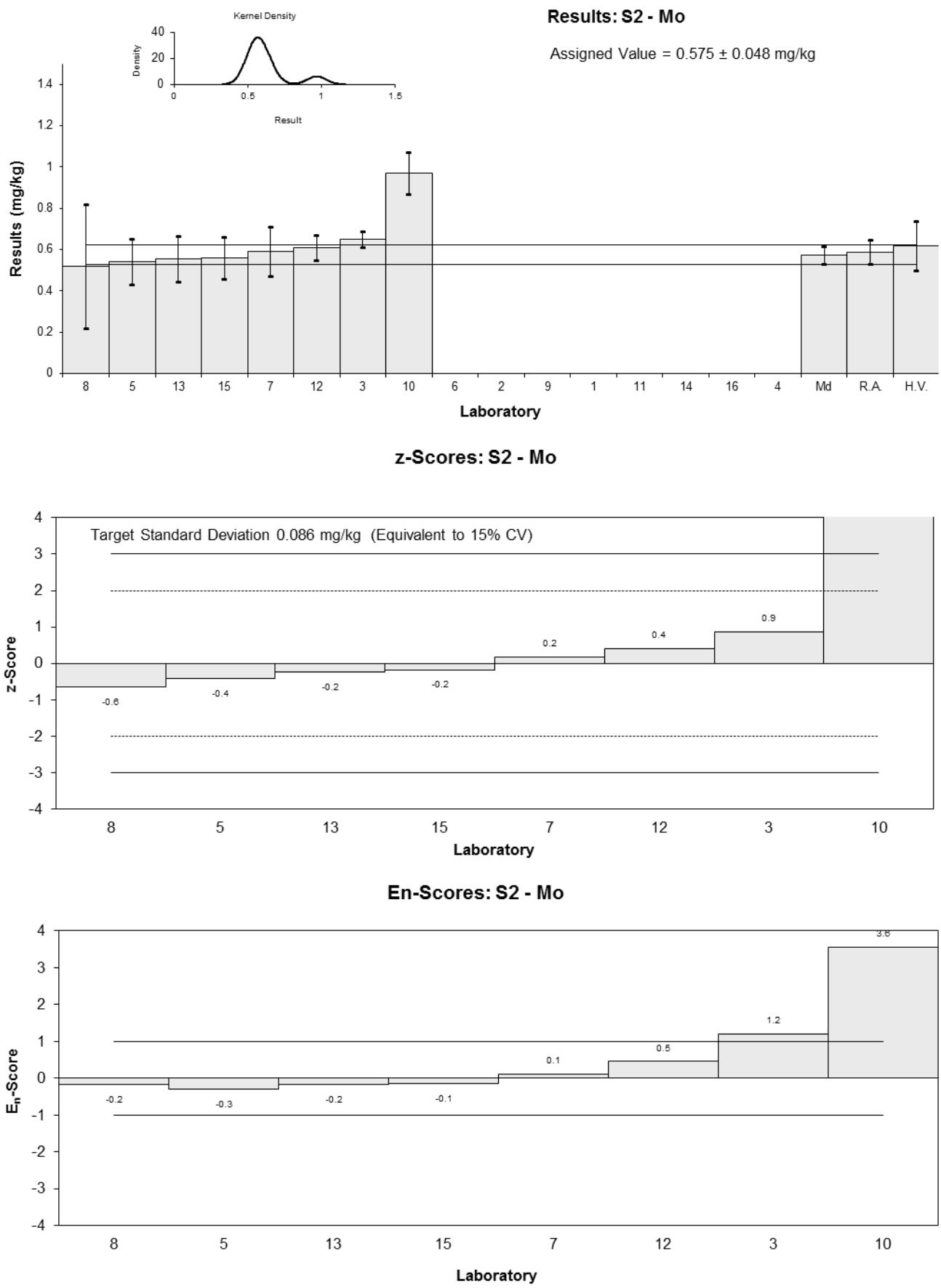


Figure 44

Table 51

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	Na
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	10.5	1.05
3	<50	5
4	NT	NT
5	20.80	4.16
6	< 50	NR
7	16	3.2
8	12	4
9	NR	NR
10	<20	20
11	NT	NT
12	NT	NT
13	16.00	3.20
14	NT	NT
15	<50	NR
16	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	13.5	2.7
Robust Average	15.1	5.1
Median	16.0	7.4
Mean	15.1	
N	5	
Max.	20.8	
Min.	10.5	
Robust SD	4.6	
Robust CV	30%	

Results: S2 - Na

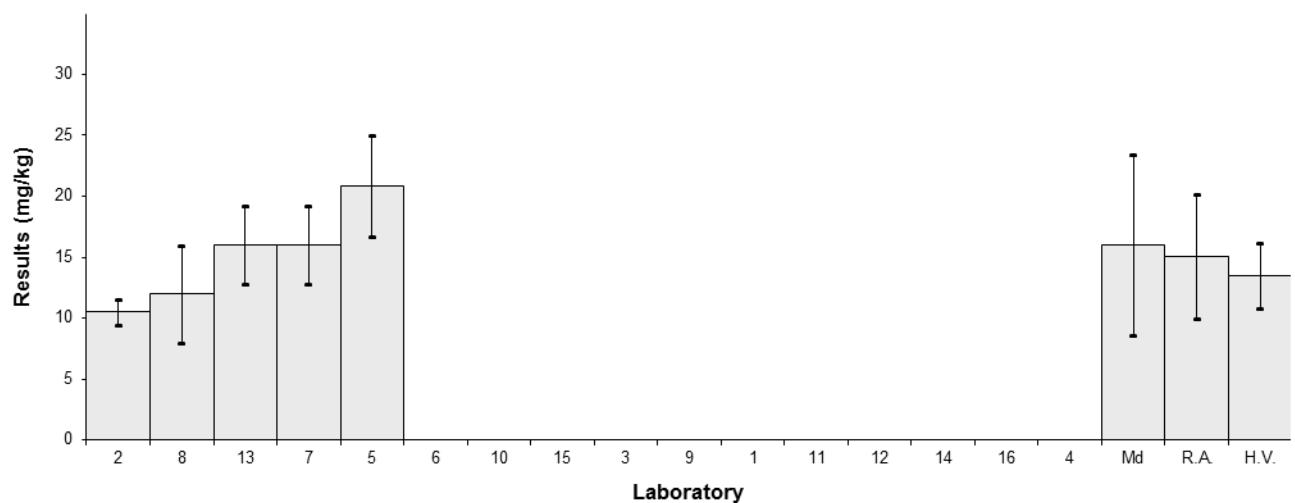


Figure 45

Table 52

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	0.724	0.072	0.19	0.34
3	0.68	0.10	-0.12	-0.16
4	NT	NT		
5	0.70	0.14	0.02	0.02
6	< 5	NR		
7	0.69	0.14	-0.05	-0.05
8	0.62	0.3	-0.55	-0.25
9	NR	NR		
10	0.76	0.2	0.45	0.31
11	NT	NT		
12	0.625	0.063	-0.52	-1
13	0.686	0.14	-0.08	-0.08
14	NT	NT		
15	0.61	0.2	-0.62	-0.43
16	NT	NT		

Statistics

Assigned Value*	0.697	0.035
Spike	Not Spiked	
Reference Value*	0.697	0.035
Robust Average	0.677	0.048
Median	0.686	0.043
Mean	0.677	
N	9	
Max.	0.76	
Min.	0.61	
Robust SD	0.057	
Robust CV	8.4%	

*Reference Value by IDMS

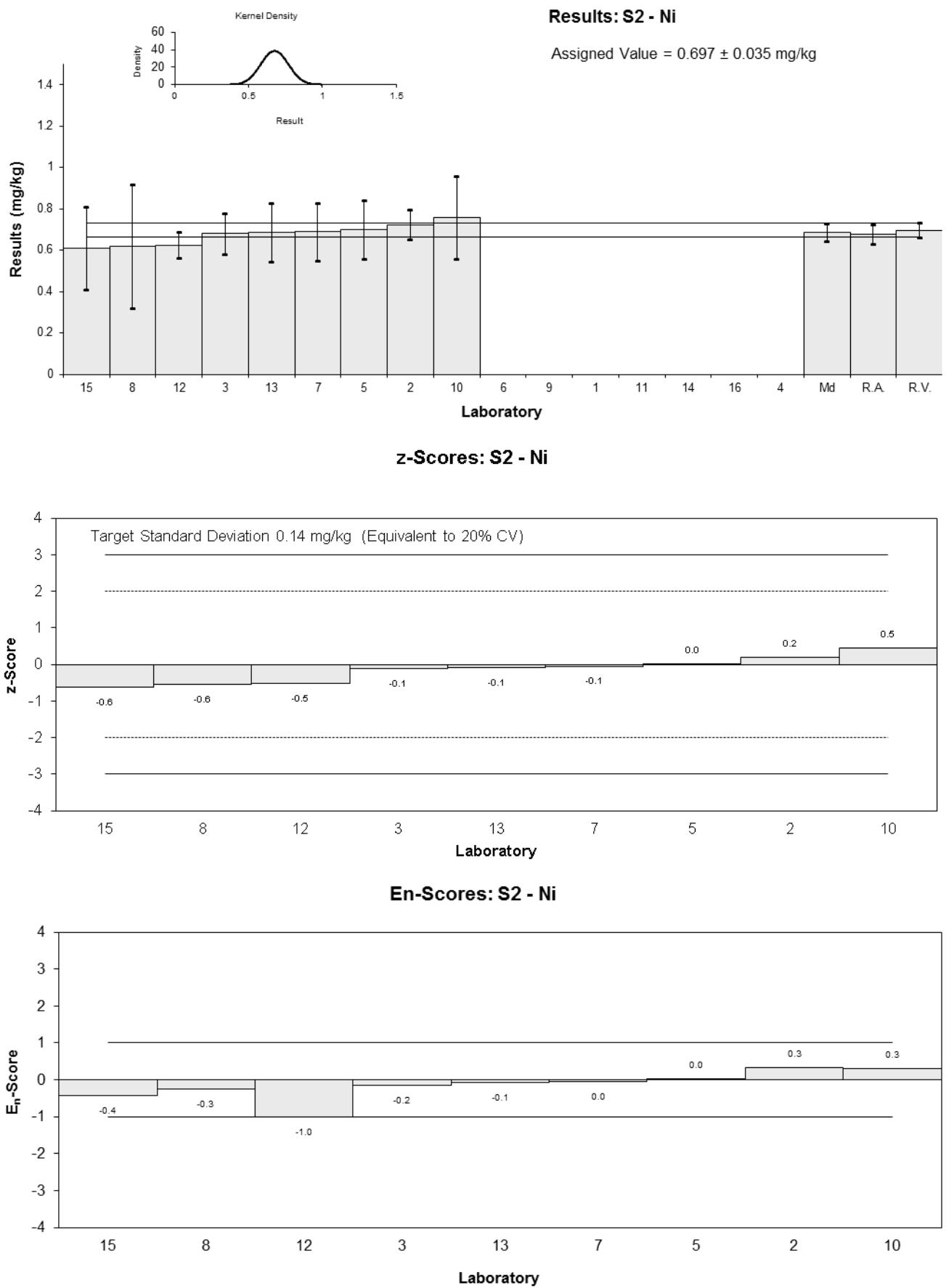


Figure 46

Table 53

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	P
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	NR	NR		
3	6750	560	1.23	1.17
4	NT	NT		
5	5631	1126.20	-0.63	-0.33
6	6030	900	0.03	0.02
7	5780	1160	-0.38	-0.19
8	5600	1900	-0.68	-0.21
9	NT	NT		
10	6035	416	0.04	0.05
11	NT	NT		
12	6277	628	0.44	0.39
13	5979.85	1195.97	-0.05	-0.02
14	NT	NT		
15	6230	750	0.37	0.27
16	NT	NT		

Statistics

Assigned Value	6010	290
Spike	Not Spiked	
Homogeneity Value	5560	670
Robust Average	6010	290
Median	6030	280
Mean	6035	
N	9	
Max.	6750	
Min.	5600	
Robust SD	350	
Robust CV	5.8%	

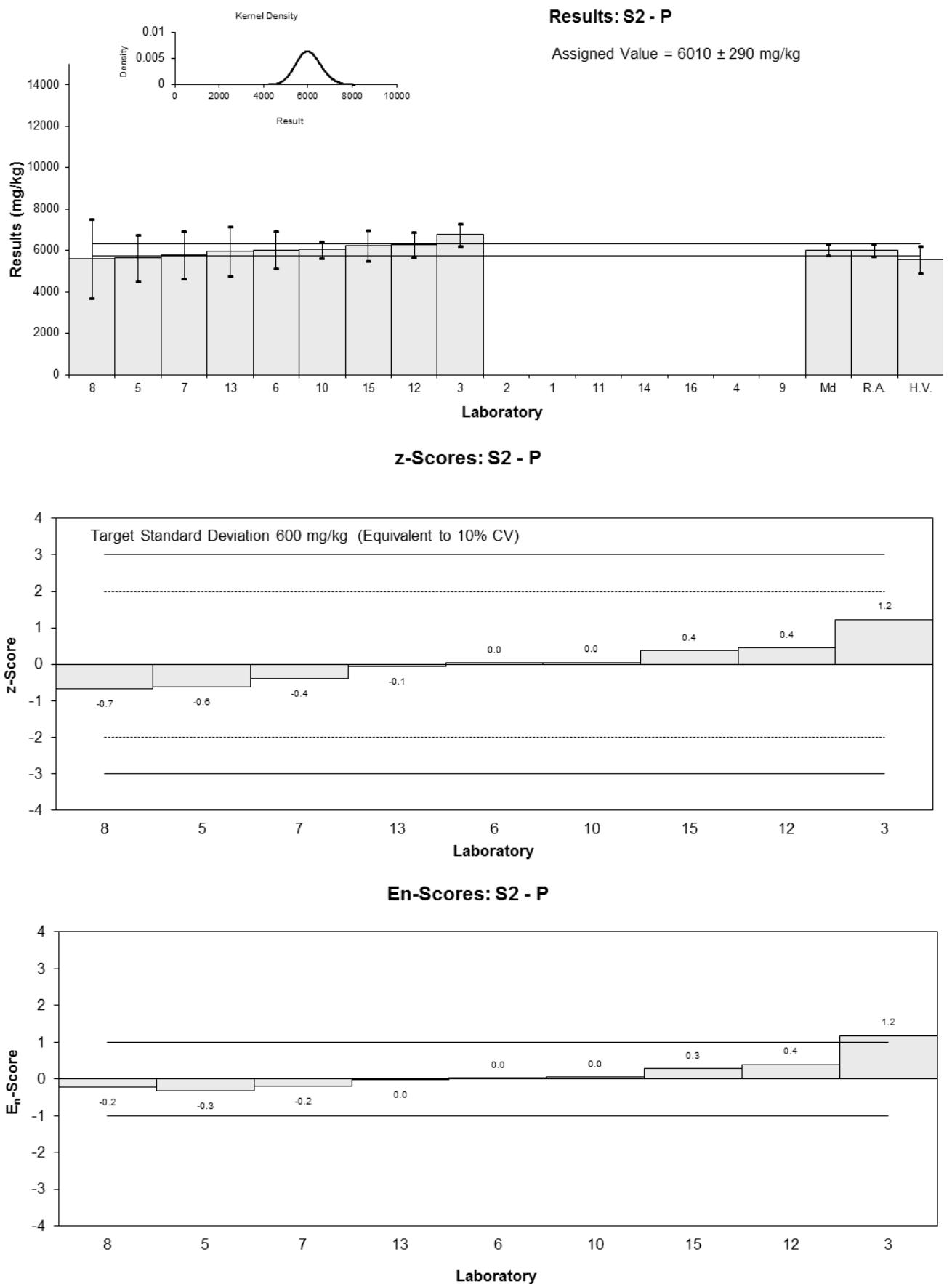


Figure 47

Table 54

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	0.015	0.002	-0.67	-0.91
3	0.018	0.010	0	0
4	NT	NT		
5	0.02	0.00	0.44	0.77
6	< 5	NR		
7	0.011	0.002	-1.56	-2.13
8	<0.1	NR		
9	0.015	0.006	-0.67	-0.46
10	0.02	0.01	0.44	0.19
11	NT	NT		
12	0.016	0.002	-0.44	-0.61
13	0.024	0.00	1.33	2.31
14	NT	NT		
15	<0.1	NR		
16	NT	NT		

Statistics

Assigned Value*	0.0180	0.0026
Spike	Not Spiked	
Reference Value*	0.0180	0.0026
Robust Average	0.0174	0.0040
Median	0.0170	0.0031
Mean	0.0174	
N	8	
Max.	0.024	
Min.	0.011	
Robust SD	0.0045	
Robust CV	26%	

*Reference Value by SA-ICP-MS

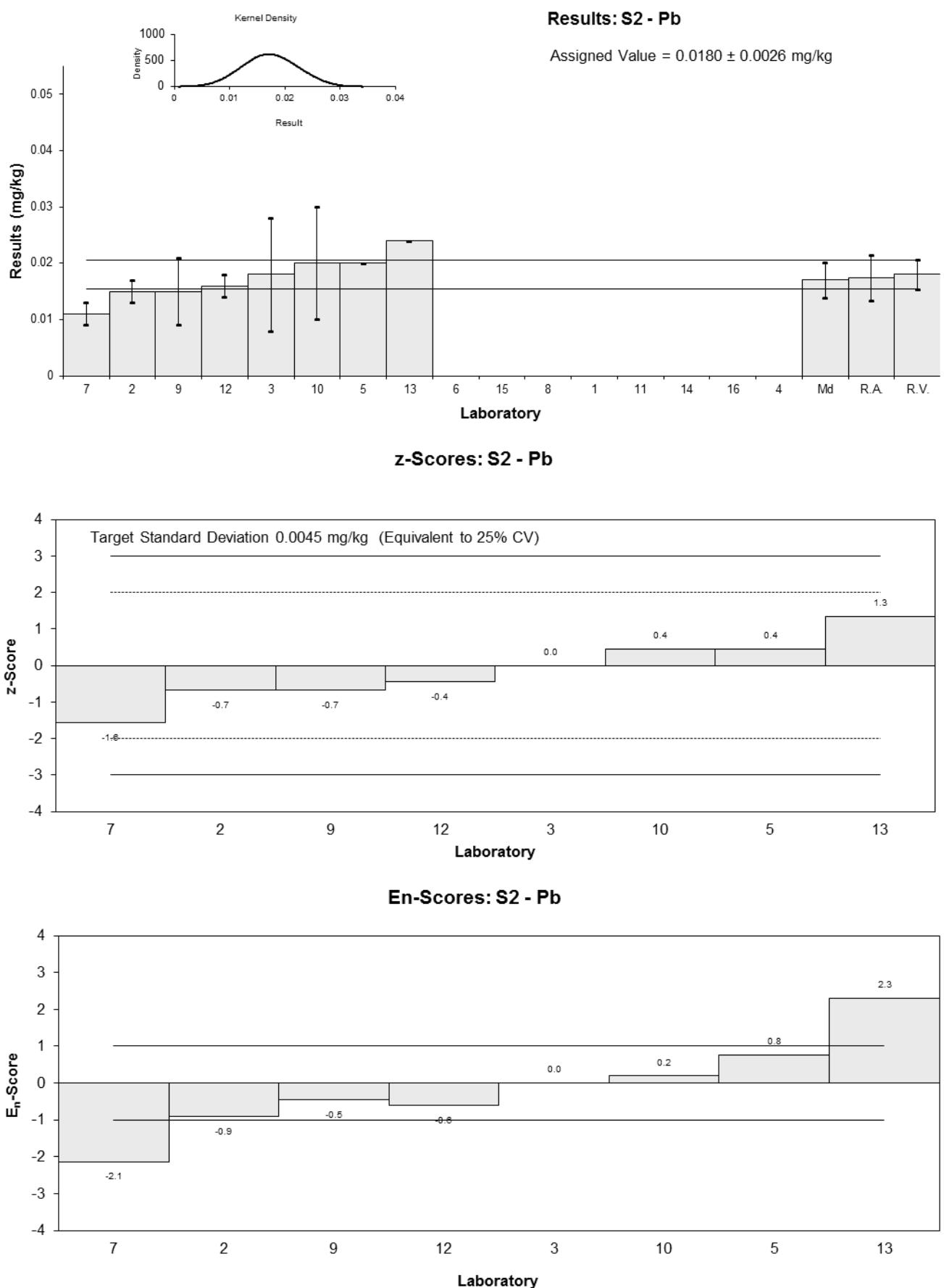


Figure 48

Table 55

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	S
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	NR	NR		
3	2370	150	1.07	1.08
4	NT	NT		
5	2114	422.80	-0.12	-0.06
6	2010	300	-0.61	-0.39
7	1940	388	-0.93	-0.48
8	1900	630	-1.12	-0.37
9	2034	63	-0.50	-0.65
10	2339	159	0.93	0.91
11	NT	NT		
12	2284	228	0.67	0.53
13	2107.255	421.45	-0.15	-0.07
14	NT	NT		
15	2260	280	0.56	0.38
16	NT	NT		

Statistics

Assigned Value	2140	150
Spike	Not Spiked	
Robust Average	2140	150
Median	2110	170
Mean	2136	
N	10	
Max.	2370	
Min.	1900	
Robust SD	190	
Robust CV	8.9%	

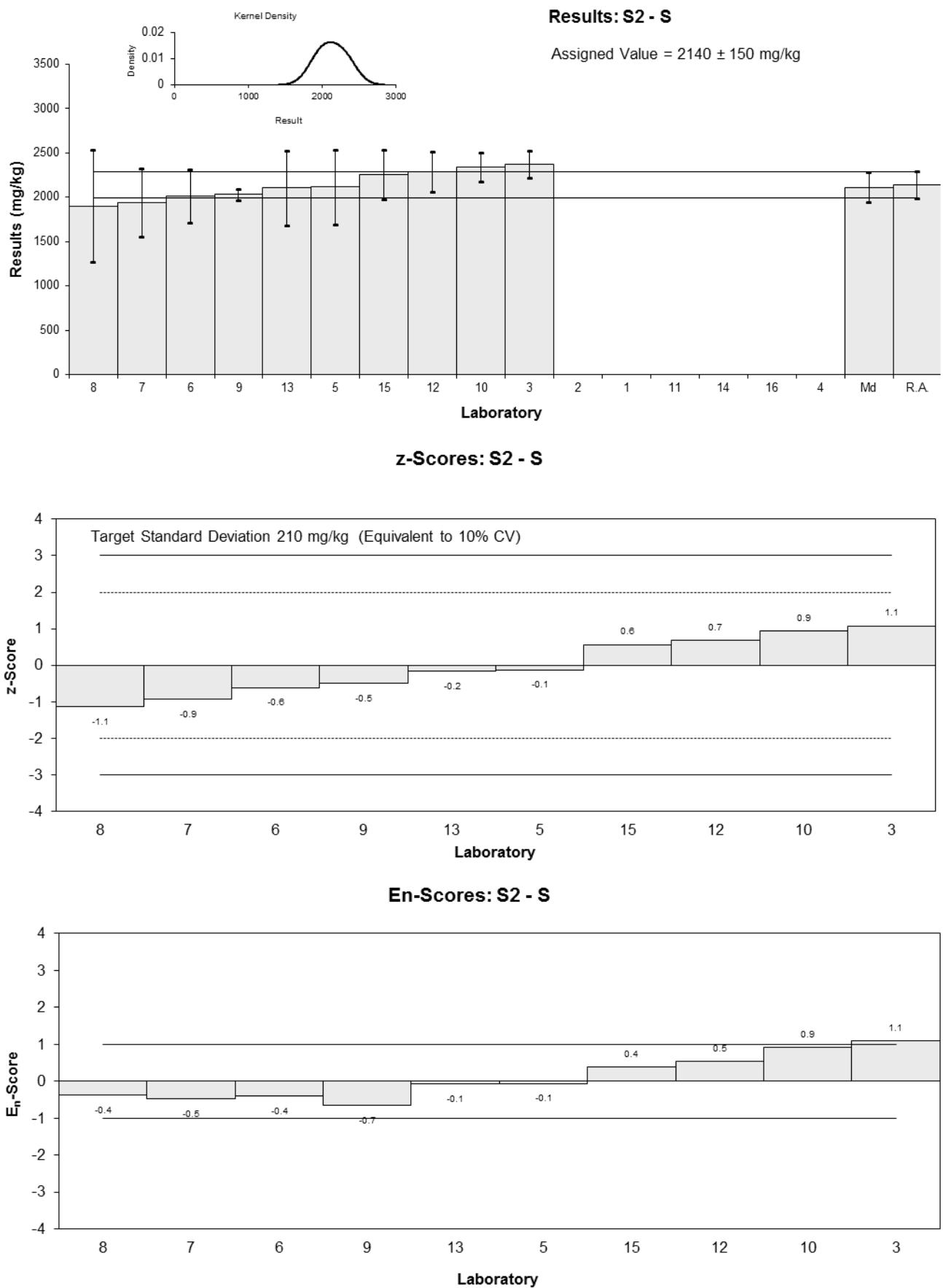


Figure 49

Table 56

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	Sb
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	0.005	0.0005
3	<0.01	0.005
4	NT	NT
5	<0.01	0.00
6	<10	NR
7	<0.01	NR
8	<0.1	NR
9	NR	NR
10	<0.02	0.02
11	NT	NT
12	0.003	0.0005
13	NT	NT
14	NT	NT
15	<1	NR
16	NT	NT

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.0033	0.0007
Mean	0.0040	
N	2	

*Insufficient data to calculate statistics

Results: S2 - Sb

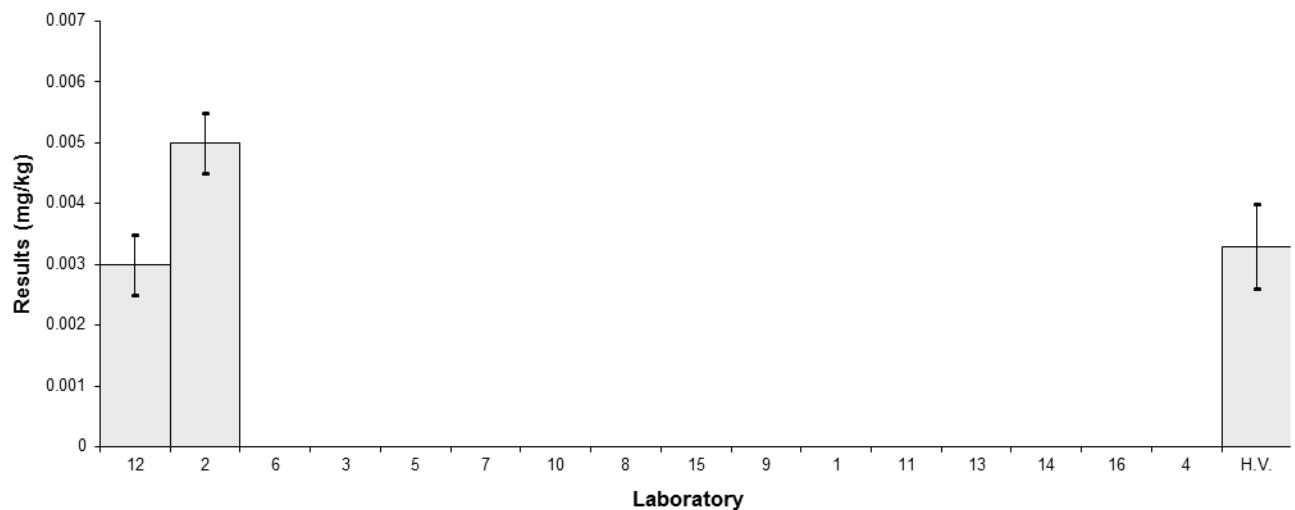


Figure 50

Table 57

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	0.060	0.006	0.45	0.40
3	0.047	0.011	-0.73	-0.51
4	NT	NT		
5	0.05	0.01	-0.45	-0.34
6	< 5	NR		
7	<0.05	NR		
8	0.06	0.04	0.45	0.12
9	0.07	0.03	1.36	0.47
10	0.04	0.02	-1.36	-0.66
11	NT	NT		
12	0.059	0.006	0.36	0.32
13	NT	NT		
14	NT	NT		
15	<0.5	NR		
16	NT	NT		

Statistics

Assigned Value	0.055	0.011
Spike	Not Spiked	
Homogeneity Value	0.041	0.008
Robust Average	0.055	0.011
Median	0.059	0.012
Mean	0.055	
N	7	
Max.	0.07	
Min.	0.04	
Robust SD	0.011	
Robust CV	20%	

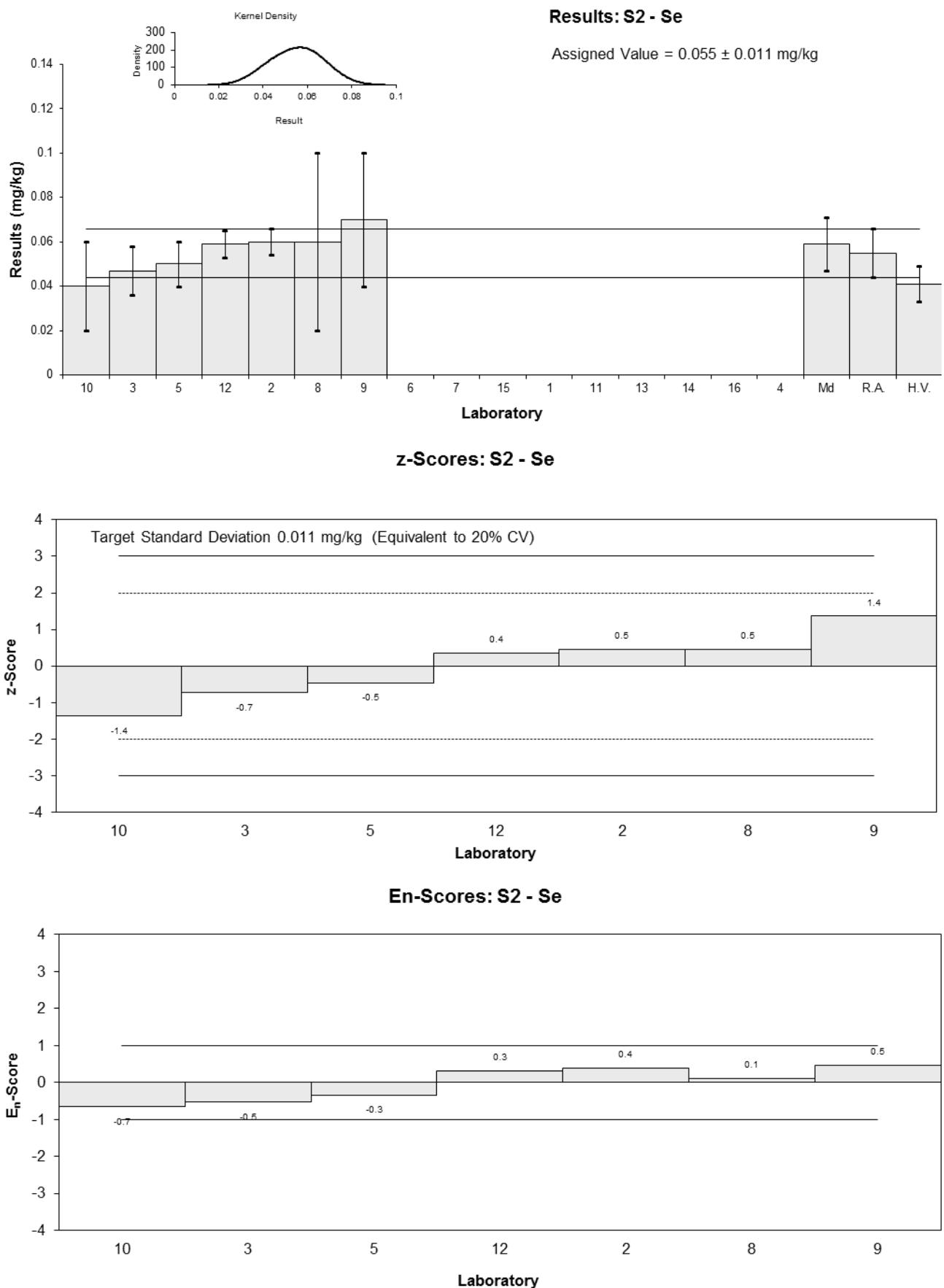


Figure 51

Table 58

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	Sr
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	NR	NR		
3	NT	NT		
4	NT	NT		
5	23.46	4.69	-0.62	-0.31
6	< 50	NR		
7	28	5.6	1.20	0.52
8	24	8	-0.40	-0.12
9	26.7	0.8	0.68	1.05
10	25	3	0.00	0.00
11	NT	NT		
12	24.5	2.45	-0.20	-0.18
13	25.204	5.04	0.08	0.04
14	NT	NT		
15	23.6	2.8	-0.56	-0.45
16	NT	NT		

Statistics

Assigned Value	25.0	1.4
Spike	Not Spiked	
Homogeneity Value	25.6	5.1
Robust Average	25.0	1.4
Median	24.8	1.2
Mean	25.1	
N	8	
Max.	28	
Min.	23.46	
Robust SD	1.6	
Robust CV	6.4%	

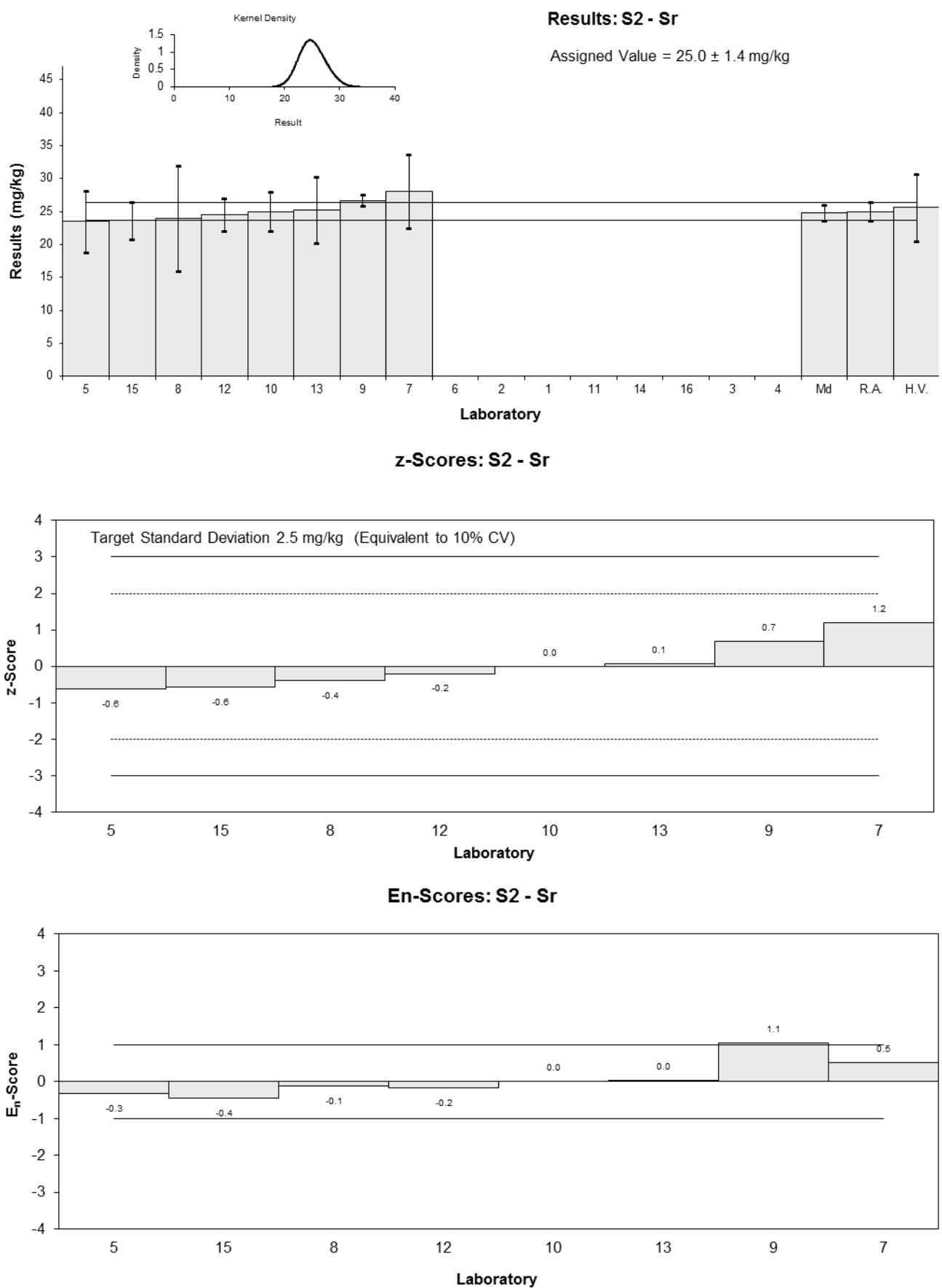


Figure 52

Table 59

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	TKN
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	NR	NR
3	35800	800
4	NT	NT
5	3.52	0.70
6	NT	NT
7	36000	5400
8	35000	12000
9	NR	NR
10	31000	4600
11	NT	NT
12	NR	NR
13	NT	NT
14	NT	NT
15	36200	3000
16	NT	NT

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Robust Average	35100	1900
Median	35800	740
Mean	34800	
N	5	
Max.	36200	
Min.	3.52	
Robust SD	1700	
Robust CV	4.8%	

*The result reported by Laboratory 5 was omitted from statistical calculation (gross error)

Results: S2 - TKN

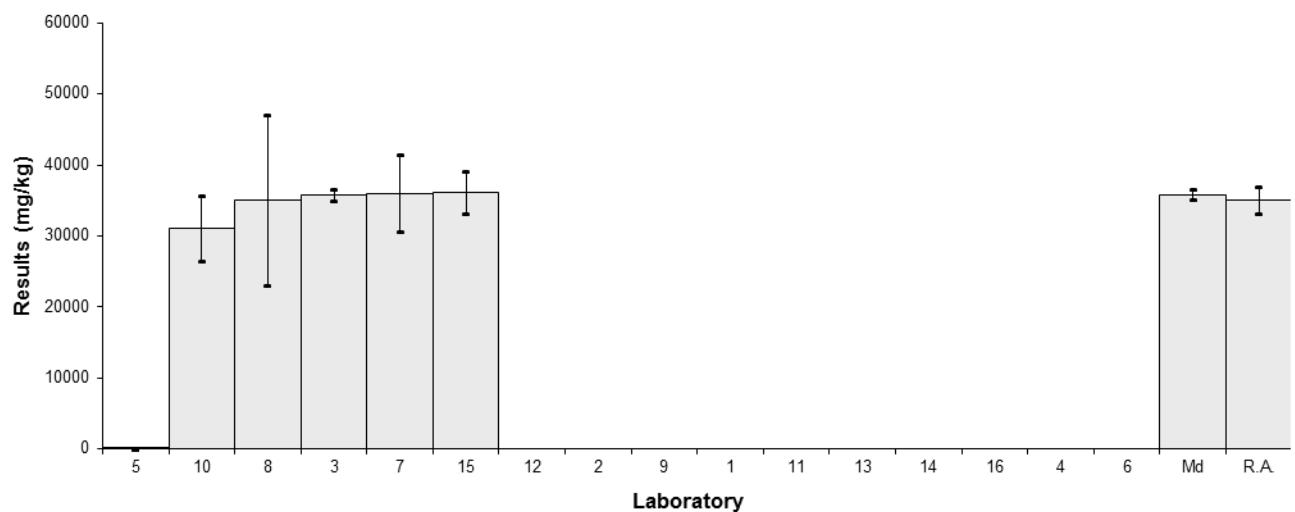


Figure 53

Table 60

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	TOC
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	NR	NR
3	NT	NT
4	NT	NT
5	48.76	9.75
6	NT	NT
7	420000	63000
8	550000	180000
9	NR	NR
10	434000	15184
11	NT	NT
12	NR	NR
13	NT	NT
14	NT	NT
15	476000	40000
16	NT	NT

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Robust Average	470000	83000
Median	455000	66000
Mean	470000	
N	4	
Max.	550000	
Min.	48.76	
Robust SD	66000	
Robust CV	14%	

*The result reported by Laboratory 5 was omitted from statistical calculation (gross error)

Results: S2 - TOC

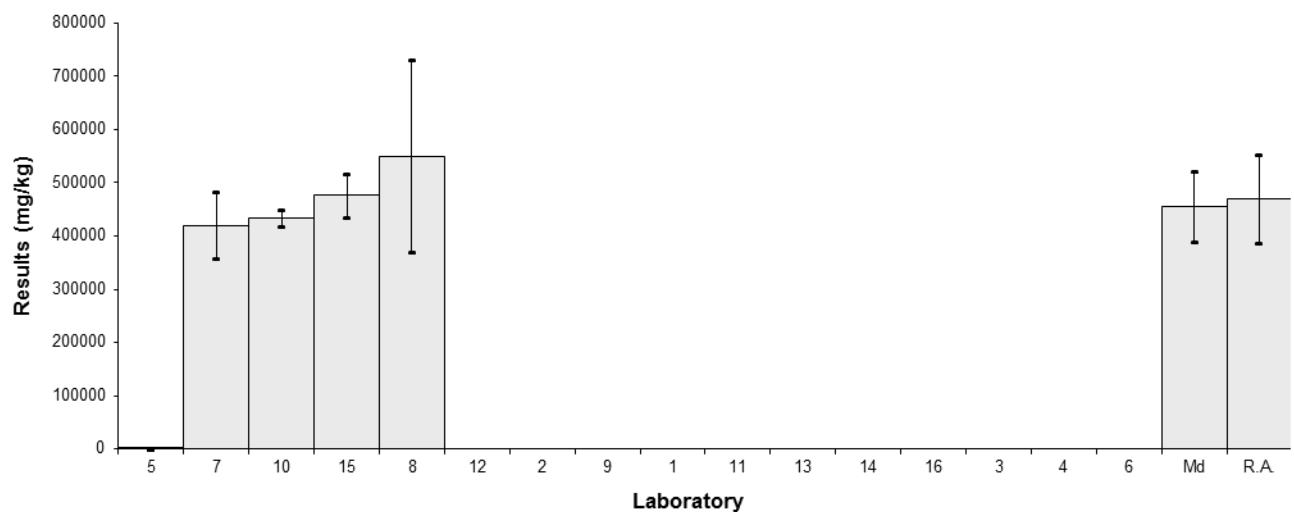


Figure 54

Table 61

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	Total Ash
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	NR	NR
3	5.0	0.2
4	NT	NT
5	5.02	1.00
6	NT	NT
7	5.1	0.5
8	51000	17000
9	NR	NR
10	50500	6064
11	NT	NT
12	NR	NR
13	NT	NT
14	NT	NT
15	52800	5000
16	NT	NT

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Robust Average	51400	2000
Median	51000	1800
Mean	51400	
N	3	
Max.	52800	
Min.	5	
Robust SD	1400	
Robust CV	2.7%	

*The results reported by Laboratories 3, 5 and 7 were omitted from statistical calculation (gross error)

Results: S2 - Total Ash

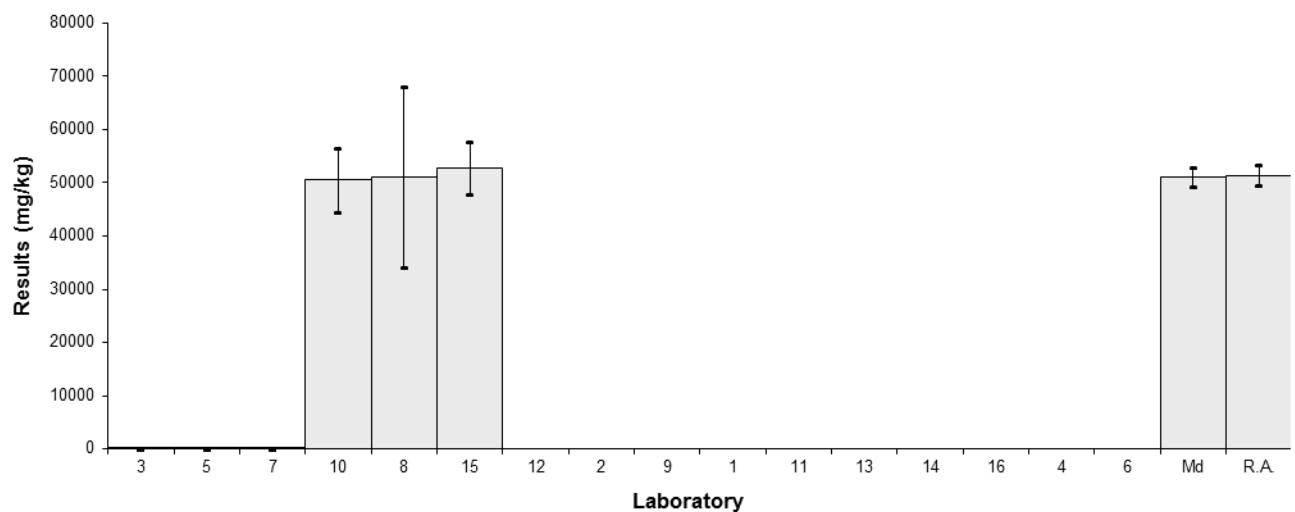


Figure 55

Table 62

Sample Details

Sample No.	S2
Matrix.	Hemp
Analyte.	V
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	NR	NR
3	NT	NT
4	NT	NT
5	0.09	0.02
6	< 5	NR
7	<0.1	NR
8	<0.5	NR
9	NT	NT
10	<0.2	0.2
11	NT	NT
12	0.017	0.002
13	NT	NT
14	NT	NT
15	<1	NR
16	NT	NT

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Information Value**	0.0212	0.0017
Mean	0.0535	
N	2	

*Insufficient data to calculate statistics;

**Information Value by SA-ICP-MS

Results: S2 - V

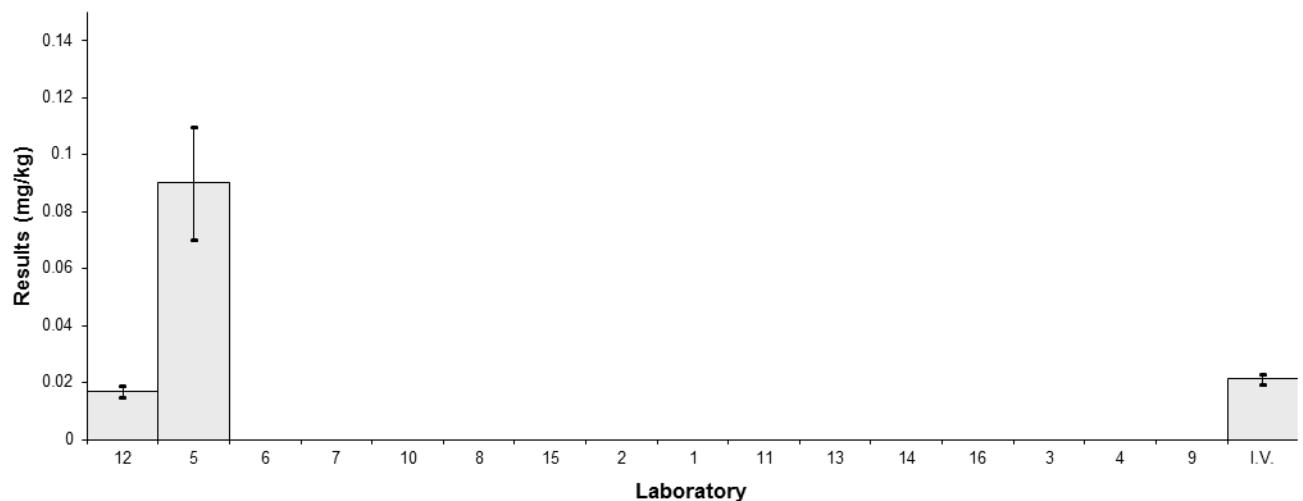


Figure 56

Table 63

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	NT	NT		
2	47.5	4.75	0.94	0.81
3	45	5	0.37	0.30
4	NT	NT		
5	41.32	8.26	-0.48	-0.25
6	45.1	6.8	0.39	0.24
7	40	8	-0.78	-0.41
8	43	15	-0.09	-0.03
9	41.5	1.8	-0.44	-0.75
10	43	6	-0.09	-0.06
11	NT	NT		
12	45	4.5	0.37	0.33
13	42.449	8.49	-0.22	-0.11
14	NT	NT		
15	44.5	7.0	0.25	0.15
16	NT	NT		

Statistics

Assigned Value	43.4	1.8
Spike	Not Spiked	
Homogeneity Value	55	11
Robust Average	43.4	1.8
Median	43.0	1.7
Mean	43.5	
N	11	
Max.	47.5	
Min.	40	
Robust SD	2.3	
Robust CV	5.3%	

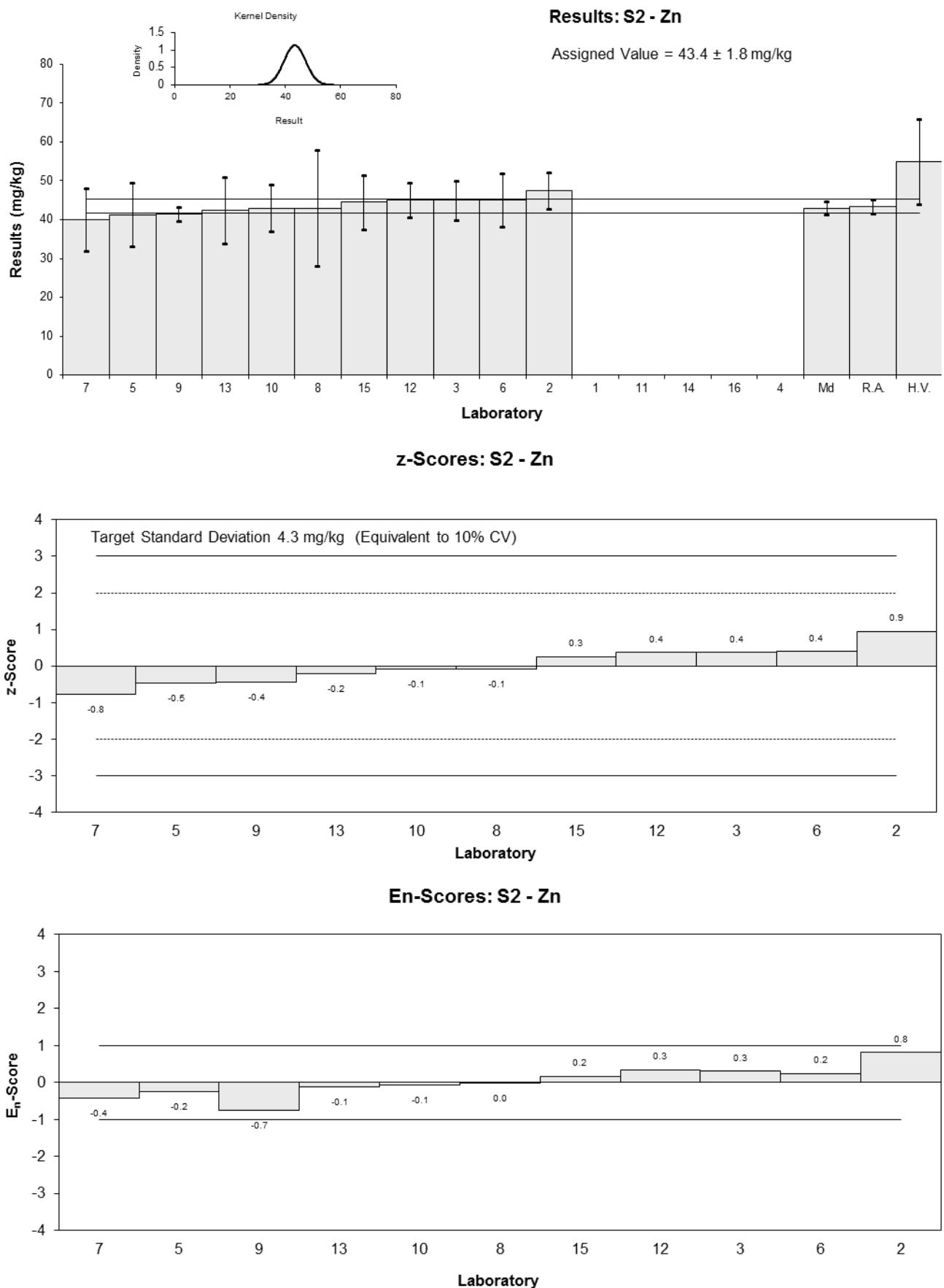


Figure 57

7 DISCUSSION OF RESULTS

7.1 Assigned Value

Sample S1 – was freeze dried fish. Assigned values were the robust average of participants' results. The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO13528:2015(E), "Statistical methods for use in proficiency testing by interlaboratory comparisons". Results less than 50% and more than 150% of the robust average were removed before calculation of the assigned value.⁵ Appendix 2 sets out the calculation for the robust average of As in Sample S1 and its associated uncertainty.

No assigned values were calculated for B and Rb in this sample because the reported results were too few. Descriptive statistics together with homogeneity values for these elements are presented in Chapter 6.

Sample S2 - was hemp. Assigned values for As, Cd, and Pb in S2 were reference values from measurements made using standard addition ICP-MS and for Cr and Ni were reference values measured using IDMS (Appendix 3).

Assigned values for: Ba, Ca, Co, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, Pb, S, Se, Sr and Zn were the robust average of participants' results calculated as for Sample S1.

Indicative values from SA-ICP-MS measurements were also provided for Al and V in this sample.

No assigned values were calculated for Ag, Al, B, Cs, Na, Sb TKN, TOC, total ash and V in S2 because the reported results were too few or too variable. However, participants may still compare their reported results for these elements with the robust average of participants' results and/or the homogeneity value or information value. Descriptive statistics for these elements are presented in Section 6. No descriptive statistics were presented for Ag and Cs in S2 because no results were reported for Ag in this sample and only one (0.04 mg/kg) has been reported for Cs.

Traceability of the reference values for As, Cd, Cr, Ni and Pb in S2 rely on gravimetric sample preparation and elemental quantification by ICP-MS. Gravimetric measurements were calibrated using Australian standards for mass and are traceable to the SI unit for mass (kg). ICP-MS measurements calibrated with standard addition and isotope dilution are traceable to the SI units for mass (kg) through the primary calibration standard certified by NIST (USA) and the SI unit for amount of substance (mol) through data for isotopic composition and relative atomic mass. Isotopic compositions are traceable to IUPAC published data with the exception of Pb which is traceable to the certified isotopic composition of an isotopically certified material from NIST (USA).

The consensus of participants' results (robust average) is not traceable to any external reference. So although expressed in SI units, the metrological traceability of these assigned values has not been established.

7.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded measurement uncertainty associated with their results. Of 515 numerical results, 506 (98%) were reported with an expanded measurement uncertainty, indicating that the majority of laboratories have addressed this requirement of ISO/IEC 17025.⁸ The magnitude of these expanded uncertainties was within the range 1.5% to 146% of the reported value. The participants used a wide variety of procedures to estimate the expanded measurement uncertainty. These are presented in Table 6.

Approaches to estimating measurement uncertainty include: standard deviation of replicate analysis, Horwitz formula, 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 comparisons studies.^{1, 9-15}

Proficiency tests allow a check of the reasonableness of uncertainty estimates. Results and the expanded MU are presented in the bar charts for each analyte (Figure 2 to 57). In this study, the reported expanded measurement uncertainty may have been over-estimated in some cases (e.g. Lab 11 for Ni in S1) or under-estimated (e.g. Lab 12 for Ni in S1). As a simple rule of thumb, when the uncertainty estimate is either smaller than the uncertainty of the assigned value or larger than the uncertainty of the assigned value plus twice the target standard deviation then this should be reviewed as suspect.

Double counting the precision uncertainty components and overestimation of the laboratory or method bias are the most common errors seen in the laboratories' estimated uncertainty budgets. According to General Accreditation Guidance, Estimating and reporting measurement uncertainty of chemical test results¹¹ and to NORDTEST TR 537⁹ the most common experimental data used for estimating the precision component for the measurement uncertainty calculation in the top down approach are from:

- Stable control samples that cover the whole analytical process (including extraction) and **have a matrix similar** to the samples; **or**
- Stable control samples **and** duplicate analyses if control samples do not cover whole analytical process (e.g. the control sample is a synthetic sample- we have to take into consideration uncertainties arising from different matrices); **or**
- When control samples are not stable, from analysis of natural duplicates (gives within-day variation for sampling and measurement) and long-term uncertainty component from the variation in the instrument calibration; **or**
- Replicate analyses performed on the same sample at different times to obtain estimates of intermediate precision; within-batch replication provides estimates of repeatability only.

The most common sources for estimating the method bias component for the measurement uncertainty calculation are from:

- Certified reference material recoveries; **or**
- Participation in PT studies (laboratory bias from at least 6 successful PT studies); **or**
- From sample spike recoveries.

Some laboratories attached an estimate of the expanded measurement uncertainty for results reported as less than their limit of detection. An estimate of uncertainty expressed as a value cannot be attached to a result expressed as a range.¹

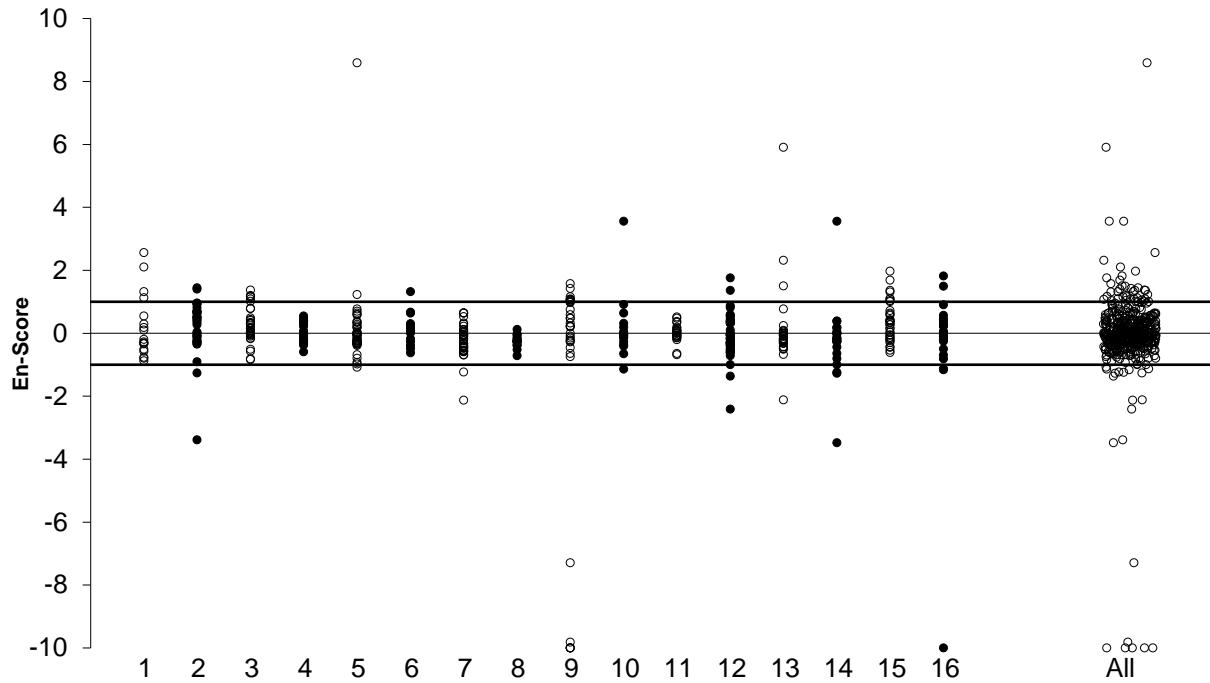
In some cases the results were reported with an inappropriate number of significant figures. The recommended format is to write uncertainty to no more than two significant figures and then to write the result with the corresponding number of decimal places. For example, instead of 0.626 ± 0.21 mg/kg, it is better to report 0.63 ± 0.21 mg/kg or instead of 155 ± 15.5 mg/kg, it is better to report 155 ± 16 mg/kg.¹

7.3 E_n-score

E_n-score should be interpreted only in conjunction with z-scores. The E_n-score indicates how closely a result agrees with the assigned value taking into account the respective uncertainties. An unsatisfactory E_n score for an analyte can either be caused by an inappropriate measurement, an inappropriate estimation of measurement uncertainty, or both.

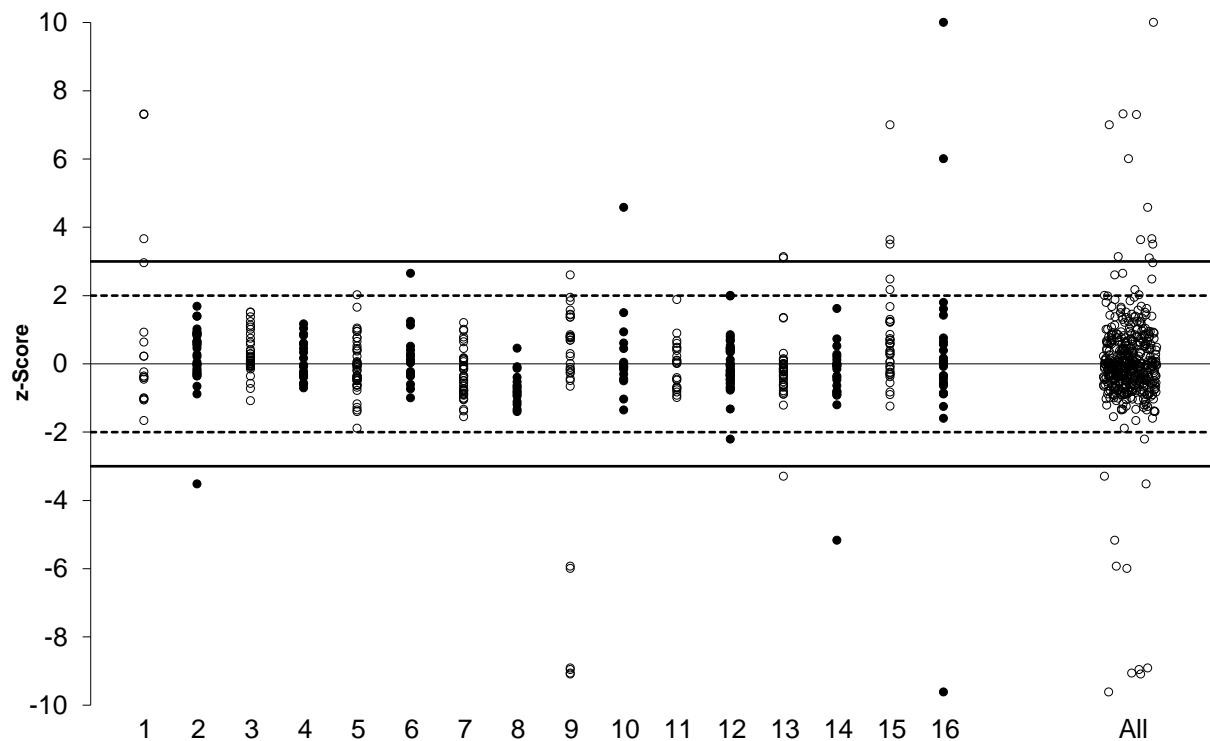
The dispersal of participants' E_n -scores is graphically presented in Figure 58. Where a laboratory did not report an expanded uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the E_n -score.

Of 464 results for which E_n -scores were calculated, 404 (87%) returned a satisfactory score of $|E_n| \leq 1.0$ indicating agreement of the participants' results with the assigned values within their respective expanded measurement uncertainties.



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

Figure 58 E_n -Score Dispersal by Laboratory



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

Figure 59 z-Score Dispersal by Laboratory

7.4 z-Score

The z-score compares participants' 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 10%, 15%, 20% and 25% PCV were used to calculate z-scores. Unlike the standard deviation based on between laboratories CV, setting the target standard deviation as a realistic set value enables z-scores to be used as a fixed reference value point for assessment of laboratory performance, independent of group performance.

The between laboratory coefficient of variation predicted by the Thompson equation⁷ and the between laboratory coefficient of variation resulted in this study are presented for comparison in Table 64. The dispersal of participants' z-scores is presented in Figure 59 (by laboratory code) and in Figure 60 (by test). Of 464 results for which z-scores were calculated, 435 (94%) returned a satisfactory score of $|z| \leq 2.0$ and 8 (2%) were questionable of $2.0 < |z| < 3.0$. Participants with multiple z-scores larger than 2 or smaller than -2 should check for laboratory bias.

Table 64 Between Laboratory CV of this study, Thompson CV and Set Target PCV

Sample	Test	Assigned value (mg/kg)	Between Laboratories CV*	Thompson/ Horwitz CV	Target SD (as CV)
S1	Ag	0.161	8.7%	21%	15%
S1	As	2.60	14%	14%	15%
S1	B	Not Set	21%	NA	Not Set
S1	Ba	3.62	4.4%	13%	15%
S1	Ca	1830	6.3%	5.2%	10%
S1	Cd	0.127	7.8%	22%	15%
S1	Co	0.627	7.8%	17%	10%
S1	Cr	1.47	18%	15%	20%
S1	Cu	1.41	7.8%	15%	10%
S1	Fe	52.7	9%	8.8%	10%
S1	Hg	0.415	8%	18%	10%
S1	K	18600	5.6%	3.6%	10%
S1	La	0.132	19%	22%	20%
S1	Li	0.230	14%	20%	20%
S1	Mg	1390	5.9%	5.4%	10%
S1	Mn	0.948	7.6%	16%	10%
S1	Mo	0.050	23%	22%	20%
S1	Na	3180	4.7%	4.8%	10%
S1	Ni	0.62	22%	17%	20%
S1	P	9470	8%	4%	10%
S1	Pb	0.186	13%	21%	15%
S1	Rb	Not Set	5.4%	NA	Not Set
S1	S	12000	13%	3.9%	10%
S1	Sb	0.100	8.6%	22%	10%
S1	Se	2.96	16%	14%	15%
S1	Sn	0.310	14%	19%	15%
S1	U	0.0615	14%	22%	15%
S1	V	0.364	27%	19%	20%
S1	Zn	34.8	5.5%	9.4%	10%
S2	Al	12.8**	51%	12%	Not Set
S2	As	0.01416	18%	22%	25%
S2	B	Not Set	26%	NA	Not Set
S2	Ba	1.96	6.6%	14%	10%

Sample	Test	Assigned value (mg/kg)	Between Laboratories CV*	Thompson/ Horwitz CV	Target SD (as CV)
S2	Ca	1080	11%	5.6%	10%
S2	Cd	0.00505	34%	22%	25%
S2	Co	0.0613	15%	22%	15%
S2	Cr	0.263	13%	20%	20%
S2	Cu	13.1	6.7%	11%	10%
S2	Fe	121	11%	7.8%	10%
S2	K	6720	16%	4.3%	15%
S2	Mg	3150	11%	4.8%	15%
S2	Mn	88.5	7.9%	8.2%	10%
S2	Mo	0.575	8.8%	17%	15%
S2	Na	Not Set	30%	NA	Not Set
S2	Ni	0.697	8.4%	17%	20%
S2	P	6010	5.8%	4.3%	10%
S2	Pb	0.0180	26%	22%	25%
S2	S	2140	8.9%	5%	10%
S2	Se	0.055	20%	22%	20%
S2	Sr	25.0	6.4%	9.9%	10%
S2	TKN	Not Set	4.8%	NA	Not Set
S2	TOC	Not Set	14%	NA	Not Set
S2	Total Ash	Not Set	2.7%	NA	Not Set
S2	Zn	43.4	5.3%	9.1%	10%

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

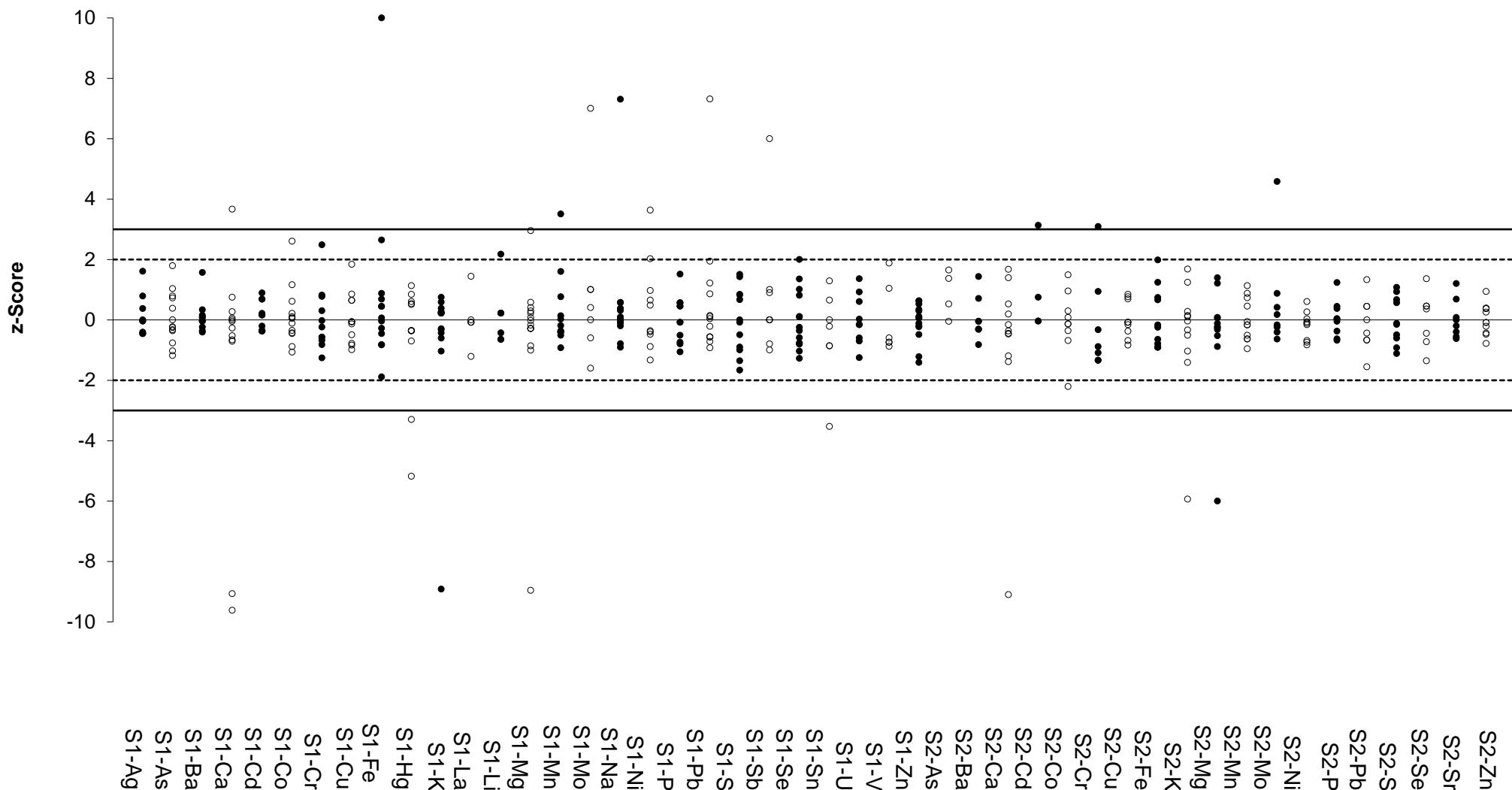
7.5 Participants' Results and Analytical Methods for Total Elements

A summary of participants' performance in the two study samples is presented in Figure 59 and Tables 65 and 66.

Reporting errors and problems with the dilution, standard preparation and/or calculation procedure were likely the main cause for the majority of the unsatisfactory results reported.

Measurements of total Al in S2 and of total B in S1 and S2 presented the most analytical difficulty. No assigned value could be set for these elements in the present and previous studies.

Measurements of low level Ag, Cd, Cs and Na in S2 also challenged participants' analytical methods. No result was reported for Ag and only one was reported for Cs; the between laboratory coefficient of variation for Cd and Na were high, at 34% and 30% respectively.



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

Figure 60 z-Score Dispersal by Analyte

Table 65 Summary of Participants' Results and Performance in S1

Lab Code	Ag (mg/kg)	As (mg/kg)	B (mg/kg)	Ba (mg/kg)	Ca (mg/kg)	Cd (mg/kg)	Co (mg/kg)	Cr (mg/kg)	Cu (mg/kg)	Fe (mg/kg)	Hg (mg/kg)	K (mg/kg)	La (mg/kg)	Li (mg/kg)	Mg (mg/kg)
A.V.	0.161	2.60	Not Set	3.62	1830	0.127	0.627	1.47	1.41	52.7	0.415	18600	0.132	0.230	1390
H.V.	0.153	2.57	1.77	3.53	2100	0.130	0.613	1.42	1.28	51.5	0.428	19100	0.138	0.207	1400
S.V.	0.175	2.49	2.00	3.88	Not Spiked	0.138	Not Spiked	1.50	Not Spiked	Not Spiked	Not Spiked	Not Spiked	0.148	0.230	Not Spiked
1	0.15	2.2	NT	3.4	2500	0.12	0.56	1.4	NT	NT	NT	19000	NT	0.24	1800
2	0.161	2.51	NR	NR	NR	0.131	0.665	1.71	1.39	57.3	0.437	18050	NR	NR	1350
3	0.16	2.6	NT	NT	NT	0.12	0.62	1.3	1.4	53	0.462	19000	NT	NT	1430
4	0.16	3	1.9	3.8	1700	0.13	0.7	1.3	1.4	55	0.40	NR	NR	<2	1350
5	0.17	2.14	2.24	3.42	1968	0.12	0.60	1.46	1.34	42.74	0.40	17792	0.13	0.24	1446
6	< 10	< 5	< 50	< 10	1880	< 1	< 5	< 5	< 20	66.6	0.436	19000	NT	< 10	1350
7	NR	2.3	2.2	3.7	1710	0.12	0.633	1.7	1.5	55	0.4	16670	0.13	0.21	1250
8	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
9	0.18	2.91	2.2	4.47	171	0.14	0.79	NR	1.67	51.2	0.45	2018	0.17	0.24	145
10	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
11	0.151	2.75	< 3	3.67	1840	0.144	0.598	1.27	1.27	48.3	0.386	19700	0.132	< 0.5	1420
12	0.15	2.5	NT	3.6	1820	0.12	0.605	1.7	1.3	52.5	0.4	18014	NT	NT	1365
13	NT	2.458	NT	3.490	1733.14	0.123	0.571	1.558	1.292	50.293	0.278	17462.70	NT	NT	1386.12
14	0.2	2.88	<5	3.6	1830	0.12	0.64	1.23	1.4	48.4	0.2	19100	0.1	0.2	1270
15	<1	2.47	4.14	3.65	1780	0.14	0.63	2.20	1.53	56.3	0.44	19300	NT	0.33	1470
16	0.15	3.3	NT	3.6	69	0.13	0.63	1.1	1.5	120	0.4	20000	0.13	0.2	1400

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

Table 65 Summary of Participants' Results and Performance in S1 (continued)

Lab Code	Mn (mg/kg)	Mo (mg/kg)	Na (mg/kg)	Ni (mg/kg)	P (mg/kg)	Pb (mg/kg)	Rb (mg/kg)	S (mg/kg)	Sb (mg/kg)	Se (mg/kg)	Sn (mg/kg)	U (mg/kg)	V (mg/kg)	Zn (mg/kg)
A.V.	0.948	0.050	3180	0.62	9470	0.186	Not Set	12000	0.100	2.96	0.310	0.0615	0.364	34.8
H.V.	1.00	0.042	3180	0.61	10000	0.185	7.07	NA	0.108	2.75	0.280	0.0603	0.328	32.8
S.V.	Not Spiked	0.201	Not Spiked	Not Spiked	0.111	2.69	0.320	0.0657	0.385	Not Spiked				
1	NT	NT	5500	0.57	NT	0.39	NT	10000	0.09	2.5	NT	0.07	NT	37
2	0.913	NR	3360	0.701	NR	0.187	NR	NR	0.109	3.41	0.146	NR	NR	34.1
3	0.95	0.054	3360	NT	10900	0.19	NT	13800	0.11	3.0	0.31	NT	NT	37
4	0.91	NR	3150	<0.7	10000	0.21	NR	13000	<2	2.7	<2	0.067	0.32	34
5	1.02	0.06	3115	0.87	8987	0.18	6.37	10371	0.10	2.39	0.34	<0.05	0.44	29.90
6	< 10	< 10	3190	< 5	8770	< 5	NT	10800	< 10	< 5	< 10	< 10	< 5	35.2
7	0.96	0.06	2930	0.74	8470	0.17	7.0	11400	0.092	2.6	0.27	0.06	0.31	35
8	NT	NT	NT	NT										
9	0.93	NR	NR	NR	NT	0.24	NR	12000	NR	3.32	NR	0.074	NT	34.4
10	NT	NT	NT	NT										
11	0.86	< 0.10	3210	0.68	9900	0.166	6.67	12800	< 0.5	2.62	< 0.5	0.0617	0.501	33.1
12	0.9	0.044	3150	0.455	9894	0.17	NT	13004	0.1	3.85	0.3	0.055	0.31	36
13	0.910	0.050	3180.64	0.575	8715.005	0.189	NT	10915.50	NT	3.560	NT	NT	NT	30.562
14	0.91	<0.05	2890	0.56	9390	0.16	7.0	NT	0.1	3.01	<0.5	0.06	<0.5	36.6
15	1.28	0.12	3280	1.07	9390	0.22	NT	11900	<1	2.85	0.37	0.05	<1	35.8
16	1.1	0.034	3300	0.51	10000	0.17	7.2	13700	0.16	2.8	0.27	0.056	0.3	35

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

Table 66 Summary of Participants' Results and Performance in S2

Lab Code	Al (mg/kg)	As (mg/kg)	B (mg/kg)	Ba (mg/kg)	Ca (mg/kg)	Cd (mg/kg)	Co (mg/kg)	Cr (mg/kg)	Cs (mg/kg)	Cu (mg/kg)	Fe (mg/kg)	K (mg/kg)	Mg (mg/kg)	Mn (mg/kg)
A.V.	Not Set	0.01416	Not Set	1.96	1080	0.00505	0.0613	0.263	Not Set	13.1	121	6720	3150	88.5
H.V./R.V./I.V.	12.8	0.01416	15.4	1.76	1100	0.00505	0.0650	0.263	0.0360	12.6	119	5990	3700	81
S.V.	Not Spiked													
1	NT													
2	7.01	0.014	NR	NR	1230	0.005	0.058	0.228	NR	14.2	129	8410	3806	96.2
3	10	0.019	NT	NT	1100	<0.01	0.062	0.22	NT	13	130	7000	3190	88
4	NT													
5	4.30	0.02	11.81	1.95	1136	<0.01	0.07	0.30	0.04	12.19	111.42	6683	3176	82.82
6	< 20	< 5	< 50	< 10	1040	< 1	< 5	< 5	NT	< 20	136	6380	3040	98.5
7	6.9	<0.05	NR	2.1	930	<0.01	0.06	0.21	<0.5	13	110	6210	2730	87
8	5.1	<0.1	15	1.8	950	<0.1	0.06	0.21	<0.1	12	110	5300	2900	80
9	NR	NT	17.1	2.24	96.8	0.006	0.064	NR	NR	14.1	118	736	314	87.0
10	13.34	<0.2	16	1.9	1031	<0.02	0.075	<1	NT	13	119	5675	3091	84
11	NT													
12	9.6	0.016	NT	1.9	1029	0.005	0.041	0.25	NT	14	145	6826	3025	92.5
13	4.191	< 0.5	NT	1.951	1063.02	0.009	0.055	0.385	NT	12.886	113.181	6859.00	2999.86	82.925
14	NT													
15	14.1	<1	22.0	1.90	1260	<0.1	<0.1	<1	NT	12.6	110	7970	3720	95.0
16	NT													

Shaded cells are results which returned a questionable or unsatisfactory z-score. A.V. = Assigned Value, H.V. = Homogeneity Value, R.V. = Reference Value, I.V. = Information Value, S.V. = Spike Value

Table 66 Summary of Participants' Results and Performance in S2 (continued)

Lab Code	Mo (mg/kg)	Na (mg/kg)	Ni (mg/kg)	P (mg/kg)	Pb (mg/kg)	S (mg/kg)	Sb (mg/kg)	Se (mg/kg)	Sr (mg/kg)	TKN (mg/kg)	TOC (mg/kg)	Total Ash (mg/kg)	V (mg/kg)	Zn (mg/kg)
A.V.	0.575	Not Set	0.697	6010	0.0180	2140	Not Set	0.055	25.0	Not Set	Not Set	Not Set	Not Set	43.4
H.V./R.V./I.V.	0.62	13.5	0.697	5560	0.0180	NA	0.0033	0.041	25.6	NA	NA	NA	0.0212	55
S.V.	Not Spiked	Not Spiked	Not Spiked	Not Spiked	Not Spiked									
1	NT	NT	NT	NT	NT									
2	NR	10.5	0.724	NR	0.015	NR	0.005	0.060	NR	NR	NR	NR	NR	47.5
3	0.65	<50	0.68	6750	0.018	2370	<0.01	0.047	NT	35800	NT	5.0	NT	45
4	NT	NT	NT	NT	NT									
5	0.54	20.80	0.70	5631	0.02	2114	<0.01	0.05	23.46	3.52	48.76	5.02	0.09	41.32
6	< 10	< 50	< 5	6030	< 5	2010	< 10	< 5	< 50	NT	NT	NT	< 5	45.1
7	0.59	16	0.69	5780	0.011	1940	<0.01	<0.05	28	36000	420000	5.1	<0.1	40
8	0.52	12	0.62	5600	<0.1	1900	<0.1	0.06	24	35000	550000	51000	<0.5	43
9	NR	NR	NR	NT	0.015	2034	NR	0.07	26.7	NR	NR	NR	NT	41.5
10	0.97	<20	0.76	6035	0.02	2339	<0.02	0.04	25	31000	434000	50500	<0.2	43
11	NT	NT	NT	NT	NT									
12	0.61	NT	0.625	6277	0.016	2284	0.003	0.059	24.5	NR	NR	NR	0.017	45
13	0.554	16.00	0.686	5979.85	0.024	2107.255	NT	NT	25.204	NT	NT	NT	NT	42.449
14	NT	NT	NT	NT	NT									
15	0.56	<50	0.61	6230	<0.1	2260	<1	<0.5	23.6	36200	476000	52800	<1	44.5
16	NT	NT	NT	NT	NT									

Shaded cells are results which returned a questionable or unsatisfactory z-score. A.V. = Assigned Value, H.V. = Homogeneity Value, R.V. = Reference Value, I.V. = Information Value, S.V. = Spike Value, NA = Not Available

Extraction Method

The Codex Alimentarius Commission recommendation for the measurement of elemental impurities in food samples by ICP is “digestion until extraction is complete”. Laboratories are expected to report total elements in food samples.¹⁶ In previous NMI PT studies participants used various extraction methods and the results produced were compatible except for Al, Cr, Fe, Ni and V in some types of food (Figure 61).

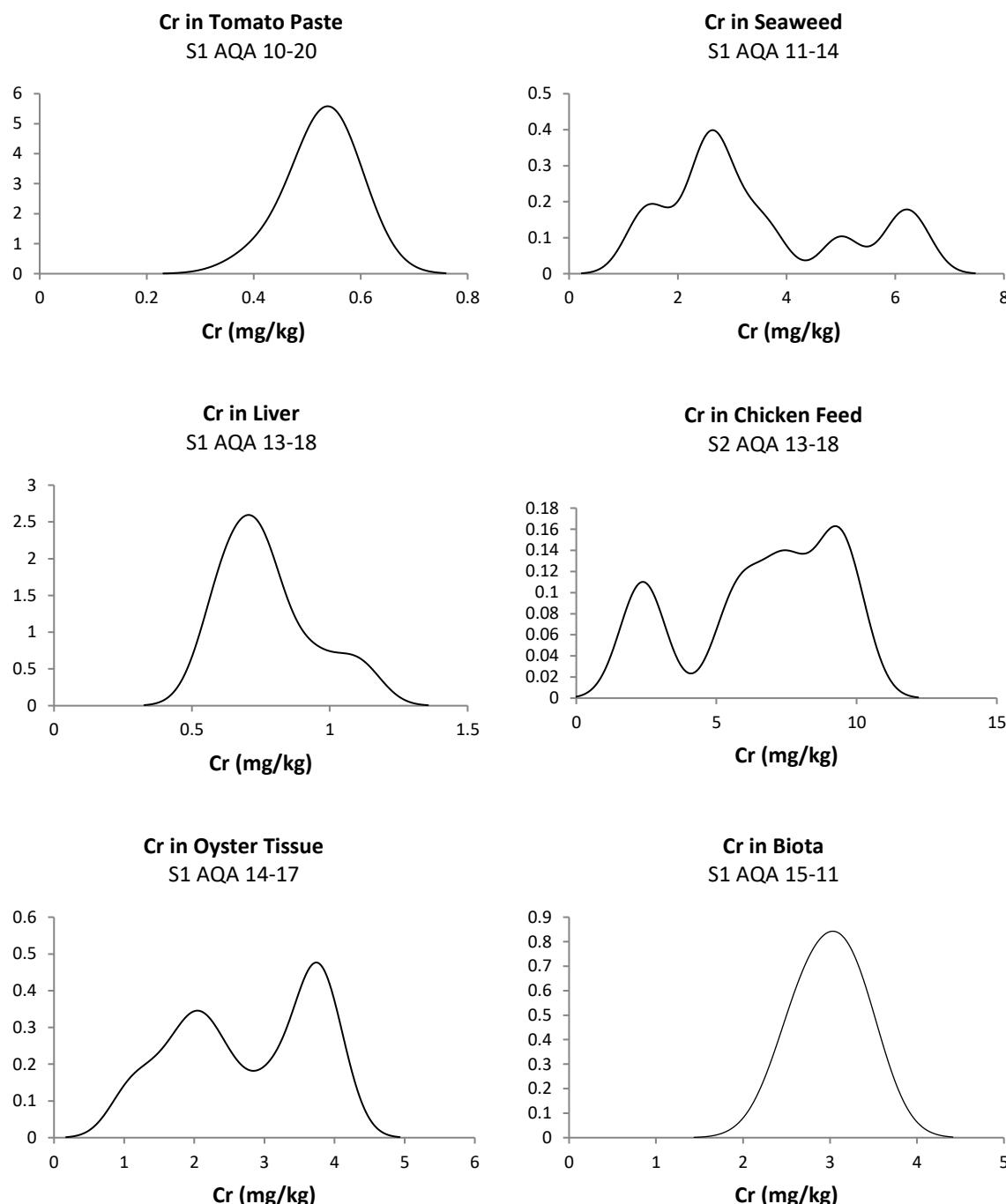


Figure 61 Density Plots of Cr Results

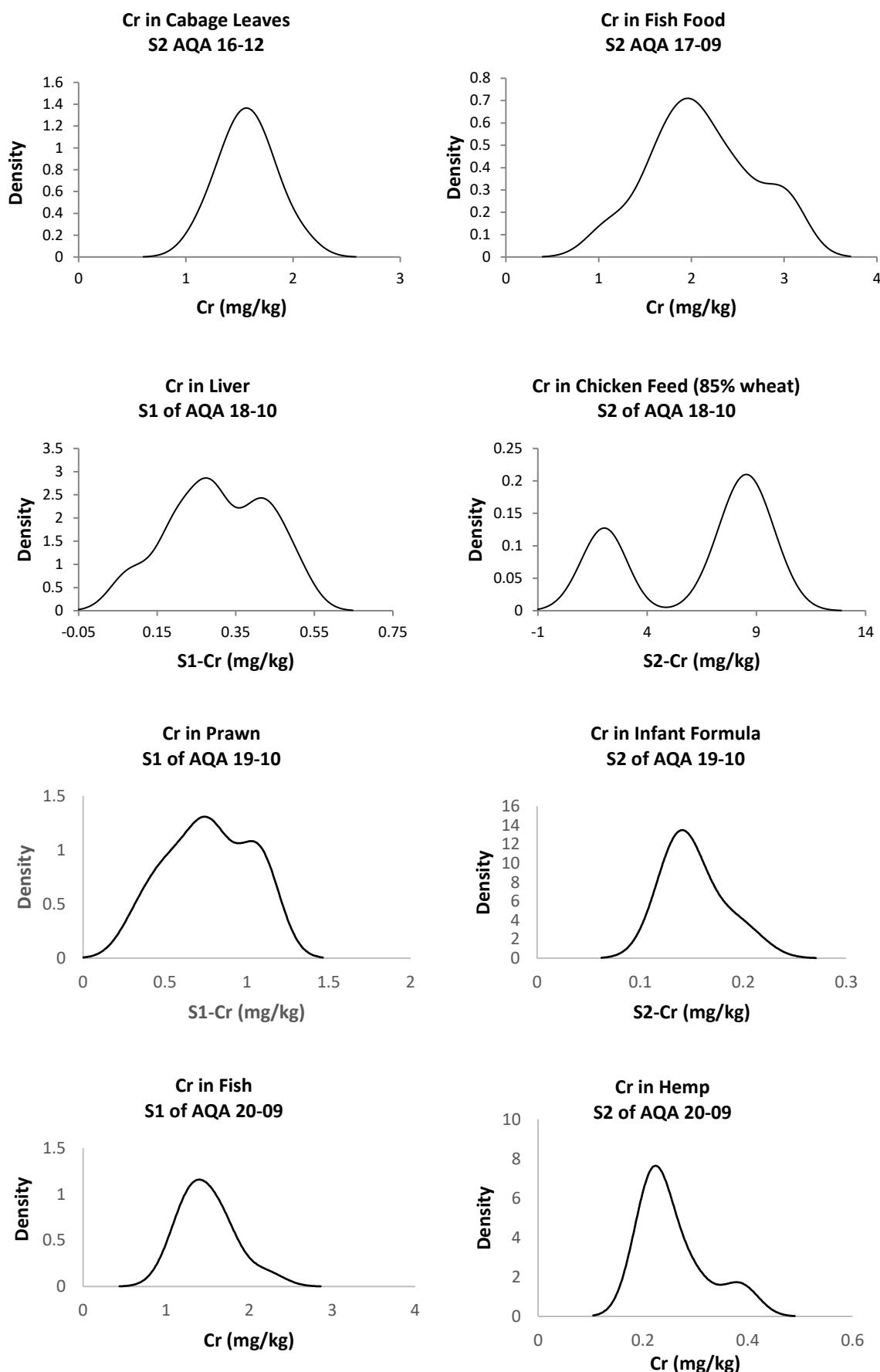


Figure 61 Density Plots of Cr Results (continued)

The extraction of these elements is strongly dependent on digestion regime, especially when the food material has high silica content. An aggressive digestion regime nitric acid, a high digestion temperature (larger than approximately 170°C) and/or hydrofluoric acid is usually recommended for the complete extraction of these elements.

Food laboratories have to test for total elements in a large number of samples and often simultaneously in the same batch. It is a challenge for them to find a method/extraction regime suitable to all types of food samples for all total elements. The use of HF is banned in many laboratories and microwave digesters allow only a limited number of samples to be digested at a time. Evidence was found in previous studies of the importance of using (in addition to nitric acid) a high ratio HCl (mL)/sample size (g) when a high digestion temperature (> 170°C) or when HF cannot be used for total Cr, Fe, Ni and V extraction.

In the present study, most participants (12) used a high digestion temperature of 165°C or higher and/or a high ratio HCl/sample size of 4 or larger. Laboratory 12 reported using 165°C for “trace elements” and 85°C for “major elements”.

The amount of sample taken for analysis by most participants was 0.5 g. One reported using 0.1 g and two used 0.2 g. Caution should be exercised when a small sample size is taken for analysis as this might not be representative of the whole sample. Caution should also be exercised if using too much sample mass and not enough digestion acid for the sample mass.

Two participants used only nitric acid for extraction. Of these two, one used a digestion temperature of 180°C and the other a digestion temperature of 165°C.

Laboratory 9 reported for Sample S1: “Our laboratory noticed that microwave digestion of sample S1 at 200°C gave higher results than a block digestion at 95°C for the following elements: Cr, Mo, Na, Ni, Rb and Sn.” For Sample S2, Laboratory 9 also reported: “We noticed that microwave digestion of sample S2 at 200°C gave higher results than a block digestion at 95°C for the following elements: Al, Cr, Cs, Mo, Na and Ni.” For hot block digestion, Laboratory 9 used a temperature of 95°C for 30 min only and no HCl. The results from the experiments conducted by Laboratory 9 are presented in Table 67.

Table 67 Lab 9 Experiment Results: Cr, Ni and V Results in S1 vs. Reagents

Sample ID	Digestion Regime	Cr (mg/kg)	Ni (mg/kg)	Rb (mg/kg)	Sn (mg/kg)
S1 (fish)	0.1g, 200°C, 18 min, 10 mL HNO ₃	1.56	0.64	8.33	0.40
	0.1g, 95°C, 30 min, 10 mL HNO ₃	1.03	0.40	7.35	0.30
		1.47*	0.62*	Not Set	0.310
S2 (hemp)	0.1g, 200°C, 18 min, 10 mL HNO ₃	0.314	0.844		
	0.1g, 95°C, 30 min, 10 mL HNO ₃	0.097	0.692		
	0.5 g, 260°C, 30 min, 3 mL HNO ₃	0.263**	0.697**		

*Assigned Value calculated from consensus of participants' results; **Reference Value

Aluminium is one of the most difficult elements to analyse in food samples. In previous PT studies, no assigned values could be set in the wheat, oyster tissue, freeze dried liver, biota and freeze dried prawn samples because the reported results varied too much. Incomplete dissolution of silicate compounds might explain the variability of the results.

In the present study, the results reported for Al in hemp were variable (CV_{rob} 51%). The robust average of reported results for Al in S2 was 8.28 mg/kg while the NMI information value was 12.8 mg/kg. As the request was for “digestion until extraction was complete” the values closest to the “true” value are most likely those reported by Laboratories 12, 3, 10 and

15 whose results were 9.6 mg/kg, 10 mg/kg, 13.34 mg/kg and 14.1 mg/kg respectively. All four sets of results were also in good agreement with the NMI information value of 12.8 mg/kg (Figure 62). All results were produced under digestion temperatures of 165°C or higher, except for one.

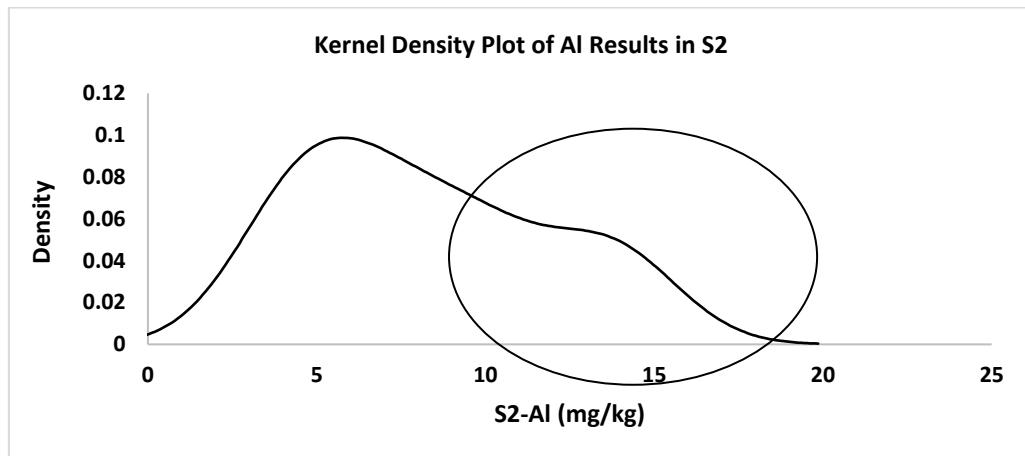


Figure 62 Participants' Results versus Digestion Regime

Lab code	S2 Al Results mg/kg	Digestion Regime					
		Sample Mass (g)	Temp °C	Time (min.)	HNO ₃ (mL)	HCl (mL)	H ₂ O ₂ (mL)
13	4.191	1	85	240	5	5	
5	4.30	0.3	85	240	3	2	
8	5.1	0.7	90-98	60	3	3	
7	6.9	0.5	100	120	3	1	
2	7.01	1	110	60	5	1.5	
12	9.6	0.5	165	50	5		
3	10	0.5	180	30	6		1
10	13.34	0.5	210	15	7 (1:1)	0.5	1
15	14.1	0.4	120	60	2.5	7.5	
NMI Information Value							
	12.8	0.5	260	30	3		

Chromium, Iron, Nickel and Vanadium are four elements often observed to be strongly dependent on the digestion regime, especially when the sample has high silica content.

In the present study, most of the reported results in fish and hemp samples were from digestion regimes that involved a high digestion temperature of 165°C or higher and/or a high ration HCl sample size of 4 or more. Most of the reported results for these elements were in good agreement with each other and with the assigned value, homogeneity value and/or reference or informative value. The robust between laboratories CV for these elements was also found to be in relative good agreement with Thomson CV, with the exception of V in S1. Unsolved ³⁵Cl¹⁶O⁺ interferences on low level ⁵¹V may explain the results variability.

Total Al, Cr, Fe, Ni and V are some of the most difficult elements to analyse in food samples. According to Eurachem/CITAC Guide CG 4, laboratories should consider using matrix matched control samples to assess their extraction efficiency (the bias of their analytical method). Bias can be expressed as recovery and should be corrected for or included in the uncertainty estimate.¹

Instrumental Techniques

Plots of participants' results/performance with the instrumental technique used are presented in Figures 63 to 69.

Aluminium, Chromium, Nickel No association between Al, Cr and Ni results/performance in S1 and S2 and the instrumental technique used was apparent (Figure 63).

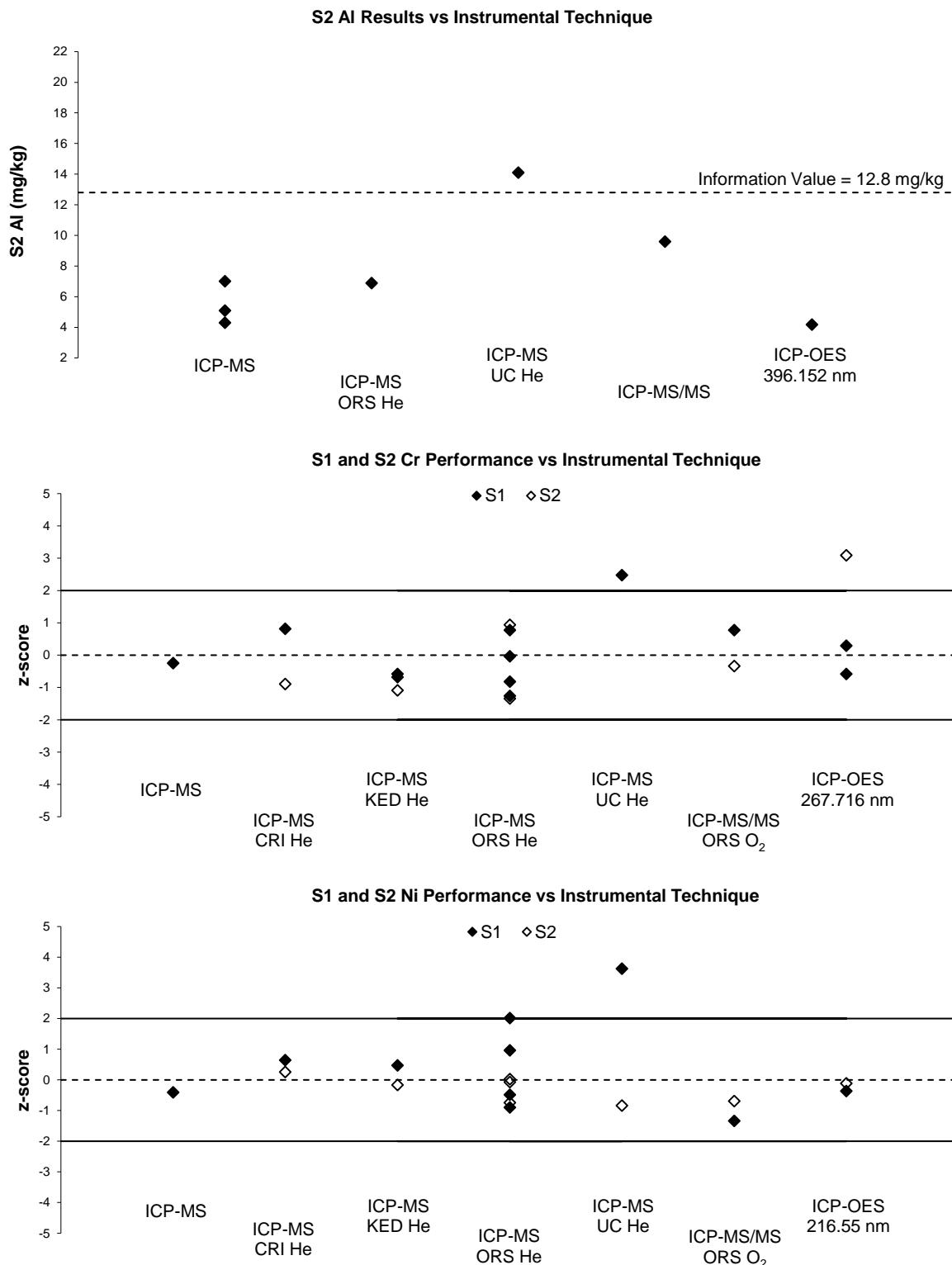


Figure 63 Al, Cr and Ni Results/Performance vs. Instrumental Technique

Arsenic Participants used a wide variety of instrumental techniques for As measurements in S1 and all produced satisfactory results (Figure 64).

Arsenic level in Sample S2 was low (0.01416 mg/kg) and challenged participants' instrumental techniques. Only four results were reported for this test and all were in good agreement with each other and with the reference value.

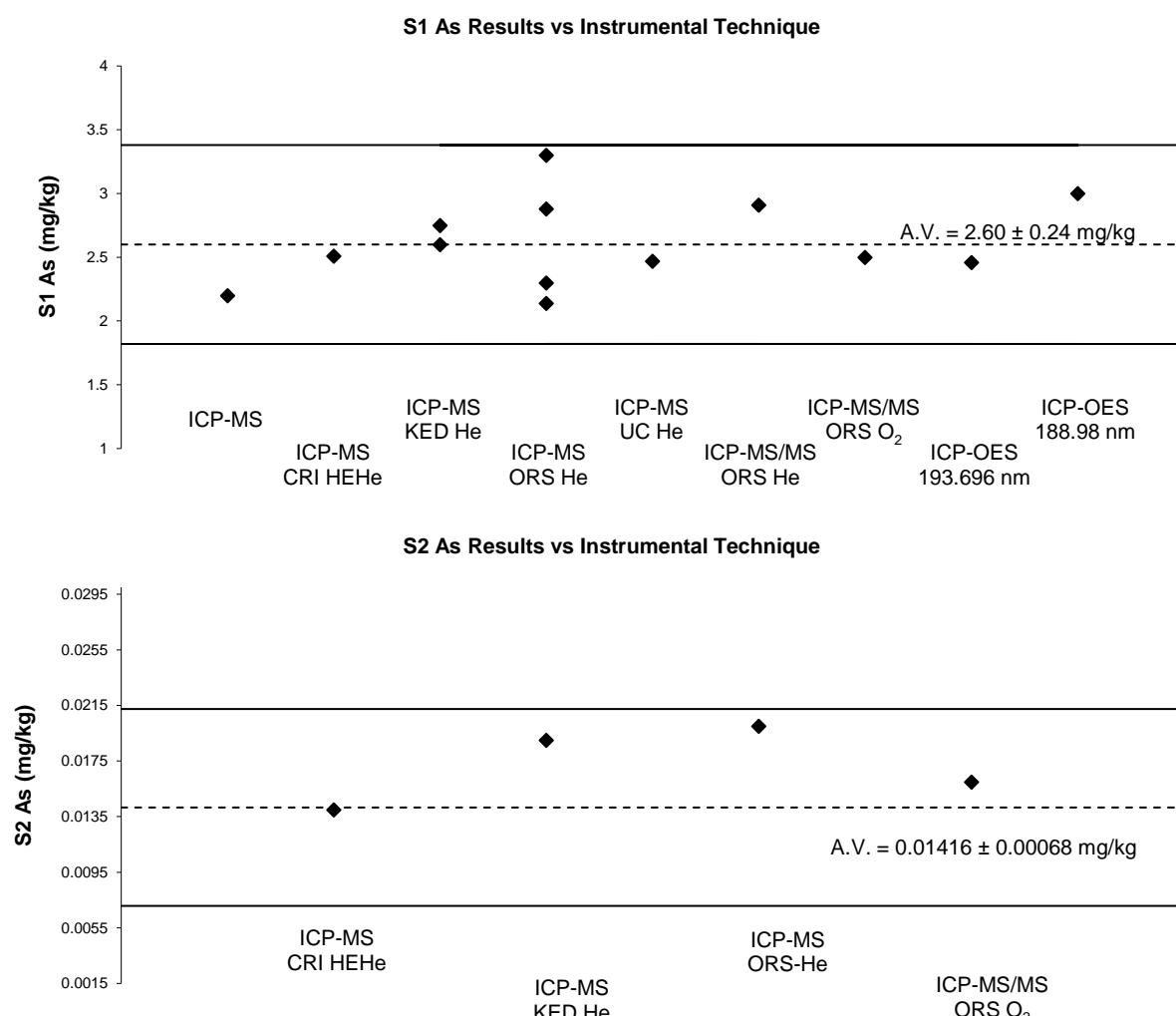


Figure 64: S1 and S2 As Results vs. Instrumental Technique

Boron is one of the elements that presented the most analytical difficulty to participating laboratories.

Only 5 results were reported for B in S1, and all were in agreement with each other and with the robust average (2.32 mg/kg) except for one. One participant used for B measurements ICP-OES with wavelength 249.678 nm (Figure 65)

Five participants reported results for B in S2 and all reported results were in agreement with each other centred on a value of 16.4 mg/kg value.

Caesium level in S2 was below the level of reporting of all participants with the exception of one. The result reported by laboratory 5 for this element was in good agreement with the homogeneity value of 0.036 mg/kg.

Cadmium level in hemp was likely too low (0.00505 mg/kg) to be reliably measured by ICP-OES (Figure 66).

S1 B Results vs Instrumental Technique

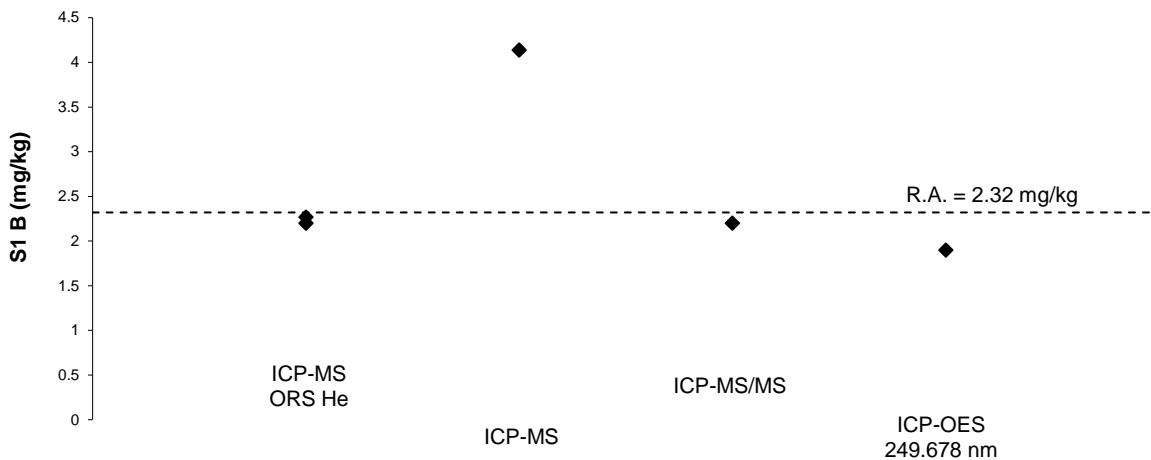


Figure 65: S1 B Results vs. Instrumental Technique

S2 Cd Results vs Instrumental Technique

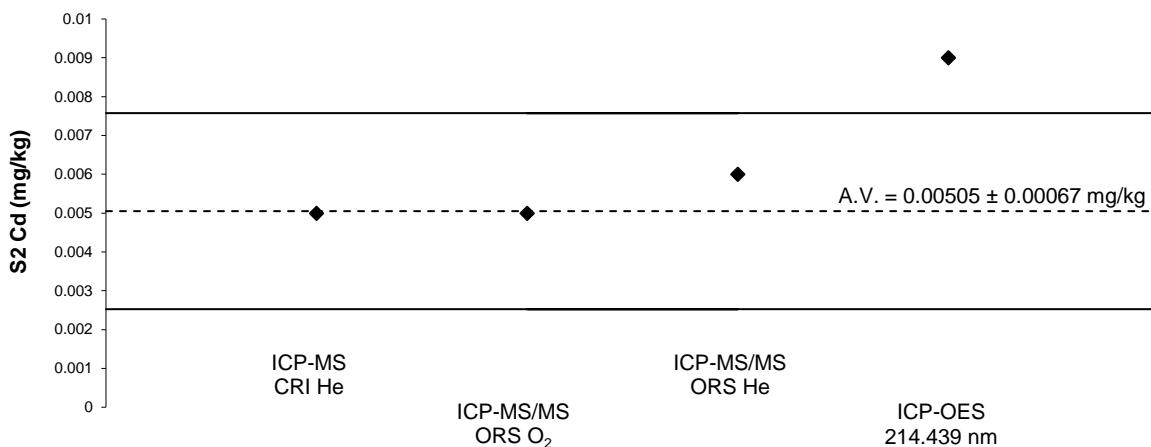
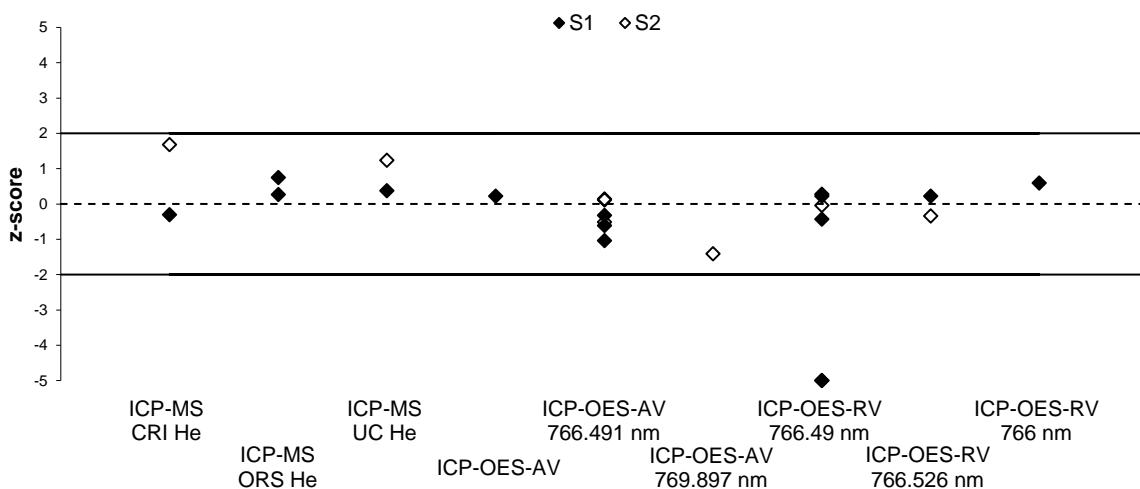


Figure 66: S1 Cd Results vs. Instrumental Technique

S1 and S2 K Performance vs Instrumental Technique*



* z-Scores smaller than -5 have been plotted as -5.

Figure 67 S1 and S2 K z-Scores vs. Instrumental Technique

Calcium, Magnesium, Potassium, Sodium Problems with the sample preparation, dilution standard preparation and/or calculation procedure were likely the main causes of the majority of unsatisfactory results reported for these elements in S1.

No association between Ca, Mg, K and Na results/performance in S1 and S2 and the instrumental technique used was apparent.

Sodium level in S2 was low, 15.1 mg/kg and challenge participants' analytical techniques. Of 16 laboratories, only 5 reported results for this element in S2. All participants used ICP-OES for low-level Na measurements with the exception of one (Figure 68).

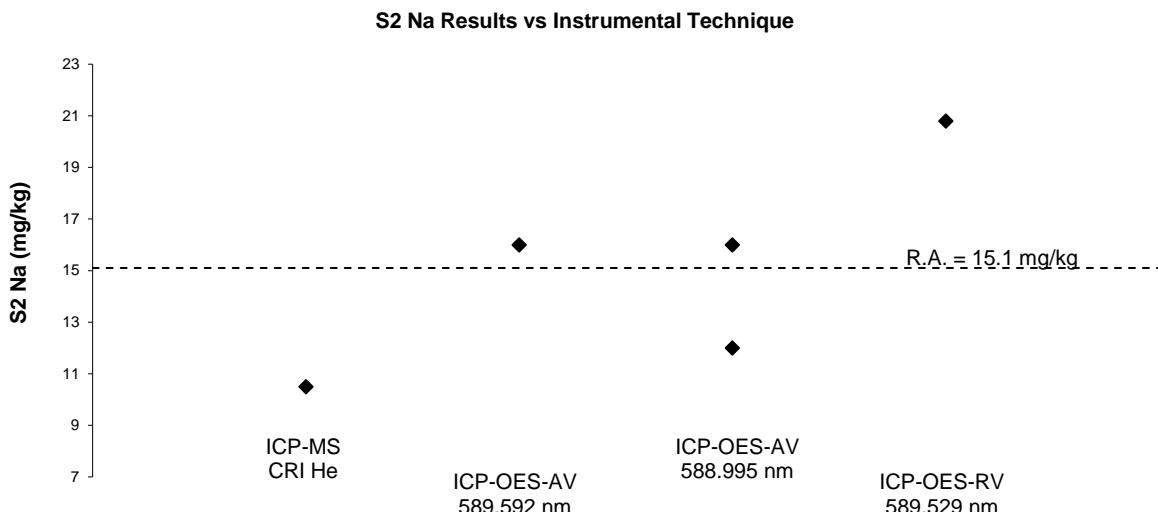


Figure 68: S2 Na Results vs. Instrumental Technique

Mercury measurements did not present analytical difficulty to laboratories. Participants used a wide variety of extraction regimes and instrumental techniques and all produced comparable results (Figure 69).

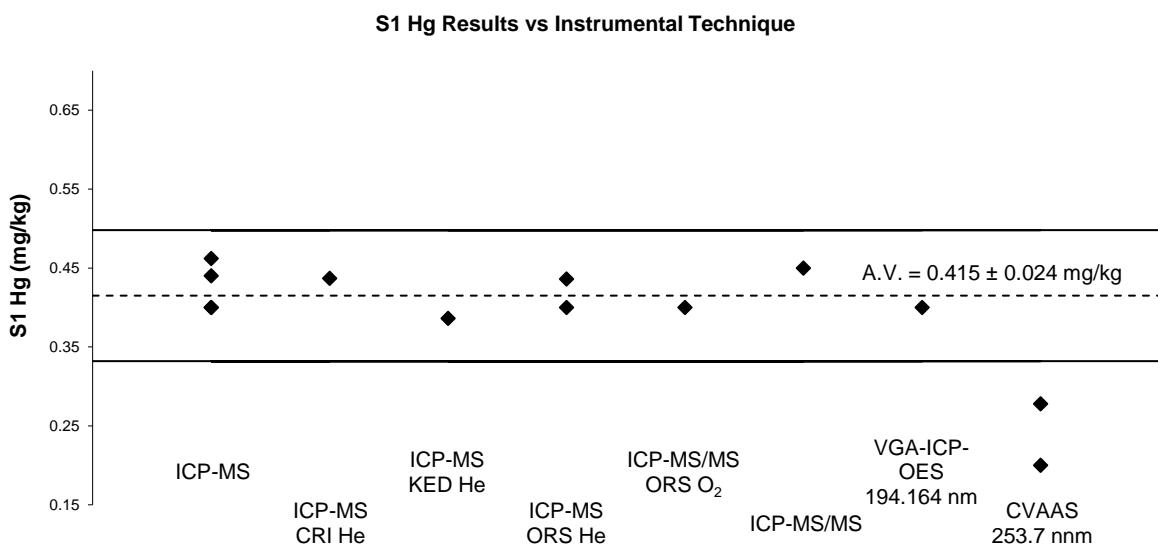


Figure 69: S1 Hg Results vs. Instrumental Technique

All participants used the same digestion regime as for the other elements, with the exception of one. One participant reported "Mercury digestion performed using hot block at 95C, digested for 45 mins 3.5mL HNO₃, 3.5mL H₂O₂, and 0.5 g sample size"

Selenium Apart from molecular and polyatomic interferences whose effects may be reduced by using a collision reaction cell, matrix effects are another main factor that can hamper accurate measurements of elements in food samples when complete digestion cannot be achieved. Matrix effects are common in food analyses using ICP-MS; they take place in the plasma and consist of signal enhancement caused by charge transfer reactions from charged carbon species to atoms like Se with a lower ionization potential.¹⁷ One participant might have struggled to overcome the interference problem in S1 (Figure 70).

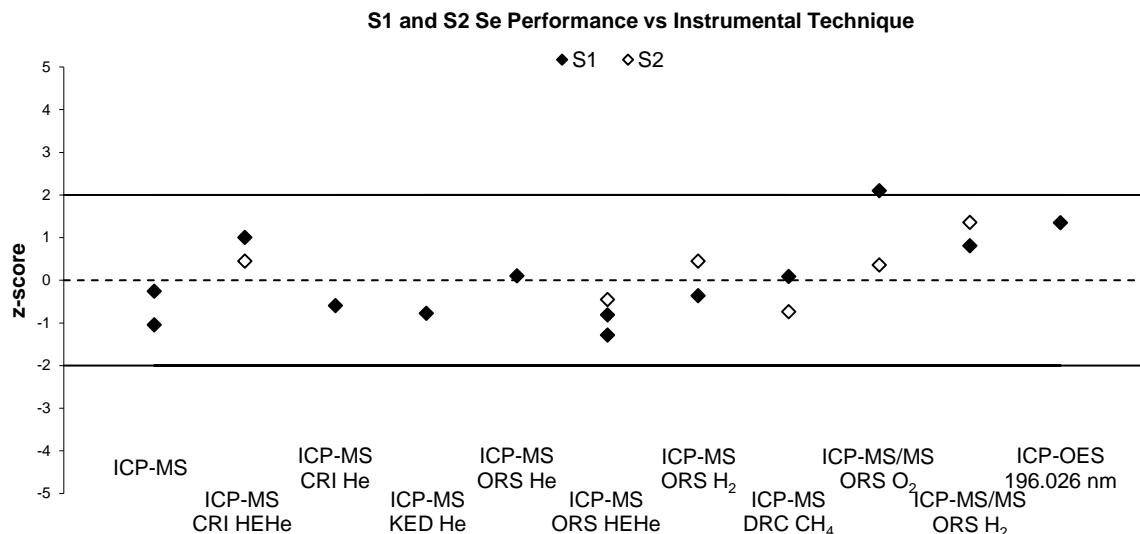


Figure 70: S1 and S2 Se z-Scores vs. Instrumental Technique

Vanadium No assigned value could be set for low-level V in S2 because only two participants reported results for this test. Overcoming $^{35}\text{Cl}^{16}\text{O}^+$ interferences on low-level ^{51}V was the main challenge for participating laboratories.

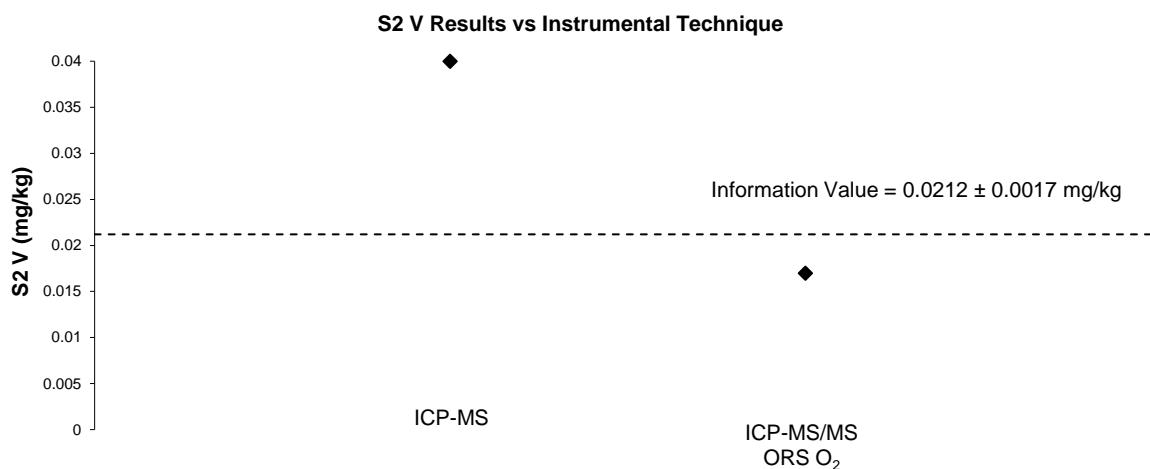


Figure 71: S2-V vs. Instrumental Technique

ICP-MS with collision/reaction cells and He as collision gas is the most popular instrumental technique used to solve interferences. While He is generally an effective interference removal strategy for many polyatomic interferences, when a typical flow rate is used, it might struggle to effectively remove $^{35}\text{Cl}^{16}\text{O}^+$ interferences often present in food matrices with high salt content.

Table 68 presents the results of V from measurements conducted by NMI using two instrumental conditions.

Table 68 NMI V results vs. Instrumental Technique

Instrumental technique used	ICP-MS-ORS He 5.8 mL/min	ICP-MS/MS-ORS O ₂ +H ₂ (70% gas; 0.5 mL/min)
Result (mg/kg)	0.0207	0.0212

7.6 Comparison with the Previous Proficiency Studies of Metals and Nutrients in Soil

AQA 20-09 is the fifteenth NMI study of elements in food. Laboratories improved their performance in the measurement of Cr, Ni and Fe in food samples. The results reported for these elements in the hemp sample S2 were in good agreement with each other and with the assigned values/reference values with the exception of one. The between laboratory CV for these elements were also in good agreement with Horwitz CV.

The participants' performance in measurements of trace elements in food over time is presented in Figure 65. Despite differences in matrices and concentrations laboratories' overall performance has remained fairly constant.

Over time laboratories should expect at least 95% of its scores to be $|z| \leq 2.0$. Scores in the range $2.0 < |z| \leq 3.0$ 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.

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.

7.7 Reference Materials and Certified Reference Materials

Proficiency testing and matrix matched control samples taken through all steps of the analytical process are highly valuable quality control tools for assessing extraction efficiency. Control samples used by participants in this study are presented in Table 69.

Table 69 Control Samples Used by Participants

Lab. Code	Description of Control Samples
1	CRM
2	CRM – DORM3 – Fish Protein; NIST SRM 1567B – Wheat Flour
3	RM
4	RM
5	Spiked Sample
7	Spiked Sample
9	NIST 2976, DORM-3, DOLT-4 and BCR-279
10	CRM
11	CRM – Apple Leaves NIST 1515, Oyster Tissue NIST 1566b, Rice Flour NIST 1568a, Mushroom CS-M-3, Dairy Proficiency – Micronutrients, NRS Metals in Liver, FAPAS, MBM and Meat Paste- Global Proficiency
12	RM – In-house reference materials (from previous PT program) and Certified reference materials (ERM and NIST)

Lab. Code	Description of Control Samples
13	CRM
14	RM
15	RM
16	NIST SRM1566b, NIST SRM2976

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'¹⁸

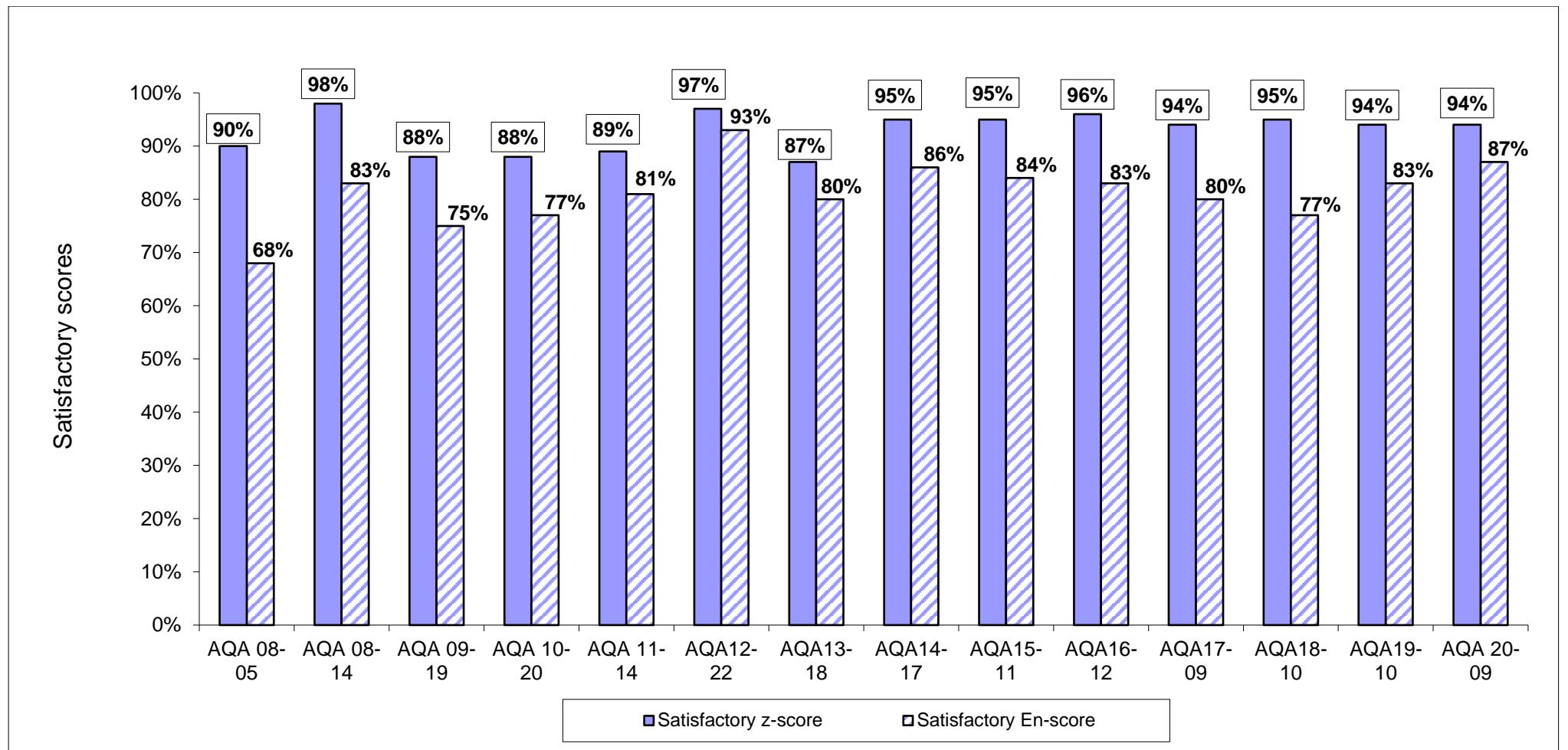


Figure 72 Participants' Performance over Time

AQA 20-09 Metals in Food

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APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING

A1.1 Sample Preparation

Sample S1 – was ground, sieved and freeze dried fish.

Sample S2 – was hemp.

A1.2 Sample Analysis and Homogeneity Testing

The same procedure was followed for the preparation of Samples S1 as in previous NMI PT studies. Partial homogeneity testing was conducted for elements of interest, except S. Three bottles were analysed in duplicate and the average of the results was reported as the homogeneity value. Measurements were made under repeatability conditions in random order.

A full homogeneity test was conducted for Sample S2 for all elements except Ag, S, TKN, TOC and total ash. Homogeneity testing was based on that described in the International Protocol.⁵ Six sample bottles 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. Table 70 sets out an example for the testing of the homogeneity of Sb in Sample S2.

Table 70 Homogeneity Testing of Sb in Sample S2

BOTTLE	A Sb (mg/kg)	B Sb (mg/kg)
2	0.00327	0.00326
5	0.00336	0.00378
11	0.00371	0.00399
13	0.00282	0.00313
22	0.00311	0.00320
23	0.00325	0.00348

	Value	Critical	Result
Cochran	0.43	0.73	Pass
S_{an}/σ	0.21	0.50	Pass
s^2_{sam}	0.00001	0.00001	Pass

Homogeneity values for As, Cd, Cr, Ni and Se in S2 were the reference value while for Al and V were the information value (Appendix 3).

Sample Analysis for Total Elements in S1 and S2

Approximately 0.5 g of sample was weighed and digested at 260°C for 1 hour with 3 mL HNO₃ and 1 mL HCl. After digestion, each sample was diluted to 40 mL with ultra-high purity water and then further diluted as necessary for ICP-MS determination. A summary of the instruments used and the ion monitored for each analyte is given in Table 71.

Table 71 Instrumental Techniques Used for Total Elements in S1 and S2

Analyte	Instrument	Internal Standard	Reaction/ Collision Cell (if applicable)	Cell Mode/ Gas (if applicable)	S1 Final Dilution Factor	S2 Final Dilution Factor	Ion
Ag	ICP-MS	Rh	ORS	He	400	NA	107 m/z
As	ICP-MS	Rh	ORS	He	NA	400	75 m/z
B	ICP-MS	Rh	NA	NA	400	400	11 m/z
Ba	ICP-MS	Rh	ORS	He	400	400	137 m/z

Element	Method	Reactor	Source	Gas	Flow Rate	Flow Rate	m/z
Ca	ICP-MS	Rh	ORS	He	400	400	44 m/z
Cd	ICP-MS	Rh	ORS	He	400	NA	114 m/z
Co	ICP-MS	Rh	ORS	He	400	400	59 m/z
Cr	ICP-MS	Rh	ORS	He	400	NA	53 m/z
Cs	ICP-MS	Rh	ORS	He	NA	400	133 m/z
Cu	ICP-MS	Rh	ORS	He	400	400	65 m/z
Fe	ICP-MS	Rh	ORS	He	400	400	56 m/z
Hg	ICP-MS	Rh	NA	NA	400	NA	202 m/z
K	ICP-MS	Rh	ORS	He	400	400	39 m/z
La	ICP-MS	Rh	ORS	He	400	NA	139 m/z
Li	ICP-MS	Rh	ORS	He	400	NA	7 m/z
Mg	ICP-MS	Rh	ORS	He	400	400	24 m/z
Mn	ICP-MS	Rh	ORS	He	400	400	55 m/z
Mo	ICP-MS	Rh	ORS	He	400	400	95 m/z
Na	ICP-MS	Rh	ORS	He	400	400	23 m/z
P	ICP-MS	Rh	ORS	HEHe	400	400	31 m/z
Pb	ICP-MS	Ir	ORS	He	400	NA	Average of 206, 207, 208 m/z
Se	ICP-MS	Rh	ORS	HEHe	400	400	78 m/z
Sn	ICP-MS	Rh	ORS	He	400	NA	118 m/z
Sr	ICP-MS	Rh	ORS	He	NA	400	88 m/z
U	ICP-MS	Ir	ORS	He	400	NA	238 m/z
V	ICP-MS	Rh	ORS	He	400	NA	51 m/z
Zn	ICP-MS	Rh	ORS	He	NA	400	213 m/z

NA- Not Applicable

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

Assigned Value

The assigned value was calculated as the robust average using the procedure described in ‘ISO13528:2015(E), Statistical methods for use in proficiency testing by inter-laboratory comparisons – Annex C’.⁵ 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 72.

Table 72 Uncertainty of Assigned Value for As in Sample S1

No. results (p)	13
Robust Average	2.60 mg/kg
$S_{rob\ av}$	0.35 mg/kg
$u_{rob\ av}$	0.12 mg/kg
k	2
$U_{rob\ av}$	0.24 mg/kg

The assigned value for As in Sample S1 is **2.60 ± 0.24 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 2 and Equation 3 respectively (see page 11).

A worked example is set out below in Table 73.

Table 73 z-Score and E_n-score for As result reported by Laboratory 5 in S1

As Result mg/kg	Assigned Value mg/kg	Set Target Standard Deviation	z-Score	E _n -Score
2.14 ± 0.43	2.60 ± 0.24	15% as CV or 0.15 x 2.60 = 0.39 mg/kg	$z = \frac{(2.14 - 2.60)}{0.39}$ $z = -1.18$	$E_n = \frac{(2.14 - 2.60)}{\sqrt{0.43^2 + 0.24^2}}$ $E_n = -0.93$

APPENDIX 3 – REFERENCE VALUES

A.3.1 Description of Method of Analysis

All analytes were quantified by either double isotope dilution ICP-MS (Cr and Ni) or the method of standard additions using ICP-MS (Al, As, Cd, Pb and V). NIST 3100 series primary calibration materials were used, see table below for details, and these were diluted gravimetrically to working concentrations.

Analyte	Standard Name	Lot No.
Al	NIST 3101a	140903
As	NIST 3103a	100818
Cd	NIST 3108	130116
Cr	NIST 3112a	170630
Ni	NIST 3136	120619
Pb	NIST 3128	101026
V	NIST 3165	160906

Isotope dilution was performed by spiking calibration standards and undigested samples with isotopically enriched ^{61}Ni (Oakridge) and ^{53}Cr (Isoflex) standards. For standard addition analysis a mixed internal standard was spiked into the undigested samples.

In both methods approximately 0.5 g of sample was weighed into a PTFE microwave vessel along with the internal standards followed by the addition of 3 mL HNO₃ (69%) digestion reagents. Samples were then digested in a microwave digester (260 °C, 90 bar, 30 min ramp, 30 min hold). Digests were then diluted to 25 mL with UHP water to produce a clear and colourless solution. This digest solution contained fine white particulates assumed to be undigested silica.

For standard addition analysis this solution was then gravimetrically separated into ‘spiked’ and ‘unspiked’ solutions with gravimetric additions of standards to the ‘spiked’ solutions.

Each experimental batch contained CRMs and method blanks prepared using the same procedures. Isotope ratios were measured by ICP-MS-MS using various conditions described in the table below. Measurements for each analyte under multiple conditions were taken to estimate potential biases in the instrumental measurement. For isotope dilution ICP-MS measurements all samples were measured bracketed on either side by the calibration solutions. For standard addition all ICP-MS measurements of the ‘spiked’ solutions were measured bracketed on either side by the ‘unspiked’ solution.

Analytes	Gas	Gas Flow Rates	Analysis Mode	Mass Shift
Al, As, Cr, Ni, Cd, Pb	He	4 mL/min	Single Quadrupole	No
V	He	5.8 mL/min	Single Quadrupole	No
Al, As, Cr, Ni	O ₂ + H ₂	30% + 0.5 mL/min	MS/MS	Yes
Cd	O ₂ + H ₂	30% + 0.5 mL/min	MS/MS	No
V	O ₂ + H ₂	70% + 0.5 mL/min	MS/MS	Yes
Pb	None	n/a	Single Quadrupole	No

A.3.2 Reference Values

The reference values and associated measurement uncertainty estimates for AQA 20-09 Sample S2 are presented below. The reference values come from the analysis of 7 bottles in duplicate. Measurement uncertainty is given as a 95% level of confidence. Measurements are based on the sample as received, no specific correction or uncertainty for moisture content variation has been made.

Sample	Analyte	Reference Value (mg/kg)	Expanded Uncertainty (95%) (mg/kg)	Relative Expanded Uncertainty	Coverage Factor (95%)
AQA 20-09 S2	As	0.01416	0.00068	4.8%	2.18
	Cd	0.00505	0.00067	13%	2.02
	Cr	0.263	0.015	5.7%	2.16
	Ni	0.697	0.035	5.0%	2.03
	Pb	0.0180	0.0026	14%	2.36

A.3.3 Information Values

Information values and associated measurement uncertainty estimates for Al and V are also provided below. These information values come from the analysis of 7 bottles in duplicate. Measurement uncertainty is given as a 95% level of confidence. Measurements are based on the sample as received (wet mass), no specific correction or uncertainty for moisture content variation has been made.

Sample	Analyte	Information Value (mg/kg)	Expanded Uncertainty (95%) (mg/kg)	Relative Expanded Uncertainty	Coverage Factor (95%)
AQA 20-09 S2	Al	12.8	0.51	4.0%	2.31
	V	0.0212	0.0017	8.0%	2.08

Based on CRM recoveries for these elements, see table below, insufficient evidence is currently available to confidently state that total recoveries have been achieved. The digestion method (see A.3.1. Description of Method of Analysis) should represent a '*practical*' total extraction (i.e. not using hydrofluoric acid).

Analyte	NIST 1515 (Recovery)	NIST 1547 (Recovery)	NIST 1570a (Recovery)	NIST 1573a (Recovery)
Al	102.0%	100.5%	87.5%	89.1%
V	89.0%	91.8%	96.4%	86.4%

A.3.4 Homogeneity Assessment

Homogeneity was assessed based on the analysis of 7 bottles in duplicate. The experimental data was tested using a single factor ANOVA. At the 95% confidence interval no significant evidence of between bottle inhomogeneity was found for Al, As, Cd, Cr, Ni and V. At this same confidence interval significant evidence of between bottle inhomogeneity was found for Pb. However the sample passes the homogeneity test for this analyte at a PCV of 25%. In all

cases an uncertainty component related to between bottle inhomogeneity was estimated and included into the measurement uncertainty budget.

A.3.5 Stability Assessment

Stability was not assessed.

A.3.6 Reference Value and Informative Value Measurement Uncertainty

The measurement uncertainty associated with the reference values and informative values takes into account all factors that can reasonably be expected to affect the measurement result. Briefly, these include the primary calibration material, gravimetric sample preparation, homogeneity, method trueness and method precision. Some variation in moisture content is expected to be covered in the method precision term but has not been specifically considered. Measurement uncertainty is reported as a 95% level of confidence.

A.3.7 Statement of Traceability

The reference values given in this report rely on gravimetric sample preparation and elemental quantification by ICP MS. Gravimetric measurements were calibrated using Australian standards for mass and are traceable to the SI unit for mass (kg). ICP-MS measurements were calibrated with isotope dilution (Cr and Ni) and standard addition (Al, As, Cd, Pb and V) and are traceable to (i) the SI unit for mass (kg) through the primary calibration standards certified by NIST (USA) and (ii) the SI unit for amount of substance (mol) through gravimetric measurement and data for isotopic composition and relative atomic mass. Isotopic compositions are traceable to IUPAC published data with the exception of Pb which is traceable to certified isotopic composition of an isotopically certified material NIST (USA),

APPENDIX 4 - ACRONYMS AND ABBREVIATIONS

CRM	Certified Reference Material
CV	Coefficient of Variation
CV _{Rob}	Robust Coefficient of Variation
DRC	Dynamic Reaction Cell
GUM	Guide to the Expression of Uncertainty in Measurement
HEHe	High energy helium
HV	Homogeneity Value
IV	Informative Value
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
ICP-MS/MS	Inductively Coupled Plasma – Tandem Mass Spectrometry
ICP-OES-AV	Inductively Coupled Plasma - Optical Emission Spectrometry- axial view
ICP-OES-AV-buffer	Inductively Coupled Plasma - Optical Emission Spectrometry- axial view with buffer
ICP-OES-RV	Inductively Coupled Plasma - Optical Emission Spectrometry- radial view
IDMS	Isotope Dilution Mass Spectrometry
ISO/IEC	International Organisation for Standardisation / International Electrotechnical Commission
IUPAC	International Union of Pure and Applied Chemists
Max	Maximum value in a set of results
Md	Median
Min	Minimum value in a set of results
MU	Measurement Uncertainty
N	Number of Participants
NIST	National Institute of Standards and Technology
NMI	National Measurement Institute (Australia)
NR	Not Reported
NT	Not Tested
ORS	Octopole Reaction System
PCV	Performance Coefficient of Variation
PFAS	Polyfluoroalkyl Substances
PT	Proficiency Test
RM	Reference Material
RV	Reference Value
SA-ICP-MS	Standard Addition - Inductively Coupled Plasma - Mass spectrometry
SV	Spiked or formulated concentration of a PT sample
SD _{Rob}	Robust Standard Deviation
SI	The International System of Units
s ² _{sam}	Sampling variance
s _a /σ	Analytical standard deviation divided by the target standard deviation
SRM	Standard Reference Material (Trademark of NIST)
Target SD	Target standard deviation (symbol: σ)
UC	Universal Cell
VGA-ICP-OES	Vapor Generation Accessory- Inductively Coupled Plasma - Optical Emission Spectrometry

APPENDIX 5 - INSTRUMENT DETAILS

Table 74 Instrument Conditions for Ag in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS					
2	ICP-MS	Rh			39.86	107
3	ICP-MS	Rh103				109
4	ICP-MS	103	Collision	He	100	107
5	ICP-MS	Ge 72	ORS		200	107m/z
6	ICP-MS	Indium	ORS	He	81.6327	107
7	N/A	N/A	N/A	N/A	N/A	N/A
8	NA	NA	NA	NA	NA	NA
9	ICP-MS/MS	none			1700	107
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Rh	KED	He	481.7	109
12	ICP-MS/MS	Rh 103	ORS	O2	80	108 m/z
13						
14	ICP-MS	103	ORS	He	50	107
15	ICP-MS	Rh	NA	NA	625	109
16	ICP-MS	Rh	ORS	He	1000	107

Table 75 Instrument Conditions for As in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS					
2	ICP-MS	Te		HEHe	39.86	75
3	ICP-MS	Te125	KED	He		75
4	ICP-OES-AV-buffer	Lu			100	188.98
5	ICP-MS	Ge 72	ORS		4000	75m/z
6	ICP-MS	Indium	ORS	He	81.6327	75
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	75
8	NA	NA	NA	NA	NA	NA
9	ICP-MS/MS	none		He	1700	75
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Te	KED	He	481.7	75
12	ICP-MS/MS	Rh 103	ORS	O2	80	91 m/z
13	ICP-OES-AV	Y371 / Te214			100	193.696 nm
14	ICP-MS	45	ORS	He	50	75
15	ICP-MS	Ge	UC	He	625	75
16	ICP-MS	72	ORS	He	200	75

Table 76 Instrument Conditions for B in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						
2						
3						
4	ICP-OES-AV-buffer	Lu			100	249.678
5	ICP-MS	Ge 72	ORS		200	11m/z
6	ICP-MS	Scandium	ORS		81.6327	11
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	11
8	NA	NA	NA	NA	NA	NA
9	ICP-MS/MS	none				11
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Sc	KED	He	481.7	10
12	ICP-MS/MS	Sc 45	ORS	NA	80	11 m/z
13						
14	ICP-MS	45			50	11
15	ICP-MS	Sc	NA	NA	625	10
16						

Table 77 Instrument Conditions for Ba in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS					
2						
3						
4	ICP-OES-AV-buffer	Lu			100	233.527
5	ICP-MS	Rh 103	ORS		200	137m/z
6	ICP-MS	Indium	ORS	He	81.6327	137
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	134
8	NA	NA	NA	NA	NA	NA
9	ICP-MS/MS	none		He	1700	137
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Rh	KED	He	481.7	137
12	ICP-MS/MS	Rh 103	ORS	O2	80	153 m/z
13	ICP-OES-AV	Y371 / Te214			100	585.367 nm
14	ICP-MS	193	ORS	He	50	137
15	ICP-MS	Rh	NA	NA	625	138
16	ICP-MS	Tb	ORS	He	200	137

Table 78 Instrument Conditions for Ca in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV					
2						
3	ICP-OES-RV	Sc				317.933
4	ICP-OES-AV-buffer	Lu			100	
5	ICP-OES-RV	Y324	NA		200	396.847nm
6	ICP-OES-RV	Lutetium			81.6327	317.94
7	ICP-OES-AV	Y	N/A	N/A	20	315.887
8	NA	NA	NA	NA	NA	NA
9	ICP-OES-RV	none			1700	315.887
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-RV	N/A	NA	NA	96.34	184
12	ICP-OES-AV	Eu290.667			80	370.602
13	ICP-OES-AV	Y371 / Te214			100	315.887 nm
14	ICP-MS	45	ORS	He	50	44
15	ICP-MS	Sc	UC	He	625	44
16	ICP-MS	Sc	ORS	H2	1000	40

Table 79 Instrument Conditions for Cd in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS					
2	ICP-MS	Rh		He	39.86	111
3	ICP-MS	Rh103				111
4	ICP-OES-AV-buffer	Lu			100	214.439
5	ICP-MS	Rh 103	ORS		200	111m/z
6	ICP-MS	Indium	ORS	He	81.6327	111
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	111
8	NA	NA	NA	NA	NA	NA
9	ICP-MS/MS	none		He	1700	111
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Rh	KED	He	481.7	111
12	ICP-MS/MS	Rh 103	ORS	O2	80	111 m/z
13	ICP-OES-AV	Y371 / Te214			100	214.439 nm
14	ICP-MS	103	ORS	He	50	111
15	ICP-MS	Rh	NA	NA	625	111
16	ICP-MS	Rh	ORS	He	200	111

Table 80 Instrument Conditions for Co in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS					
2	ICP-MS	Rh		He	39.86	59
3	ICP-MS	Sc45	KED	He		59
4	ICP-OES-AV-buffer	Lu			100	228.615
5	ICP-MS	Ge 72	ORS		200	59m/z
6	ICP-MS	Scandium	ORS	He	81.6327	59
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	59
8	NA	NA	NA	NA	NA	NA
9	ICP-MS/MS	none		He	1700	59
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Ga	KED	He	481.7	59
12	ICP-MS/MS	Rh 103	ORS	O2	80	59 m/z
13	ICP-OES-AV	Y371 / Te214			100	228.615 nm
14	ICP-MS	45	ORS	He	50	59
15	ICP-MS	Ge	UC	He	625	59
16	ICP-MS	Ge	ORS	He	200	59

Table 81 Instrument Conditions for Cr in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS					
2	ICP-MS	Rh		He	39.86	52
3	ICP-MS	Sc45	KED	He		52
4	ICP-OES-AV-buffer	Lu			100	267.716
5	ICP-MS	Ge 72	ORS		4000	52m/z
6	ICP-MS	Scandium	ORS	He	81.6327	52
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	52
8	NA	NA	NA	NA	NA	NA
9						
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Sc	KED	He	481.7	52
12	ICP-MS/MS	Rh 103	ORS	O2	80	52 m/z
13	ICP-OES-AV	Y371 / Te214			100	267.716 nm
14	ICP-MS	45	ORS	He	50	52
15	ICP-MS	Sc	UC	He	625	52
16	ICP-MS	Ge	ORS	He	200	52

Table 82 Instrument Conditions for Cu in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						
2	ICP-MS	Rh		He	39.86	63
3	ICP-OES-AV	Sc				327.393
4	ICP-OES-AV-buffer	Lu			100	327.395
5	ICP-MS	Ge 72	ORS		200	63m/z
6	ICP-MS	Scandium	ORS	He	81.6327	63
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	65
8	NA	NA	NA	NA	NA	NA
9	ICP-MS/MS	none		He	1700	63
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Ga	KED	He	481.7	63
12	ICP-MS/MS	Rh 103	ORS	O2	80	63 m/z
13	ICP-OES-AV	Y371 / Te214			100	324.754 nm
14	ICP-MS	45	ORS	He	50	63
15	ICP-MS	Ge	UC	He	625	63
16	ICP-MS	Rh	ORS	He	1000	63

Table 83 Instrument Conditions for Fe in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						
2	ICP-MS	Rh		HEHe	39.86	56
3	ICP-OES-AV	Sc				238.204
4	ICP-OES-AV-buffer	Lu			100	238.204
5	ICP-MS	Ge 72	ORS		4000	56m/z
6	ICP-MS	Scandium	ORS	He	81.6327	56
7	ICP-OES-AV	Y	N/A	N/A	2	238.204
8	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	none			1700	238.204
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-RV	N/A	NA	NA	96.34	240
12	ICP-MS/MS	Rh 103	ORS	O2	80	56 m/z
13	ICP-OES-AV	Y371 / Te214			100	240.489 nm
14	ICP-MS	45	ORS	He	50	56
15	ICP-MS	Sc	UC	He	625	56
16	ICP-MS	Rh	ORS	H2	200	56

Table 84 Instrument Conditions for Hg in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						
2	ICP-MS	Ir		He	39.86	201
3	ICP-MS	Ir193				202
4	VGA-ICP-OES				100	194.164
5	ICP-MS	Ir 193	ORS		200	202m/z
6	ICP-MS	Indium	ORS	He	81.6327	201
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	202
8	NA	NA	NA	NA	NA	NA
9	ICP-MS/MS	none			1700	202
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Tb	KED	He	481.7	201
12	ICP-MS/MS	Ir 193	ORS	O2	80	202 m/z
13	CVAAS				100	253.7 nm
14	CVAAS				50	253.7
15	ICP-MS	Ir	NA	NA	625	201
16	ICP-MS	Ir			200	201

Table 85 Instrument Conditions for K in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV					
2	ICP-MS	Rh		He	39.86	39
3	ICP-OES-RV	Sc				766.49
4						
5	ICP-OES-RV	Y324	NA		200	766.49nm
6	ICP-OES-RV	Lutetium			816.327	766.526
7	ICP-OES-AV	Y	N/A	N/A	50	766.491
8	NA	NA	NA	NA	NA	NA
9	ICP-OES-RV	none			1700	766.491
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-RV	N/A	NA	NA	96.34	766
12	ICP-OES-AV	Cs697.327			80	766.491
13	ICP-OES-AV	Y371 / Te214			1000	766.491 nm
14	ICP-MS	45	ORS	He	50	39
15	ICP-MS	Sc	UC	He	625	39
16	ICP-MS	Sc	ORS	He	1000	39

Table 86 Instrument Conditions for La in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						
2						
3						
4						
5	ICP-MS	Rh 103	ORS		200	139m/z
6						
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	139
8	NA	NA	NA	NA	NA	NA
9	ICP-MS/MS	none			1700	
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Rh	KED	He	481.7	139
12	NT	NT	NT	NT	NT	NT
13						
14	ICP-MS	193	ORS	He	50	139
15						
16	ICP-MS	Tb	ORS	He	200	139

Table 87 Instrument Conditions for Li in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS					
2						
3						
4	ICP-OES-AV-buffer	Lu			100	670.783
5	ICP-MS	Ge 72	ORS		200	7m/z
6	ICP-MS	Scandium	ORS		81.6327	7
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	7
8	NA	NA	NA	NA	NA	NA
9	ICP-MS/MS	none			1700	7
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Sc	KED	He	481.7	7
12	ICP-MS/MS	Sc 45	ORS	NA	80	7 m/z
13						
14	ICP-MS	45			50	7
15	ICP-MS	Sc	NA	NA	625	7
16	ICP-MS	Sc	ORS	H2	200	7

Table 88 Instrument Conditions for Mg in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV					
2	ICP-MS	Sc		He	398.6	24
3	ICP-OES-RV	Sc				285.213
4	ICP-OES-AV-buffer	Lu			100	279.078
5	ICP-OES-RV	Y324	NA		200	285.213nm
6	ICP-OES-RV	Lutetium			81.6327	285.213
7	ICP-OES-AV	Y	N/A	N/A	20	279.8
8	NA	NA	NA	NA	NA	NA
9	ICP-OES-RV	none			1700	279.553
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-RV	N/A	NA	NA	96.34	285
12	ICP-OES-AV	Eu390.711			80	383.829
13	ICP-OES-AV	Y371 / Te214			100	280.27 nm
14	ICP-MS	45	ORS	He	50	25
15	ICP-MS	Sc	UC	He	625	25
16	ICP-MS	Y	ORS	H2	200	24

Table 89 Instrument Conditions for Mn in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						
2	ICP-MS	Sc		He	39.86	55
3	ICP-OES-AV	Sc				257.61
4	ICP-OES-AV-buffer	Lu			100	257.61
5	ICP-MS	Ge 72	ORS		4000	55m/z
6	ICP-MS	Scandium	ORS	He	81.6327	55
7	ICP-OES-AV	Y	N/A	N/A	2	257.61
8	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	none			1700	257.61
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Sc	KED	He	481.7	55
12	ICP-MS/MS	Rh 103	ORS	O2	80	55 m/z
13	ICP-OES-AV	Y371 / Te214			100	257.61 nm
14	ICP-MS	45	ORS	He	50	55
15	ICP-MS	Sc	UC	He	625	55
16	ICP-MS	Ge			200	55

Table 90 Instrument Conditions for Mo in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						
2						
3	ICP-MS	Rh103				98
4						
5	ICP-MS	Ge 72	ORS		200	95m/z
6	ICP-MS	Indium	ORS	He	81.6327	95
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	95
8	NA	NA	NA	NA	NA	NA
9						
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Rh	KED	He	481.7	98
12	ICP-MS/MS	Rh 103	ORS	O2	80	95 m/z
13	ICP-OES-AV	Y371 / Te214			100	202.032 nm
14	ICP-MS	103	ORS	He	50	95
15	ICP-MS	Rh	NA	NA	625	95
16	ICP-MS	Y	ORS	He	200	95

Table 91 Instrument Conditions for Na in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV					
2	ICP-MS	Sc		He	398.6	23
3	ICP-OES-RV	Sc				589.592
4	ICP-OES-AV-buffer	Lu			100	589.592
5	ICP-OES-RV	Y324	NA		200	589.592nm
6	ICP-OES-RV	Lutetium			81.6327	589.61
7	ICP-OES-AV	Y	N/A	N/A	50	589.592
8	NA	NA	NA	NA	NA	NA
9						
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-RV	N/A	NA	NA	96.34	590
12	ICP-OES-AV	N/A			80	589.592
13	ICP-OES-AV	Y371 / Te214			100	588.995 nm
14	ICP-MS	45	ORS	He	50	23
15	ICP-MS	Sc	UC	He	625	23
16	ICP-MS	Sc	ORS	H2	200	23

Table 92 Instrument Conditions for Ni in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS					
2	ICP-MS	Rh		He	39.86	60
3	ICP-MS	Sc45	KED	He		60
4	ICP-OES-AV-buffer	Lu			100	227.021
5	ICP-MS	Ge 72	ORS		200	60m/z
6	ICP-MS	Scandium	ORS	He	81.6327	60
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	60
8	NA	NA	NA	NA	NA	NA
9						
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Ga	KED	He	481.7	60
12	ICP-MS/MS	Rh 103	ORS	O2	80	60 m/z
13	ICP-OES-AV	Y371 / Te214			100	216.55 nm
14	ICP-MS	45	ORS	He	50	60
15	ICP-MS	Sc	UC	He	625	60
16	ICP-MS	Rh	ORS	He	200	60

Table 93 Instrument Conditions for P in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						
2						
3	ICP-OES-RV	Sc				178.221
4	ICP-OES-AV-buffer	Lu			500	213.618
5	ICP-OES-RV	Te214	NA		200	185.942nm
6	ICP-OES-RV	Lutetium			816.327	178.226
7	ICP-OES-AV	Y	N/A	N/A	50	213.618
8	NA	NA	NA	NA	NA	NA
9						
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-RV	N/A	NA	NA	96.34	186
12	ICP-OES-AV	N/A			80	185.878
13	ICP-OES-AV	Y371 / Te214			100	213.618 nm
14	ICP-OES-AV	Eu			50	185.8
15	ICP-MS	Sc	UC	He	625	31
16	ICP-MS	Ge	ORS	He	1000	31

Table 94 Instrument Conditions for Pb in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS					
2	ICP-MS	Ir			39.86	208
3	ICP-MS	Ir193				206+207+208
4	ICP-MS	193	Collision	He	100	208
5	ICP-MS	Ir 193	ORS		200	208m/z
6	ICP-MS	Lutetium	ORS	He	81.6327	208
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	208
8	NA	NA	NA	NA	NA	NA
9	ICP-MS/MS	none			1700	208
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Tb	KED	He	481.7	206+207+208
12	ICP-MS/MS	Ir 193	ORS	O2	80	208 m/z
13	ICP-OES-AV	Y371 / Te214			100	220.353 nm
14	ICP-MS	193	ORS	He	50	208
15	ICP-MS	Ir	NA	NA	625	206+207+208
16	ICP-MS	Tb			200	206+207+208

Table 95 Instrument Conditions for Rb in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						
2						
3						
4						
5	ICP-MS	Rh 103	ORS		200	85m/z
6						
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	85
8	NA	NA	NA	NA	NA	NA
9						
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Rh	KED	He	481.7	85
12	NT	NT	NT	NT	NT	NT
13						
14	ICP-MS	103	ORS	He	50	85
15						
16	ICP-MS	Y	ORS	He	200	85

Table 96 Instrument Conditions for S in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV					
2						
3	ICP-OES-RV	Sc				181.975
4	ICP-OES-AV-buffer	Lu			500	181.972
5	ICP-OES-RV	Te214	NA		4000	180.669nm
6	ICP-OES-RV	Lutetium			816.327	180.673
7	ICP-OES-AV	Y	N/A	N/A	50	181.972
8	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	none				181.972
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-RV	N/A	NA	NA	96.34	182
12	ICP-OES-AV	N/A			80	181.972 nm
13	ICP-OES-AV	Y371 / Te214			1000	180.669 nm
14						
15	ICP-OES-AV	Y	NA	NA	62.5	181.975
16	ICPMS_MS	Y	ORS	O2	100000	48

Table 97 Instrument Conditions for Sb in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS					
2	ICP-MS	Rh			39.86	121
3	ICP-MS	Rh103				121
4	ICP-OES-AV-buffer	Lu			100	206.834
5	ICP-MS	Rh 103	ORS		200	121m/z
6	ICP-MS	Indium	ORS	He	81.6327	121
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	121
8	NA	NA	NA	NA	NA	NA
9						
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Rh	KED	He	481.7	121
12	ICP-MS/MS	Rh 103	ORS	O2	80	121 m/z
13						
14	ICP-MS	103	ORS	He	50	121
15	ICP-MS	Rh	NA	NA	625	121
16	ICP-MS	Ir			200	121

Table 98 Instrument Conditions for Se in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS					
2	ICP-MS	Te		HEHe	78.72	78
3	ICP-MS	Te125	DRC	CH4		80
4	ICP-MS	103	Collision	He	100	78
5	ICP-MS	Rh 103	ORS		4000	78m/z
6	ICP-MS	Rhodium	ORS	He	81.6327	78
7	ICP-MS	Sc, Rh, Ir	ORS	HEHe	2	78
8	NA	NA	NA	NA	NA	NA
9	ICP-MS/MS	none		H2	1700	78
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Te	KED	He	99.6	82
12	ICP-MS/MS	Rh 103	ORS	O2	80	94 m/z
13	ICP-OES-AV	Y371 / Te214			100	196.026 nm
14	ICP-MS	45	ORS	He	50	78
15	ICP-MS	Rh	NA	NA	625	82
16	ICP-MS	Ge	ORS	H2	1000	78

Table 99 Instrument Conditions for Sn in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						
2	ICP-MS	Te		He	39.86	118
3	ICP-MS	Rh103				120
4	ICP-OES-AV-buffer	Lu			100	189.925
5	ICP-MS	Rh 103	ORS		200	118m/z
6	ICP-MS	Indium	ORS	He	81.6327	118
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	118
8	NA	NA	NA	NA	NA	NA
9						
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Rh	KED	He	481.7	120
12	ICP-MS/MS	Rh 103	ORS	O2	80	134 m/z
13						
14	ICP-MS	103	ORS	He	50	118
15	ICP-MS	Rh	NA	NA	625	118
16	ICP-MS	Rh	ORS	He	200	118

Table 100 Instrument Conditions for U in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-MS					
2						
3						
4	ICP-MS	193	Collision	He	100	238
5	ICP-MS	Ir 193	ORS		200	238m/z
6	ICP-MS	Lutetium	ORS	He	81.6327	238
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	238
8	NA	NA	NA	NA	NA	NA
9	ICP-MS/MS	none			1700	238
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Tb	KED	He	481.7	238
12	ICP-MS/MS	Ir 193	ORS	HEHe	80	238 m/z
13						
14	ICP-MS	193	ORS	He	50	238
15	ICP-MS	Ir	NA	NA	625	238
16	ICP-MS	Tb			200	238

Table 101 Instrument Conditions for V in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1						
2						
3						
4	ICP-OES-AV-buffer	Lu			100	292.401
5	ICP-MS	Ge 72	ORS		200	51m/z
6	ICP-MS	Scandium	ORS	He	81.6327	51
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	51
8	NA	NA	NA	NA	NA	NA
9						
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Sc	KED	He	481.7	51
12	ICP-MS/MS	Rh 103	ORS	O2	80	67 m/z
13						
14	ICP-MS	45	ORS	He	50	51
15	ICP-MS	Sc	UC	He	625	51
16	ICP-MS	Ge	ORS	He	200	51

Table 102 Instrument Conditions for Zn in S1

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	ICP-OES-AV					
2	ICP-MS	Rh		He	398.6	66
3	ICP-OES-AV	Sc				206.2
4	ICP-OES-AV-buffer	Lu			100	213.857
5	ICP-MS	Ge 72	ORS		4000	66m/z
6	ICP-MS	Scandium	ORS	He	81.6327	66
7	ICP-OES-AV	Y	N/A	N/A	2	213.857
8	NA	NA	NA	NA	NA	NA
9	ICP-OES-AV	none			1700	213.857
10	NA	NA	NA	NA	NA	NA
11	ICP-MS	Te	KED	He	481.7	66
12	ICP-MS/MS	Rh 103	ORS	O2	80	66 m/z
13	ICP-OES-AV	Y371 / Te214			100	206.2 nm
14	ICP-MS	45	ORS	He	50	66
15	ICP-MS	Ge	UC	He	625	66
16	ICP-MS	Ge	ORS	He	200	66

Table 103 Instrument Conditions for Ag in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Rh			39.55	107
3	ICP-MS	Rh103				109
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Ge 72	ORS		167	107m/z
6	ICP-MS	Indium	ORS	He	81.6327	107
7	N/A	N/A	N/A	N/A	N/A	N/A
8	ICP-MS	In	ORS		1440	107
9	ICP-MS/MS	none			1700	107
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	108 m/z
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rh	NA	NA	625	109
16	NA	NA	NA	NA	NA	NA

Table 104 Instrument Conditions for Al in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Rh			39.55	27
3						
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Ge 72	ORS		3340	27m/z
6	ICP-MS	Scandium	ORS	He	81.6327	27
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	27
8	ICP-MS	Ge	ORS		1440	27
9					1700	
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Sc 45	ORS	NA	80	27 m/z
13	ICP-OES-AV	Y371 / Te214			50	396.152 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Sc	UC	He	625	27
16	NA	NA	NA	NA	NA	NA

Table 105 Instrument Conditions for As in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Te		HEHe	39.55	75
3	ICP-MS	Te125	KED	He		75
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Ge 72	ORS		167	75m/z
6	ICP-MS	Indium	ORS	He	81.6327	75
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	75
8	ICP-MS	Ge	ORS	He	1440	75
9						
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	91 m/z
13	ICP-OES-AV	Y371 / Te214			50	193.696 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Ge	UC	He	625	75
16	NA	NA	NA	NA	NA	NA

Table 106 Instrument Conditions for B in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2						
3						
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Ge 72	ORS		3340	11m/z
6	ICP-MS	Scandium	ORS		81.6327	11
7	N/A	N/A	N/A	N/A	N/A	N/A
8	ICP-MS	Ge	ORS		1440	11
9	ICP-MS/MS	none		He	1700	11
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Sc 45	ORS	NA	80	11 m/z
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Sc	NA	NA	625	9
16	NA	NA	NA	NA	NA	NA

Table 107 Instrument Conditions for Ba in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2						
3						
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Rh 103	ORS		167	137m/z
6	ICP-MS	Indium	ORS	He	81.6327	137
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	134
8	ICP-MS	Lu	ORS		1440	135
9	ICP-MS/MS	none		He	1700	137
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	153 m/z
13	ICP-OES-AV	Y371 / Te214			50	585.367 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rh	NA	NA	625	138
16	NA	NA	NA	NA	NA	NA

Table 108 Instrument Conditions for Ca in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Sc			395.5	44
3	ICP-OES-RV	Sc				317.933
4	NA	NA	NA	NA	NA	NA
5	ICP-OES-RV	Y324	NA		167	396.847nm
6	ICP-OES-RV	Lutetium			81.6327	317.94
7	ICP-OES-AV	Y	N/A	N/A	10	315.887
8	ICP-OES-AV	Lu			72	315.887
9	ICP-OES-RV				1700	315.887
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-OES-AV	Eu290.667			80	370.602nm
13	ICP-OES-AV	Y371 / Te214			50	315.887 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Sc	UC	He	625	44
16	NA	NA	NA	NA	NA	NA

Table 109 Instrument Conditions for Cd in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Rh		He	39.55	111
3	ICP-MS	Rh103				111
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Rh 103	ORS		167	111m/z
6	ICP-MS	Indium	ORS	He	81.6327	111
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	111
8	ICP-MS	Rh	ORS	He	1440	111
9	ICP-MS/MS	none		He	1700	111
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	111 m/z
13	ICP-OES-AV	Y371 / Te214			50	214.439 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rh	NA	NA	625	111
16	NA	NA	NA	NA	NA	NA

Table 110 Instrument Conditions for Co in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Rh		He	39.55	59
3	ICP-MS	Sc45	KED	He		59
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Ge 72	ORS		167	59m/z
6	ICP-MS	Scandium	ORS	He	81.6327	59
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	59
8	ICP-MS	Ge	ORS	He	1440	59
9	ICP-MS/MS	none		He	1700	59
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	59 m/z
13	ICP-OES-AV	Y371 / Te214			50	228.615 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Ge	UC	He	625	59
16	NA	NA	NA	NA	NA	NA

Table 111 Instrument Conditions for Cr in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Rh		He	39.55	52
3	ICP-MS	Sc45	KED	He		52
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Ge 72	ORS		167	52m/z
6	ICP-MS	Scandium	ORS	He	81.6327	52
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	52
8	ICP-MS	Ge	ORS	He	1440	52
9						
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	52 m/z
13	ICP-OES-AV	Y371 / Te214			50	267.716 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Sc	UC	He	625	52
16	NA	NA	NA	NA	NA	NA

Table 112 Instrument Conditions for Cs in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2						
3						
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Rh 103	ORS		167	133m/z
6						
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	52
8	ICP-MS	Ge	ORS		1440	133
9						
10						
11	NA	NA	NA	NA	NA	NA
12	NT	NT	NT	NT	NT	NT
13						
14	NA	NA	NA	NA	NA	NA
15						
16	NA	NA	NA	NA	NA	NA

Table 113 Instrument Conditions for Cu in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Rh		He	39.55	63
3	ICP-OES-AV	Sc				327.393
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Ge 72	ORS		3340	63m/z
6	ICP-MS	Scandium	ORS	He	81.6327	63
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	65
8	ICP-MS	Ge	ORS	He	1440	63
9	ICP-MS/MS	none		He	1700	63
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	63 m/z
13	ICP-OES-AV	Y371 / Te214			50	324.754 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Ge	UC	He	625	63
16	NA	NA	NA	NA	NA	NA

Table 114 Instrument Conditions for Fe in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Rh		HEHe	39.55	56
3	ICP-OES-AV	Sc				238.204
4	NA	NA	NA	NA	NA	NA
5	ICP-OES-AV	Y324	NA		167	259.94nm
6	ICP-MS	Scandium	ORS	He	81.6327	56
7	ICP-OES-AV	Y	N/A	N/A	2	238.204
8	ICP-MS	Ge	ORS	H2	1440	56
9	ICP-OES-AV	none			1700	238.204
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	56 m/z
13	ICP-OES-AV	Y371 / Te214			50	240.489 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Sc	UC	He	625	56
16	NA	NA	NA	NA	NA	NA

Table 115 Instrument Conditions for K in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Rh		He	395.5	39
3	ICP-OES-RV	Sc				766.49
4	NA	NA	NA	NA	NA	NA
5	ICP-OES-RV	Y324	NA		167	766.49nm
6	ICP-OES-RV	Lutetium			81.6327	766.526
7	ICP-OES-AV	Y	N/A	N/A	100	766.491
8	ICP-OES-AV	Cs			72	769.897
9	ICP-OES-RV	none			1700	766.491
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-OES-AV	Cs697.327			80	766.491nm
13	ICP-OES-AV	Y371 / Te214			500	766.491 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Sc	UC	He	625	39
16	NA	NA	NA	NA	NA	NA

Table 116 Instrument Conditions for Mg in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Sc		He	395.5	24
3	ICP-OES-RV	Sc				285.213
4	NA	NA	NA	NA	NA	NA
5	ICP-OES-RV	Y324	NA		167	285.213nm
6	ICP-OES-RV	Lutetium			81.6327	285.213
7	ICP-OES-AV	Y	N/A	N/A	10	279.8
8	ICP-OES-AV	Lu			72	279.8
9	ICP-OES-RV	none			1700	279.078
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-OES-AV	Eu390.711	ORS	O2	80	383.829nm
13	ICP-OES-AV	Y371 / Te214			50	280.27 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Sc	UC	He	625	25
16	NA	NA	NA	NA	NA	NA

Table 117 Instrument Conditions for Mn in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Sc		He	39.55	55
3	ICP-OES-AV	Sc				257.61
4	NA	NA	NA	NA	NA	NA
5	ICP-OES-RV	Y324	NA		167	257.61nm
6	ICP-MS	Scandium	ORS	He	81.6327	55
7	ICP-OES-AV	Y	N/A	N/A	2	257.61
8	ICP-MS	Ge	ORS	He	1440	55
9	ICP-OES-AV	none			1700	257.61
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	55 m/z
13	ICP-OES-AV	Y371 / Te214			50	257.61 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Sc	UC	He	625	55
16	NA	NA	NA	NA	NA	NA

Table 118 Instrument Conditions for Mo in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2						
3	ICP-MS	Rh103				98
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Ge 72	ORS		167	95m/z
6	ICP-MS	Indium	ORS	He	81.6327	95
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	95
8	ICP-MS	Rh	ORS	He	1440	95
9						
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	95 m/z
13	ICP-OES-AV	Y371 / Te214			50	202.032 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rh	NA	NA	625	95
16	NA	NA	NA	NA	NA	NA

Table 119 Instrument Conditions for Na in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Sc		He	39.55	23
3	ICP-OES-RV	Sc				589.592
4	NA	NA	NA	NA	NA	NA
5	ICP-OES-RV	Y324	NA		167	589.529nm
6	ICP-OES-RV	Lutetium			81.6327	589.61
7	ICP-OES-AV	Y	N/A	N/A	2	589.592
8	ICP-OES-AV	Lu			72	588.995
9						
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-OES-AV	N/A			80	589.592nm
13	ICP-OES-AV	Y371 / Te214			50	588.995 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Sc	UC	He	625	23
16	NA	NA	NA	NA	NA	NA

Table 120 Instrument Conditions for Ni in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Rh		He	39.55	60
3	ICP-MS	Sc45	KED	He		60
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Ge 72	ORS		167	60m/z
6	ICP-MS	Scandium	ORS	He	81.6327	60
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	60
8	ICP-MS	Ge	ORS	He	1440	60
9						
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	60 m/z
13	ICP-OES-AV	Y371 / Te214			50	216.55 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Ge	UC	He	625	60
16	NA	NA	NA	NA	NA	NA

Table 121 Instrument Conditions for P in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2						
3	ICP-OES-RV	Sc				178.221
4	NA	NA	NA	NA	NA	NA
5	ICP-OES-RV	Te214	NA		167	185.942nm
6	ICP-OES-RV	Lutetium			816.327	178.226
7	ICP-OES-AV	Y	N/A	N/A	100	213.618
8	ICP-OES-AV	Lu			72	213.618
9						
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-OES-AV	N/A			80	185.878nm
13	ICP-OES-AV	Y371 / Te214			500	213.618 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Sc	UC	He	625	31
16	NA	NA	NA	NA	NA	NA

Table 122 Instrument Conditions for Pb in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Ir			39.55	208
3	ICP-MS	Ir193				206+207+208
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Ir 193	ORS		167	208m/z
6	ICP-MS	Lutetium	ORS	He	81.6327	208
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	208
8	ICP-MS	Lu	ORS		1440	208
9	ICP-MS/MS	none			1700	208
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Ir 193	ORS	O2	80	208 m/z
13	ICP-OES-AV	Y371 / Te214			50	220.353 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Ir	NA	NA	625	206+207+208
16	NA	NA	NA	NA	NA	NA

Table 123 Instrument Conditions for S in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2						
3	ICP-OES-RV	Sc				181.975
4	NA	NA	NA	NA	NA	NA
5	ICP-OES-RV	Te214	NA		167	180.669nm
6	ICP-OES-RV	Lutetium			816.327	180.673
7	ICP-OES-AV	Y	N/A	N/A	10	181.972
8	ICP-OES-AV	Lu			72	181.972
9						
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-OES-AV	N/A			80	181.972 nm
13	ICP-OES-AV	Y371 / Te214			500	180.669 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-OES-AV	Y	NA	NA	62.5	181.975
16	NA	NA	NA	NA	NA	NA

Table 124 Instrument Conditions for Sb in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Rh			39.55	121
3	ICP-MS	Rh103				121
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Rh 103	ORS		167	121m/z
6	ICP-MS	Indium	ORS	He	81.6327	121
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	121
8	ICP-MS	In	ORS		1440	121
9						
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	121 m/z
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rh	NA	NA	625	121
16	NA	NA	NA	NA	NA	NA

Table 125 Instrument Conditions for Se in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Te		HEHe	79.19	78
3	ICP-MS	Te125	DRC	CH4		80
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Rh 103	ORS		167	78m/z
6	ICP-MS	Rhodium	ORS	He	81.6327	78
7	ICP-MS	Sc, Rh, Ir	ORS	HEHe	2	78
8	ICP-MS	Ge	ORS	H2	1440	78
9	ICP-MS/MS	none		H2	1700	78
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	94 m/z
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rh	NA	NA	625	82
16	NA	NA	NA	NA	NA	NA

Table 126 Instrument Conditions for Sr in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2						
3						
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Ge 72	ORS		3340	88m/z
6	ICP-MS	Indium	ORS	He	81.6327	88
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	88
8	ICP-MS	Ge	ORS		1440	88
9						
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	88 m/z
13	ICP-OES-AV	Y371 / Te214			100	421.552 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rh	NA	NA	625	88
16	NA	NA	NA	NA	NA	NA

Table 127 Instrument Conditions for V in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2						
3						
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Ge 72	ORS		167	51m/z
6	ICP-MS	Scandium	ORS	He	81.6327	51
7	ICP-MS	Sc, Rh, Ir	ORS	He	2	51
8	ICP-MS	Ge	ORS	He	1440	51
9						
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	67 m/z
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Sc	UC	He	625	51
16	NA	NA	NA	NA	NA	NA

Table 128 Instrument Conditions for Zn in S2

Laboratory Code	Instrument	Internal standard	Reaction Cell	Reaction Gas	Final Dilution Factor	Wavelength (nm)/ Ion(m/z)/ Absorbance(nm)
1	NA	NA	NA	NA	NA	NA
2	ICP-MS	Rh		He	395.49	66
3	ICP-OES-AV	Sc				206.2
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Ge 72	ORS		3340	66m/z
6	ICP-MS	Scandium	ORS	He	81.6327	66
7	ICP-OES-AV	Y	N/A	N/A	2	213.857
8	ICP-MS	Ge	ORS	He	1440	66
9	ICP-OES-AV	none			1700	213.857
10						
11	NA	NA	NA	NA	NA	NA
12	ICP-MS/MS	Rh 103	ORS	O2	80	66 m/z
13	ICP-OES-AV	Y371 / Te214			50	206.2 nm
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Ge	UC	He	625	66
16	NA	NA	NA	NA	NA	NA

END OF REPORT