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Proficiency Test Report

AQA 18-10

Elements in Food

September 2018

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

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

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Accredited for compliance with ISO/IEC 17043

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SUMMARY

This report presents the results of the proficiency test AQA 18-10 – Inorganic Contaminants and Nutrients in Food. The study focused on the measurement of inorganic As and total: Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, Hg, K, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Se, Sb, Sn, Sr, Th, U, V and Zn in a freeze dried liver sample and of total: Al, As, B, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, P, Pb, S, Se, Zn, total Kjeldahl nitrogen (TKN) and total organic carbon (TOC) in a plant material (wheat).

Measurement of inorganic arsenic was included as a pilot program.

Seventeen laboratories registered to participate and all submitted results.

The assigned values were the robust averages of participants' results, except Cr, Ni and Mo in S1. The associated uncertainties were estimated from the robust standard deviation of the participants' results. For Ni and Mo in S1 the assigned value was a reference value measured using double isotope dilution inductively coupled plasma mass spectrometry. For Cr in S1 the assigned value was an information value measured using the same technique.

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 511 z-scores, 483 (95%) were satisfactory with |z| ≤ 2.

Of 511 E_n-scores, 395 (77%) were satisfactory with |E_n| ≤ 1.

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

Some participants may need to reassess their extraction method since they only recovered a fraction of Al and/or Cr from the samples. A high ratio of HCl/sample size is essential for complete extraction of Cr in food samples when high digestion temperatures cannot be employed. In addition, Al extraction is highly dependent on digestion temperature, and as such, sufficiently high temperatures (typically > 200 °C) should be employed to ensure complete extraction.

Measurement of low level Se in food samples presented analytical difficulty to participating laboratories.

- iii. evaluate the laboratories' methods used in determination of inorganic As in food;

The results reported for inorganic As in S1 were in good agreement with each other and with the homogeneity value.

- iv. evaluate within laboratory reproducibility;

Samples S2 of the present study was distributed as Sample S2 of AQA 13-18. Although the assigned values set for the two samples were not significantly different, in some cases, the results reported were significantly different.

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

Despite differences in matrices and laboratories, performance has remained fairly constant. Most participants were found to use the same instrumental and extraction techniques from one PT study to another, regardless of test sample matrix.

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

Of 556 numerical results, 481 (87%) were reported with an expanded measurement uncertainty.

1 INTRODUCTION

1.1 NMI Proficiency Testing Program

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

Proficiency testing (PT): “is evaluation of participant performance against pre-established criteria by means of interlaboratory comparison.”¹ NMI PT studies target chemical testing in areas of high public significance such as trade, environment and food safety. NMI offers studies in:

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

AQA 18-10 is the twelfth NMI proficiency test on inorganic contaminants and nutrients in food.

1.2 Study Aims

The aims of the study were to:

- compare the performance of participant laboratories and assess their accuracy;
- evaluate the laboratories' methods used in determination of inorganic analytes in food;
- compare the performance of participant laboratories with their past performance;
- evaluate within-laboratory reproducibility; and
- develop the practical application of traceability and measurement uncertainty;

1.3 Study Conduct

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

NMI is accredited by National Association of Testing Authorities, Australia (NATA) to ISO/IEC 17043 as a provider of proficiency testing schemes.

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

2 STUDY INFORMATION

2.1 Selection of Matrices and Inorganic Analytes

The 54 tests studied in the two 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.

2.2 Participation

Seventeen laboratories participated and submitted results.

The timetable of the study was:

Invitation issued: 05 June 2018
Samples dispatched: 25 June 2018
Results due: 20 July 2018
Interim report issued: 24 July 2018

2.3 Test Material Specification

Two samples were provided for analysis:

- Sample S1 was 15 g of freeze dried bovine liver, a reference material previously prepared by NMI; and
- Sample S2 was 20 g of plant material (85% wheat), previously distributed as Sample S2 of proficiency testing AQA 13-18.⁵

2.4 Laboratory Code

All participant laboratories were assigned a confidential code number.

2.5 Sample Preparation, Analysis and Homogeneity Testing

Sample S1 was a reference material and Sample S2 was previously distributed as Sample S2 of PT study AQA 13-18.⁵ Although these samples were formerly tested for homogeneity by NMI a partial homogeneity testing was still conducted. The results from the partial homogeneity testing are reported in this study as homogeneity value. No homogeneity testing was conducted for B in S1 and S, TKN and TOC in S2

Results returned by participants gave no reason to question the homogeneity of the test samples.

The preparation and analysis are described in Appendix 1.

2.6 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.^{5,6} Results of this study gave no reason to question the stability of the test samples.

2.7 Sample Storage, Dispatch and Receipt

The samples were dispatched by courier on 25 June 2018.

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

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

2.8 Instructions to Participants

Participants were instructed as follows:

- Quantitatively analyse the samples using your normal test method.
- Report the results in units of mg/kg on as received basis.
- Report results for:

SAMPLE S1 (liver)		SAMPLE S2 (wheat)	
Test TOTAL	Approximate Conc. Range (as received basis) mg/kg	Test TOTAL	Approximate Conc. Range (as received basis) mg/kg
Ag	0.05 – 2	Al	50 – 1250
Al	0.5 – 20	As	0.05 – 1.25
As	0.05 – 2	B	1 – 75
B	0.5 – 20	Ba	1 – 75
Ba	0.05 – 2	Ca	25000 – 1500000
Be	0.05 – 2	Cd	0.025 – 2
Bi	0.05 – 2	Co	0.2 – 15
Ca	10 – 400	Cr	1 – 75
Cd	0.05 – 2	Cu	1 – 75
Co	0.05 – 2	Fe	50 – 1250
Cr	0.05 – 2	K	750 – 15000
Cu	10 – 400	Mg	750 – 15000
Fe	50 – 2000	Mn	50 – 1250
Hg	0.05 – 2	Mo	0.2 – 15
K	500 – 20000	Na	750 – 15000
Li	0.05 – 2	P	750 – 15000
Mg	50 – 2000	Pb	0.05 – 1.25
Mn	0.5 – 20	S	750 – 15000
Mo	0.5 – 20	Se	0.05 – 1.25
Na	500 – 20000	Zn	50 – 1250
Ni	0.05 – 2	Total Kjeldahl Nitrogen	NA
P	500 – 20000	Total Organic Carbon	NA
Pb	0.05 – 2		
Se	0.5 – 20		
Sb	0.05 – 2		
Sn	0.05 – 2		
Sr	0.05 – 2		
Th	0.05 – 2		
U	0.05 – 2		
V	0.05 – 2		
Zn	50 – 2000		
Inorganic As	0.05 – 2		

NA-Not Available

Report results using the electronic results sheet emailed to you:

- Report results as you would report them to a client. For each analyte in each sample, report the expanded measurement uncertainty associated with your analytical result (e.g. 5.01 ± 0.52 mg/kg).
- Please send us the requested details regarding the test method and the basis of your uncertainty estimate.
- Return the completed results sheet by e-mail by 20 July 2018. Late results cannot be included in the report.

2.9 Interim Report

An interim report was e-mailed to participants on 24 July 2018.

3 PARTICIPANT LABORATORY INFORMATION

3.1 Test Method Summaries

Summaries of test methods are transcribed in Tables 1 to 6.

Table 1 Methodology for Total Elements in S1

Lab. Code	Method Reference	Sample Mass (g)	Digestion Temp. (°C)	Digestion Time (min)	Vol. HNO ₃ (mL)	Vol. HNO ₃ (1:1) (mL)	Vol. HCl (mL)	Vol. H ₂ O ₂ (mL)
1	In-house method. Microwave digestion for trace elements and Hot block digestion for major elements. Analysed by ICP-MS and ICP-OES	0.5	110-160	45	4			
3 ^a	EN 13805:2002	1	210	15		7	0.5	1
5 ^a		1	500	24 h	1			
6 ^a	AACC Official Methods No. 40-70 and 40-71, AOAC 975.03, 985.35	0.4	190	30	5		2	
7	In house reverse aqua regia digestion	1	110	60	5		1.5	
8	In house method	0.99	112.5	120	10		10	
9	In house	0.5	85	60	2.5		0.5	
10 ^a			95	120	7.5		5	2
13	In House S6 - referencing APHA 3125	0.4	120	60	10			
14	U.S.Food and Drug Administration, Elemental Analysis Manual, Section 4.7, Version 1.1, March, 2015.	0.5	220	45	5		1	
16	In-house method	0.54	98	90	3			
17	AOAC 990.10 Ch9.1.08	0.5	85	240	5			

^a Additional information in Table 6

Table 2 Methodology for Inorganic As in S1

Lab. Code	Method Reference	Preparation Method	Reagents used	Determination Technique
1	In-house method; arsenic speciation in seafood samples by HPLC-ICPMS	acid extraction	0.28 M HNO ₃	HPLC-ICP-MS (75 m/z)
3 ^a		distillation	KI and HCL	VGA-AAS (193.7 nm)
9	The Analyst, 1999,124	acid extraction	HCl, Chloroform	ICP-MS (75 m/z)

^a Additional information in Table 6

Table 3 Methodology for Total Elements in S2

Lab. Code	Method Reference	Sample Mass (g)	Digestion Temp. (°C)	Digestion Time (min)	Vol. HNO ₃ (mL)	Vol. HNO ₃ (1:1) (mL)	Vol. HCl (mL)	Vol. H ₂ O ₂ (mL)
1	In-house method. Microwave digestion for trace elements and Hot block digestion for major elements. Analysed by ICP-MS and ICP-OES	0.5	110-160	45	4			
2	FC0074	1	95	120	5			1
3	EN 13805:2002	0.5	210	15		7	0.5	1
4 ^a	USEPA 6020, USEPA 6010B	0.2	95	60	3		1	
5 ^a		1	500	24 h	1			
6 ^a	AACC Official Methods No. 40-70 and 40-71 AOAC 975.03, 985.35	0.4	190	30	5		2 2 (1:1)	
7	In house reverse aqua regia digestion	1	110	60	5		1.5	
8	In house method	0.985	112.5	120	10		10	
9	In house	0.5	85	60	2.5		0.5	
11	USEPA 200.3	0.2	120	240	6		1	2
12		0.5	95-100	120	3			
13	In House S6 - referencing APHA 3125	0.4	120	60	10			
14	U.S.Food and Drug Administration, Elemental Analysis Manual, Section 4.7, Version 1.1, March, 2015.	0.5, 0.2	220	45	5		1	
15 ^a	In-house Method	1	220	30	5		1	
16 ^a	In-house method	0.5	98	90	3			
17	AOAC 990.10 Ch9.1.08	0.5	85	240	5			

^a Additional information in Table 6

Table 4 Methodology for TKN in S2

Lab. Code	Measurement Method	Instrument	Method Reference
4	Kjeldahl Method	Kjeltec 8400 Distillation Titration Unit	AOAC
11		FIA	
12	H ₂ SO ₄ digestion		
13	Dumas combustion	LECO	In House S4a
17		Kjeldahl	AS 2300.1.2.1

Table 5 Methodology for TOC in S2

Lab. Code	Measurement Method	Instrument	Method Reference
11	Combustion	TOC IR Analyser	
12	Combustion at 1200C		
13	Dumas combustion	LECO	In House S15b

3.2 Instruments Used for Measurements

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

3.3 Additional Information

Participants had the option to report additional information for each sample analysed. These are transcribed in Table 6.

Table 6 Additional Information

Lab. Code	Additional Information
3	For Sample S1: Digested to completion by microwave; For Inorganic As: Distillate collected in Di water, analysed on VG using inorganic method APHA 3114B
4	For Sample S2: Microwave step included at 250 watts for 20 min followed by venting for 8 mib on MILESTONE START D
5	For Sample S1: Samples were ashed in a muffle furnace for 24 hours @ 500degrees C. Samples assayed by Atomic Absorption and Atomic Emission; For Sample S2: Samples were ashed in a muffle furnace @ 500 degrees C for 24 hours then dissolved in 1 ml HNO ₃ . Samples assayed by Atomic Absorption and Atomic Emission
6	For Samples S1 and S2: Sample was digested using a microwave digestor. Microwave digestion ramped up to 190°C (with 25 Bar pressure) held for 30mins.
10	For Sample S1: Left to sit in the HNO ₃ overnight before heating.
15	For Sample S2: Ultrawave Digester used
16	For Sample S1: FASS-TIL Winlims assigned Sample ID: W2018001447; For Sample S2: Note, 3 ml of HNO ₃ and 1 ml of HCl gave better recovery results for Al, Fe and P. Hence, these results were reported.

3.4 Basis of Participants' Measurement Uncertainty Estimates

Participants were requested to provide information about the basis of their uncertainty estimates (Table 7).

Table 7 Basis of Uncertainty Estimate

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation		Guide Document for Estimating MU
		Precision ^a	Method Bias ^a	
1	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples - RM	CRM	NATA Technical Note 33
2	Standard deviation of replicate analyses multiplied by 2 or 3			NATA Technical Note 33
3	Top Down - precision and estimates of the method and laboratory bias	Control Samples – CRM Duplicate Analysis	CRM	NMI Uncertainty Course

Table 7 Basis of Uncertainty Estimate (continued)

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation		Guide Document for Estimating MU
		Precision ^a	Method Bias ^a	
4	Bottom Up (ISO/GUM, fish bone/ cause and effect diagram)	Control Samples – CRM Duplicate Analysis Instrument Calibration	CRM Recoveries of SS	ISO/GUM
5	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples Duplicate Analysis Instrument Calibration	CRM Recoveries of SS Instrument Calibration Standard Purity	IANZ Technical Guide
6	Horwitz formula	Control Samples Duplicate Analysis Instrument Calibration	CRM Recoveries of SS	NATA Technical Note 33
7	Top Down - precision and estimates of the method and laboratory bias	Control Samples – CRM Duplicate Analysis	CRM Laboratory bias from PT Recoveries of SS Instrument Calibration Standard Purity Variation in sample moisture content	ISO/GUM
8	Professional judgment	Control Samples – RM Duplicate Analysis Instrument Calibration	CRM Recoveries of SS Instrument Calibration	NATA Technical Note 33
9	Top Down - precision and estimates of the method and laboratory bias	Control Samples – CRM Duplicate Analysis	CRM Laboratory bias from PT studies	NMI Uncertainty Course
10	Professional judgment	Duplicate Analysis Instrument Calibration		NATA Technical Note 33
11		Control Samples – RM Duplicate Analysis		
12	Top Down - precision and estimates of the method and laboratory bias	Control Samples Duplicate Analysis	Instrument Calibration Recoveries of SS	Nordtest Report TR537
13	Top Down - precision and estimates of the method and laboratory bias	Control Samples – RM Duplicate Analysis	Instrument Calibration	Nordtest Report TR537
15	Top Down - precision and estimates of the method and laboratory bias	Control Samples – SS Duplicate Analysis	Recoveries of SS	Eurachem/CITA C Guide
16	Top Down - precision and estimates of the method and laboratory bias	Control Samples Duplicate Analysis Instrument Calibration	CRM Recoveries of SS Standard Purity	NMI Uncertainty Course
17	Top Down - precision and estimates of the method and laboratory bias	Control Samples – SS Duplicate Analysis	Laboratory bias from PT studies	NATA Technical Note 33

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

3.5 Participant Comments on this PT Study or Suggestions for Future Studies

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

No comments were received for this study.

4 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

4.1 Results Summary

Participants' results are listed in Tables 8 to 61 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 55. An example chart with interpretation guide is shown in Figure 1.

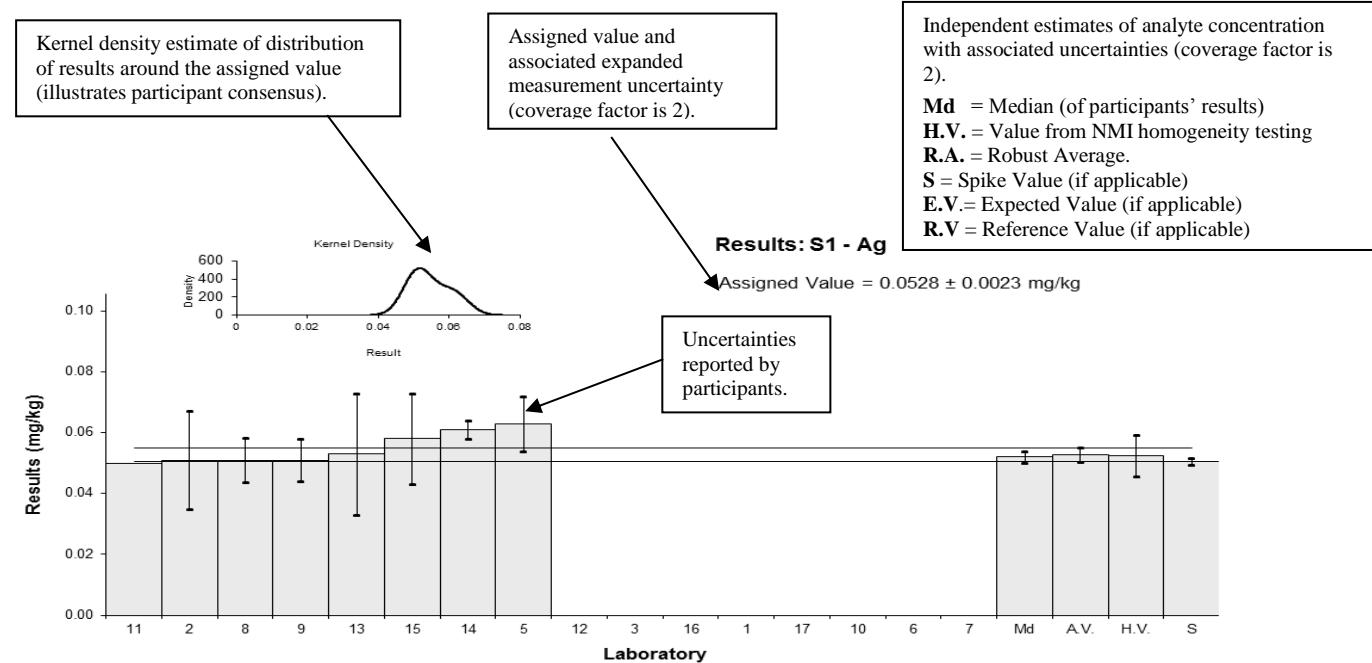


Figure 1 Guide to Presentation of Results

4.2 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 interlaboratory comparisons, ISO13528:2015(E).'⁷

4.3 Robust Between-Laboratory Coefficient of Variation

The robust between-laboratory coefficient of variation (CV) is a measure of the variability of participants' results and was calculated using the procedure described in ISO13528:2015(E).⁷

4.4 Assigned Value

An example of an assigned value calculation using data from the present study is given in Appendix 2. The assigned value is defined as: 'the value attributed to a particular property of a proficiency test item.'¹ In this study the property is the mass fraction of analyte. Assigned values were the robust average of participants' results; the expanded uncertainties were estimated from the associated robust standard deviations. For Ni and Mo in S1 the assigned value was a reference value measured using double isotope dilution inductively coupled plasma mass spectrometry (d-IDMS). For Cr in S1 the assigned value was an information value measured using the same technique.

4.5 Target Standard Deviation

The target standard deviation (σ) is used in the calculation of z-scores and provides scaling for laboratory deviation from the assigned value. It is important to note that the target standard deviation for this study is a fixed value established by the study coordinator and is not the standard deviation of participants' results. The fixed value set for the target standard deviation is based on existing regulation, the acceptance criteria indicated by the methods, the matrix, the concentration level of the analyte and on experience from previous studies. It is backed up by mathematical models such as Thompson - Horwitz equation.⁸

z-Score

An example of a 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 1 below:

$$z = \frac{(\chi - X)}{\sigma} \quad \text{Equation 1}$$

where:

- χ is participant result
- X is the study assigned value
- σ is the target standard deviation

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

- $|z| \leq 2$ is satisfactory;
- $2 < |z| < 3$ is questionable;
- $|z| \geq 3$ is unsatisfactory.

4.6 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 2 below:

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

where:

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

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

- $|E_n| \leq 1$ is satisfactory;
- $|E_n| > 1$ is unsatisfactory.

4.7 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025:2001⁹ 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.¹⁰

5 TABLES AND FIGURES

Table 8

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Ag
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.87	0.08	9.33	5.05
2	NT	NT		
3	0.43	0.16	-0.44	-0.12
4	NT	NT		
5	NR	NR		
6	NT	NT		
7	0.454	0.045	0.09	0.08
8	NT	NT		
9	0.46	0.08	0.22	0.12
10	0.5	0.5	1.11	0.10
11	NT	NT		
12	NT	NT		
13	0.45	0.1	0.00	0.00
14	NT	NT		
15	NT	NT		
16	0.419	0.04	-0.69	-0.67
17	0.45	0.07	0.00	0.00

Statistics

Assigned Value*	0.450	0.023
Spike	Not Spiked	
Homogeneity Value	0.490	0.074
Robust Average	0.460	0.033
Median	0.452	0.019
Mean	0.504	
N	8	
Max.	0.87	
Min.	0.419	
Robust SD	0.025	
Robust CV	5.4%	

*Robust Average excluding Laboratory 1.

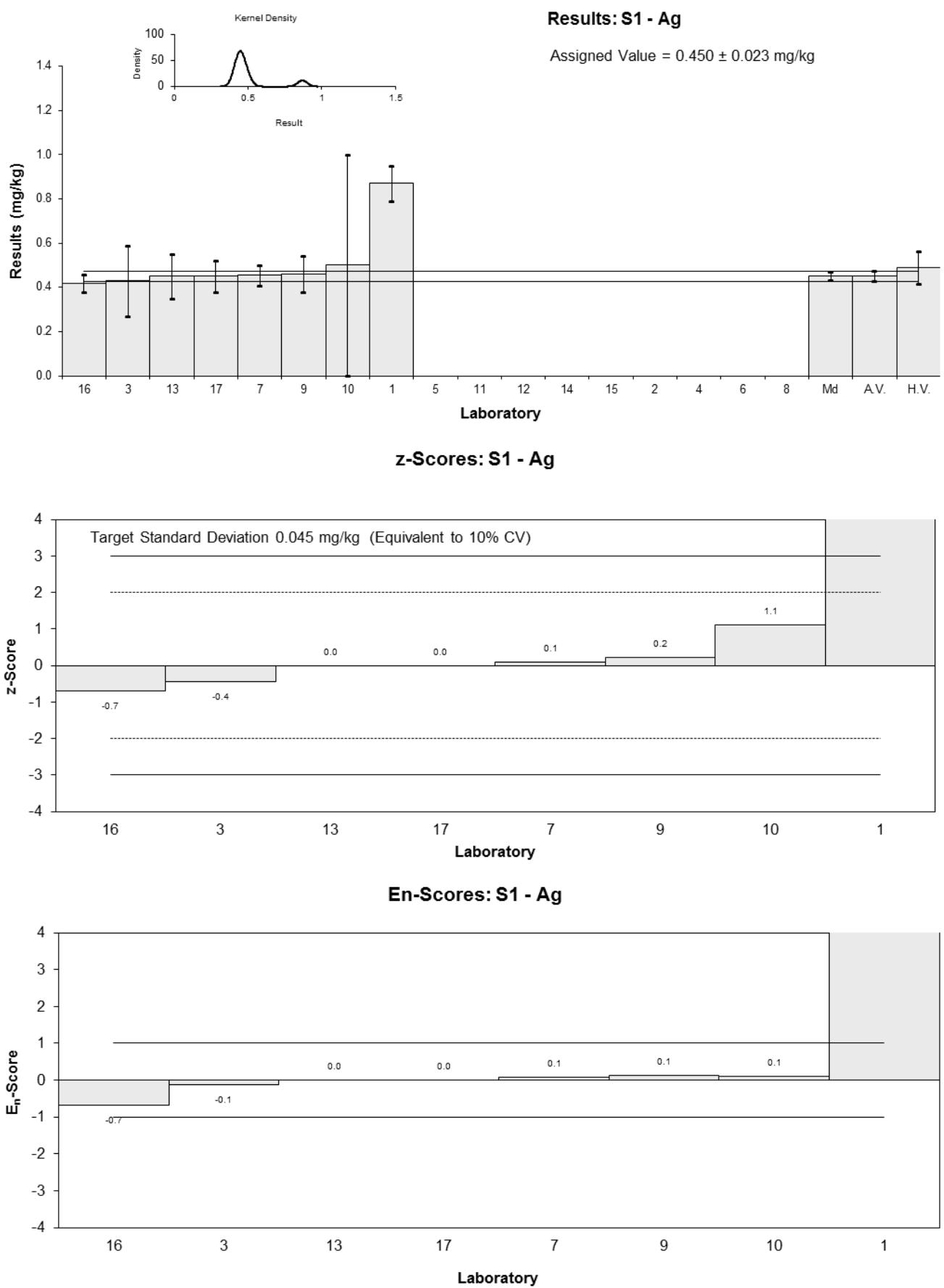


Figure 2

Table 9

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	AI
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	3.4	0.2
2	NT	NT
3	4.2	2
4	NT	NT
5	NR	NR
6	NT	NT
7	3.49	0.349
8	NT	NT
9	3.0	0.6
10	4	4
11	NT	NT
12	NT	NT
13	<10	NR
14	5.811	NR
15	NT	NT
16	3.979	0.16
17	2.78	0.42

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	5.30	1.06
Robust Average	3.72	0.69
Median	3.73	0.50
Mean	3.83	
N	8	
Max.	5.81	
Min.	2.78	
Robust SD	0.78	
Robust CV	21%	

Results: S1 - Al

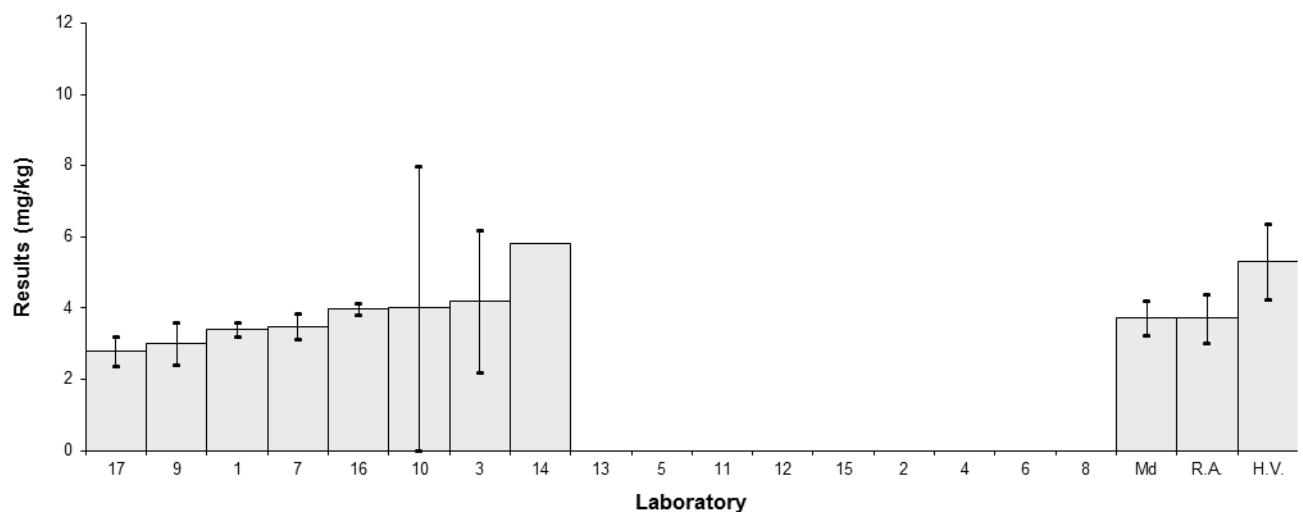


Figure 3

Table 10

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	As
Units	mg/kg

Participant Results

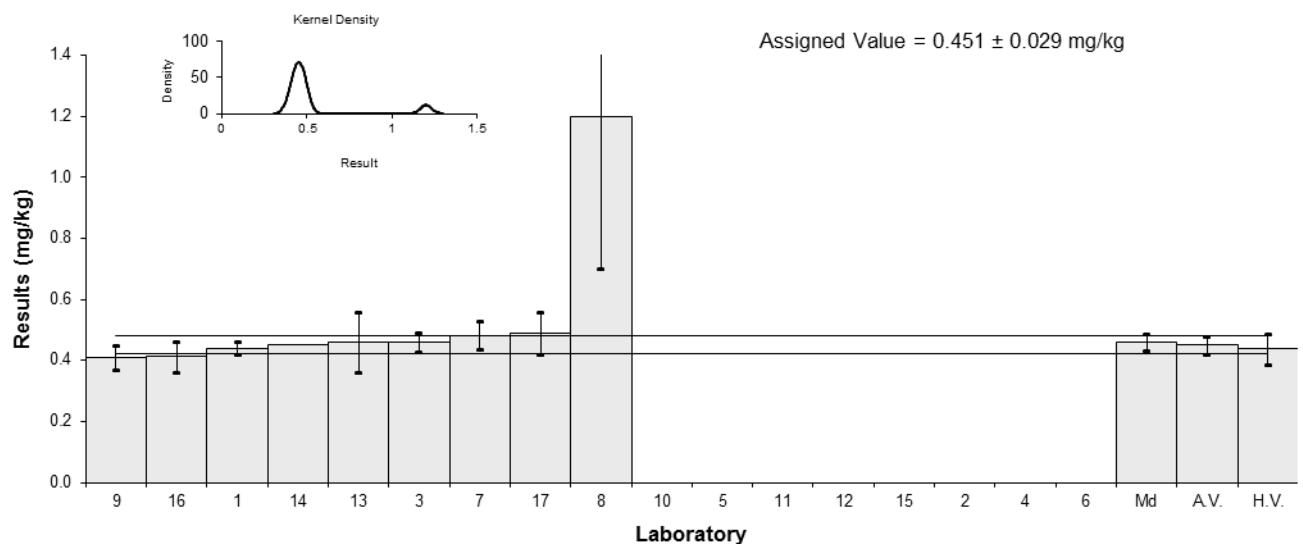
Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.44	0.02	-0.24	-0.31
2	NT	NT		
3	0.46	0.03	0.20	0.22
4	NT	NT		
5	NR	NR		
6	NT	NT		
7	0.483	0.048	0.71	0.57
8	1.2	0.5	16.61	1.50
9	0.41	0.04	-0.91	-0.83
10	<3	NR		
11	NT	NT		
12	NT	NT		
13	0.46	0.1	0.20	0.09
14	0.451	NR	0.00	0.00
15	NT	NT		
16	0.413	0.05	-0.84	-0.66
17	0.49	0.07	0.86	0.51

Statistics

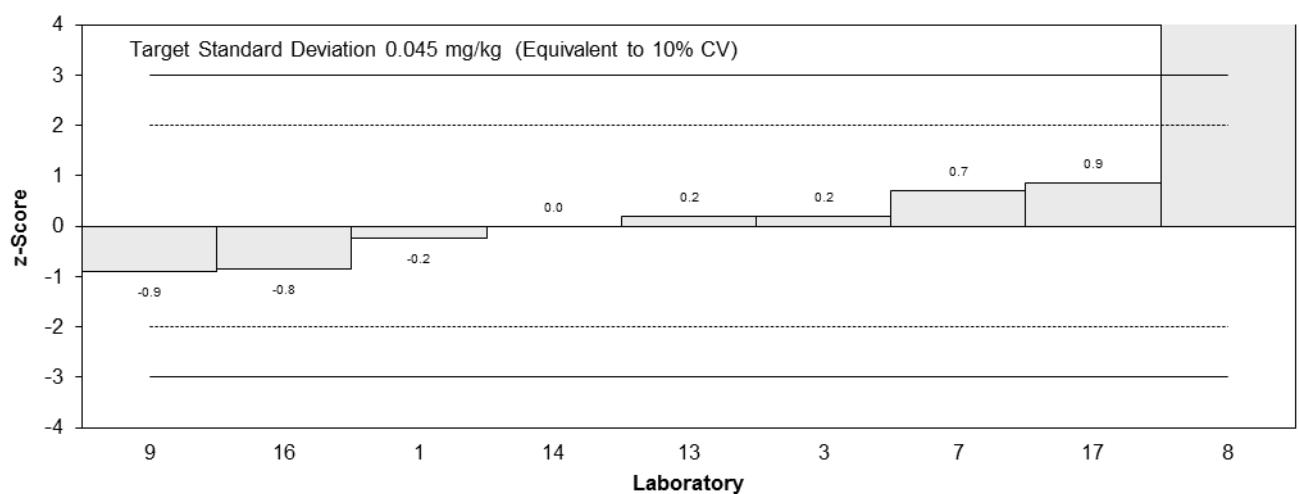
Assigned Value*	0.451	0.029
Spike	Not Spiked	
Homogeneity Value	0.437	0.052
Robust Average	0.458	0.033
Median	0.460	0.026
Mean	0.534	
N	9	
Max.	1.2	
Min.	0.41	
Robust SD	0.033	
Robust CV	7.2%	

*Robust Average excluding Laboratory 8.

Results: S1 - As



z-Scores: S1 - As



En-Scores: S1 - As

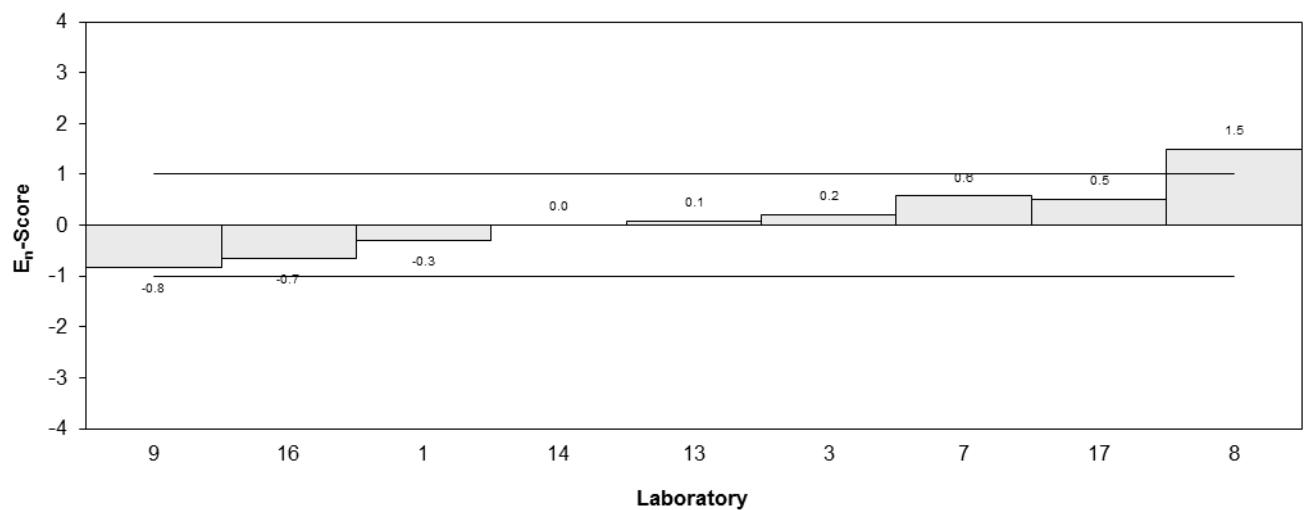


Figure 4

Table 11

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	B
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	0.69	0.05
2	NT	NT
3	NR	NR
4	NT	NT
5	NR	NR
6	NT	NT
7	0.632	0.063
8	NT	NT
9	<1	NR
10	<2	NR
11	NT	NT
12	NT	NT
13	0.51	0.2
14	NT	NT
15	NT	NT
16	0.626	0.21
17	0.49	0.07

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Robust Average	0.59	0.11
Median	0.63	0.12
Mean	0.59	
N	5	
Max.	0.69	
Min.	0.49	
Robust SD	0.097	
Robust CV	16%	

Results: S1 - B

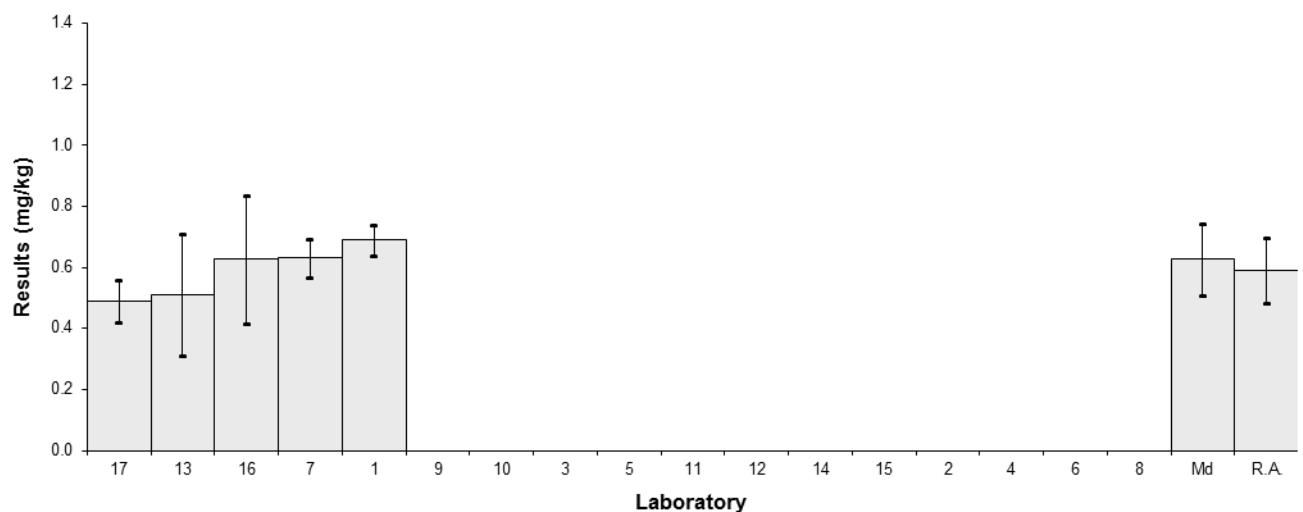


Figure 5

Table 12

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Ba
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.63	0.06	-0.88	-0.76
2	NT	NT		
3	NR	NR		
4	NT	NT		
5	NR	NR		
6	0.7	NR	0.13	0.17
7	0.687	0.069	-0.06	-0.05
8	<1.0	NR		
9	0.64	0.09	-0.74	-0.49
10	0.8	0.8	1.58	0.14
11	NT	NT		
12	NT	NT		
13	0.65	0.1	-0.59	-0.36
14	0.652	NR	-0.56	-0.74
15	NT	NT		
16	0.772	0.06	1.17	1.01
17	0.7	0.11	0.13	0.07

Statistics

Assigned Value	0.691	0.053
Spike	Not Spiked	
Homogeneity Value	0.580	0.070
Robust Average	0.691	0.053
Median	0.687	0.042
Mean	0.692	
N	9	
Max.	0.8	
Min.	0.63	
Robust SD	0.055	
Robust CV	8%	

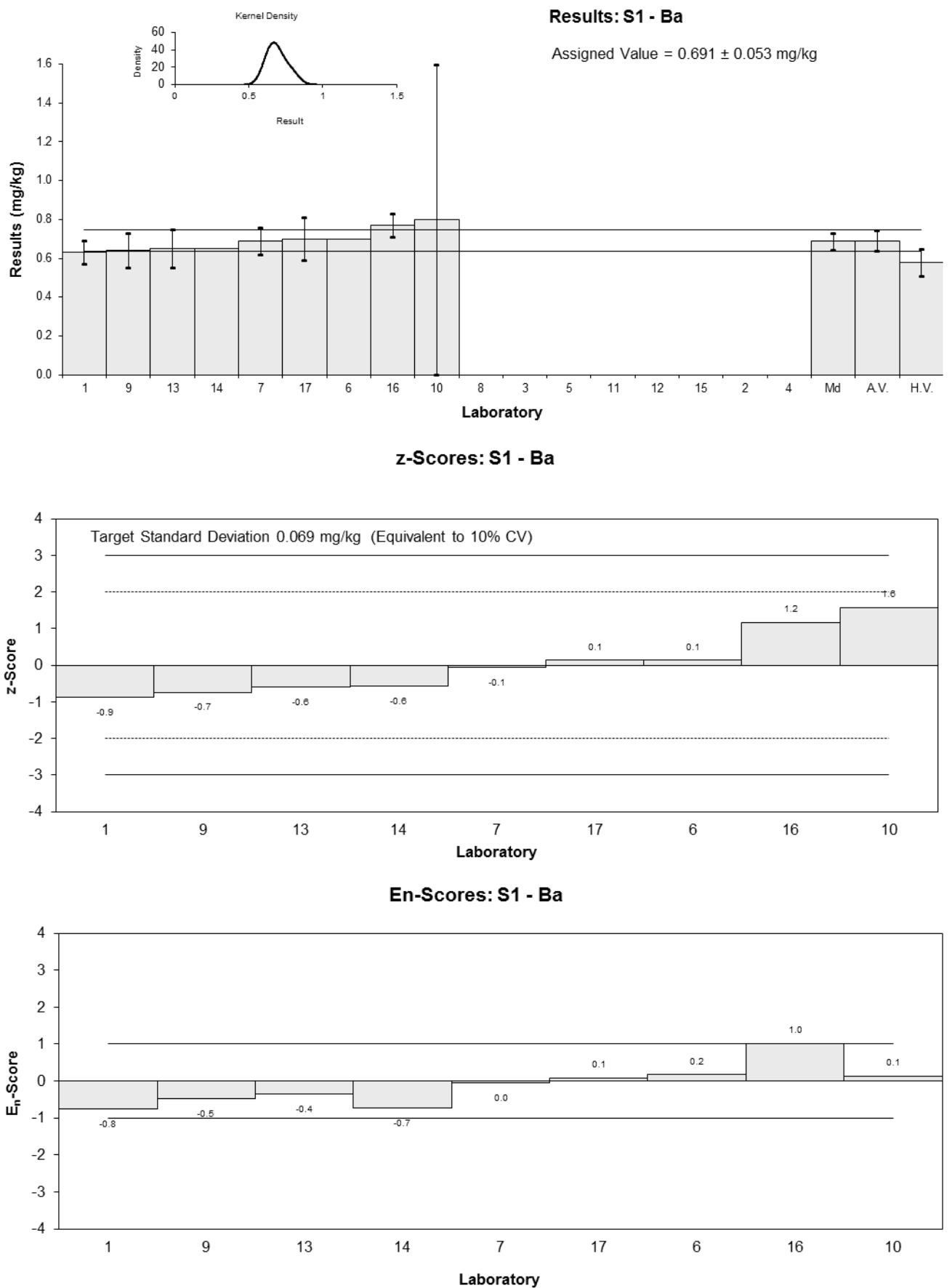


Figure 6

Table 13

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Be
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.32	0.03	-0.28	-0.24
2	NT	NT		
3	0.32	0.02	-0.28	-0.26
4	NT	NT		
5	NR	NR		
6	NT	NT		
7	0.328	0.033	-0.12	-0.10
8	<1.0	NR		
9	0.40	0.09	1.32	0.64
10	0.4	0.4	1.32	0.16
11	NT	NT		
12	NT	NT		
13	0.35	0.07	0.32	0.19
14	0.305	NR	-0.58	-0.58
15	NT	NT		
16	NR	NR		
17	0.23	0.03	-2.08	-1.78

Statistics

Assigned Value	0.334	0.050
Spike	Not Spiked	
Homogeneity Value	0.348	0.042
Robust Average	0.334	0.050
Median	0.324	0.028
Mean	0.332	
N	8	
Max.	0.4	
Min.	0.23	
Robust SD	0.057	
Robust CV	17%	

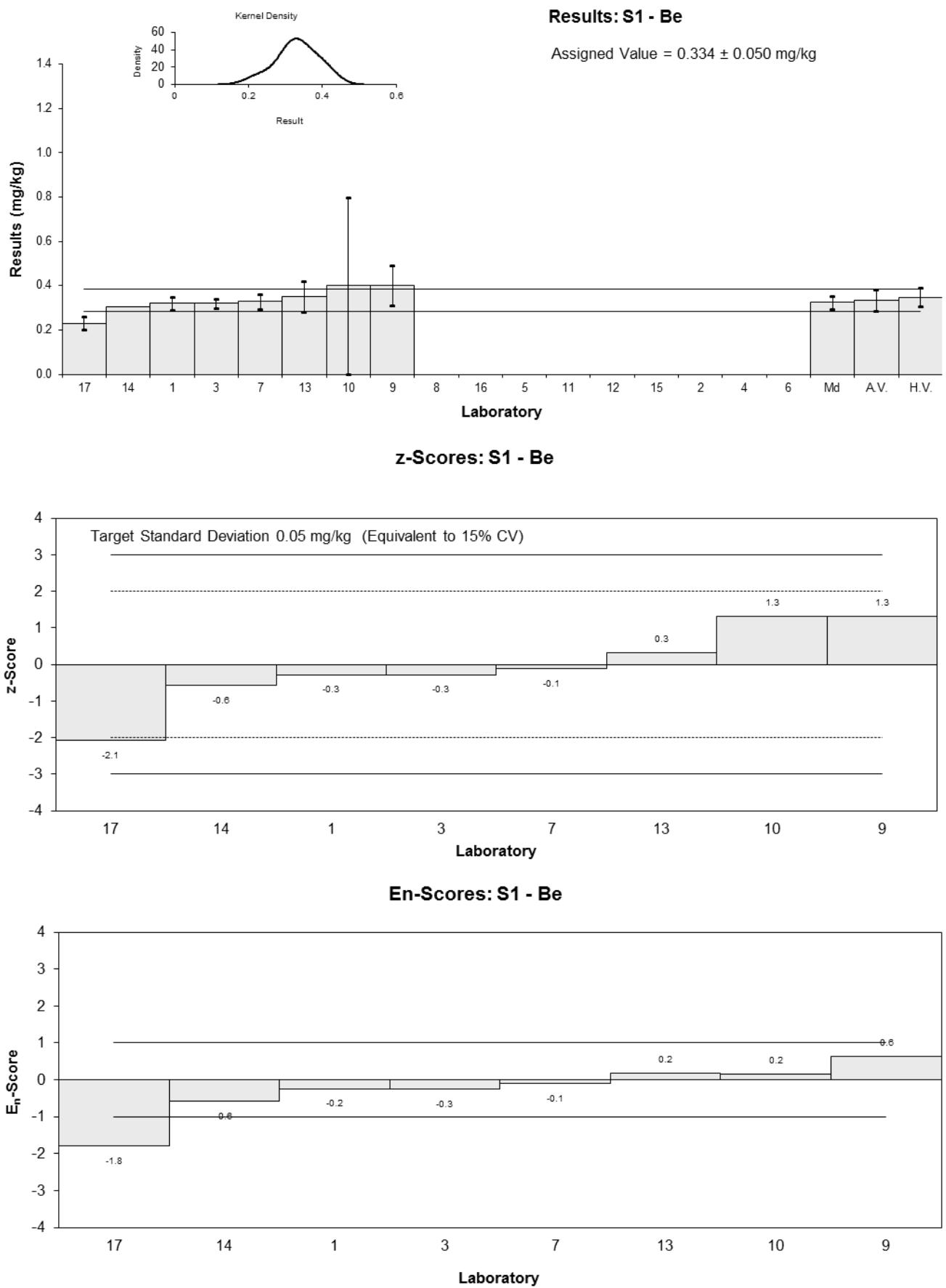


Figure 7

Table 14

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Bi
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.34	0.03	-0.23	-0.25
2	NT	NT		
3	0.35	0.05	0.06	0.04
4	NT	NT		
5	NR	NR		
6	NT	NT		
7	0.336	0.034	-0.34	-0.33
8	NT	NT		
9	0.345	0.028	-0.09	-0.10
10	NT	NT		
11	NT	NT		
12	NT	NT		
13	0.35	0.07	0.06	0.03
14	NT	NT		
15	NT	NT		
16	NR	NR		
17	0.38	0.06	0.92	0.52

Statistics

Assigned Value	0.348	0.012
Spike	Not Spiked	
Homogeneity Value	0.363	0.044
Robust Average	0.348	0.012
Median	0.348	0.008
Mean	0.350	
N	6	
Max.	0.38	
Min.	0.336	
Robust SD	0.011	
Robust CV	3.2%	

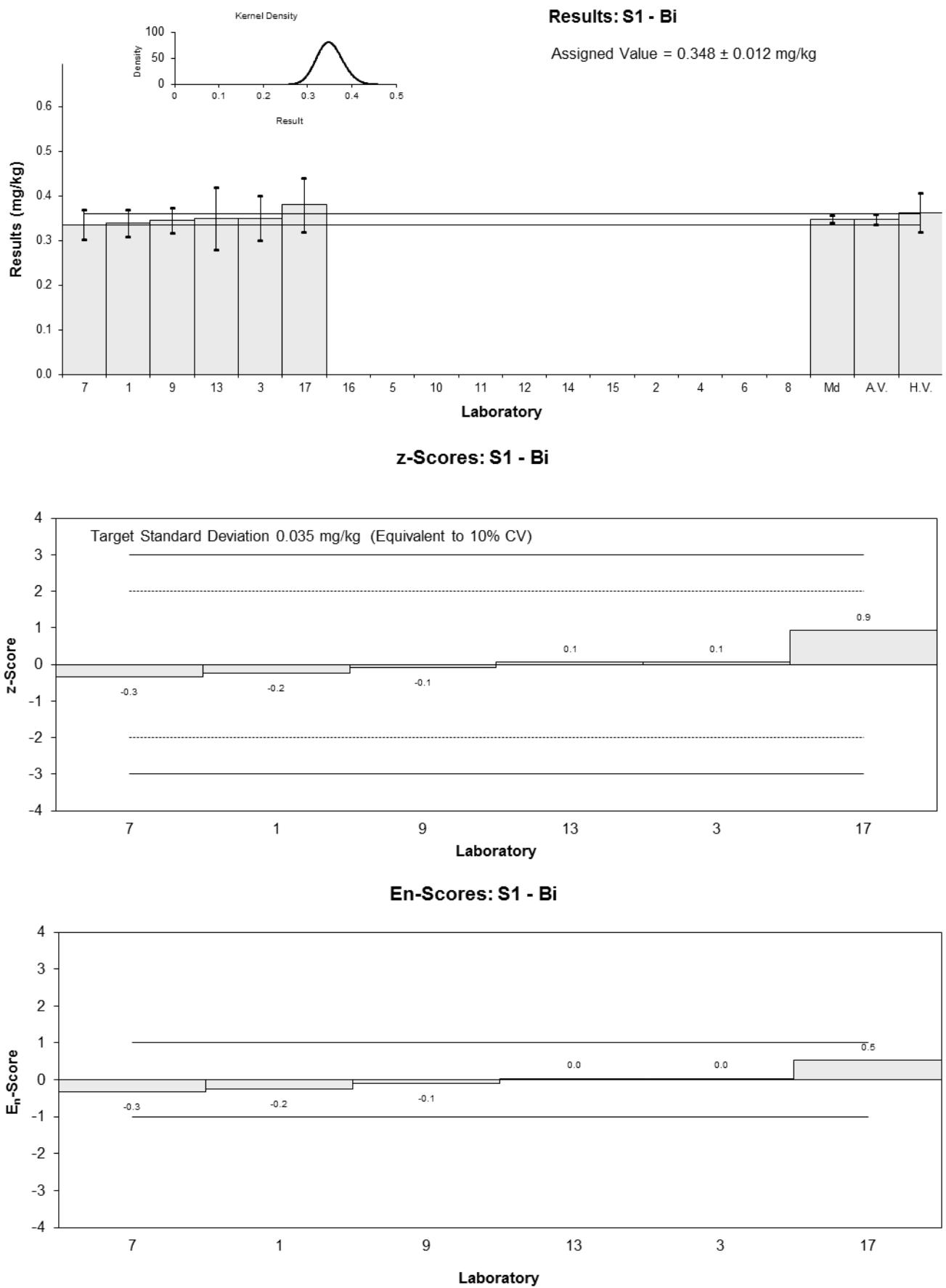


Figure 8

Table 15

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Ca
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	160	8.0	-0.42	-0.55
2	NT	NT		
3	152	20	-0.90	-0.67
4	NT	NT		
5	171.69	7	0.28	0.38
6	181.5	NR	0.87	1.45
7	167	16.7	0.00	0.00
8	NT	NT		
9	158	35	-0.54	-0.25
10	170	20	0.18	0.13
11	NT	NT		
12	NT	NT		
13	215	45	2.87	1.04
14	177.478	NR	0.63	1.05
15	NT	NT		
16	155	17	-0.72	-0.61
17	156	23	-0.66	-0.44

Statistics

Assigned Value	167	10
Spike	Not Spiked	
Homogeneity Value	165	17
Robust Average	167	10
Median	167	10
Mean	169	
N	11	
Max.	215	
Min.	152	
Robust SD	13	
Robust CV	7.8%	

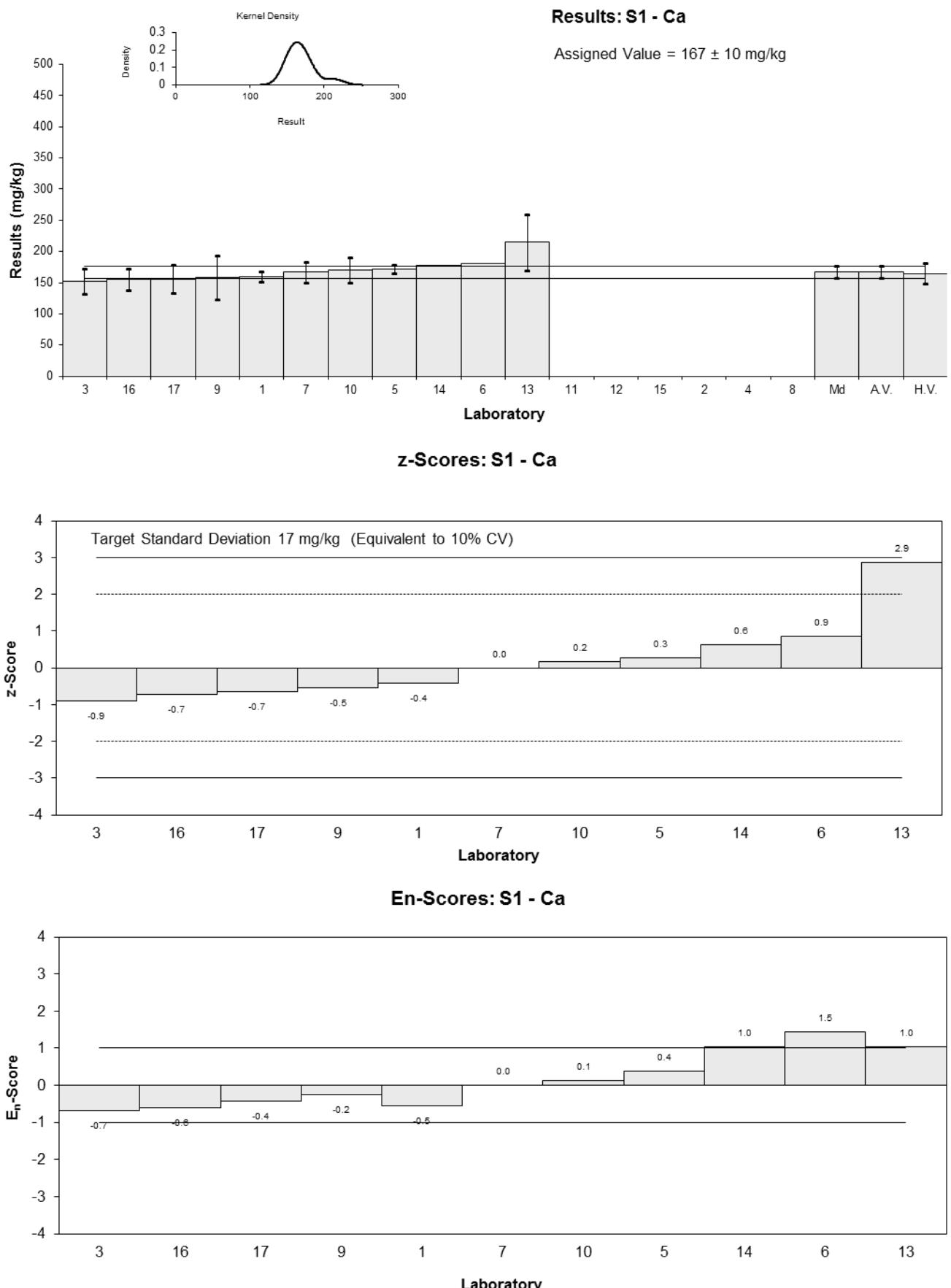


Figure 9

Table 16

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Cd
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.95	0.08	-0.15	-0.15
2	NT	NT		
3	0.89	0.21	-0.77	-0.34
4	NT	NT		
5	1.037	0.1	0.76	0.65
6	1.07	NR	1.10	2.12
7	1.03	0.103	0.68	0.58
8	<1.0	NR		
9	0.93	0.08	-0.35	-0.36
10	0.9	0.9	-0.66	-0.07
11	NT	NT		
12	NT	NT		
13	0.91	0.2	-0.56	-0.26
14	0.954	NR	-0.10	-0.20
15	NT	NT		
16	0.957	0.04	-0.07	-0.11
17	0.98	0.15	0.17	0.10

Statistics

Assigned Value	0.964	0.050
Spike	Not Spiked	
Homogeneity Value	0.95	0.11
Robust Average	0.964	0.050
Median	0.954	0.044
Mean	0.964	
N	11	
Max.	1.07	
Min.	0.89	
Robust SD	0.066	
Robust CV	6.8%	

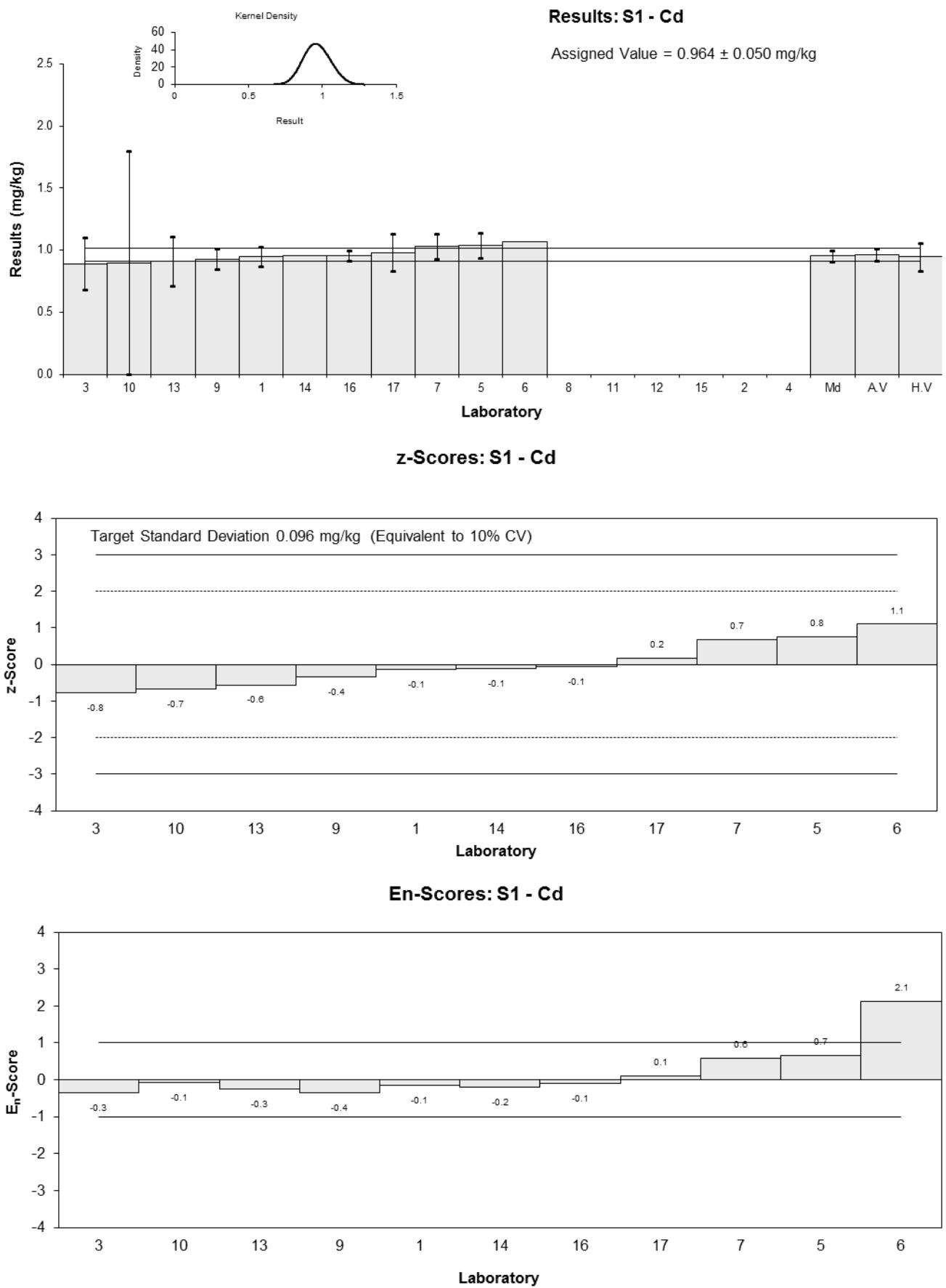


Figure 10

Table 17

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Co
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.63	0.03	-0.03	-0.04
2	NT	NT		
3	0.62	0.07	-0.19	-0.14
4	NT	NT		
5	NR	NR		
6	0.65	NR	0.28	0.40
7	0.580	0.058	-0.82	-0.71
8	<1.0	NR		
9	0.61	0.09	-0.35	-0.22
10	0.7	0.7	1.08	0.10
11	NT	NT		
12	NT	NT		
13	0.70	0.1	1.08	0.62
14	0.552	NR	-1.27	-1.78
15	NT	NT		
16	0.677	0.05	0.71	0.67
17	0.60	0.09	-0.51	-0.32

Statistics

Assigned Value	0.632	0.045
Spike	Not Spiked	
Homogeneity Value	0.617	0.062
Robust Average	0.632	0.045
Median	0.625	0.037
Mean	0.632	
N	10	
Max.	0.7	
Min.	0.552	
Robust SD	0.057	
Robust CV	9%	

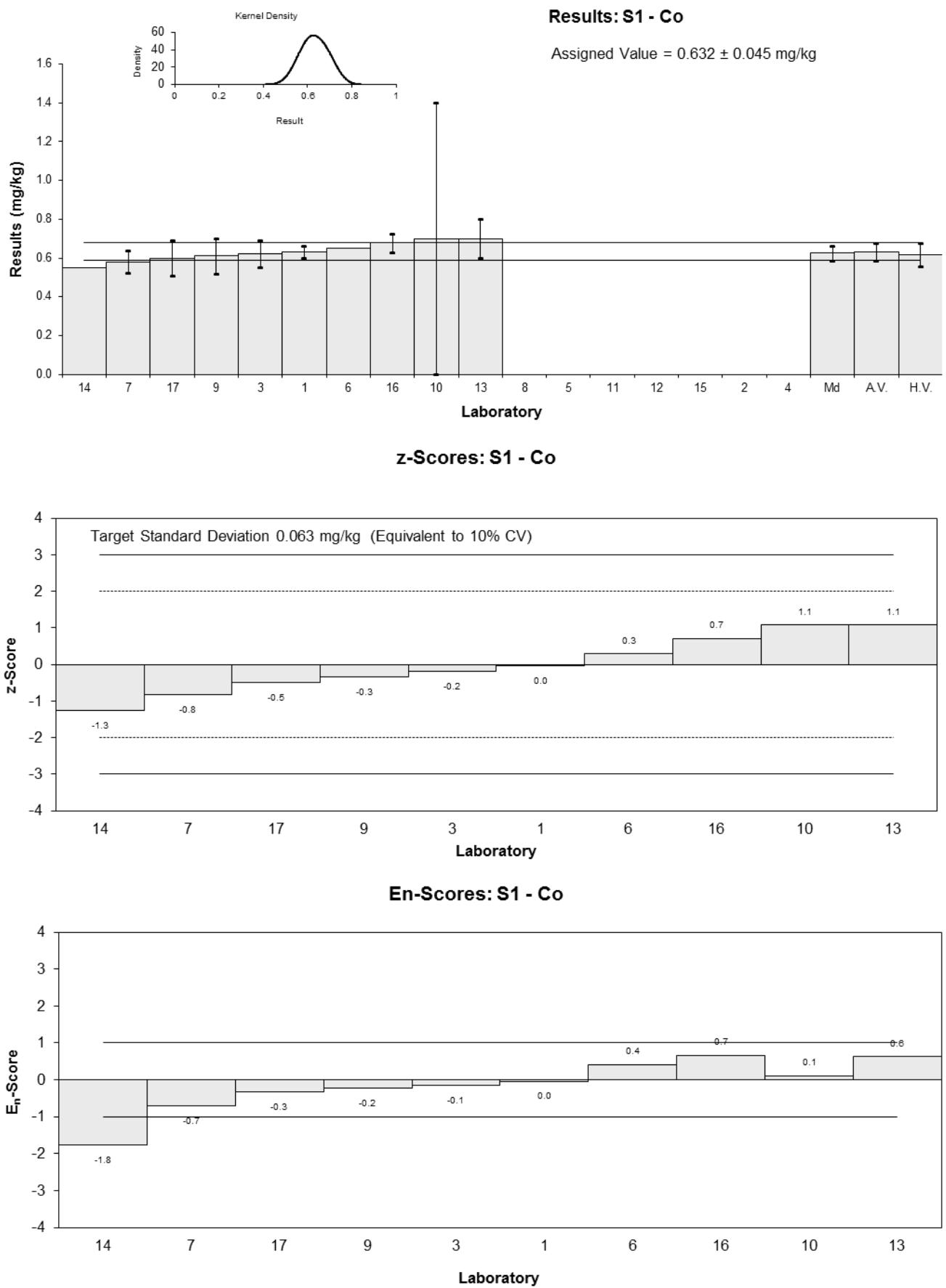


Figure 11

Table 18

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Cr
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.20	0.02	-2.72	-7.62
2	NT	NT		
3	0.38	0.05	-0.66	-1.05
4	NT	NT		
5	NR	NR		
6	0.50	NR	0.71	2.58
7	0.295	0.03	-1.63	-3.72
8	<1.0	NR		
9	0.19	0.05	-2.83	-4.47
10	<1	NR		
11	NT	NT		
12	NT	NT		
13	0.27	0.1	-1.92	-1.63
14	0.415	NR	-0.26	-0.96
15	NT	NT		
16	0.08	0.005	-4.09	-14.60
17	0.29	0.04	-1.69	-3.17

Statistics

Assigned Value*	0.438	0.024
Spike	Not Spiked	
Homogeneity Value	0.438	0.024
Robust Average	0.29	0.12
Median	0.29	0.10
Mean	0.29	
N	9	
Max.	0.5	
Min.	0.08	
Robust SD	0.095	
Robust CV	33%	

*Information Value measured by d-IDMS.

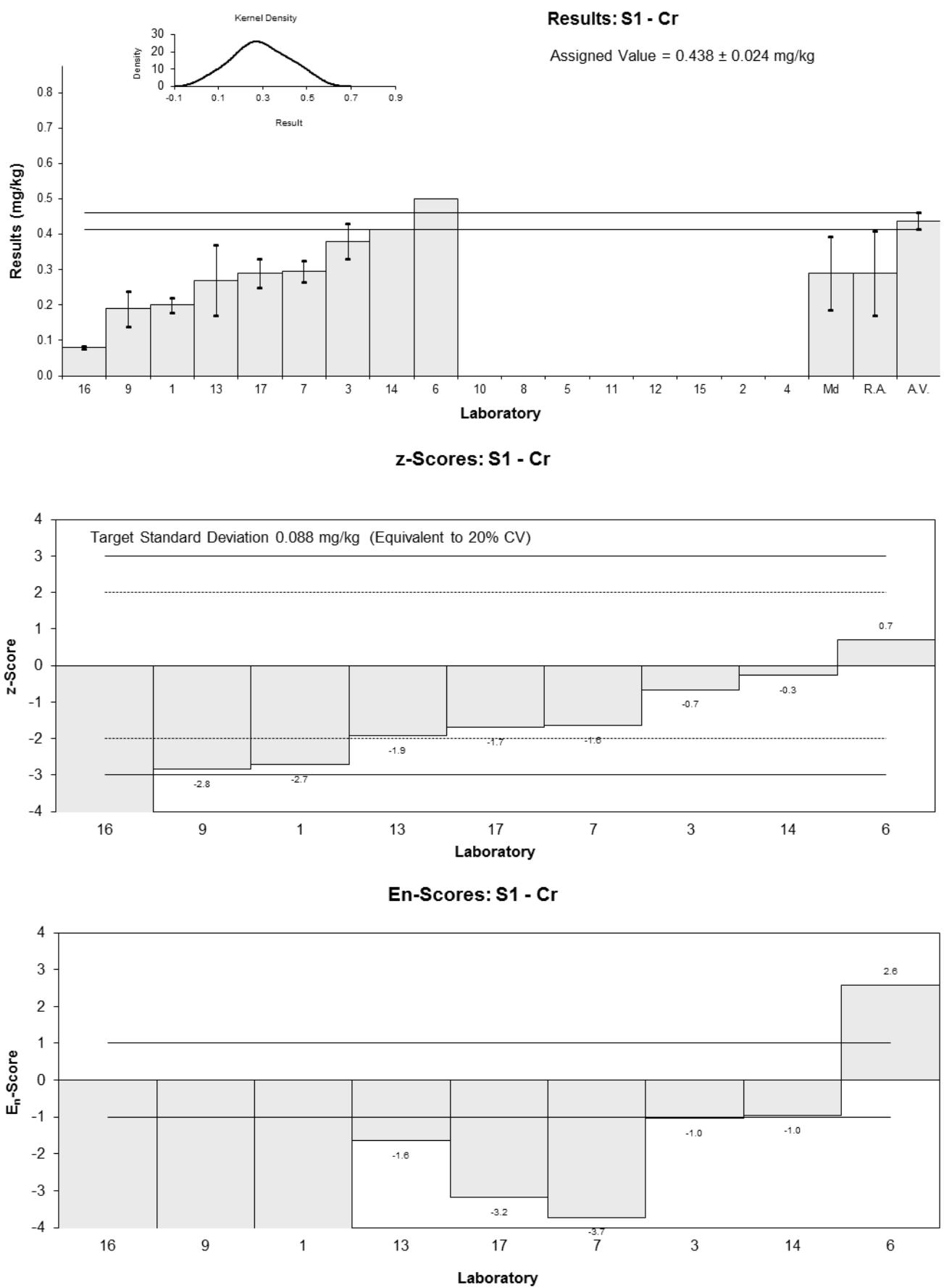


Figure 12

Table 19

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Cu
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	160	8.0	-0.18	-0.34
2	NT	NT		
3	163	12	0.00	0.00
4	NT	NT		
5	175.36	13	0.76	0.91
6	166.1	NR	0.19	0.77
7	155	15.5	-0.49	-0.50
8	157	25	-0.37	-0.24
9	161	13	-0.12	-0.15
10	160	20	-0.18	-0.15
11	NT	NT		
12	NT	NT		
13	164	20	0.06	0.05
14	162.930	NR	0.00	-0.02
15	NT	NT		
16	185	0.7	1.35	5.42
17	161	24	-0.12	-0.08

Statistics

Assigned Value	163	4
Spike	Not Spiked	
Homogeneity Value	168	13
Robust Average	163	4
Median	162	2
Mean	164	
N	12	
Max.	185	
Min.	155	
Robust SD	5.5	
Robust CV	3.4%	

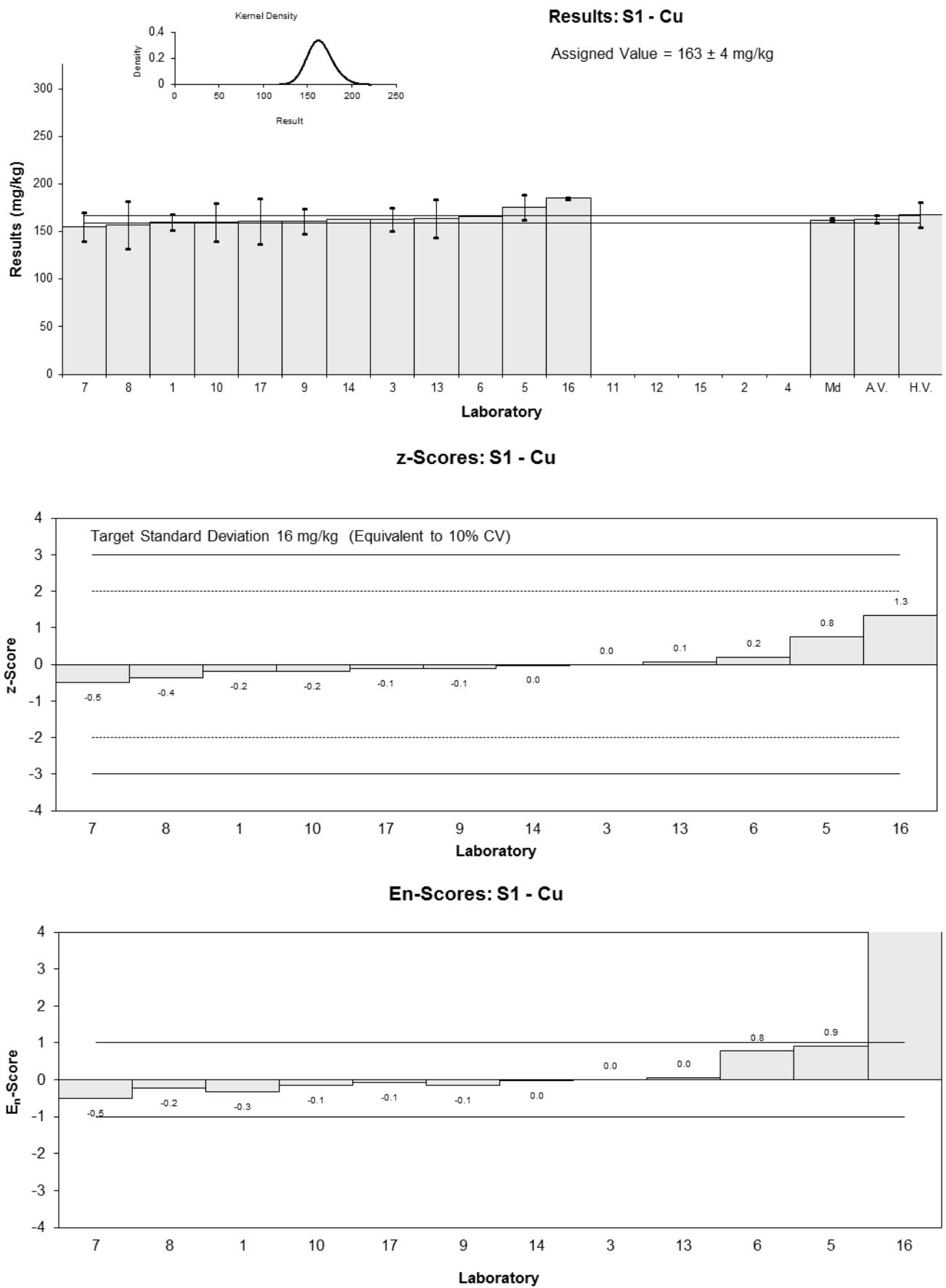


Figure 13

Table 20

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Fe
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	510	20	0.65	1.18
2	NT	NT		
3	461	36	-0.38	-0.45
4	NT	NT		
5	90.54	6	-8.11	-21.55
6	474.4	NR	-0.10	-0.27
7	448	44.8	-0.65	-0.65
8	488	122	0.19	0.07
9	469	42	-0.21	-0.22
10	510	50	0.65	0.59
11	NT	NT		
12	NT	NT		
13	463	50	-0.33	-0.30
14	478.367	NR	-0.01	-0.04
15	NT	NT		
16	492	61	0.27	0.21
17	470	70	-0.19	-0.12

Statistics

Assigned Value*	479	17
Spike	Not Spiked	
Homogeneity Value	503	50
Robust Average	475	18
Median	472	13
Mean	446	
N	12	
Max.	510	
Min.	90.54	
Robust SD	22	
Robust CV	4.6%	

*Robust Average excluding Laboratory 5.

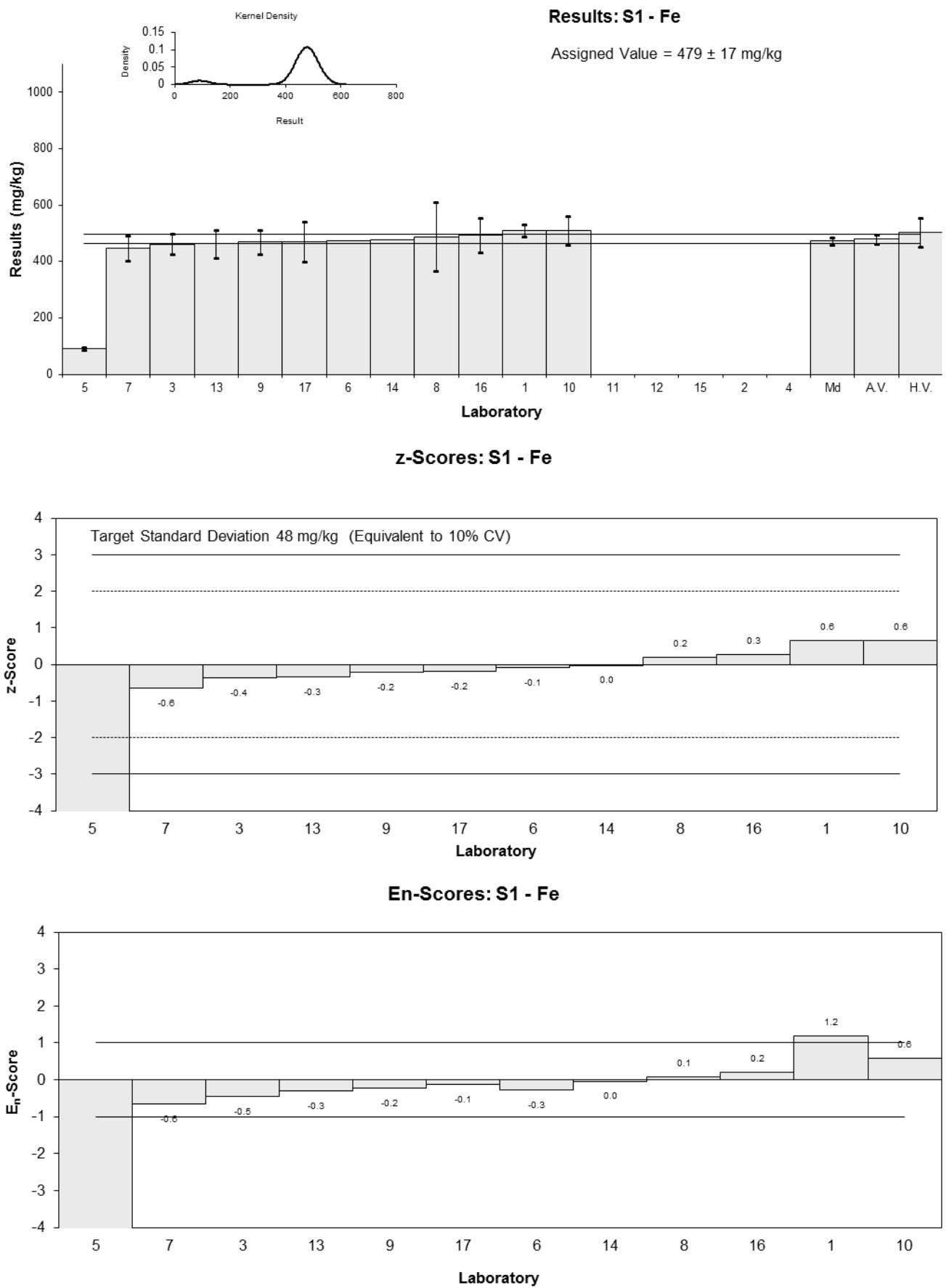


Figure 14

Table 21

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Hg
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.39	0.03	0.10	0.13
2	NT	NT		
3	0.36	0.07	-0.42	-0.31
4	NT	NT		
5	NR	NR		
6	0.40	NR	0.28	0.47
7	0.446	0.045	1.08	1.10
8	0.33	0.10	-0.94	-0.51
9	0.38	0.05	-0.07	-0.07
10	0.21	0.05	-3.02	-2.88
11	NT	NT		
12	NT	NT		
13	0.44	0.1	0.97	0.53
14	0.3738	NR	-0.18	-0.30
15	NT	NT		
16	0.382	0.015	-0.03	-0.05
17	0.41	0.06	0.45	0.38

Statistics

Assigned Value	0.384	0.034
Spike	Not Spiked	
Homogeneity Value	0.380	0.046
Robust Average	0.384	0.034
Median	0.382	0.022
Mean	0.375	
N	11	
Max.	0.446	
Min.	0.21	
Robust SD	0.046	
Robust CV	12%	

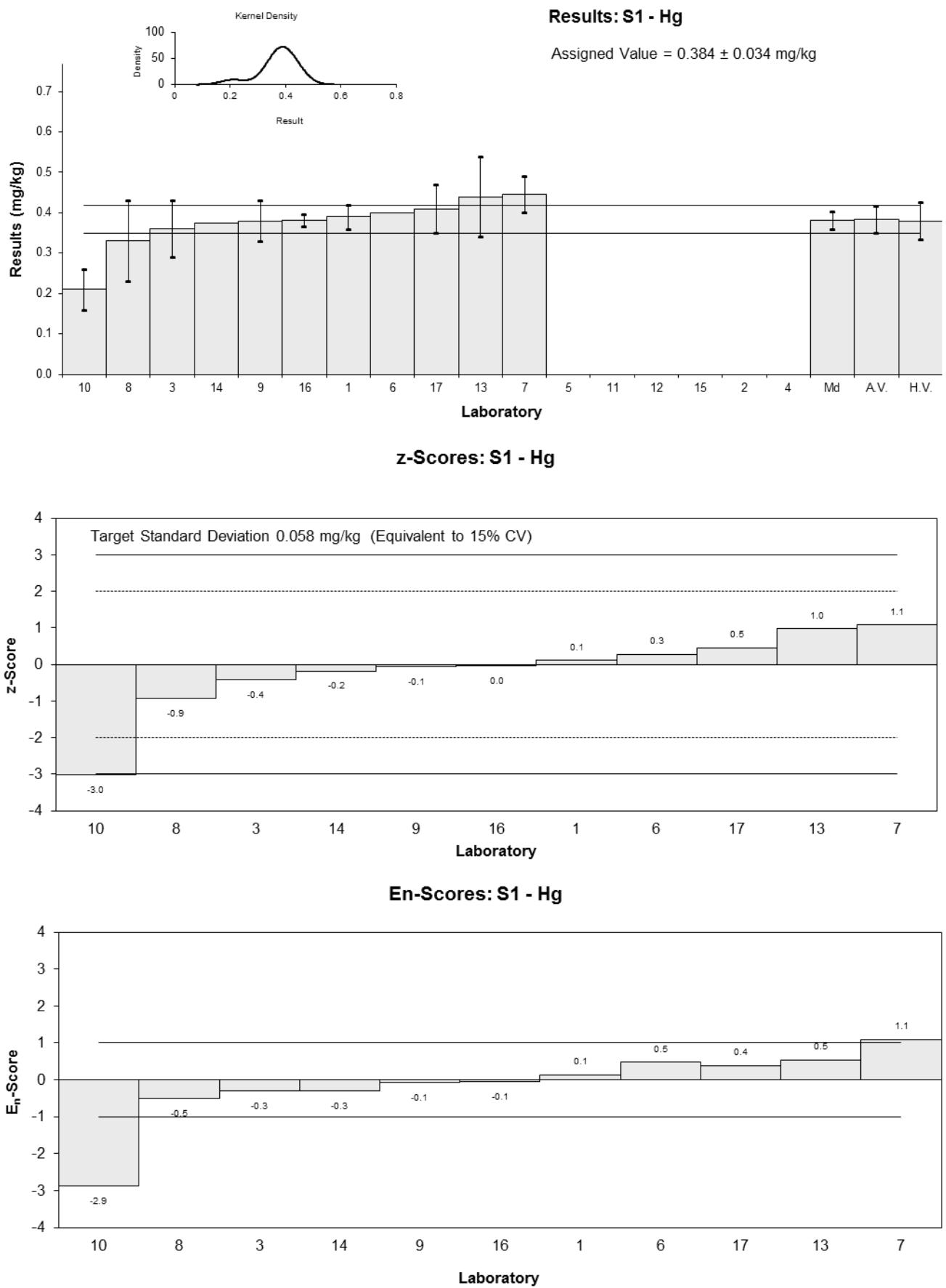


Figure 15

Table 22

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Inorganic As
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	0.40	0.04
2	NT	NT
3	0.33	0.1
4	NT	NT
5	NR	NR
6	NT	NT
7	NT	NT
8	NT	NT
9	0.28	0.04
10	NT	NT
11	NT	NT
12	NT	NT
13	NT	NT
14	NT	NT
15	NT	NT
16	NR	NR
17	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.330	0.010
Robust Average	0.337	0.099
Median	0.33	0.18
Mean	0.337	
N	3	
Max.	0.4	
Min.	0.28	
Robust SD	0.068	
Robust CV	20%	

Results: S1 - Inorganic As

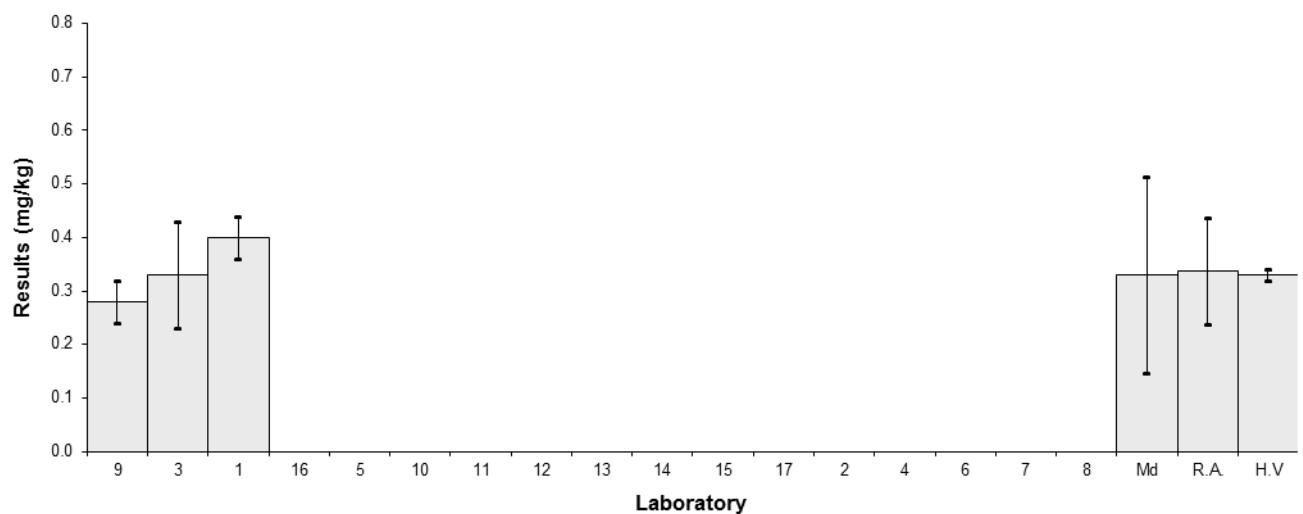


Figure 16

Table 23

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	K
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	9100	300	-0.55	-0.77
2	NT	NT		
3	8617	629	-1.05	-1.15
4	NT	NT		
5	5210.3	540	-4.59	-5.38
6	10510	NR	0.91	1.42
7	9690	969	0.06	0.05
8	NT	NT		
9	10200	911	0.59	0.52
10	8400	1000	-1.28	-1.05
11	NT	NT		
12	NT	NT		
13	9950	900	0.33	0.29
14	9862.011	NR	0.24	0.37
15	NT	NT		
16	10200	550	0.59	0.69
17	9740	1461	0.11	0.07

Statistics

Assigned Value*	9630	620
Spike	Not Spiked	
Homogeneity Value	10300	1000
Robust Average	9490	690
Median	9740	460
Mean	9230	
N	11	
Max.	10510	
Min.	5210.3	
Robust SD	910	
Robust CV	9.6%	

*Robust Average excluding Laboratory 5.

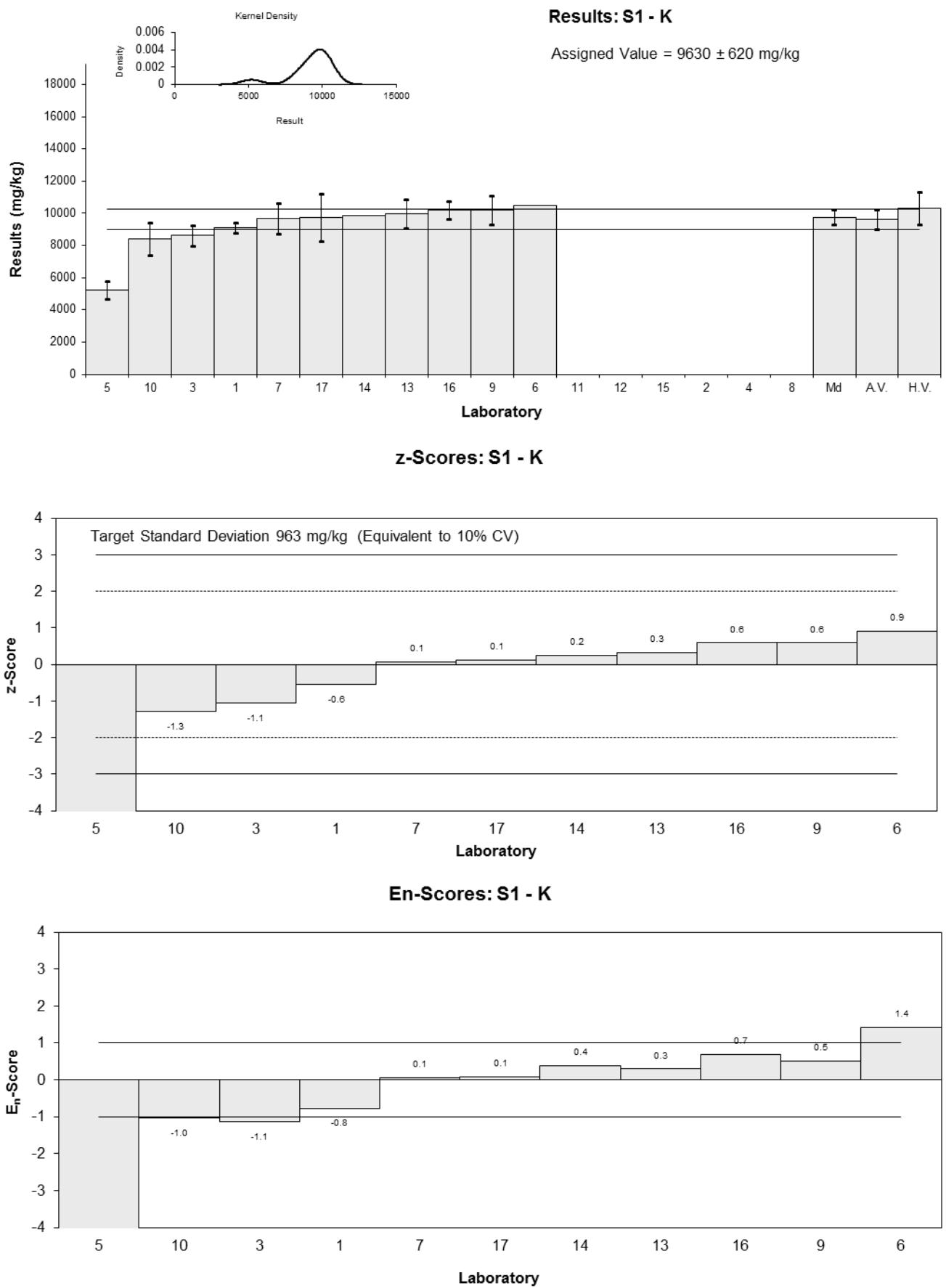


Figure 17

Table 24

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Li
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.30	0.03	-0.38	-0.43
2	NT	NT		
3	0.35	0.04	0.38	0.39
4	NT	NT		
5	NR	NR		
6	0.30	NR	-0.38	-0.50
7	0.338	0.034	0.20	0.22
8	NT	NT		
9	0.37	0.04	0.69	0.70
10	0.3	0.3	-0.38	-0.08
11	NT	NT		
12	NT	NT		
13	0.40	0.1	1.15	0.67
14	NT	NT		
15	NT	NT		
16	NR	NR		
17	0.22	0.03	-1.62	-1.80

Statistics

Assigned Value	0.325	0.050
Spike	Not Spiked	
Homogeneity Value	0.370	0.055
Robust Average	0.325	0.050
Median	0.319	0.031
Mean	0.322	
N	8	
Max.	0.4	
Min.	0.22	
Robust SD	0.057	
Robust CV	18%	

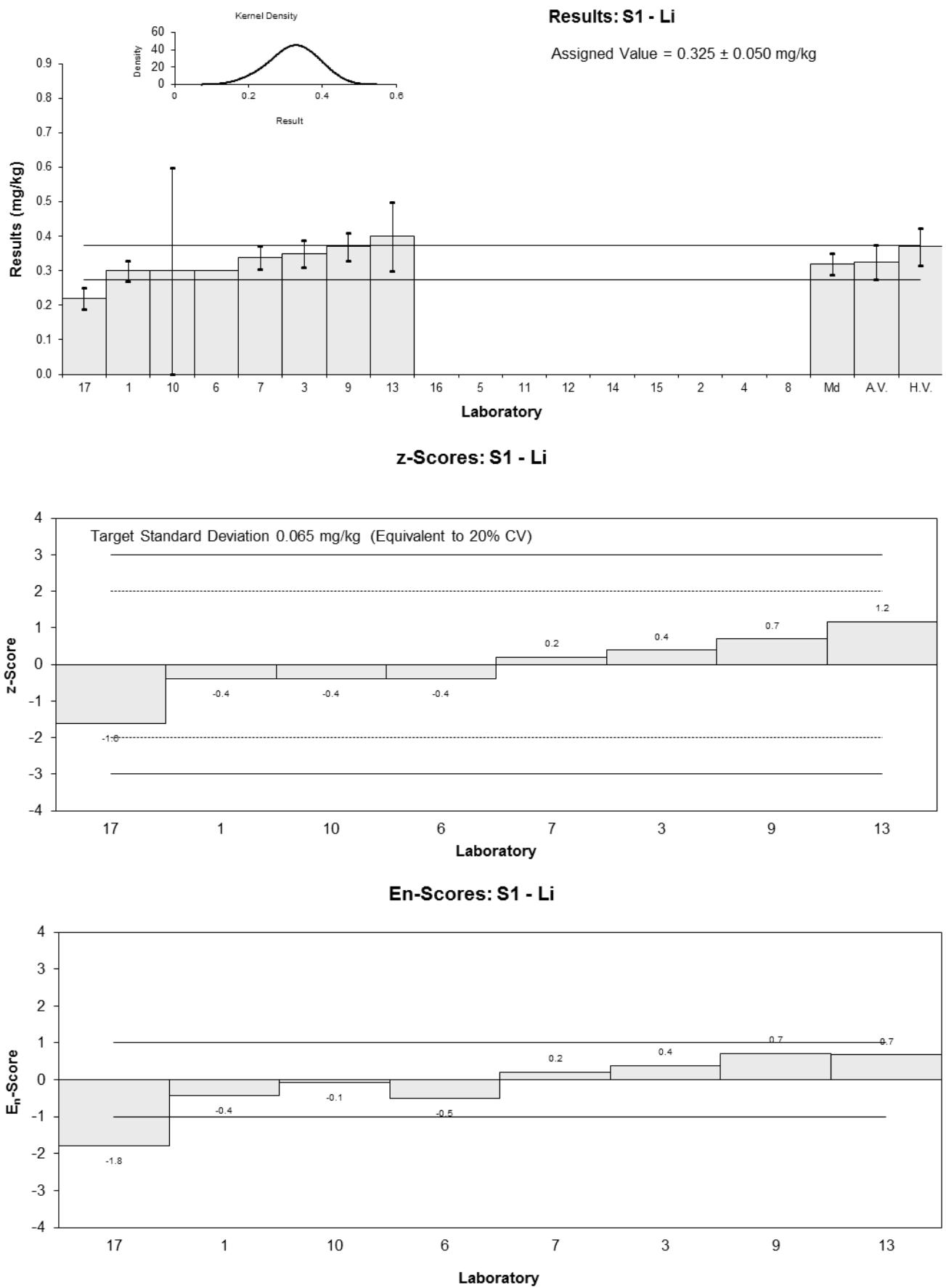


Figure 18

Table 25

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Mg
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	570	25	-0.44	-0.67
2	NT	NT		
3	594	71	-0.03	-0.03
4	NT	NT		
5	608.32	78	0.21	0.15
6	663	NR	1.12	2.23
7	558	55.8	-0.64	-0.60
8	NT	NT		
9	650	59	0.91	0.82
10	560	170	-0.60	-0.21
11	NT	NT		
12	NT	NT		
13	607	60	0.18	0.16
14	564.475	NR	-0.53	-1.05
15	NT	NT		
16	616	8	0.34	0.64
17	573	86	-0.39	-0.25

Statistics

Assigned Value	596	30
Spike	Not Spiked	
Homogeneity Value	653	65
Robust Average	596	30
Median	594	24
Mean	597	
N	11	
Max.	663	
Min.	558	
Robust SD	39	
Robust CV	6.5%	

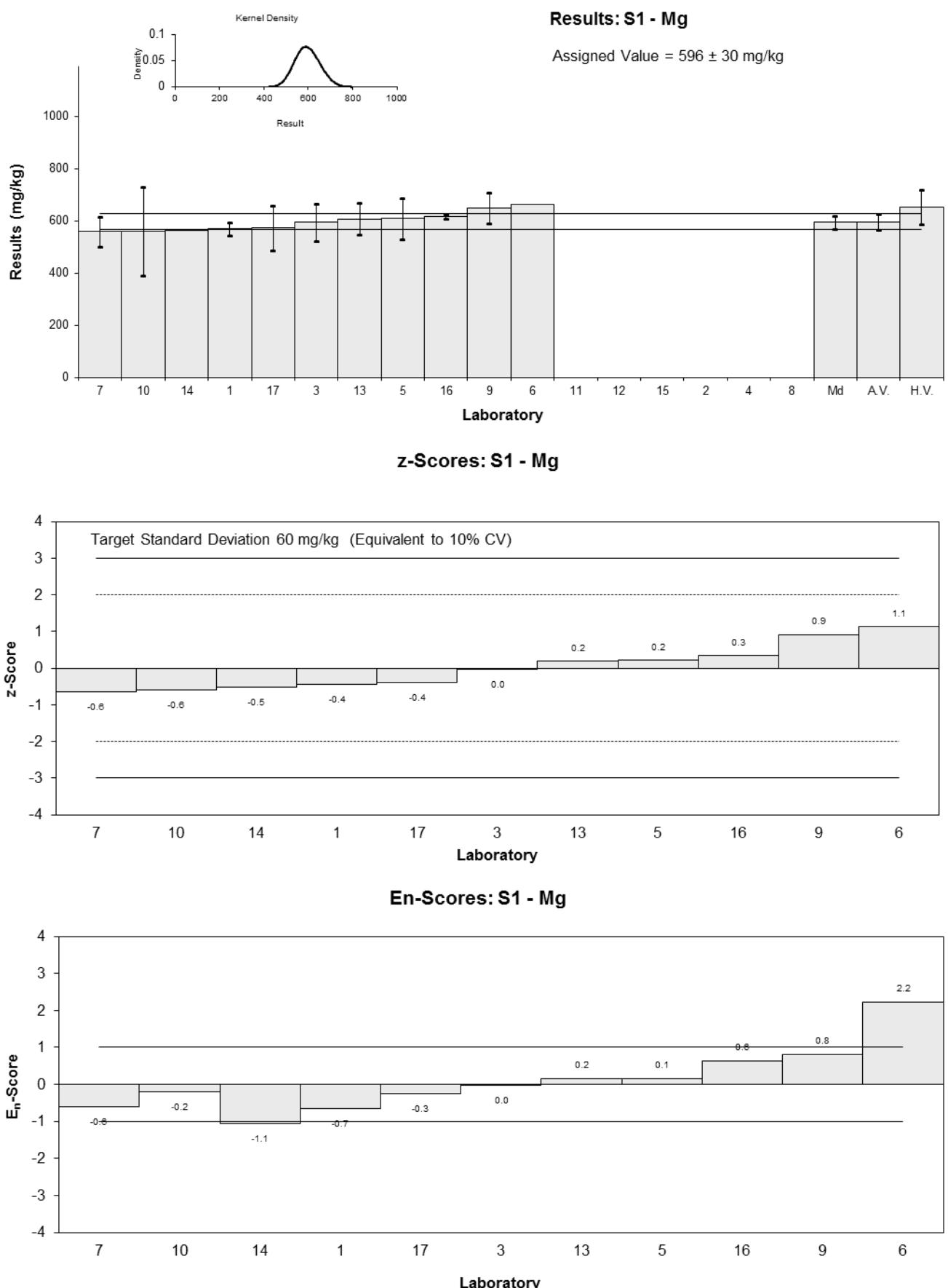


Figure 19

Table 26

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Mn
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	10	1.0	0.16	0.14
2	NT	NT		
3	9.8	1.3	-0.04	-0.03
4	NT	NT		
5	NR	NR		
6	9.74	NR	-0.10	-0.20
7	9.45	0.945	-0.40	-0.36
8	10.4	2.6	0.57	0.21
9	9.88	1.03	0.04	0.03
10	11	3	1.18	0.38
11	NT	NT		
12	NT	NT		
13	9.69	0.9	-0.15	-0.15
14	10.462	NR	0.63	1.24
15	NT	NT		
16	8.70	0.7	-1.16	-1.33
17	9.13	1.37	-0.72	-0.49

Statistics

Assigned Value	9.84	0.50
Spike	Not Spiked	
Homogeneity Value	10.8	1.1
Robust Average	9.84	0.50
Median	9.80	0.35
Mean	9.84	
N	11	
Max.	11	
Min.	8.7	
Robust SD	0.66	
Robust CV	6.7%	

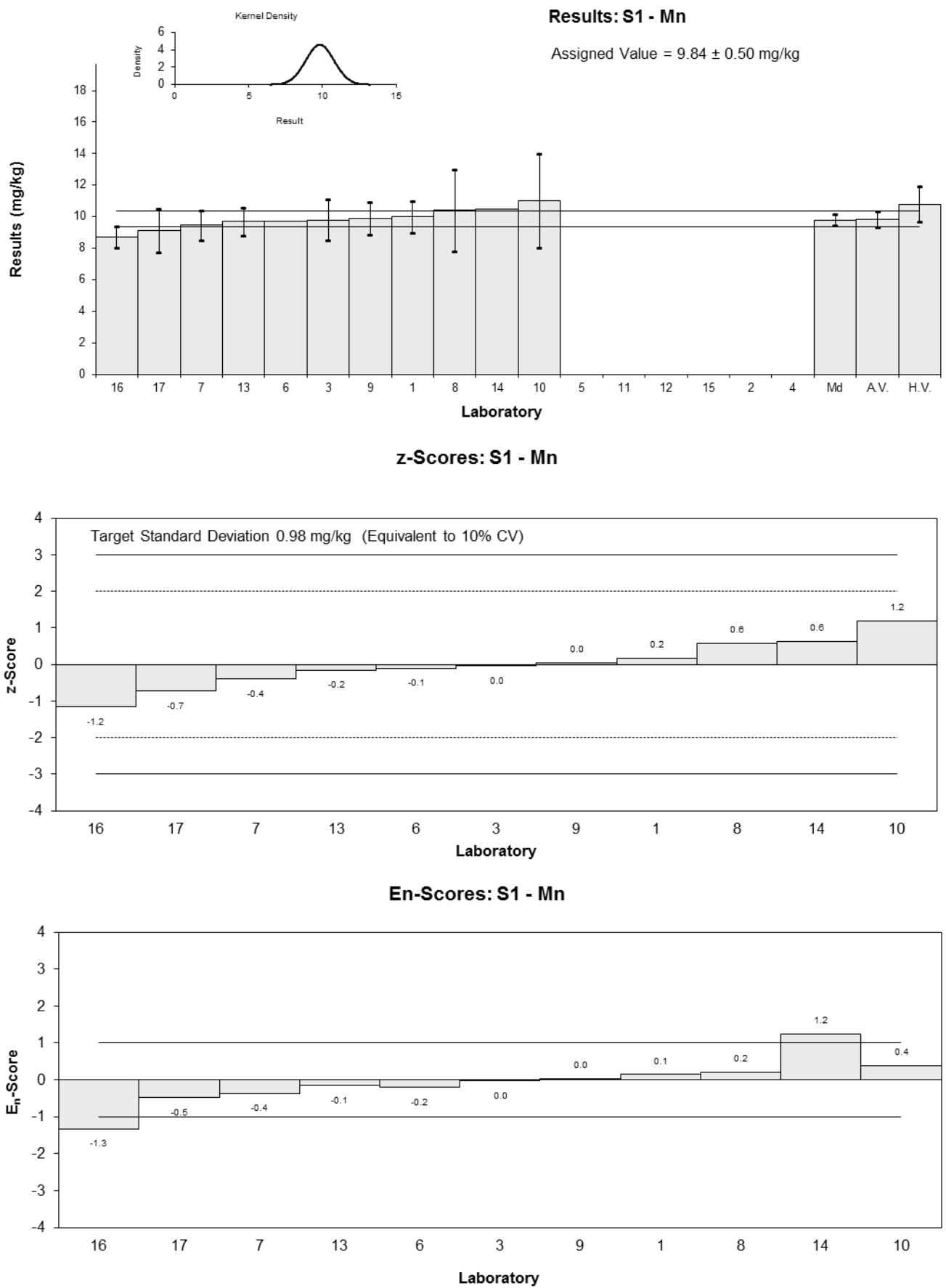


Figure 20

Table 27

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Mo
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	4.1	0.3	0.50	0.64
2	NT	NT		
3	3.5	0.5	-1.03	-0.80
4	NT	NT		
5	NR	NR		
6	4.10	NR	0.50	2.94
7	3.87	0.387	-0.08	-0.08
8	3.8	1.0	-0.26	-0.10
9	3.81	0.48	-0.24	-0.19
10	4	3	0.25	0.03
11	NT	NT		
12	NT	NT		
13	3.95	0.6	0.12	0.08
14	NT	NT		
15	NT	NT		
16	4.10	0.04	0.50	2.52
17	3.73	0.56	-0.44	-0.31

Statistics

Assigned Value*	3.903	0.067
Spike	Not Spiked	
Homogeneity Value	3.903	0.067
Robust Average	3.91	0.15
Median	3.91	0.15
Mean	3.90	
N	10	
Max.	4.1	
Min.	3.5	
Robust SD	0.19	
Robust CV	4.9%	

*Reference Value measured by d-IDMS.

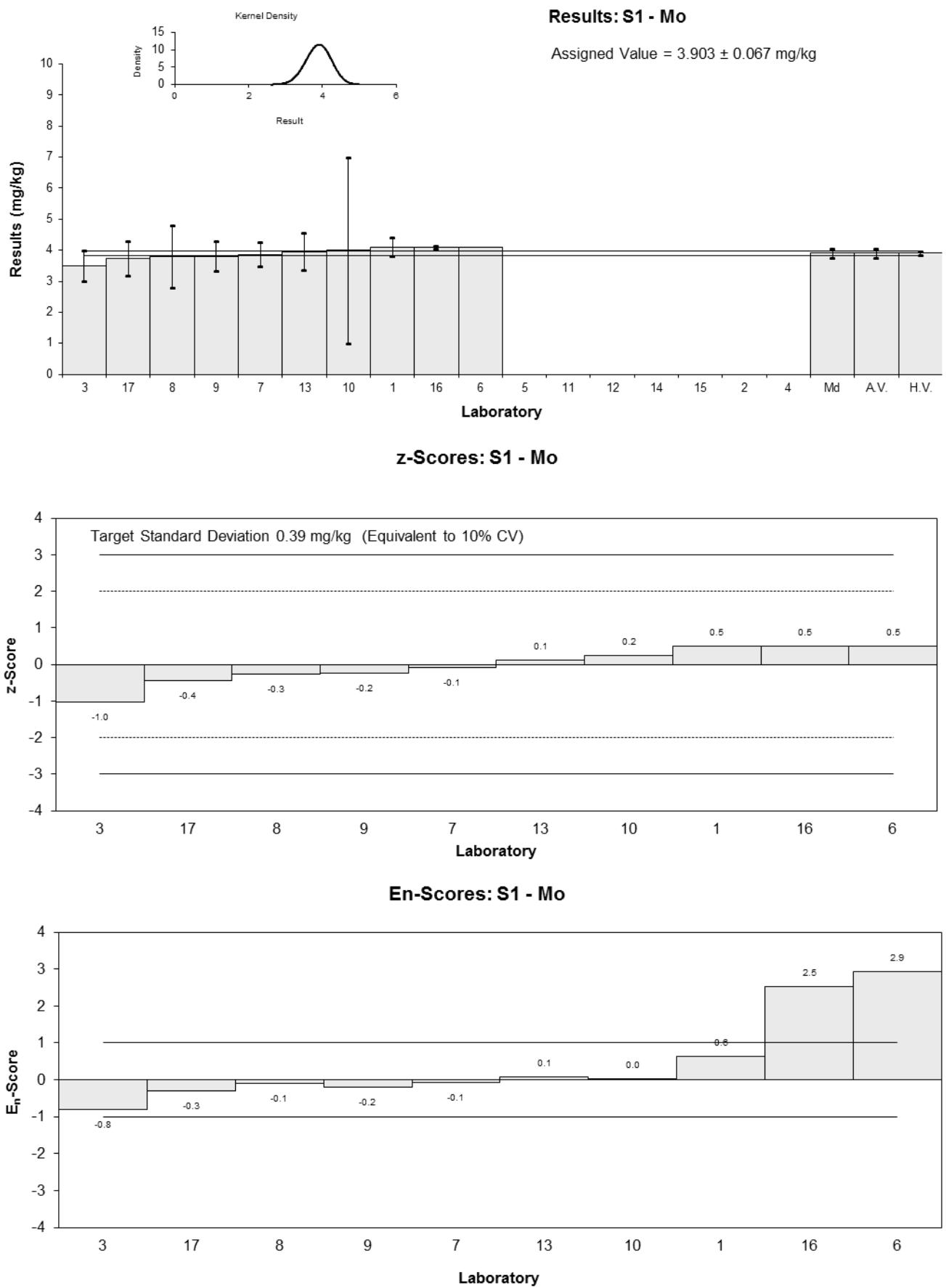


Figure 21

Table 28

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Na
Units	mg/kg

Participant Results

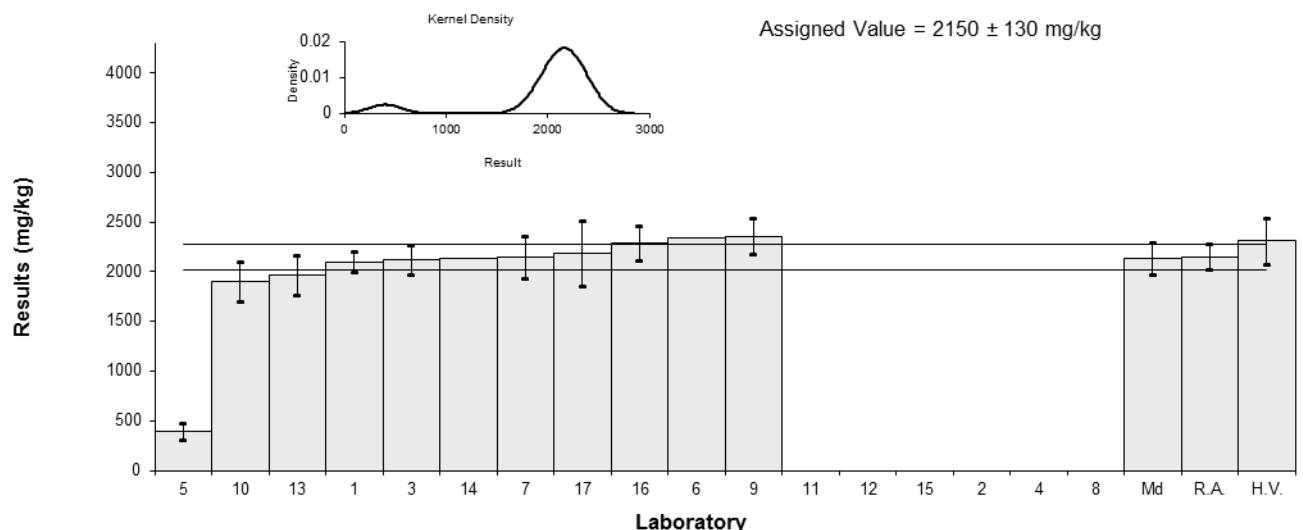
Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	2100	100	-0.23	-0.30
2	NT	NT		
3	2118	148	-0.15	-0.16
4	NT	NT		
5	396.4	83	-8.16	-11.37
6	2336	NR	0.87	1.43
7	2150	215	0.00	0.00
8	NT	NT		
9	2360	178	0.98	0.95
10	1900	200	-1.16	-1.05
11	NT	NT		
12	NT	NT		
13	1970	200	-0.84	-0.75
14	2130.458	NR	-0.09	-0.15
15	NT	NT		
16	2290	180	0.65	0.63
17	2183	327	0.15	0.09

Statistics

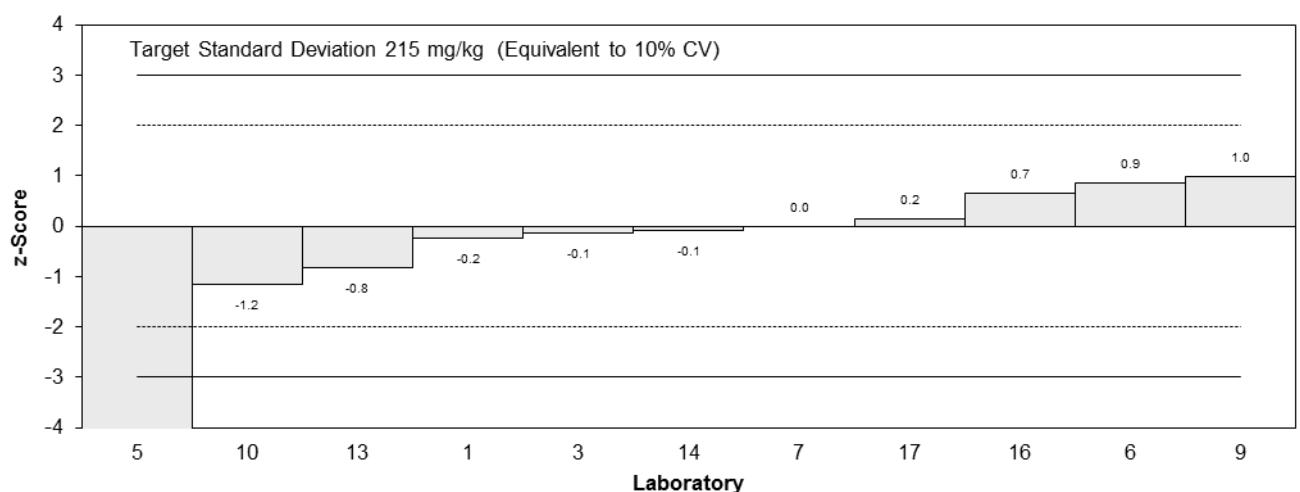
Assigned Value*	2150	130
Spike	Not Spiked	
Homogeneity Value	2310	230
Robust Average	2120	150
Median	2130	160
Mean	1990	
N	11	
Max.	2360	
Min.	396.4	
Robust SD	168	
Robust CV	7.9%	

*Robust Average excluding Laboratory 5.

Results: S1 - Na



z-Scores: S1 - Na



En-Scores: S1 - Na

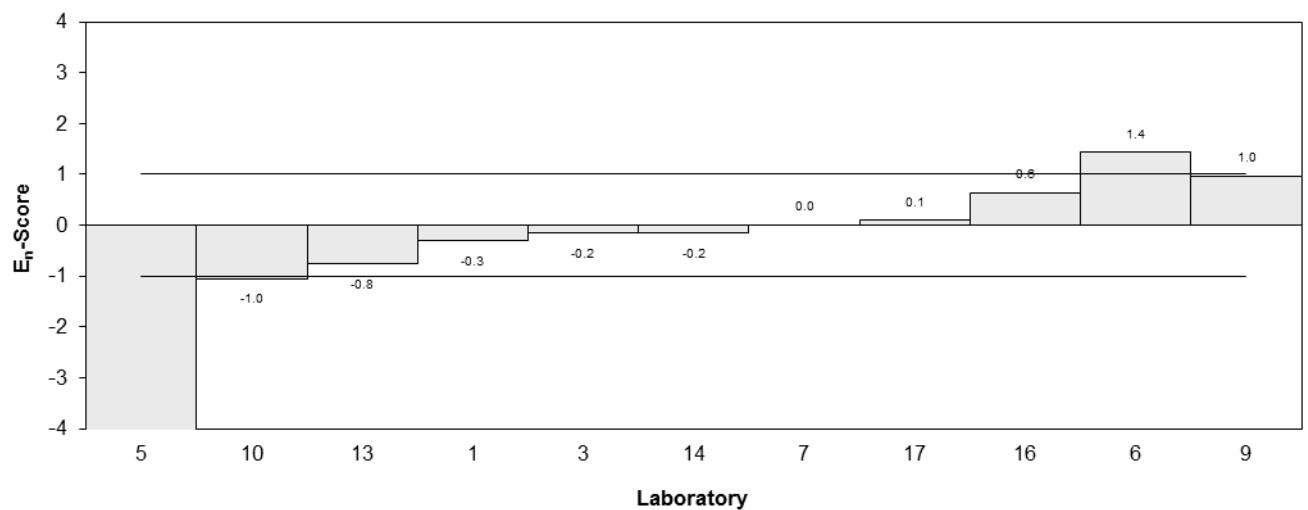


Figure 22

Table 29

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Ni
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.57	0.03	-0.97	-3.09
2	NT	NT		
3	1.45	0.34	5.24	2.17
4	NT	NT		
5	NR	NR		
6	0.80	NR	0.65	2.79
7	0.627	0.063	-0.57	-1.14
8	<1.0	NR		
9	0.55	0.10	-1.12	-1.50
10	0.9	0.9	1.36	0.21
11	NT	NT		
12	NT	NT		
13	0.57	0.1	-0.97	-1.31
14	0.697	NR	-0.08	-0.33
15	NT	NT		
16	0.611	0.07	-0.69	-1.25
17	0.6	0.09	-0.76	-1.13

Statistics

Assigned Value*	0.708	0.033
Spike	Not Spiked	
Homogeneity Value	0.708	0.033
Robust Average	0.69	0.13
Median	0.619	0.063
Mean	0.738	
N	10	
Max.	1.45	
Min.	0.55	
Robust SD	0.16	
Robust CV	23%	

*Reference Value measured by d-IDMS.

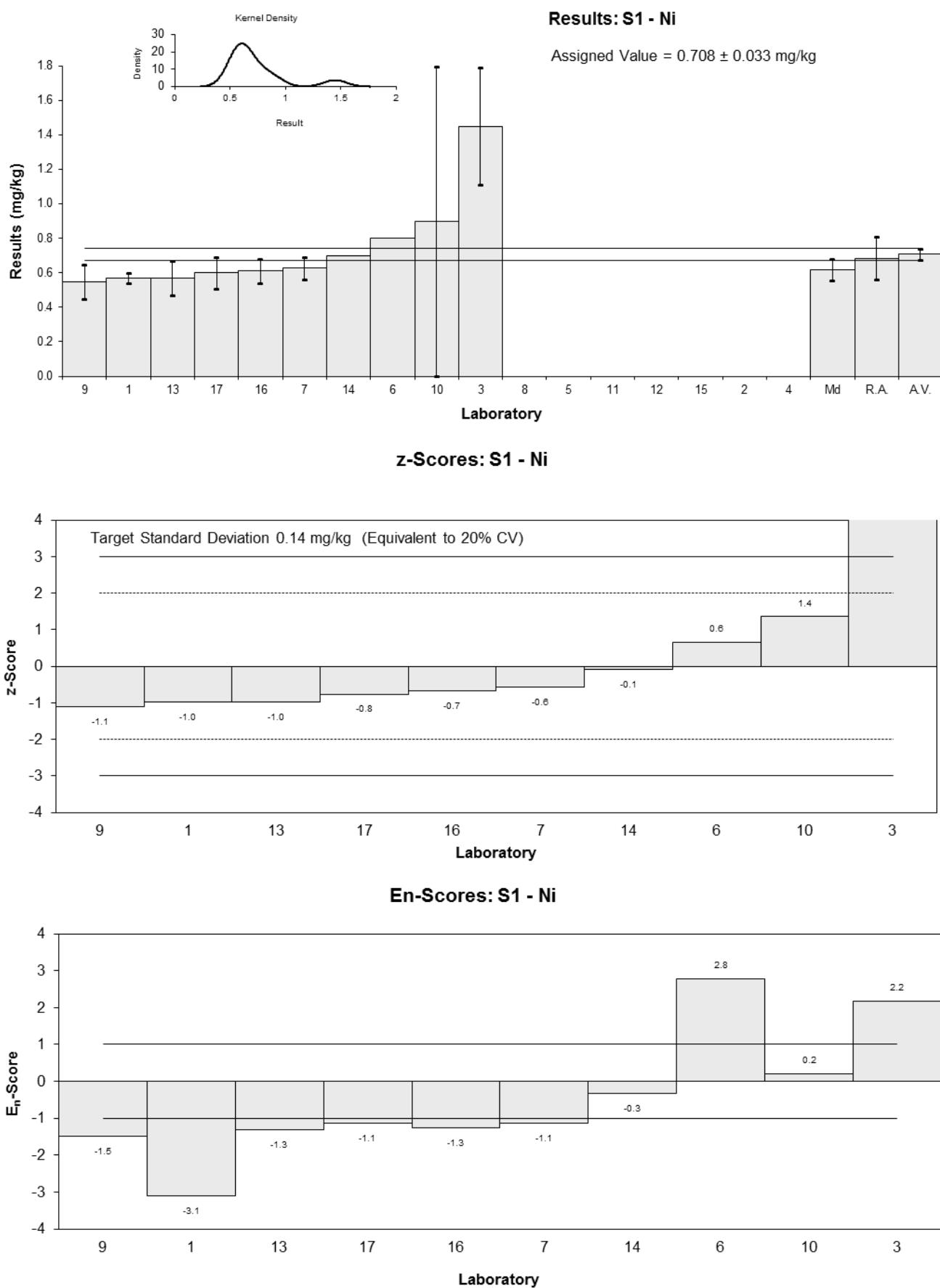


Figure 23

Table 30

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	P
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	11700	500	-0.25	-0.32
2	NT	NT		
3	11608	801	-0.33	-0.34
4	NT	NT		
5	NR	NR		
6	13550	NR	1.29	1.91
7	11000	1000	-0.83	-0.78
8	12900	3230	0.75	0.27
9	11920	679	-0.07	-0.08
10	12000	1000	0.00	0.00
11	NT	NT		
12	NT	NT		
13	11300	1000	-0.58	-0.54
14	NT	NT		
15	NT	NT		
16	13150	300	0.96	1.33
17	10943	1641	-0.88	-0.58

Statistics

Assigned Value	12000	810
Spike	Not Spiked	
Homogeneity Value	12400	1200
Robust Average	12000	810
Median	11800	700
Mean	12007	
N	10	
Max.	13550	
Min.	10943	
Robust SD	1027	
Robust CV	8.6%	

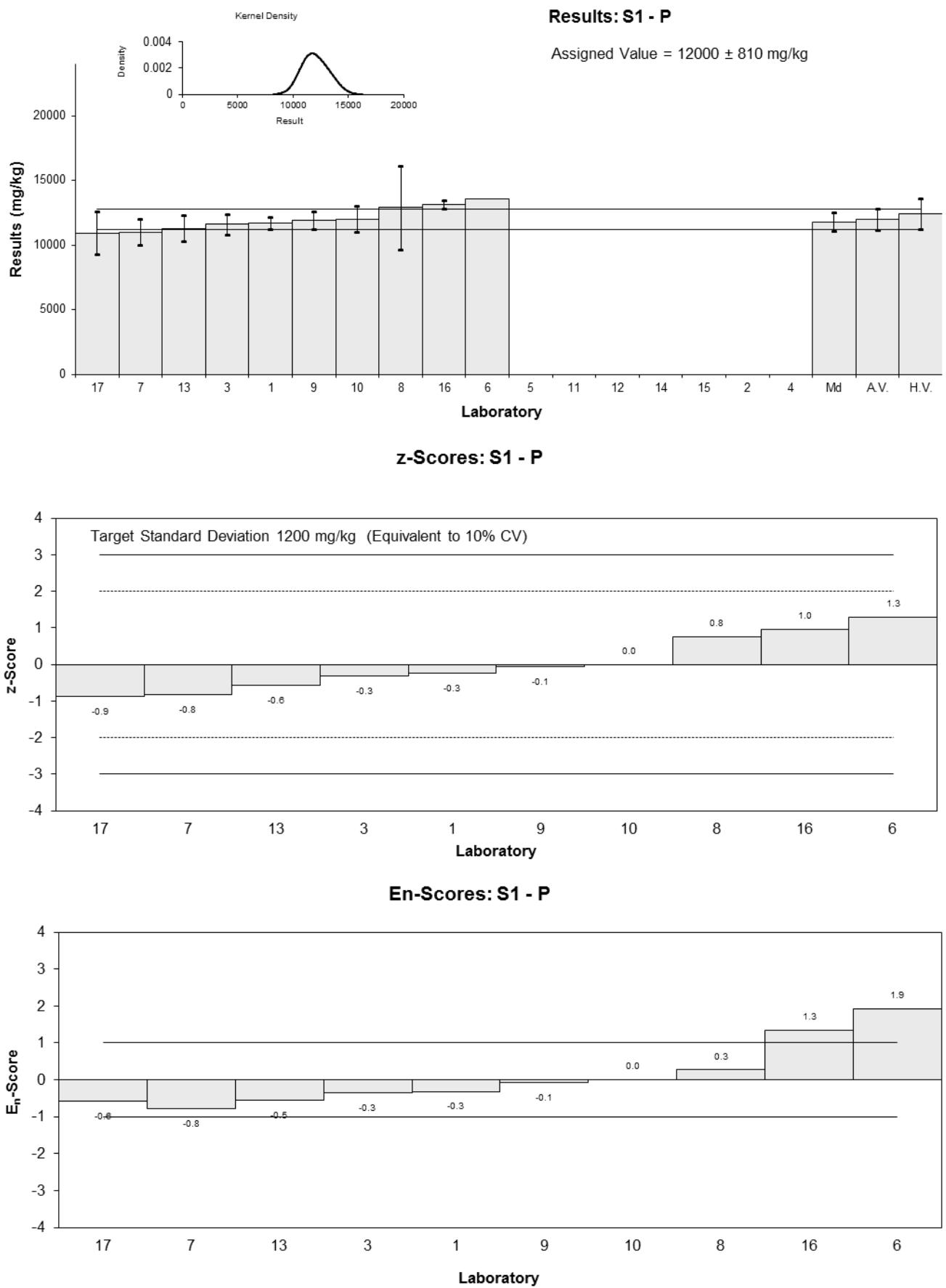


Figure 24

Table 31

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Pb
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.79	0.08	-0.06	-0.06
2	NT	NT		
3	0.76	0.12	-0.44	-0.28
4	NT	NT		
5	NR	NR		
6	0.80	NR	0.06	0.18
7	0.763	0.076	-0.40	-0.40
8	<1.0	NR		
9	0.79	0.14	-0.06	-0.04
10	<2	NR		
11	NT	NT		
12	NT	NT		
13	0.82	0.1	0.31	0.24
14	0.765	NR	-0.38	-1.07
15	NT	NT		
16	0.871	0.006	0.96	2.65
17	0.82	0.12	0.31	0.20

Statistics

Assigned Value	0.795	0.028
Spike	Not Spiked	
Homogeneity Value	0.875	0.088
Robust Average	0.795	0.028
Median	0.790	0.031
Mean	0.798	
N	9	
Max.	0.871	
Min.	0.76	
Robust SD	0.034	
Robust CV	4.2%	

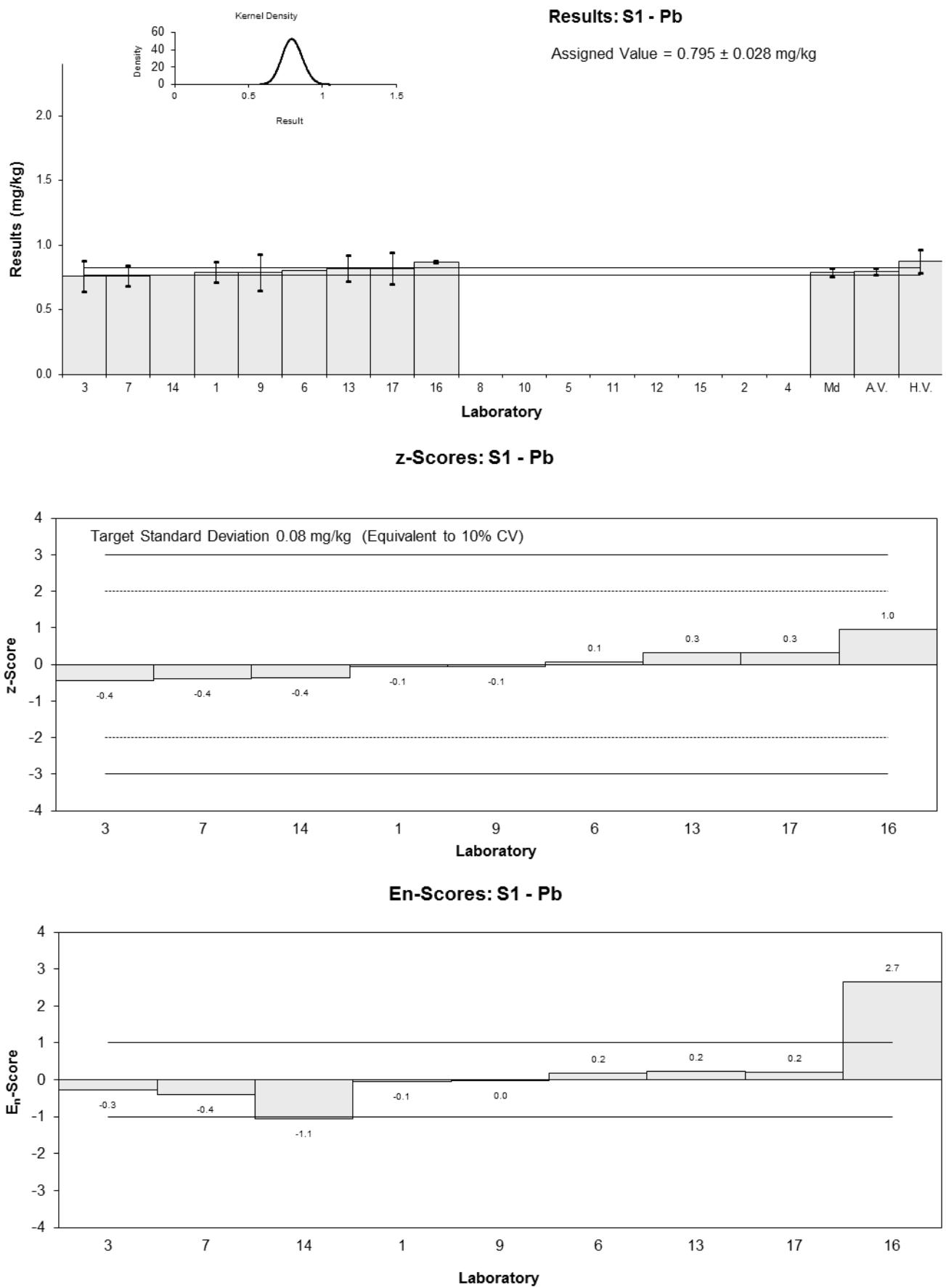


Figure 25

Table 32

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Sb
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.37	0.03	-0.49	-0.45
2	NT	NT		
3	0.40	0.06	0.28	0.16
4	NT	NT		
5	NR	NR		
6	0.46	NR	1.83	2.37
7	0.367	0.037	-0.57	-0.46
8	NT	NT		
9	0.37	0.11	-0.49	-0.17
10	<2	NR		
11	NT	NT		
12	NT	NT		
13	0.41	0.2	0.54	0.10
14	0.365	NR	-0.62	-0.80
15	NT	NT		
16	0.425	0.015	0.93	1.07
17	0.35	0.05	-1.00	-0.67

Statistics

Assigned Value	0.389	0.030
Spike	Not Spiked	
Homogeneity Value	0.413	0.050
Robust Average	0.389	0.030
Median	0.370	0.023
Mean	0.391	
N	9	
Max.	0.46	
Min.	0.35	
Robust SD	0.036	
Robust CV	9.3%	

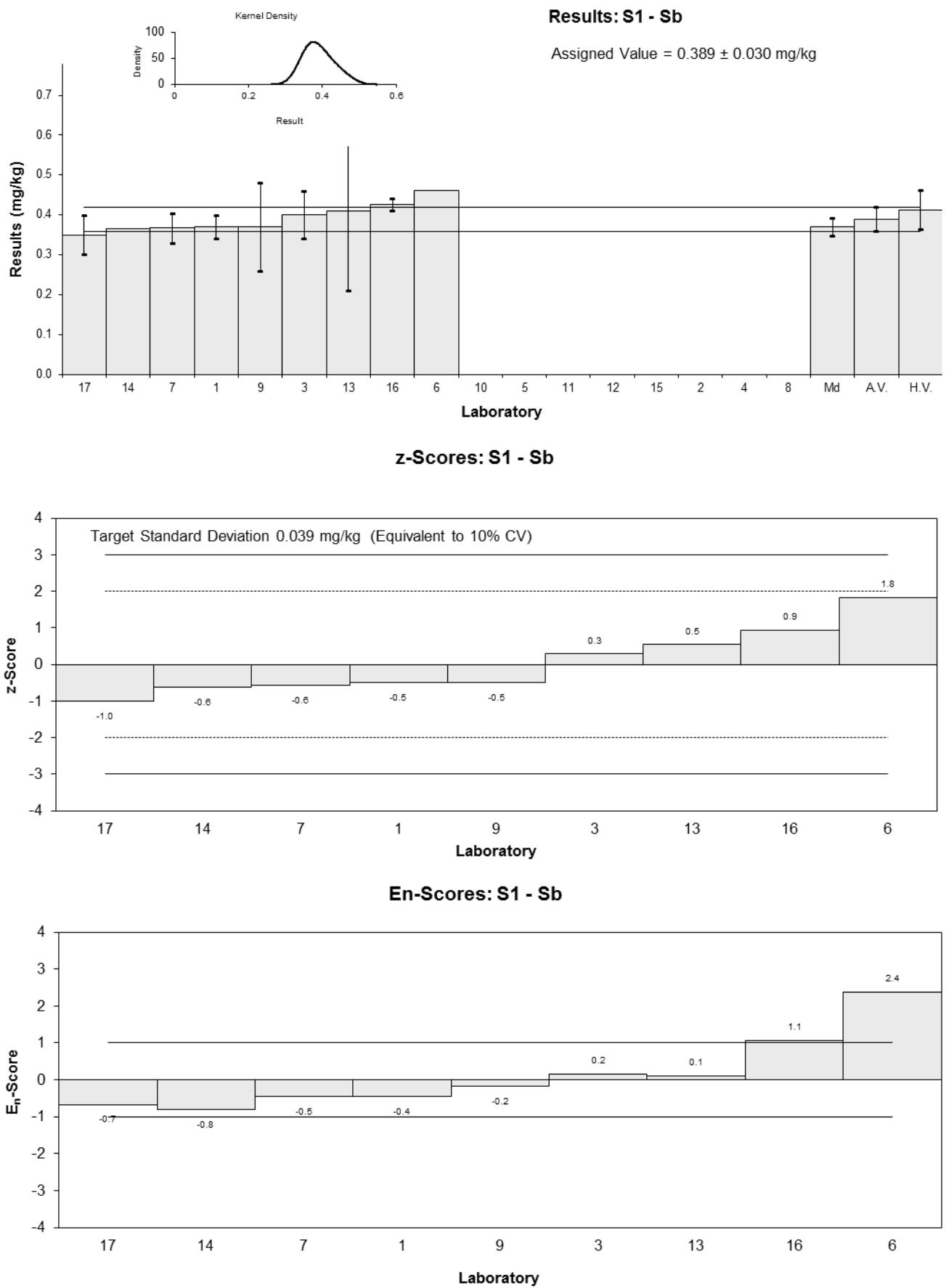


Figure 26

Table 33

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Se
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	1.7	0.1	0.18	0.15
2	NT	NT		
3	1.4	0.2	-0.73	-0.55
4	NT	NT		
5	NR	NR		
6	1.84	NR	0.61	0.51
7	2.20	0.22	1.71	1.25
8	<5.0	NR		
9	1.37	0.29	-0.82	-0.56
10	<4	NR		
11	NT	NT		
12	NT	NT		
13	1.00	0.4	-1.95	-1.15
14	1.56	NR	-0.24	-0.21
15	NT	NT		
16	1.464	0.03	-0.54	-0.45
17	2.27	0.34	1.92	1.22

Statistics

Assigned Value	1.64	0.39
Spike	Not Spiked	
Homogeneity Value	1.49	0.30
Robust Average	1.64	0.39
Median	1.56	0.22
Mean	1.64	
N	9	
Max.	2.27	
Min.	1	
Robust SD	0.46	
Robust CV	28%	

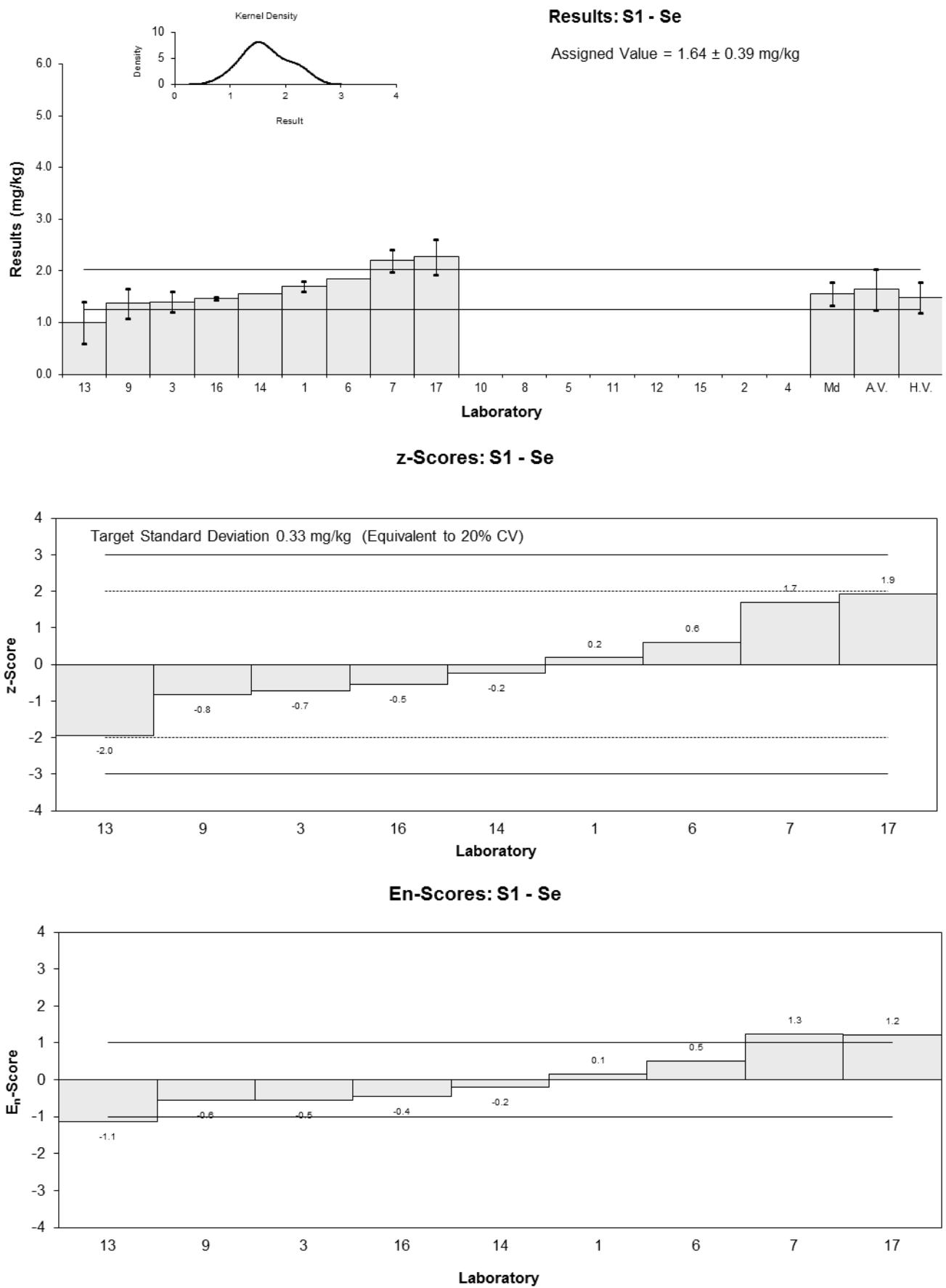


Figure 27

Table 34

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Sn
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	0.045	0.006
2	NT	NT
3	NR	NR
4	NT	NT
5	NR	NR
6	0.90	NR
7	0.061	0.006
8	NT	NT
9	<0.1	NR
10	<2	NR
11	NT	NT
12	NT	NT
13	<0.5	NR
14	0.067	NR
15	NT	NT
16	NR	NR
17	0.11	0.02

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.053	0.011
Robust Average	0.104	0.098
Median	0.067	0.041
Mean	0.237	
N	5	
Max.	0.9	
Min.	0.045	
Robust SD	0.088	
Robust CV	84%	

Results: S1 - Sn

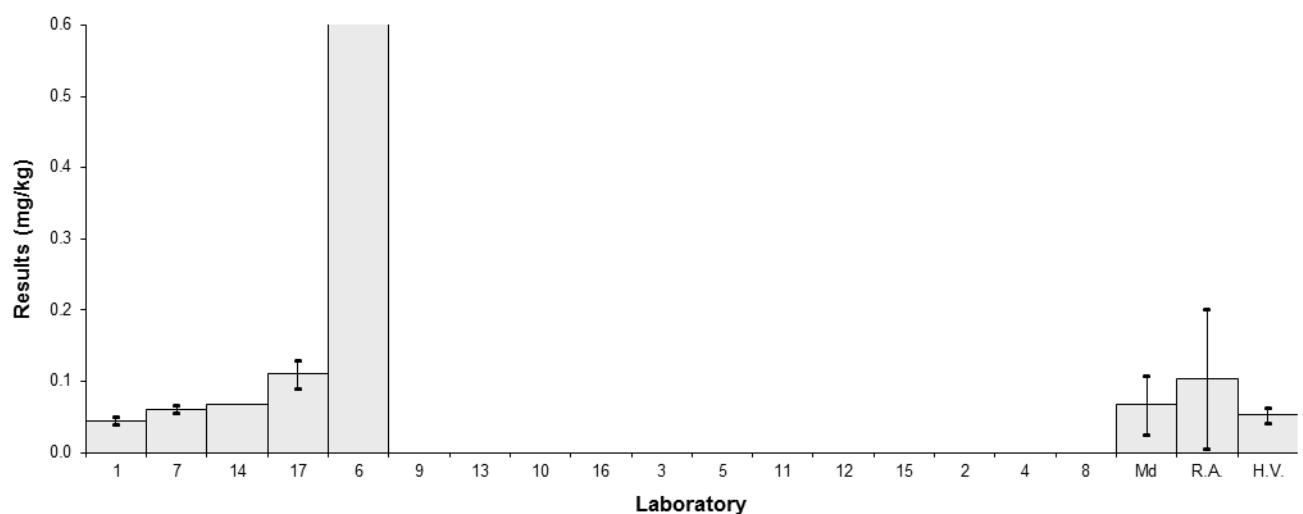


Figure 28

Table 35

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Sr
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.65	0.04	0.02	0.02
2	NT	NT		
3	0.62	0.07	-0.45	-0.39
4	NT	NT		
5	NR	NR		
6	NT	NT		
7	0.665	0.067	0.25	0.22
8	NT	NT		
9	0.68	0.16	0.48	0.19
10	<1	NR		
11	NT	NT		
12	NT	NT		
13	0.63	0.1	-0.29	-0.18
14	NT	NT		
15	NT	NT		
16	NR	NR		
17	0.65	0.10	0.02	0.01

Statistics

Assigned Value	0.649	0.026
Spike	Not Spiked	
Homogeneity Value	0.646	0.065
Robust Average	0.649	0.026
Median	0.650	0.027
Mean	0.649	
N	6	
Max.	0.68	
Min.	0.62	
Robust SD	0.026	
Robust CV	3.9%	

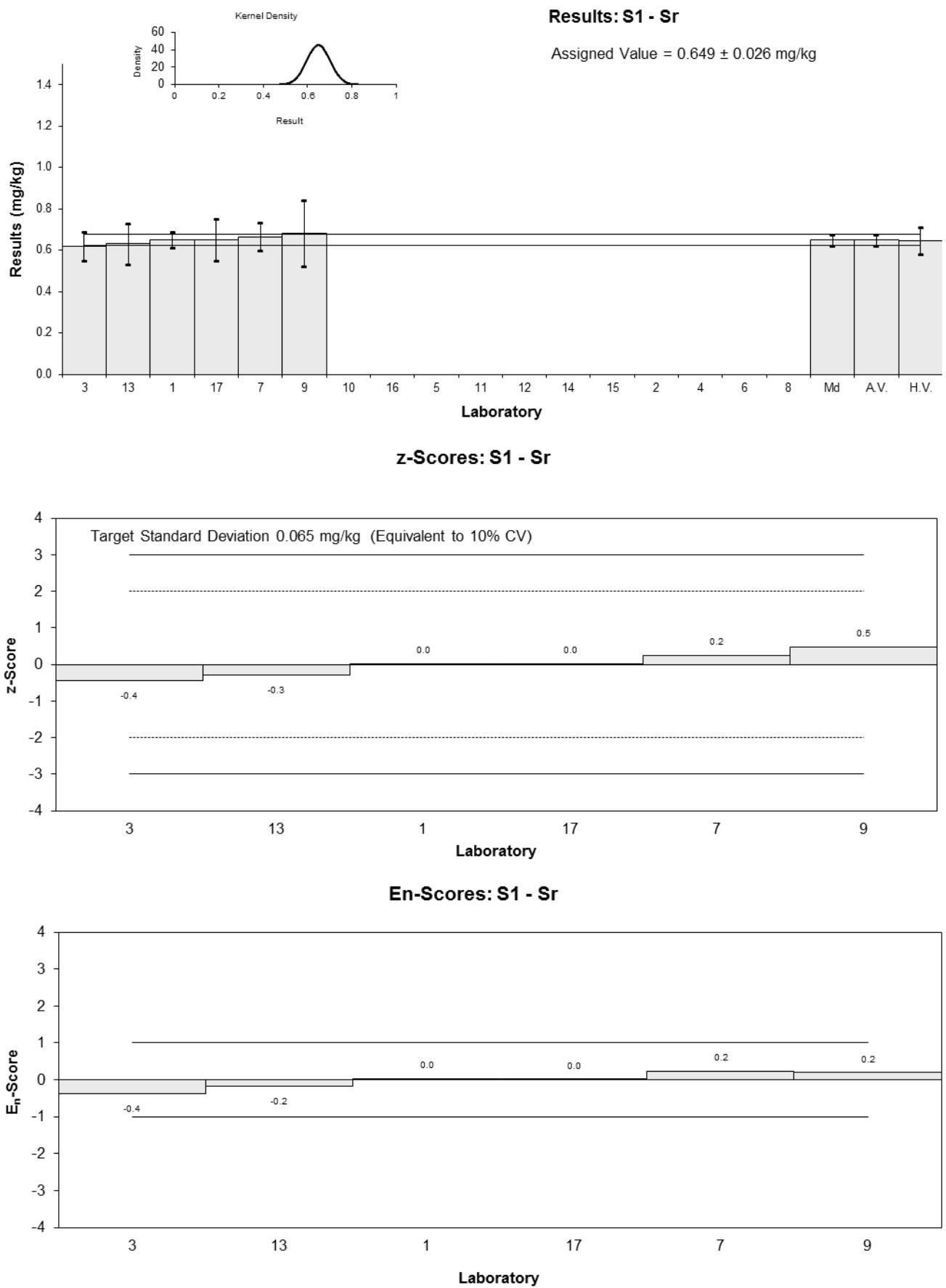


Figure 29

Table 36

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Th
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	0.37	0.04
2	NT	NT
3	0.33	0.05
4	NT	NT
5	NR	NR
6	NT	NT
7	NT	NT
8	NT	NT
9	NT	NT
10	NT	NT
11	NT	NT
12	NT	NT
13	0.39	0.2
14	NT	NT
15	NT	NT
16	NR	NR
17	0.34	0.05

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.268	0.027
Robust Average	0.358	0.039
Median	0.355	0.047
Mean	0.358	
N	4	
Max.	0.39	
Min.	0.33	
Robust SD	0.031	
Robust CV	8.7%	

Results: S1 - Th

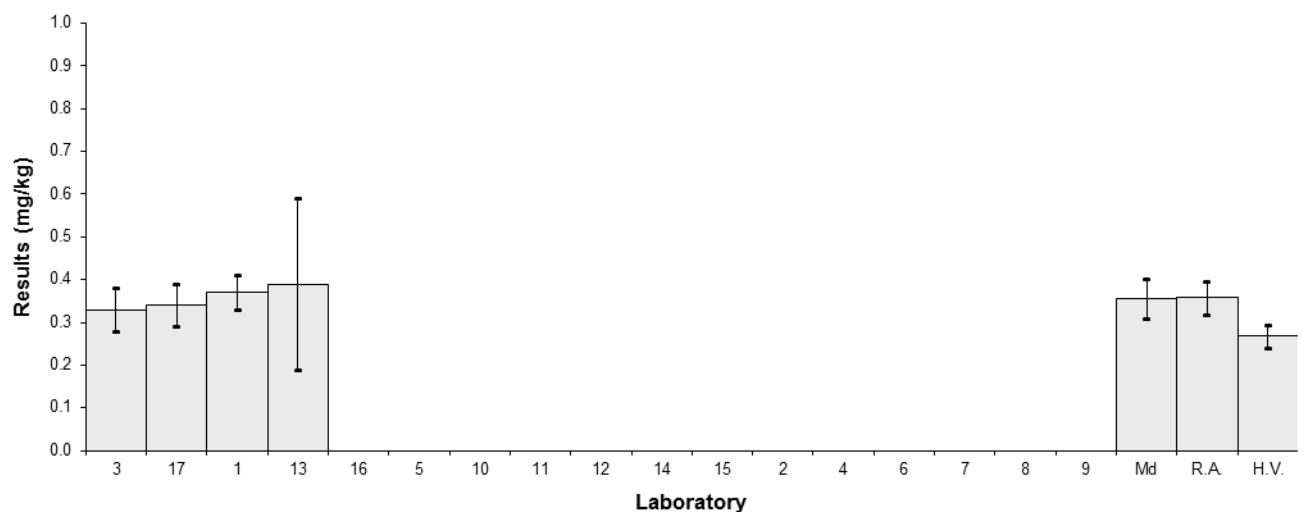


Figure 30

Table 37

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	U
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.35	0.03	-0.91	-0.91
2	NT	NT		
3	0.39	0.05	0.13	0.09
4	NT	NT		
5	NR	NR		
6	NT	NT		
7	0.395	0.040	0.26	0.21
8	NT	NT		
9	0.385	0.044	0.00	0.00
10	NT	NT		
11	NT	NT		
12	NT	NT		
13	0.37	0.08	-0.39	-0.18
14	NT	NT		
15	NT	NT		
16	0.477	0.05	2.39	1.66
17	0.38	0.06	-0.13	-0.08

Statistics

Assigned Value	0.385	0.024
Spike	Not Spiked	
Homogeneity Value	0.422	0.051
Robust Average	0.385	0.024
Median	0.385	0.014
Mean	0.392	
N	7	
Max.	0.48	
Min.	0.35	
Robust SD	0.026	
Robust CV	6.6%	

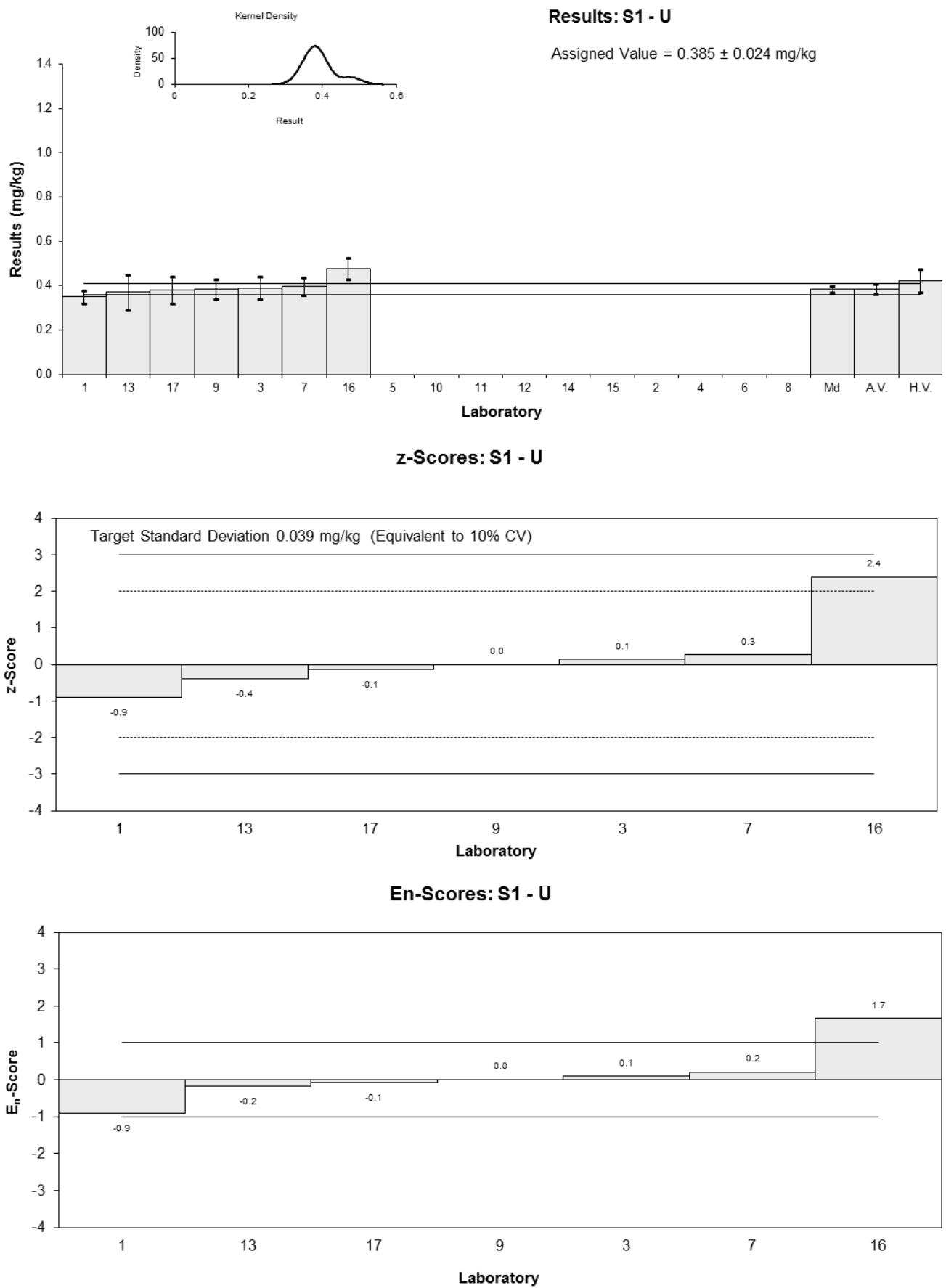


Figure 31

Table 38

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	V
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.37	0.03	-1.23	-1.06
2	NT	NT		
3	0.45	0.06	0.66	0.39
4	NT	NT		
5	NR	NR		
6	<0.1	NR		
7	0.396	0.04	-0.62	-0.47
8	<1.0	NR		
9	0.43	0.09	0.19	0.08
10	0.4	0.4	-0.52	-0.05
11	NT	NT		
12	NT	NT		
13	0.43	0.08	0.19	0.09
14	NT	NT		
15	NT	NT		
16	0.861	0.08	10.40	4.93
17	0.48	0.07	1.37	0.72

Statistics

Assigned Value*	0.422	0.039
Spike	Not Spiked	
Homogeneity Value	0.455	0.055
Robust Average	0.434	0.047
Median	0.430	0.040
Mean	0.477	
N	8	
Max.	0.86	
Min.	0.37	
Robust SD	0.042	
Robust CV	9.6%	

*Robust Average excluding Laboratory 16.

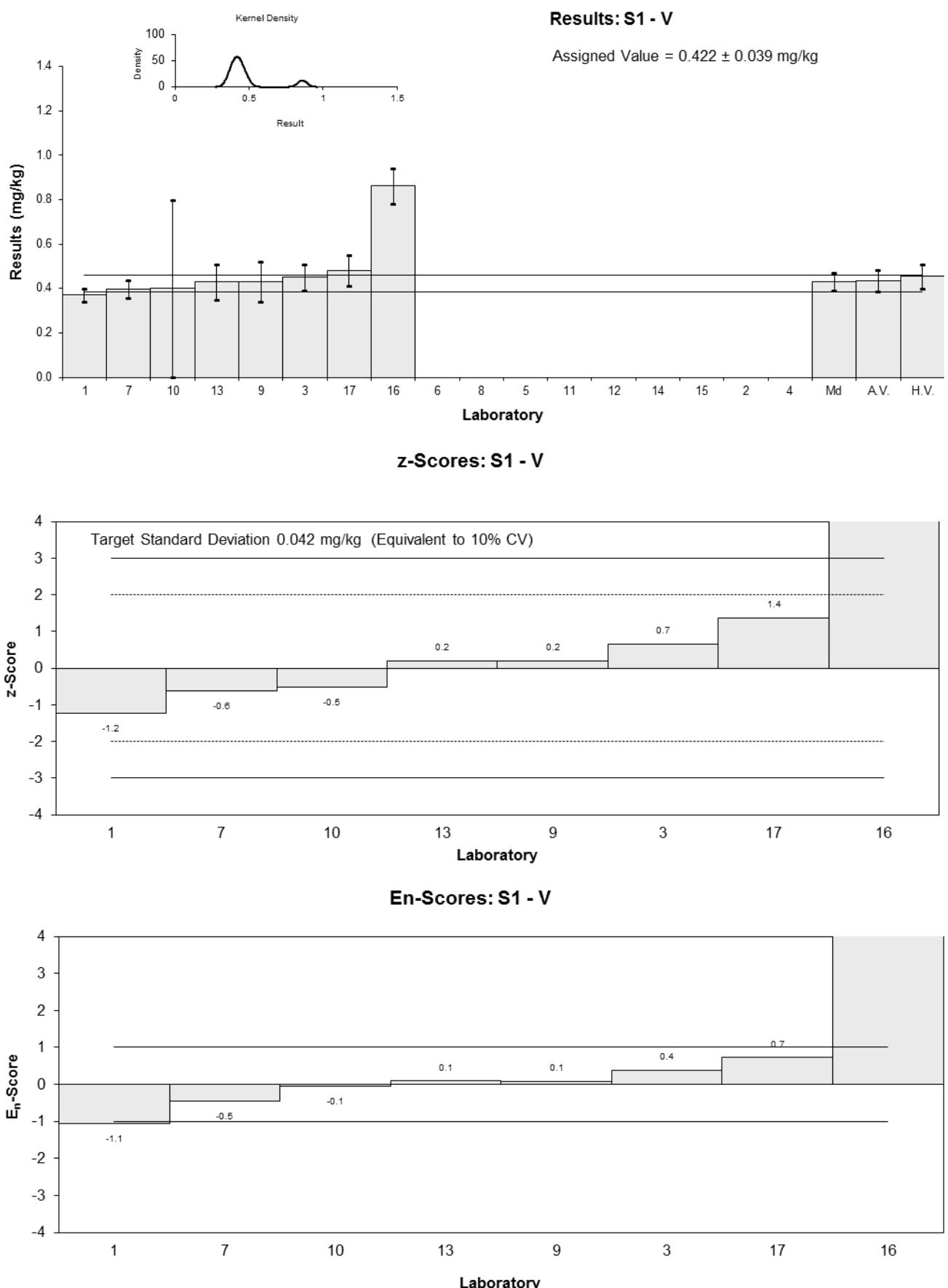


Figure 32

Table 39

Sample Details

Sample No.	S1
Matrix.	Bovine Liver
Analyte.	Zn
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	160	6.0	0.13	0.28
2	NT	NT		
3	153	20	-0.32	-0.25
4	NT	NT		
5	156.15	3	-0.12	-0.37
6	156.6	NR	-0.09	-0.35
7	163	16.3	0.32	0.30
8	152	21	-0.38	-0.28
9	163	12	0.32	0.40
10	150	20	-0.51	-0.39
11	NT	NT		
12	NT	NT		
13	158	20	0.00	0.00
14	164.504	NR	0.41	1.63
15	NT	NT		
16	159	8	0.06	0.11
17	159	24	0.06	0.04

Statistics

Assigned Value	158	4
Spike	Not Spiked	
Homogeneity Value	165	20
Robust Average	158	4
Median	159	3
Mean	158	
N	12	
Max.	165	
Min.	150	
Robust SD	5.14	
Robust CV	3.3%	

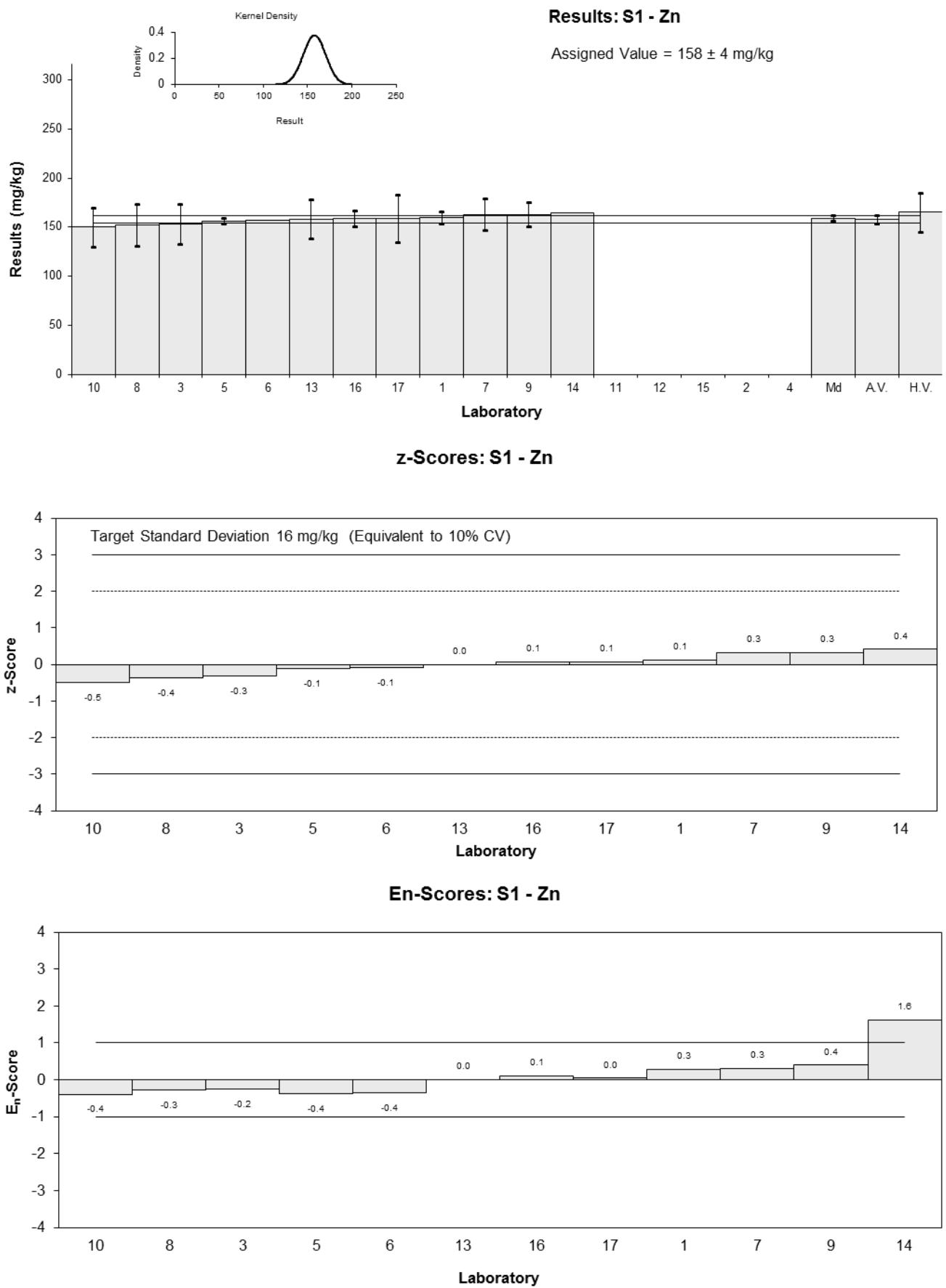


Figure 33

Table 40

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	220	30
2	NT	NT
3	385	46
4	NT	NT
5	NR	NR
6	NT	NT
7	191	19.1
8	NT	NT
9	215	24
10	NT	NT
11	229	64
12	185	37
13	276	50
14	422.174	NR
15	380	52
16	216	6
17	142	21

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	479	96
Robust Average	260	79
Median	220	35
Mean	260	
N	11	
Max.	422	
Min.	142	
Robust SD	105	
Robust CV	40%	

Results: S2 - Al

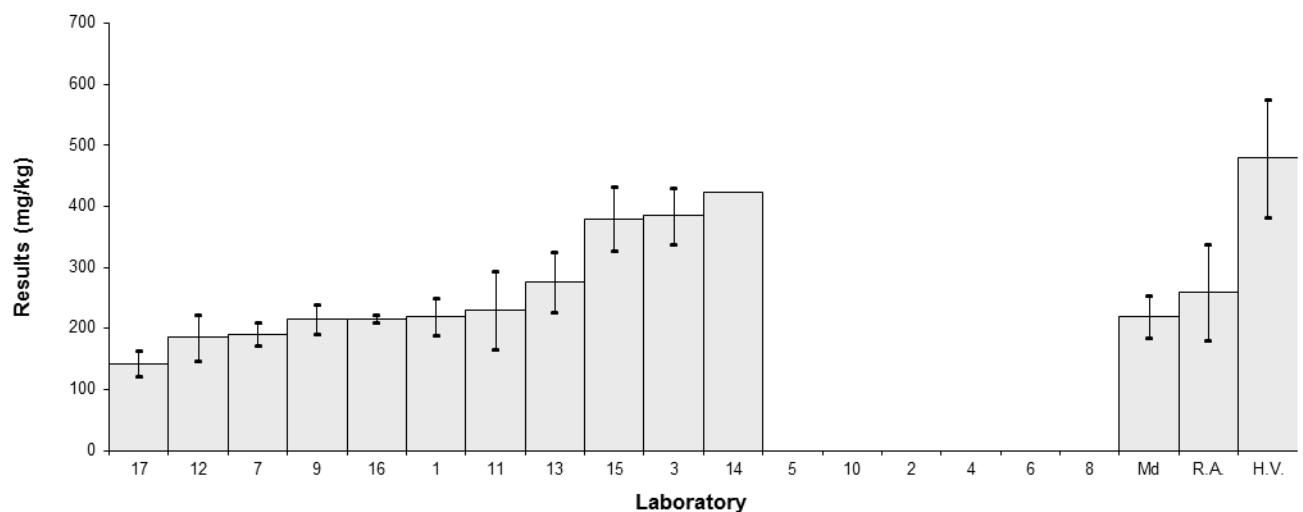


Figure 34

Table 41

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.48	0.03	-0.64	-1.17
2	0.57	0.028	0.18	0.34
3	0.56	0.2	0.09	0.05
4	0.501	0.14	-0.45	-0.33
5	NR	NR		
6	0.5	NR	-0.45	-0.96
7	0.714	0.071	1.49	1.86
8	1.1	0.5	5.00	1.09
9	0.51	0.05	-0.36	-0.55
10	NT	NT		
11	<2	NR		
12	0.627	0.125	0.70	0.57
13	0.63	0.1	0.73	0.71
14	0.498	NR	-0.47	-1.00
15	0.46	0.087	-0.82	-0.89
16	0.62	0.02	0.64	1.26
17	0.53	0.08	-0.18	-0.21

Statistics

Assigned Value*	0.550	0.052
Spike	Not Spiked	
Homogeneity Value	0.536	0.080
Robust Average	0.563	0.060
Median	0.545	0.048
Mean	0.593	
N	14	
Max.	1.1	
Min.	0.46	
Robust SD	0.052	
Robust CV	9.3%	

*Robust Average excluding Laboratory 8.

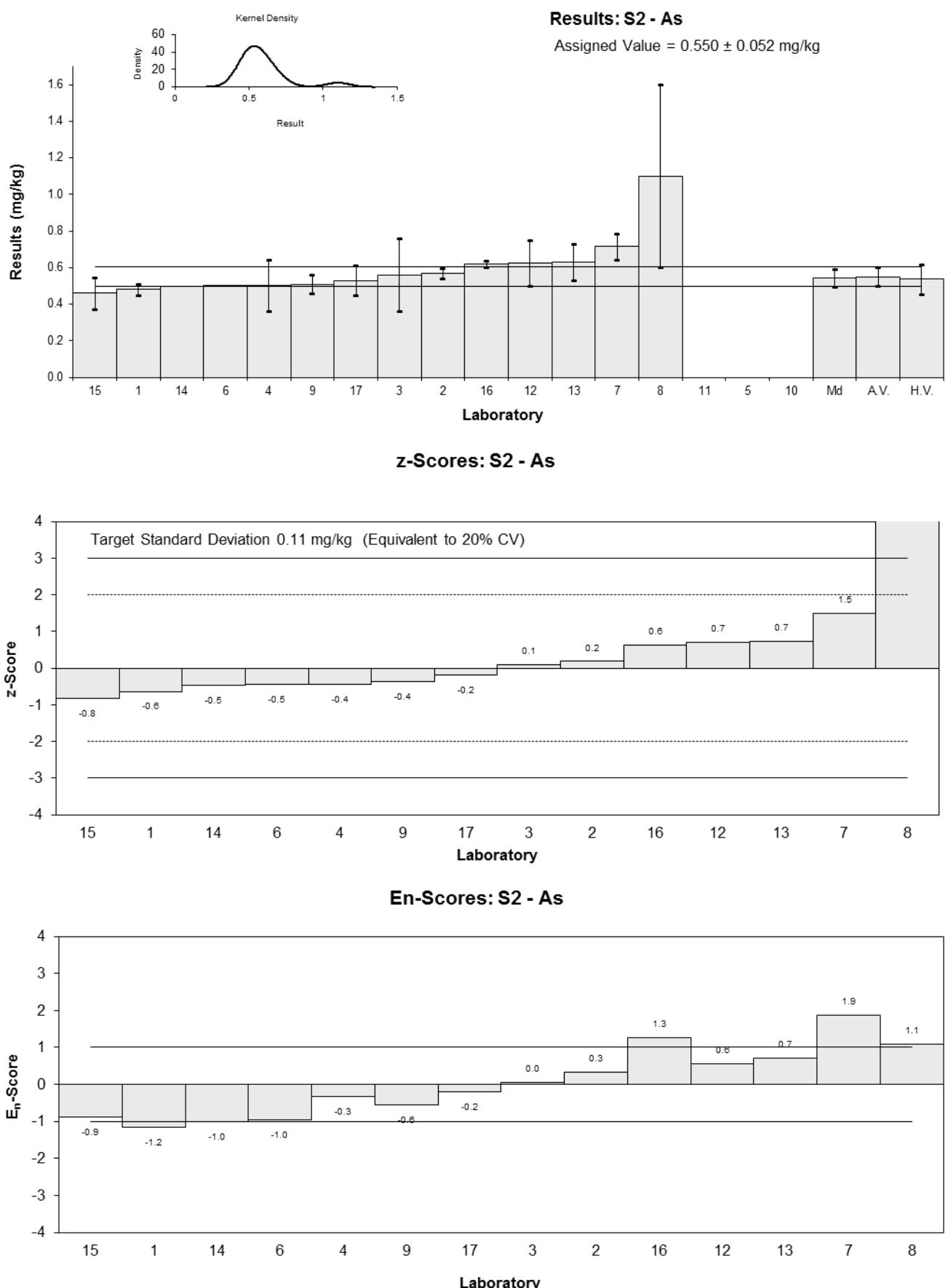


Figure 35

Table 42

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	14	1.0	0.30	0.25
2	NT	NT		
3	11.7	0.7	-0.85	-0.74
4	NT	NT		
5	NR	NR		
6	NT	NT		
7	13.4	1.34	0.00	0.00
8	NT	NT		
9	18.5	2.1	2.54	1.68
10	NT	NT		
11	11.7	2.3	-0.85	-0.53
12	13.6	2.7	0.10	0.06
13	13	3.0	-0.20	-0.11
14	NT	NT		
15	17	2.8	1.79	1.01
16	12.81	1.1	-0.29	-0.24
17	8.41	1.26	-2.48	-1.97

Statistics

Assigned Value	13.4	2.2
Spike	Not Spiked	
Homogeneity Value	13.2	2.6
Robust Average	13.4	2.2
Median	13.2	1.2
Mean	13.4	
N	10	
Max.	18.5	
Min.	8.4	
Robust SD	2.8	
Robust CV	21%	

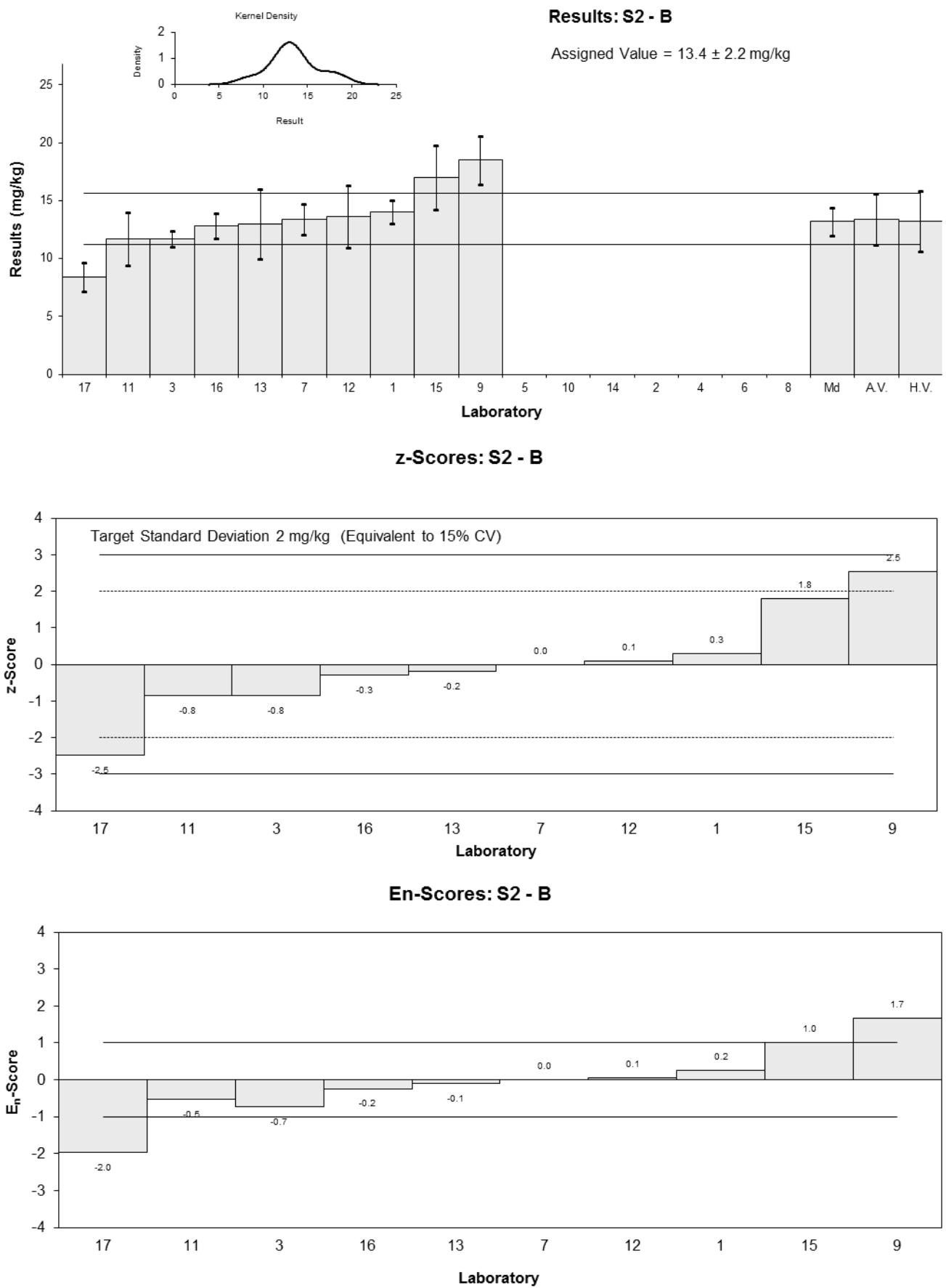


Figure 36

Table 43

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	7.1	0.6	-0.27	-0.29
2	NT	NT		
3	6.9	0.8	-0.55	-0.46
4	6.93	1.94	-0.51	-0.19
5	NR	NR		
6	7.9	NR	0.82	1.71
7	7.31	0.731	0.01	0.01
8	6.8	1.7	-0.68	-0.29
9	7.1	0.5	-0.27	-0.33
10	NT	NT		
11	7.1	1.4	-0.27	-0.14
12	7.52	1.50	0.30	0.14
13	7.50	1.00	0.27	0.19
14	8.059	NR	1.04	2.17
15	6.6	0.67	-0.96	-0.93
16	7.24	0.16	-0.08	-0.16
17	8.10	1.22	1.10	0.63

Statistics

Assigned Value	7.30	0.35
Spike	Not Spiked	
Homogeneity Value	7.64	0.76
Robust Average	7.30	0.35
Median	7.17	0.26
Mean	7.30	
N	14	
Max.	8.1	
Min.	6.6	
Robust SD	0.53	
Robust CV	7.2%	

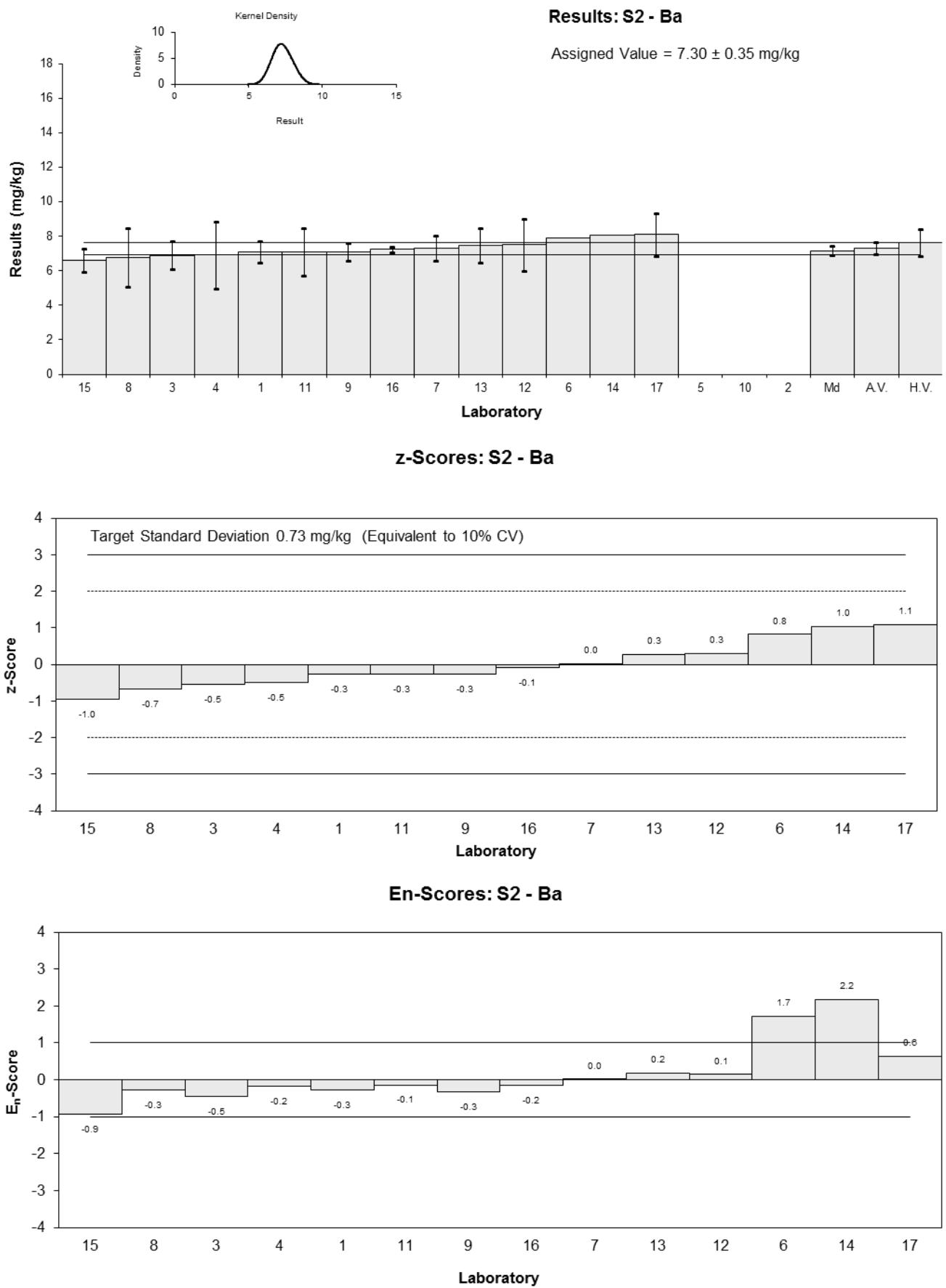


Figure 37

Table 44

Sample Details

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

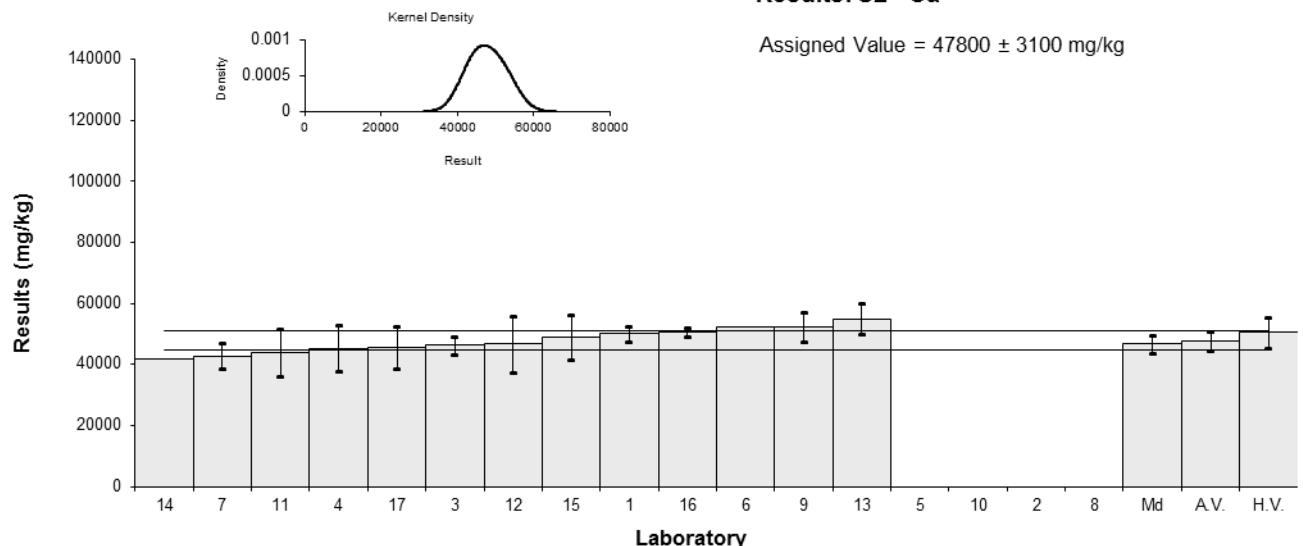
Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	50000	2500	0.46	0.55
2	NT	NT		
3	46246	2868	-0.33	-0.37
4	45300	7700	-0.52	-0.30
5	NR	NR		
6	52090	NR	0.90	1.38
7	42800	4280	-1.05	-0.95
8	NT	NT		
9	52390	4769	0.96	0.81
10	NT	NT		
11	43900	7900	-0.82	-0.46
12	46650	9330	-0.24	-0.12
13	55000	5000	1.51	1.22
14	41881.063	NR	-1.24	-1.91
15	49000	7400	0.25	0.15
16	50700	1500	0.61	0.84
17	45600	6838	-0.46	-0.29

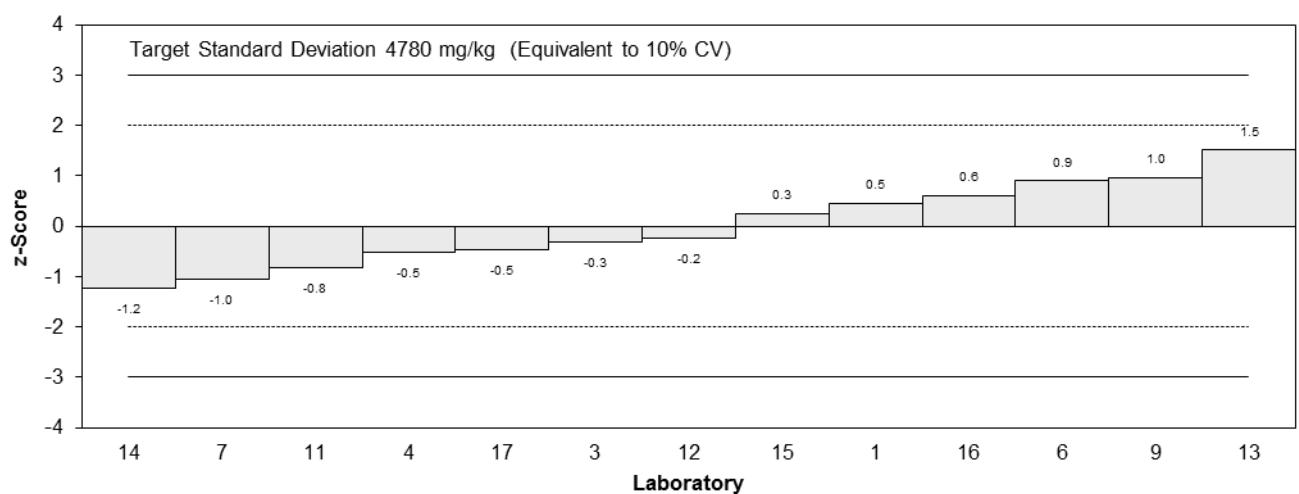
Statistics

Assigned Value	47800	3100
Spike	Not Spiked	
Homogeneity Value	50500	5100
Robust Average	47800	3100
Median	46700	3000
Mean	47800	
N	13	
Max.	55000	
Min.	41900	
Robust SD	4507	
Robust CV	9.4%	

Results: S2 - Ca



z-Scores: S2 - Ca



En-Scores: S2 - Ca

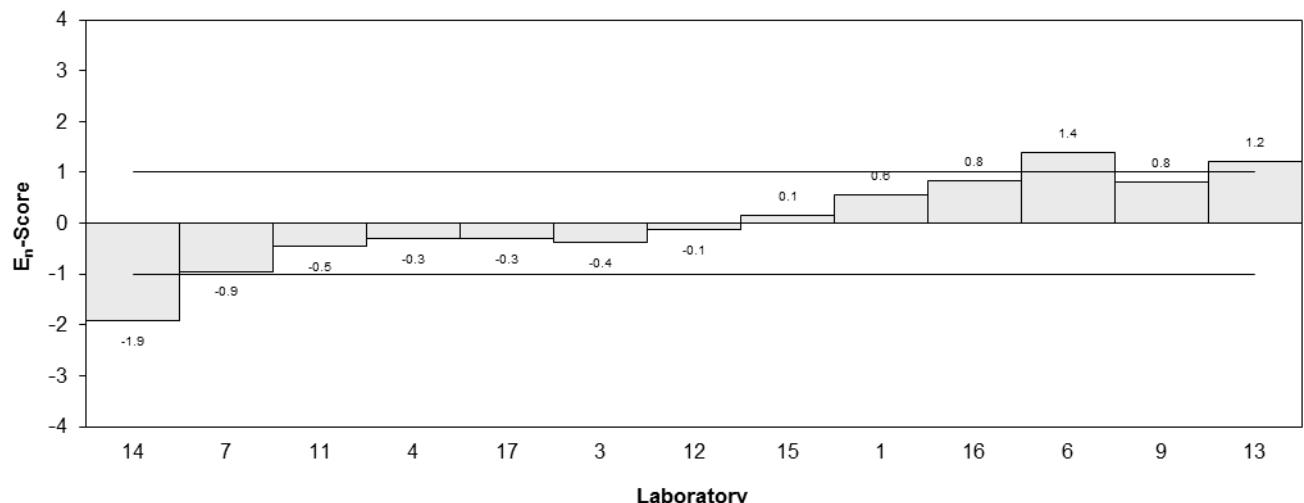


Figure 38

Table 45

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.049	0.004	-0.53	-0.75
2	0.05	0.023	-0.40	-0.14
3	0.046	0.02	-0.90	-0.35
4	0.0551	0.01	0.24	0.18
5	<0.1	0.1		
6	0.06	NR	0.85	1.74
7	0.049	0.005	-0.53	-0.66
8	<1.0	NR		
9	0.051	0.014	-0.28	-0.15
10	NT	NT		
11	<0.1	NR		
12	0.055	0.011	0.23	0.15
13	0.05	0.02	-0.40	-0.16
14	0.056	NR	0.35	0.72
15	0.060	0.0056	0.85	1.00
16	0.061	0.005	0.98	1.23
17	0.05	0.01	-0.40	-0.30

Statistics

Assigned Value	0.0532	0.0039
Spike	Not Spiked	
Homogeneity Value	0.0560	0.0060
Robust Average	0.0532	0.0039
Median	0.0510	0.0036
Mean	0.0532	
N	13	
Max.	0.061	
Min.	0.046	
Robust SD	0.0056	
Robust CV	11%	

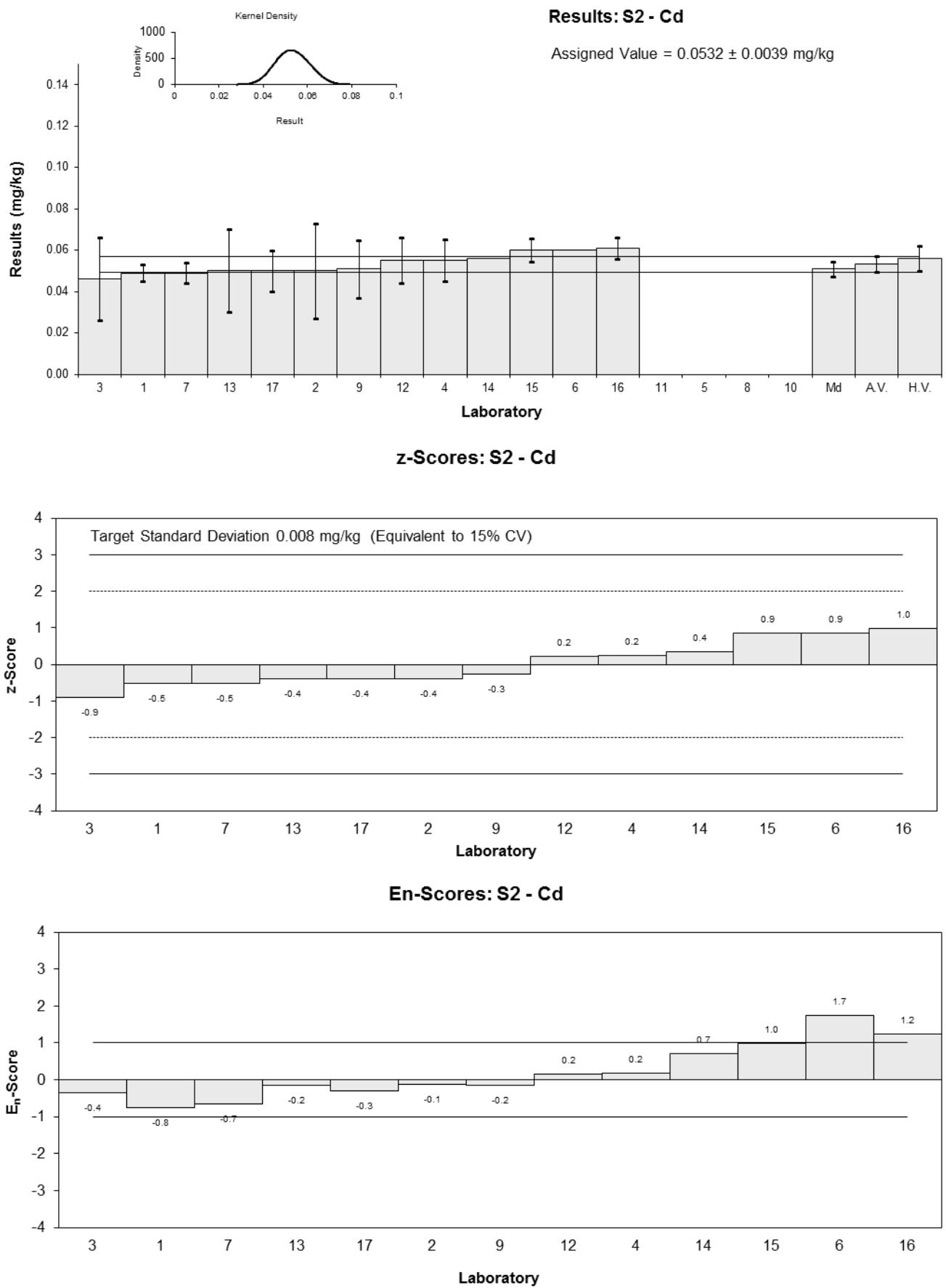


Figure 39

Table 46

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.46	0.03	-0.13	-0.17
2	NT	NT		
3	0.51	0.10	0.58	0.38
4	0.414	0.066	-0.78	-0.70
5	NR	NR		
6	0.46	NR	-0.13	-0.21
7	0.588	0.059	1.69	1.63
8	<1.0	NR		
9	0.46	0.07	-0.13	-0.11
10	NT	NT		
11	0.5	0.2	0.44	0.15
12	0.456	0.091	-0.18	-0.13
13	0.44	0.1	-0.41	-0.27
14	0.403	NR	-0.94	-1.53
15	NT	NT		
16	0.556	0.05	1.24	1.32
17	0.41	0.06	-0.84	-0.80

Statistics

Assigned Value	0.469	0.043
Spike	Not Spiked	
Homogeneity Value	0.501	0.075
Robust Average	0.469	0.043
Median	0.460	0.041
Mean	0.469	
N	12	
Max.	0.59	
Min.	0.40	
Robust SD	0.06	
Robust CV	13%	

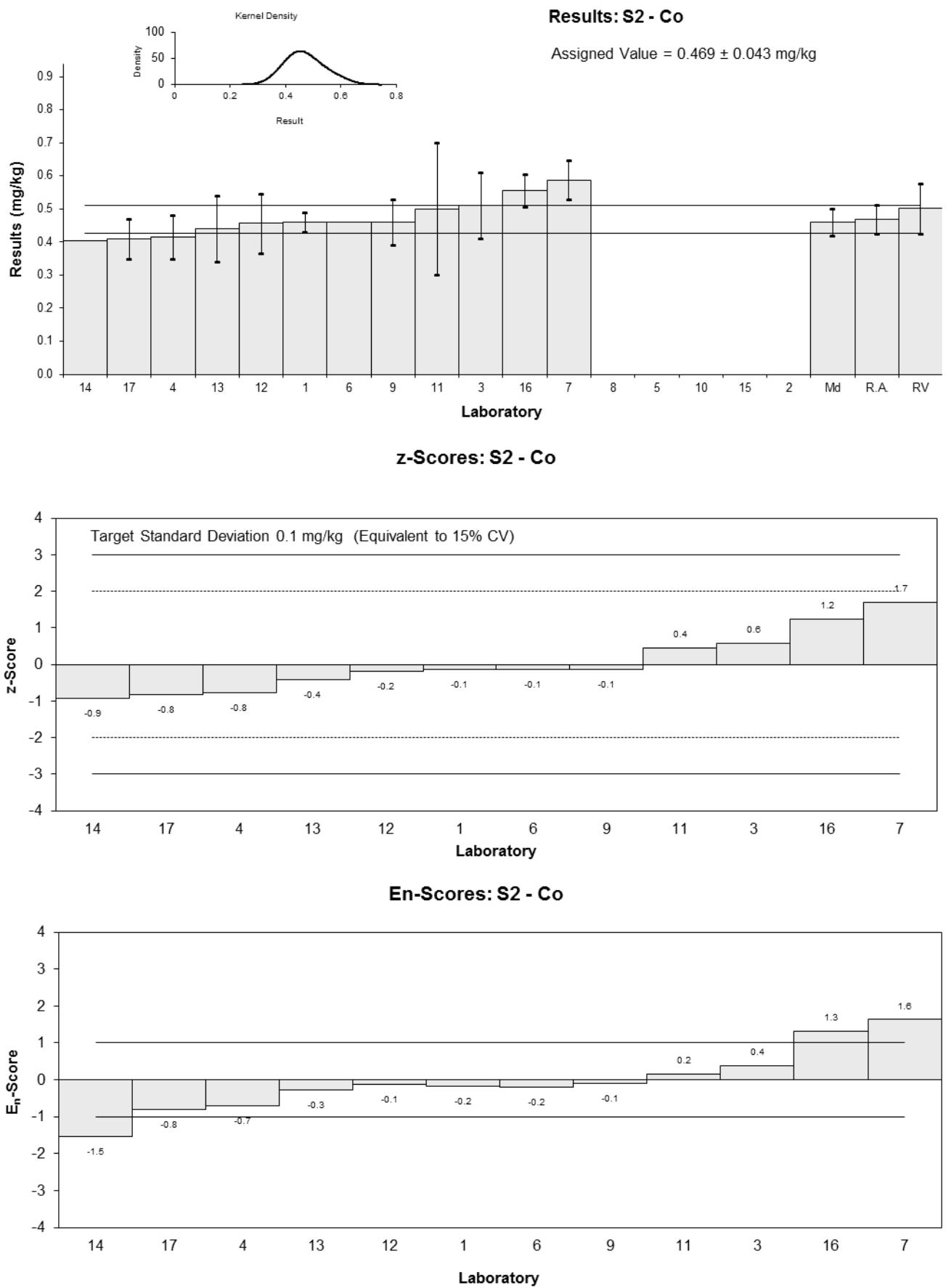


Figure 40

Table 47

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	2.3	0.2	-3.64	-7.52
2	NT	NT		
3	8.5	1.0	0.04	0.05
4	8.18	1.88	-0.15	-0.12
5	NR	NR		
6	9.2	NR	0.46	0.97
7	8.61	0.861	0.11	0.15
8	8.0	1.2	-0.26	-0.30
9	7.7	1.1	-0.43	-0.54
10	NT	NT		
11	7.0	1.8	-0.85	-0.73
12	2.28	0.46	-3.65	-6.73
13	1.77	0.4	-3.95	-7.52
14	10.047	NR	0.96	2.05
15	8.8	0.82	0.22	0.32
16	2.39	0.015	-3.58	-7.64
17	1.26	0.19	-4.25	-8.82

Statistics

Assigned Value*	8.43	0.79
Spike	Not Spiked	
Homogeneity Value	9.3	1.4
Robust Average	6.2	2.5
Median	7.85	0.98
Mean	6.2	
N	14	
Max.	10	
Min.	1.3	
Robust SD	3.7	
Robust CV	61%	

*Robust Average excluding Laboratories 1, 12, 13, 16 and 17.

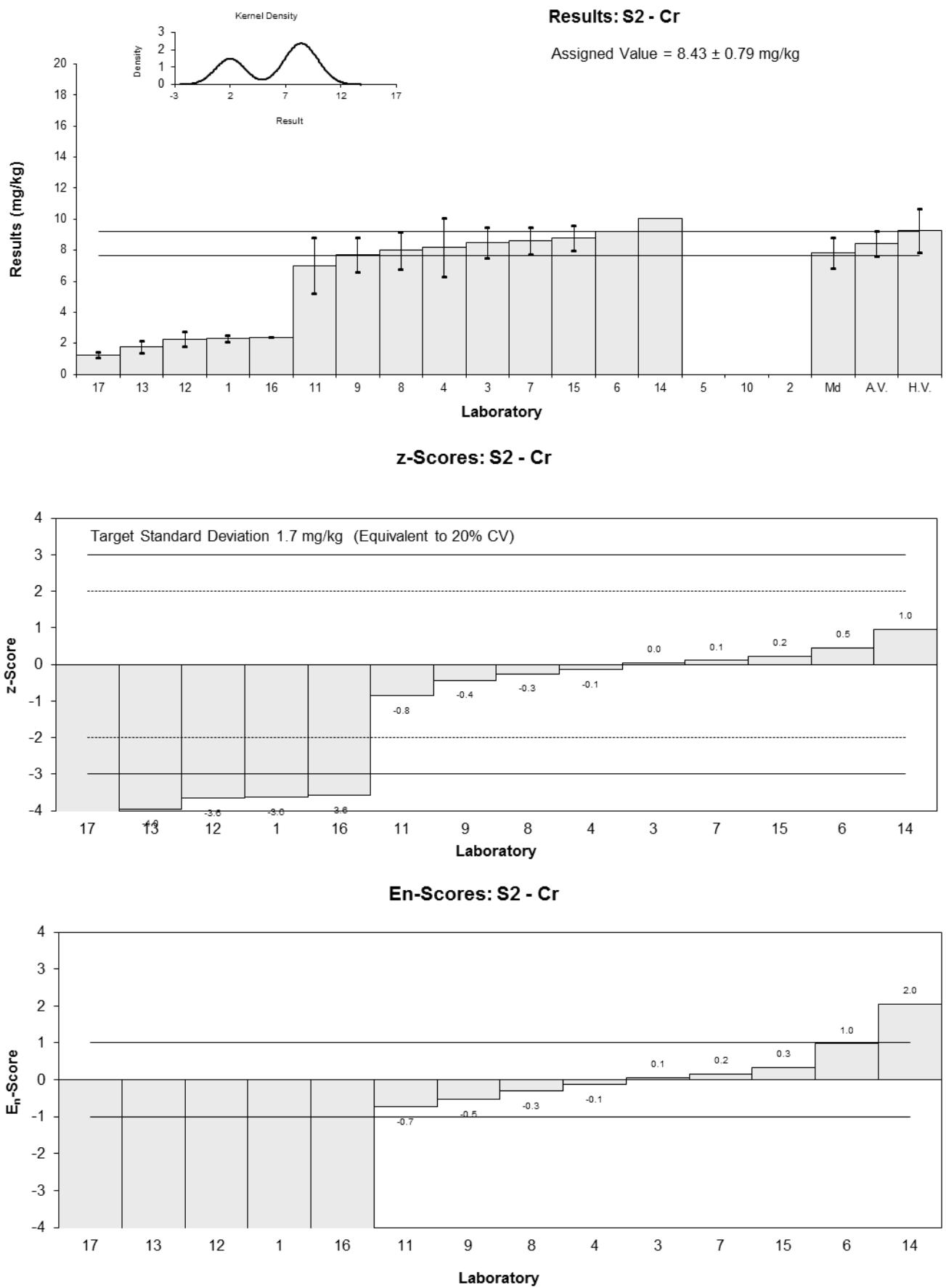


Figure 41

Table 48

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	15	0.7	0.34	0.44
2	14.20	0.028	-0.21	-0.33
3	14.3	1.1	-0.14	-0.14
4	13.0	2.6	-1.03	-0.55
5	17.24	4	1.89	0.67
6	13.39	NR	-0.77	-1.23
7	14.3	1.43	-0.14	-0.12
8	14.2	2.3	-0.21	-0.12
9	15.0	1.7	0.34	0.26
10	NT	NT		
11	11.7	1.6	-1.93	-1.53
12	17.2	3.4	1.86	0.77
13	14.6	2.0	0.07	0.05
14	16.501	NR	1.38	2.22
15	13	1.8	-1.03	-0.75
16	14.7	0.1	0.14	0.22
17	14.6	2.19	0.07	0.04

Statistics

Assigned Value	14.5	0.9
Spike	Not Spiked	
Homogeneity Testing	14.3	1.7
Robust Average	14.5	0.9
Median	14.5	0.4
Mean	14.6	
N	16	
Max.	17.2	
Min.	11.7	
Robust SD	1.5	
Robust CV	10%	

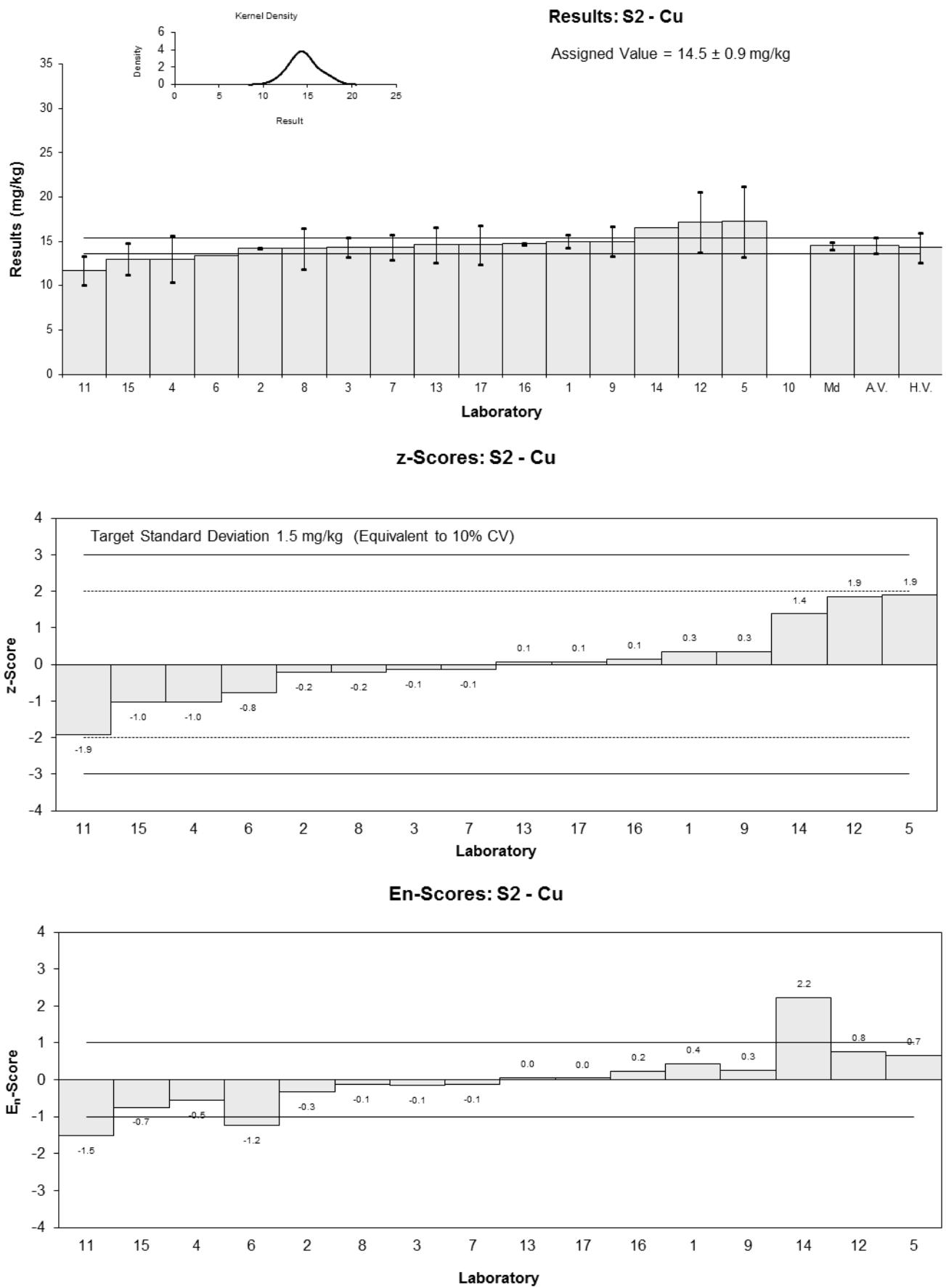


Figure 42

Table 49

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	500	30	0.11	0.22
2	NT	NT		
3	507	40	0.20	0.33
4	514	82.2	0.30	0.26
5	354.48	27	-1.86	-4.02
6	475.8	NR	-0.22	-0.77
7	482	48.2	-0.14	-0.19
8	506	96	0.19	0.14
9	540	49	0.65	0.90
10	NT	NT		
11	477	62	-0.20	-0.23
12	452	90	-0.54	-0.43
13	462	50	-0.41	-0.55
14	561.652	NR	0.94	3.32
15	500	80	0.11	0.10
16	506	61	0.19	0.22
17	476	71	-0.22	-0.22

Statistics

Assigned Value	492	21
Spike	Not Spiked	
Homogeneity Value	528	79
Robust Average	492	21
Median	500	19
Mean	488	
N	15	
Max.	562	
Min.	354	
Robust SD	32.8	
Robust CV	6.7%	

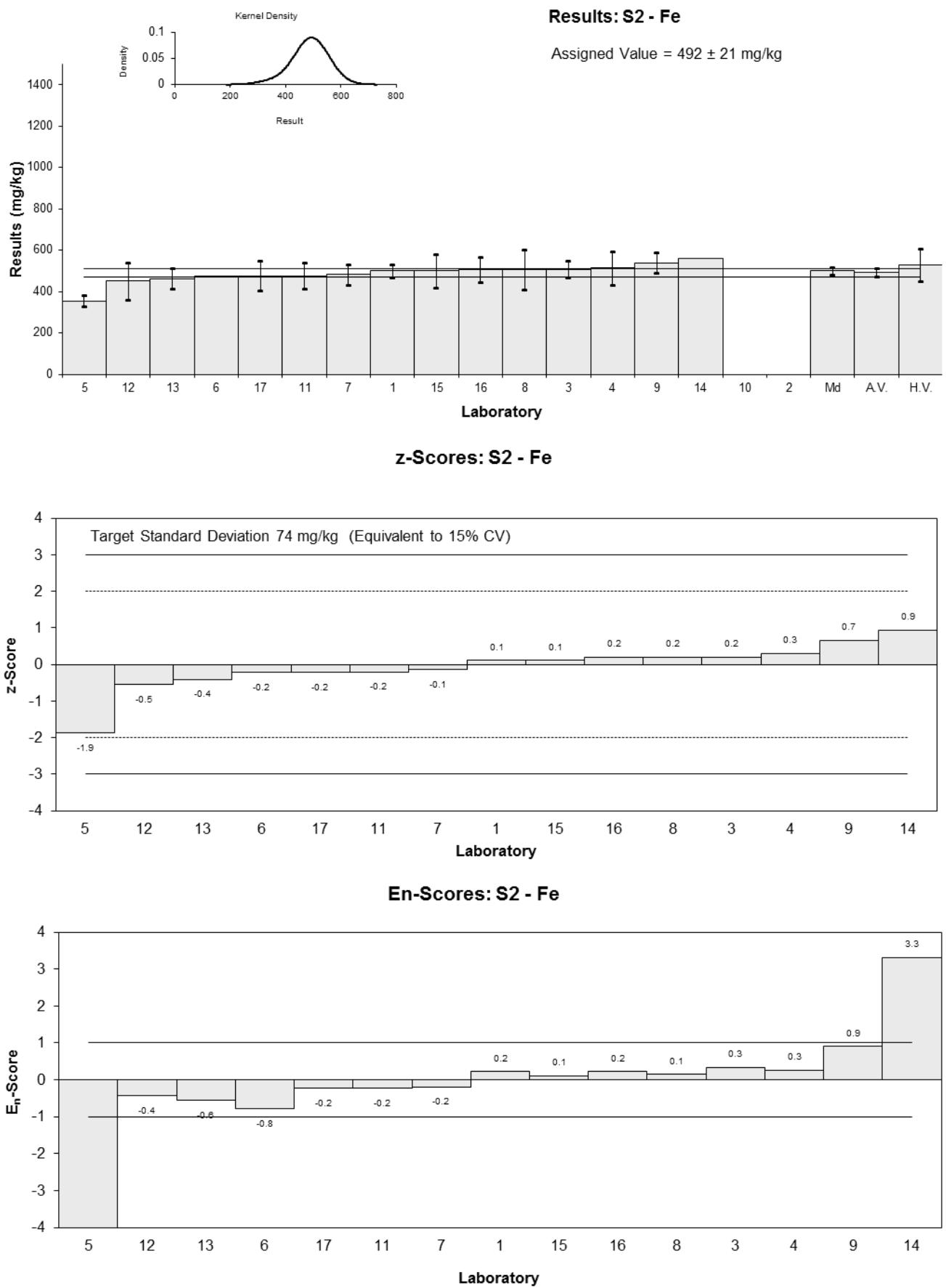


Figure 43

Table 50

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	7900	500	-0.38	-0.48
2	NT	NT		
3	7377	539	-1.01	-1.24
4	7860	1100	-0.43	-0.30
5	7473.1	66	-0.90	-1.82
6	8442	NR	0.28	0.58
7	8070	807	-0.17	-0.16
8	NT	NT		
9	9350	548	1.39	1.68
10	NT	NT		
11	8360	1680	0.18	0.09
12	7950	1590	-0.32	-0.16
13	9000	900	0.96	0.80
14	8074.876	NR	-0.16	-0.34
15	8900	800	0.84	0.77
16	8400	350	0.23	0.36
17	8040	1206	-0.21	-0.13

Statistics

Assigned Value	8210	400
Spike	Not Spiked	
Homogeneity Value	8490	310
Robust Average	8210	400
Median	8070	260
Mean	8228	
N	14	
Max.	9350	
Min.	7377	
Robust SD	597	
Robust CV	7.3%	

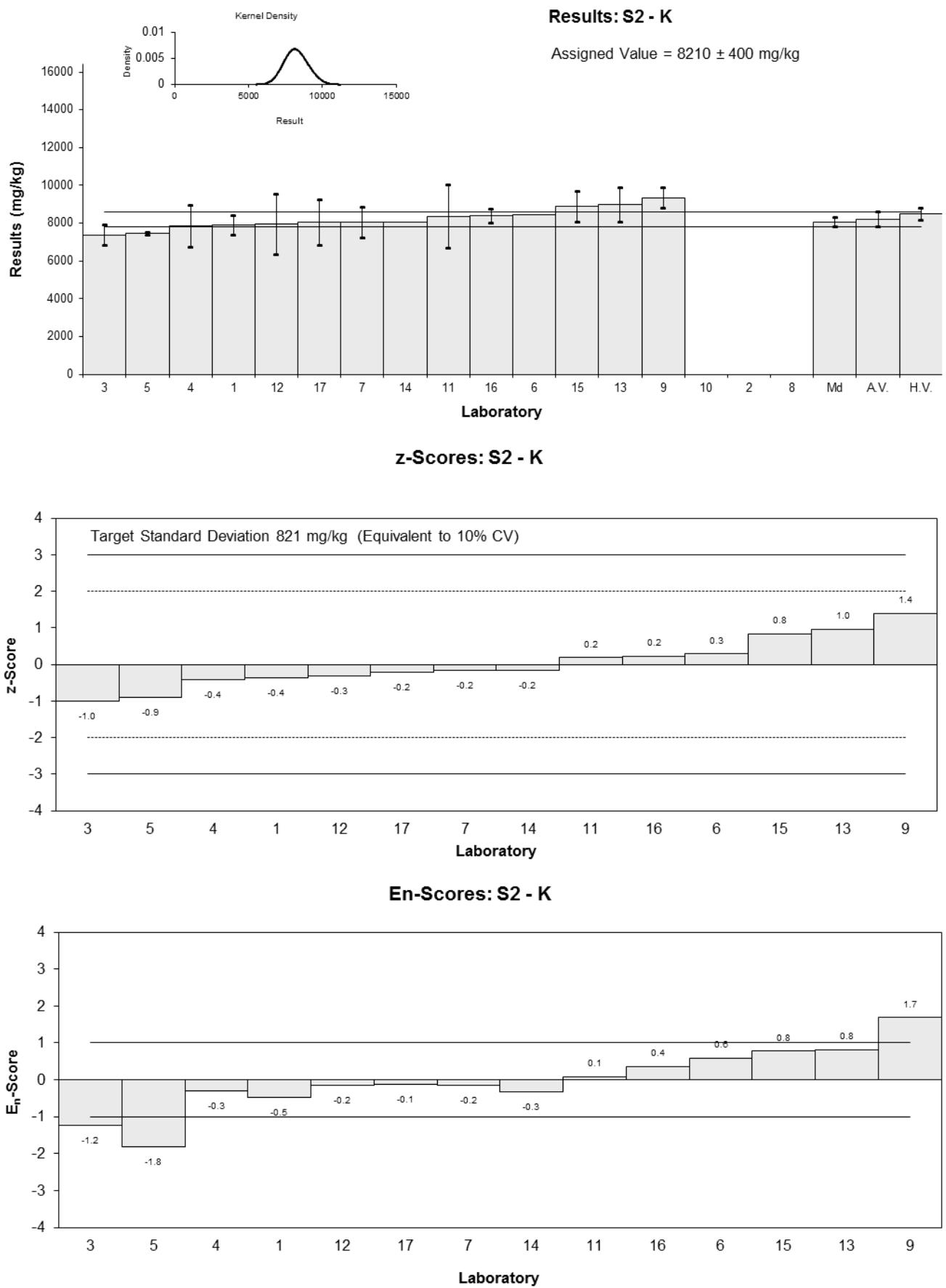


Figure 44

Table 51

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	2000	200	0.20	0.19
2	NT	NT		
3	1916	230	-0.22	-0.18
4	2000	180	0.20	0.21
5	1984.0	195	0.12	0.12
6	2032	NR	0.37	1.03
7	1900	190	-0.31	-0.30
8	NT	NT		
9	2400	175	2.24	2.33
10	NT	NT		
11	1760	180	-1.02	-1.04
12	1930	390	-0.15	-0.08
13	2250	220	1.48	1.26
14	1843.066	NR	-0.60	-1.67
15	2000	240	0.20	0.16
16	1960	25	0.00	0.00
17	1890	284	-0.36	-0.24

Statistics

Assigned Value	1960	70
Spike	Not Spiked	
Homogeneity Value	2380	270
Robust Average	1960	69
Median	1970	50
Mean	1990	
N	14	
Max.	2400	
Min.	1760	
Robust SD	105	
Robust CV	5.4%	

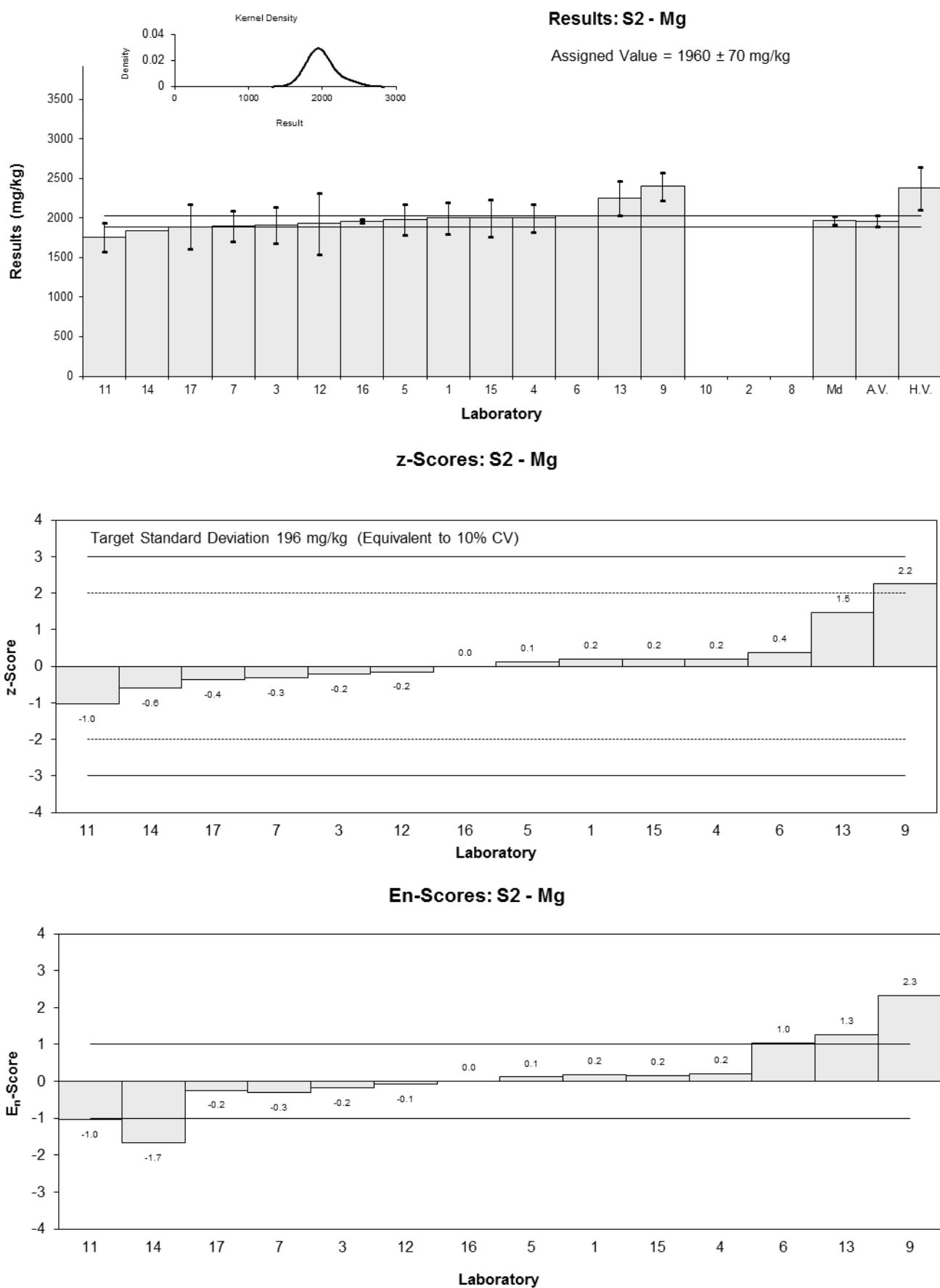


Figure 45

Table 52

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	175	10	0.42	0.52
2	NT	NT		
3	153	20	-0.89	-0.68
4	151	37.8	-1.01	-0.44
5	NR	NR		
6	160.6	NR	-0.44	-0.82
7	173	17.3	0.30	0.26
8	168	42	0.00	0.00
9	182	12	0.83	0.93
10	NT	NT		
11	161	25	-0.42	-0.26
12	195	39	1.61	0.67
13	177	20	0.54	0.41
14	183.763	NR	0.94	1.75
15	150	18	-1.07	-0.89
16	163	15	-0.30	-0.29
17	165	25	-0.18	-0.11

Statistics

Assigned Value	168	9
Spike	Not Spiked	
Homogeneity Value	185	22
Robust Average	168	9
Median	167	8
Mean	168	
N	14	
Max.	195	
Min.	150	
Robust SD	14	
Robust CV	8.3%	

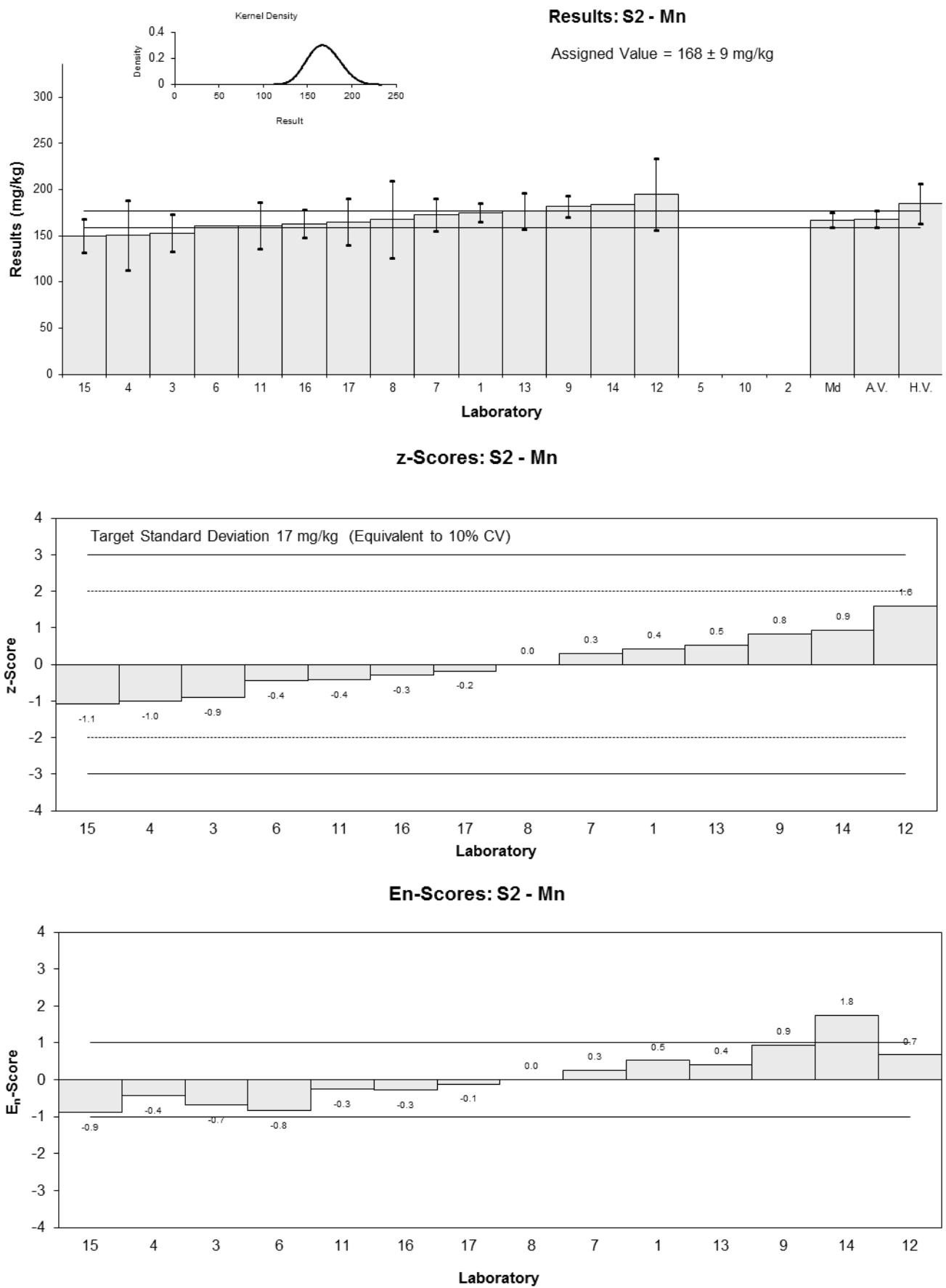


Figure 46

Table 53

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	3.0	0.2	-0.22	-0.36
2	2.98	0.025	-0.26	-0.63
3	2.8	0.4	-0.65	-0.68
4	3.25	0.65	0.32	0.22
5	NR	NR		
6	3.4	NR	0.65	1.58
7	3.29	0.329	0.41	0.50
8	3.3	1.0	0.43	0.20
9	3.11	0.39	0.02	0.02
10	NT	NT		
11	3.0	0.8	-0.22	-0.12
12	3.0	0.6	-0.22	-0.16
13	2.80	0.5	-0.65	-0.56
14	NT	NT		
15	4.2	0.59	2.37	1.77
16	3.245	0.04	0.31	0.75
17	2.50	0.38	-1.29	-1.41

Statistics

Assigned Value	3.10	0.19
Spike	Not Spiked	
Homogeneity Value	3.07	0.37
Robust Average	3.10	0.19
Median	3.06	0.18
Mean	3.13	
N	14	
Max.	4.2	
Min.	2.5	
Robust SD	0.28	
Robust CV	9%	

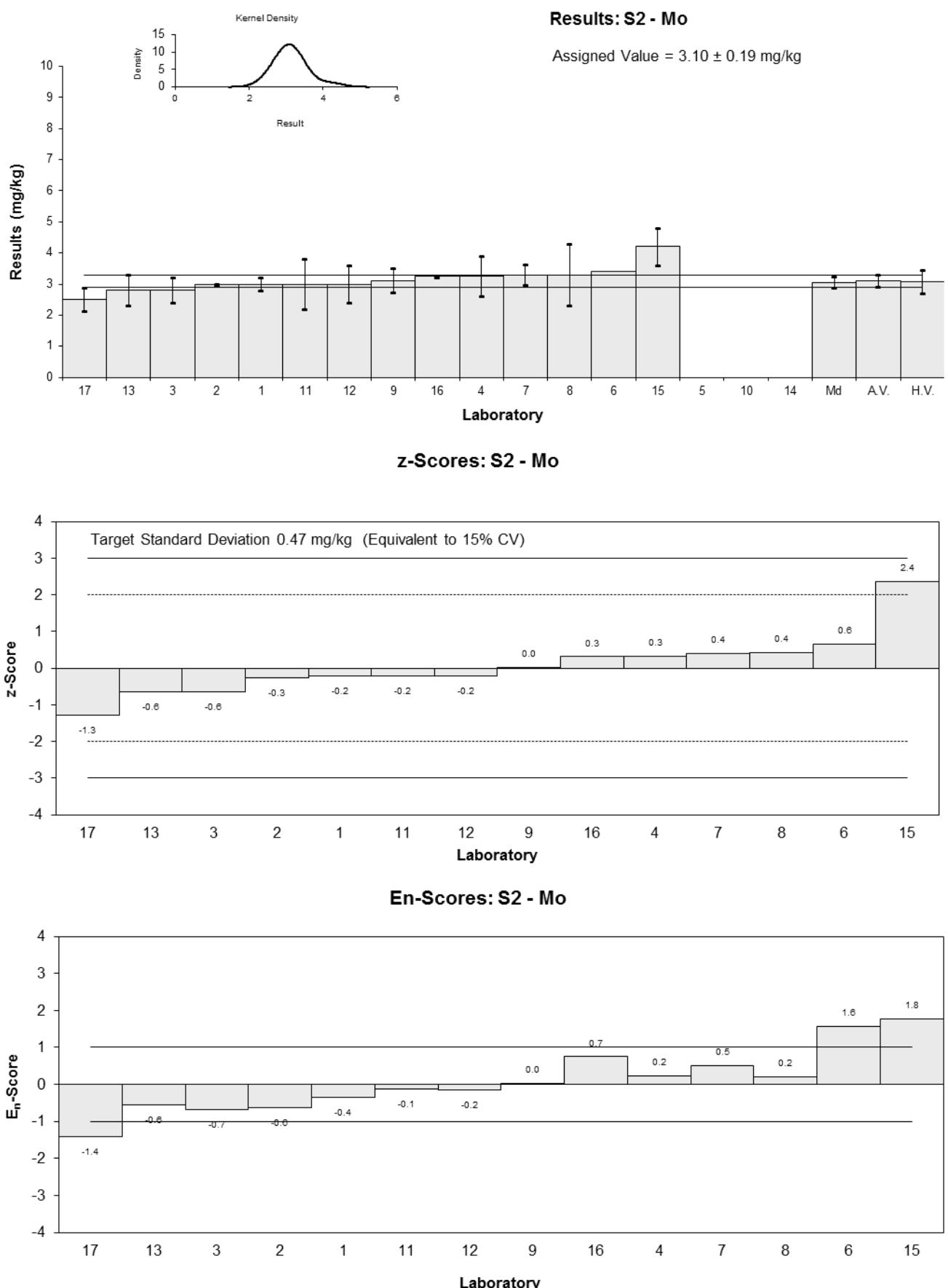


Figure 47

Table 54

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	2000	200	0.54	0.66
2	NT	NT		
3	1661	116	-0.68	-1.18
4	1840	313	-0.04	-0.03
5	1691.8	213	-0.57	-0.66
6	1919	NR	0.25	0.63
7	1770	177	-0.29	-0.38
8	NT	NT		
9	2150	162	1.08	1.53
10	NT	NT		
11	1810	370	-0.14	-0.10
12	1670	330	-0.65	-0.52
13	1780	200	-0.25	-0.31
14	1765.607	NR	-0.30	-0.77
15	2200	260	1.26	1.24
16	1890	170	0.14	0.20
17	1900	285	0.18	0.16

Statistics

Assigned Value	1850	110
Spike	Not Spiked	
Homogeneity Value	2210	440
Robust Average	1850	110
Median	1830	70
Mean	1861	
N	14	
Max.	2200	
Min.	1661	
Robust SD	159	
Robust CV	8.6%	

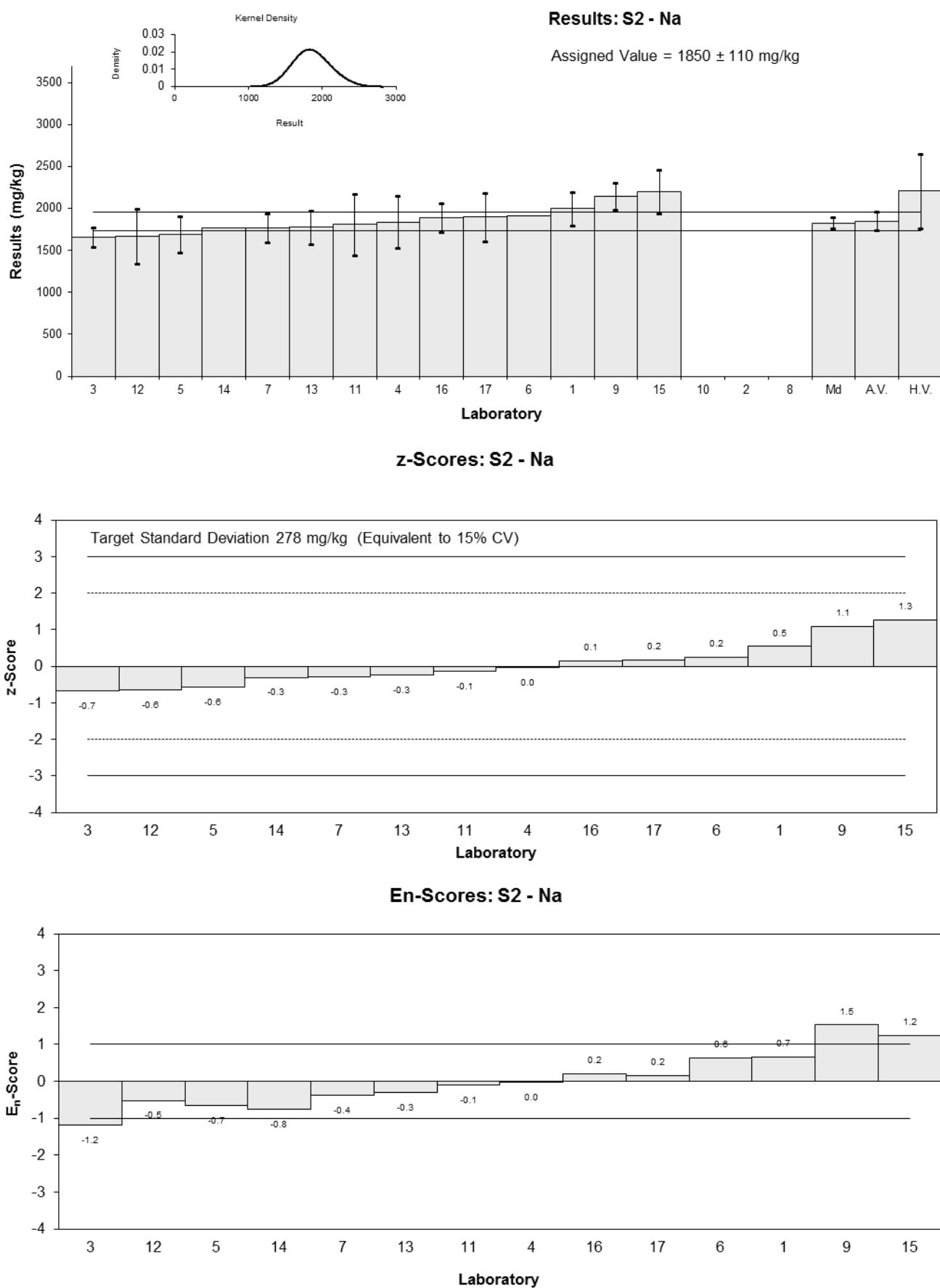


Figure 48

Table 55

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	5000	300	-1.18	-1.48
2	NT	NT		
3	5240	361	-0.76	-0.87
4	5920	1480	0.44	0.16
5	NR	NR		
6	6124	NR	0.80	1.34
7	5190	519	-0.85	-0.77
8	5830	1460	0.28	0.11
9	6140	407	0.83	0.89
10	NT	NT		
11	5540	670	-0.23	-0.17
12	5700	1140	0.05	0.03
13	5820	580	0.26	0.22
14	NT	NT		
15	5700	260	0.05	0.07
16	6940	120	2.24	3.52
17	5120	768	-0.97	-0.65

Statistics

Assigned Value	5670	340
Spike	Not Spiked	
Homogeneity Value	6360	950
Robust Average	5670	340
Median	5700	380
Mean	5713	
N	13	
Max.	6940	
Min.	5000	
Robust SD	490	
Robust CV	8.6%	

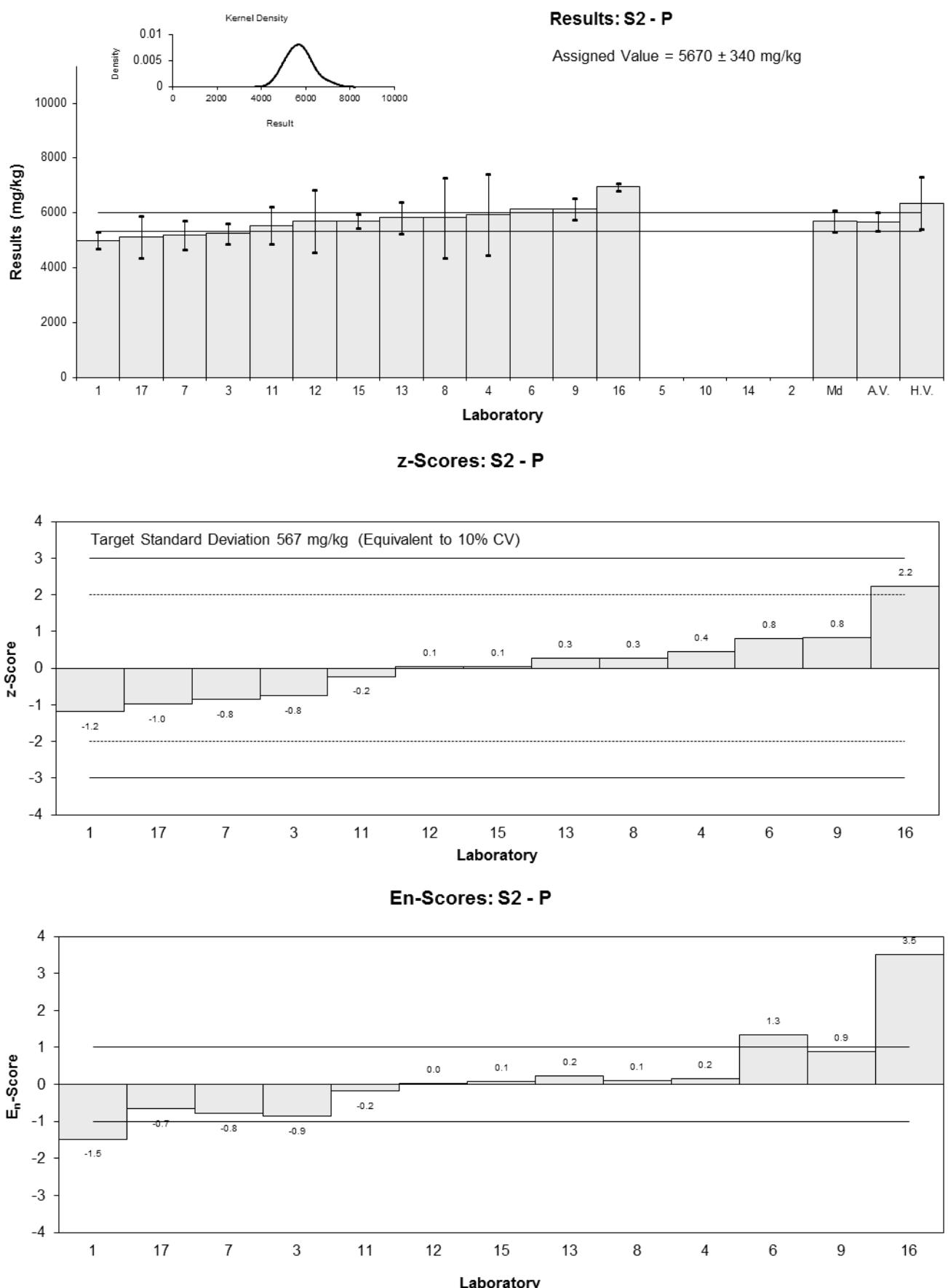


Figure 49

Table 56

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.41	0.03	-1.01	-1.30
2	0.44	0.025	-0.35	-0.51
3	0.44	0.07	-0.35	-0.22
4	0.505	0.11	1.07	0.44
5	NR	NR		
6	0.47	NR	0.31	0.74
7	0.470	0.047	0.31	0.28
8	<1.0	NR		
9	0.49	0.09	0.75	0.37
10	NT	NT		
11	<1	NR		
12	0.457	0.091	0.02	0.01
13	0.47	0.1	0.31	0.14
14	0.430	NR	-0.57	-1.37
15	0.44	0.066	-0.35	-0.23
16	0.473	0.003	0.37	0.88
17	0.44	0.07	-0.35	-0.22

Statistics

Assigned Value	0.456	0.019
Spike	Not Spiked	
Homogeneity Value	0.459	0.055
Robust Average	0.456	0.019
Median	0.457	0.015
Mean	0.457	
N	13	
Max.	0.505	
Min.	0.41	
Robust SD	0.028	
Robust CV	6.1%	

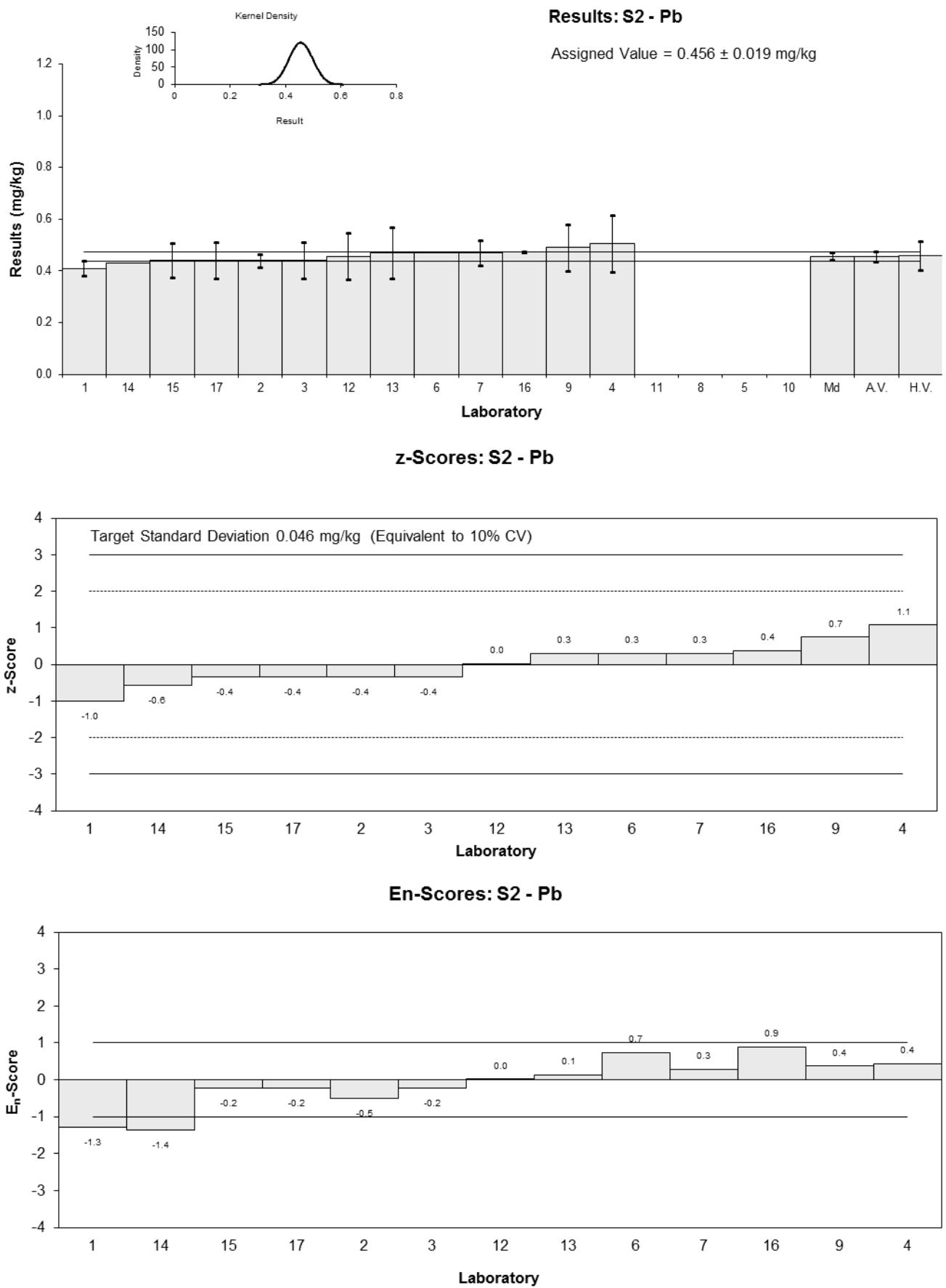


Figure 50

Table 57

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	2200	110	-1.27	-1.58
2	NT	NT		
3	2461	167	-0.23	-0.25
4	NT	NT		
5	NR	NR		
6	NT	NT		
7	2430	243	-0.36	-0.30
8	2310	580	-0.83	-0.35
9	3450	599	3.69	1.49
10	NT	NT		
11	2390	310	-0.52	-0.37
12	2540	510	0.08	0.04
13	2500	250	-0.08	-0.07
14	NT	NT		
15	2800	340	1.11	0.74
16	2660	15	0.56	0.82
17	2580	388	0.24	0.14

Statistics

Assigned Value	2520	170
Spike	Not Spiked	
Robust Average	2520	170
Median	2500	110
Mean	2575	
N	11	
Max.	3450	
Min.	2200	
Robust SD	224	
Robust CV	8.9%	

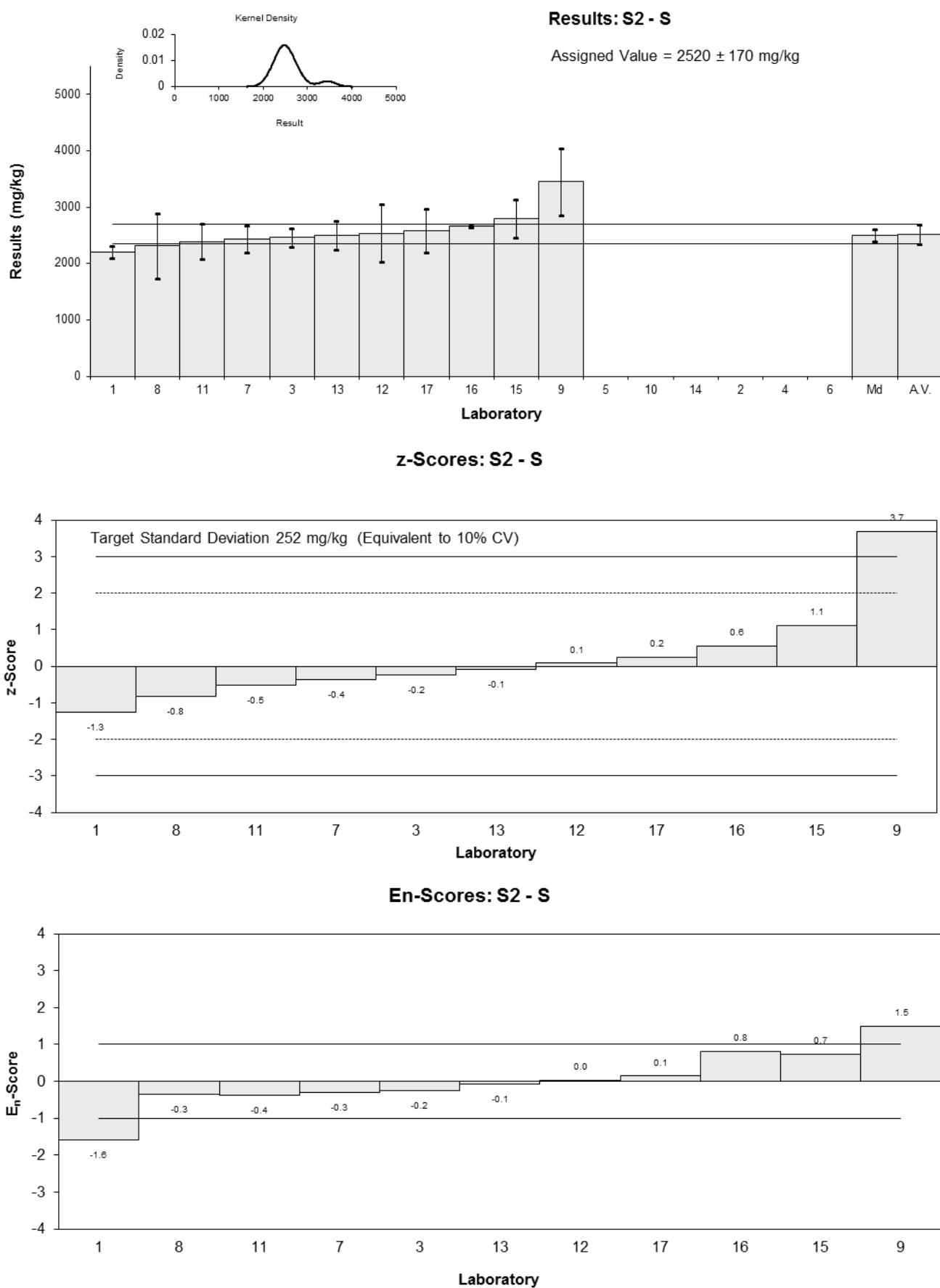


Figure 51

Table 58

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	0.35	0.03	-0.22	-0.33
2	NT	NT		
3	0.35	0.04	-0.22	-0.29
4	0.365	0.095	-0.01	-0.01
5	NR	NR		
6	0.31	NR	-0.77	-1.47
7	0.450	0.045	1.15	1.43
8	<5.0	NR		
9	0.64	0.13	3.74	2.02
10	NT	NT		
11	<2	NR		
12	0.349	0.070	-0.23	-0.21
13	<0.5	NR		
14	0.409	NR	0.59	1.13
15	NT	NT		
16	0.368	0.015	0.03	0.05
17	0.58	0.09	2.92	2.19

Statistics

Assigned Value*	0.366	0.038
Spike	Not Spiked	
Homogeneity Value	0.348	0.052
Robust Average	0.405	0.076
Median	0.367	0.032
Mean	0.417	
N	10	
Max.	0.64	
Min.	0.31	
Robust SD	0.096	
Robust CV	24%	

*Robust Average excluding Laboratories 9 and 17.

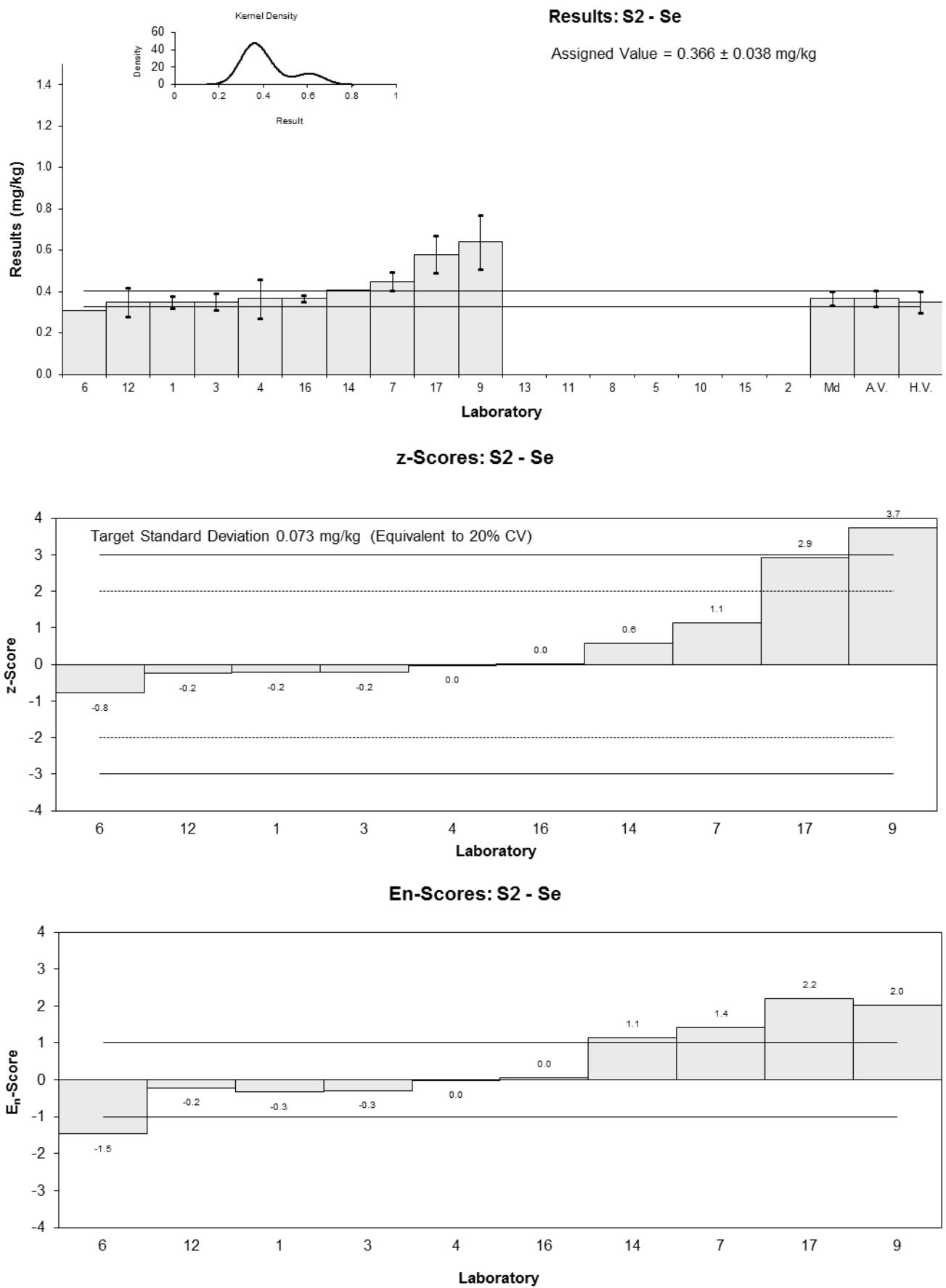


Figure 52

Table 59

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	NR	NR
2	NT	NT
3	NT	NT
4	33400	1200
5	NR	NR
6	NT	NT
7	NT	NT
8	NT	NT
9	NT	NT
10	NT	NT
11	3240	900
12	31000	4700
13	31700	3170
14	NT	NT
15	NT	NT
16	NR	NR
17	2.99	0.45

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Robust Average	19900	21000
Median	31000	4400
Mean	19869	
N	5	
Max.	33400	
Min.	2.99	
Robust SD	19000	
Robust CV	95%	

Results: S2 - TKN

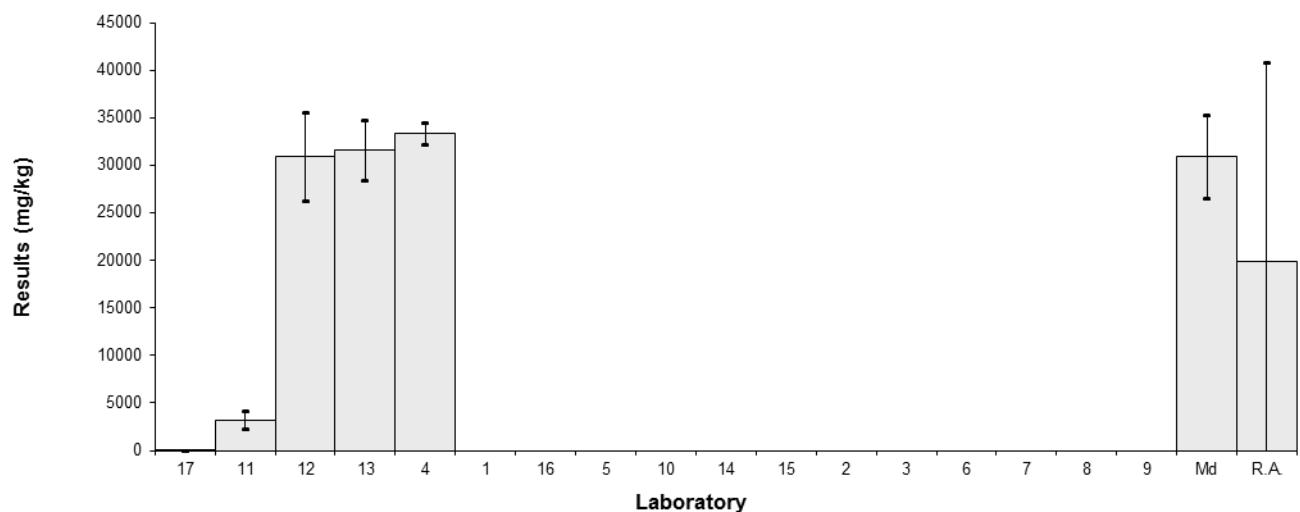


Figure 53

Table 60

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	NR	NR
2	NT	NT
3	390000	1560
4	NT	NT
5	NR	NR
6	NT	NT
7	NT	NT
8	NT	NT
9	NT	NT
10	NT	NT
11	36100	4000
12	324000	32000
13	290000	15000
14	NT	NT
15	NT	NT
16	NR	NR
17	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Robust Average	260000	220000
Median	310000	120000
Mean	260025	
N	4	
Max.	390000	
Min.	36100	
Robust SD	220000	
Robust CV	85%	

Results: S2 - TOC

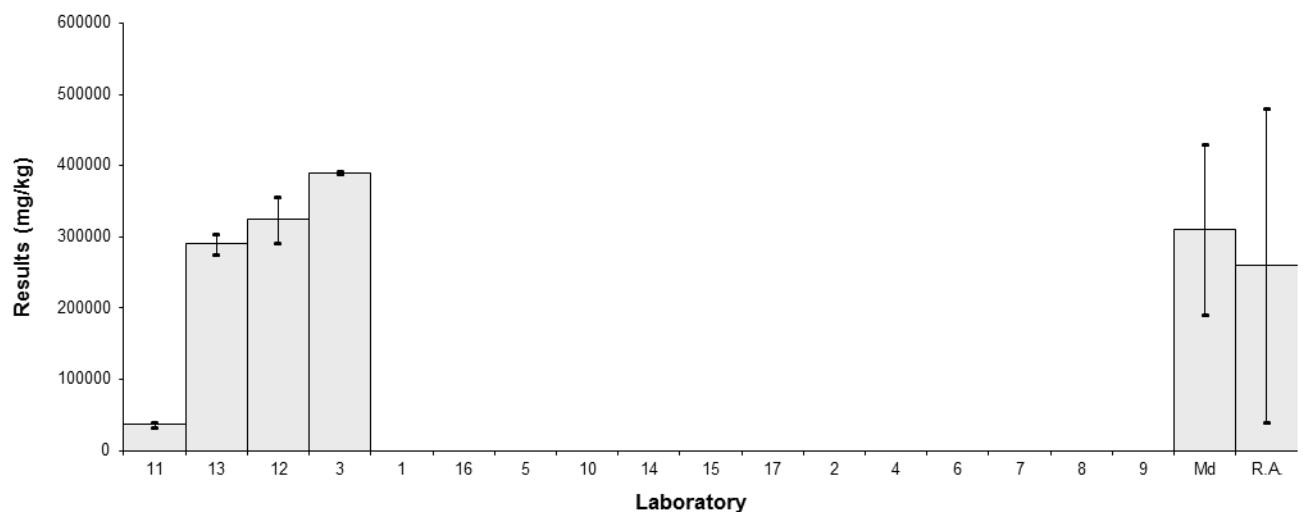


Figure 54

Table 61

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E _n -Score
1	93	5.0	-0.36	-0.59
2	NT	NT		
3	92	12	-0.47	-0.36
4	93.9	9.39	-0.27	-0.26
5	98.8	2	0.24	0.61
6	84.47	NR	-1.25	-3.76
7	103	10.3	0.67	0.60
8	99.1	13.9	0.27	0.18
9	110	8	1.40	1.57
10	NT	NT		
11	91	12	-0.57	-0.44
12	98.1	19.6	0.17	0.08
13	98	15	0.16	0.10
14	99.809	NR	0.34	1.03
15	98	14	0.16	0.10
16	96	4	-0.05	-0.10
17	93.3	14	-0.33	-0.22

Statistics

Assigned Value	96.5	3.2
Spike	Not Spiked	
Homogeneity Value	111	13
Robust Average	96.5	3.2
Median	98.0	3.4
Mean	96.6	
N	15	
Max.	110	
Min.	84.5	
Robust SD	4.9	
Robust CV	5.1%	

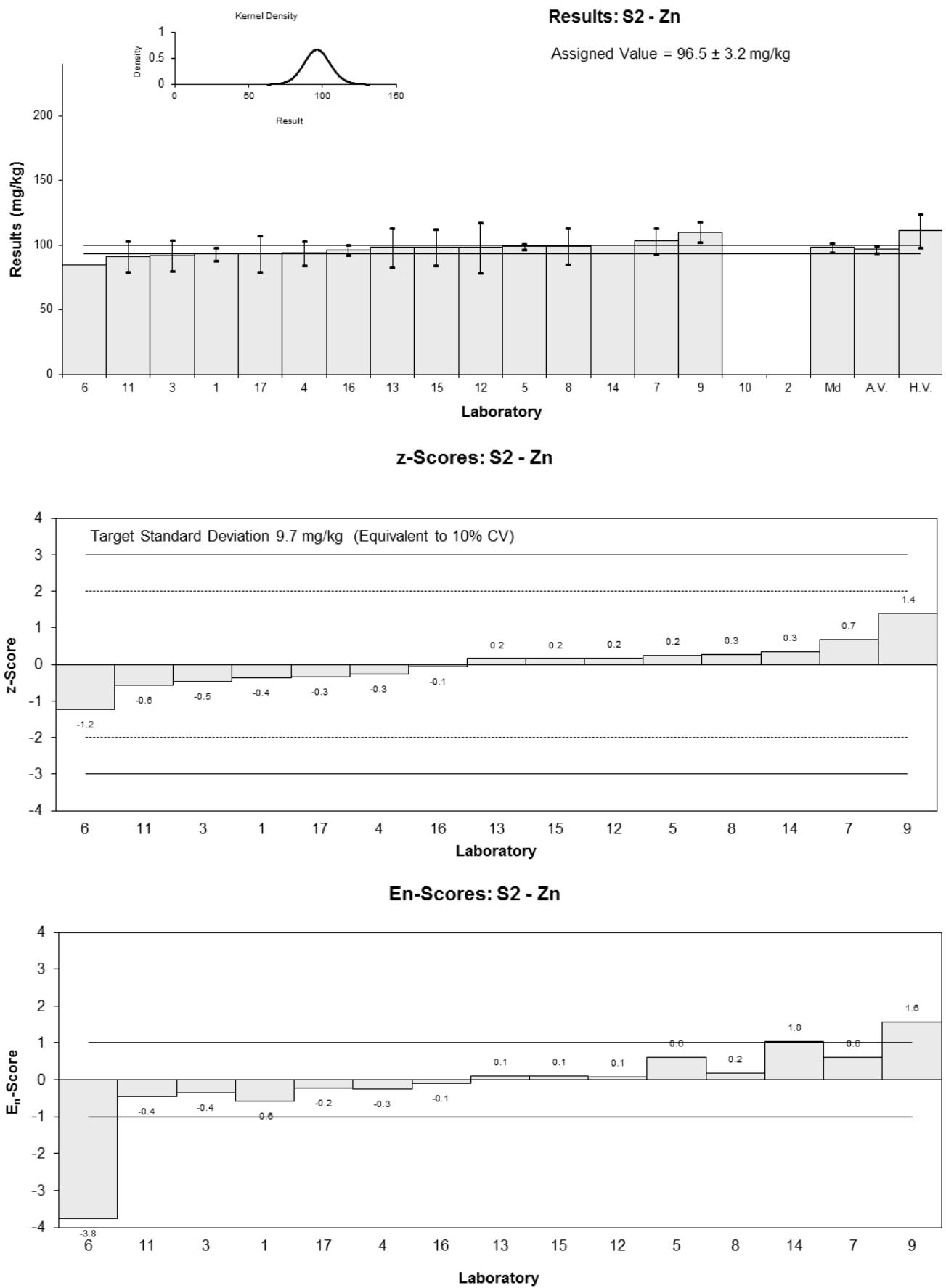


Figure 55

6 DISCUSSION OF RESULTS

6.1 Assigned Value

Samples S1 – was freeze dried bovine liver, a reference material previously prepared by NMI.

Sample S2 – was a plant material (85 % wheat). This material was also distributed as Sample S2 of AQA 13-18.

Assigned Values were the robust averages of participants' results, except Cr, Ni and Mo in S1. The robust averages used as assigned values and their associated expanded uncertainties were calculated using the procedure described in 'Statistical methods for use in proficiency testing by interlaboratory comparisons, ISO13528:2015(E)'. Results less than 50% and more than 150% of the robust average were investigated and then removed before calculation of the assigned value.⁷ Appendix 2 sets out the calculation for the robust average of U in Sample S1 and its associated uncertainty.

The assigned values for Ni, and Mo in Sample S1 were reference values from measurements made using d-IDMS; the assigned value for Cr in S1 was an information value from measurements made using the same technique (See Appendix 3).

No assigned value was set for Al in S1 and S2 as most participants could not achieve a complete recovery of this element from the study samples. No assigned value was also set for B, Sn, Th, TKN and TOC because too few results were reported for these tests.

No assigned value was set for inorganic-As because this was included as a pilot program.

Traceability of the reference values for Ni and Mo in S1 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 and 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.

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.

6.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded measurement uncertainty associated with their results. Of 556 numerical results, 481 (87%) were reported with an expanded measurement uncertainty, indicating that the majority of laboratories have addressed this requirement of ISO 17025.⁹ The participants used a wide variety of procedures to estimate the expanded measurement uncertainty. These are presented in Table 4.

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.^{10 – 17}

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 55). In this study, the reported expanded measurement uncertainty has been over-estimated in some cases (e.g. Lab 10 for Ag, Ba, Be, Co) or under-estimated (e.g. Lab 3 for Be in S1 or Lab 16 for Cu in S1). As a simple rule of thumb, when the uncertainty estimate is smaller than the assigned uncertainty 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 NATA Technical Note 33¹⁴ and to NORDTEST TR 537,¹¹ 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.

Laboratory 5 attached an estimate of the expanded measurement uncertainty for Cd result in S2 reported as less than their limit of detection. An estimate of uncertainty expressed as a value cannot be attached to a result expressed as a range.¹⁰

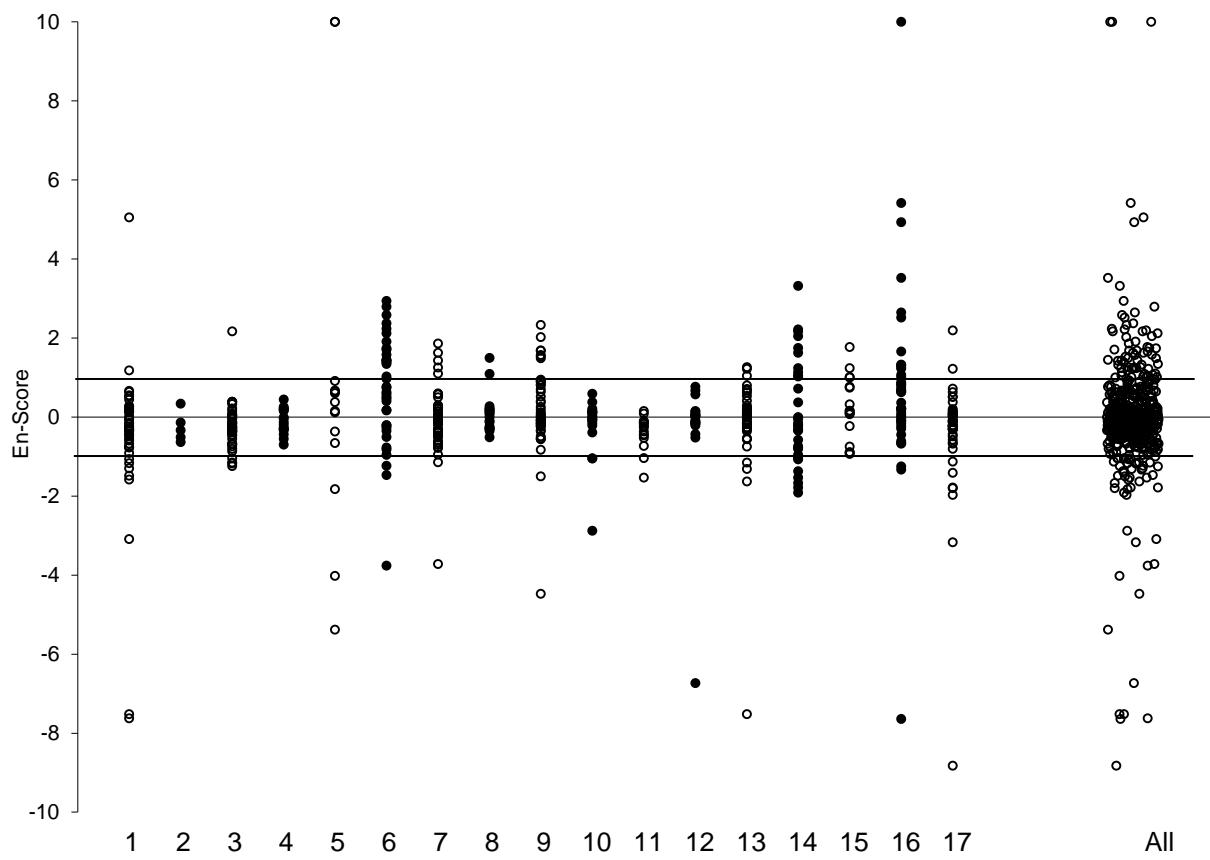
In some cases the results were reported with an inappropriate number of significant figures. The recommended format is to write uncertainty to no more than two significant figures and then to write the result with the corresponding number of decimal places. For example, instead of 0.626 ± 0.21 mg/kg, 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.¹⁰

6.3 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.

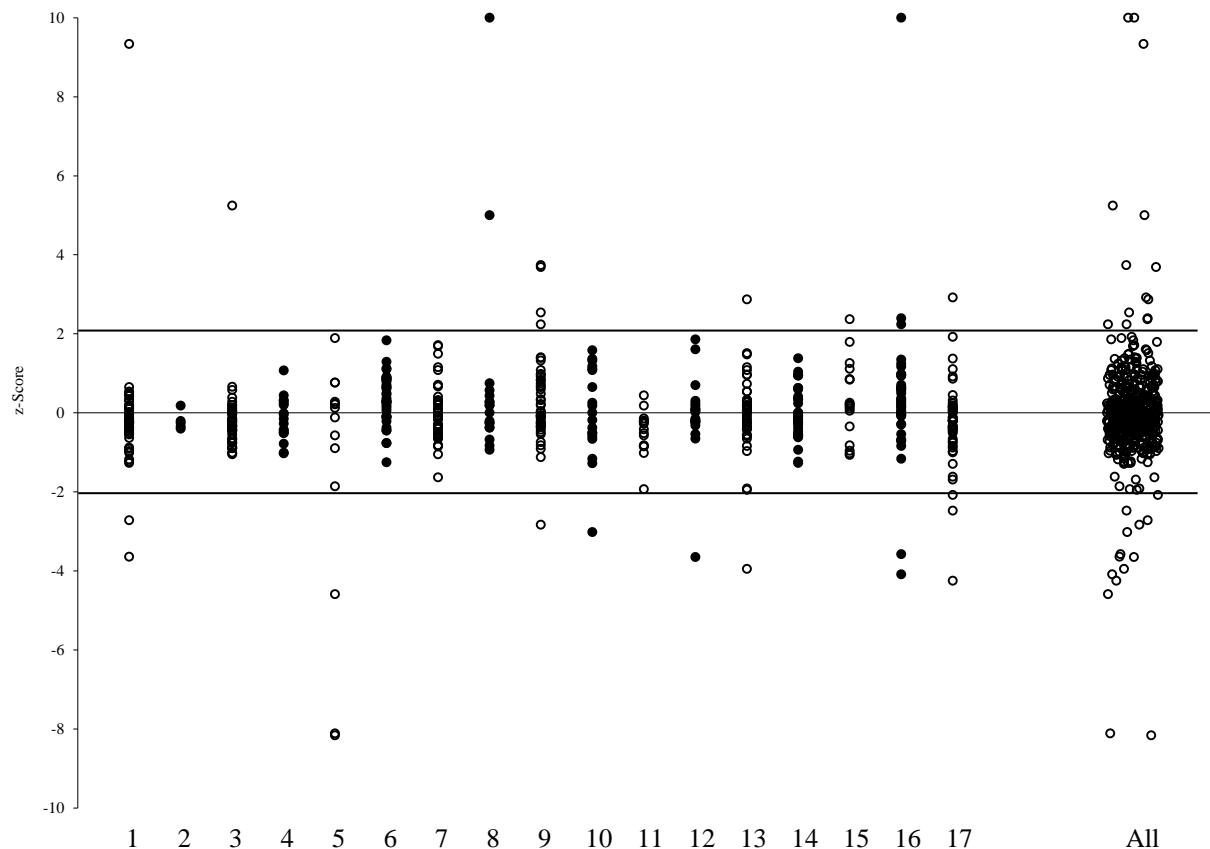
The dispersal of participants' E_n-scores is graphically presented in Figure 56. Where a laboratory did not report an expanded uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the E_n-score.

Of 511 results for which E_n-scores were calculated, 395 (77%) returned a satisfactory score of $|E_n| \leq 1$ indicating agreement of the participants' results with the assigned values within their respective expanded measurement uncertainties.



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

Figure 56 E_n-Score Dispersal by Laboratory



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

Figure 57 z-Score Dispersal by Laboratory

6.4 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% and 20% CV were used to calculate z-scores. Sample S2 in this study was Sample S2 of AQA 13-18. The same standard deviation was set for this sample as in the previous study. 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 equation⁹ and the between laboratory coefficient of variation resulted in this study are presented for comparison in Table 62. The dispersal of participants' z-scores is presented in Figure 57 (by laboratory code) and in Figure 58 (by test). Of 511 results for which z-scores were calculated, 483 (95%) returned a satisfactory score of $|z| \leq 2$ and 11 (3%) were questionable of $2 < |z| \leq 3$.

Participants with multiple z-scores larger than 2 or smaller than -2 should check for laboratory bias.

Laboratories **2, 4, 6, 7, 11** and **14** returned satisfactory z-scores for all analytes reported.

Table 62 Between Laboratory CV of this study, Thompson CV and Set Target CV

Sample	Analyte	Assigned value (mg/kg)	Between Laboratories CV	Thompson CV	Target SD (as CV)
S1	Ag	0.45	5.4%	18%	10%
S1	Al	Not Set	21%	NA	Not Set
S1	As	0.451	7.2%	18%	10%
S1	B	Not Set	16%	NA	Not Set
S1	Ba	0.691	8%	17%	10%
S1	Be	0.334	17%	19%	15%
S1	Bi	0.348	3.2%	19%	10%
S1	Ca	167	7.8%	7%	10%
S1	Cd	0.964	6.8%	16%	10%
S1	Co	0.632	9%	17%	10%
S1	Cr	0.438	33%	18%	20%
S1	Cu	163	3.4%	7%	10%
S1	Fe	479	4.6%	6%	10%
S1	Hg	0.384	12%	18%	15%
S1	Inorganic As	Not Set	20%	NA	Not Set
S1	K	9630	9.6%	4%	10%
S1	Li	0.325	18%	19%	20%
S1	Mg	596	6.5%	6%	10%
S1	Mn	9.84	6.7%	11%	10%
S1	Mo	3.903	4.9%	13%	10%
S1	Na	2150	7.9%	5%	10%
S1	Ni	0.708	23%	17%	20%
S1	P	12000	8.6%	4%	10%
S1	Pb	0.795	4.2%	17%	10%
S1	Sb	0.389	9.3%	18%	10%

S1	Se	1.64	28%	15%	20%
S1	Sn	Not Set	84%	NA	Not Set
S1	Sr	0.649	3.9%	17%	10%
S1	Th	Not Set	8.7%	NA	Not Set
S1	U	0.385	6.6%	18%	10%
S1	V	0.422	9.6%	18%	10%
S1	Zn	158	3.3%	7%	10%
S2	Al	Not Set	40%	NA	Not Set
S2	As	0.55	9.3%	18%	20%
S2	B	13.4	21%	11%	15%
S2	Ba	7.3	7.2%	12%	10%
S2	Ca	47800	9.4%	3%	10%
S2	Cd	0.0532	11%	22%	15%
S2	Co	0.469	13%	18%	15%
S2	Cr	8.43	61%	12%	20%
S2	Cu	14.5	10%	11%	10%
S2	Fe	492	6.7%	6%	15%
S2	K	8210	7.3%	4%	10%
S2	Mg	1960	5.4%	5%	10%
S2	Mn	168	8.3%	7%	10%
S2	Mo	3.1	9%	13%	15%
S2	Na	1850	8.6%	5%	15%
S2	P	5670	8.6%	4%	10%
S2	Pb	0.456	6.1%	18%	10%
S2	S	2520	8.9%	5%	10%
S2	Se	0.366	24%	19%	20%
S2	TKN	Not Set	95%	NA	Not Set
S2	TOC	Not Set	85%	NA	Not Set
S2	Zn	96.5	5.1%	8%	10%

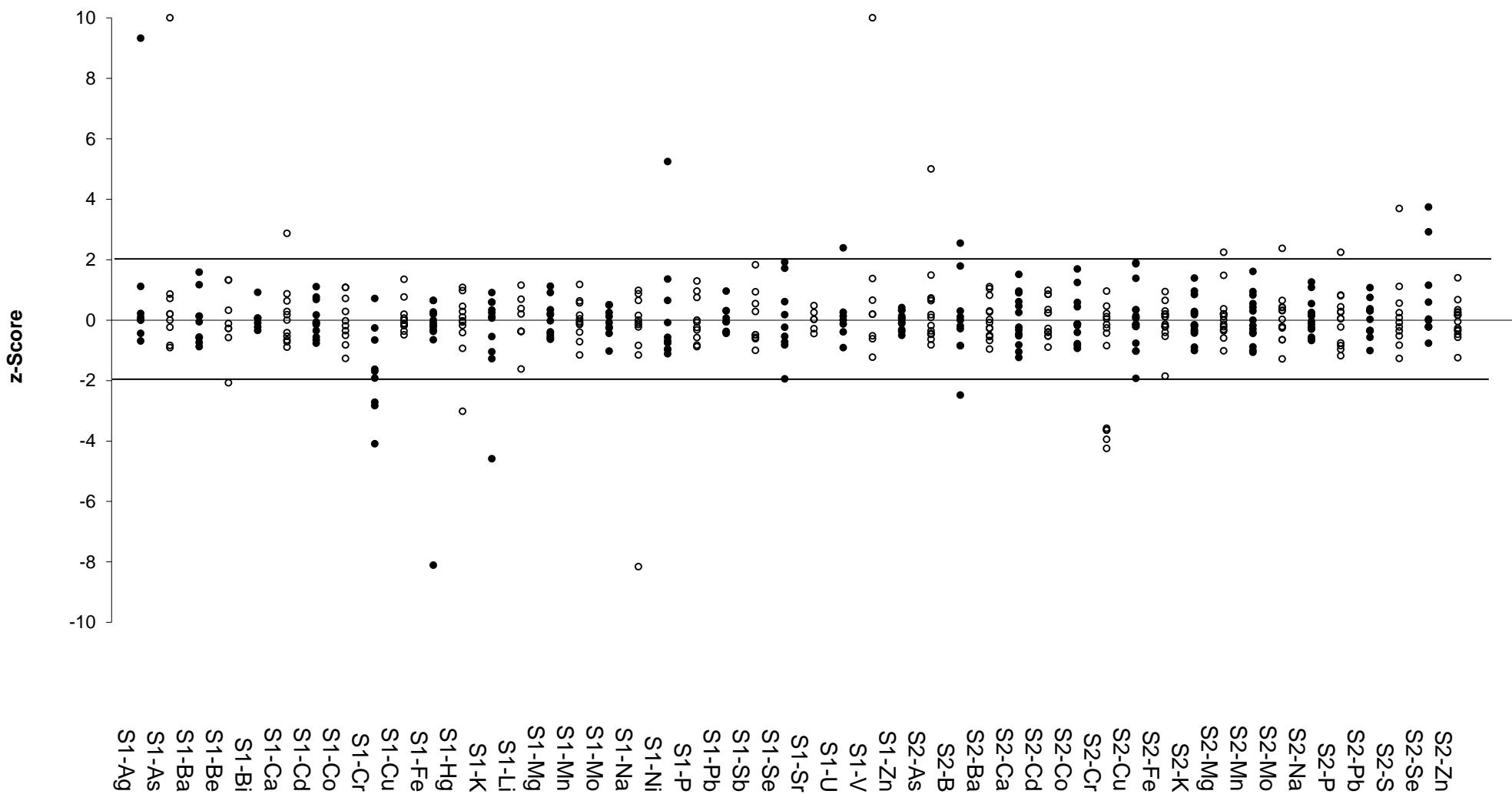
6.5 Participants' Results and Analytical Methods for Total Elements

A summary of participants' performance in the two study samples is presented in Figure 58 and Tables 63 and 64.

Measurements of total Al and Cr in the liver and plant samples presented most analytical difficulty.

Selenium measurement at low level in food samples challenged participants' instrumental techniques, the between laboratory coefficient of variation for this element was high, 28% in S1 and 24% in S2.

The method descriptions provided by participants are presented in Tables 1 to 3; the instrumental conditions are presented in Appendix 5.



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

Figure 58 z-Score Dispersal by Analyte

Table 63 Summary of Participants' Results and Performance in S1

Lab. Code	S1-Ag	S1-Al	S1-As	S1-B	S1-Ba	S1-Be	S1-Bi	S1-Ca	S1-Cd	S1-Co	S1-Cr	S1-Cu
Units	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
H.V./I.V.	0.49	5.3	0.437	NA	0.58	0.348	0.363	165	0.947	0.617	0.438	168
A.V.	0.45	Not Set	0.451	Not Set	0.691	0.334	0.348	167	0.964	0.632	0.438	163
1	0.87	3.4	0.44	0.69	0.63	0.32	0.34	160	0.95	0.63	0.2	160
2	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
3	0.43	4.2	0.46	NR	NR	0.32	0.35	152	0.89	0.62	0.38	163
4	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
5	NR	NR	NR	NR	NR	NR	NR	171.69	1.037	NR	NR	175.36
6	NT	NT	NT	NT	0.7	NT	NT	181.5	1.07	0.65	0.5	166.1
7	0.454	3.49	0.483	0.632	0.687	0.328	0.336	167	1.03	0.58	0.295	155
8	NT	NT	1.2	NT	<1.0	<1.0	NT	NT	<1.0	<1.0	<1.0	157
9	0.46	3	0.41	<1	0.64	0.4	0.345	158	0.93	0.61	0.19	161
10	0.5	4	<3	<2	0.8	0.4	NT	170	0.9	0.7	<1	160
11	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
12	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
13	0.45	<10	0.46	0.51	0.65	0.35	0.35	215	0.91	0.7	0.27	164
14	NT	5.811	0.451	NT	0.652	0.305	NT	177.478	0.954	0.552	0.415	162.93
15	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
16	0.419	3.979	0.413	0.626	0.772	NA	NA	155	0.957	0.677	0.08	185
17	0.45	2.78	0.49	0.49	0.7	0.23	0.38	156	0.98	0.6	0.29	161

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

Table 63 Summary of Participants' Results and Performance in S1 (continued)

Lab. Code	S1-Fe	S1-Hg	S1-K	S1-Li	S1-Mg	S1-Mn	S1-Mo	S1-Na	S1-Ni	S1-P	S1-Pb	S1-Se
Units	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
H.V./R.V.	503	0.38	10300	0.37	653	10.8	3.903	2310	0.708	12400	0.875	1.49
A.V.	479	0.384	9630	0.325	596	9.84	3.903	2150	0.708	12000	0.795	1.64
1	510	0.39	9100	0.3	570	10	4.1	2100	0.57	11700	0.79	1.7
2	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
3	461	0.36	8617	0.35	594	9.8	3.5	2118	1.45	11608	0.76	1.4
4	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
5	90.54	NR	5210.3	NR	608.32	NR	NR	396.4	NR	NR	NR	NR
6	474.4	0.4	10510	0.3	663	9.74	4.1	2336	0.8	13550	0.8	1.84
7	448	0.446	9690	0.338	558	9.45	3.87	2150	0.627	11000	0.763	2.2
8	488	0.33	NT	NT	NT	10.4	3.8	NT	<1.0	12900	<1.0	<5.0
9	469	0.38	10200	0.37	650	9.88	3.81	2360	0.55	11920	0.79	1.37
10	510	0.21	8400	0.3	560	11	4	1900	0.9	12000	<2	<4
11	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
12	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
13	463	0.44	9950	0.4	607	9.69	3.95	1970	0.57	11300	0.82	1
14	478.367	0.3738	9862.011	NT	564.475	10.462	NT	2130.458	0.697	NT	0.765	1.56
15	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
16	492	0.382	10200	NA	616	8.7	4.1	2290	0.611	13150	0.871	1.464
17	470	0.41	9740	0.22	573	9.13	3.73	2183	0.6	10943	0.82	2.27

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

Table 63 Summary of Participants' Results and Performance in S1 (continued)

Lab. Code	S1-Sb	S1-Sn	S1-Sr	S1-Th	S1-U	S1-V	S1-Zn	S1-Inorganic As
Units	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
H.V.	0.413	0.0525	0.646	0.268	0.422	0.455	165	0.33
A.V.	0.389	Not Set	0.649	Not Set	0.385	0.422	158	Not Set
1	0.37	0.045	0.65	0.37	0.35	0.37	160	0.4
2	NT	NT	NT	NT	NT	NT	NT	NT
3	0.4	NR	0.62	0.33	0.39	0.45	153	0.33
4	NT	NT	NT	NT	NT	NT	NT	NT
5	NR	NR	NR	NR	NR	NR	156.15	NR
6	0.46	0.9	NT	NT	NT	<0.1	156.6	NT
7	0.367	0.061	0.665	NT	0.395	0.396	163	NT
8	NT	NT	NT	NT	NT	<1.0	152	NT
9	0.37	<0.1	0.68	NT	0.385	0.43	163	0.28
10	<2	<2	<1	NT	NT	0.4	150	NT
11	NT	NT	NT	NT	NT	NT	NT	NT
12	NT	NT	NT	NT	NT	NT	NT	NT
13	0.41	<0.5	0.63	0.39	0.37	0.43	158	NT
14	0.365	0.067	NT	NT	NT	NT	164.504	NT
15	NT	NT	NT	NT	NT	NT	NT	NT
16	0.425	NA	NA	NA	0.477	0.861	159	NA
17	0.35	0.11	0.65	0.34	0.38	0.48	159	NT

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

Table 64 Summary of Participants' Results and Performance in S2

Lab. Code	S2-Al	S2-As	S2-B	S2-Ba	S2-Ca	S2-Cd	S2-Co	S2-Cr	S2-Cu	S2-Fe	S2-K
Units	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
H.V.	479	0.536	13.2	7.64	50500	0.056	0.501	9.29	14.3	528	8490
A.V.	Not Set	0.55	13.4	7.3	47800	0.0532	0.469	8.43	14.5	492	8210
1	220	0.48	14	7.1	50000	0.049	0.46	2.3	15	500	7900
2	NT	0.57	NT	NT	NT	0.05	NT	NT	14.2	NT	NT
3	385	0.56	11.7	6.9	46246	0.046	0.51	8.5	14.3	507	7377
4	NT	0.501	NT	6.93	45300	0.0551	0.414	8.18	13	514	7860
5	NR	NR	NR	NR	NR	<0.1	NR	NR	17.24	354.48	7473.1
6	NT	0.5	NT	7.9	52090	0.06	0.46	9.2	13.39	475.8	8442
7	191	0.714	13.4	7.31	42800	0.049	0.588	8.61	14.3	482	8070
8	NT	1.1	NT	6.8	NT	<1.0	<1.0	8	14.2	506	NT
9	215	0.51	18.5	7.1	52390	0.051	0.46	7.7	15	540	9350
10	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
11	229	<2	11.7	7.1	43900	<0.1	0.5	7	11.7	477	8360
12	185	0.627	13.6	7.52	46650	0.055	0.456	2.28	17.2	452	7950
13	276	0.63	13	7.5	55000	0.05	0.44	1.77	14.6	462	9000
14	422.174	0.498	NT	8.059	41881.06	0.056	0.403	10.047	16.501	561.652	8074.876
15	380	0.46	17	6.6	49000	0.06	NT	8.8	13	500	8900
16	216	0.62	12.81	7.24	50700	0.061	0.556	2.39	14.7	506	8400
17	142	0.53	8.41	8.1	45600	0.05	0.41	1.26	14.6	476	8040

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

Table 64 Summary of Participants' Results and Performance in S2 (continued)

Lab. Code	S2-Mg	S2-Mn	S2-Mo	S2-Na	S2-P	S2-Pb	S2-S	S2-Se	S2-TKN	S2-TOC	S2-Zn
Units	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
H.V.	2380	185	3.07	2210	6360	0.459	NA	0.348	NA	NA	111
A.V.	1960	168	3.1	1850	5670	0.456	2520	0.366	Not Set	Not Set	96.5
1	2000	175	3	2000	5000	0.41	2200	0.35	NA	NA	93
2	NT	NT	2.98	NT	NT	0.44	NT	NT	NT	NT	NT
3	1916	153	2.8	1661	5240	0.44	2461	0.35	NT	390000	92
4	2000	151	3.25	1840	5920	0.505	NT	0.365	33400	NT	93.9
5	1984	NR	NR	1691.8	NR	NR	NR	NR	NR	NR	98.8
6	2032	160.6	3.4	1919	6124	0.47	NT	0.31	NT	NT	84.47
7	1900	173	3.29	1770	5190	0.47	2430	0.45	NT	NT	103
8	NT	168	3.3	NT	5830	<1.0	2310	<5.0	NT	NT	99.1
9	2400	182	3.11	2150	6140	0.49	3450	0.64	NT	NT	110
10	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
11	1760	161	3	1810	5540	<1	2390	<2	3240	36100	91
12	1930	195	3	1670	5700	0.457	2540	0.349	31000	324000	98.1
13	2250	177	2.8	1780	5820	0.47	2500	<0.5	31700	290000	98
14	1843.066	183.763	NT	1765.607	NT	0.43	NT	0.409	NT	NT	99.809
15	2000	150	4.2	2200	5700	0.44	2800	NT	NT	NT	98
16	1960	163	3.245	1890	6940	0.473	2660	0.368	NA	NA	96
17	1890	165	2.5	1900	5120	0.44	2580	0.58	2.99	NT	93.3

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

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.¹⁸

11 laboratories reported results in both samples and all used the same extraction regime for both samples

In previous NMI PT studies of metals in food, results were compatible except for Al, Cr, Fe, Ni and V in seaweed, plant material (chicken feed) and oyster tissue. The extraction of these elements is strongly dependent on the digestion regime especially when the plant-based food material has high silica content; an aggressive digestion regime (nitric acid, a high digestion temperature and/or hydrofluoric acid) is recommended for the complete extraction of these elements.

Food laboratories have to test for total elements for a large number of samples and often at the same time 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 this and previous studies of the importance of using a high ratio HCl (mL)/sample size (g) when a high digestion temperature (> 170°C) or if HF cannot be used for Cr, Fe, Ni and V extraction.

Five participants used a digestion temperature higher than 170°C; one participant ashed the samples in a muffle furnace for 24 hours at 500°C.

Six participants used only nitric acid for extraction and except for one all used a digestion temperature lower of 160°C or less.

Laboratory 3 reported: “Sample digested to completion by microwave”.

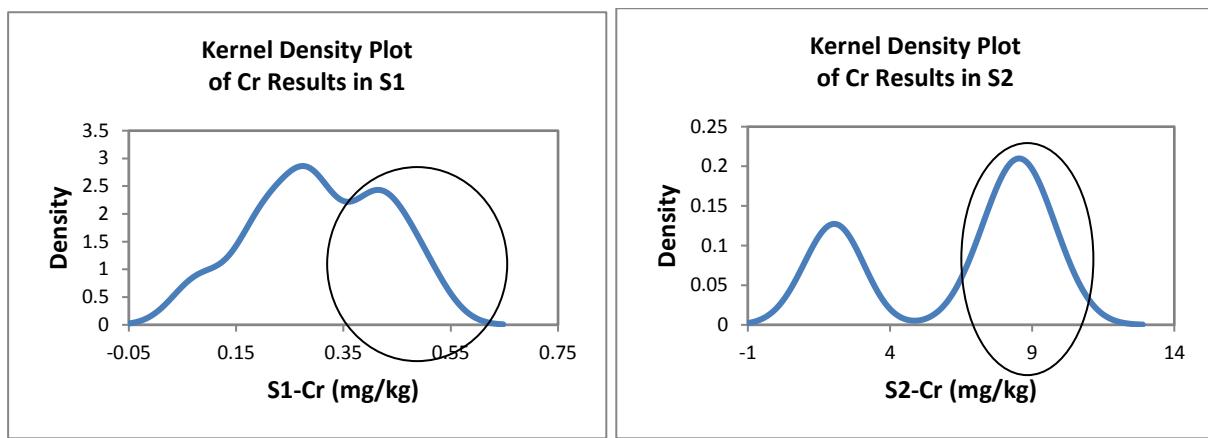
Chromium The results for total Cr in S1 and S2 had a similar bimodal distribution, with all satisfactory results coming from the same laboratories: those which used more aggressive digestion regimes (high digestion temperature and/or a ratio high HCl/sample size).

Similar to previous studies, there was agreement between the results coming from high digestion temperatures and the results coming from a ratio of HCl/sample size of 1 and over (Figure 59).

No association between the results for Cr and the instrumental techniques was apparent.

Aluminium is one of the most difficult elements to analyse in food samples. In the previous PT studies, no assigned values could be set in the plant material (chicken feed), oyster tissue and biota 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 the two study samples were variable, had a bimodal distribution and no assigned value could be set (Figure 60). As the request made was “digestion until extraction was complete” the true value for Al in S1 is most likely similar to the results reported by laboratories 3 and 14, 4.2 mg/kg and 5.811 mg/kg respectively. These results were also in good agreement with the homogeneity value (5.3 mg/kg).



S1 and S2-Cr Participants Performance

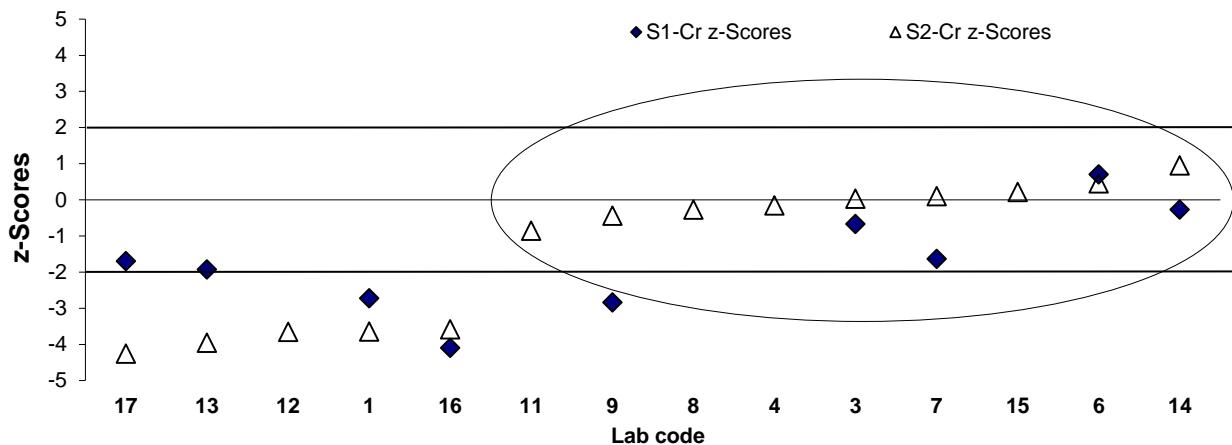


Figure 59 Participants' results/performance versus Digestion Regime

Lab. Code	S1 Cr mg/kg	S2-Cr mg/kg	Digestion Regime				
			HCl (mL)/sample size (g)	Temp (°C)	Time (min)	Sample Size (g)	Reagents Used
17	0.29	1.26	0	85	240	0.5	5 mL HNO ₃ ,
13	0.27	1.77	0	120	60	0.4	10 mL HNO ₃
12	NR	2.28	0	100		0.5	3 mL HNO ₃
9	0.19		0.5	85	60	0.5-1	2.5 mL HNO ₃ , 0.5 mL HCl
1	0.2	2.3	0	160	45	0.5	4 mL HNO ₃
16	0.08	2.39	0	98	90	0.54	3 mL HNO ₃ ,
11	NR	7	5	120	240	0.2	6 mL HNO ₃ , 1 mL HCl, 2 mL H ₂ O ₂
9		7.7	1	85	60	0.5-1	2.5 mL HNO ₃ , 0.5 mL HCl
8	NR	8	10	112.5	120	0.99	10 mL HNO ₃ , 10 mL HCl
4	NR	8.18	5	95	60	0.2	3 mL HNO ₃ , 1 mL HCl
3	0.38	8.5	1	210	15	0.5	7 (1:1) mL HNO ₃ , 0.5 mL HCl, 1 mL H ₂ O ₂
7	0.293	8.61	1.5	110	60	1	5 mL HNO ₃ , 1.5 mL HCl
15	NR	8.8	1	220	30	1	5 mL HNO ₃ , 1 mL HCl
6	0.5	9.2	5	190	30	0.4	5 mL HNO ₃ , 2 mL HCl, 2 (1:1) mL HCl
IV/HV	0.438	9.29	2	260	45	0.5	7 mL HNO ₃ , 1 mL HCl
14	0.415	10.047	1.4	220	45	0.35	

I.V.= Information Value by d-IDMS; H.V.= Homogeneity Value

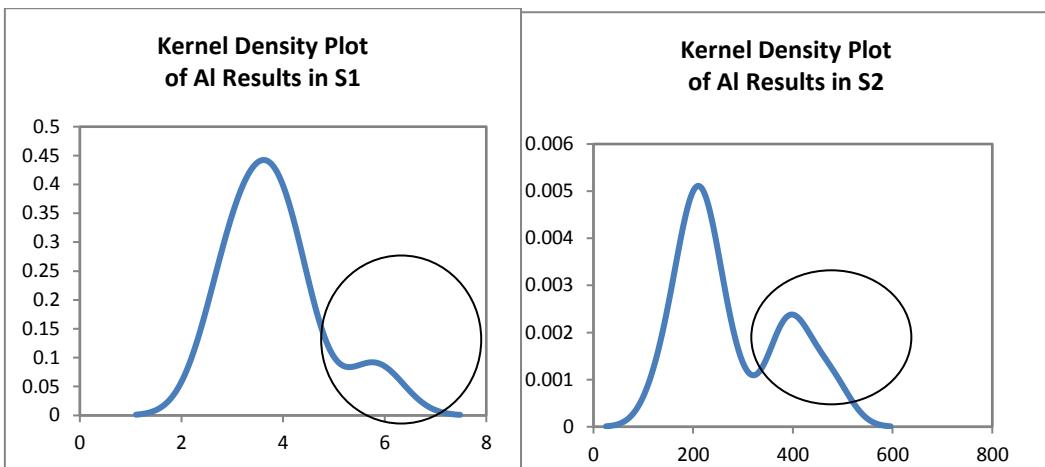


Figure 60 Kernel Density Plot of Al Results in S1 and S2

Lab code	S1 Al Results mg/kg	S2 Al Results mg/kg	Digestion Regime					
			Sample Mass (g)	Temp °C	Time (min.)	HNO ₃ (mL)	HCl (mL)	H ₂ O ₂ (mL)
17	2.78	142	0.5	85	240	5		
12	NR	185	0.5	100		3		
7	3.49	191	0.2	120	240	6	1	
9	3	215	0.5	85	60	2.5	0.5	
16	3.979	216	0.54	98	90	3		
1	3.4	220	0.5	135	45	4		
10	4	NR	1	95	120	7.5	5	
11	NR	229	0.2	120	240	6	1	2
13	NR	276	0.4	120	60	10		
15	NR	380	1	220	30	5	1	
3	4.2	385	0.5	210	15	7 (1:1)	0.5	1
14	5.811	422.174	0.35	220	45	5	0.5	
HV	5.3	479	0.5	260	45	7	1	

HV=Homogeneity Value

The closest value to the true value for Al in S2 is most likely 396 mg/kg, the value on which the high results from the minor mode are distributed. However, based on consensus of these results, no assigned value could be set because their number was too small. All high Al results are from laboratories which used a high digestion temperature of 210°C or more.

Chromium and Aluminium were some of the most difficult elements to analyse in the plant material sample. The plant material was previously distributed as S2 of AQA 13-18.⁶ This allowed for evaluation of laboratories capabilities for measurement of these above analytes in time.

Of 17 laboratories who reported results in the present study, 7 reported results in S2 of AQA 13-18 for Al and Cr (Laboratories 1, 3, 8, 9, 12, 13 and 17).

Laboratories 1, 3, 8, 9 and 12 used the same extraction method in the present study and in AQA 13-18, while Laboratories 13 and 17 excluded HCl from their digestion regime.

Results from the two studies reported by participants for Al and Cr in plant material along with the associated uncertainties, are presented in Figure 61. In some cases, the reported results and the expanded measurement uncertainty in the two study samples are significantly different.

No agreement was found between the results reported for Al in either of the two studies.

The assigned value for Cr in S2 of the present study was 8.43 mg/kg. No assigned value could be set for Cr in the previous study because the reported results varied too much.

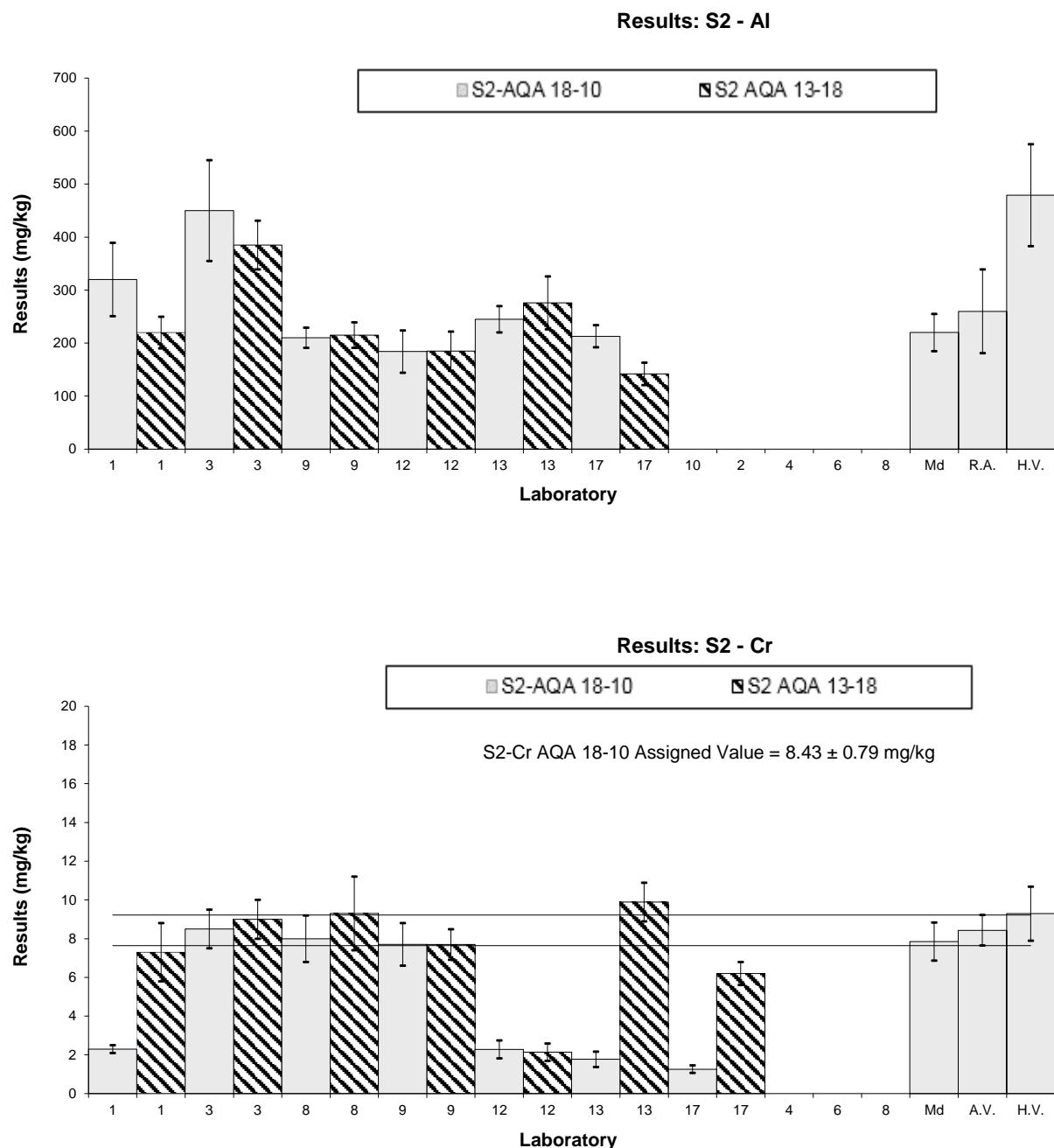
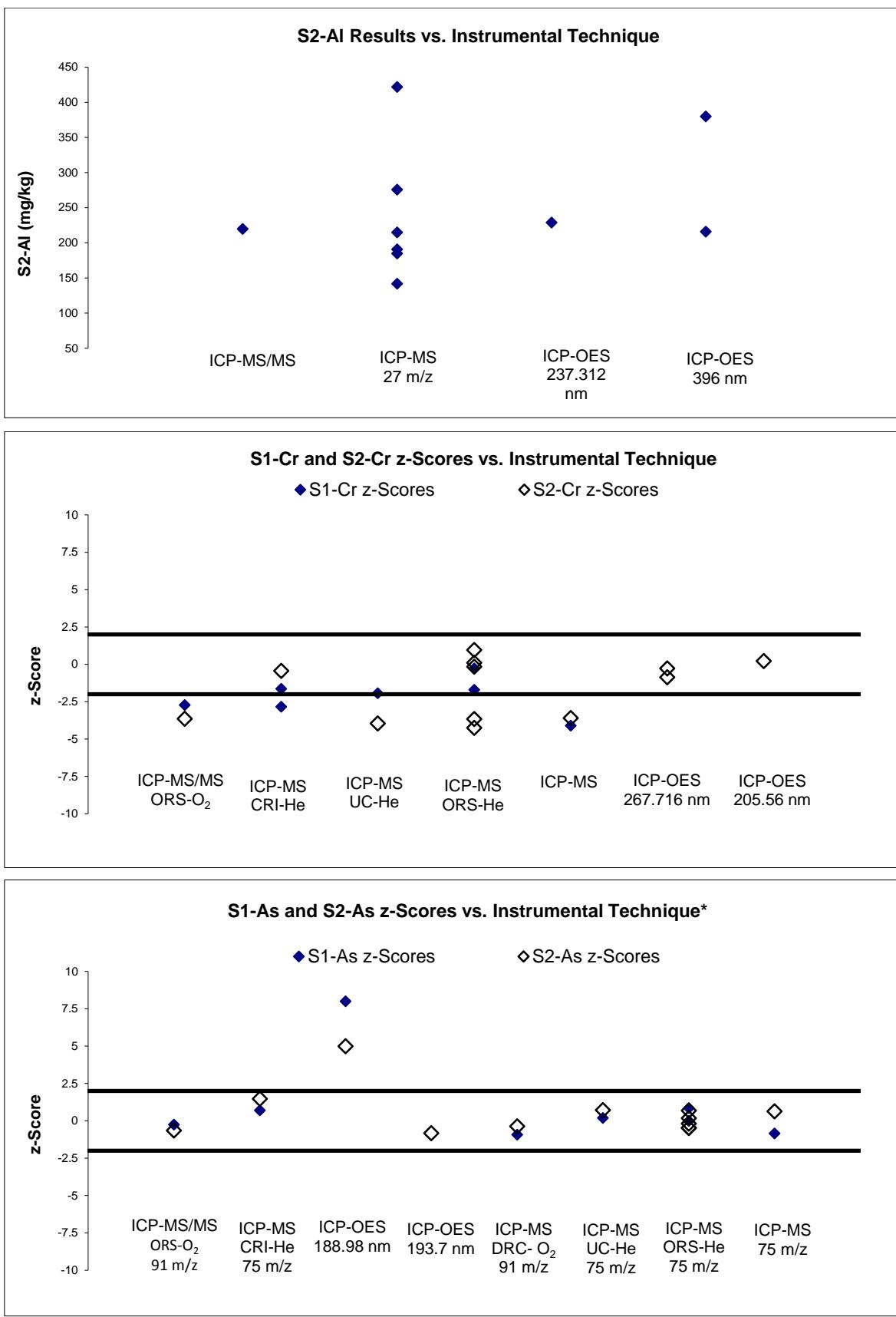


Figure 61 Bar charts of Results in S2 of AQA 13-18 and S2 of AQA 18-10

Instrumental Techniques

Plots of participants' results/ performance with the instrumental technique used are presented in Figure 62.



*z-score larger than 8 has been plotted as 8.

Figure 62: Participants' Results/Performance vs. Instrumental Technique

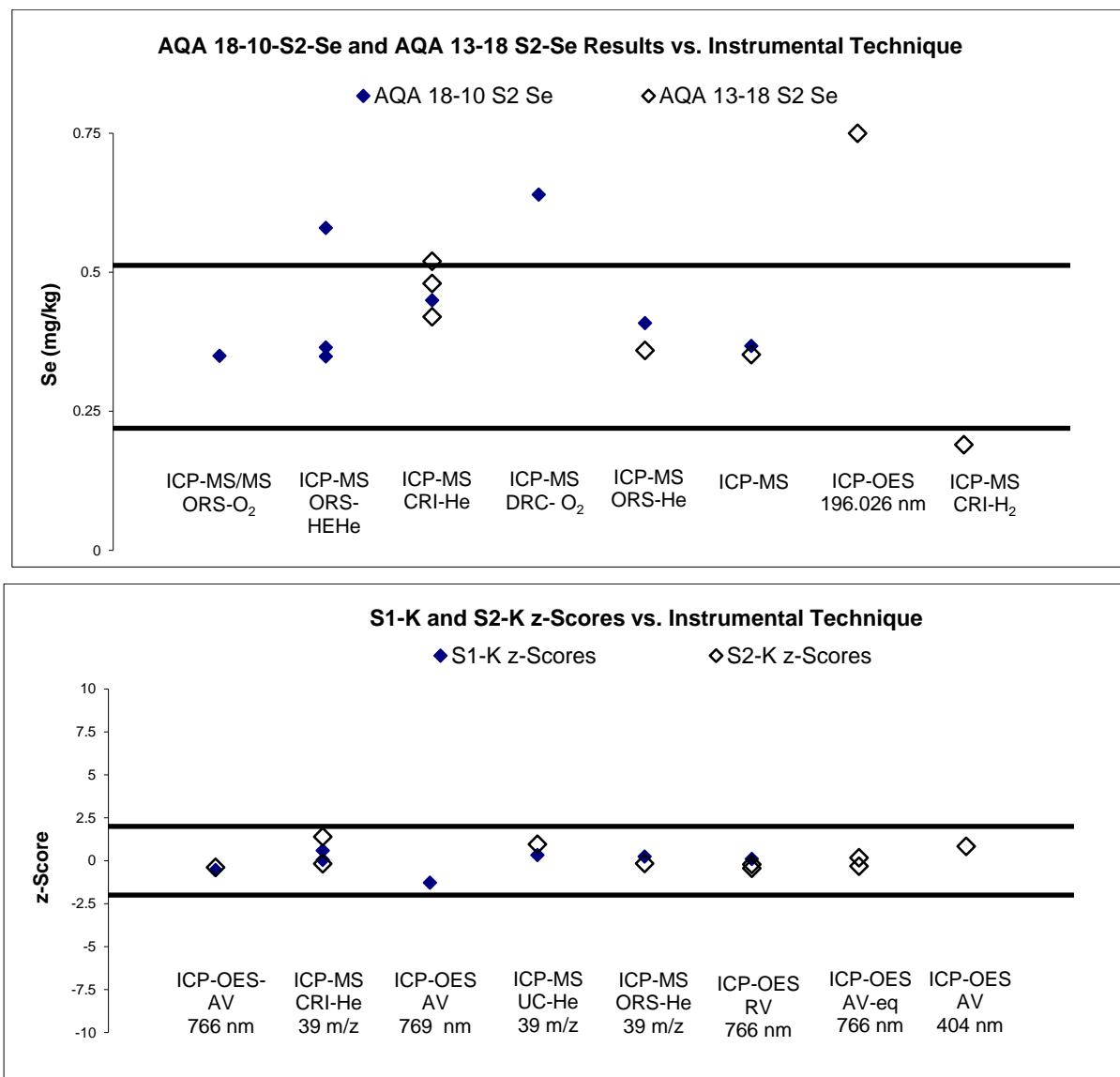


Figure 62: Performance vs. Instrumental Technique (Continued)

Aluminium and Chromium No association between Al and Cr results and the instrumental technique used was apparent (Figure 62).

Arsenic Except for two all the results reported for As in the liver and plant material sample returned satisfactory z-scores. ICP-OES may not be the right technique for measurement of low level As in food samples (Figure 62).

Potassium Participants used a wide variety of measurement techniques for K measurements in the two study samples and all produced comparable results.

Selenium concentration in the plant material sample was low (0.366 mg/kg) and no assigned value could be set for this analyte in the previous study AQA 13-18. Measurement of low level Se in the plant material sample S2 also challenged participants in the present study. Of the 10 reported results 8 returned satisfactory z-scores.

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 from charged carbon species to atoms like Se with a lower ionization potential.¹⁹ Molecular or polyatomic interferences are the other main factor which can affect Se measurements in food. Usually these are: ⁴⁰Ar³⁷Cl on ⁷⁷Se, ¹H⁸¹Br, ¹²C³⁵Cl₂ and ³⁴S¹⁶O₃ on ⁸²Se; ⁴⁰Ar³⁸Ar on ⁷⁸Se; ⁴⁰Ar⁴⁰Ar and ³²S¹⁶O₃ on ⁸⁰Se.¹¹ Concentrations much higher than the true values are frequently obtained when measurements are made by ICP-MS without the help of collision/reaction cell and/or are not corrected for matrix effects.

A plot of participants' results for Se in the two studies samples (S2 of the present study and S2 of AQA 13-18) versus instrumental technique used is presented in Figure 62. Some participants may not have overcome the interference problem.

Sulphur One participant used ICP-MS with collation/reaction cell for S measurement, He as collision gas and Sc as internal standard

6.6 Participants' Results and Analytical Methods for Inorganic As

Inorganic Arsenic was included as a pilot program. 3 participants reported results and all were in good agreement with each other and with homogeneity value (0.330 ± 0.010 mg/kg). Figure 63 presents plots of participants' results versus method used.

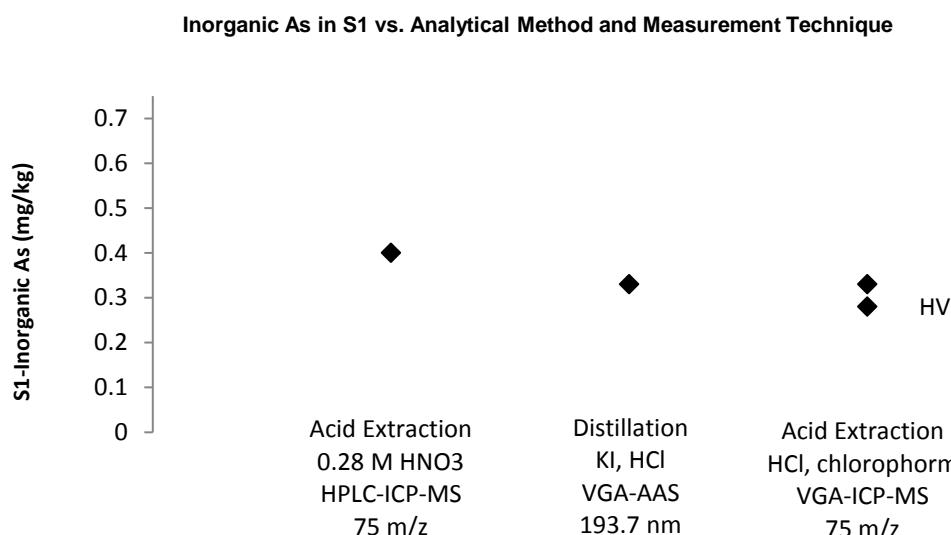


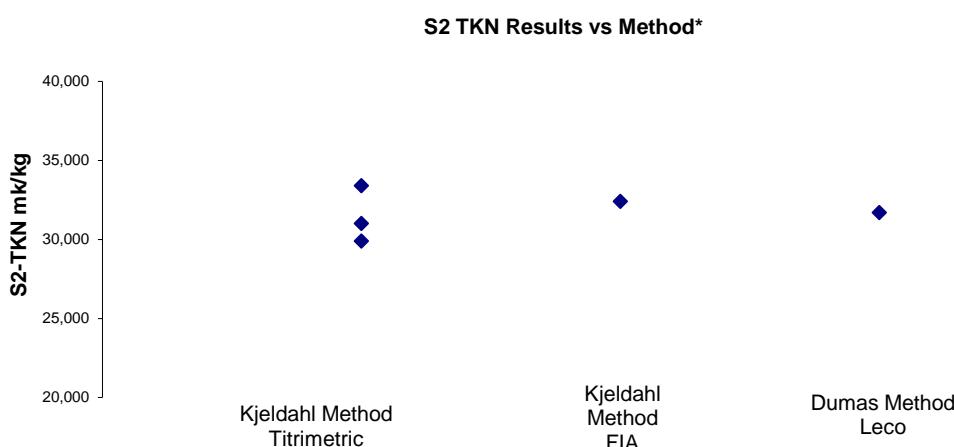
Figure 63: Participants' Results for Inorganic As in S1 vs. Method

6.7 Participants' Results and Analytical Methods for Total Kjeldahl Nitrogen

Only 5 results were reported for TKN in S2 and 3 of those were in good agreement with each other, centred on the 32000 mg/kg value.

Laboratory 17 correctly measured TKN in S2, but reported the result in the wrong units.

Plots of participants' results versus analytical method and measurement technique are presented in Figure 64.



*Results of 3240 mg/kg and 2.99 mg/kg have been plotted as 32400 mg/kg and 29900 mg/kg respectively.

Figure 64: Participants' Results for TKN in S2 vs. Method

6.8 Participants' Results and Analytical Methods for Total Organic Carbon

No assigned value could be set for TOC in S2 because only four participants reported results for this test. Except for one all reported results were in good agreement with each other, centred on the 335000 mg/kg value.

6.9 Participants' Within-Laboratory Reproducibility

Sample S2 was a plant material sample previously distributed as S2 of AQA 13-18. The same target standard deviation was used to calculate z-scores for analytes in both samples. This allowed evaluation of the within laboratory reproducibility of participants.

Of 17 laboratories who reported results in the present study, 8 reported results in AQA 13-18 (Laboratories 1, 3, 8, 9, 10, 12, 13 and 17).

Scatter plots of z-scores in Sample S2 of AQA 18-10 and S2 of AQA 13-18 are presented in Figure 65. Points close to the diagonal axis represent excellent reproducibility and points close to zero represent excellent reproducibility and accuracy.

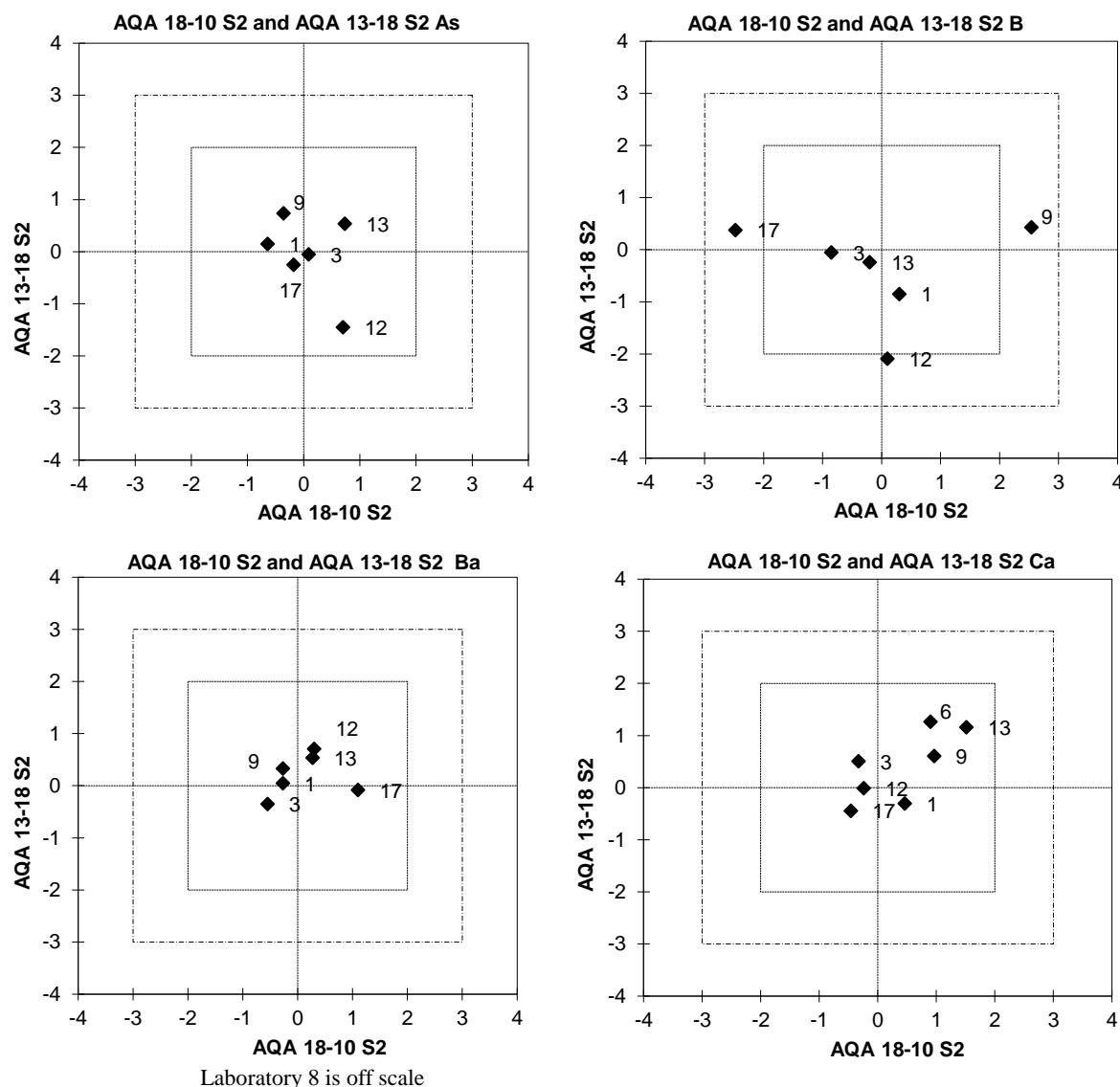


Figure 65 Scatter Plots of: z-Score in S2 of AQA 18-10 and S2 of AQA 13-18

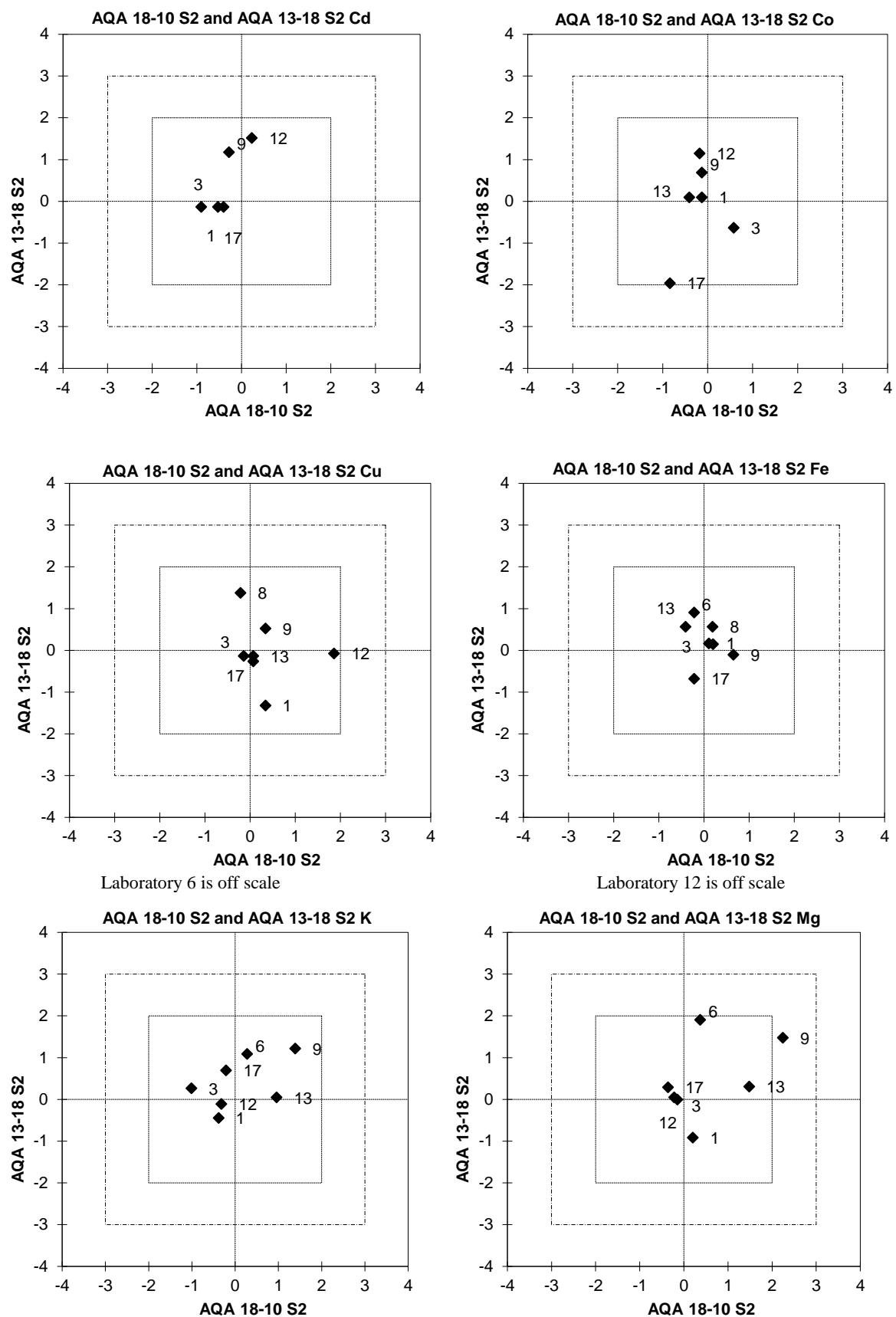


Figure 65 Scatter Plots of: z-Score in S2 of AQA 18-10 and S2 of AQA 13-18 (continued)

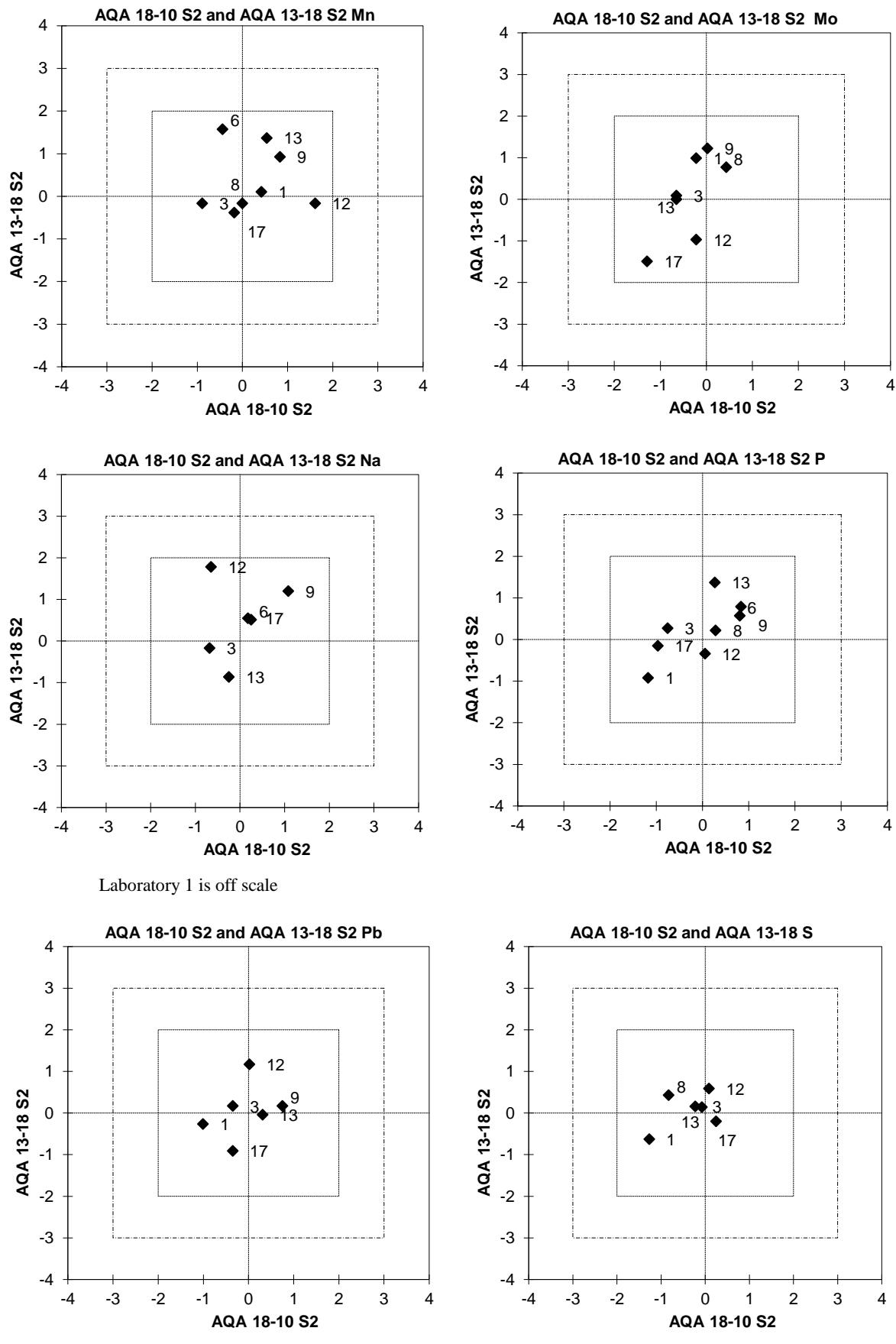


Figure 65 Scatter Plots of: z-Score in S2 of AQA18-10 and S2 of AQA 13-18 (continued)

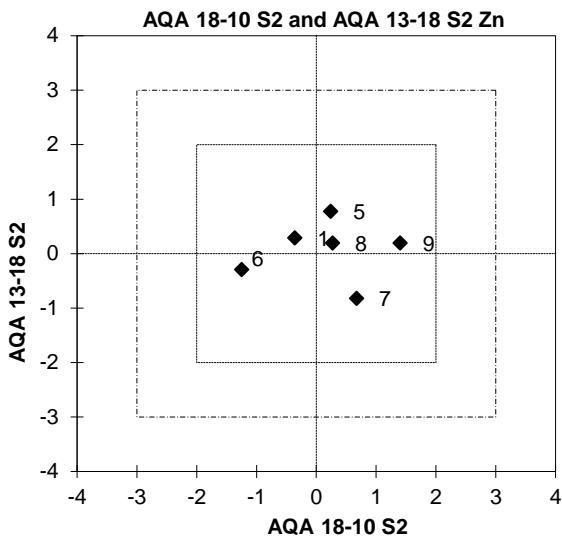


Figure 65 Scatter Plots of: z-Score in S2 of AQA18-10 and S2 of AQA 13-18 (continued)

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

AQA 18-10 is the twelfth NMI study of elements in food. The participants' performance in measurements of trace elements in food over time is presented in Figure 66. Despite differences in matrices and laboratories, performance has remained fairly constant.

Over time laboratories should expect at least 95% of its scores to lay with the range $|z| \leq 2$. Scores in the range $2 < |z| \leq 3$ 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.

6.11 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 65.

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'*²⁰

Table 65 Control Samples Used by Participants

Lab Code	Description of Control Sample
1	RM: In-house reference materials (FQC 311 mussel tissue etc)
4	CRM: agal3, agal4, agal6, agal7, pharmtab9
7	CRM: NIST SRM 1486 (Bone Meal) and 1567B (Wheat Flour)
9	CRM: In house reference samples, NIST 2976, NIST 1568a
12	Previous PT study samples

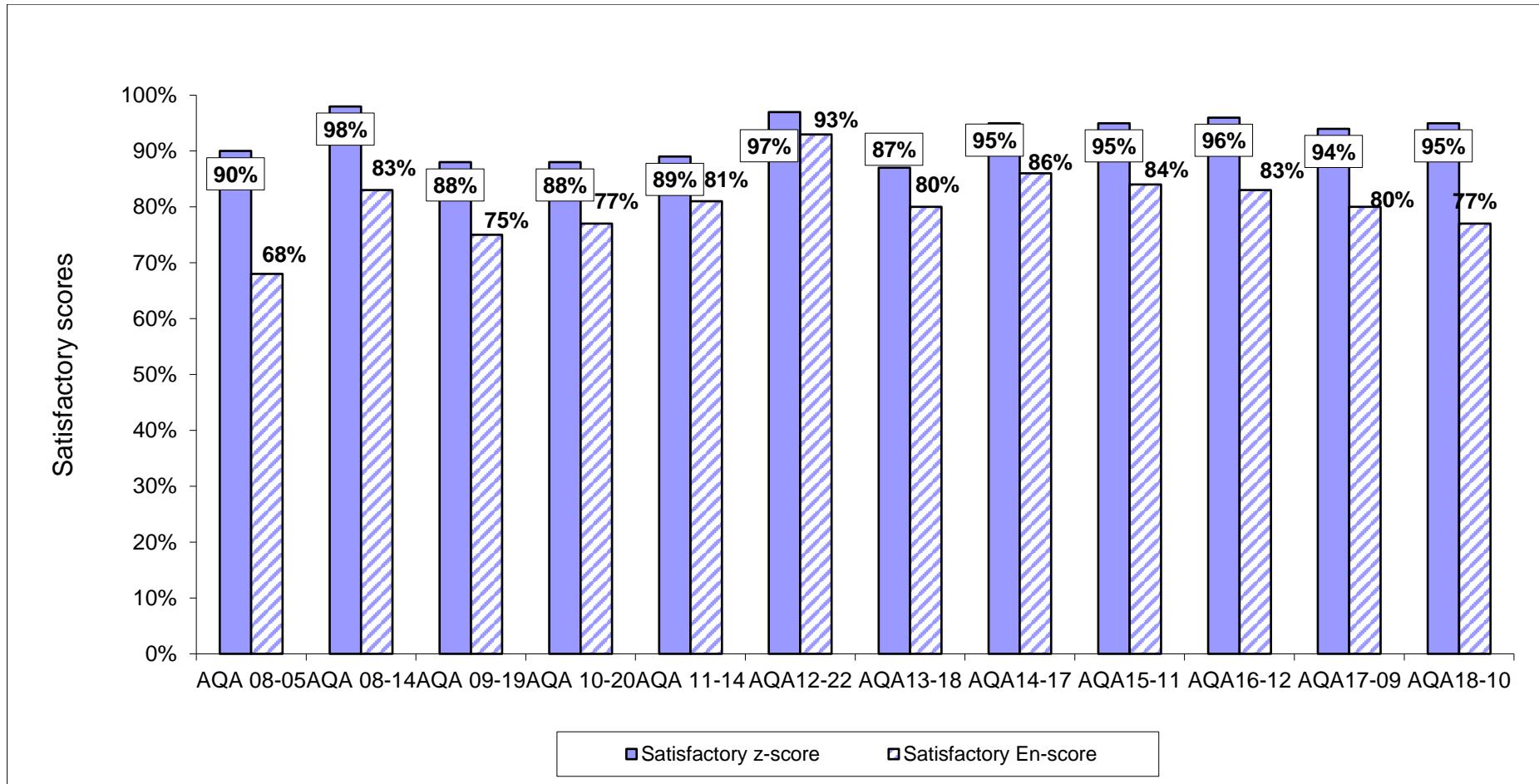


Figure 66 Participants' Performance over Time

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APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING

A1.1 Sample Preparation

Sample S1 was a freeze dried liver, a reference material sample previously prepared by NMI.

Sample S2 was previously distributed as Sample S2 in AQA 13-18. The procedures for preparation and analysis of this material were provided in the report of AQA 13-18.⁵

A1.2 Sample Analysis and Homogeneity Testing

Although the study samples were formerly tested for homogeneity by NMI a partial homogeneity testing was still conducted. The results from the partial homogeneity testing are reported in this study as the homogeneity value. No homogeneity testing was conducted for B in S1 and S, TKN and TOC in S2

Sample Analysis for Total Elements in S1 and S2

Approximately 0.5 g of sample was weighed and digested at 260°C for 45 min with 7 mL HNO₃ and 1 mL HCl. Digestion solutions were clear and colourless. 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 66.

Table 66 Instrumental Techniques Used for Total Elements in S1 and S2 (continued)

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
Al	ICP-MS	Rh	NA	NA	400	400	27 m/z
As	ICP-MS	Rh	ORS	He	400	400	75 m/z
B	ICP-MS	Rh	NA	NA	NA	400	11 m/z
Ba	ICP-MS	Rh	ORS	He	400	400	137 m/z
Be	ICP-MS	Rh	NA	NA	400	NA	9 m/z
Bi	ICP-MS	Ir	OES	He	400	NA	209 m/z
Ca	ICP-MS	Rh	ORS	He	400	400	44 m/z
Cd	ICP-MS	Rh	ORS	He	NA	400	114 m/z
Co	ICP-MS	Rh	ORS	He	NA	400	59 m/z
Cr	ICP-MS	Rh	ORS	He	400	400	53 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	Ir	ORS	He	400	NA	201 m/z
K	ICP-MS	Rh	ORS	He	400	400	39 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	NA	400	95 m/z
Na	ICP-MS	Rh	ORS	He	400	400	23 m/z
Ni	ICP-MS	Rh	ORS	He	400	NA	60 m/z
P	ICP-MS	Rh	ORS	HEHe	400	400	31 m/z
Pb	ICP-MS	Ir	ORS	He	400	400	Average of 206, 207, 208 m/z
Se	ICP-MS	Rh	ORS	HEHe	400	400	78 m/z

Table 66 Instrumental Techniques Used for Total Elements in S1 and S2 (continued)

Analyte	Instrument	Internal Standard	Reaction/Collision Cell (if applicable)	Cell Mode/Gas (if applicable)	S1 Final Dilution Factor	S2 Final Dilution Factor	Ion
Sb	ICP-MS	Rh	ORS	He	400	NA	121 m/z
Sr	ICP-MS	Rh	ORS	He	400	NA	88 m/z
Sn	ICP-MS	Rh	ORS	He	400	NA	118 m/z
Th	ICP-MS	Ir	ORS	He	400	NA	232 m/z
Zn	ICP-MS	Rh	ORS	He	400	400	213 m/z

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 ‘ISO13258:2005(E), Statistical methods for use in proficiency testing by interlaboratory comparisons – Annex C⁷ the uncertainty was estimated as:

$$u_{rob\ av} = 1.25 * S_{rob\ av} / \sqrt{p} \quad \text{Equation 3}$$

where:

- $u_{rob\ av}$ robust average standard uncertainty
- $S_{rob\ mean}$ robust average standard deviation
- p number of results

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

A worked example is set out below in Table 67.

Table 67 Uncertainty of Assigned Value for U in Sample S1

No. results (p)	8
Robust Average	0.385 mg/kg
$S_{rob\ av}$	0.026 mg/kg
$u_{rob\ av}$	0.012 mg/kg
k	2
$U_{rob\ av}$	0.024 mg/kg

The assigned value for U in Sample S1 is **0.385 ± 0.024 mg/kg**.

z-Score and E_n-score

For each participant’s result a z-score and E_n-score are calculated according to Equation 1 and Equation 2 respectively (see page 15).

A worked example is set out below in Table 68.

Table 68 z-Score and E_n-score for U result reported by Laboratory 3 in S1

U Result mg/kg	Assigned Value mg/kg	Set Target Standard Deviation	z-Score	E _n -Score
0.39 ± 0.05	0.385 ± 0.024	10% as CV or 0.1 x 0.385 = 0.0385 mg/kg	$z = \frac{(0.39 - 0.385)}{0.0385}$ $z = 0.130$	$E_n = \frac{(0.39 - 0.385)}{\sqrt{0.05^2 + 0.024^2}}$ $E_n = 0.090$

APPENDIX 3 – REFERENCE VALUES

A.3.1 Description of Method of Analysis

All analytes were quantified by double isotope dilution ICP-MS. NIST SRM 3136 (lot #120619), NIST SRM 3134 (lot #130418) and NIST SRM 3112a (lot #030730) were used as the primary calibration materials and diluted gravimetrically to working concentrations. Isotope dilution was performed by spiking calibration standards and undigested samples with isotopically enriched 61Ni (Oakridge), 100Mo (Oakridge) and 53Cr (Isoflex) standards. Approximately 0.5 g of sample was weighed into a Teflon microwave digestion tube and digested by microwave-assisted pressurised acid digestion using HNO₃ (69% w/w, 7 mL) and HCl (35% w/w, 1 mL) with a maximum digestion temperature of 260°C. Samples were diluted to 50 mL with ultra-high purity water. Digestion solutions were clear and colourless. Experiments also contained QC samples including CRMs and method blanks prepared using the same procedures. Isotope ratios were measured by ICP-SF-MS using medium resolution. For all isotope dilution ICP-MS measurements, sample solutions were bracketed by spiked calibration solutions.

A.3.2 Reference Values

The reference values and associated measurement uncertainty estimates for AQA 18-10 Sample S1 are presented below. The reference values come from the analysis of three bottles in quadruplicate. Measurement uncertainty is given as a 95% level of confidence.

Sample	Analyte	Reference Value	Units	Expanded Uncertainty (95%)	Relative Expanded Uncertainty	Coverage Factor (95%)
AQA 18-10 S1	Ni	0.708	mg/kg	0.033	4.7%	2.03
	Mo	3.903	mg/kg	0.067	1.7%	1.97

Measurements are based on the sample as received (wet mass), no specific assessment for moisture content variation has been made.

A.3.3 Information Values

The primary calibration material used in Cr analysis, SRM 3112a (lot #030730) expired on 01 July 2018 so whilst there is no reason to question the scientific validity of this standard, results are not directly traceable to the SI via this SRM, as such the results have been provided as information values. This information value and associated measurement uncertainty estimates is presented below. The information values come from analysis of three bottles in quadruplicate. Additionally it is noted that the uncertainty is dominated by variation within the sample. Measurement uncertainty is given as a 95% level of confidence.

Sample	Analyte	Reference Value	Units	Expanded Uncertainty (95%)	Relative Expanded Uncertainty	Coverage Factor (95%)
AQA 18-10 S1	Cr	0.438	mg/kg	0.024	5.5%	2.18

Measurements are based on the sample as received (wet mass), no specific assessment for moisture content variation has been made.

A.3.4 Measurement Uncertainty

The measurement uncertainty associated with the reference values takes into account all factors that can reasonably be expected to affect the measurement result. Briefly, these include the accuracy of the primary calibration material, gravimetric preparation, method precision, method trueness and isotopic composition. Measurement uncertainty is reported as a 95% level of confidence using the coverage factors given.

A.3.5 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 and are traceable to the SI unit 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.

APPENDIX 4 - ACRONYMS AND ABBREVIATIONS

CRI	Collision Reaction Interface
COC	Codex Alimentarius Commission
df	Dilution factor
DRC	Dynamic Reaction Cell
ICP-MS	Inductively Coupled Plasma - Mass Spectrometry
ICP-SF-MS	Inductively Coupled Plasma – Sector Field-Mass Spectrometry
ICP-OES-AV	Inductively Coupled Plasma - Optical Emission Spectrometry- axial view
ICP-OES-AV-eq.	Inductively Coupled Plasma - Optical Emission Spectrometry- axial view with correction equation
ICP-OES-RV	Inductively Coupled Plasma - Optical Emission Spectrometry- radial view
Max	Maximum value in a set of results
Md	Median
Min	Minimum value in a set of results
NMI	National Measurement Institute (Australia)
NR	Not Reported
NT	Not Tested
ORS	Octopole Reaction System
PT	Proficiency Test
RM	Reference Material
Robust CV	Robust Coefficient of Variation
Robust SD	Robust Standard Deviation
S	Spiked or formulated concentration of a PT sample
SI	The International System of Units
s_{sam}^2	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
UC	Universal Cell
σ	Target standard deviation

APPENDIX 5 - INSTRUMENT DETAILS

Table 69 Instrument Conditions for Ag in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	27 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	Rh	NA	NA	2000	109
10	ICP-OES-AV	Y			25	328.068
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Rh	NA	NA	625	109
14						
15	NA	NA	NA	NA	NA	NA
16	MS	Ir193			80	109
17	ICP-MS	Ge 72	ORS		50	107m/z

Table 70 Instrument Conditions for Al in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Sc 45	ORS	NA	80	27 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Sc	CRI		40	
8						
9	ICP-MS	Sc	NA	NA	2000	27
10	ICP-OES-AV	Y			25	167.078
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Sc	UC	He	625	27
14	ICP-MS	72Ge	ORS	NA	100	27
15	NA	NA	NA	NA	NA	NA
16	MS	Y89			80	27
17	ICP-MS	Ge 72	ORS		50	27m/z

Table 71 Instrument Conditions for As in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	91 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	188.98
9	ICP-MS	Rh	DRC	O2	2000	91
10	ICP-OES-AV	Y			25	189.042
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Ge	UC	He	625	75
14	ICP-MS	72Ge	ORS	He	100	75
15	NA	NA	NA	NA	NA	NA
16	MS	Y89			80	75
17	ICP-MS	Ge 72	ORS		50	75m/z

Table 72 Instrument Conditions for B in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Sc 45	ORS	NA	80	11 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	Sc	NA	NA	2000	10
10	ICP-OES-AV	Y			25	208.959
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Sc	NA	NA	625	10
14						
15	NA	NA	NA	NA	NA	NA
16	MS	Y89			80	10
17	ICP-MS	Ge 72	ORS		50	11m/z

Table 73 Instrument Conditions for Ba in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	153 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI		40	
8	ICP-OES-AV	Y 371.029			50	233.527
9	ICP-MS	Lu	NA	NA	2000	137
10	ICP-OES-AV	Y			25	233.527
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Rh	NA	NA	625	138
14	ICP-MS	103Rh	ORS	He	100	137
15	NA	NA	NA	NA	NA	NA
16	MS	Ir193			80	137
17	ICP-MS	Rh 103	ORS		50	137m/z

Table 74 Instrument Conditions for Be in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Sc 45	ORS	NA	80	9 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	Sc	NA	NA	2000	9
10	ICP-OES-AV	Y			25	313.042
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Sc	NA	NA	625	9
14	ICP-MS	6Li	ORS	NA	100	9
15	NA	NA	NA	NA	NA	NA
16	NA					
17	ICP-MS	Ge 72	ORS		50	9m/z

Table 75 Instrument Conditions for Bi in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Ir 193	ORS	O2	80	209 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	Ir	NA	NA	2000	209
10						
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Ir	NA	NA	625	209
14						
15	NA	NA	NA	NA	NA	NA
16	NA					
17	ICP-MS	Rh 103	ORS		50	209m/z

Table 76 Instrument Conditions for Ca in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-OES-AV	Eu290.667			80	370.602
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	Sc	NA	NA	2000	43
10	ICP-OES-AV	Y			25	317.933
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Sc	UC	He	625	43
14	ICP-MS	72Ge	ORS	He	5000	44
15	NA	NA	NA	NA	NA	NA
16	OES	Lu			80	315.887
17	ICP-OES-RV	Y377	NA		50	393.366 nm

Table 77 Instrument Conditions for Cd in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	111 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI		40	
8	ICP-OES-AV	Y 371.029			50	228.802
9	ICP-MS	Rh	NA	NA	2000	111
10	ICP-OES-AV	Y			25	228.802
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Rh	NA	NA	625	111
14	ICP-MS	103Rh	ORS	He	100	111
15	NA	NA	NA	NA	NA	NA
16	MS	In115			80	111
17	ICP-MS	Rh 103	ORS		50	111m/z

Table 78 Instrument Conditions for Co in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	59 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	228.615
9	ICP-MS	Sc	CRI	He	2000	59
10	ICP-OES-AV	Y			25	230.786
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Ge	UC	He	625	59
14	ICP-MS	72Ge	ORS	He	100	59
15	NA	NA	NA	NA	NA	NA
16	MS	Y89			80	59
17	ICP-MS	Ge 72	ORS		50	59m/z

Table 79 Instrument Conditions for Cr in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	52 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	267.716
9	ICP-MS	Sc	CRI	He	2000	52
10	ICP-OES-AV	Y			25	205.618
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Sc	UC	He	625	52
14	ICP-MS	103Rh	ORS	He	100	52
15	NA	NA	NA	NA	NA	NA
16	MS	Y89			80	53
17	ICP-MS	Ge 72	ORS		50	52m/z

Table 80 Instrument Conditions for Cu in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	63 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	327.395
9	ICP-MS	Sc	CRI	He	2000	63
10	ICP-OES-AV	Y			25	324.754
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Ge	UC	He	625	63
14	ICP-MS	103Rh	ORS	He	5000	63
15	NA	NA	NA	NA	NA	NA
16	OES	Lu			80	327.393
17	ICP-OES-RV	Y377	NA		50	327.395nm

Table 81 Instrument Conditions for Fe in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	56 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI	HEHe	400	
8	ICP-OES-AV	Y371.029			50	259.94
9	ICP-MS	Sc	CRI	He	2000	56
10	ICP-OES-AV	Y			25	259.941
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Sc	UC	He	625	56
14	ICP-MS	72Ge	ORS	He	5000	56
15	NA	NA	NA	NA	NA	NA
16	OES	Lu			80	238.204
17	ICP-OES-AV	Te214	NA		50	238.204nm

Table 82 Instrument Conditions for Hg in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Ir 193	ORS	O2	80	202 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Ir	CRI		40	
8	CVAFS				500	253.7
9	ICP-MS	Ir	NA	NA	2000	102
10	AAS				2	253.7
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Ir	NA	NA	625	201
14	ICP-MS	159Tb	ORS	He	100	202
15	NA	NA	NA	NA	NA	NA
16	MS	Ir193			80	201
17	ICP-MS	Ir 193	ORS		50	202m/z

Table 83 Instrument Conditions for K in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-OES-AV	Cs697.327			80	766.491
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI	He	4000	
8						
9	ICP-MS	Sc	CRI	He	2000	39
10	ICP-OES-AV	Y			25	769.896
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Sc	UC	He	625	39
14	ICP-MS	72Ge	ORS	He	10000	39
15	NA	NA	NA	NA	NA	NA
16	OES	Lu			800	766.49
17	ICP-OES-RV	Y377	NA		1000	766.491nm

Table 84 Instrument Conditions for Li in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Sc 45	ORS	NA	80	7 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	Sc	NA	NA	2000	7
10	ICP-OES-AV	Y			25	670.78
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Sc	NA	NA	625	7
14						
15	NA	NA	NA	NA	NA	NA
16	NA					
17	ICP-MS	Ge 72	ORS		50	7m/z

Table 85 Instrument Conditions for Mg in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-OES-AV	Eu390.711			80	383.829
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI	He	400	
8						
9	ICP-MS	Sc	CRI	He	2000	24
10	ICP-OES-AV	Y			25	285.213
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Sc	UC	He	625	25
14	ICP-MS	72Ge	ORS	NA	5000	24
15	NA	NA	NA	NA	NA	NA
16	OES	Lu			80	285.213
17	ICP-OES-RV	Y377	NA		50	280.270nm

Table 86 Instrument Conditions for Mn in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	55 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	293.931
9	ICP-MS	Sc	CRI	He	2000	55
10	ICP-OES-AV	Y			25	257.611
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Sc	UC	He	625	55
14	ICP-MS	103Rh	ORS	He	100	55
15	NA	NA	NA	NA	NA	NA
16	MS	Y89			80	55
17	ICP-MS	Ge 72	ORS		50	55m/z

Table 87 Instrument Conditions for Mo in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	95 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	Rh	NA	NA	2000	98
10	ICP-OES-AV	Y			25	202.095
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Rh	NA	NA	625	95
14						
15	NA	NA	NA	NA	NA	NA
16	MS	Y89			80	95
17	ICP-MS	Ge 72	ORS		50	95m/z

Table 88 Instrument Conditions for Na in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-OES-AV	N/A			80	589.592
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI	He	400	
8						
9	ICP-MS	Sc	CRI	He	2000	23
10	ICP-OES-AV	Y			25	589.592
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Sc	UC	He	625	23
14	ICP-MS	72Ge	ORS	NA	5000	23
15	NA	NA	NA	NA	NA	NA
16	OES	Lu			80	589.592
17	ICP-OES-RV	Y377	NA		1000	589.592nm

Table 89 Instrument Conditions for Ni in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	60 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	231.604
9	ICP-MS	Sc	CRI	He	2000	60
10	ICP-OES-AV	Y			25	231.604
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Ge	UC	He	625	60
14	ICP-MS	103Rh	ORS	He	100	60
15	NA	NA	NA	NA	NA	NA
16	MS	Y89			80	60
17	ICP-MS	Ge 72	ORS		50	60m/z

Table 90 Instrument Conditions for P in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-OES-AV	N/A			80	185.878
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-OES-AV				400	
8	ICP-OES-AV	Y 371.029			2500	213.618
9	ICP-MS	Sc	CRI	He	2000	31
10	ICP-OES-AV	Y			25	177.495
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Sc	UC	He	625	31
14						
15	NA	NA	NA	NA	NA	NA
16	OES	Lu			80	178.221
17	ICP-OES-AV	Te214	NA		1000	178.222nm

Table 91 Instrument Conditions for Pb in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Ir 193	ORS	O2	80	208 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Tb	CRI		40	
8	ICP-OES-AV	Y 371.029			50	220.353
9	ICP-MS	Ir	NA	NA	2000	206+207+208
10	ICP-OES-AV	Y			25	220.353
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Ir	NA	NA	625	206+207+208
14	ICP-MS	159Tb	ORS	He	100	208
15	NA	NA	NA	NA	NA	NA
16	MS	Ir193			80	206+207+208
17	ICP-MS	Ir 193	ORS		50	208m/z

Table 92 Instrument Conditions for Se in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	94 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI	HEHe	40	
8	ICP-OES-AV	Y 371.029			50	196.026
9	ICP-MS	Rh	DRC	O2	2000	78
10	ICP-OES-AV	Y			25	196.09
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Rh	NA	NA	625	82
14	ICP-MS	103Rh	ORS	He	100	78
15	NA	NA	NA	NA	NA	NA
16	MS	Y89			80	78
17	ICP-MS	Rh 103	ORS	HEHe	50	78m/z

Table 93 Instrument Conditions for Sb in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	121 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	In	NA	NA	2000	121
10	ICP-OES-AV	Y			25	217.581
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Rh	NA	NA	625	121
14	ICP-MS	103Rh	ORS	He	100	121
15	NA	NA	NA	NA	NA	NA
16	MS	Y89			80	121
17	ICP-MS	Rh 103	ORS		50	121m/z

Table 94 Instrument Conditions for Sn in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	134 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	In	NA	NA	2000	120
10	ICP-OES-AV	Y			25	189.991
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Rh	NA	NA	625	118
14	ICP-MS	103Rh	ORS	He	100	118
15	NA	NA	NA	NA	NA	NA
16	NA					
17	ICP-MS	Rh 103	ORS		50	118m/z

Table 95 Instrument Conditions for Sr in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	88 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	Rh	NA	NA	2000	88
10	ICP-OES-AV	Y			25	407.771
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Rh	NA	NA	625	88
14						
15	NA	NA	NA	NA	NA	NA
16	NA					
17	ICP-MS	Ge 72	ORS		50	88m/z

Table 96 Instrument Conditions for Th in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Ir 193	ORS	HEHe	80	232 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7						
8						
9						
10						
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Ir	NA	NA	625	232
14						
15	NA	NA	NA	NA	NA	NA
16	NA					
17	ICP-MS	Ir 193	ORS		50	232m/z

Table 97 Instrument Conditions for U in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Ir 193	ORS	HeHe	80	238 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	Ir	NA	NA	2000	238
10						
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Ir	NA	NA	625	238
14						
15	NA	NA	NA	NA	NA	NA
16	MS	Ir193			80	238
17	ICP-MS	Ir 193	ORS		50	238m/z

Table 98 Instrument Conditions for V in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	67 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	311.837
9	ICP-MS	Sc	CRI	He	2000	51
10	ICP-OES-AV	Y			25	292.402
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Sc	UC	He	625	51
14						
15	NA	NA	NA	NA	NA	NA
16	MS	Y89			80	51
17	ICP-MS	Ge 72	ORS		50	51m/z

Table 99 Instrument Conditions for Zn in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	66 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	NA	NA	NA	NA	NA	NA
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	213.857
9	ICP-MS	Sc	CRI	He	2000	66
10	ICP-OES-AV	Y			25	231.856
11	NA	NA	NA	NA	NA	NA
12	NA	NA	NA	NA	NA	NA
13	ICP-MS	Ge	UC	He	625	66
14	ICP-MS	103Rh	ORS	He	5000	66
15	NA	NA	NA	NA	NA	NA
16	OES	Lu			80	213.857
17	ICP-OES-AV	Te214	NA		50	206.2nm

Table 100 Instrument Conditions for Al in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Sc 45	ORS	NA	80	27 m/z
2	NA	NA	NA	NA	NA	NA
3						
4						
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	Sc	NA	NA	2000	27
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	237.312
12	icp-ms	Rh			x20	27
13	ICP-MS	Sc	UC	He	625	27
14	ICP-MS	72Ge	ORS	NA	5000	27
15	ICP-OES-AV	NA	NA	NA	50	396.15
16	OES	Lu			80	396.153
17	ICP-MS	Ge 72	ORS		1000	27m/z

Table 101 Instrument Conditions for As in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	91 m/z
2	ICP-MS 7900	Li,Rh,In,Te,Lu	Quadrupole	He	50	75
3						
4	ICP-MS	Germanium	ORS	He	700	75
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	188.98
9	ICP-MS	Rh	DRC	O2	2000	91
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	188.98
12	icp-ms	Rh	ORS		x2	75
13	ICP-MS	Ge	UC	He	625	75
14	ICP-MS	72Ge	ORS	He	100	75
15	ICP-OES-AV	NA	NA	NA	50	193.7
16	MS	In115			80	75
17	ICP-MS	Ge 72	ORS		50	75m/z

Table 102 Instrument Conditions for B in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Sc 45	ORS	NA	80	11 m/z
2	NA	NA	NA	NA	NA	NA
3						
4						
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	Sc	NA	NA	2000	10
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	249.678
12	icp-aes	Y			x2	249.772
13	ICP-MS	Sc	NA	NA	625	10
14						
15	ICP-OES-AV	NA	NA	NA	50	249.68
16	MS	In115			80	10
17	ICP-MS	Ge 72	ORS		50	11m/z

Table 103 Instrument Conditions for Ba in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	153 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	ICP-MS	Rhodium			70	136
5						
6						
7	ICP-MS	Rh	CRI		40	
8	ICP-OES-AV	Y 371.029			50	233.527
9	ICP-MS	Lu	NA	NA	2000	137
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	493.408
12	icp-aes	Y			x2	455.403
13	ICP-MS	Rh	NA	NA	625	138
14	ICP-MS	103Rh	ORS	He	100	137
15	ICP-OES-AV	NA	NA	NA	50	233.53
16	MS	Ir193			80	137
17	ICP-MS	Rh 103	ORS		50	137m/z

Table 104 Instrument Conditions for Ca in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-OES-AV	Eu290.667			80	370.602nm
2	NA	NA	NA	NA	NA	NA
3						
4	ICP-OES-RV	Lutetium			700	315, 317
5						
6						
7	ICP-MS	Rh	CRI		40	
8						
9	ICP-MS	Sc	NA	NA	2000	43
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			500	430.253
12	icp-aes	Y			x100	315.887
13	ICP-MS	Sc	UC	He	625	43
14	ICP-MS	72Ge	ORS	He	40000	44
15	ICP-OES-AV	NA	NA	NA	50	318.13
16	OES	Lu			800	315.887
17	ICP-OES-RV	Y377	NA		1000	393.366nm

Table 105 Instrument Conditions for Cd in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	111 m/z
2	ICP-MS 7900	Li,Rh,In,Te,Lu	Quadrupole	He	50	111
3						
4	ICP-MS	Rhodium			70	111114
5						
6						
7	ICP-MS	Rh	CRI		40	
8	ICP-OES-AV	Y 371.029			50	228.802
9	ICP-MS	Rh	NA	NA	2000	111
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	214.439
12	icp-ms	Rh	ORS		x2	111
13	ICP-MS	Rh	NA	NA	625	111
14	ICP-MS	103Rh	ORS	He	100	111
15	ICP-OES-AV	NA	NA	NA	50	214.44
16	MS	In115			80	111
17	ICP-MS	Rh 103	ORS		50	111m/z

Table 106 Instrument Conditions for Co in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	59 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	ICP-MS	Germanium	ORS	He	70	59
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	228.615
9	ICP-MS	Sc	CRI	He	2000	59
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	231.16
12	icp-ms	Rh	ORS		x2	59
13	ICP-MS	Ge	UC	He	625	59
14	ICP-MS	72Ge	ORS	He	100	59
15	ICP-OES-AV	NA	NA	NA	50	NA
16	MS	In115			80	59
17	ICP-MS	Ge 72	ORS		50	59m/z

Table 107 Instrument Conditions for Cr in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	52 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	ICP-MS	Germanium	ORS	He	700	52, 53
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	267.716
9	ICP-MS	Sc	CRI	He	2000	52
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	267.716
12	icp-ms	Rh	ORS		x2	52
13	ICP-MS	Sc	UC	He	625	52
14	ICP-MS	103Rh	ORS	He	100	52
15	ICP-OES-AV	NA	NA	NA	50	205.56
16	MS	In115			80	53
17	ICP-MS	Ge 72	ORS		50	52m/z

Table 108 Instrument Conditions for Cu in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	63 m/z
2	ICP-MS 7900	Li,Rh,In,Te,Lu	Quadrupole	He	50	63
3						
4	ICP-MS	Germanium	ORS	He	700	63,65
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	327.395
9	ICP-MS	Sc	CRI	He	2000	63
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	327.395
12	icp-ms	Rh	ORS		x2	65
13	ICP-MS	Ge	UC	He	625	63
14	ICP-MS	103Rh	ORS	He	2000	63
15	ICP-OES-AV	NA	NA	NA	50	327.4
16	MS	In115			80	65
17	ICP-MS	Ge 72	ORS		1000	63m/z

Table 109 Instrument Conditions for Fe in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	56 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	ICP-OES-AV	Lutetium			700	259
5						
6						
7	ICP-MS	Rh	CRI	HeHe	400	
8	ICP-OES-AV	Y 371.029			50	259.94
9	ICP-MS	Sc	CRI	He	2000	59
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	238.204
12	icp-aes	Y			x20	238.204
13	ICP-MS	Sc	UC	He	625	56
14	ICP-MS	72Ge	ORS	He	2000	56
15	ICP-OES-AV	NA	NA	NA	50	261.19
16	OES	Lu			80	238.204
17	ICP-OES-AV	Te214	NA		50	238.204nm

Table 110 Instrument Conditions for K in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-OES-AV	Cs697.327			80	766.491nm
2	NA	NA	NA	NA	NA	NA
3						
4	ICP-OES-RV	Lutetium			700	766
5						
6						
7	ICP-MS	Rh	CRI	He	4000	
8						
9	ICP-MS	Sc	CRI	He	2000	39
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	766.491
12	icp-aes	Y			x20	766.491
13	ICP-MS	Sc	UC	He	625	39
14	ICP-MS	72Ge	ORS	He	10000	39
15	ICP-OES-AV	NA	NA	NA	50	404.72
16	OES	Lu			80	766.49
17	ICP-OES-RV	Y377	NA		1000	766.491nm

Table 111 Instrument Conditions for Mg in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-OES-AV	Eu390.711	ORS	O2	80	383.829nm
2	NA	NA	NA	NA	NA	NA
3						
4	ICP-OES-RV	Lutetium			70	285
5						
6						
7	ICP-MS	Rh	CRI	He	400	
8						
9	ICP-MS	Sc	CRI	He	2000	24
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	279.078
12	icp-aes	Y			x20	279.8
13	ICP-MS	Sc	UC	He	625	25
14	ICP-MS	72Ge	ORS	NA	5000	24
15	ICP-OES-AV	NA	NA	NA	50	279.08
16	OES	Lu			80	285.213
17	ICP-OES-RV	Y377	NA		50	280.270nm

Table 112 Instrument Conditions for Mn in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	55 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	ICP-OES-AV	Lutetium			70	257
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	293.931
9	ICP-MS	Sc	CRI	He	2000	55
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	257.61
12	icp-ms	Rh	ORS		x2	55
13	ICP-MS	Sc	UC	He	625	55
14	ICP-MS	103Rh	ORS	He	5000	55
15	ICP-OES-AV	NA	NA	NA	50	293.93
16	OES	Lu			80	257.61
17	ICP-OES-AV	Te214	NA		50	191.466

Table 113 Instrument Conditions for Mo in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	95 m/z
2	ICP-MS 7900	Li,Rh,In,Te,Lu	Quadrupole	He	50	95
3						
4	ICP-MS	Rhodium			70	98
5						
6						
7	ICP-MS	Rh	CRI		40	
8	ICP-OES-AV	Y 371.029			50	202.032
9	ICP-MS	Rh	NA	NA	2000	98
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	202.032
12	icp-ms	Rh	ORS		x2	95
13	ICP-MS	Rh	NA	NA	625	95
14						
15	ICP-OES-AV	NA	NA	NA	50	204.6
16	MS	In115			80	95
17	ICP-MS	Ge 72	ORS		50	95m/z

Table 114 Instrument Conditions for Na in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-OES-AV	N/A			80	589.592nm
2	NA	NA	NA	NA	NA	NA
3						
4	ICP-OES-RV	Lutetium			70	589
5						
6						
7	ICP-MS	Rh	CRI	He	400	
8						
9	ICP-MS	Sc	CRI	He	2000	23
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	589.592
12	icp-aes	Y			x20	588.995
13	ICP-MS	Sc	UC	He	625	23
14	ICP-MS	72Ge	ORS	NA	5000	23
15	ICP-OES-AV	NA	NA	NA	50	568.82
16	OES	Lu			80	589.592
17	ICP-OES-RV	Y377	NA		50	589.592nm

Table 115 Instrument Conditions for P in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-OES-AV	N/A			80	185.878nm
2	NA	NA	NA	NA	NA	NA
3						
4	ICP-OES-AV	Lutetium			70	178
5						
6						
7	ICP-OES-AV				400	
8	ICP-OES-AV	Y 371.029			1000	213.618
9	ICP-MS	Sc	CRI	He	2000	31
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	231.618
12	icp-aes	Y			x20	177.434
13	ICP-MS	Sc	UC	He	625	31
14						
15	ICP-OES-AV	NA	NA	NA	50	185.88
16	OES	Lu			80	178.221
17	ICP-OES-AV	Te214	NA		1000	178.222nm

Table 116 Instrument Conditions for Pb in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Ir 193	ORS	O2	80	208 m/z
2	ICP-MS 7900	Li,Rh,In,Te,Lu	Quadrupole	He	50	206
3						
4	ICP-MS	Rhodium			70	206207208
5						
6						
7	ICP-MS	Tb	CRI		40	
8	ICP-OES-AV	Y 371.029			50	220.353
9	ICP-MS	Ir	NA	NA	2000	206+207+208
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	220.353
12	icp-ms	Ir	ORS		x2	207
13	ICP-MS	Ir	NA	NA	625	206+207+208
14	ICP-MS	159Tb	ORS	He	100	208
15	ICP-OES-AV	NA	NA	NA	50	220.35
16	MS	Ir193			80	206+207+208
17	ICP-MS	Ir 193	ORS		50	208m/z

Table 117 Instrument Conditions for S in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-OES-AV	N/A			80	181.972 nm
2	NA	NA	NA	NA	NA	NA
3						
4						
5						
6						
7	ICP-OES-AV				40	
8	ICP-OES-AV	Y 371.029			50	181.972
9	ICP-MS	Sc	CRI	He	2000	34
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	181.972
12	icp-aes	Y			x20	181.972
13	ICP-OES-AV	Y	NA	NA	62.5	181.975
14						
15	ICP-OES-AV	NA	NA	NA	50	182.56
16	OES	Lu			80	181.975
17	ICP-OES-AV	Te214	NA		1000	181.972nm

Table 118 Instrument Conditions for Se in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	94 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	ICP-MS	Germanium	ORS	HEHe	700	78
5						
6						
7	ICP-MS	Rh	CRI	HEHe	40	
8	ICP-OES-AV	Y 371.029			50	196.026
9	ICP-MS	Rh	DRC	O2	2000	78
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	196.026
12	icp-ms	Rh	ORS		x2	78
13	ICP-MS	Rh	NA	NA	625	82
14	ICP-MS	103Rh	ORS	He	100	78
15	ICP-OES-AV	NA	NA	NA	50	NA
16	MS	In115			80	78
17	ICP-MS	Rh 103	ORS	HEHe	50	78m/z

Table 119 Instrument Conditions for Zn in S2

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	ICP-MS/MS	Rh 103	ORS	O2	80	66 m/z
2	NA	NA	NA	NA	NA	NA
3						
4	ICP-OES-AV	Lutetium			70	206
5						
6						
7	ICP-MS	Rh	CRI	He	40	
8	ICP-OES-AV	Y 371.029			50	213.857
9	ICP-MS	Sc	CRI	He	2000	66
10	NA	NA	NA	NA	NA	NA
11	ICP-OES-AV-equation	Lu			100	206.2
12	icp-ms	Rh	ORS		x2	66
13	ICP-MS	Ge	UC	He	625	66
14	ICP-MS	103Rh	ORS	He	5000	66
15	ICP-OES-AV	NA	NA	NA	50	213.86
16	OES	Lu			80	213.857
17	ICP-OES-AV	Te214	NA		50	206.200nm

END OF REPORT