Australian Government
Department of Industry, Innovation and Science

National Measurement Institute

Proficiency Test Report
AQA 19-10
Total Elements in Food

November 2019

ACKNOWLEDGMENTS

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

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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1 SUMMARY

This report presents the results of the proficiency test AQA 19-10 – Inorganic Contaminants and Nutrients in Food. The study focused on the measurement of inorganic As and total: Ag, Al, As, B, Ba, Bi, Ca, Cd, Cr, Cs, Cu, Fe, Hg, K, La, Li, Mg, Mn, Na, Ni, P, Pb, Rb, S, Se, Sb, Sn, Sr, U, V and Zn in a freeze dried prawn sample, the NMI CRM MX009 and of total: Al, As, B, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, P, Pb, S, Se, Sr, V, Zn and water soluble iodine in infant formula. Measurement of inorganic arsenic was included as a pilot program.

Fifteen laboratories registered to participate and all submitted results.

The assigned values were the robust averages of participants' results, except for As, Cr, Cu, Fe, Hg, Mn, Ni, and Se in the prawn Sample S1. The associated uncertainties were estimated from the robust standard deviation of the participants' results. For As, Cr, Cu, Fe, Mn, Ni and Se in S1 the assigned values were reference values measured using standard addition mass spectrometry (Appendix 3). For Hg in S1 the assigned value was the certified value of MX009 for Hg previously measured by NMI using isotope dilution mass spectrometry (Appendix 4).

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 401 z-scores, 376 (94%) were satisfactory with $|z| \leq 2$.

Of 401 E_n-scores, 334 (83%) were satisfactory with $|E_n| \leq 1$.

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

Evidence was found in the previous and present studies of the importance of using digestion temperature of 170ºC or higher and/or a digestion regime which involves a high ratio HCl (mL)/sample size (g) when total elements except Al is requested.

Some participants may need to reassess their extraction method since they only recovered a fraction of Al, Cr, Ni and V from the freeze dried prawn sample. According to Eurachem/CITAC Guide CG 4, laboratories should consider using matrix matched control samples to assess their extraction efficiency (the bias of their analytical methods). Bias can be expressed as recovery and should be corrected for or included in the uncertainty estimate.[1]

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

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.

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

Of 460 numerical results, 437 (95%) were reported with an expanded measurement *uncertainty.*

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

Sample S1 of the present study is the NMI certified reference material MX009 which is to be certified for 8 more elements (As, Co, Cr, Cu, Fe, Mn, Ni, Se). Further investigation will be conducted on Al, V and Zn in this material for possible certification. MX009 (with the reissued certificate) will be available for sale from NMI in 2020. Sample S2 of this study was checked for homogeneity and is well characterised, both by in-house testing and from the results of the proficiency round. Surplus of this test sample is available from NMI.

2 INTRODUCTION

2.1 NMI Proficiency Testing Program

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

Proficiency testing (PT): "is evaluation of participant performance against pre-established criteria by means of interlaboratory comparison."[2] NMI PT studies target chemical testing in areas of high public significance such as trade, environment and food safety. NMI offers studies in:

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

AQA 19-10 is the thirteenth NMI proficiency test on inorganic contaminants and nutrients in food.

2.2 Study Aims

The aims of the study were to:

- compare the performance of participant laboratories and assess their accuracy;
- evaluate the laboratories' methods used in determination of inorganic analytes in food;
- compare the performance of participant laboratories with their past performance; and
- develop the practical application of traceability and measurement uncertainty.
- produce materials that can be used in method validation and as control samples.

2.3 Study Conduct

The conduct of NMI proficiency tests is described in the NMI Chemical Proficiency Testing Study Protocol.[3] The statistical methods used are described in the NMI Chemical Proficiency Statistical Manual.[4] These documents have been prepared with reference to ISO Standard 17043[2] and The International Harmonised Protocol for Proficiency Testing of (Chemical) Analytical Laboratories.[5]

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

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

3 STUDY INFORMATION

3.1 Selection of Matrices and Inorganic Analytes

The 55 tests in the study samples were representative of those commonly measured in food, and included toxic elements such as Cd and Pb and nutrient elements such as Na, P and Mg.

3.2 Participation

Fifteen laboratories participated and submitted results.

The timetable of the study was:

Invitation issued:	11 June 2019
Samples dispatched:	1 July 2019
Results due:	2 August 2019
Interim report issued:	8 August 2019

3.3 Test Material Specification

Two samples were provided for analysis:

- Sample S1 was 15 g of freeze dried prawn, the certified reference material MX009 previously prepared by NMI; and
- Sample S2 was 25 g of infant formula.

3.4 Laboratory Code

All participant laboratories were assigned a confidential code number.

3.5 Sample Preparation, Analysis and Homogeneity Testing

A partial homogeneity testing was conducted in this study. The same sample preparation procedure was followed as in previous studies.[2] The test samples from the previous studies were demonstrated to be sufficiently homogeneous for evaluation of participants' performance.[6, 7] The results from the partial homogeneity testing are reported in this study as homogeneity values. For Al, As, Cr, Cu, Fe, Hg, Mn, Ni, Se, V and Zn in S1 homogeneity values are the reference and information values (see Appendices 3 and 4). No homogeneity testing was conducted for Rb, S and Sb in S1 and Iodine, Pb and S 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.

3.6 Stability of Analytes

No stability study was carried out during the period of the present study. Stability studies conducted for the previous proficiency tests of metals and nutrients in food found no significant changes in any of the analytes' concentration.[6,7] Results of this study gave no reason to question the stability of the test samples.

3.7 Sample Storage, Dispatch and Receipt

The samples were dispatched by courier on 1st July 2019.

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

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

3.8 Instructions to Participants

Participants were instructed as follows:

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

SAMPLE S1		SAMPLE S2	
Test (Total)	mg/kg	Test (Total)	mg/kg
Ag	0.005-0.2	Al	0.5-20
Al	50-2000	As	<0.05
As (total)	0.25-7.5	B	0.05-2

Inorganic As	0.05- 2	Ba	0.05-2
B	0.05- 2	Ca	500-20000
Ba	0.10-4	Co	<0.05
Bi	0.005-0.25	Cd	<0.05
Ca	1500-7500	Cr	0.05-2
Cd	0.05-2	Cu	0.5-20
Cr	0.05-2	Fe	50-2000
Cs	0.005-0.2	Iodine (water soluble)	0.25-10
Cu	0.5-20	K	500-20000
Fe	50-2000	Mg	50-2000
Hg	0.05-2	Mn	0.5-20
K	500-20000	Mo	0.05-2
La	0.005-0.25	Na	500-20000
Li	0.05-2	P	500-20000
Mg	500-20000	Pb	<0.05
Mn	0.5-20	S	NA
Na	500-20000	Se	0.05-2
Ni	0.05-2	Sr	0.5-20
P	500-20000	V	0.005-2
Pb	0.05-2	Zn	25-1000
Rb	0.05-2		
S	500-20000		
Sb	0.005-2		
Se	0.25-7.5		
Sn	0.05-2		
Sr	25-1000		
U	0.005-0.2		
V	0.05-2		
Zn	25-1000		

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 2nd August 2019. Late results cannot be included in the report.

3.9 Interim Report

An interim report was e-mailed to participants on 8th August 2019.

4 PARTICIPANT LABORATORY INFORMATION

4.1 Test Method Summaries

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

Table 1 Methodology for Total Elements in S1

Lab. Code	Method Reference	Sample Mass (g)	Digestion Temp. (ºC)	Digestion Time (min)	Vol. HNO_3 (mL)	Vol. HNO_3 (1:1) (mL)	Vol. HCl (mL)	Vol. HCl dilute (mL)	Other
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]	In House based on APHA 3125	0.5	95	120	8		1		2 mL H_2O_2
4[a]	In House	0.51	105	60	2.5		0.5		2 mL H_2O
7	In house	0.5	85	60	2.5		0.5		
8[a]		0.1	200	18	10				
9[a]	EN 13805:2002	0.5	210	15		7	0.5		
10	In house reverse aqua rega digestion	0.5	110	60	5		1.5		
11	In house method based on AS 3641.2	0.5	220	30	5		1		
12	AOAC 990.10 Ch 9.1.08	0.5	85	240	5		5		
13	In House	1	112.5	120	10		10		
14	USEPA method 200.2 Revision 2.8	1	95	60		2		10 (1:4)	2 mL H_2O_2

[a] Additional information in Table 5

Table 2 Methodology for Inorganic As in S1

Lab. Code	Method Reference	Preparation Method	Reagents used	Determination Technique
1	In-house method (QIS 27644) arsenic speciation in seafood samples by HPLC-ICPMS	acid extraction	2% HNO_3 + 3% H_2O_2	HPLC-ICP-MS (75 m/z)
7	The Analyst, 1999 124	acid extraction	HCl, Chloroform	ICP-MS (75 m/z)
12		water extraction	Sodium phosphate buffer	HPLC-ICP-MS (75 m/z)

Table 3 Methodology for Water Soluble Iodine in S2

Lab. Code	Preparation Method	Determination Technique	Method Reference	Wavelength/ Ion absorbance
12	TMAH Digestion	ICP-MS		27
15			Microwave step included at 250 watts for 20 min followed by venting for 8 min on Milestone Start D	

Table 4 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_2O_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			
5[a]	GB 5009.268	0.5	180	80	10			
6		0.5			3		1	
7	In house	0.5	85	60	2.5		0.5	
10[a]	In house reverse aqua regia digestion	0.5-1	110	60	5		1.5	
11	In house method based on AS 3641.2	0.5	220	30	5		1	
12	AOAC 990.10 Ch9.1.08	0.5	85	240	5			
13	In house	1	112.5	120	10		10	
15	USEPA 6020, USEPA 6010B	0.2	95	60	3		1	

a Additional information in Table 5

4.2 Instruments Used for Measurements

The instruments and settings used by participants are presented in Appendix 6.

4.3 Additional Information

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

Table 5 Additional Information

Lab. Code	Additional Information
3	Sample S1: Sample is digested with the acids for 1 hour at 95°C and then the H2O2 is added and the sample digested for a further hour at 85°C
4	Sample S1: Note: Selenium and Total Arsenic were prepared using a different extraction method: sample mass = 0.5 g, extraction with 5 mL of 12.5% Tetramethylammonium hydroxide, digestion temp 115°C for 60 mins. IT S1: As-75 corrected for Nd, Sm; Se-82 corrected for S, Br; Sr-88 corrected for Ca; Cd-111 corrected for Mo.
5	Uncertainty: Estimation of MU from within-laboratory data on bias and precision has been calculated by using the procedures outlined in ASTM E2554-13 Standard Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques
8	Sample S1: Ramp time to 200°C 18 minutes
9	Sample S1: Digested to completion by microwave
10	Sample S2: 0.5 g sample used for Se. 1 g sample used for all other elements
14	Uncertainty: Macro MU calculation pack based on QC data

4.4 Basis of Participants' Measurement Uncertainty Estimates

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

Table 6 Basis of Uncertainty Estimate

Lab. Code	Approach to Estimating MU	Information Sources for MU Estimation		Guide Document for Estimating MU
		Precision[a]	Method Bias[a]	
1	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples – RM Duplicate Analysis	CRM Laboratory bias from PT Recoveries of SS Standard Purity Variation in sample moisture content	NATA Technical Note 33
2	Standard deviation of replicate analyses multiplied by 2 or 3	Standard deviation from PT studies only		NATA Technical Note 33
3	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples – CRM	CRM	IANZ Technical Guide
4	Standard deviation of replicate analyses multiplied by 2 or 3	Control Samples – CRM Duplicate Analysis Instrument Calibration	CRM Instrument Calibration Recoveries of SS	Eurachem/CITAC Guide
5*	Refer to Table 5	Control Samples - CRM	CRM Instrument Calibration	Refer to Table 5
6		Control Samples Duplicate Analysis Instrument Calibration		
7	Top Down - precision and estimates of the method and laboratory bias		CRM	IANZ Technical Guide
8	Standard deviation of replicate analyses multiplied by 2 or 3	Duplicate Analysis	CRM	Nordtest Report TR537
9	Top Down - precision and estimates of the method and laboratory bias	Control Samples – CRM Duplicate Analysis	CRM	NMI Uncertainty Course
10	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
11	Top Down - precision and estimates of the method and laboratory bias	Control Samples – SS	CRM Recoveries of SS	Eurachem/CITAC Guide
12	Top Down - precision and estimates of the method and laboratory bias	Control Samples – SS Duplicate Analysis	Laboratory bias from PT Recoveries of SS	NATA Technical Note 33

Table 6 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]	
13	Professional Judgment	Control Samples – RM Duplicate Analysis Instrument Calibration	CRM Laboratory bias from PT Recoveries of SS	NATA Technical Note 33
14*	Top Down - precision and estimates of the method and laboratory bias	Control Samples – RM Duplicate Analysis Instrument Calibration		NATA Technical Note 33
15	Bottom Up (ISO/GUM, fish bone/ cause and effect diagram)	Control Samples Duplicate Analysis Instrument Calibration	CRM Recoveries of SS Instrument Calibration	ISO/GUM

[a] RM = Reference Material, CRM = Certified Reference Material, SS = Spiked Samples.

* Additional comments made in Table 5

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

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

No comments were received for this study.

5 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

5.1 Results Summary

Participants' results are listed in Tables 7 to 60 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.

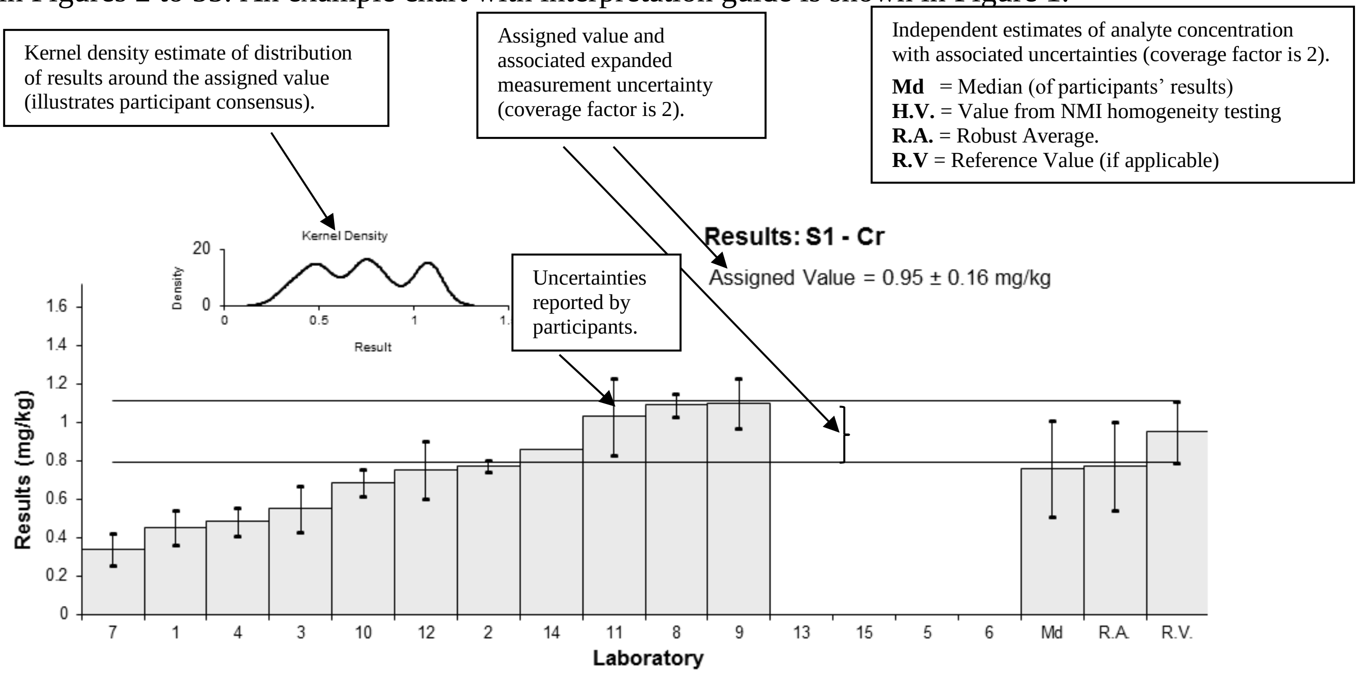


Figure 1 Guide to Presentation of Results

5.2 Assigned Value

An example of the assigned value calculation using data from the present study is given in Appendix 2. The assigned value is defined as: 'the value attributed to a particular property of a proficiency test item.'[2] In this study the property is the mass fraction of analyte. Assigned values were the robust average of participants' results; the expanded uncertainties were estimated from the associated robust standard deviations. For As, Cr, Cu, Fe, Mn, Ni and Se, in S1 the assigned values were reference values measured using standard addition mass spectrometry. For Hg in S1 the assigned value was reference value measured using isotope dilution mass spectrometry.

5.3 Robust Average

The robust averages and associated expanded measurement uncertainties were calculated using the procedure described in 'Statistical methods for use in proficiency testing by interlaboratory comparisons, ISO13528:2015(E)'.[8]

5.4 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).[8]

5.5 Target Standard Deviation

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

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

This value is used for calculation of participant z-score and provides scaling for laboratory deviation from the assigned value. It is important to note that the PCV is a fixed value and is not the standard deviation of participants' results. The fixed value set for PCV is based on the existing regulation, the acceptance criteria indicated by the methods, the matrix, and the concentration level of analyte and on experience from previous studies. It is backed up by mathematical models such as Thompson Horwitz equation.[9] By setting a fixed and realistic value for PCV, the participant's performance does not depend on other participants' performance and can be compared from study to study and against achievable performance.

5.6 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 2 below:

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

Where:

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

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

- $|z| \le 2$ is satisfactory;
- $2 < |z| < 3$ is questionable;
- $|z| \ge 3$ is unsatisfactory.

5.7 E_n-Score

An example of E_n-score calculation using data from the present study is given in Appendix 2.

The E_n-score is complementary to the z-score in assessment of laboratory performance. E_n-score includes measurement uncertainty and is calculated according to Equation 3 below:

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

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 ($|E_n|$):

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

5.8 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025:2018[10] must establish and demonstrate the traceability and measurement uncertainty associated with their test results. Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.[1]

6 TABLES AND FIGURES

Table 7

Sample Details

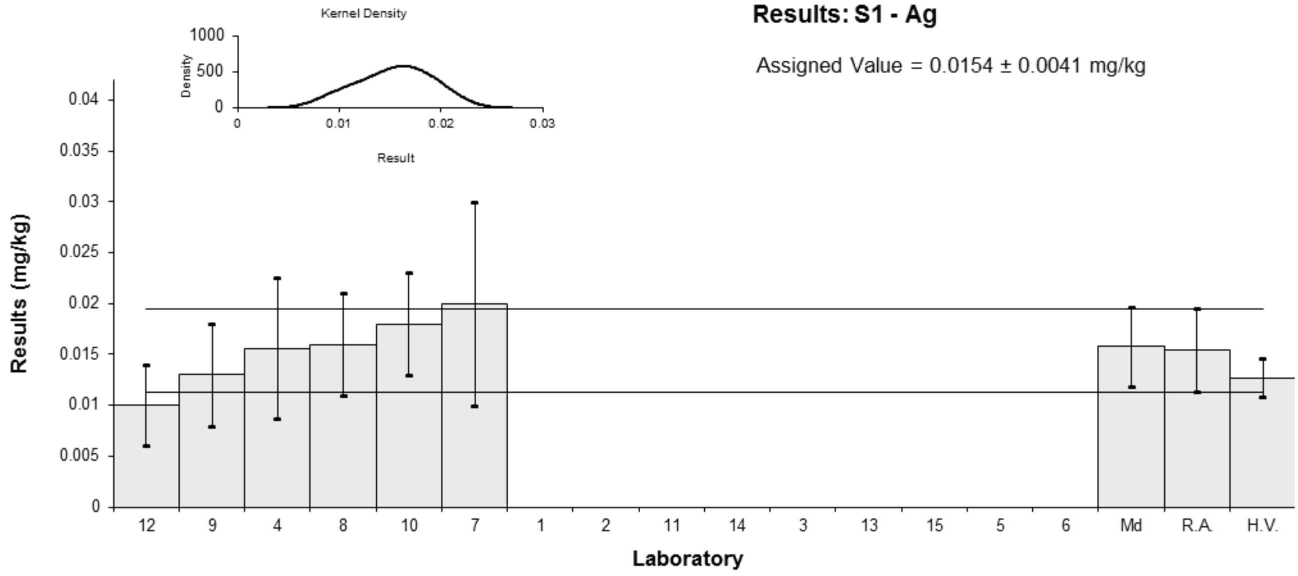
Sample No.	S1
Matrix.	Prawn
Analyte.	Ag
Units	mg/kg

Participant Results

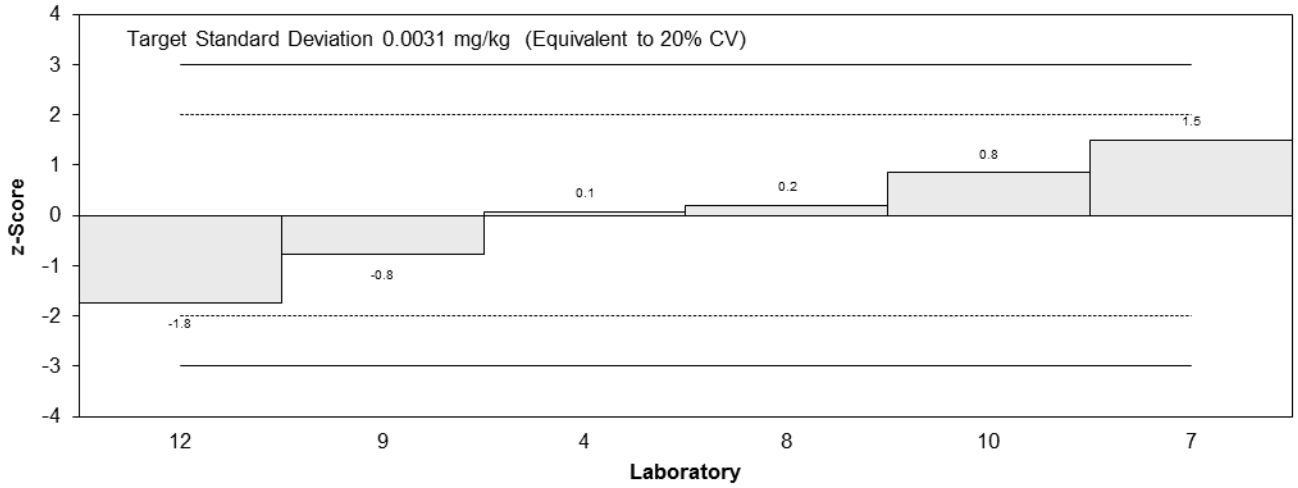
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	<0.02	NR		
2	<0.02	NR		
3	<0.2	NR		
4	0.0156	0.0069	0.06	0.02
5	NT	NT		
6	NT	NT		
7	0.02	0.01	1.49	0.43
8	0.016	0.005	0.19	0.09
9	0.013	0.005	-0.78	-0.37
10	0.018	0.005	0.84	0.40
11	<0.050	NR		
12	0.01	0.004	-1.75	-0.94
13	NT	NT		
14	<0.1	NR		
15	NT	NT		

Statistics

Assigned Value	0.0154	0.0041
Spike	Not Spiked	
Homogeneity Value	0.0127	0.0019
Robust Average	0.0154	0.0041
Median	0.0158	0.0039
Mean	0.0154	
N	6	
Max.	0.02	
Min.	0.01	
Robust SD	0.004	
Robust CV	26%	



z-Scores: S1 - Ag



En-Scores: S1 - Ag

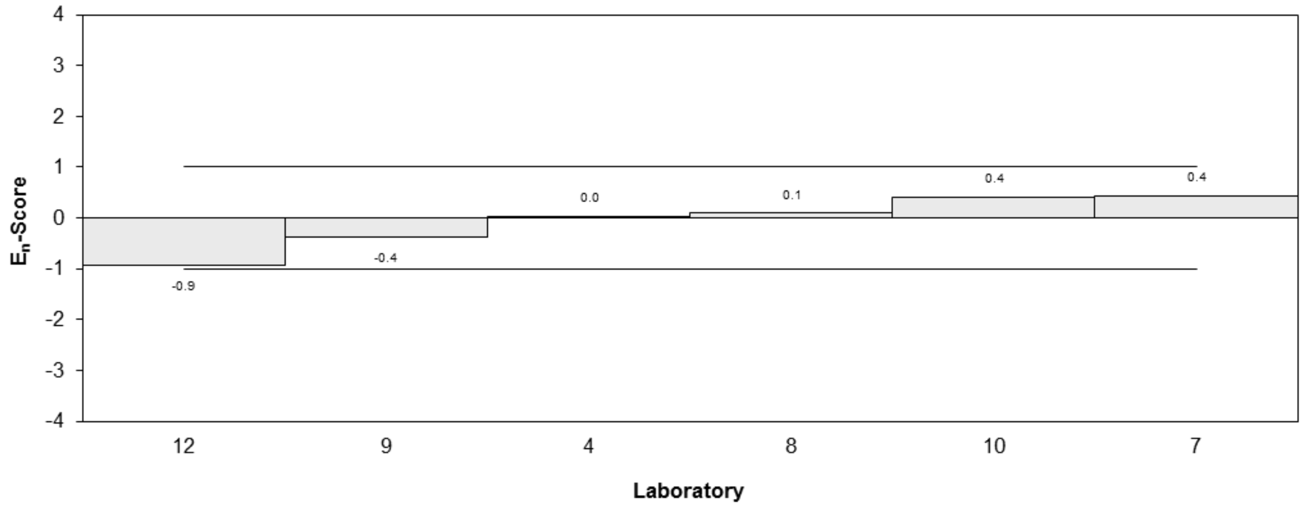


Figure 2

Table 8

Sample Details

Sample No.	S1
Matrix.	Prawn
Analyte.	Al
Units	mg/kg

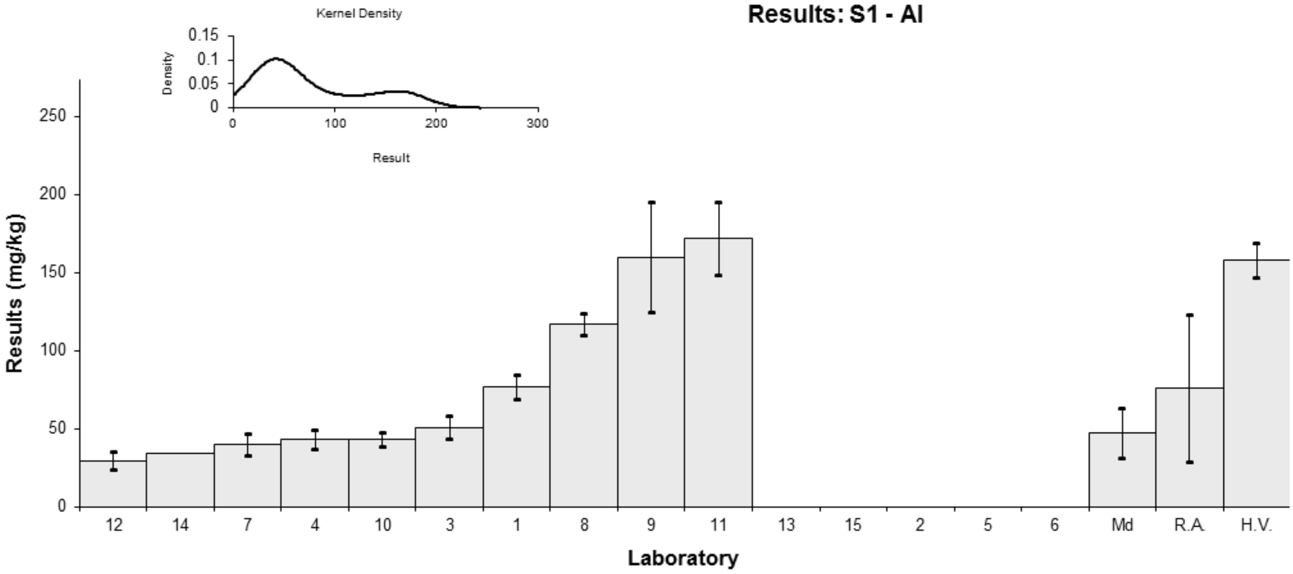
Participant Results

Lab Code	Result	Uncertainty
1	77	7.7
2	NT	NT
3	50.9	7.6
4	42.9	6.1
5	NT	NT
6	NT	NT
7	40	7
8	117	7
9	160	35
10	43.2	4.32
11	172	23.4
12	29.4	5.88
13	NT	NT
14	34.2	NR
15	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value*	158	11
Robust Average	76	47
Median	47	16
Mean	76.7	
N	10	
Max.	172	
Min.	29.4	
Robust SD	14	
Robust CV	78%	

*Information Value by SA-ICP-MS



Results: S1 - Al
Kernel Density
Density
0.15
0.1
0.05
0
0
100
200
300
Result
Results (mg/kg)
250
200
150
100
50
0
12
14
7
4
10
3
1
8
9
11
13
15
2
5
6
Md
R.A.
H.V.
Laboratory

Figure 3

Table 9

Sample Details

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

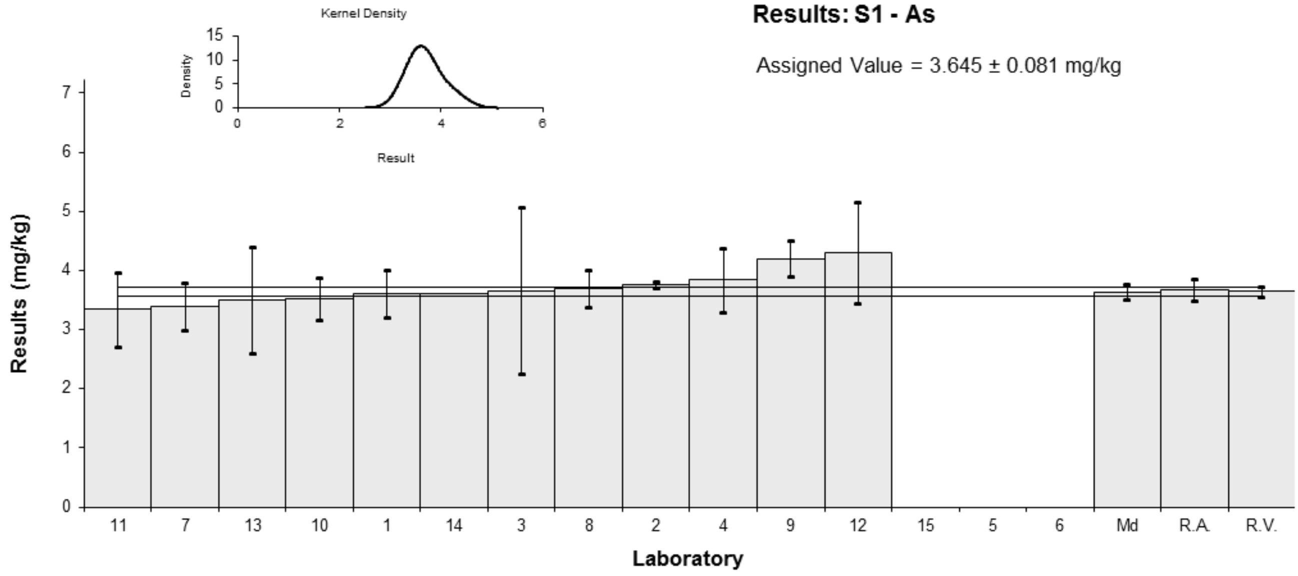
Participant Results

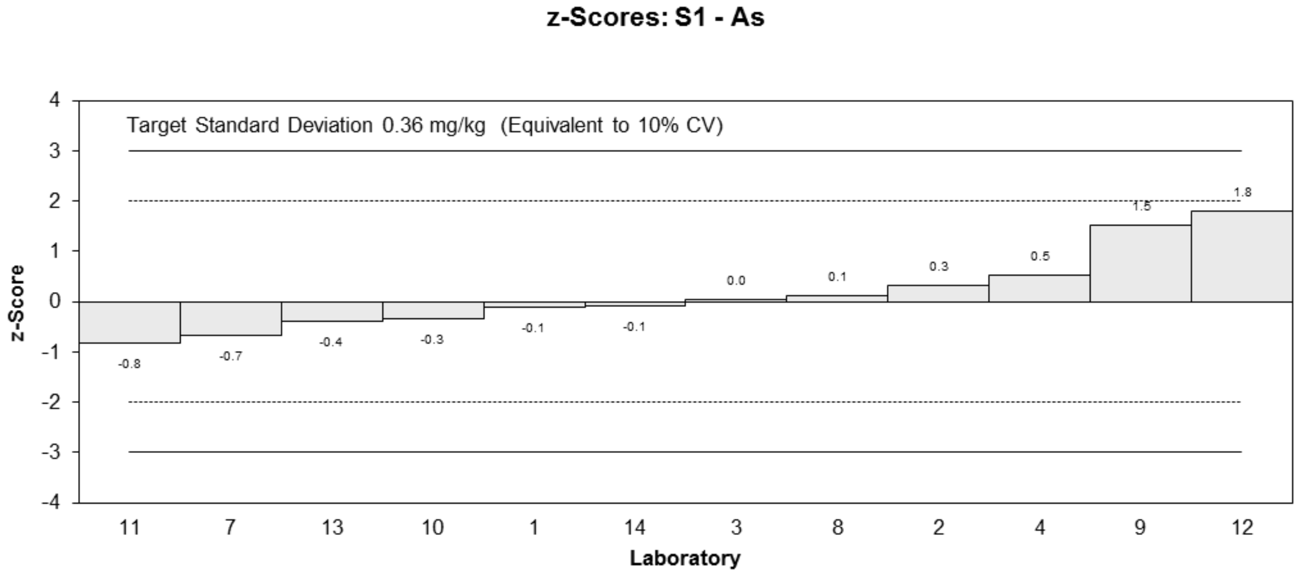
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	3.6	0.4	-0.12	-0.11
2	3.76	0.06	0.32	1.14
3	3.66	1.4	0.04	0.01
4	3.84	0.54	0.53	0.36
5	NT	NT		
6	NT	NT		
7	3.4	0.4	-0.67	-0.60
8	3.69	0.31	0.12	0.14
9	4.2	0.3	1.52	1.79
10	3.52	0.35	-0.34	-0.35
11	3.34	0.629	-0.84	-0.48
12	4.3	0.86	1.80	0.76
13	3.5	0.9	-0.40	-0.16
14	3.61	NR	-0.10	-0.43
15	NT	NT		

Statistics

Assigned Value*	3.645	0.081
Spike	Not Spiked	
Reference Value*	3.645	0.081
Robust Average	3.67	0.19
Median	3.64	0.12
Mean	3.70	
N	12	
Max.	4.3	
Min.	3.34	
Robust SD	0.26	
Robust CV	7.1%	

*Reference Value by SA-ICP-MS


Results: S1 - As
Assigned Value = 3.645 ± 0.081 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
11 7 13 10 1 14 3 8 2 4 9 12 15 5 6 Md R.A. R.V.


z-Scores: S1 - As
Target Standard Deviation 0.36 mg/kg (Equivalent to 10% CV)
z-Score
-0.8 -0.7 -0.4 -0.3 -0.1 -0.1 0.0 0.1 0.3 0.5 1.5 1.8
Laboratory
11 7 13 10 1 14 3 8 2 4 9 12

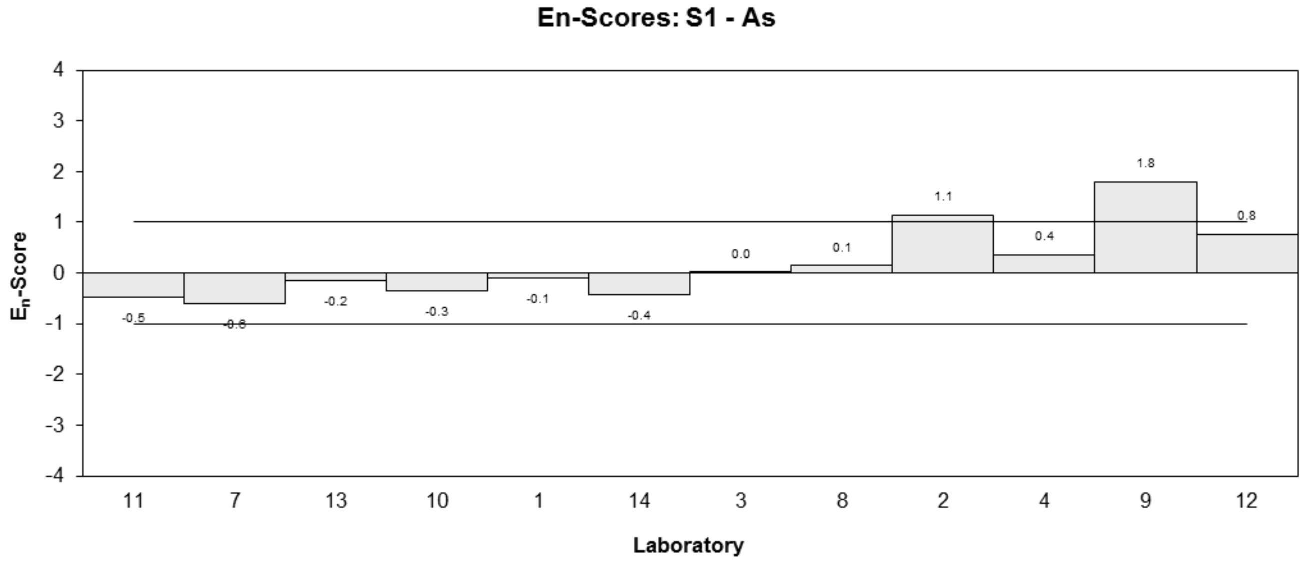

En-Scores: S1 - As
E_n-Score
-0.5 -0.6 -0.2 -0.3 -0.1 -0.4 0.0 0.1 1.1 0.4 1.8 0.8
Laboratory
11 7 13 10 1 14 3 8 2 4 9 12

Figure 4

Table 10

Sample Details

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

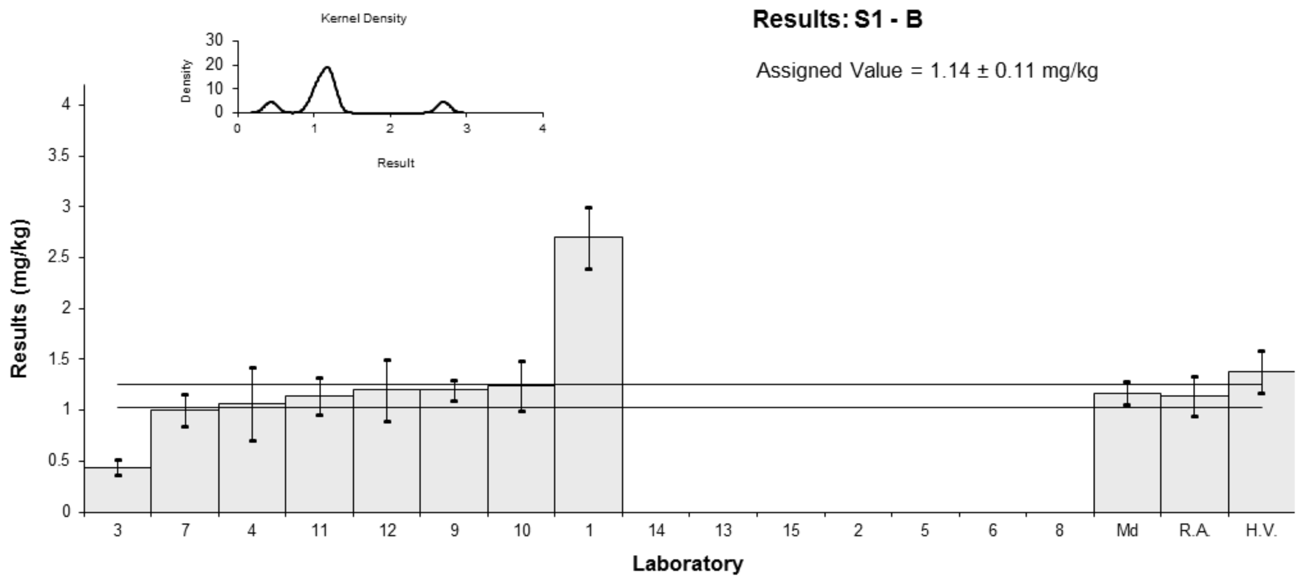
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	2.7	0.3	13.68	4.88
2	NT	NT		
3	0.44	0.08	-6.14	-5.15
4	1.06	0.36	-0.70	-0.21
5	NT	NT		
6	NT	NT		
7	1.0	0.16	-1.23	-0.72
8	NT	NT		
9	1.2	0.1	0.53	0.40
10	1.24	0.25	0.88	0.37
11	1.14	0.188	0.00	0.00
12	1.2	0.30	0.53	0.19
13	NT	NT		
14	<5	NR		
15	NT	NT		

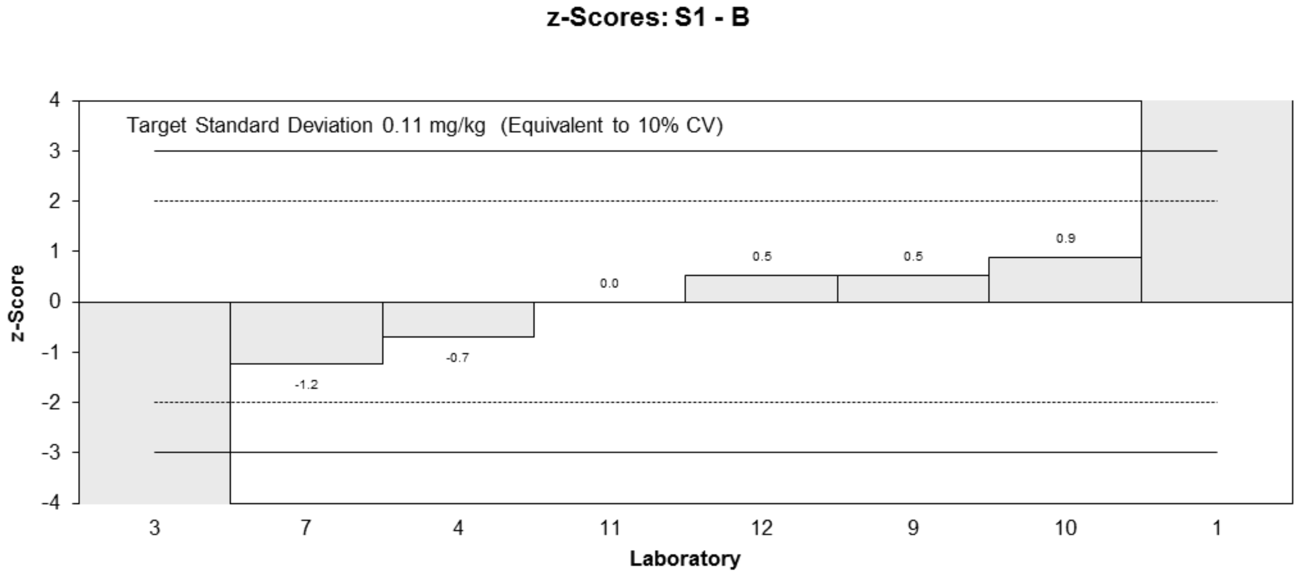
Statistics

Assigned Value*	1.14	0.11
Spike	Not Spiked	
Homogeneity Value	1.38	0.21
Robust Average	1.14	0.19
Median	1.17	0.11
Mean	1.25	
N	8	
Max.	2.7	
Min.	0.44	
Robust SD	0.11	
Robust CV	9.3%	

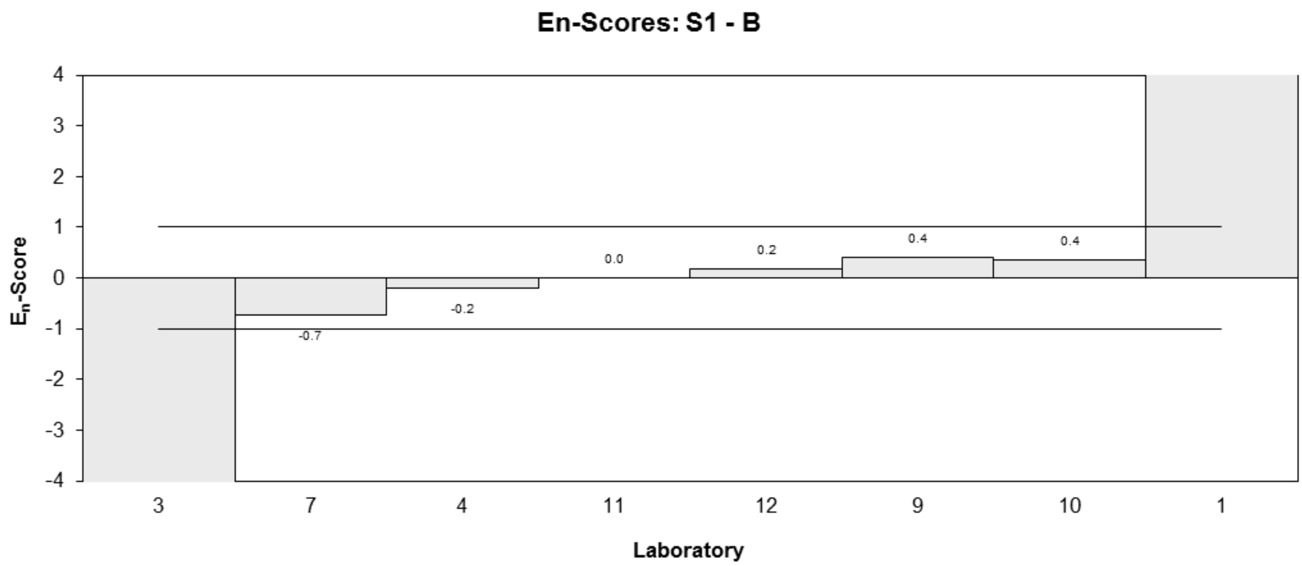
*Robust Average excluding Laboratories 1 and 3.



Results: S1 - B
Assigned Value = 1.14 ± 0.11 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
3
7
4
11
12
9
10
1
14
13
15
2
5
6
8
Md
R.A.
H.V.



z-Scores: S1 - B
Target Standard Deviation 0.11 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-1.2
-0.7
0.0
0.5
0.5
0.9



En-Scores: S1 - B
En-Score
Laboratory
-0.7
-0.2
0.0
0.2
0.4
0.4

Figure 5

Table 11

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1.1	0.15	-0.75	-0.64
2	1.46	0.02	1.18	1.36
3	1.18	0.18	-0.32	-0.25
4	1.06	0.15	-0.97	-0.82
5	NT	NT		
6	NT	NT		
7	1.1	0.2	-0.75	-0.55
8	1.48	0.01	1.29	1.50
9	1.5	0.15	1.40	1.19
10	1.33	0.13	0.48	0.44
11	1.48	0.151	1.29	1.09
12	1.02	0.20	-1.18	-0.86
13	1.1	0.3	-0.75	-0.41
14	1.1	NR	-0.75	-0.87
15	NT	NT		

Statistics

Assigned Value	1.24	0.16
Spike	Not Spiked	
Homogeneity Value	1.06	0.16
Robust Average	1.24	0.16
Median	1.14	0.09
Mean	1.24	
N	12	
Max.	1.5	
Min.	1.02	
Robust SD	0.22	
Robust CV	17%	

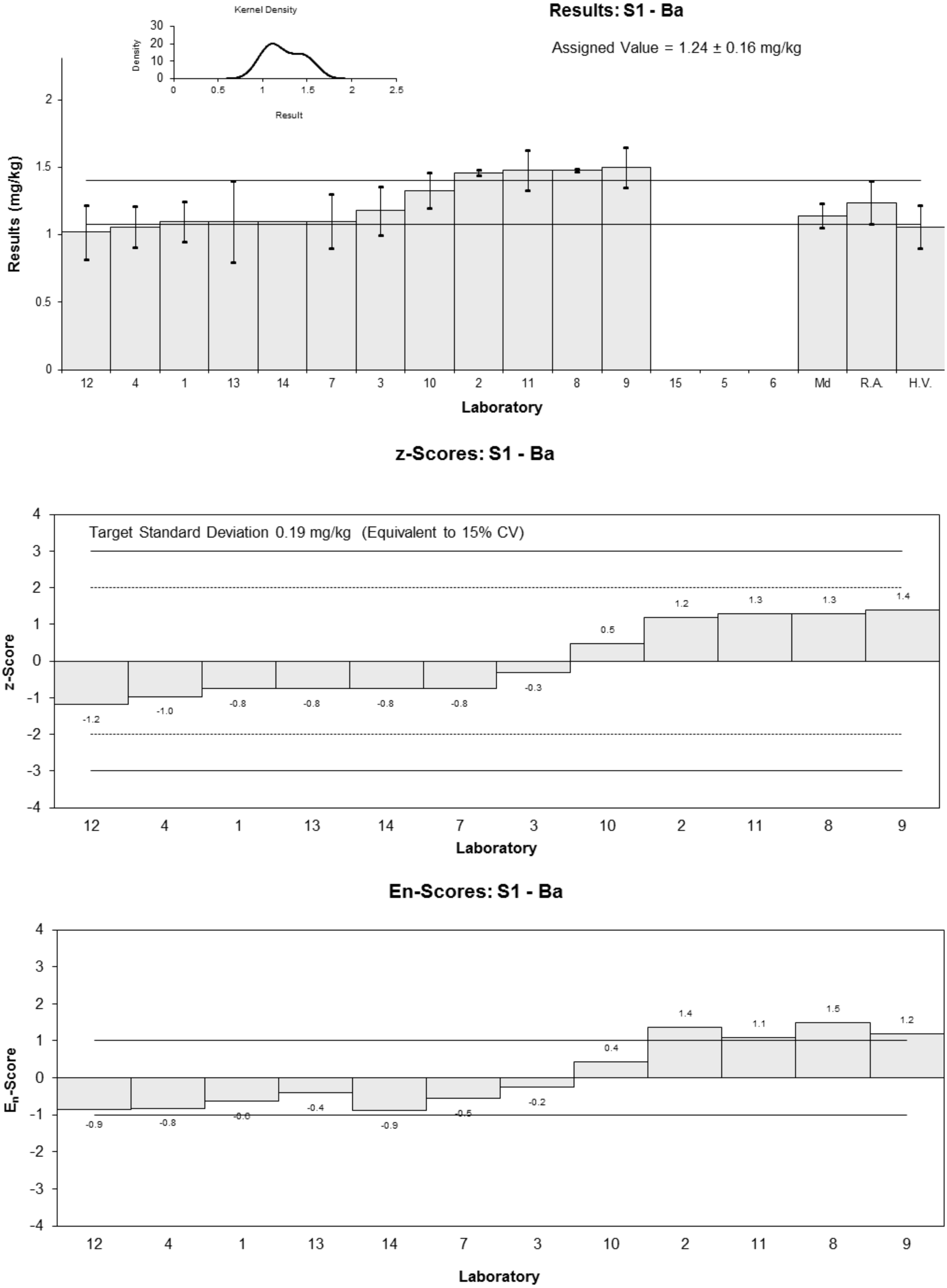

Results: S1 - Ba
Assigned Value = 1.24 ± 0.16 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
12 4 1 13 14 7 3 10 2 11 8 9 15 5 6 Md R.A. H.V.
z-Scores: S1 - Ba
Target Standard Deviation 0.19 mg/kg (Equivalent to 15% CV)
z-Score
-1.2 -1.0 -0.8 -0.8 -0.8 -0.8 -0.3 0.5 1.2 1.3 1.3 1.4
Laboratory
12 4 1 13 14 7 3 10 2 11 8 9
En-Scores: S1 - Ba
E_n-Score
-0.9 -0.8 -0.6 -0.4 -0.9 -0.5 -0.2 0.4 1.4 1.1 1.5 1.2
Laboratory
12 4 1 13 14 7 3 10 2 11 8 9

Figure 6

Table 12

Sample Details

Sample No.	S1
Matrix.	Prawn
Analyte.	Bi
Units	mg/kg

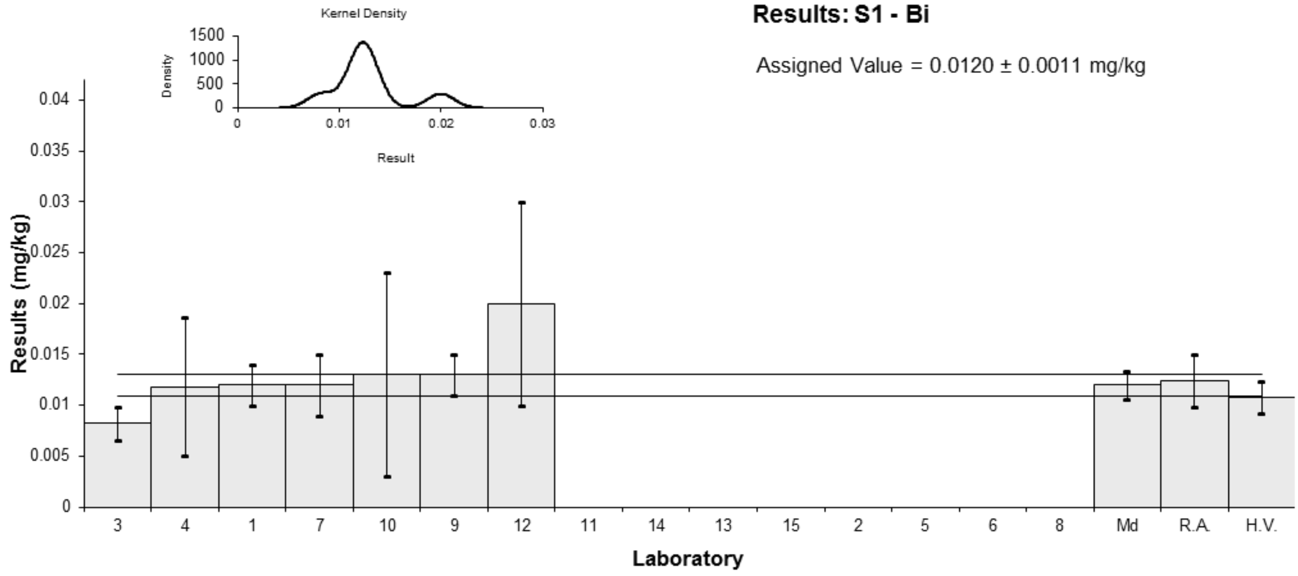
Participant Results

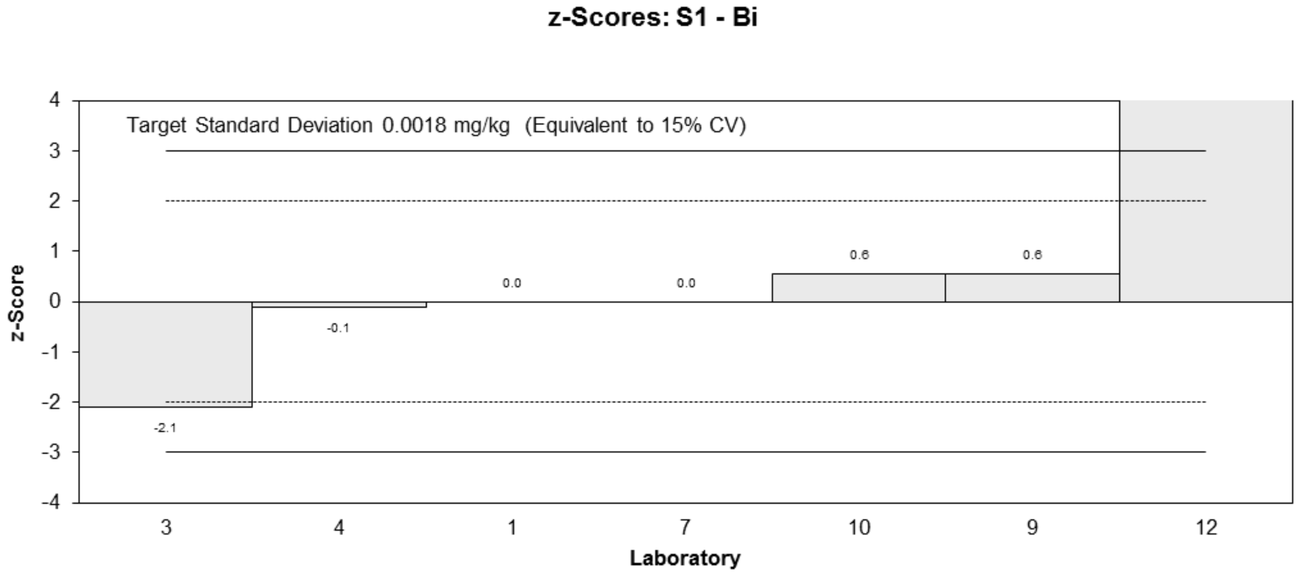
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.012	0.002	0.00	0.00
2	NT	NT		
3	0.0082	0.0016	-2.11	-1.96
4	0.0118	0.0068	-0.11	-0.03
5	NT	NT		
6	NT	NT		
7	0.012	0.003	0.00	0.00
8	NT	NT		
9	0.013	0.002	0.56	0.44
10	0.013	0.01	0.56	0.10
11	<0.050	NR		
12	0.02	0.01	4.44	0.80
13	NT	NT		
14	<0.1	NR		
15	NT	NT		

Statistics

Assigned Value*	0.0120	0.0011
Spike	Not Spiked	
Homogeneity Value	0.0108	0.0016
Robust Average	0.0124	0.0026
Median	0.0120	0.0014
Mean	0.0129	
N	7	
Max.	0.02	
Min.	0.0082	
Robust SD	0.0011	
Robust CV	8.9%	

*Robust Average excluding Laboratory 12.


Results: S1 - Bi
Assigned Value = 0.0120 ± 0.0011 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
3
4
1
7
10
9
12
11
14
13
15
2
5
6
8
Md
R.A.
H.V.


z-Scores: S1 - Bi
Target Standard Deviation 0.0018 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
-2.1
-0.1
0.0
0.0
0.6
0.6

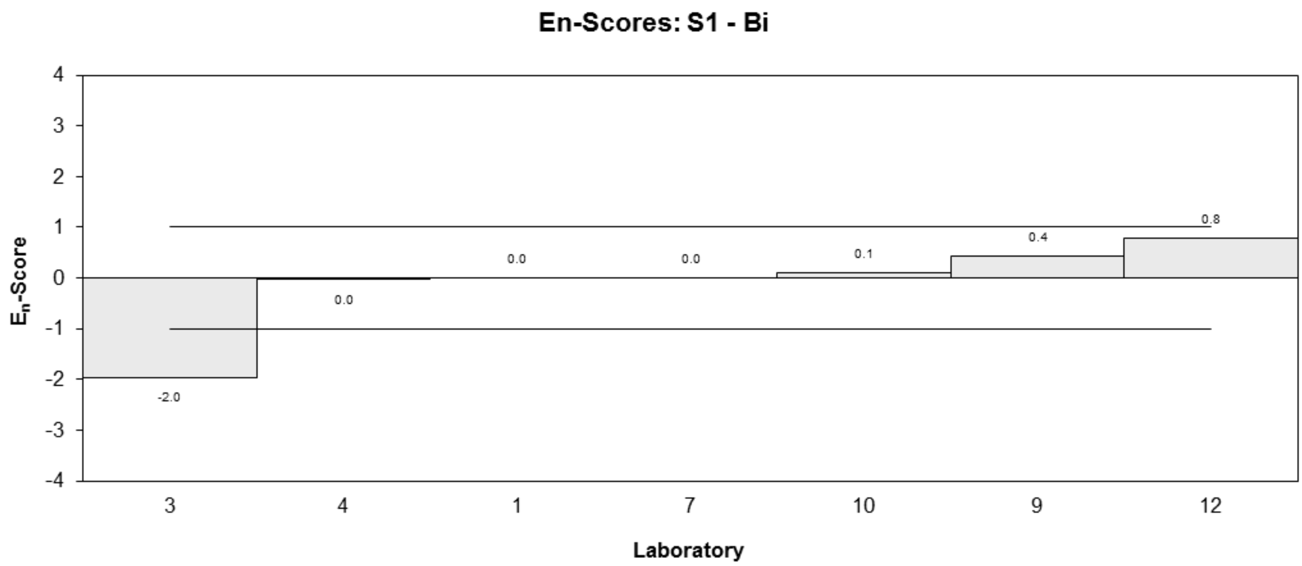

En-Scores: S1 - Bi
En-Score
Laboratory
-2.0
0.0
0.0
0.0
0.1
0.4
0.8

Figure 7

Table 13

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	5200	160	-0.21	-0.52
2	5312	270	0.00	0.01
3	5100	770	-0.40	-0.27
4	5400	550	0.17	0.16
5	NT	NT		
6	NT	NT		
7	5300	1256	-0.02	-0.01
8	5200	300	-0.21	-0.33
9	5460	340	0.28	0.41
10	5520	552	0.40	0.37
11	5380	942	0.13	0.07
12	5000	750	-0.58	-0.41
13	NT	NT		
14	5480	NR	0.32	1.21
15	NT	NT		

Statistics

Assigned Value	5310	140
Spike	Not Spiked	
Homogeneity Value	4770	720
Robust Average	5310	140
Median	5310	110
Mean	5300	
N	11	
Max.	5520	
Min.	5000	
Robust SD	180	
Robust CV	3.4%	

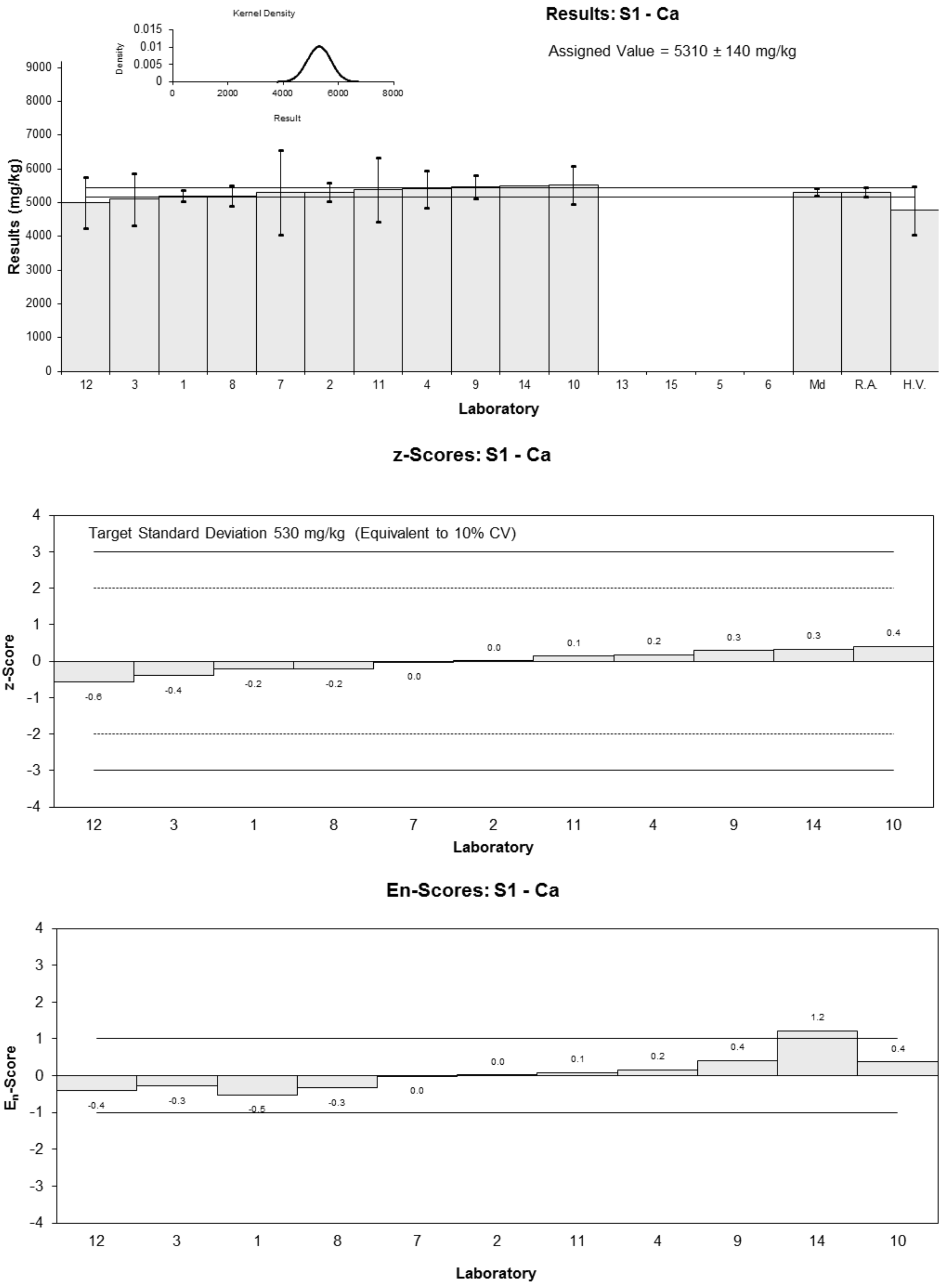

Results: S1 - Ca
Assigned Value = 5310 ± 140 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
12 3 1 8 7 2 11 4 9 14 10 13 15 5 6 Md R.A. H.V.
z-Scores: S1 - Ca
Target Standard Deviation 530 mg/kg (Equivalent to 10% CV)
z-Score
-0.6 -0.4 -0.2 -0.2 0.0 0.0 0.1 0.2 0.3 0.3 0.4
12 3 1 8 7 2 11 4 9 14 10
Laboratory
En-Scores: S1 - Ca
E_n-Score
-0.4 -0.3 -0.5 -0.3 0.0 0.0 0.1 0.2 0.4 1.2 0.4
12 3 1 8 7 2 11 4 9 14 10
Laboratory

Figure 8

Table 14

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.14	0.03	0.63	0.37
2	0.12	0.01	-0.42	-0.51
3	0.111	0.017	-0.89	-0.82
4	0.115	0.017	-0.68	-0.62
5	NT	NT		
6	NT	NT		
7	0.126	0.020	-0.10	-0.09
8	0.133	0.03	0.26	0.15
9	0.11	0.03	-0.94	-0.56
10	0.212	0.021	4.38	3.47
11	0.14	0.0277	0.63	0.40
12	0.17	0.03	2.19	1.30
13	<1	NR		
14	0.13	NR	0.10	0.17
15	NT	NT		

Statistics

Assigned Value*	0.128	0.012
Spike	Not Spiked	
Homogeneity Value	0.127	0.019
Robust Average	0.132	0.016
Median	0.130	0.010
Mean	0.137	
N	11	
Max.	0.212	
Min.	0.11	
Robust SD	0.016	
Robust CV	12%	

*Robust Average excluding Laboratory 10.

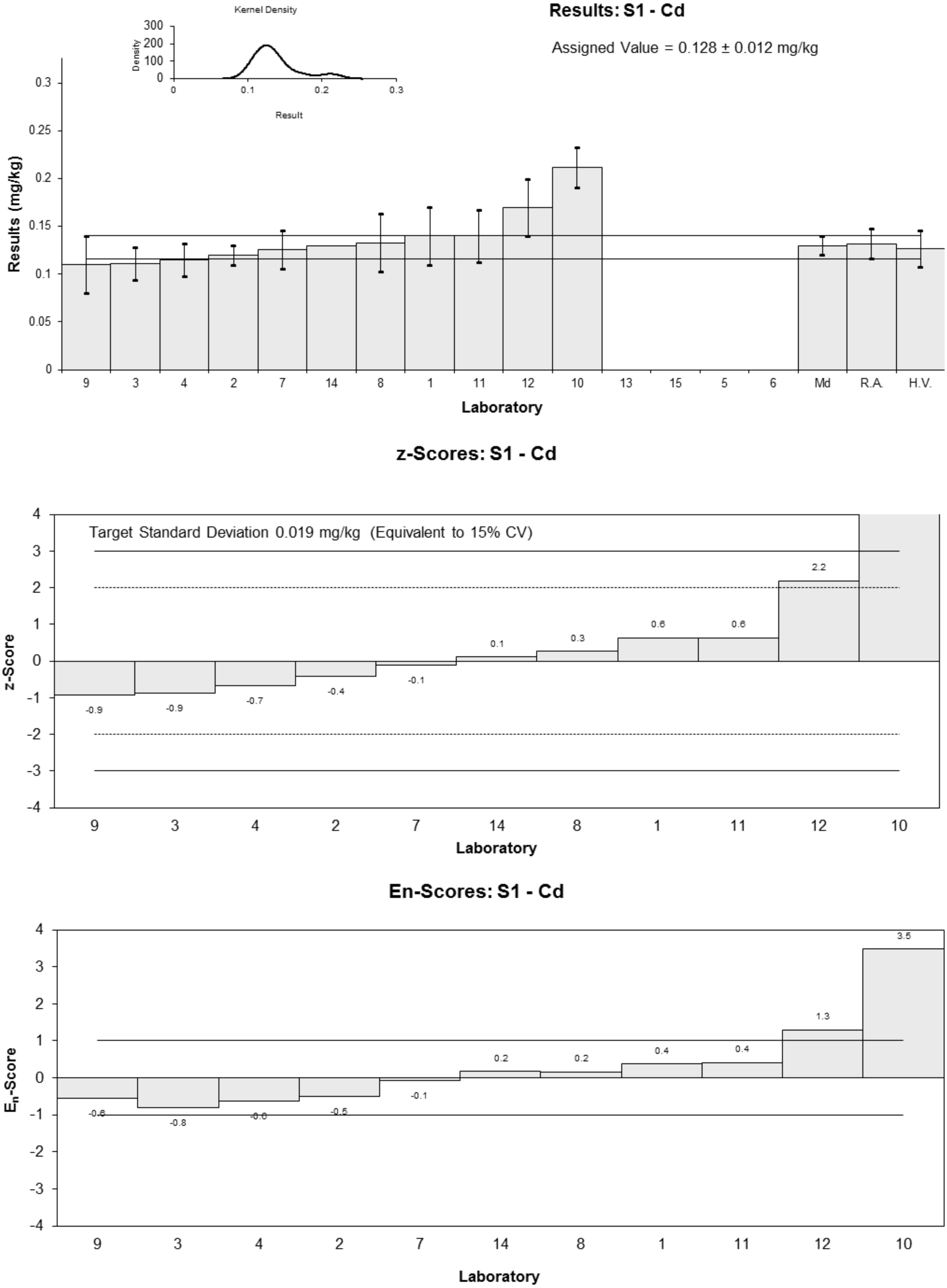

Kernel Density
Density
Result
Results: S1 - Cd
Assigned Value = 0.128 ± 0.012 mg/kg
Results (mg/kg)
Laboratory
9 3 4 2 7 14 8 1 11 12 10 13 15 5 6 Md R.A. H.V.
z-Scores: S1 - Cd
Target Standard Deviation 0.019 mg/kg (Equivalent to 15% CV)
z-Score
-0.9 -0.9 -0.7 -0.4 -0.1 0.1 0.3 0.6 0.6 2.2
Laboratory
En-Scores: S1 - Cd
E_n-Score
-0.6 -0.8 -0.6 -0.5 -0.1 0.2 0.2 0.4 0.4 1.3 3.5
Laboratory

Figure 9

Table 15

Sample Details

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

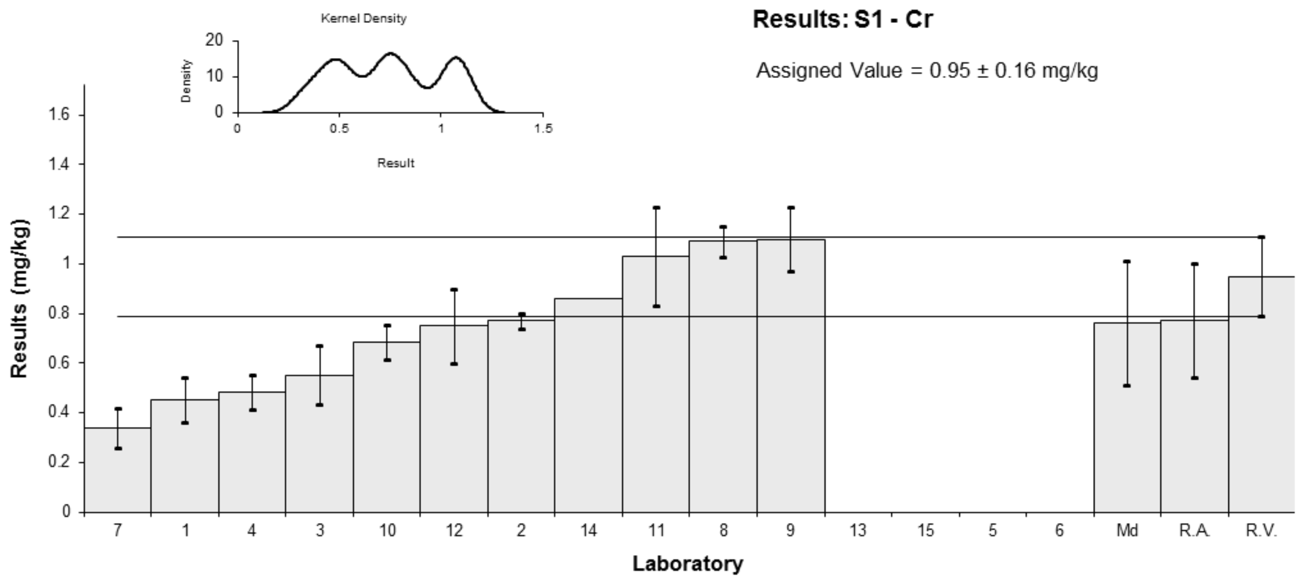
Participant Results

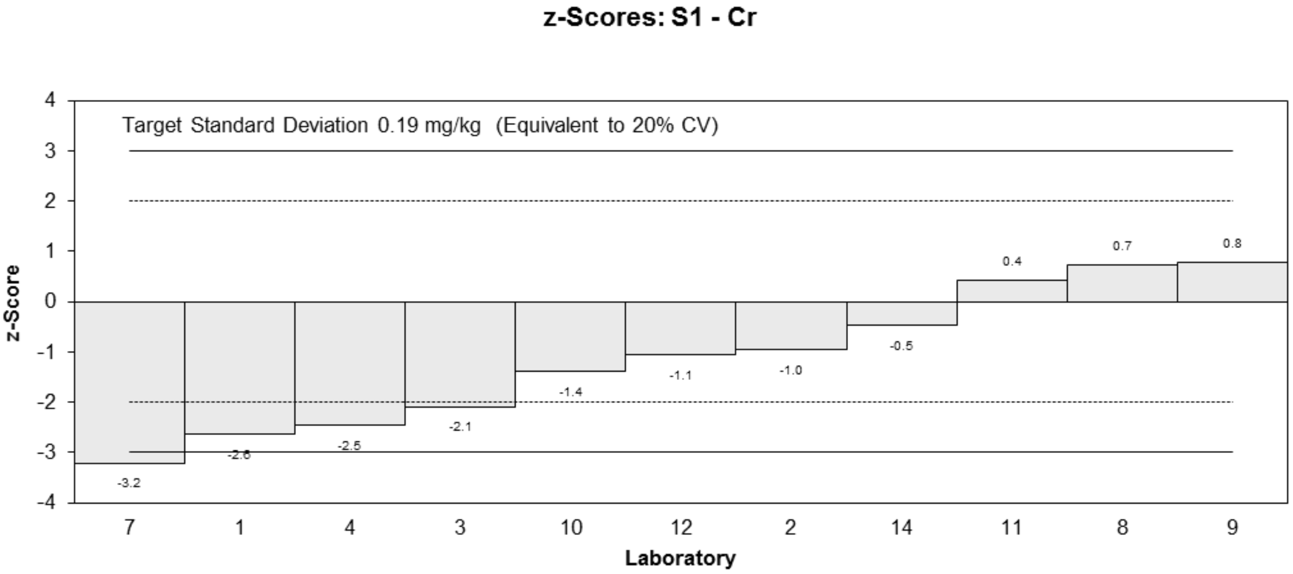
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.45	0.09	-2.63	-2.72
2	0.77	0.03	-0.95	-1.11
3	0.552	0.12	-2.09	-1.99
4	0.484	0.071	-2.45	-2.66
5	NT	NT		
6	NT	NT		
7	0.34	0.08	-3.21	-3.41
8	1.09	0.06	0.74	0.82
9	1.1	0.13	0.79	0.73
10	0.685	0.069	-1.39	-1.52
11	1.03	0.199	0.42	0.31
12	0.75	0.15	-1.05	-0.91
13	<1	NR		
14	0.86	NR	-0.47	-0.56
15	NT	NT		

Statistics

Assigned Value*	0.95	0.16
Spike	Not Spiked	
Reference Value*	0.95	0.16
Robust Average	0.77	0.23
Median	0.76	0.25
Mean	0.74	
N	11	
Max.	1.1	
Min.	0.34	
Robust SD	0.26	
Robust CV	41%	

*Reference Value by SA-ICP-MS


Results: S1 - Cr
Assigned Value = 0.95 ± 0.16 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
7
1
4
3
10
12
2
14
11
8
9
13
15
5
6
Md
R.A.
R.V.
Laboratory


z-Scores: S1 - Cr
Target Standard Deviation 0.19 mg/kg (Equivalent to 20% CV)
z-Score
-3.2
-2.6
-2.5
-2.1
-1.4
-1.1
-1.0
-0.5
0.4
0.7
0.8
Laboratory

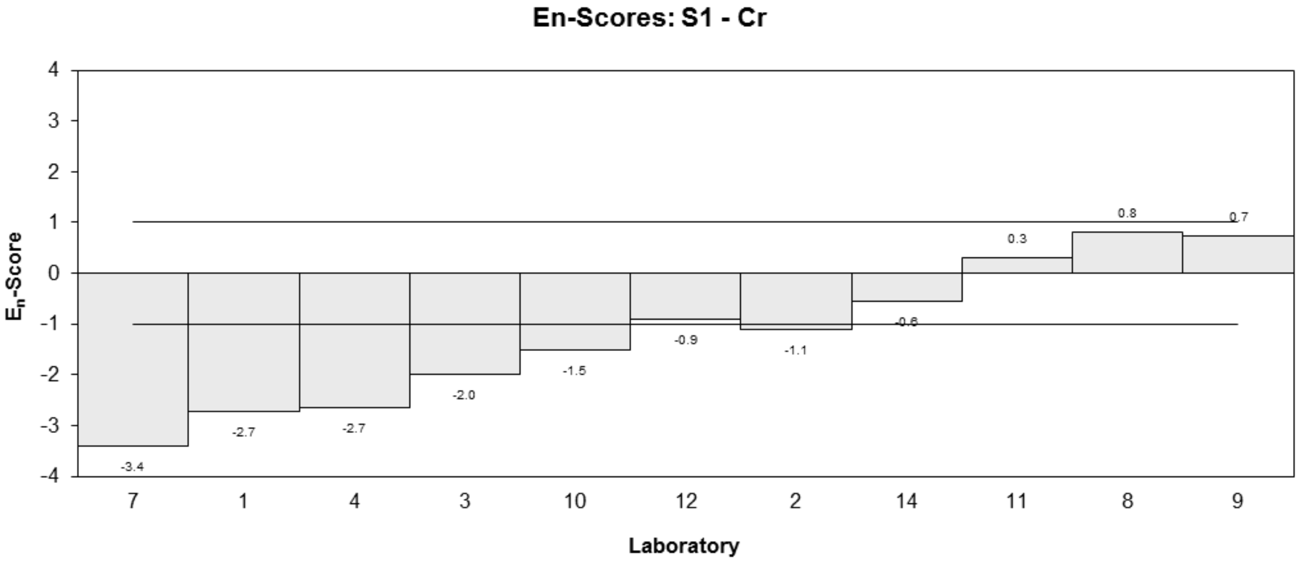

En-Scores: S1 - Cr
En-Score
-3.4
-2.7
-2.7
-2.0
-1.5
-0.9
-1.1
-0.6
0.3
0.8
0.7
Laboratory

Figure 10

Table 16

Sample Details

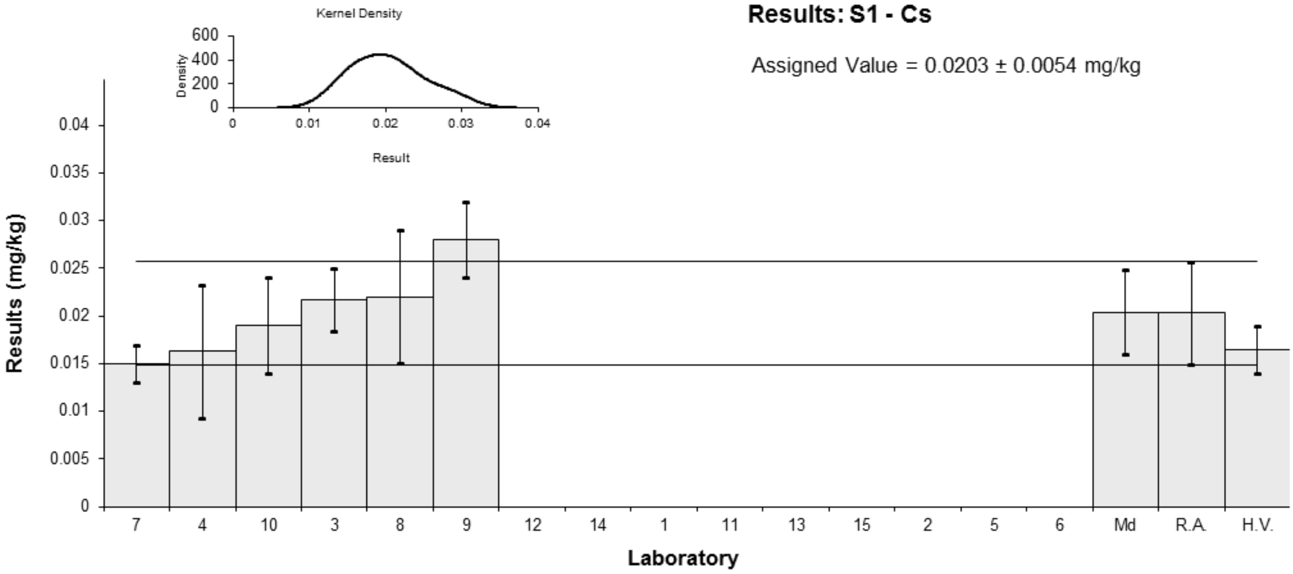
Sample No.	S1
Matrix.	Prawn
Analyte.	Cs
Units	mg/kg

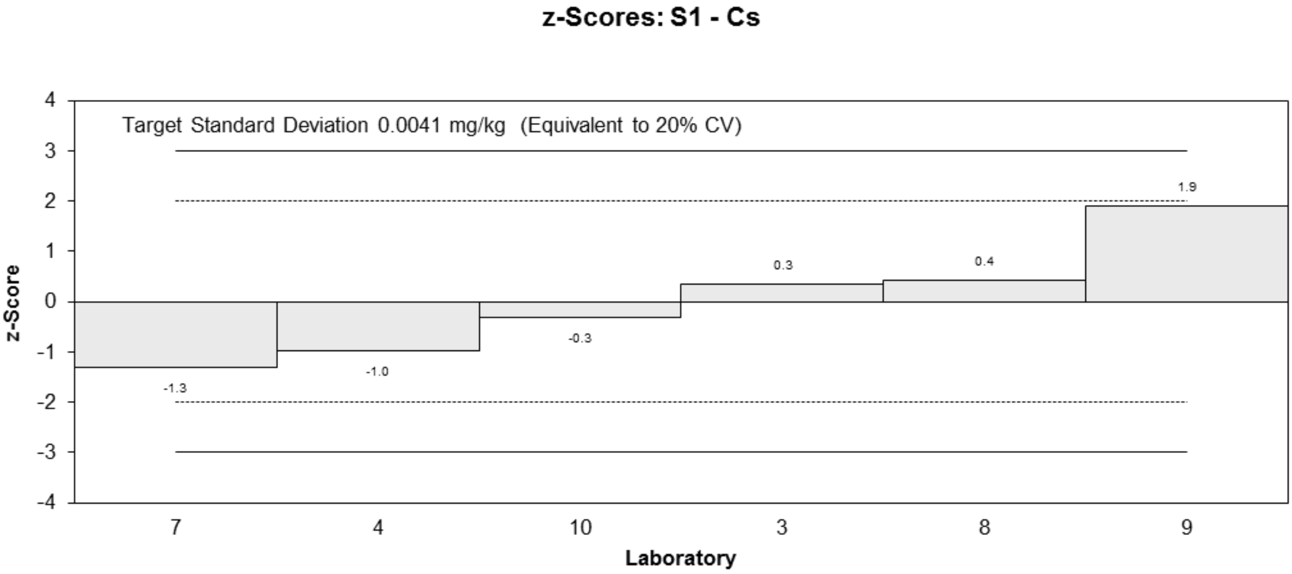
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	NT	NT		
3	0.0217	0.0033	0.34	0.22
4	0.0163	0.0070	-0.99	-0.45
5	NT	NT		
6	NT	NT		
7	0.015	0.002	-1.31	-0.92
8	0.022	0.007	0.42	0.19
9	0.028	0.004	1.90	1.15
10	0.019	0.005	-0.32	-0.18
11	NT	NT		
12	<0.05	0.02		
13	NT	NT		
14	<0.1	NR		
15	NT	NT		

Statistics

Assigned Value	0.0203	0.0054
Spike	Not Spiked	
Homogeneity Value	0.0165	0.0025
Robust Average	0.0203	0.0054
Median	0.0204	0.0044
Mean	0.0203	
N	6	
Max.	0.028	
Min.	0.015	
Robust SD	0.0053	
Robust CV	26%	


Results: S1 - Cs
Assigned Value = 0.0203 ± 0.0054 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
7
4
10
3
8
9
12
14
1
11
13
15
2
5
6
Md
R.A.
H.V.


z-Scores: S1 - Cs
Target Standard Deviation 0.0041 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
-1.3
-1.0
-0.3
0.3
0.4
1.9
7
4
10
3
8
9

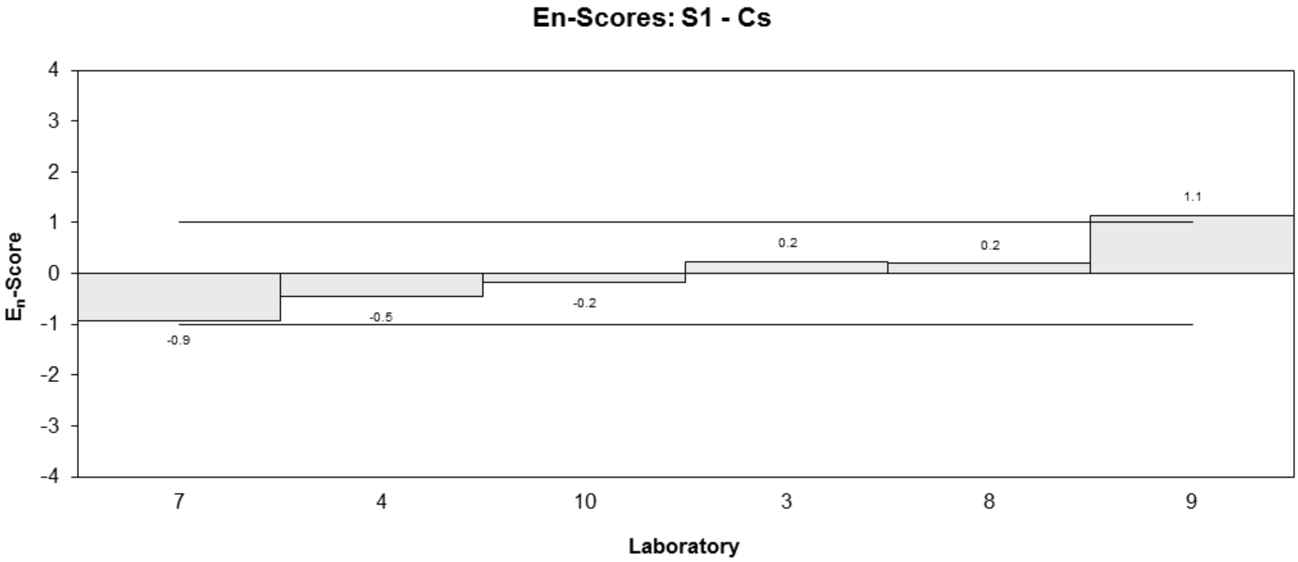

En-Scores: S1 - Cs
En-Score
Laboratory
-0.9
-0.5
-0.2
0.2
0.2
1.1
7
4
10
3
8
9

Figure 11

Table 17

Sample Details

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

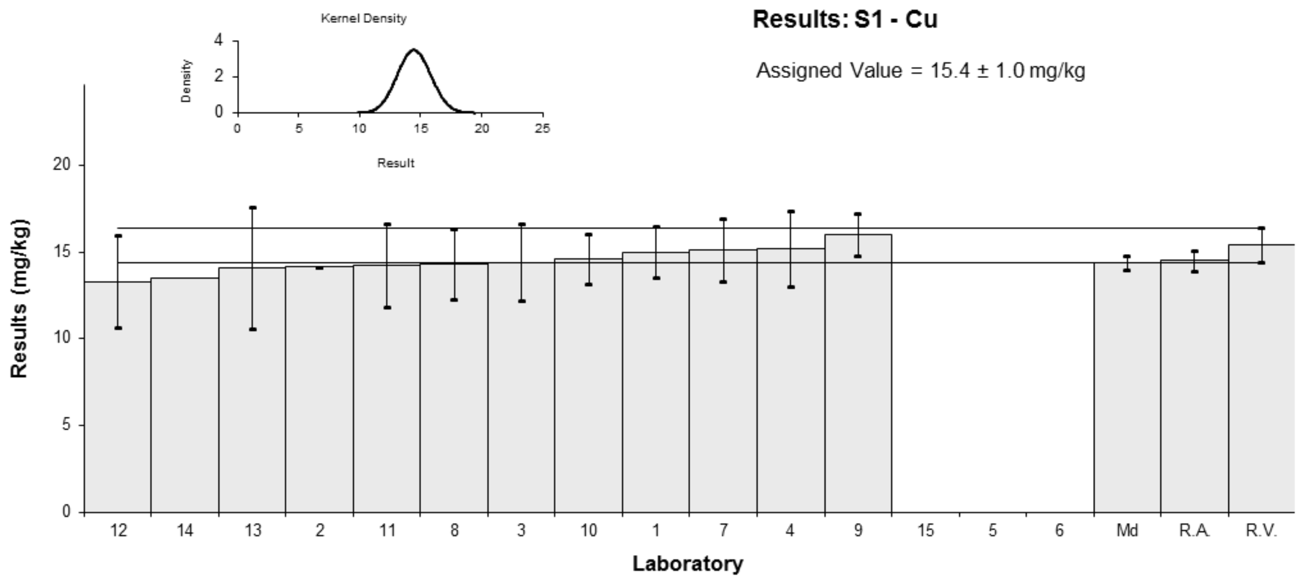
Participant Results

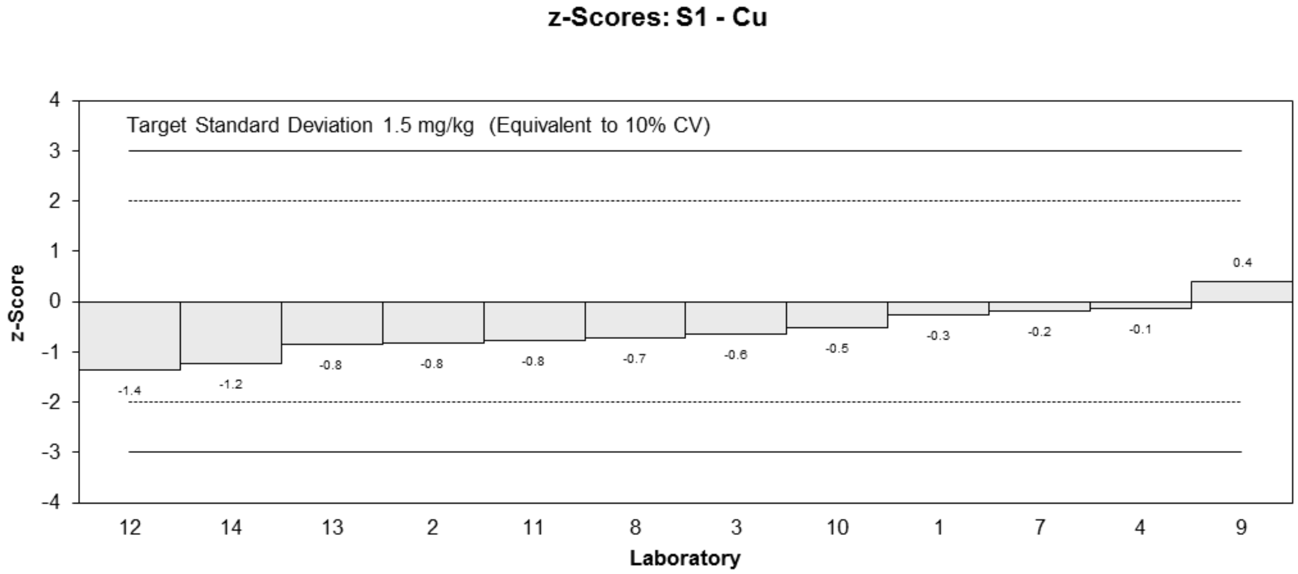
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	15	1.5	-0.26	-0.22
2	14.12	0.03	-0.83	-1.28
3	14.4	2.2	-0.65	-0.41
4	15.2	2.2	-0.13	-0.08
5	NT	NT		
6	NT	NT		
7	15.1	1.8	-0.19	-0.15
8	14.3	2.0	-0.71	-0.49
9	16	1.2	0.39	0.38
10	14.6	1.46	-0.52	-0.45
11	14.2	2.40	-0.78	-0.46
12	13.3	2.66	-1.36	-0.74
13	14.1	3.5	-0.84	-0.36
14	13.5	NR	-1.23	-1.90
15	NT	NT		

Statistics

Assigned Value*	15.4	1.0
Spike	Not Spiked	
Reference Value*	15.4	1.0
Robust Average	14.5	0.6
Median	14.4	0.4
Mean	14.5	
N	12	
Max.	16	
Min.	13.3	
Robust SD	0.8	
Robust CV	5.5%	

*Reference Value by SA-ICP-MS


Results: S1 - Cu
Assigned Value = 15.4 ± 1.0 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
12
14
13
2
11
8
3
10
1
7
4
9
15
5
6
Md
R.A.
R.V.


z-Scores: S1 - Cu
Target Standard Deviation 1.5 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-1.4
-1.2
-0.8
-0.8
-0.8
-0.7
-0.6
-0.5
-0.3
-0.2
-0.1
0.4

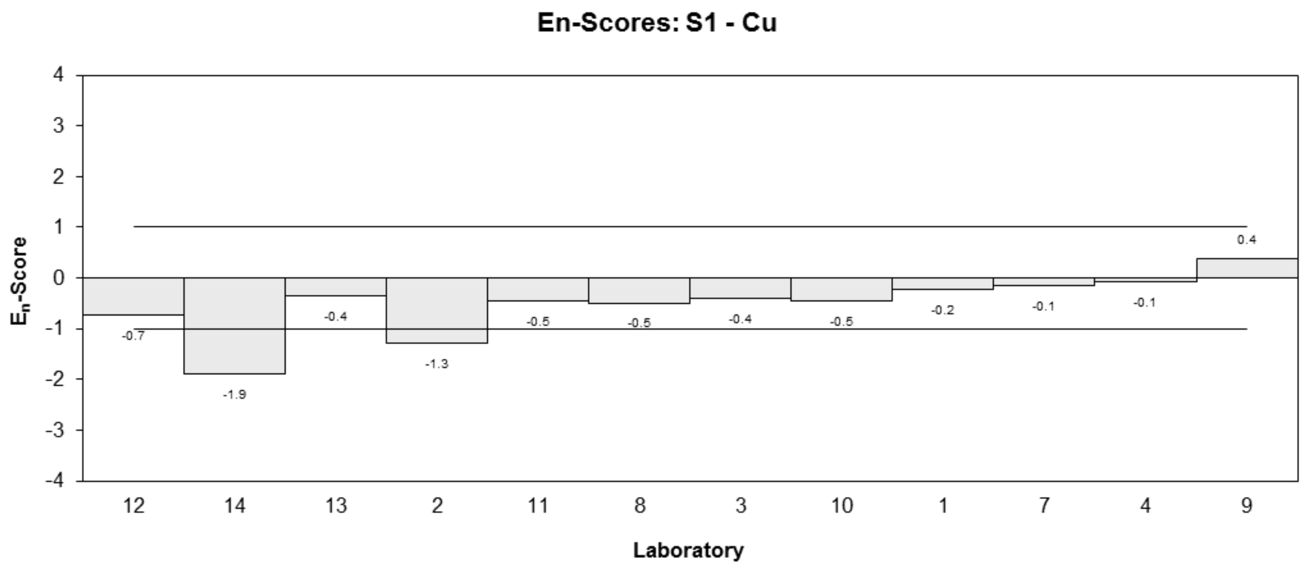

En-Scores: S1 - Cu
En-Score
Laboratory
-0.7
-1.9
-0.4
-1.3
-0.5
-0.5
-0.4
-0.5
-0.2
-0.1
-0.1
0.4

Figure 12

Table 18

Sample Details

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

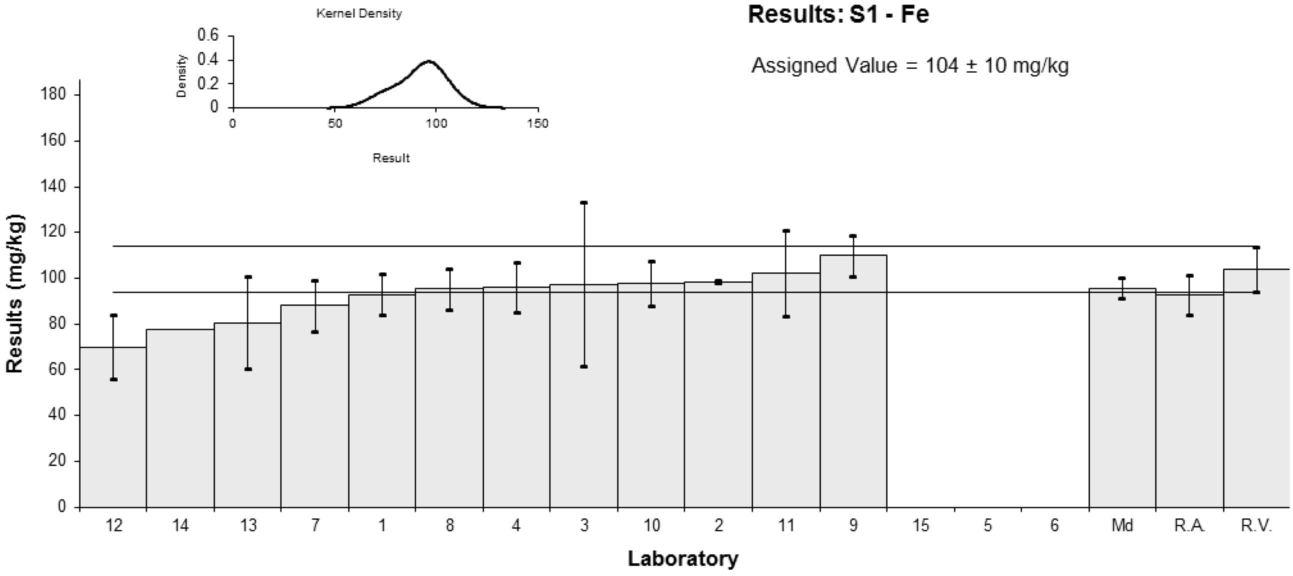
Participant Results

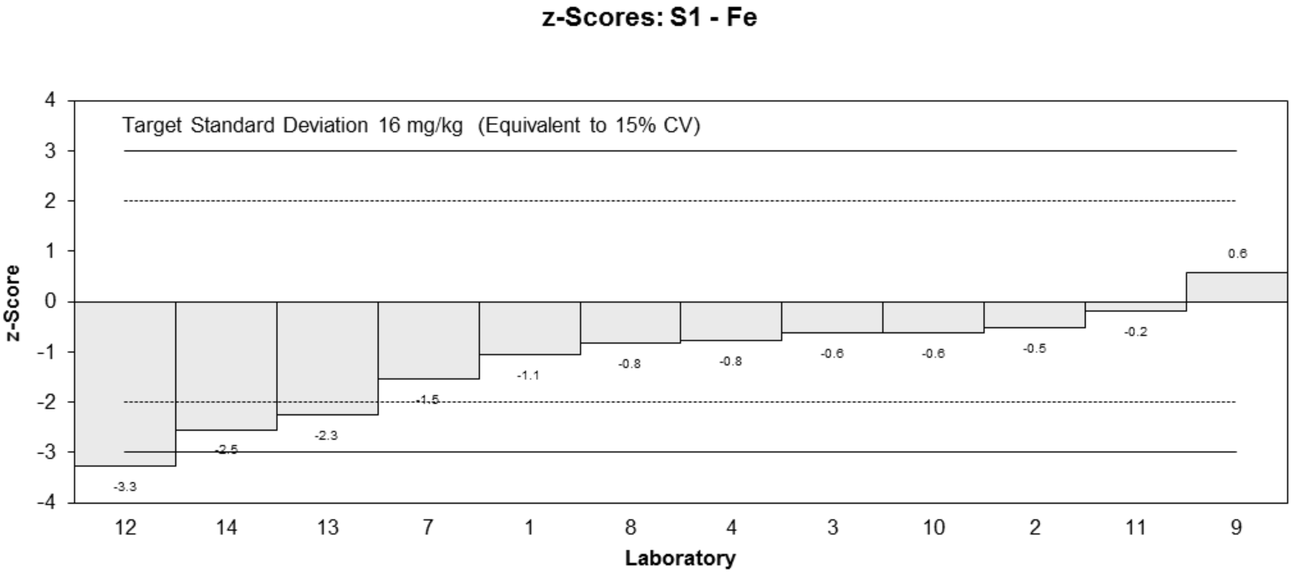
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	93	9.0	-0.71	-0.82
2	98.58	0.79	-0.35	-0.54
3	97.4	36	-0.42	-0.18
4	96	11	-0.51	-0.54
5	NT	NT		
6	NT	NT		
7	88	11	-1.03	-1.08
8	95.3	9.1	-0.56	-0.64
9	110	8.9	0.38	0.45
10	97.6	9.76	-0.41	-0.46
11	102	18.8	-0.13	-0.09
12	70	14	-2.18	-1.98
13	80.6	20.2	-1.5	-1.04
14	77.5	NR	-1.7	-2.65
15	NT	NT		

Statistics

Assigned Value*	104	10
Spike	Not Spiked	
Reference Value*	104	10
Robust Average	92.6	8.6
Median	95.7	4.4
Mean	92.2	
N	12	
Max.	110	
Min.	70	
Robust SD	12.0	
Robust CV	13%	

*Reference Value by SA-ICP-MS


Results: S1 - Fe
Assigned Value = 104 ± 10 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
12
14
13
7
1
8
4
3
10
2
11
9
15
5
6
Md
R.A.
R.V.


z-Scores: S1 - Fe
Target Standard Deviation 16 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
-3.3
-2.5
-2.3
-1.5
-1.1
-0.8
-0.8
-0.6
-0.6
-0.5
-0.2
0.6

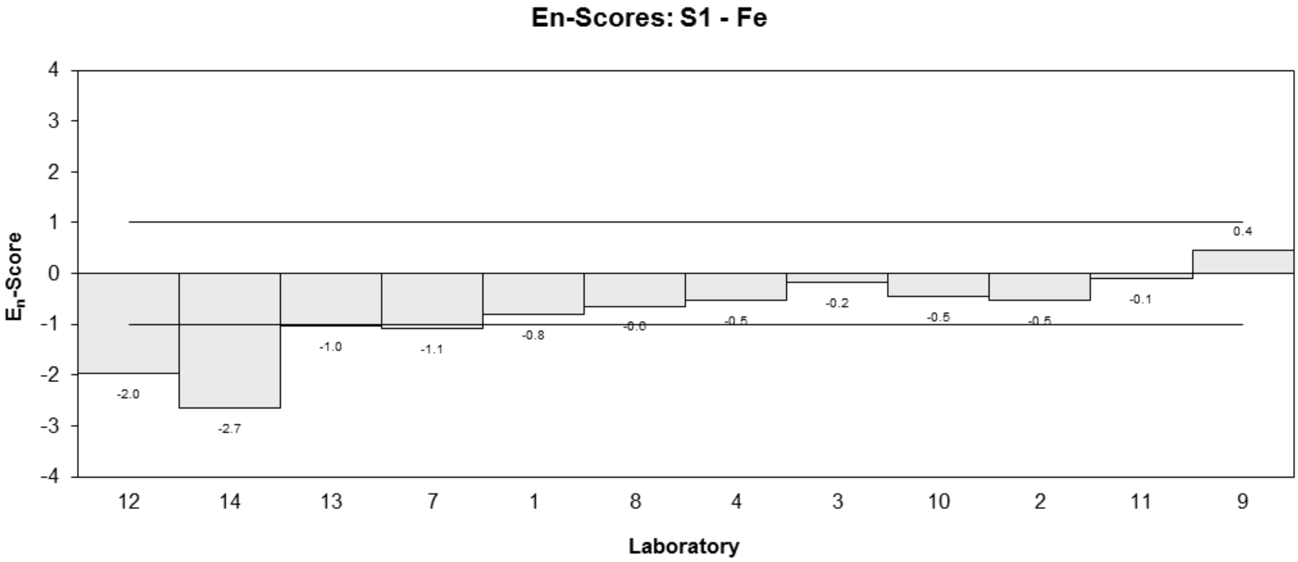

En-Scores: S1 - Fe
E_n-Score
Laboratory
-2.0
-2.7
-1.0
-1.1
-0.8
-0.5
-0.2
-0.5
-0.5
-0.1
0.4

Figure 13

Table 19

Sample Details

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

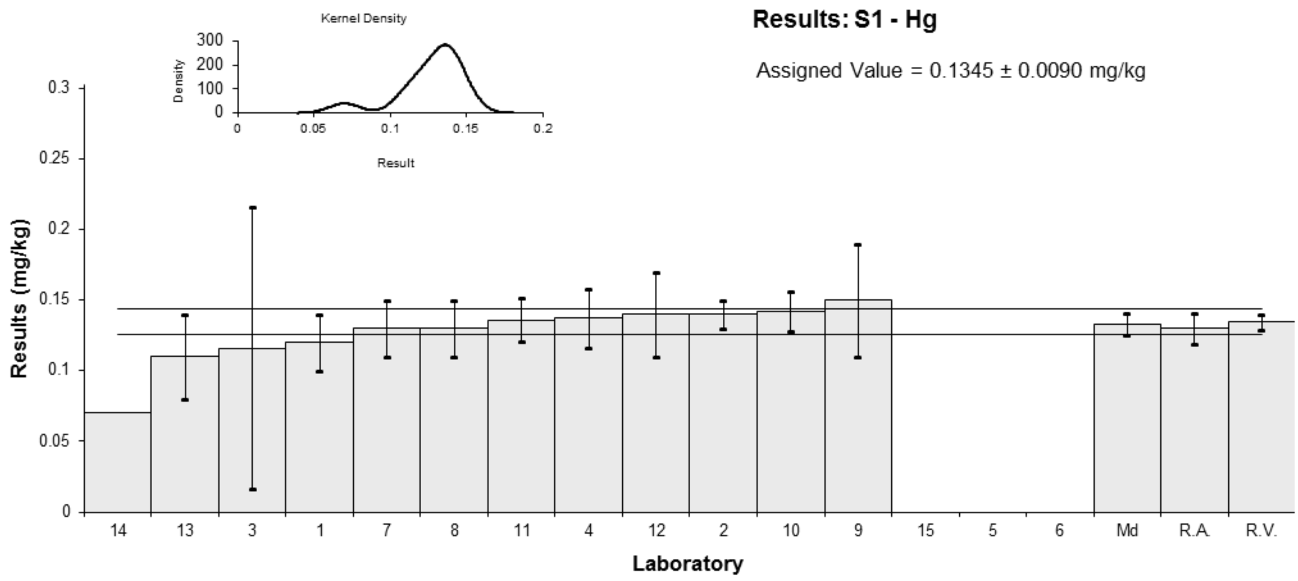
Participant Results

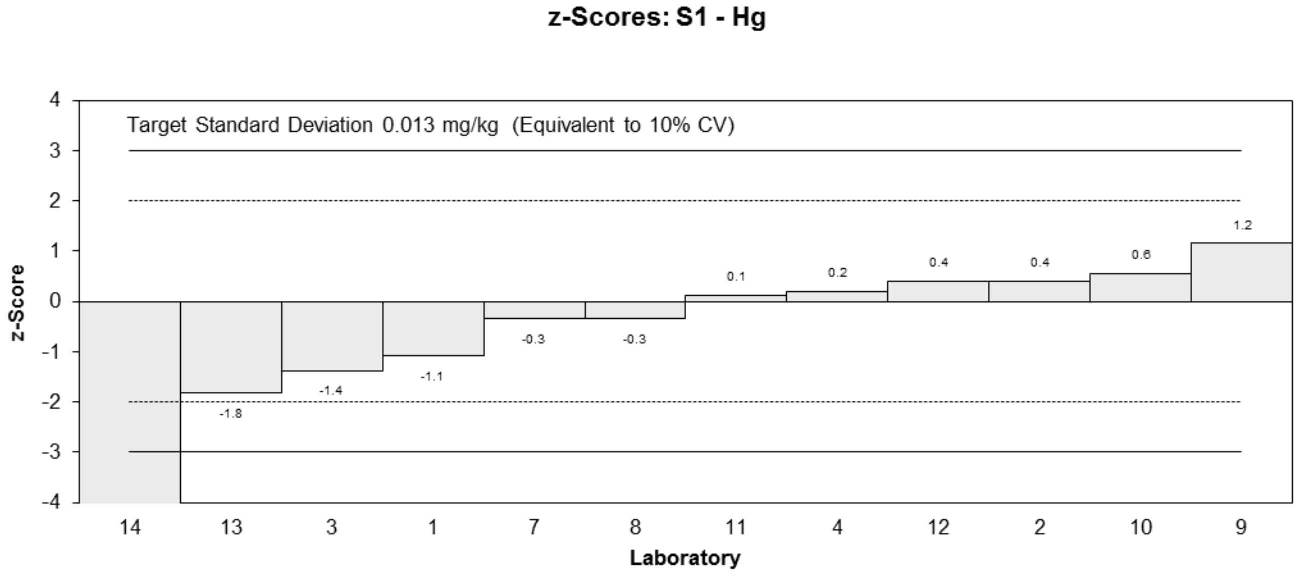
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.12	0.02	-1.08	-0.66
2	0.14	0.01	0.41	0.41
3	0.116	0.10	-1.38	-0.18
4	0.137	0.021	0.19	0.11
5	NT	NT		
6	NT	NT		
7	0.13	0.02	-0.33	-0.21
8	0.13	0.02	-0.33	-0.21
9	0.15	0.04	1.15	0.38
10	0.142	0.014	0.56	0.45
11	0.136	0.0154	0.11	0.08
12	0.14	0.03	0.41	0.18
13	0.11	0.03	-1.82	-0.78
14	0.07	NR	-4.80	-7.17
15	NT	NT		

Statistics

Assigned Value*	0.1345	0.0090
Spike	Not Spiked	
Reference Value*	0.1345	0.0090
Robust Average	0.130	0.011
Median	0.133	0.008
Mean	0.127	
N	12	
Max.	0.15	
Min.	0.07	
Robust SD	0.016	
Robust CV	12%	

*Reference Value by IDMS


Results: S1 - Hg
Assigned Value = 0.1345 ± 0.0090 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
14 13 3 1 7 8 11 4 12 2 10 9 15 5 6 Md R.A. R.V.


z-Scores: S1 - Hg
Target Standard Deviation 0.013 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
14 13 3 1 7 8 11 4 12 2 10 9

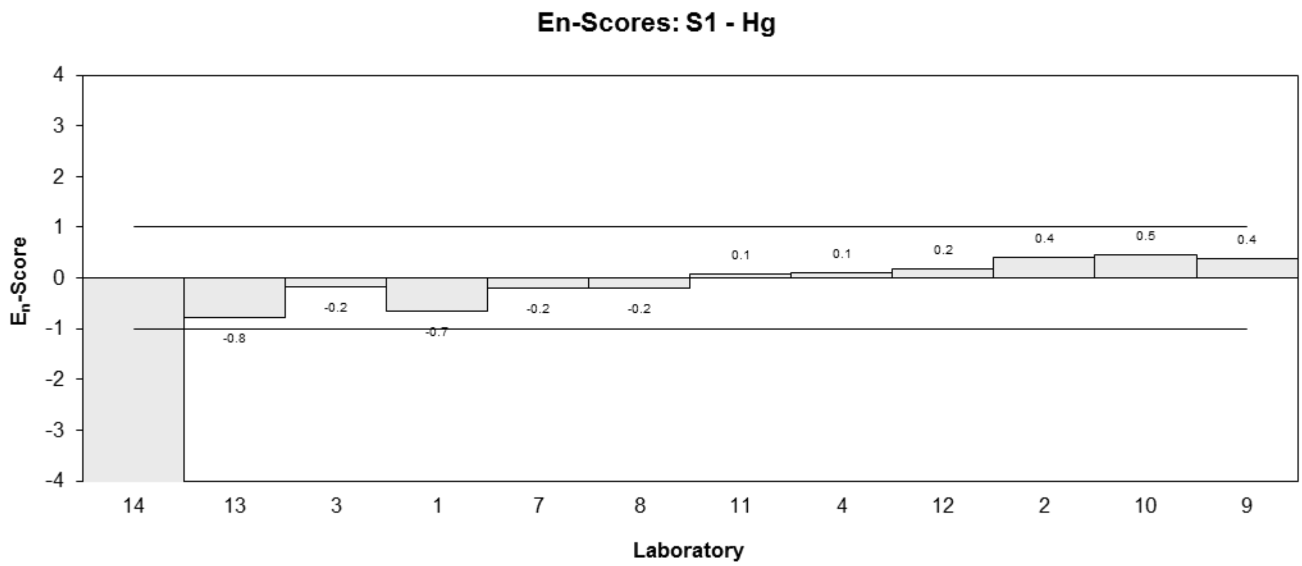

En-Scores: S1 - Hg
En-Score
Laboratory
14 13 3 1 7 8 11 4 12 2 10 9

Figure 14

Table 20

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	0.21	0.04
2	NT	NT
3	NR	NR
4	NT	NT
5	NT	NT
6	NT	NT
7	0.23	0.03
8	NT	NT
9	NR	NR
10	NT	NT
11	NT	NT
12	0.41	0.10
13	NT	NT
14	NT	NT
15	NT	NT

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Mean	0.28	
Homogeneity Value	0.16	0.02

*Insuficient data to calculate statistics

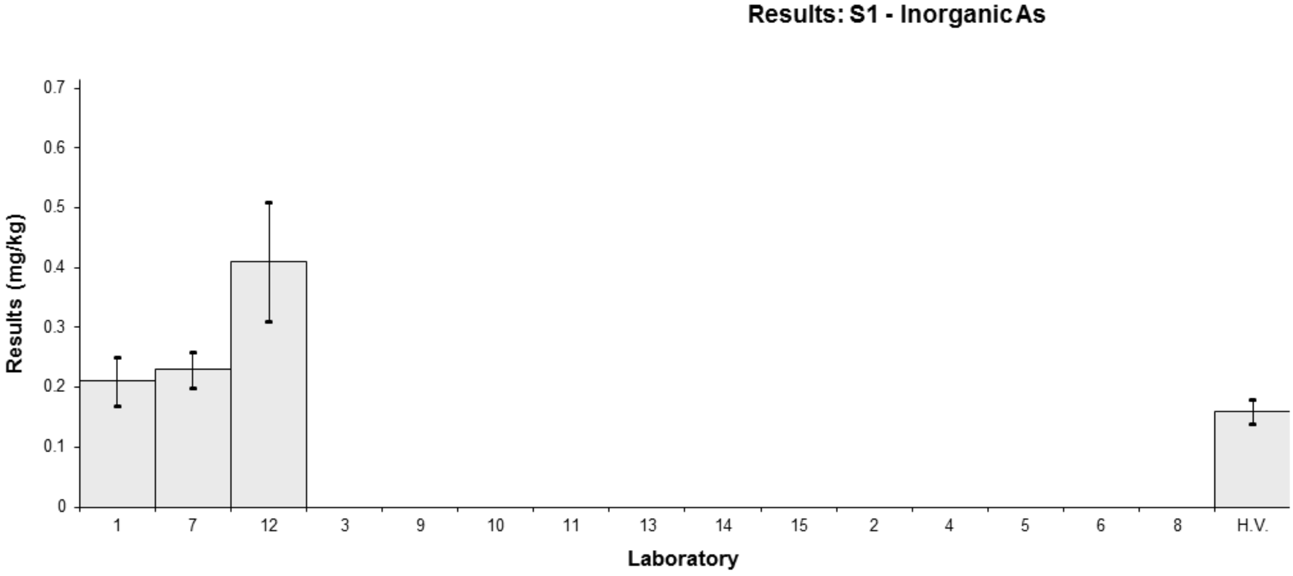

Results: S1 - Inorganic As
Results (mg/kg)
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
1
7
12
3
9
10
11
13
14
15
2
4
5
6
8
H.V.
Laboratory

Figure 15

Table 21

Sample Details

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

Participant Results

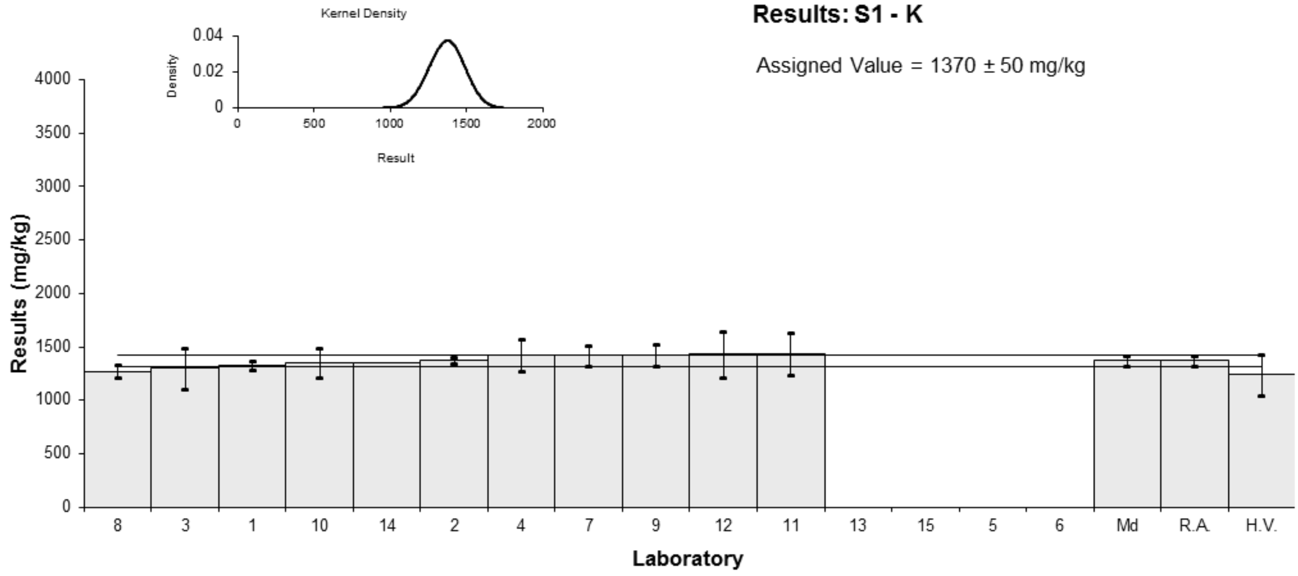
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1330	40	-0.29	-0.62
2	1373	34	0.02	0.05
3	1300	190	-0.51	-0.36
4	1420	150	0.36	0.32
5	NT	NT		
6	NT	NT		
7	1420	97	0.36	0.46
8	1270	59	-0.73	-1.29
9	1420	104	0.36	0.43
10	1350	135	-0.15	-0.14
11	1435	195	0.47	0.32
12	1430	215	0.44	0.27
13	NT	NT		
14	1350	NR	-0.15	-0.40
15	NT	NT		

Statistics

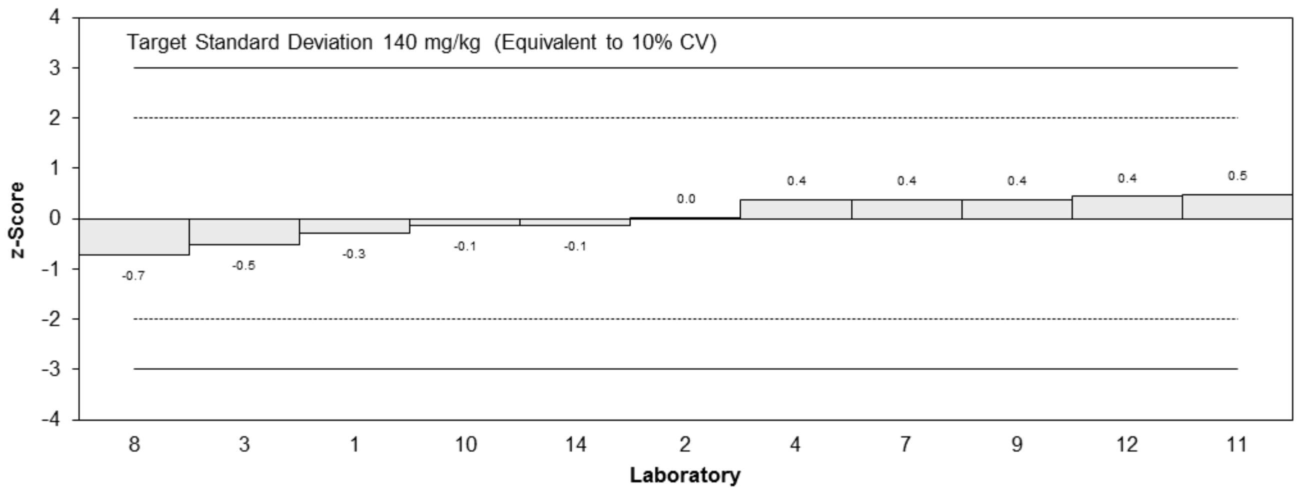
Assigned Value	1370	50
Spike	Not Spiked	
Homogeneity Value	1240	190
Robust Average	1370	50
Median	1370	50
Mean	1373	
N	11	
Max.	1435	
Min.	1270	
Robust SD	60	
Robust CV	4.6%	

Results: S1 - K

Assigned Value = 1370 ± 50 mg/kg



z-Scores: S1 - K



En-Scores: S1 - K

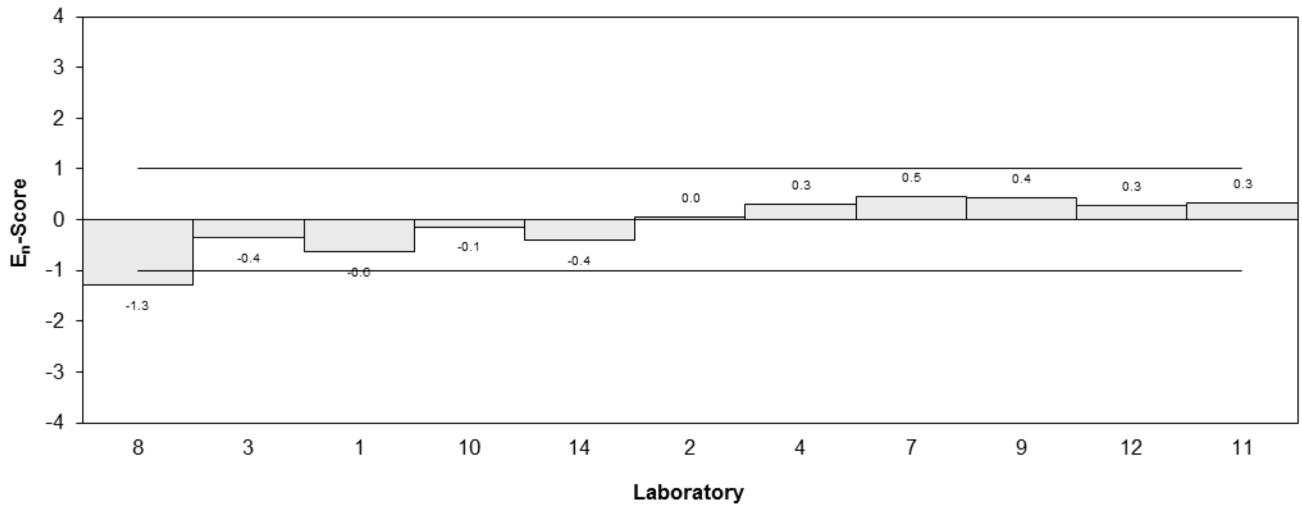


Figure 16

Table 22

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	NT	NT
3	0.0286	0.015
4	0.0311	0.0079
5	NT	NT
6	NT	NT
7	0.03	0.01
8	0.049	0.003
9	0.12	0.014
10	NT	NT
11	NT	NT
12	<0.05	0.02
13	NT	NT
14	<0.1	NR
15	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.031	0.005
Robust Average	0.046	0.033
Median	0.031	0.005
Mean	0.052	
N	5	
Max.	0.12	
Min.	0.029	
Robust SD	0.011	
Robust CV	31%	

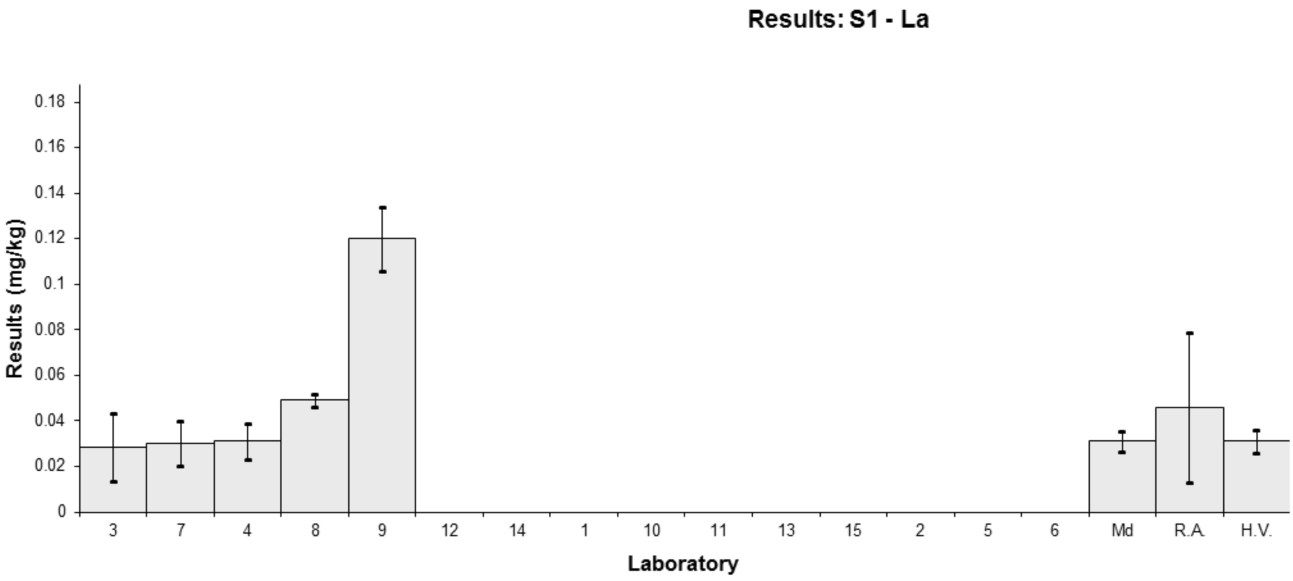

Results: S1 - La
Results (mg/kg)
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
3
7
4
8
9
12
14
1
10
11
13
15
2
5
6
Md
R.A.
H.V.
Laboratory

Figure 17

Table 23

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	0.14	0.04
2	<0.20	NR
3	<0.3	NR
4	0.105	0.067
5	NT	NT
6	NT	NT
7	0.11	0.04
8	0.15	0.01
9	0.23	0.03
10	0.152	0.015
11	NT	NT
12	0.079	0.02
13	NT	NT
14	0.1	NR
15	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.148	0.022
Robust Average	0.128	0.036
Median	0.125	0.031
Mean	0.133	
N	8	
Max.	0.23	
Min.	0.079	
Robust SD	0.032	
Robust CV	27%	

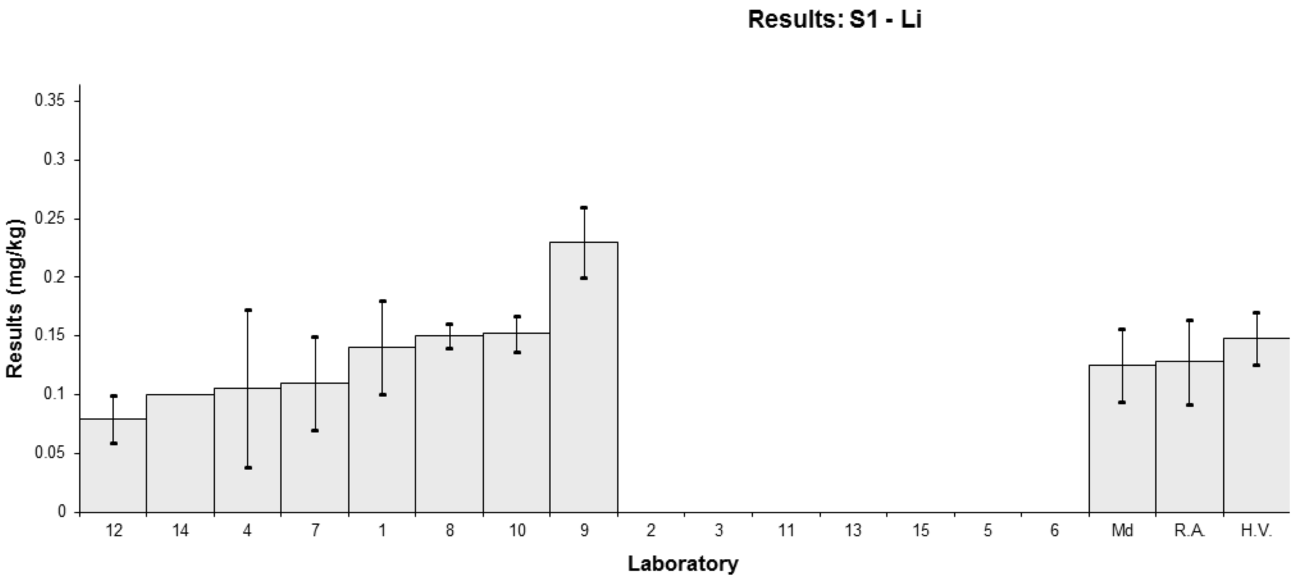

Results: S1 - Li
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
Results (mg/kg)
12
14
4
7
1
8
10
9
2
3
11
13
15
5
6
Md
R.A.
H.V.
Laboratory

Figure 18

Table 24

Sample Details

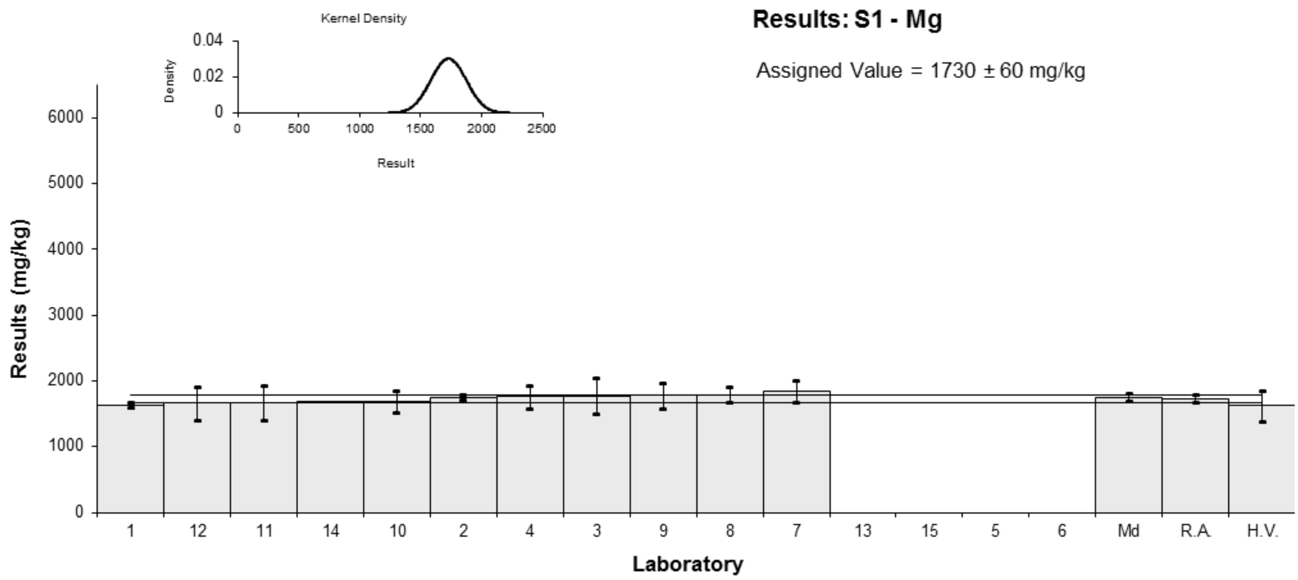
Sample No.	S1
Matrix.	Prawn
Analyte.	Mg
Units	mg/kg

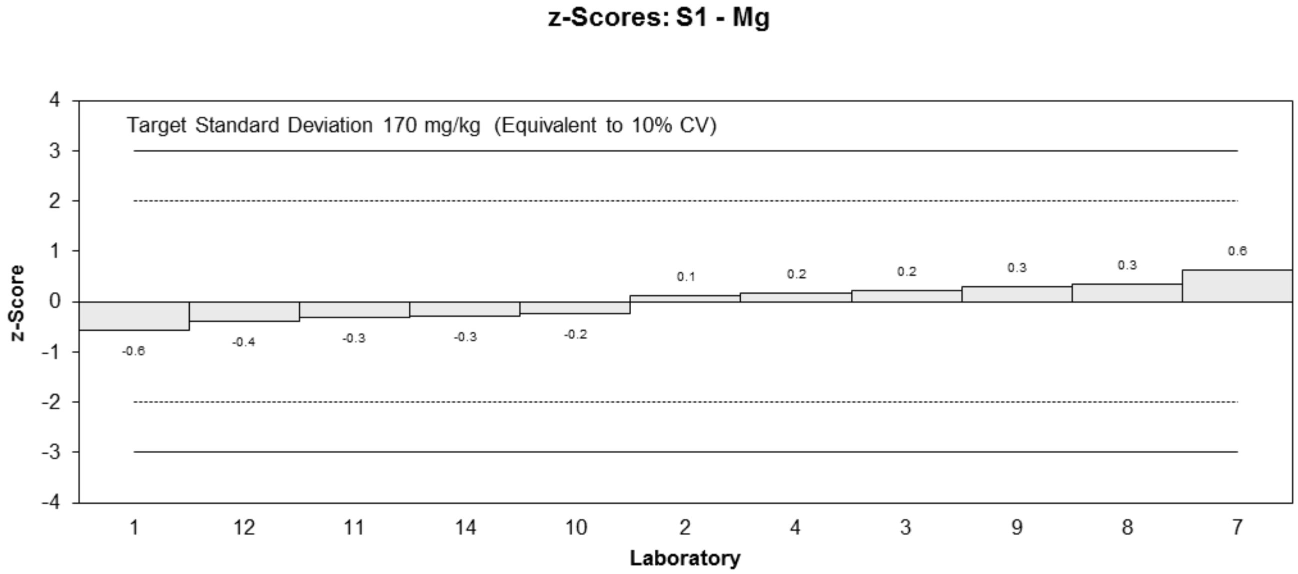
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1630	40	-0.58	-1.39
2	1752	34	0.13	0.32
3	1770	270	0.23	0.14
4	1760	180	0.17	0.16
5	NT	NT		
6	NT	NT		
7	1840	167	0.64	0.62
8	1790	120	0.35	0.45
9	1780	195	0.29	0.25
10	1690	169	-0.23	-0.22
11	1675	261	-0.32	-0.21
12	1660	249	-0.40	-0.27
13	NT	NT		
14	1680	NR	-0.29	-0.83
15	NT	NT		

Statistics

Assigned Value	1730	60
Spike	Not Spiked	
Homogeneity Value	1620	240
Robust Average	1730	60
Median	1750	60
Mean	1730	
N	11	
Max.	1840	
Min.	1630	
Robust SD	70	
Robust CV	4.3%	


Results: S1 - Mg
Assigned Value = 1730 ± 60 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
1
12
11
14
10
2
4
3
9
8
7
13
15
5
6
Md
R.A.
H.V.


z-Scores: S1 - Mg
Target Standard Deviation 170 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-0.6
-0.4
-0.3
-0.3
-0.2
0.1
0.2
0.2
0.3
0.3
0.6

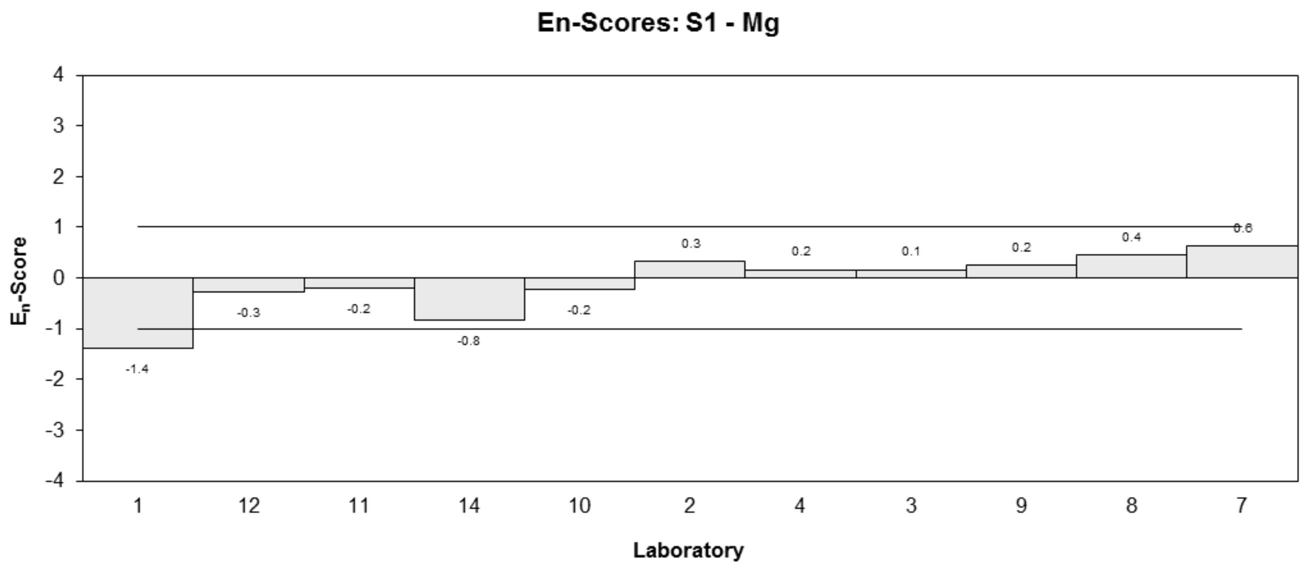

En-Scores: S1 - Mg
E_n-Score
Laboratory
-1.4
-0.3
-0.2
-0.8
-0.2
0.3
0.2
0.1
0.2
0.4
0.6

Figure 19

Table 25

Sample Details

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

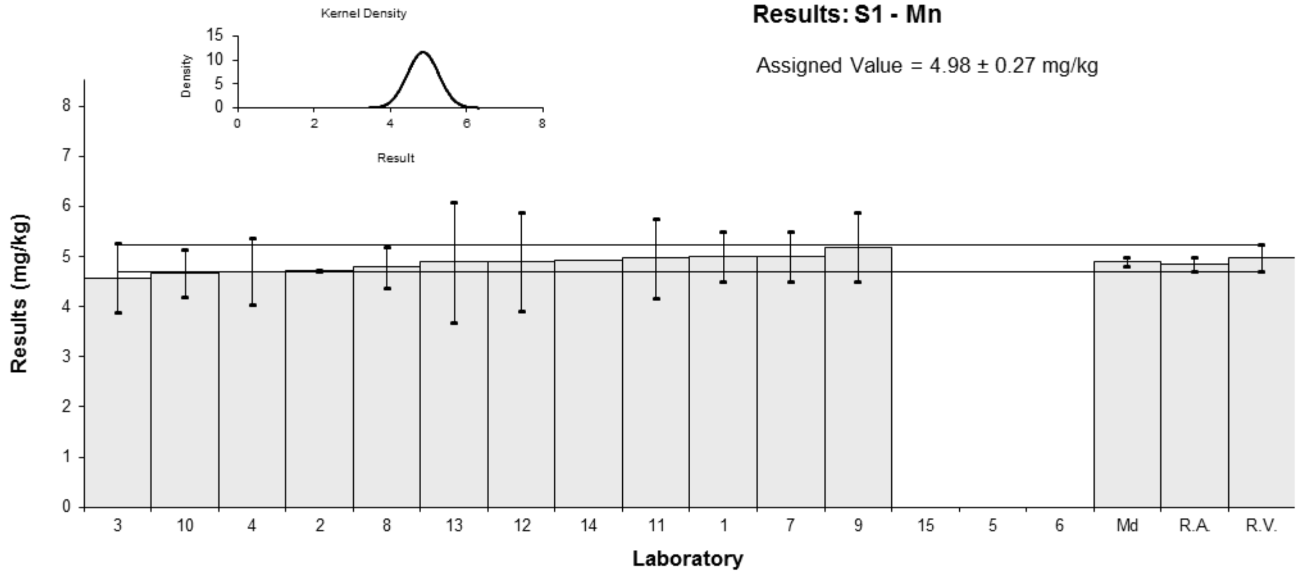
Participant Results

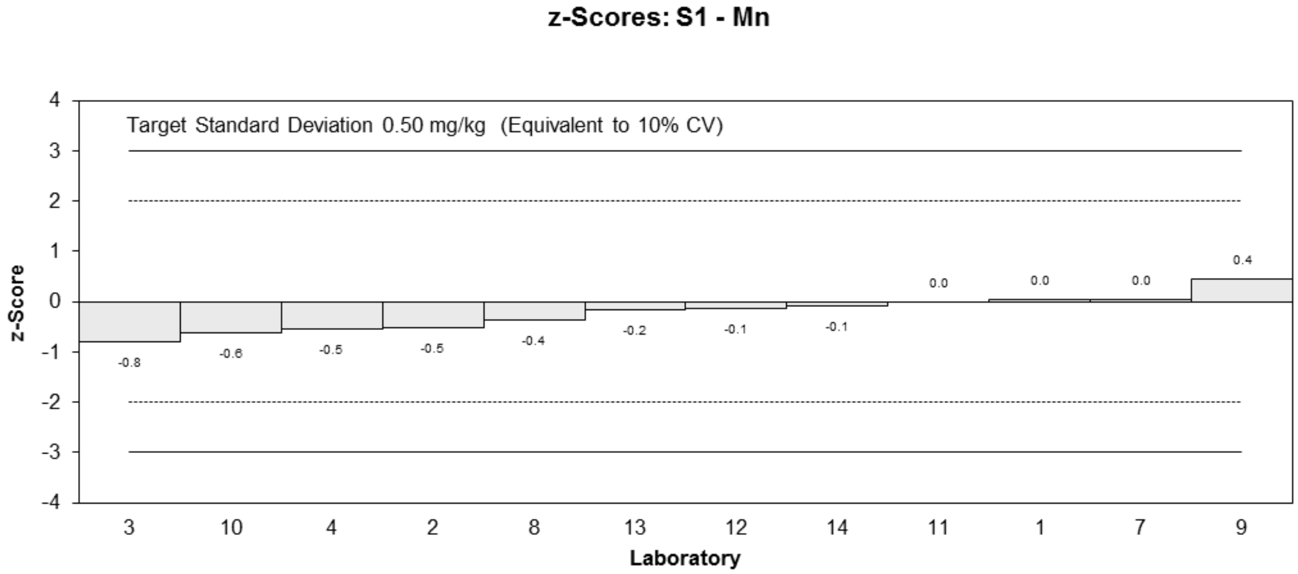
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	5.0	0.5	0.04	0.04
2	4.72	0.01	-0.52	-0.96
3	4.58	0.69	-0.80	-0.54
4	4.71	0.67	-0.54	-0.37
5	NT	NT		
6	NT	NT		
7	5.0	0.5	0.04	0.04
8	4.80	0.41	-0.36	-0.37
9	5.2	0.7	0.44	0.29
10	4.67	0.47	-0.62	-0.57
11	4.98	0.792	0.00	0.00
12	4.91	0.98	-0.14	-0.07
13	4.9	1.2	-0.16	-0.07
14	4.94	NR	-0.08	-0.15
15	NT	NT		

Statistics

Assigned Value*	4.98	0.27
Spike	Not Spiked	
Reference Value*	4.98	0.27
Robust Average	4.86	0.14
Median	4.91	0.09
Mean	4.87	
N	12	
Max.	5.2	
Min.	4.58	
Robust SD	0.19	
Robust CV	3.9%	

*Reference Value by SA-ICP-MS


Kernel Density
Density
Result
Results: S1 - Mn
Assigned Value = 4.98 ± 0.27 mg/kg
Results (mg/kg)
Laboratory
3
10
4
2
8
13
12
14
11
1
7
9
15
5
6
Md
R.A.
R.V.


z-Scores: S1 - Mn
Target Standard Deviation 0.50 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-0.8
-0.6
-0.5
-0.5
-0.4
-0.2
-0.1
-0.1
0.0
0.0
0.0
0.4

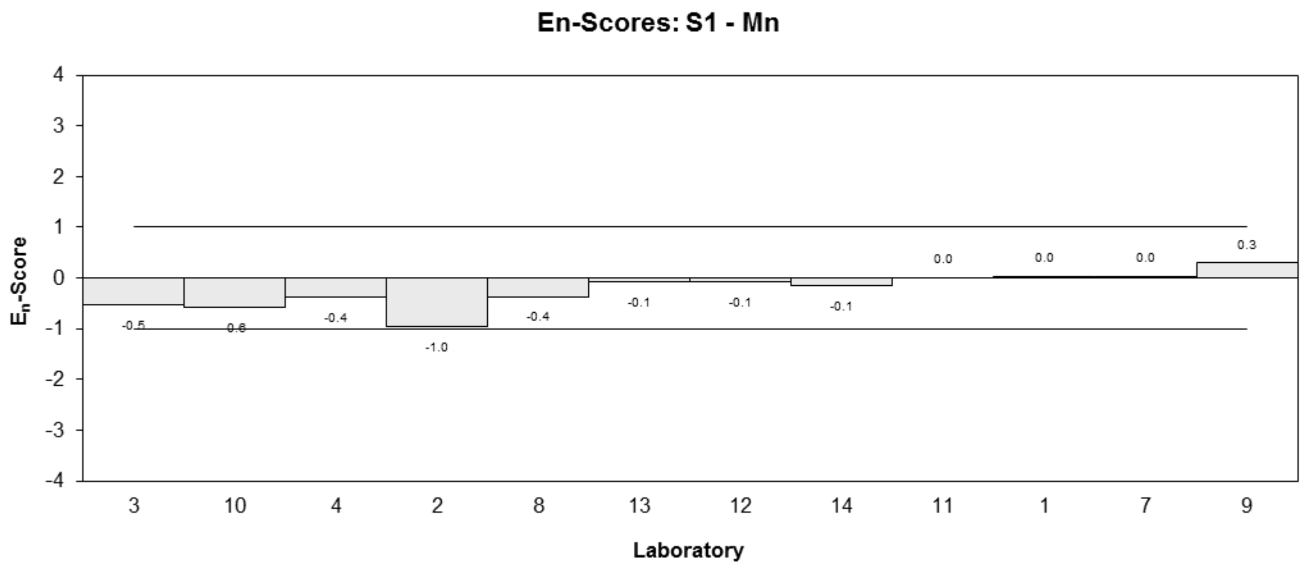

En-Scores: S1 - Mn
En-Score
Laboratory
-0.5
-0.6
-0.4
-1.0
-0.4
-0.1
-0.1
-0.1
0.0
0.0
0.0
0.3

Figure 20

Table 26

Sample Details

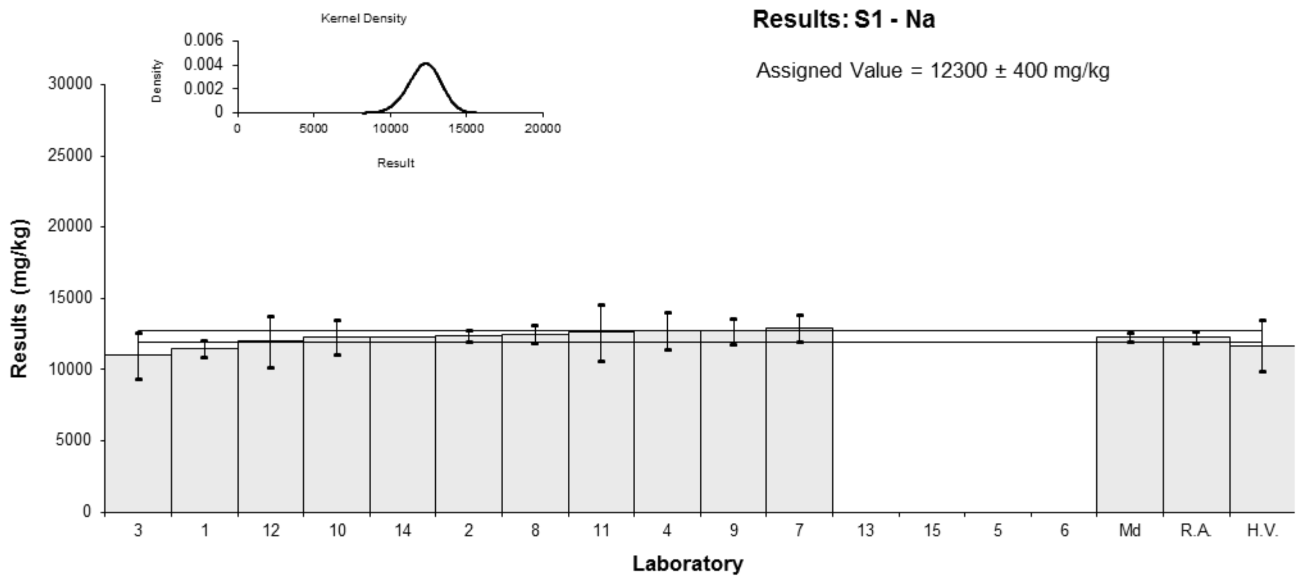
Sample No.	S1
Matrix.	Prawn
Analyte.	Na
Units	mg/kg

Participant Results

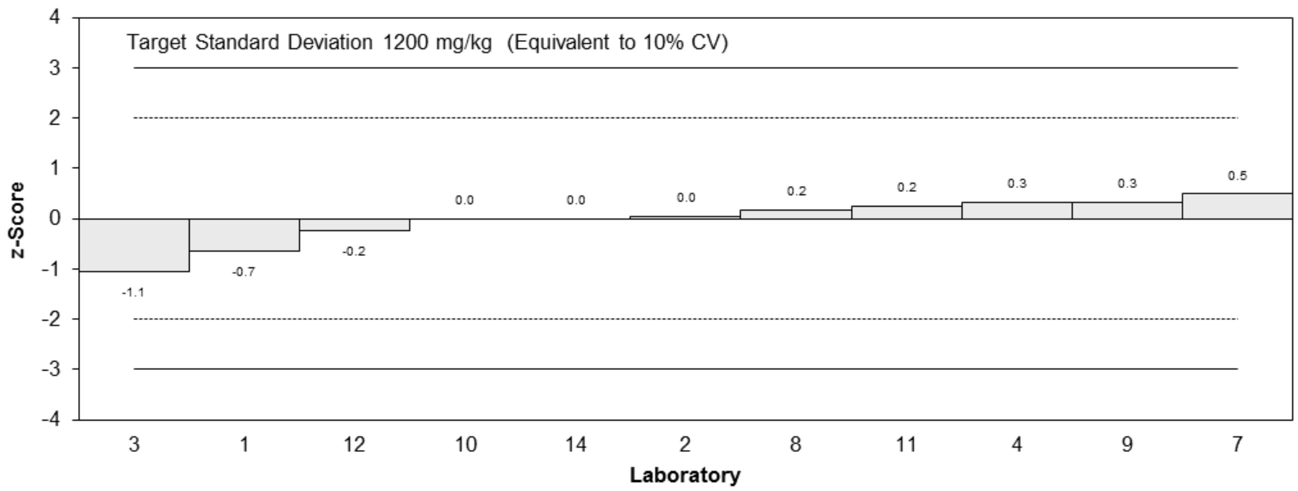
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	11500	600	-0.65	-1.11
2	12340	410	0.03	0.07
3	11000	1600	-1.06	-0.79
4	12700	1300	0.33	0.29
5	NT	NT		
6	NT	NT		
7	12900	973	0.49	0.57
8	12500	600	0.16	0.28
9	12700	890	0.33	0.41
10	12300	1230	0.00	0.00
11	12600	2020	0.24	0.15
12	12000	1800	-0.24	-0.16
13	NT	NT		
14	12300	NR	0.00	0.00
15	NT	NT		

Statistics

Assigned Value	12300	400
Spike	Not Spiked	
Homogeneity Value	11700	1800
Robust Average	12300	400
Median	12300	300
Mean	12258	
N	11	
Max.	12900	
Min.	11000	
Robust SD	500	
Robust CV	4.2%	



z-Scores: S1 - Na



En-Scores: S1 - Na

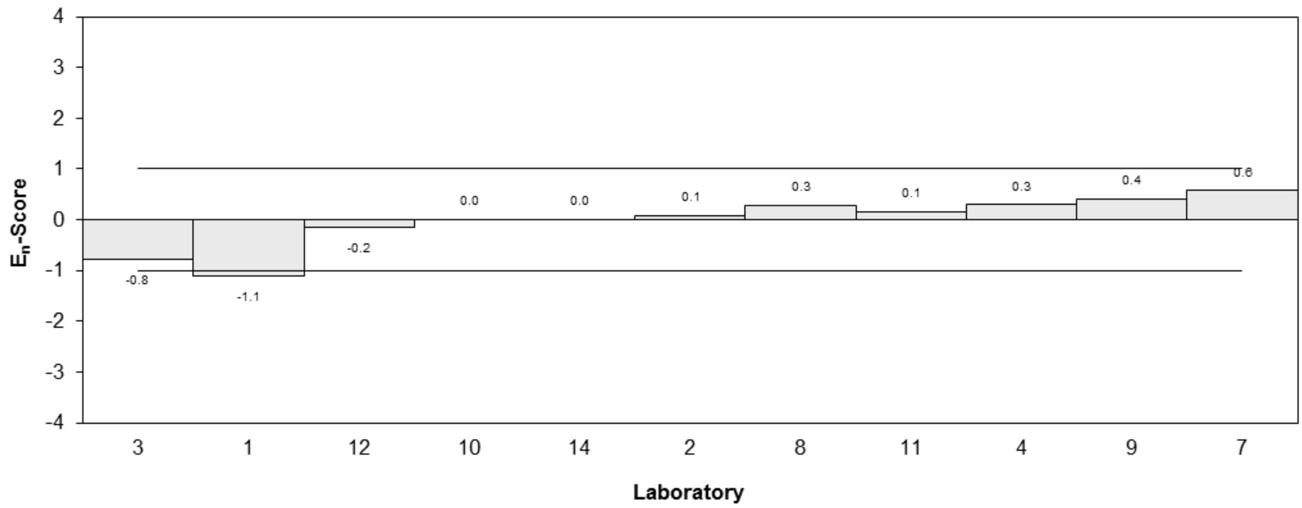


Figure 21

Table 27

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.27	0.03	-2.08	-2.82
2	0.92	0.08	4.96	4.55
3	0.391	0.059	-0.77	-0.84
4	0.216	0.072	-2.66	-2.61
5	NT	NT		
6	NT	NT		
7	0.17	0.04	-3.16	-4.00
8	0.41	0.09	-0.56	-0.48
9	NR	NR		
10	0.359	0.036	-1.11	-1.45
11	0.585	0.060	1.33	1.44
12	0.40	0.08	-0.67	-0.62
13	<1	NR		
14	0.41	NR	-0.56	-0.85
15	NT	NT		

Statistics

Assigned Value*	0.462	0.061
Spike	Not Spiked	
Reference Value*	0.462	0.061
Robust Average	0.38	0.13
Median	0.396	0.086
Mean	0.413	
N	10	
Max.	0.92	
Min.	0.17	
Robust SD	0.084	
Robust CV	43%	

*Reference Value by SA-ICP-MS

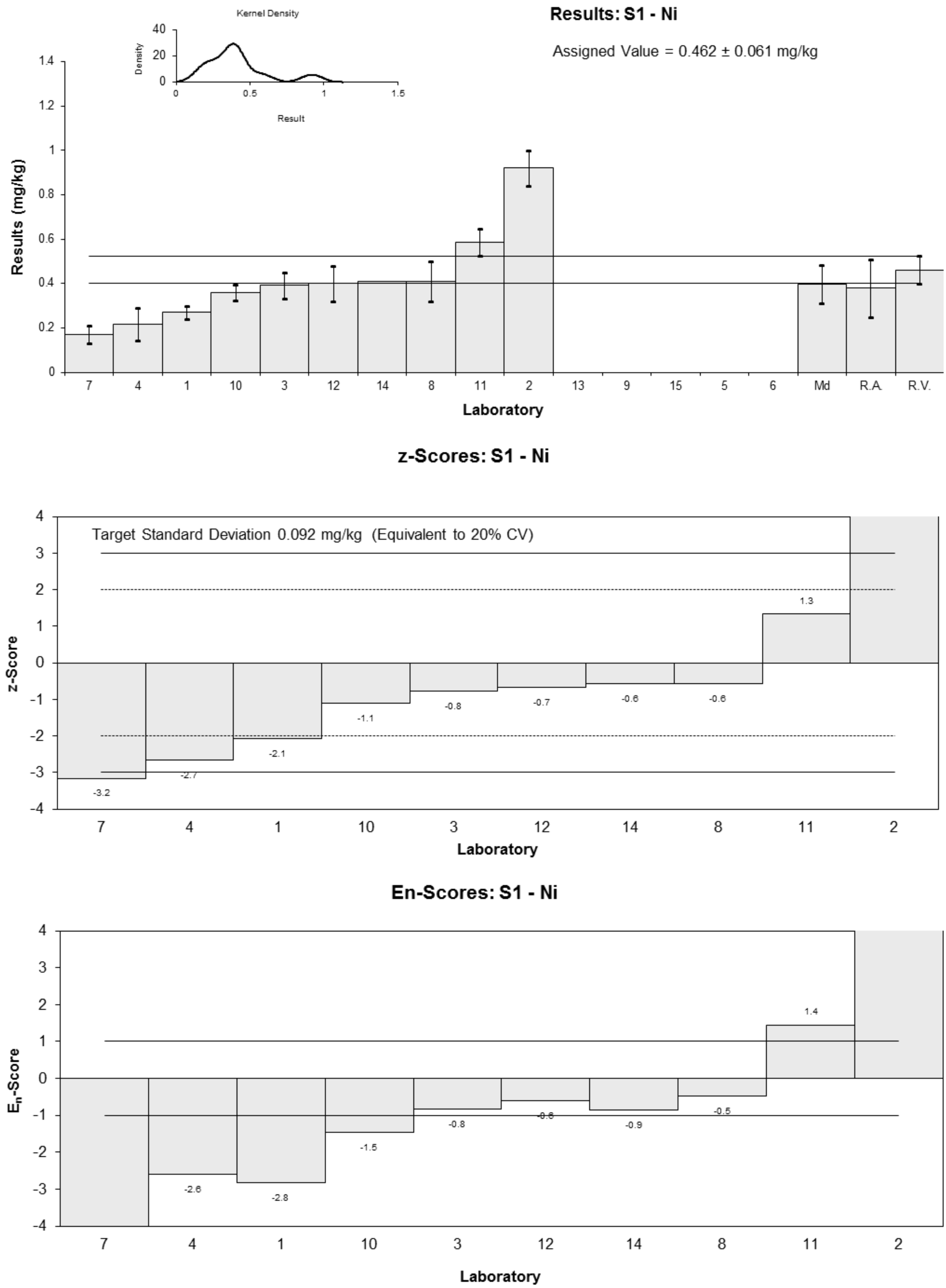

Results: S1 - Ni
Assigned Value = 0.462 ± 0.061 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
7
4
1
10
3
12
14
8
11
2
13
9
15
5
6
Md
R.A.
R.V.
z-Scores: S1 - Ni
Target Standard Deviation 0.092 mg/kg (Equivalent to 20% CV)
z-Score
-3.2
-2.1
-1.1
-0.8
-0.7
-0.6
-0.6
1.3
En-Scores: S1 - Ni
E_n-Score
-2.6
-2.8
-1.5
-0.8
-0.9
-0.5
1.4

Figure 22

Table 28

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	6000	300	-0.05	-0.08
2	6382	198	0.58	1.13
3	5900	1000	-0.22	-0.13
4	6190	620	0.27	0.24
5	NT	NT		
6	NT	NT		
7	6240	414	0.35	0.44
8	6100	400	0.12	0.15
9	6400	440	0.61	0.74
10	5670	567	-0.60	-0.58
11	5900	914	-0.22	-0.14
12	5600	840	-0.71	-0.49
13	5610	1400	-0.70	-0.30
14	6310	NR	0.46	1.17
15	NT	NT		

Statistics

Assigned Value	6030	240
Spike	Not Spiked	
Homogeneity Value	6380	960
Robust Average	6030	240
Median	6050	210
Mean	6025	
N	12	
Max.	6400	
Min.	5600	
Robust SD	330	
Robust CV	5.5%	

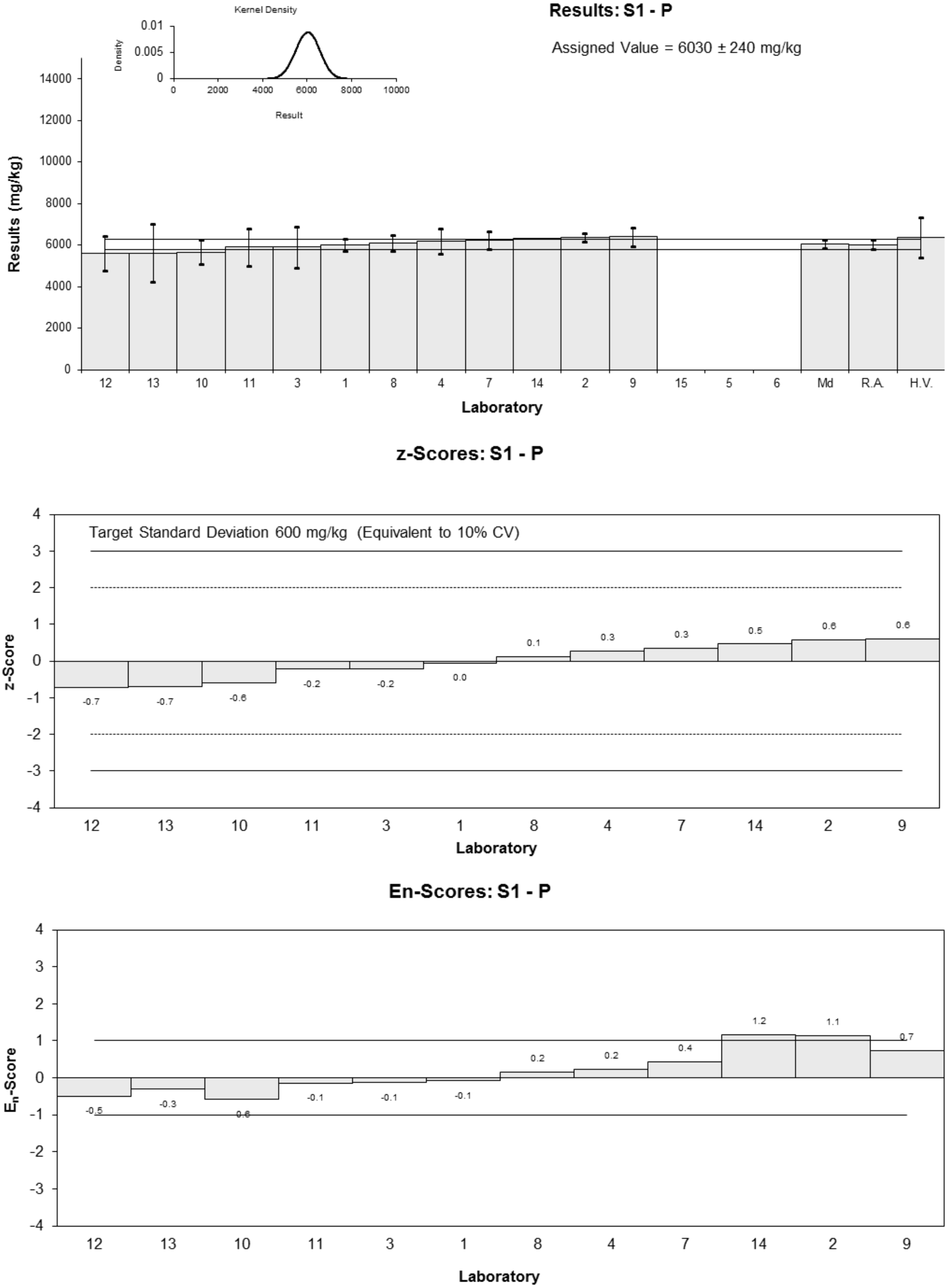

Results: S1 - P
Assigned Value = 6030 ± 240 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
12 13 10 11 3 1 8 4 7 14 2 9 15 5 6 Md R.A. H.V.
z-Scores: S1 - P
Target Standard Deviation 600 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
12 13 10 11 3 1 8 4 7 14 2 9
-0.7 -0.7 -0.6 -0.2 -0.2 0.0 0.1 0.3 0.3 0.5 0.6 0.6
En-Scores: S1 - P
E_n-Score
Laboratory
12 13 10 11 3 1 8 4 7 14 2 9
-0.5 -0.3 -0.6 -0.1 -0.1 -0.1 0.2 0.2 0.4 1.2 1.1 0.7

Figure 23

Table 29

Sample Details

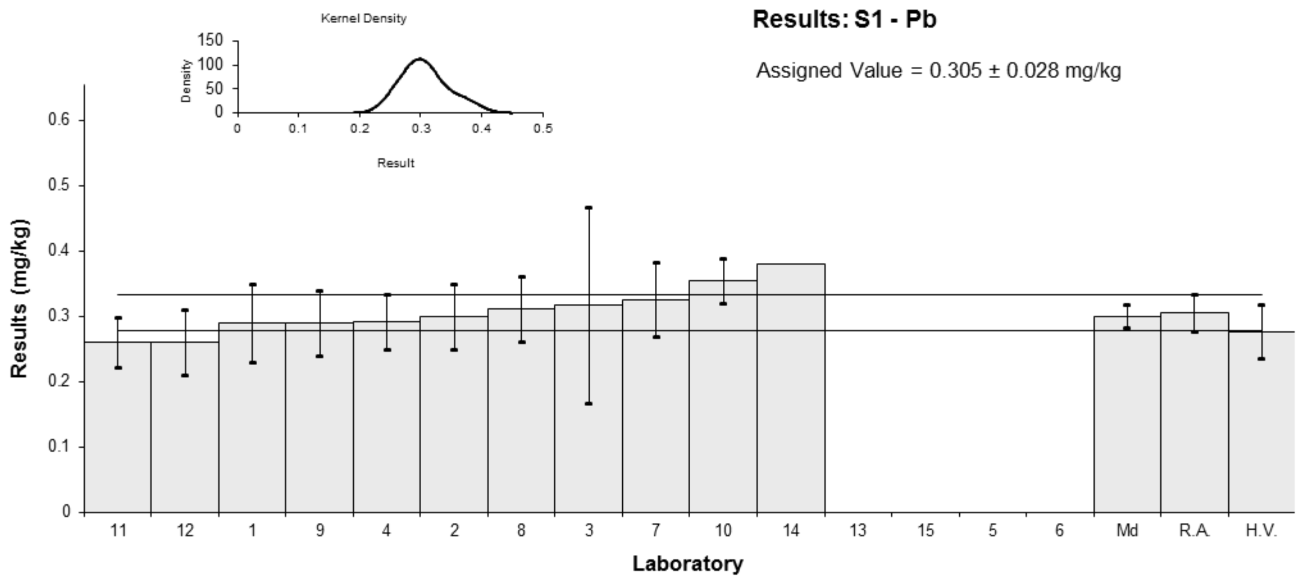
Sample No.	S1
Matrix.	Prawn
Analyte.	Pb
Units	mg/kg

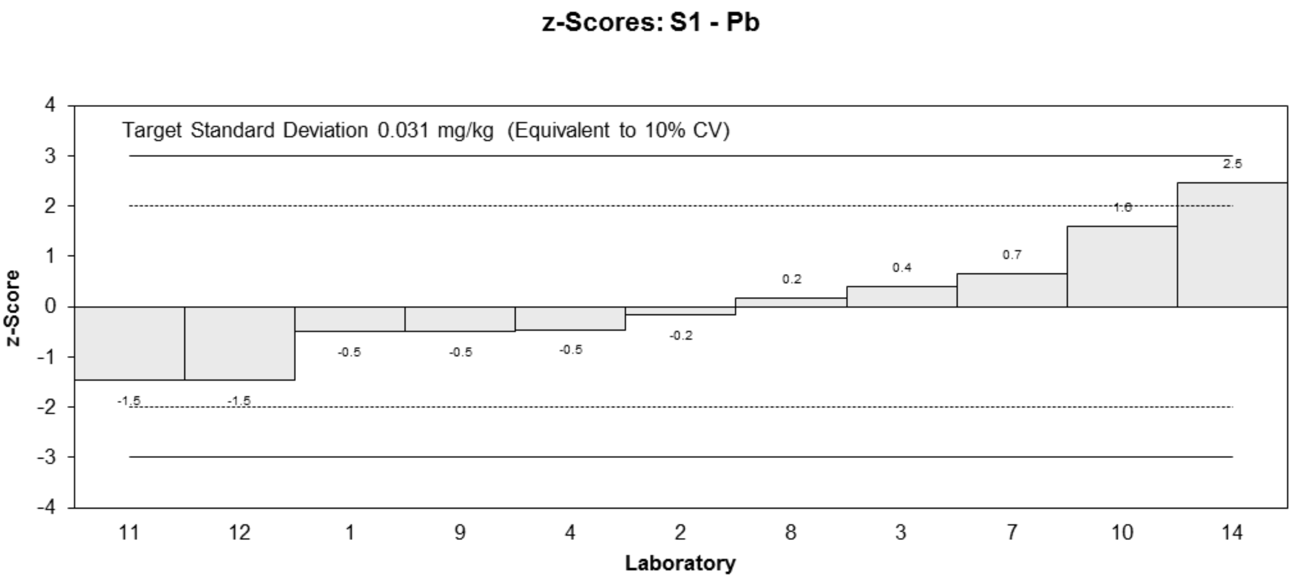
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.29	0.06	-0.49	-0.23
2	0.30	0.05	-0.16	-0.09
3	0.317	0.15	0.39	0.08
4	0.291	0.042	-0.46	-0.28
5	NT	NT		
6	NT	NT		
7	0.325	0.057	0.66	0.31
8	0.31	0.05	0.16	0.09
9	0.29	0.05	-0.49	-0.26
10	0.354	0.035	1.61	1.09
11	0.260	0.039	-1.48	-0.94
12	0.26	0.05	-1.48	-0.79
13	<1	NR		
14	0.38	NR	2.46	2.68
15	NT	NT		

Statistics

Assigned Value	0.305	0.028
Spike	Not Spiked	
Homogeneity Value	0.276	0.041
Robust Average	0.305	0.028
Median	0.300	0.017
Mean	0.307	
N	11	
Max.	0.38	
Min.	0.26	
Robust SD	0.037	
Robust CV	12%	


Results: S1 - Pb
Assigned Value = 0.305 ± 0.028 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
11 12 1 9 4 2 8 3 7 10 14 13 15 5 6 Md R.A. H.V.


z-Scores: S1 - Pb
Target Standard Deviation 0.031 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
11 12 1 9 4 2 8 3 7 10 14
-1.5 -1.5 -0.5 -0.5 -0.5 -0.2 0.2 0.4 0.7 1.6 2.5

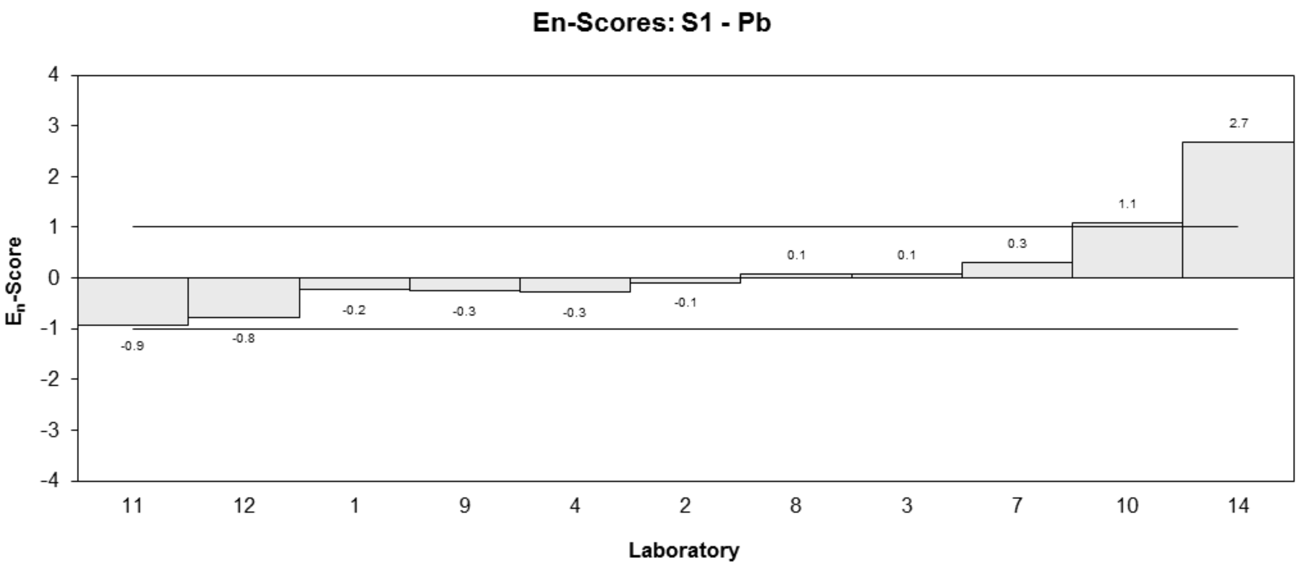

En-Scores: S1 - Pb
E_n-Score
Laboratory
11 12 1 9 4 2 8 3 7 10 14
-0.9 -0.8 -0.2 -0.3 -0.3 -0.1 0.1 0.1 0.3 1.1 2.7

Figure 24

Table 30

Sample Details

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

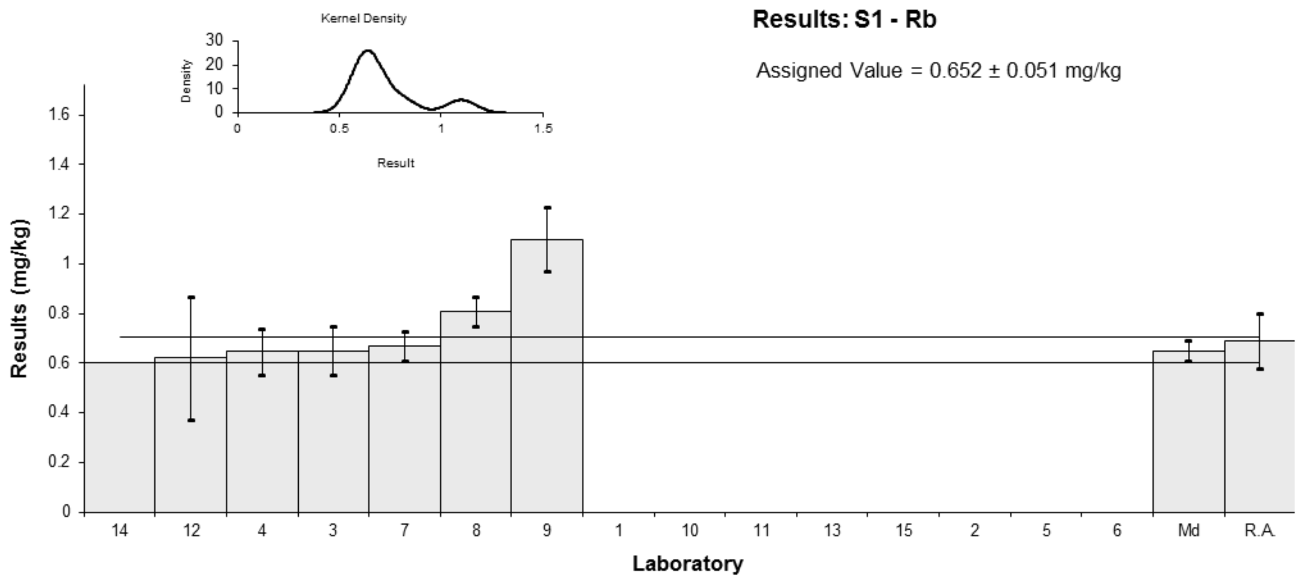
Participant Results

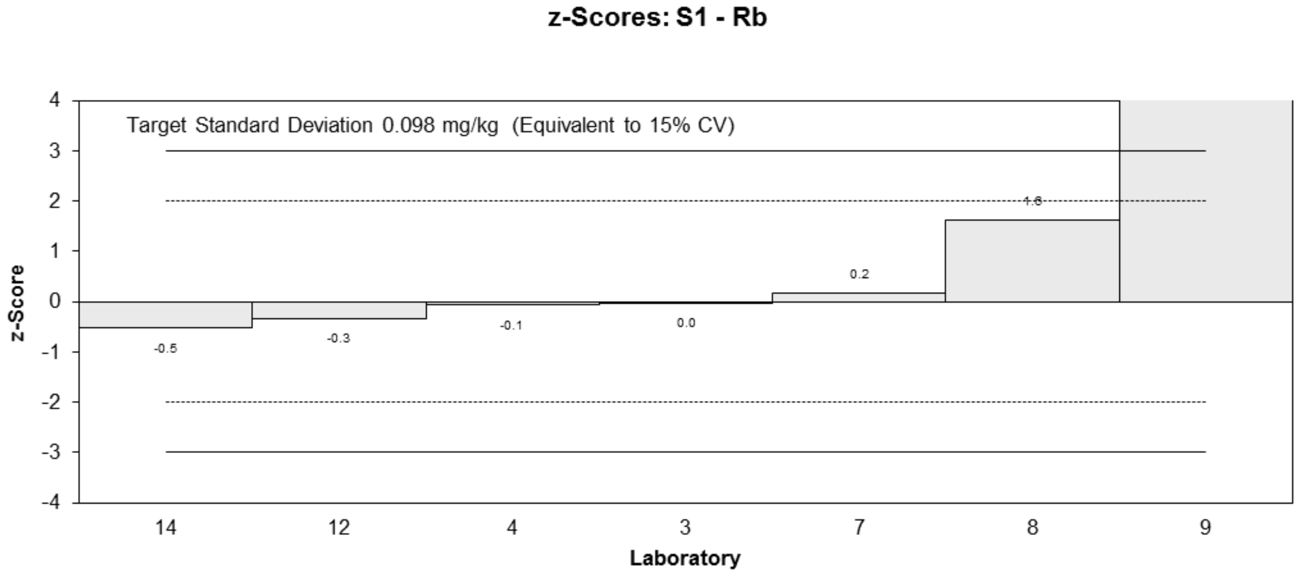
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	NT	NT		
2	NT	NT		
3	0.650	0.097	-0.02	-0.02
4	0.646	0.091	-0.06	-0.06
5	NT	NT		
6	NT	NT		
7	0.67	0.06	0.18	0.23
8	0.81	0.06	1.62	2.01
9	1.1	0.13	4.58	3.21
10	NT	NT		
11	NT	NT		
12	0.62	0.25	-0.33	-0.13
13	NT	NT		
14	0.6	NR	-0.53	-1.02
15	NT	NT		

Statistics

Assigned Value*	0.652	0.051
Spike	Not Spiked	
Robust Average	0.69	0.11
Median	0.650	0.041
Mean	0.728	
N	7	
Max.	1.1	
Min.	0.6	
Robust SD	0.05	
Robust CV	7.7%	

*Robust Average excluding Laboratory 9.


Results: S1 - Rb
Assigned Value = 0.652 ± 0.051 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
14
12
4
3
7
8
9
1
10
11
13
15
2
5
6
Md
R.A.


z-Scores: S1 - Rb
Target Standard Deviation 0.098 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
-0.5
-0.3
-0.1
0.0
0.2
1.6

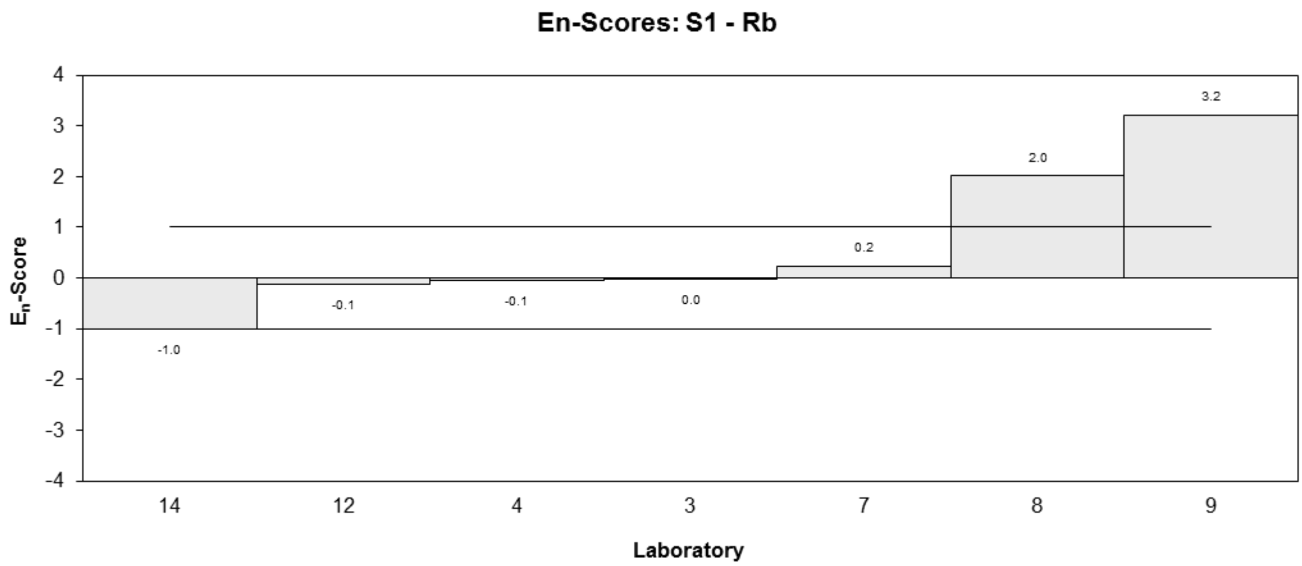

En-Scores: S1 - Rb
En-Score
Laboratory
-1.0
-0.1
-0.1
0.0
0.2
2.0
3.2

Figure 25

Table 31

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	9100	500	-0.52	-0.45
2	NT	NT		
3	NR	NR		
4	10400	1100	0.83	0.54
5	NT	NT		
6	NT	NT		
7	10900	1440	1.35	0.74
8	10300	600	0.73	0.60
9	11100	750	1.56	1.20
10	8230	823	-1.43	-1.06
11	10300	1670	0.73	0.36
12	9050	1810	-0.57	-0.27
13	8490	2120	-1.16	-0.47
14	8060	NR	-1.60	-1.54
15	NT	NT		

Statistics

Assigned Value	9600	1000
Spike	Not Spiked	
Robust Average	9600	1000
Median	9700	1000
Mean	9593	
N	10	
Max.	11100	
Min.	8060	
Robust SD	1300	
Robust CV	13%	

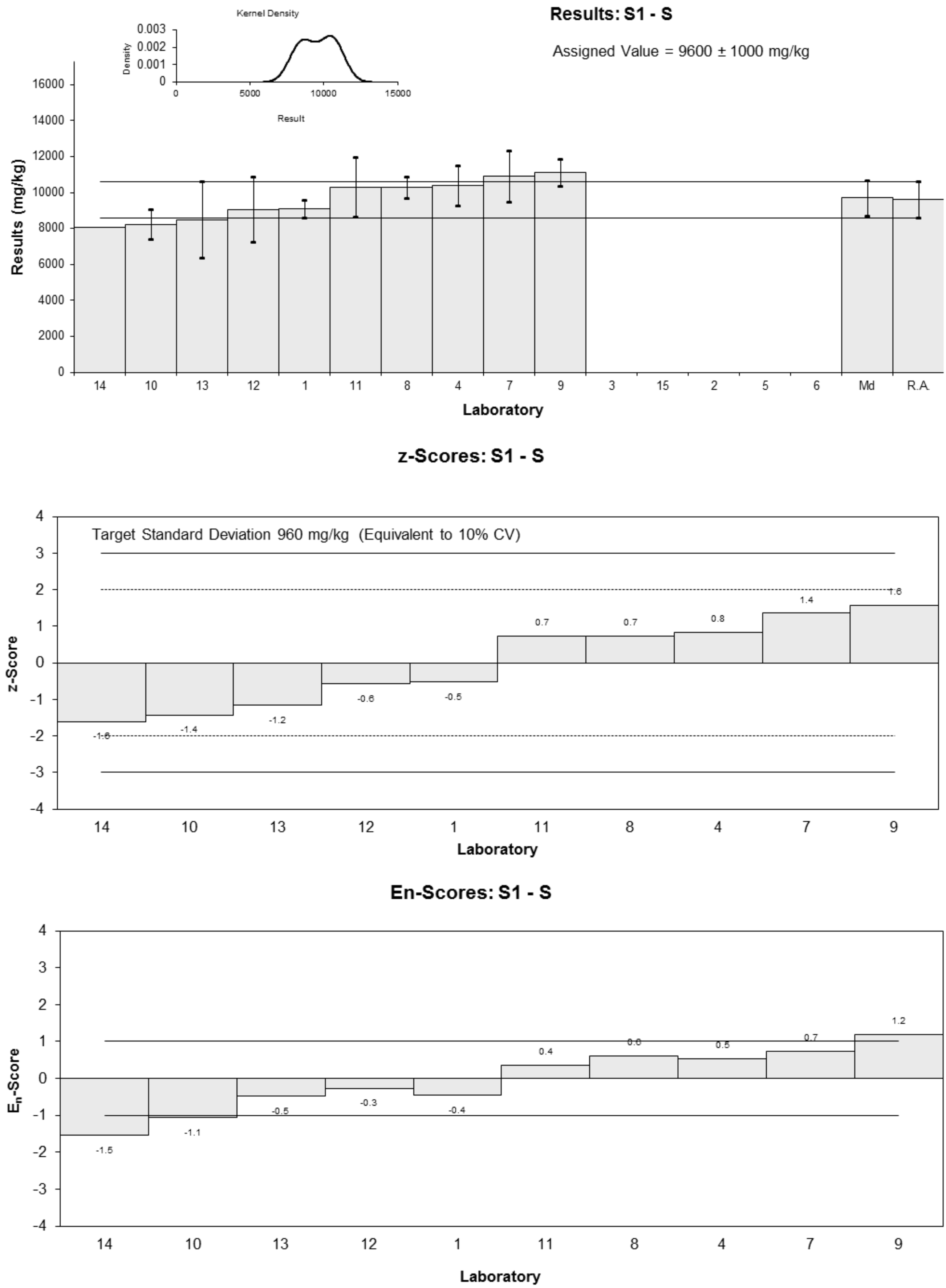

Results: S1 - S
Assigned Value = 9600 ± 1000 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
14
10
13
12
1
11
8
4
7
9
3
15
2
5
6
Md
R.A.
z-Scores: S1 - S
Target Standard Deviation 960 mg/kg (Equivalent to 10% CV)
z-Score
-1.6
-1.4
-1.2
-0.6
-0.5
0.7
0.7
0.8
1.4
1.6
En-Scores: S1 - S
E_n-Score
-1.5
-1.1
-0.5
-0.3
-0.4
0.4
0.6
0.5
0.7
1.2

Figure 26

Table 32

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	<0.01	NR
2	<0.05	NR
3	0.0513	0.0077
4	<0.10	0.068
5	NT	NT
6	NT	NT
7	<0.02	NR
8	NT	NT
9	0.018	0.003
10	0.018	0.005
11	<0.10	NR
12	<0.01	0.002
13	NT	NT
14	<0.1	NR
15	NT	NT

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Mean	0.029	

*Insufficient data to calculate statistics

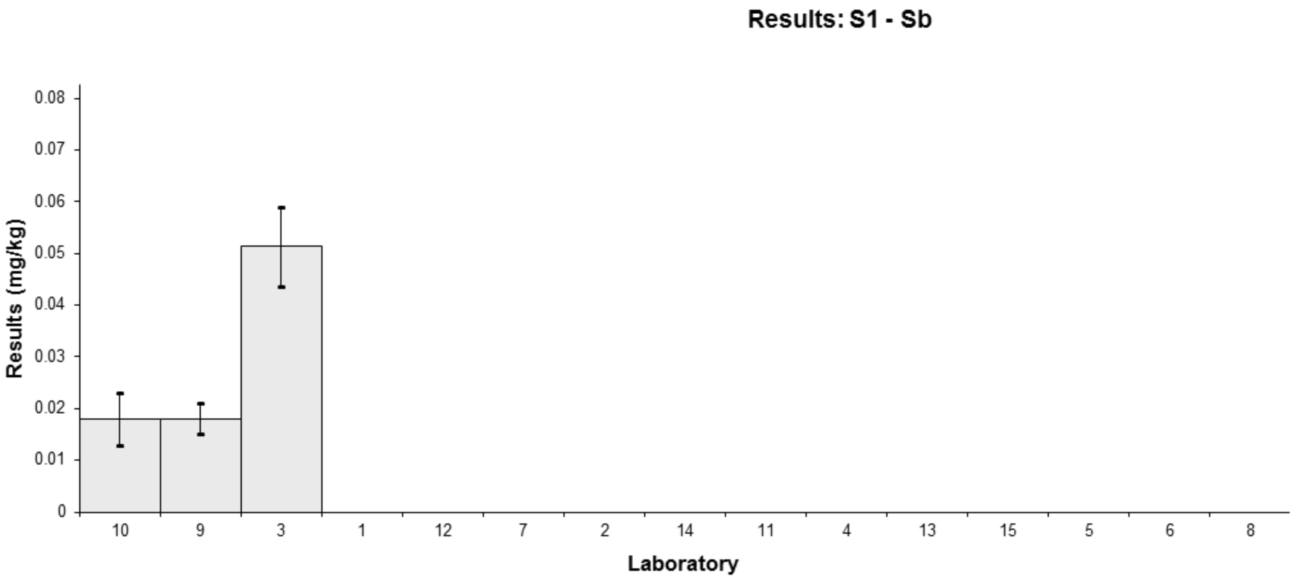

Results: S1 - Sb
Results (mg/kg)
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
10
9
3
1
12
7
2
14
11
4
13
15
5
6
8
Laboratory

Figure 27

Table 33

Sample Details

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

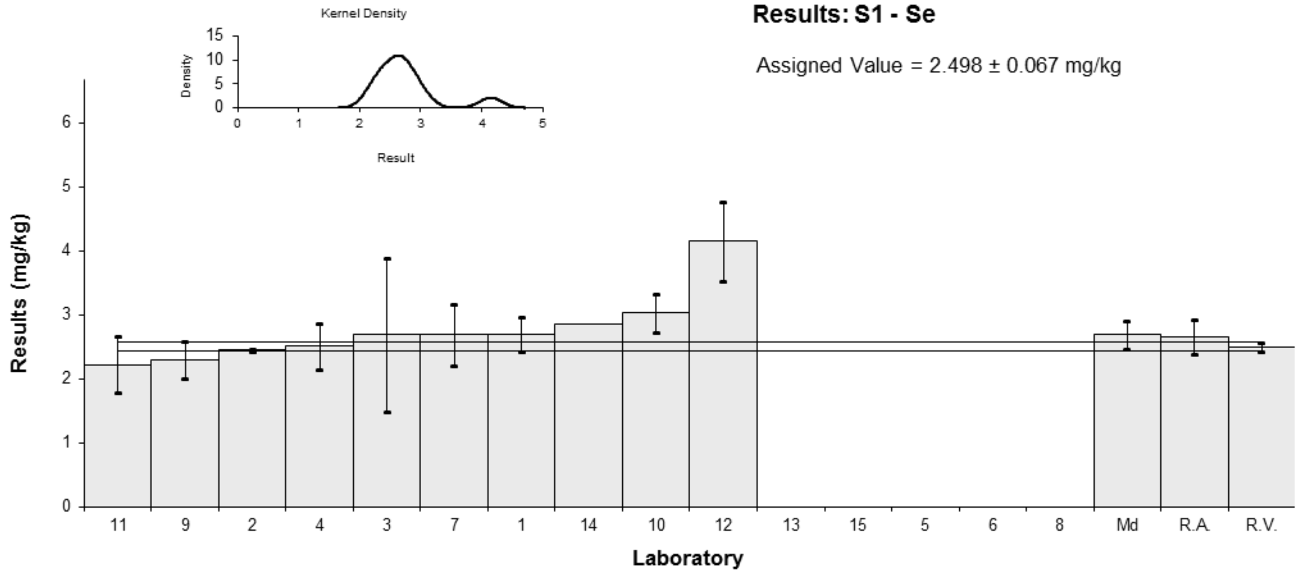
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	2.7	0.27	0.81	0.73
2	2.45	0.02	-0.19	-0.69
3	2.69	1.20	0.77	0.16
4	2.51	0.36	0.05	0.03
5	NT	NT		
6	NT	NT		
7	2.69	0.48	0.77	0.40
8	NT	NT		
9	2.3	0.29	-0.79	-0.67
10	3.03	0.30	2.13	1.73
11	2.22	0.437	-1.11	-0.63
12	4.15	0.62	6.61	2.65
13	<5	NR		
14	2.86	NR	1.45	5.40
15	NT	NT		

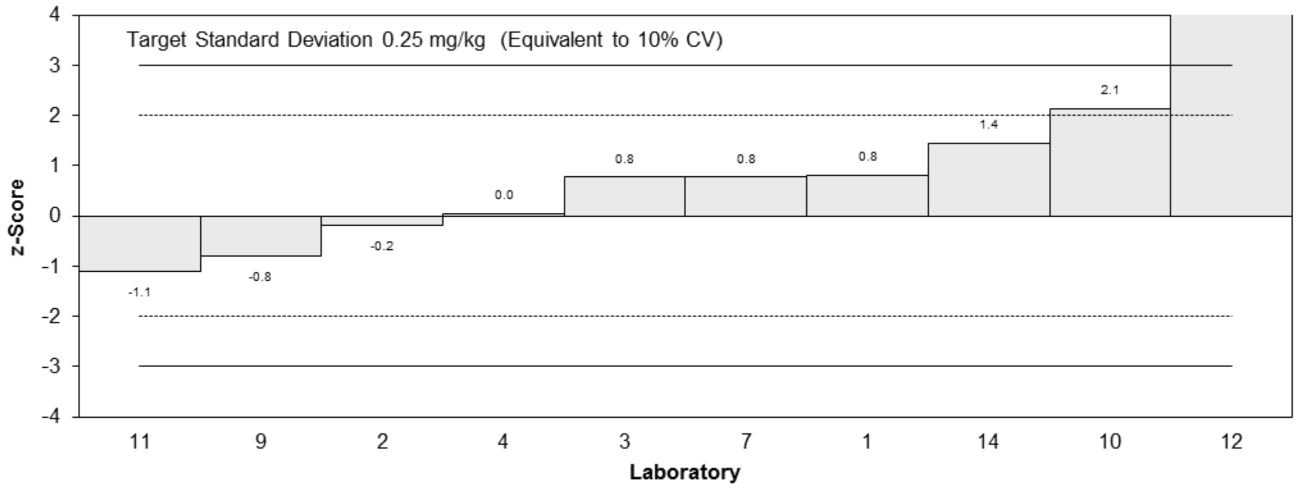
Statistics

Assigned Value*	2.498	0.067
Spike	Not Spiked	
Reference Value*	2.498	0.067
Robust Average	2.66	0.27
Median	2.69	0.22
Mean	2.76	
N	10	
Max.	4.15	
Min.	2.22	
Robust SD	0.3	
Robust CV	11%	

*Reference Value by SA-ICP-MS



z-Scores: S1 - Se



En-Scores: S1 - Se

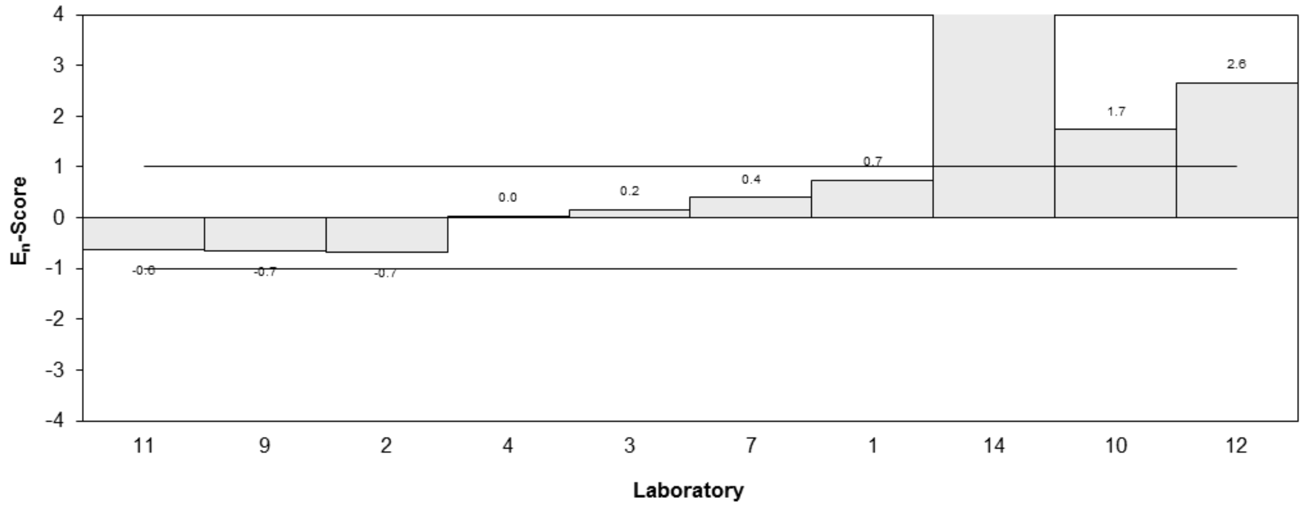


Figure 28

Table 34

Sample Details

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

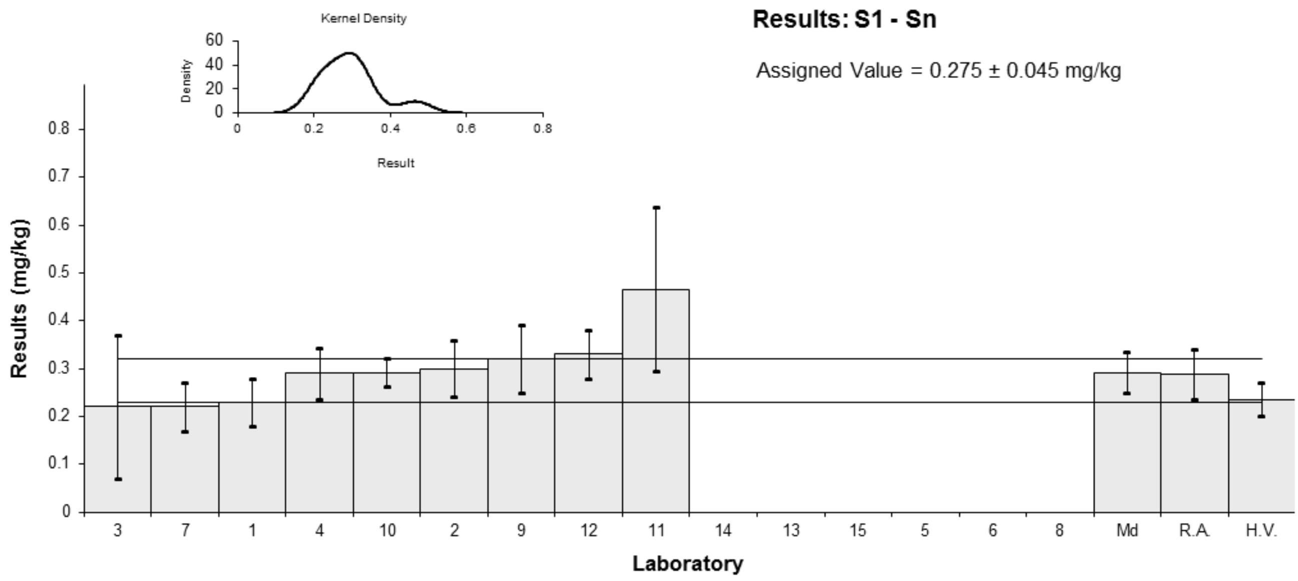
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.23	0.05	-0.82	-0.67
2	0.30	0.06	0.45	0.33
3	0.22	0.15	-1.00	-0.35
4	0.290	0.053	0.27	0.22
5	NT	NT		
6	NT	NT		
7	0.22	0.05	-1.00	-0.82
8	NT	NT		
9	0.32	0.07	0.82	0.54
10	0.292	0.029	0.31	0.32
11	0.466	0.172	3.47	1.07
12	0.33	0.05	1.00	0.82
13	NT	NT		
14	<0.5	NR		
15	NT	NT		

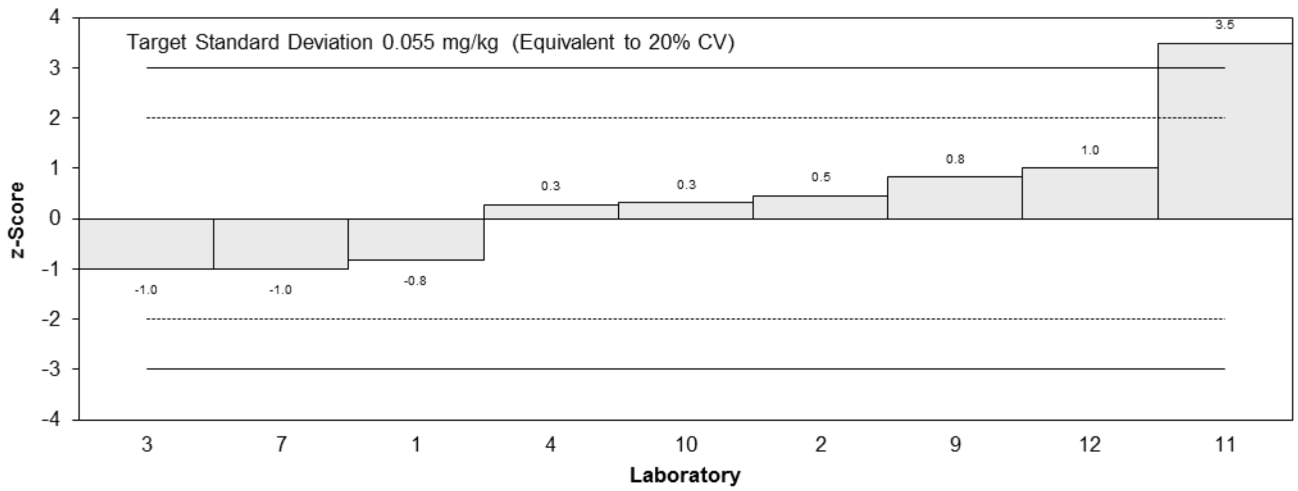
Statistics

Assigned Value*	0.275	0.045
Spike	Not Spiked	
Homogeneity Value	0.235	0.035
Robust Average	0.287	0.052
Median	0.292	0.043
Mean	0.296	
N	9	
Max.	0.466	
Min.	0.22	
Robust SD	0.051	
Robust CV	19%	

*Robust Average excluding Laboratory 11.



z-Scores: S1 - Sn



En-Scores: S1 - Sn

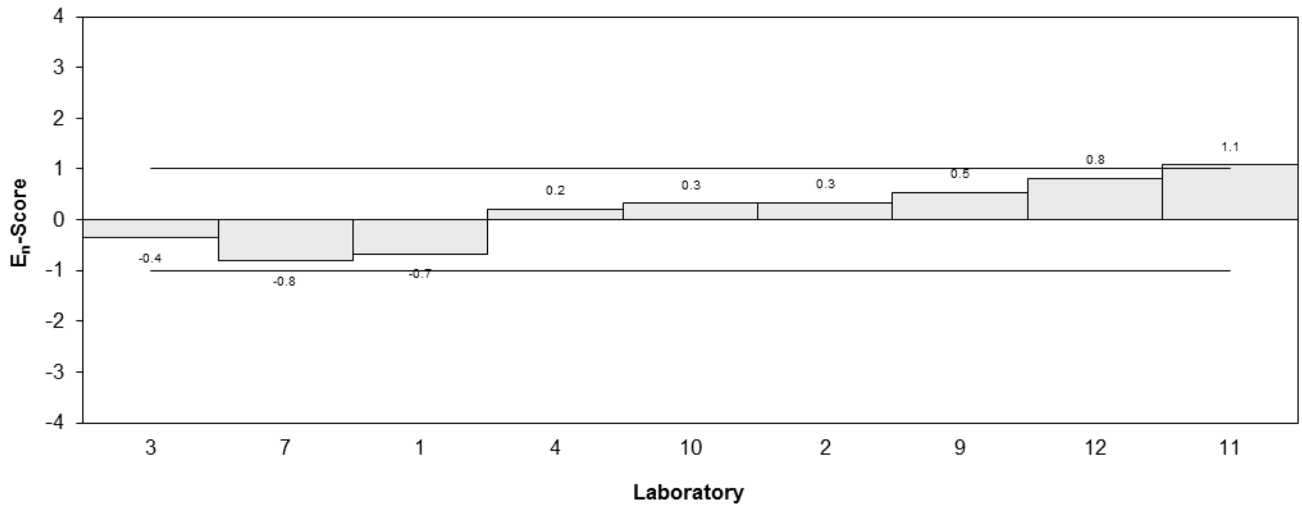


Figure 29

Table 35

Sample Details

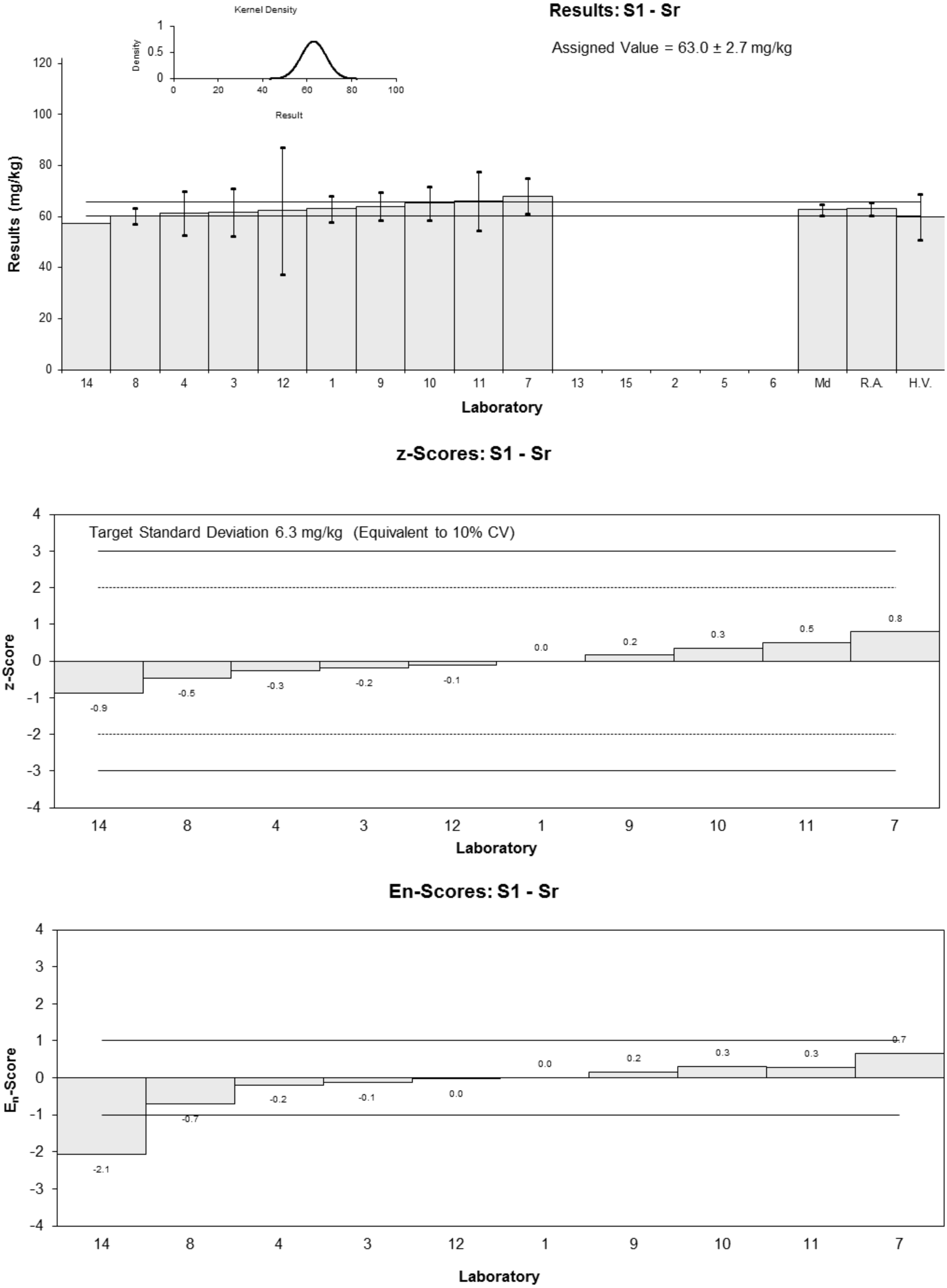
Sample No.	S1
Matrix.	Prawn
Analyte.	Sr
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	63	5.0	0.00	0.00
2	NT	NT		
3	61.8	9.3	-0.19	-0.12
4	61.3	8.6	-0.27	-0.19
5	NT	NT		
6	NT	NT		
7	68	7	0.79	0.67
8	60.1	3.1	-0.46	-0.71
9	64	5.5	0.16	0.16
10	65.2	6.52	0.35	0.31
11	66.2	11.5	0.51	0.27
12	62.3	24.9	-0.11	-0.03
13	NT	NT		
14	57.4	NR	-0.89	-2.07
15	NT	NT		

Statistics

Assigned Value	63.0	2.7
Spike	Not Spiked	
Homogeneity Value	59.8	9.0
Robust Average	63.0	2.7
Median	62.7	2.1
Mean	62.9	
N	10	
Max.	68	
Min.	57.4	
Robust SD	3.4	
Robust CV	5.4%	



Results: S1 - Sr
Assigned Value = 63.0 ± 2.7 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
14 8 4 3 12 1 9 10 11 7 13 15 2 5 6 Md R.A. H.V.
z-Scores: S1 - Sr
Target Standard Deviation 6.3 mg/kg (Equivalent to 10% CV)
z-Score
-0.9 -0.5 -0.3 -0.2 -0.1 0.0 0.2 0.3 0.5 0.8
Laboratory
14 8 4 3 12 1 9 10 11 7
En-Scores: S1 - Sr
En-Score
-2.1 -0.7 -0.2 -0.1 0.0 0.0 0.2 0.3 0.3 0.7
Laboratory
14 8 4 3 12 1 9 10 11 7

Figure 30

Table 36

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.006	0.002	-0.54	-0.34
2	NT	NT		
3	0.0067	0.0023	-0.01	-0.01
4	0.0066	0.0016	-0.09	-0.07
5	NT	NT		
6	NT	NT		
7	0.006	0.001	-0.54	-0.60
8	0.009	0.002	1.70	1.08
9	0.007	0.001	0.21	0.23
10	0.007	0.007	0.21	0.04
11	NT	NT		
12	<0.05	0.02		
13	NT	NT		
14	<0.01	NR		
15	NT	NT		

Statistics

Assigned Value	0.00672	0.00067
Spike	Not Spiked	
Homogeneity Value	0.00617	0.00093
Robust Average	0.00672	0.00067
Median	0.00670	0.00041
Mean	0.0069	
N	7	
Max.	0.009	
Min.	0.006	
Robust SD	0.0007	
Robust CV	10%	

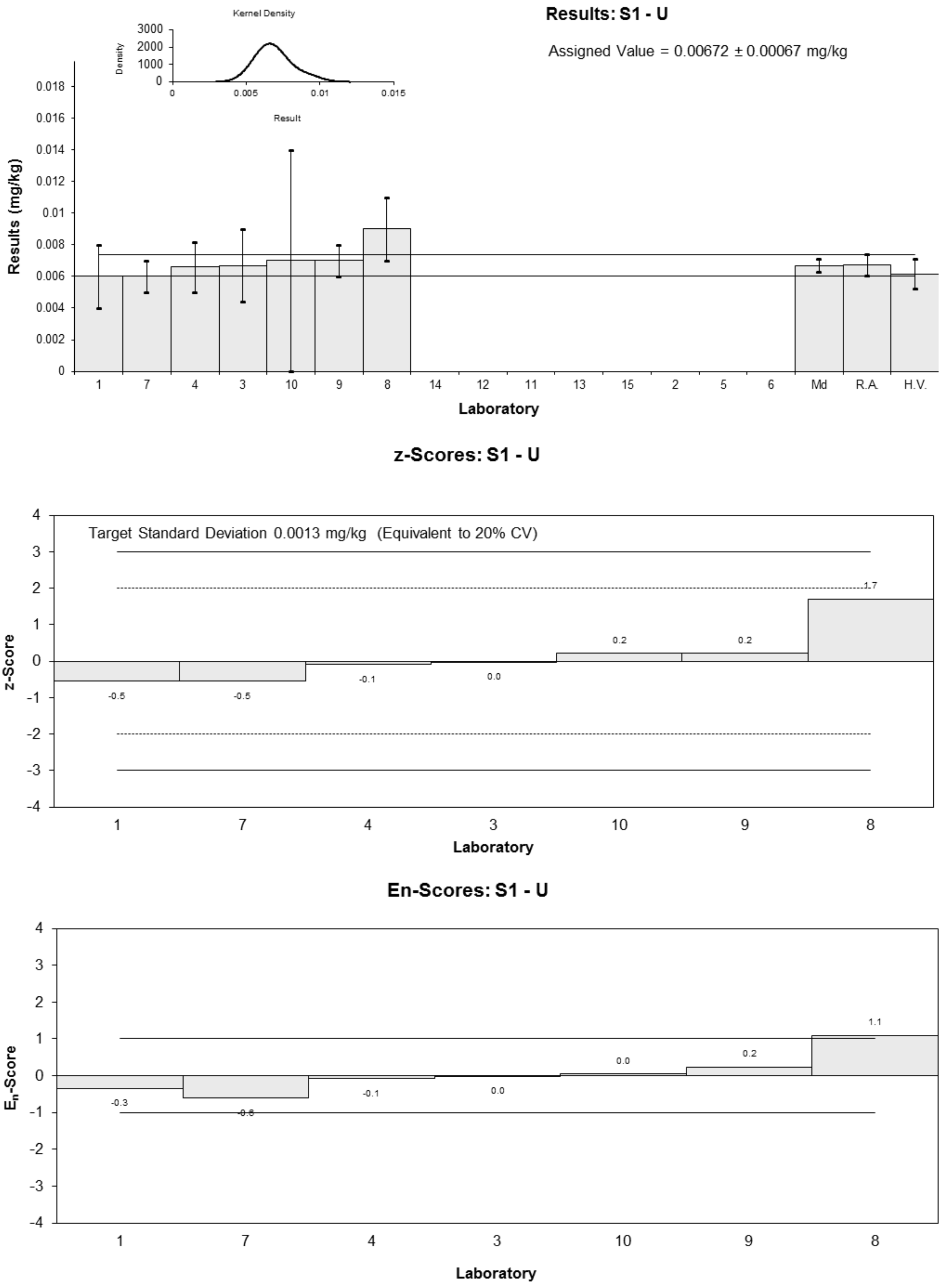

Results: S1 - U
Assigned Value = 0.00672 ± 0.00067 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
1 7 4 3 10 9 8 14 12 11 13 15 2 5 6 Md R.A. H.V.
z-Scores: S1 - U
Target Standard Deviation 0.0013 mg/kg (Equivalent to 20% CV)
z-Score
-0.5 -0.5 -0.1 0.0 0.2 0.2 1.7
1 7 4 3 10 9 8
Laboratory
En-Scores: S1 - U
En-Score
-0.3 -0.6 -0.1 0.0 0.0 0.2 1.1
1 7 4 3 10 9 8
Laboratory

Figure 31

Table 37

Sample Details

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

Participant Results

Lab Code	Result	Uncertainty
1	0.13	0.03
2	0.21	0.01
3	0.0604	0.017
4	0.095	0.036
5	NT	NT
6	NT	NT
7	0.09	0.03
8	0.65	0.06
9	0.19	0.03
10	0.115	0.012
11	0.258	0.051
12	0.22	0.09
13	<1	NR
14	<0.5	NR
15	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value*	0.252	0.021
Robust Average	0.167	0.073
Median	0.160	0.066
Mean	0.202	
N	10	
Max.	0.65	
Min.	0.060	
Robust SD	0.063	
Robust CV	55%	

*Information Value by SA-ICP-MS

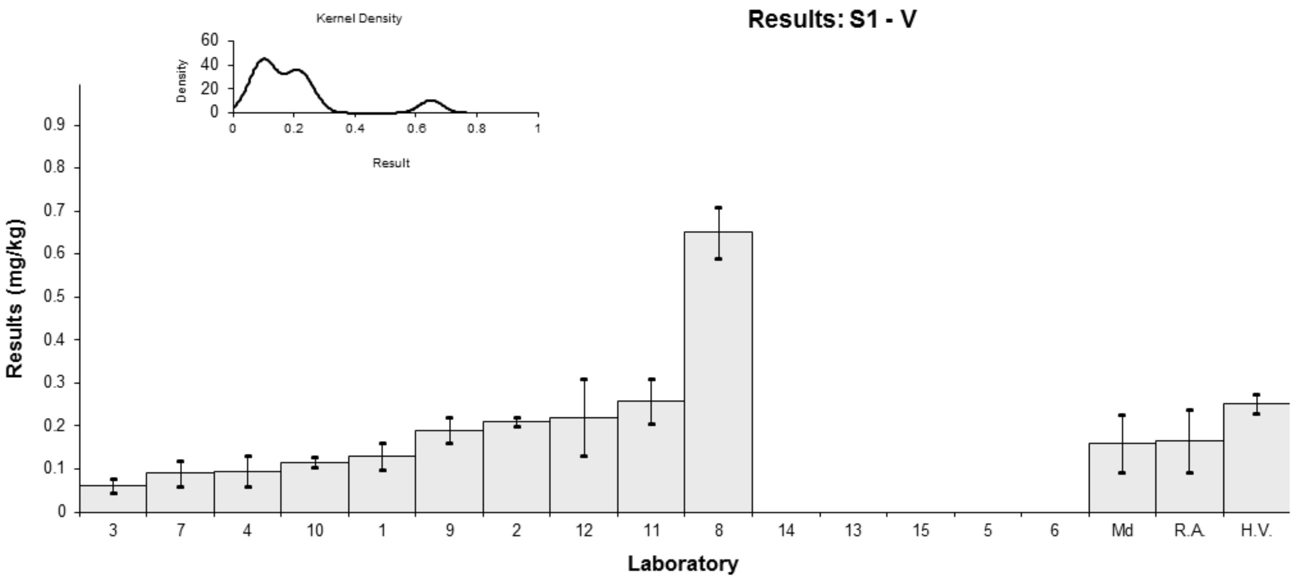

Results: S1 - V
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
3
7
4
10
1
9
2
12
11
8
14
13
15
5
6
Md
R.A.
H.V.

Figure 32

Table 38

Sample Details

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

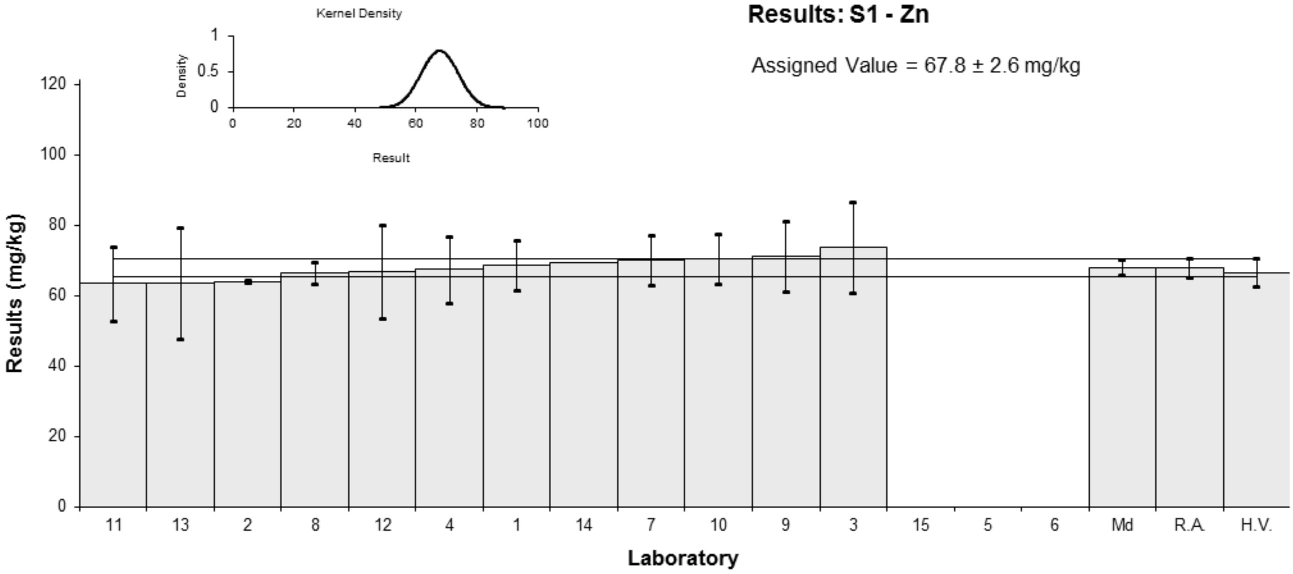
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	68.5	7.0	0.10	0.09
2	63.87	0.34	-0.58	-1.50
3	73.6	13	0.86	0.44
4	67.3	9.5	-0.07	-0.05
5	NT	NT		
6	NT	NT		
7	70	7	0.32	0.29
8	66.5	3.1	-0.19	-0.32
9	71	10	0.47	0.31
10	70.5	7.05	0.40	0.36
11	63.4	10.6	-0.65	-0.40
12	66.7	13.3	-0.16	-0.08
13	63.4	15.8	-0.65	-0.27
14	69.2	NR	0.21	0.54
15	NT	NT		

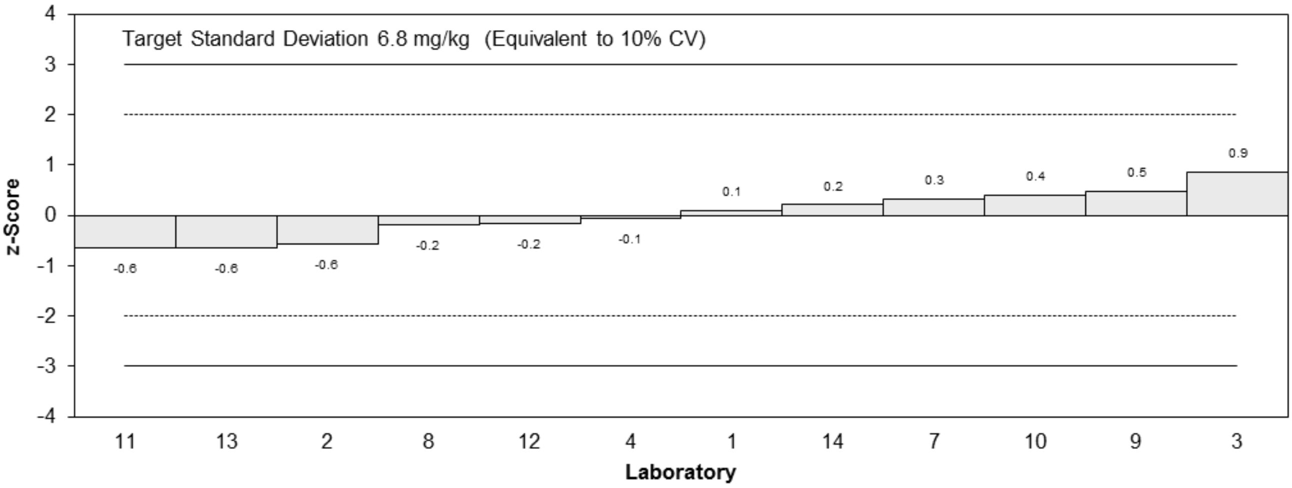
Statistics

Assigned Value	67.8	2.6
Spike	Not Spiked	
Homogeneity Value*	66.4	4.0
Robust Average	67.8	2.6
Median	67.9	2.2
Mean	67.8	
N	12	
Max.	73.6	
Min.	63.4	
Robust SD	3.6	
Robust CV	5.3%	

*Information Value by SA-ICP-MS



z-Scores: S1 - Zn



En-Scores: S1 - Zn

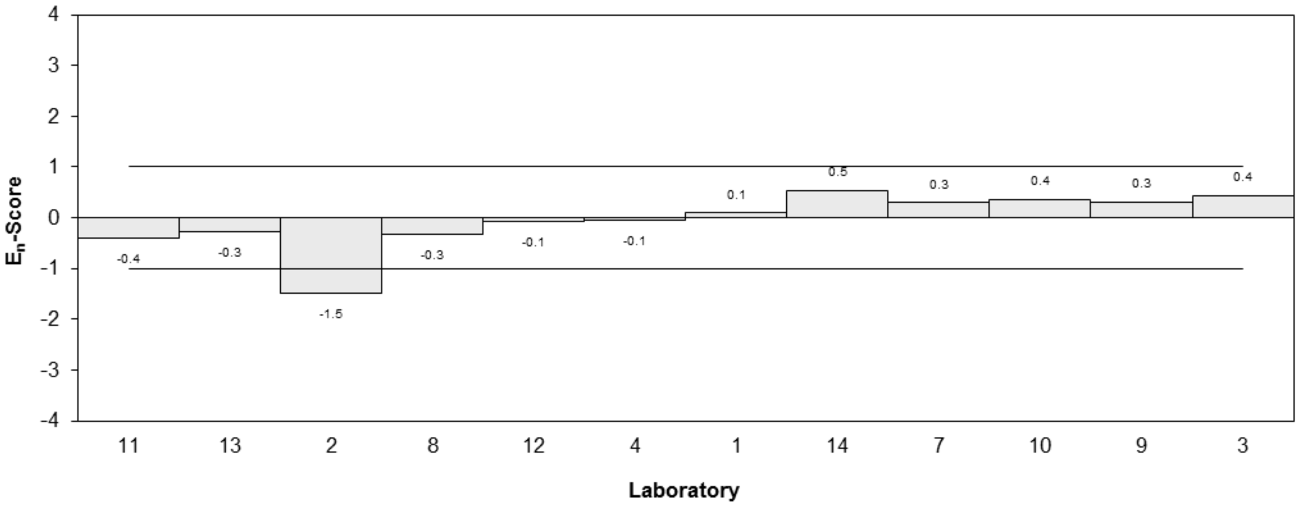


Figure 33

Table 39

Sample Details

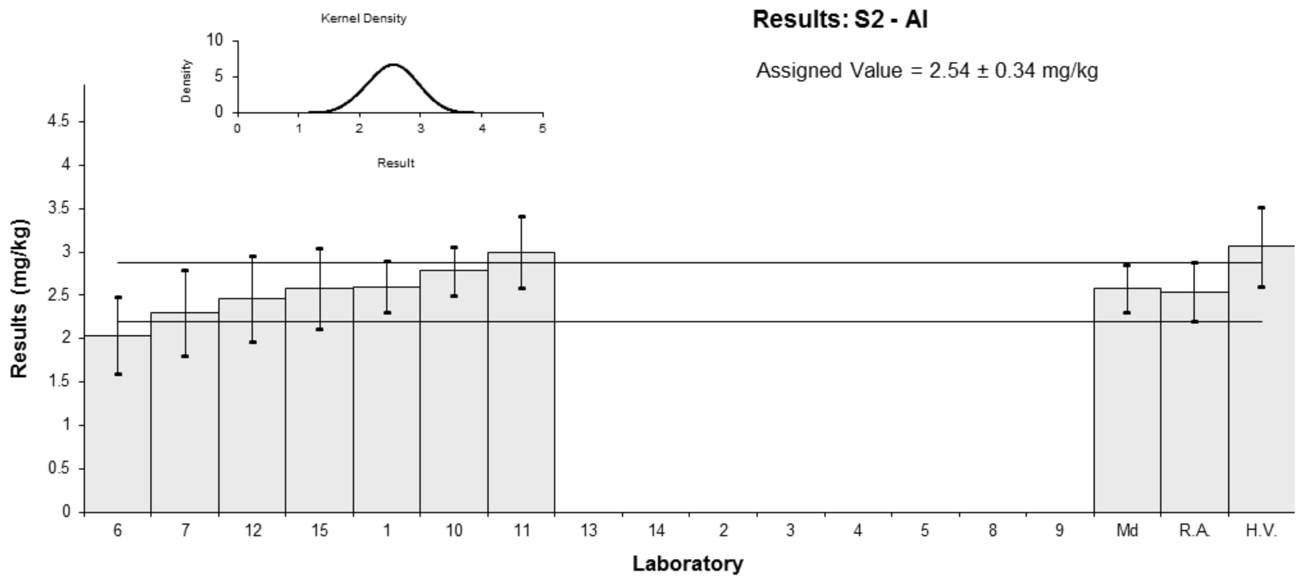
Sample No.	S2
Matrix.	Infant formula
Analyte.	Al
Units	mg/kg

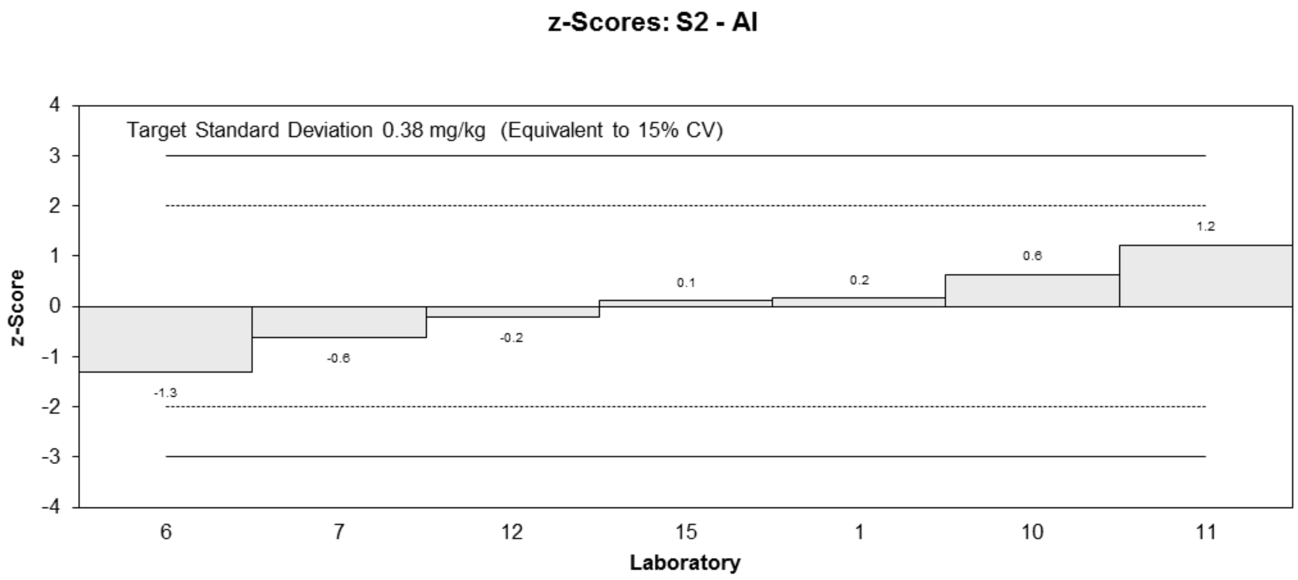
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	2.6	0.3	0.16	0.13
2	NT	NT		
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	2.04	0.45	-1.31	-0.89
7	2.3	0.5	-0.63	-0.40
8	NT	NT		
9	NT	NT		
10	2.78	0.28	0.63	0.54
11	3.00	0.408	1.21	0.87
12	2.46	0.492	-0.21	-0.13
13	NT	NT		
14	NT	NT		
15	2.58	0.47	0.10	0.07

Statistics

Assigned Value	2.54	0.34
Spike	Not Spiked	
Homogeneity Value	3.06	0.46
Robust Average	2.54	0.34
Median	2.58	0.27
Mean	2.54	
N	7	
Max.	3	
Min.	2.04	
Robust SD	0.36	
Robust CV	14%	


Results: S2 - Al
Assigned Value = 2.54 ± 0.34 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
6
7
12
15
1
10
11
13
14
2
3
4
5
8
9
Md
R.A.
H.V.


z-Scores: S2 - Al
Target Standard Deviation 0.38 mg/kg (Equivalent to 15% CV)
z-Score
Laboratory
-1.3
-0.6
-0.2
0.1
0.2
0.6
1.2
6
7
12
15
1
10
11

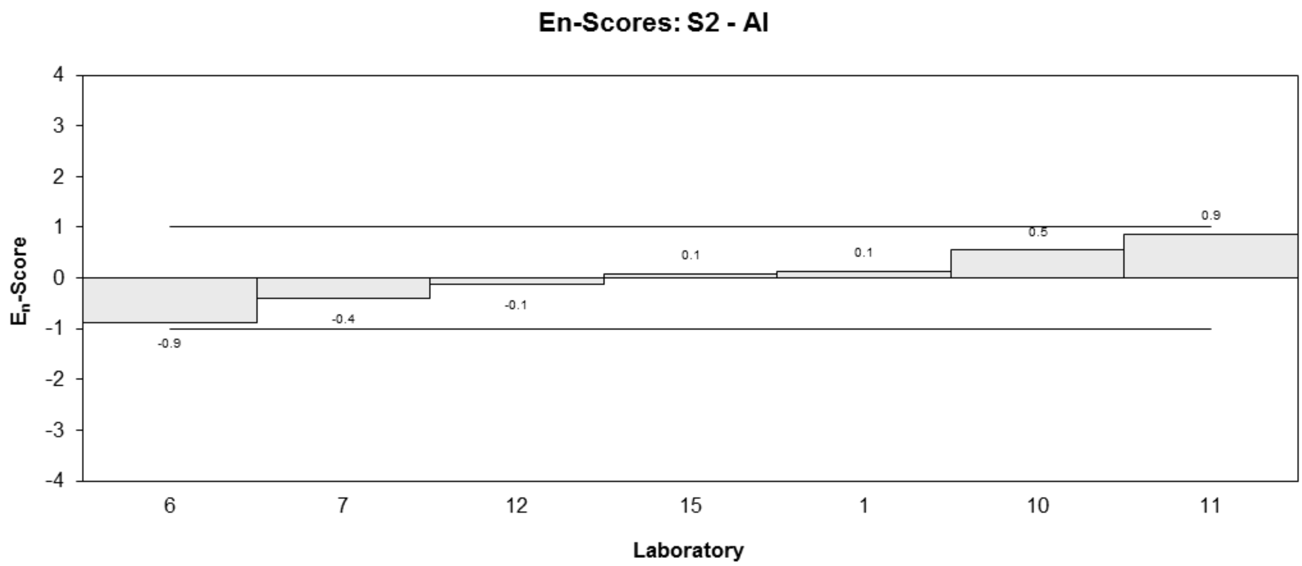

En-Scores: S2 - Al
E_n-Score
Laboratory
-0.9
-0.4
-0.1
0.1
0.1
0.5
0.9
6
7
12
15
1
10
11

Figure 34

Table 40

Sample Details

Sample No.	S2
Matrix.	Infant formula
Analyte.	As
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	0.005	0.0005
2	<0.10	NR
3	NT	NT
4	NT	NT
5	NT	NT
6	<0.01	NR
7	<0.05	NR
8	NT	NT
9	NT	NT
10	0.006	0.006
11	<0.10	NR
12	<0.025	0.005
13	NT	NT
14	NT	NT
15	<0.01	0.006

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.0060	0.0009
Mean	0.0055	

*Insufficient data to calculate statistics

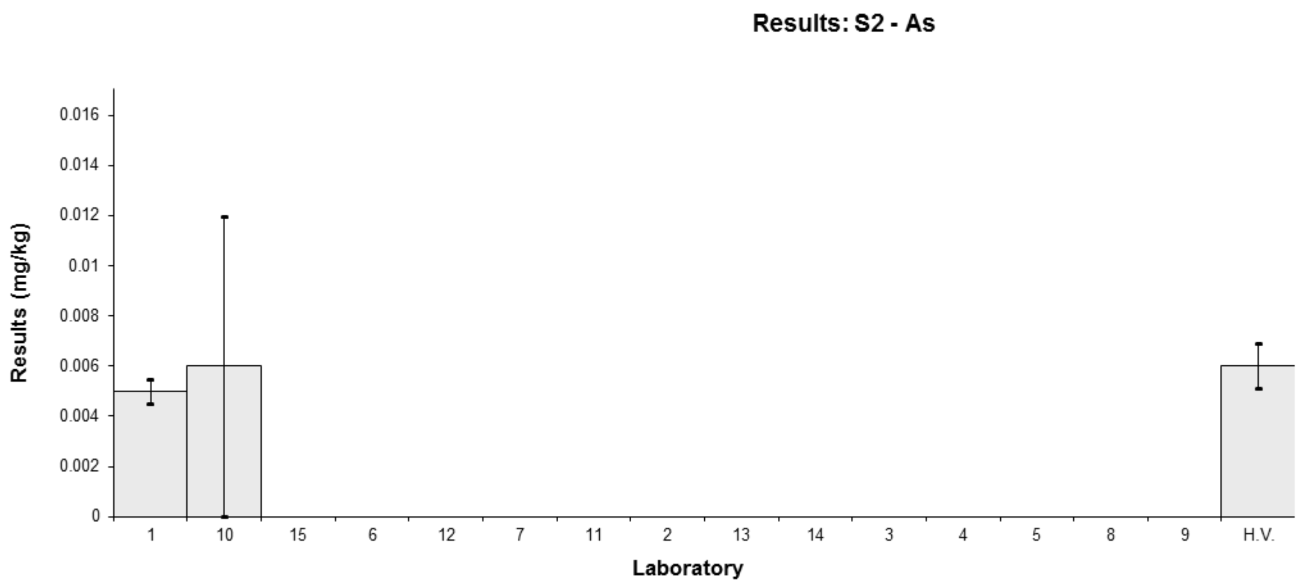

Results: S2 - As
Results (mg/kg)
0.016
0.014
0.012
0.01
0.008
0.006
0.004
0.002
0
1
10
15
6
12
7
11
2
13
14
3
4
5
8
9
H.V.
Laboratory

Figure 35

Table 41

Sample Details

Sample No.	S2
Matrix.	Infant formula
Analyte.	B
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	1.4	0.14
2	NT	NT
3	NT	NT
4	NT	NT
5	NT	NT
6	0.81	0.15
7	<1	NR
8	NT	NT
9	NT	NT
10	1.07	0.11
11	0.815	0.134
12	1.3	0.325
13	NT	NT
14	NT	NT
15	NT	NT

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	1.10	0.17
Robust Average	1.08	0.34
Median	1.07	0.47
Mean	1.08	
N	5	
Max.	1.4	
Min.	0.81	
Robust SD	0.31	
Robust CV	28%	

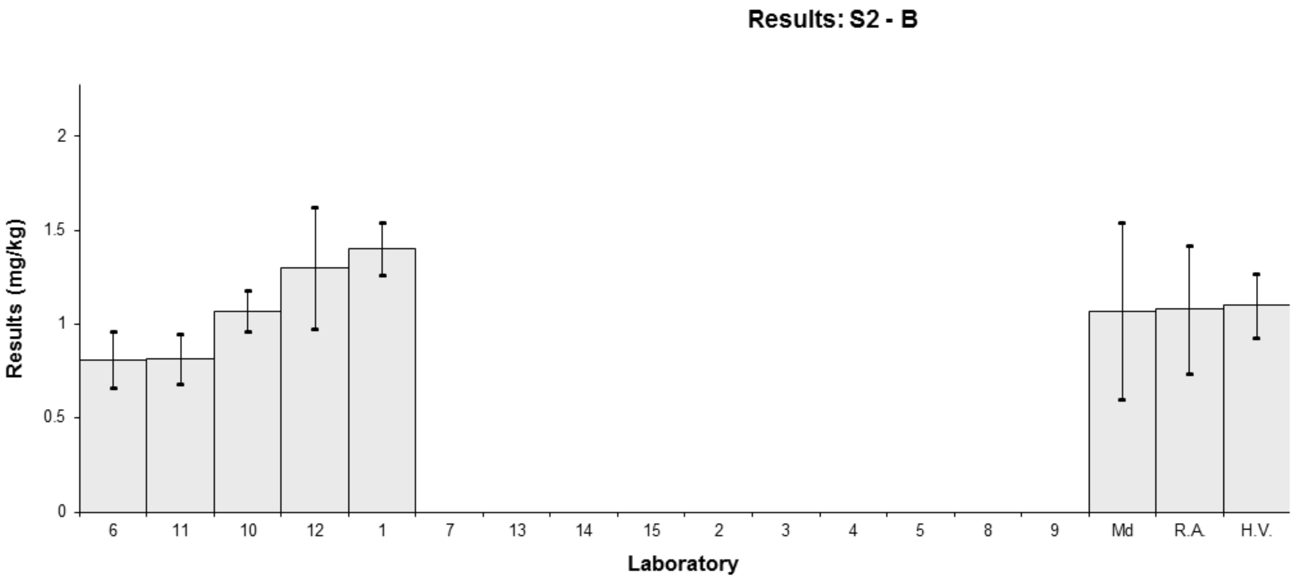

Results: S2 - B
Results (mg/kg)
2
1.5
1
0.5
0
6
11
10
12
1
7
13
14
15
2
3
4
5
8
9
Md
R.A.
H.V.
Laboratory

Figure 36

Table 42

Sample Details

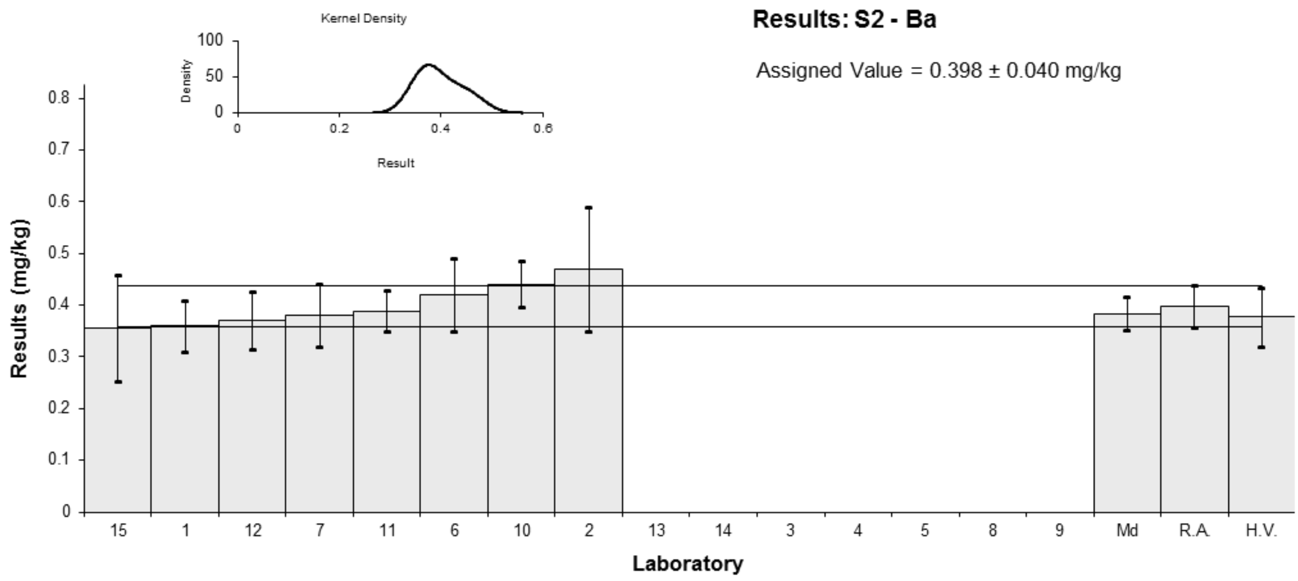
Sample No.	S2
Matrix.	Infant formula
Analyte.	Ba
Units	mg/kg

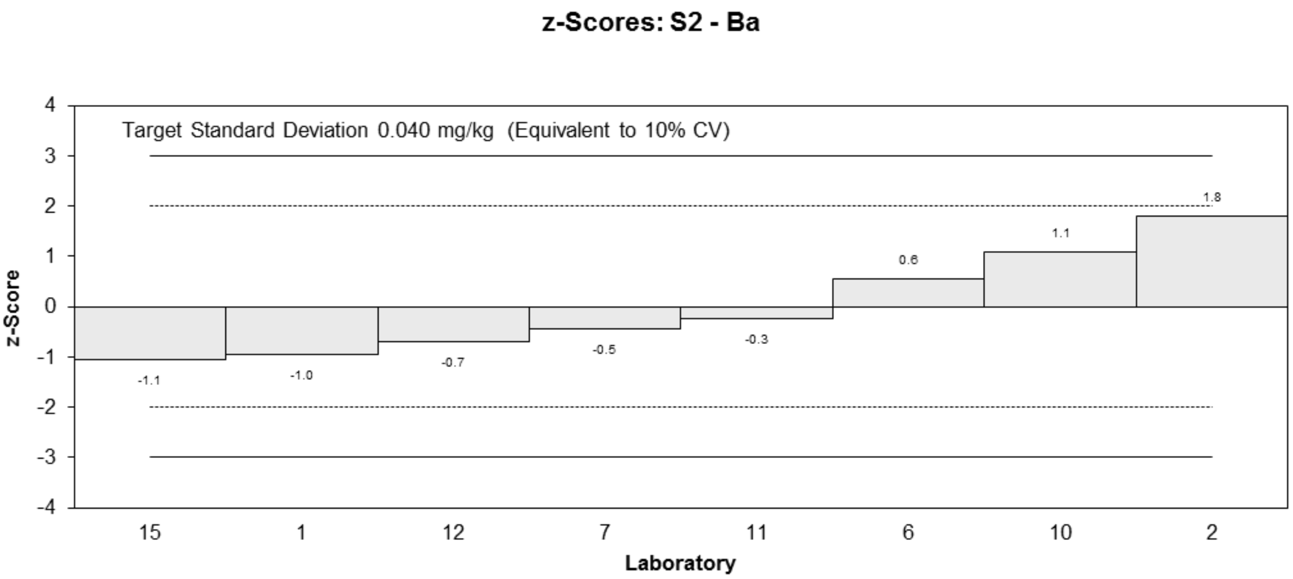
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.36	0.05	-0.95	-0.59
2	0.47	0.12	1.81	0.57
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	0.42	0.07	0.55	0.27
7	0.38	0.06	-0.45	-0.25
8	NT	NT		
9	NT	NT		
10	0.441	0.044	1.08	0.72
11	0.388	0.0396	-0.25	-0.18
12	0.37	0.056	-0.70	-0.41
13	<1	NR		
14	NT	NT		
15	0.356	0.102	-1.06	-0.38

Statistics

Assigned Value	0.398	0.040
Spike	Not Spiked	
Homogeneity Value	0.377	0.057
Robust Average	0.398	0.040
Median	0.384	0.032
Mean	0.398	
N	8	
Max.	0.47	
Min.	0.356	
Robust SD	0.046	
Robust CV	12%	


Results: S2 - Ba
Assigned Value = 0.398 ± 0.040 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
15
1
12
7
11
6
10
2
13
14
3
4
5
8
9
Md
R.A.
H.V.


z-Scores: S2 - Ba
Target Standard Deviation 0.040 mg/kg (Equivalent to 10% CV)
z-Score
-1.1
-1.0
-0.7
-0.5
-0.3
0.6
1.1
1.8
15
1
12
7
11
6
10
2
Laboratory

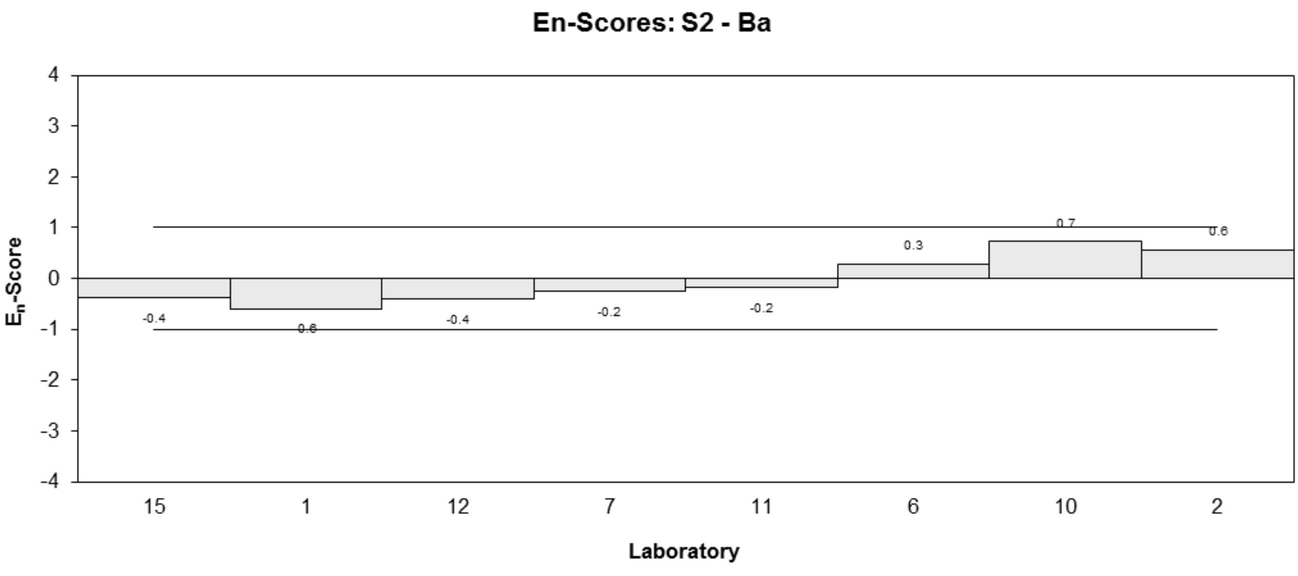

En-Scores: S2 - Ba
En-Score
-0.4
-0.6
-0.4
-0.2
-0.2
0.3
0.7
0.6
15
1
12
7
11
6
10
2
Laboratory

Figure 37

Table 43

Sample Details

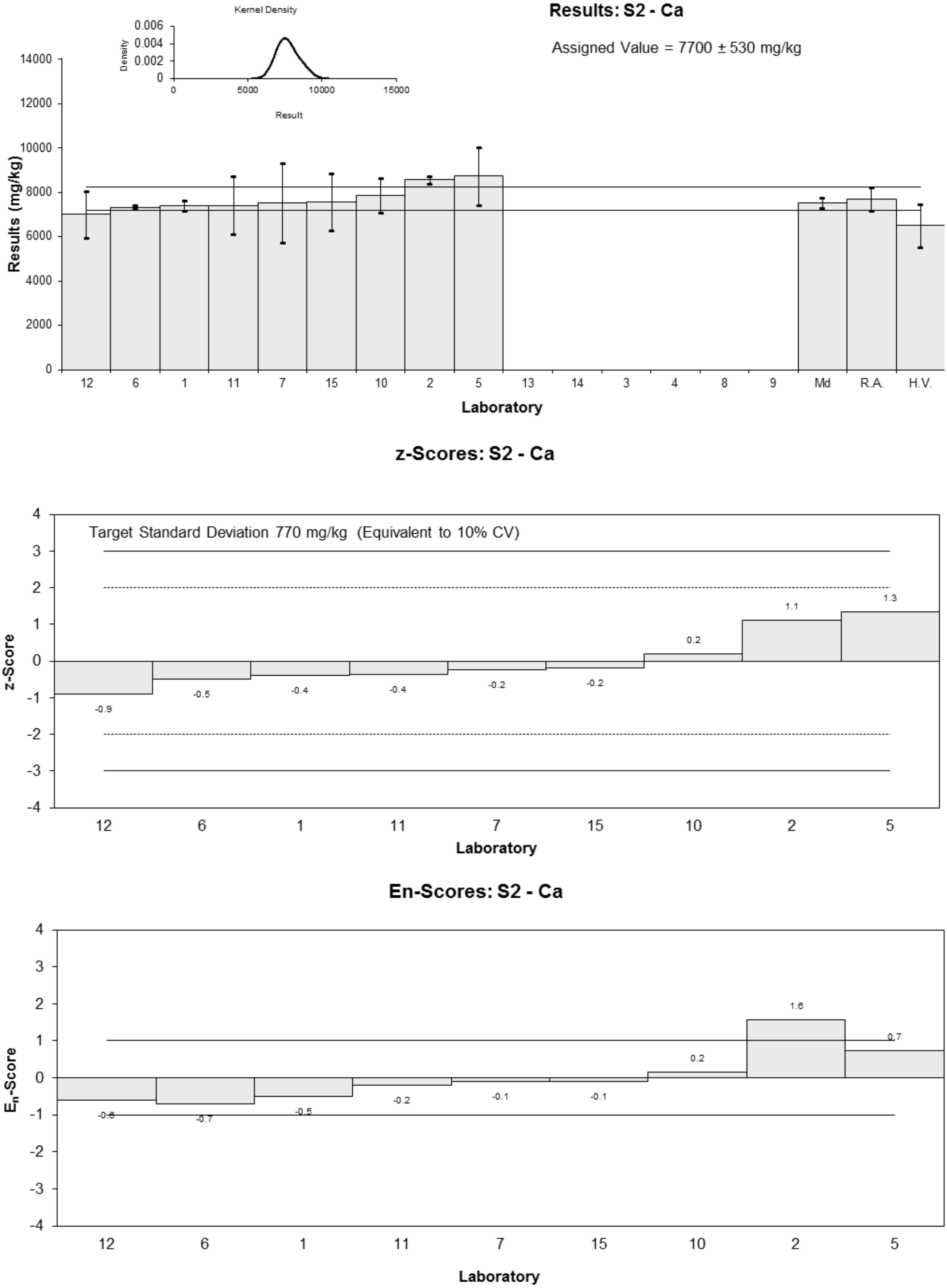
Sample No.	S2
Matrix.	Infant formula
Analyte.	Ca
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	7400	250	-0.39	-0.51
2	8564	154	1.12	1.57
3	NT	NT		
4	NT	NT		
5	8730	1310	1.34	0.73
6	7320	86	-0.49	-0.71
7	7520	1783	-0.23	-0.10
8	NT	NT		
9	NT	NT		
10	7850	785	0.19	0.16
11	7420	1290	-0.36	-0.20
12	7000	1050	-0.91	-0.60
13	NT	NT		
14	NT	NT		
15	7560	1290	-0.18	-0.10

Statistics

Assigned Value	7700	530
Spike	Not Spiked	
Homogeneity Value	6500	970
Robust Average	7700	530
Median	7520	230
Mean	7707	
N	9	
Max.	8730	
Min.	7000	
Robust SD	640	
Robust CV	8.3%	



Results: S2 - Ca
Assigned Value = 7700 ± 530 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
12
6
1
11
7
15
10
2
5
13
14
3
4
8
9
Md
R.A.
H.V.
z-Scores: S2 - Ca
Target Standard Deviation 770 mg/kg (Equivalent to 10% CV)
z-Score
-0.9
-0.5
-0.4
-0.4
-0.2
-0.2
0.2
1.1
1.3
En-Scores: S2 - Ca
En-Score
-0.6
-0.7
-0.5
-0.2
-0.1
-0.1
0.2
1.6
0.7

Figure 38

Table 44

Sample Details

Sample No.	S2
Matrix.	Infant formula
Analyte.	Cd
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	<0.005	NR
2	<0.01	NR
3	NT	NT
4	NT	NT
5	NT	NT
6	<0.01	NR
7	<0.01	NR
8	NT	NT
9	NT	NT
10	0.0014	0.002
11	<0.010	NR
12	<0.01	0.002
13	NT	NT
14	NT	NT
15	0.00150	0.0010

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.00200	0.00030
Mean	0.00145	0.00067

*Insufficient data to calculate statistics

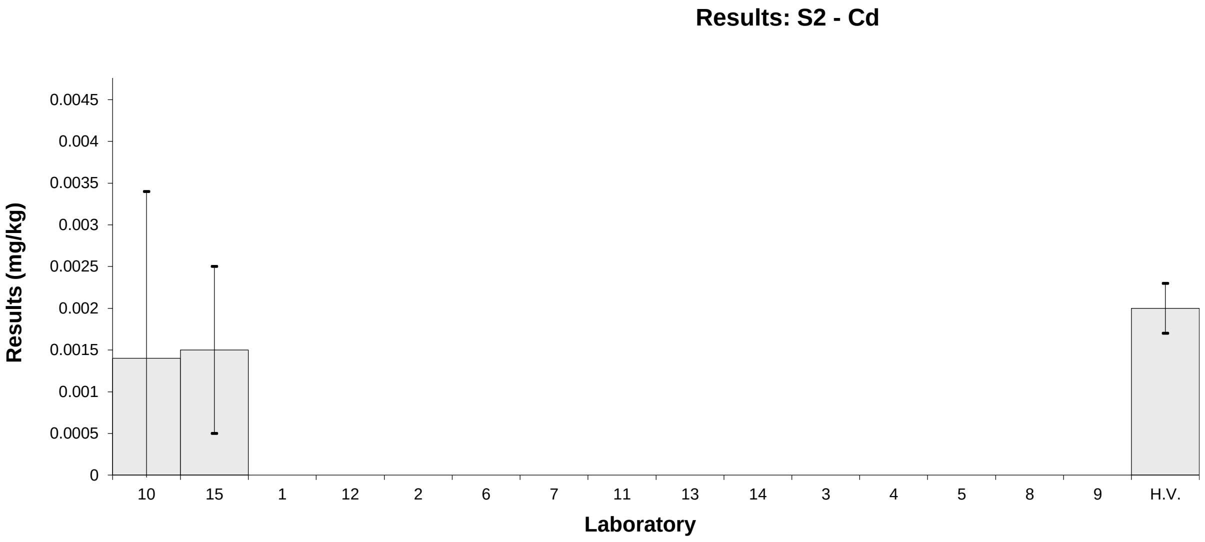

Results: S2 - Cd
Results (mg/kg)
0.0045
0.004
0.0035
0.003
0.0025
0.002
0.0015
0.001
0.0005
0
10
15
1
12
2
6
7
11
13
14
3
4
5
8
9
H.V.
Laboratory

Figure 39

Table 45

Sample Details

Sample No.	S2
Matrix.	Infant formula
Analyte.	Co
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	<0.01	NR
2	<0.05	NR
3	NT	NT
4	NT	NT
5	NT	NT
6	<0.01	NR
7	<0.02	NR
8	NT	NT
9	NT	NT
10	0.007	0.007
11	<0.010	NR
12	0.01	0.002
13	NT	NT
14	NT	NT
15	0.00806	0.0030

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.0080	0.0012
Robust Average	0.0084	0.0025
Median	0.0081	0.0039
Mean	0.0084	
N	3	
Max.	0.01	
Min.	0.007	
Robust SD	0.0017	
Robust CV	21%	

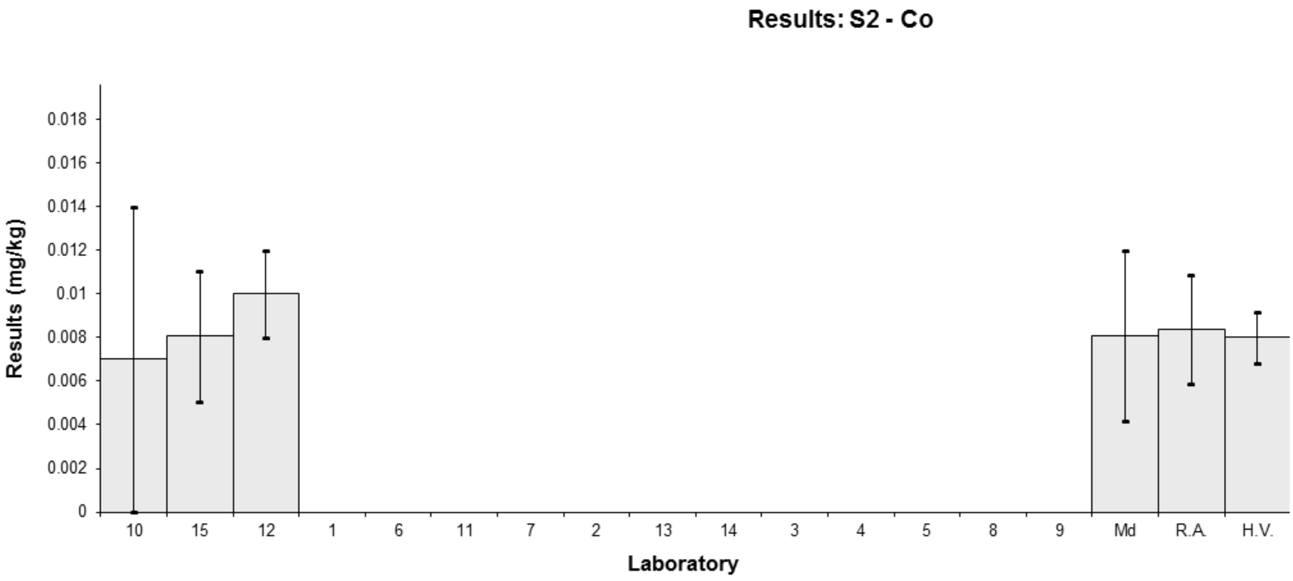

Results: S2 - Co
Results (mg/kg)
0.018
0.016
0.014
0.012
0.01
0.008
0.006
0.004
0.002
0
10
15
12
1
6
11
7
2
13
14
3
4
5
8
9
Md
R.A.
H.V.
Laboratory

Figure 40

Table 46

Sample Details

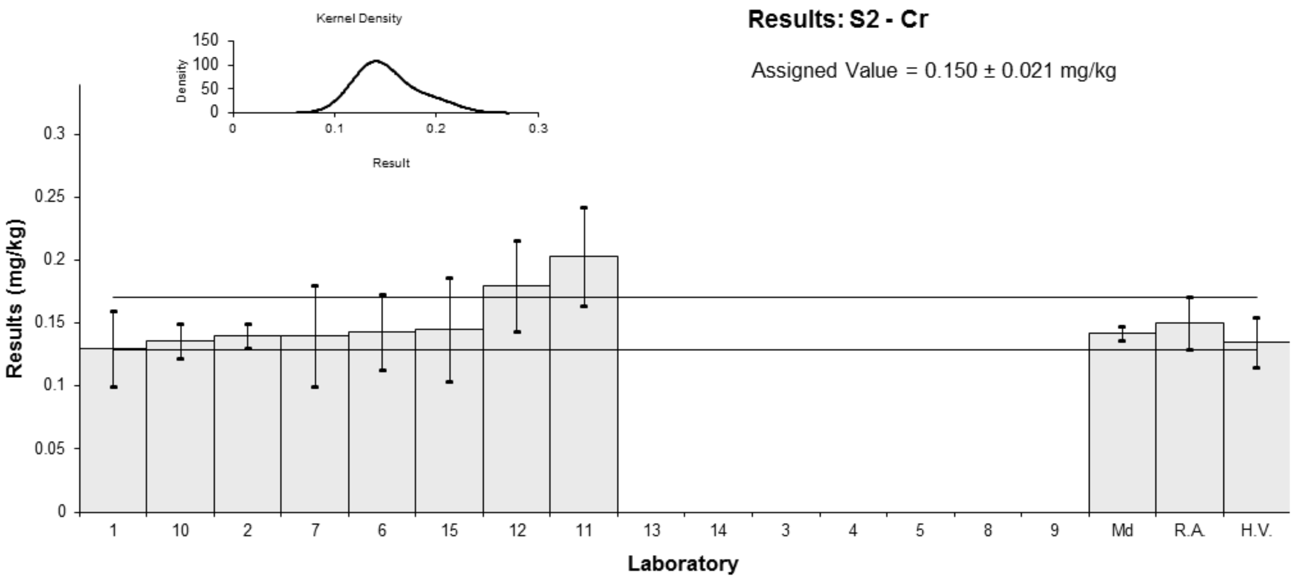
Sample No.	S2
Matrix.	Infant formula
Analyte.	Cr
Units	mg/kg

Participant Results

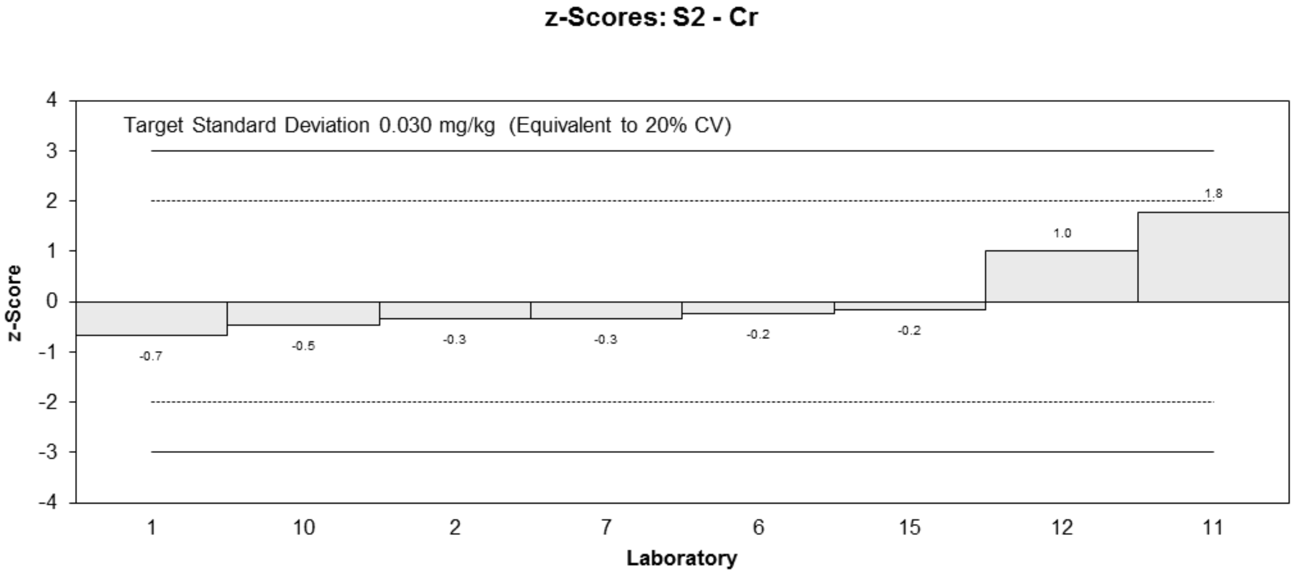
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.13	0.03	-0.67	-0.55
2	0.14	0.01	-0.33	-0.43
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	0.143	0.03	-0.23	-0.19
7	0.14	0.04	-0.33	-0.22
8	NT	NT		
9	NT	NT		
10	0.136	0.014	-0.47	-0.55
11	0.203	0.0394	1.77	1.19
12	0.18	0.036	1.00	0.72
13	<1	NR		
14	NT	NT		
15	0.145	0.0413	-0.17	-0.11

Statistics

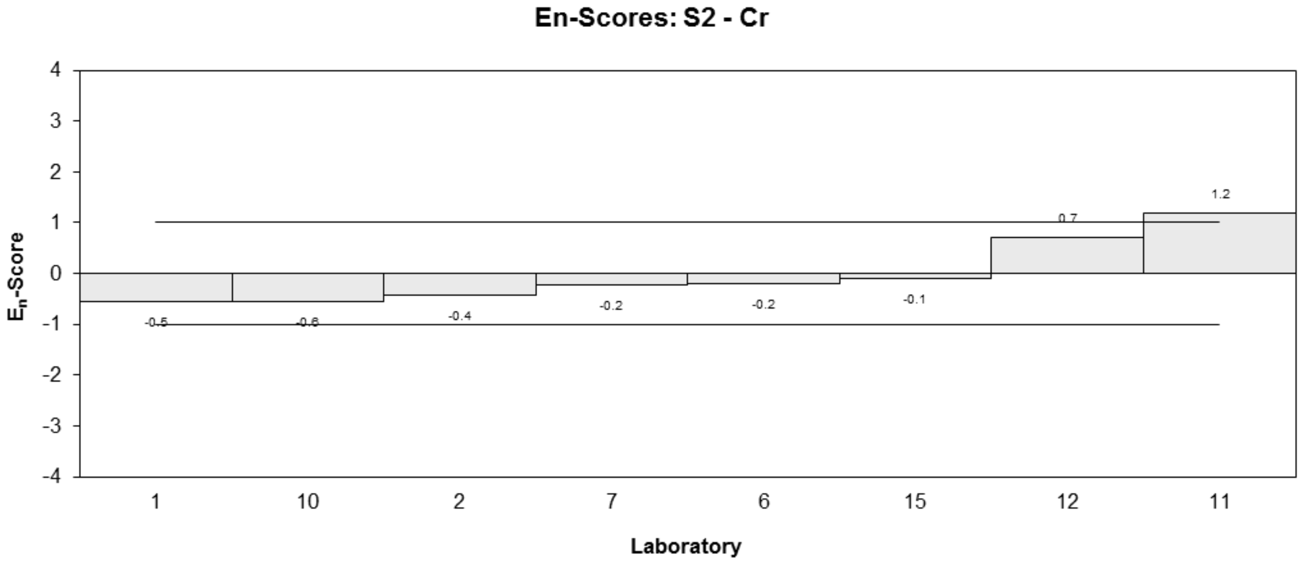
Assigned Value	0.150	0.021
Spike	Not Spiked	
Homogeneity Value	0.135	0.020
Robust Average	0.150	0.021
Median	0.142	0.006
Mean	0.152	
N	8	
Max.	0.203	
Min.	0.13	
Robust SD	0.023	
Robust CV	16%	



Results: S2 - Cr
Assigned Value = 0.150 ± 0.021 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
1 10 2 7 6 15 12 11 13 14 3 4 5 8 9 Md R.A. H.V.



z-Scores: S2 - Cr
Target Standard Deviation 0.030 mg/kg (Equivalent to 20% CV)
z-Score
Laboratory
1 10 2 7 6 15 12 11
-0.7 -0.5 -0.3 -0.3 -0.2 -0.2 1.0 1.8



En-Scores: S2 - Cr
E_n-Score
Laboratory
1 10 2 7 6 15 12 11
-0.5 -0.6 -0.4 -0.2 -0.2 -0.1 0.7 1.2

Figure 41

Table 47

Sample Details

Sample No.	S2
Matrix.	Infant formula
Analyte.	Cu
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	3.6	0.4	-0.22	-0.21
2	4.64	0.04	1.65	2.18
3	NT	NT		
4	NT	NT		
5	4.54	0.68	1.47	1.03
6	3.2	0.61	-0.93	-0.70
7	3.76	0.45	0.07	0.06
8	NT	NT		
9	NT	NT		
10	3.87	0.39	0.27	0.26
11	3.34	0.564	-0.68	-0.54
12	3.27	0.654	-0.81	-0.58
13	3.6	0.9	-0.22	-0.12
14	NT	NT		
15	3.51	0.70	-0.38	-0.26

Statistics

Assigned Value	3.72	0.42
Spike	Not Spiked	
Homogeneity Value	3.53	0.53
Robust Average	3.72	0.42
Median	3.60	0.28
Mean	3.73	
N	10	
Max.	4.64	
Min.	3.2	
Robust SD	0.53	
Robust CV	14%	

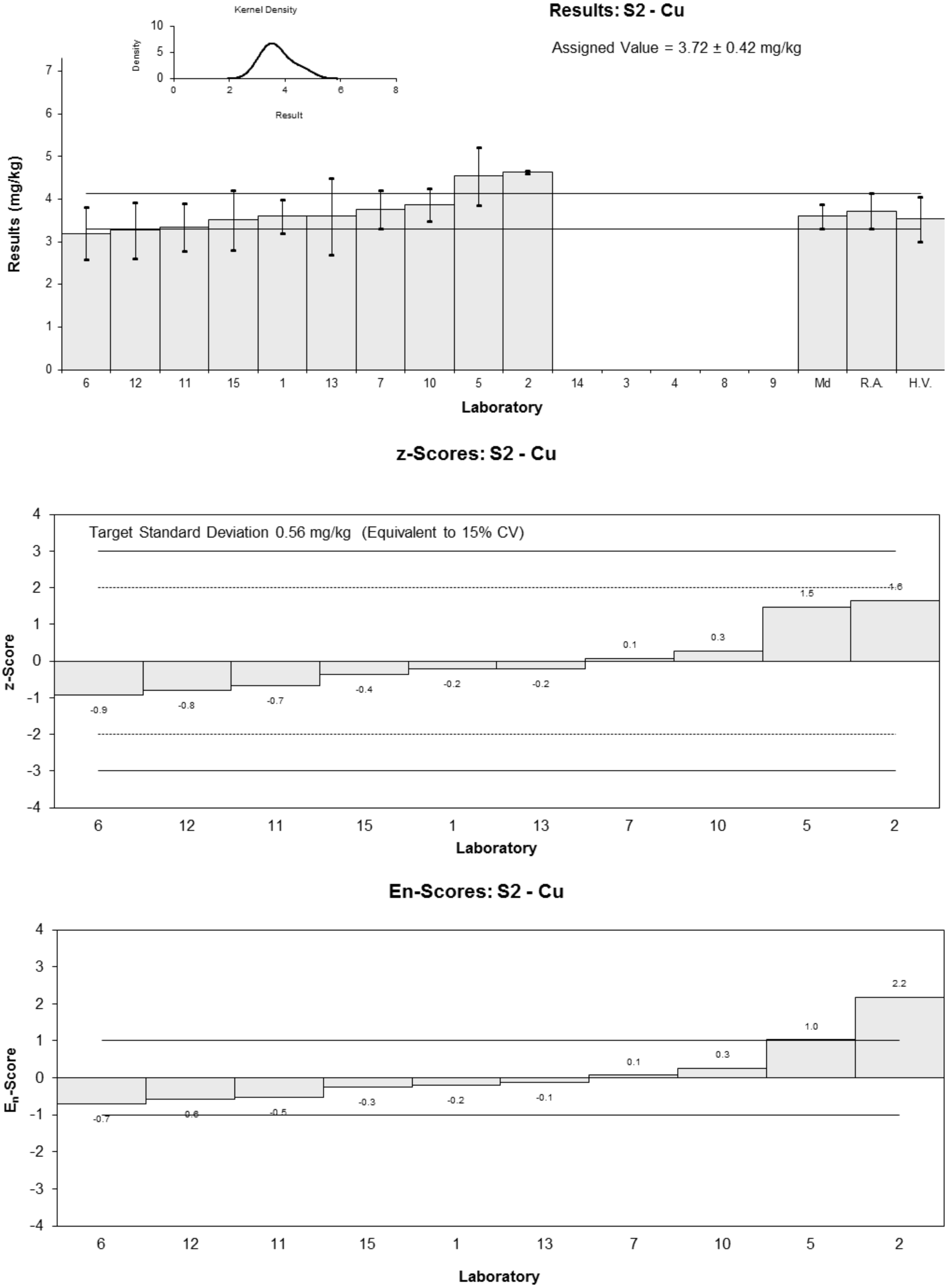

Results: S2 - Cu
Assigned Value = 3.72 ± 0.42 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
6
12
11
15
1
13
7
10
5
2
14
3
4
8
9
Md
R.A.
H.V.
z-Scores: S2 - Cu
Target Standard Deviation 0.56 mg/kg (Equivalent to 15% CV)
z-Score
-0.9
-0.8
-0.7
-0.4
-0.2
-0.2
0.1
0.3
1.5
1.6
Laboratory
En-Scores: S2 - Cu
En-Score
-0.7
-0.6
-0.5
-0.3
-0.2
-0.1
0.1
0.3
1.0
2.2
Laboratory

Figure 42

Table 48

Sample Details

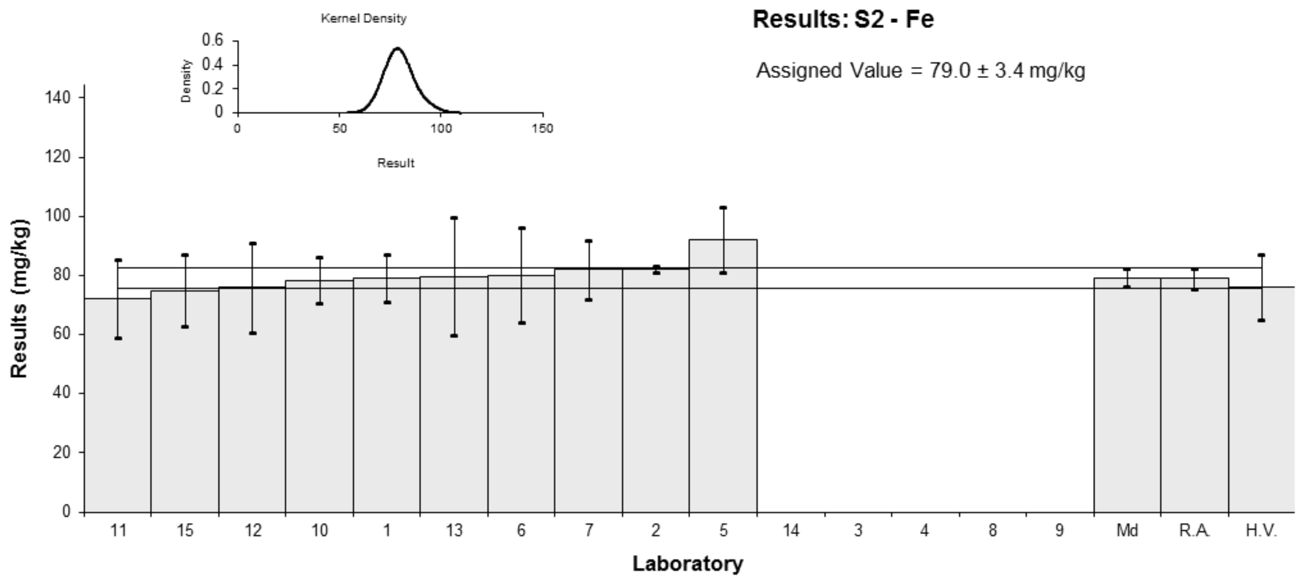
Sample No.	S2
Matrix.	Infant formula
Analyte.	Fe
Units	mg/kg

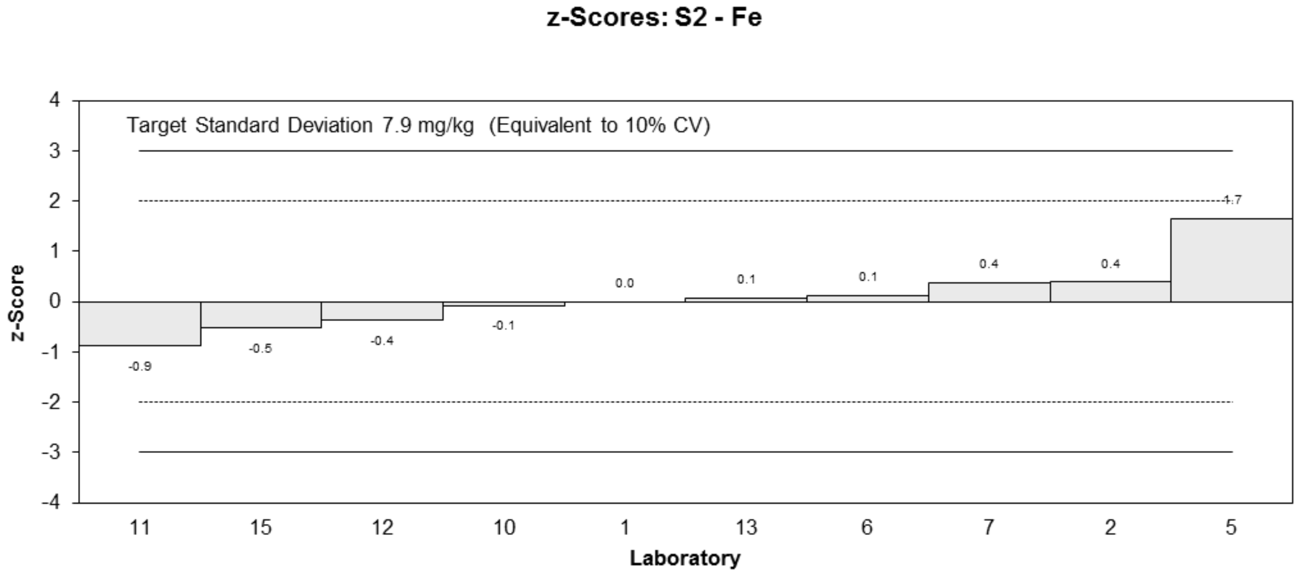
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	79	8.0	0.00	0.00
2	82.04	1.06	0.38	0.85
3	NT	NT		
4	NT	NT		
5	92.1	11.1	1.66	1.13
6	80	16	0.13	0.06
7	82	10	0.38	0.28
8	NT	NT		
9	NT	NT		
10	78.4	7.84	-0.08	-0.07
11	72.0	13.2	-0.89	-0.51
12	76	15.2	-0.38	-0.19
13	79.6	19.9	0.08	0.03
14	NT	NT		
15	74.9	12.0	-0.52	-0.33

Statistics

Assigned Value	79.0	3.4
Spike	Not Spiked	
Homogeneity Value	76	11
Robust Average	79.0	3.4
Median	79.3	2.9
Mean	79.6	
N	10	
Max.	92.1	
Min.	72	
Robust SD	4.3	
Robust CV	5.4%	


Results: S2 - Fe
Assigned Value = 79.0 ± 3.4 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
11 15 12 10 1 13 6 7 2 5 14 3 4 8 9 Md R.A. H.V.
Laboratory


z-Scores: S2 - Fe
Target Standard Deviation 7.9 mg/kg (Equivalent to 10% CV)
z-Score
-0.9 -0.5 -0.4 -0.1 0.0 0.1 0.1 0.4 0.4 1.7
11 15 12 10 1 13 6 7 2 5
Laboratory

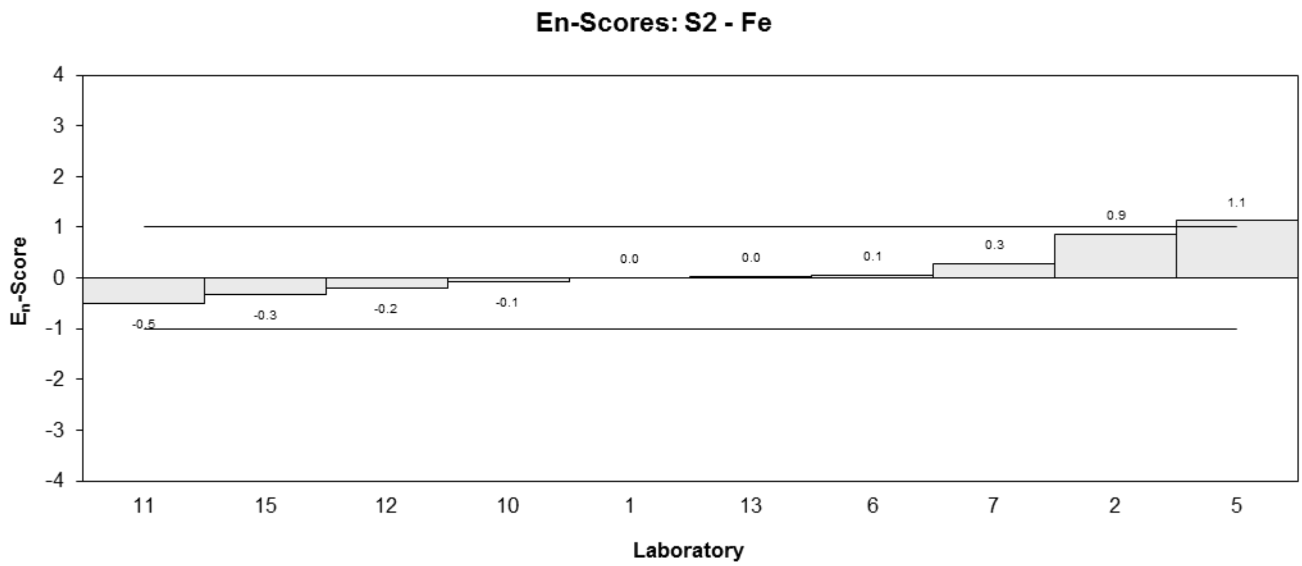

En-Scores: S2 - Fe
E_n-Score
-0.5 -0.3 -0.2 -0.1 0.0 0.0 0.1 0.3 0.9 1.1
11 15 12 10 1 13 6 7 2 5
Laboratory

Figure 43

Table 49

Sample Details

Sample No.	S2
Matrix.	Infant formula
Analyte.	Iodine
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	NT	NT
2	NT	NT
3	NT	NT
4	NT	NT
5	NT	NT
6	1.24	NR
7	NT	NT
8	NT	NT
9	NT	NT
10	NT	NT
11	NT	NT
12	1.3	0.26
13	NT	NT
14	NT	NT
15	NT	NT

Statistics*

Assigned Value	Not Set	
Spike	Not Spiked	
Mean	1.27	

*Insufficient data to calculate statistics

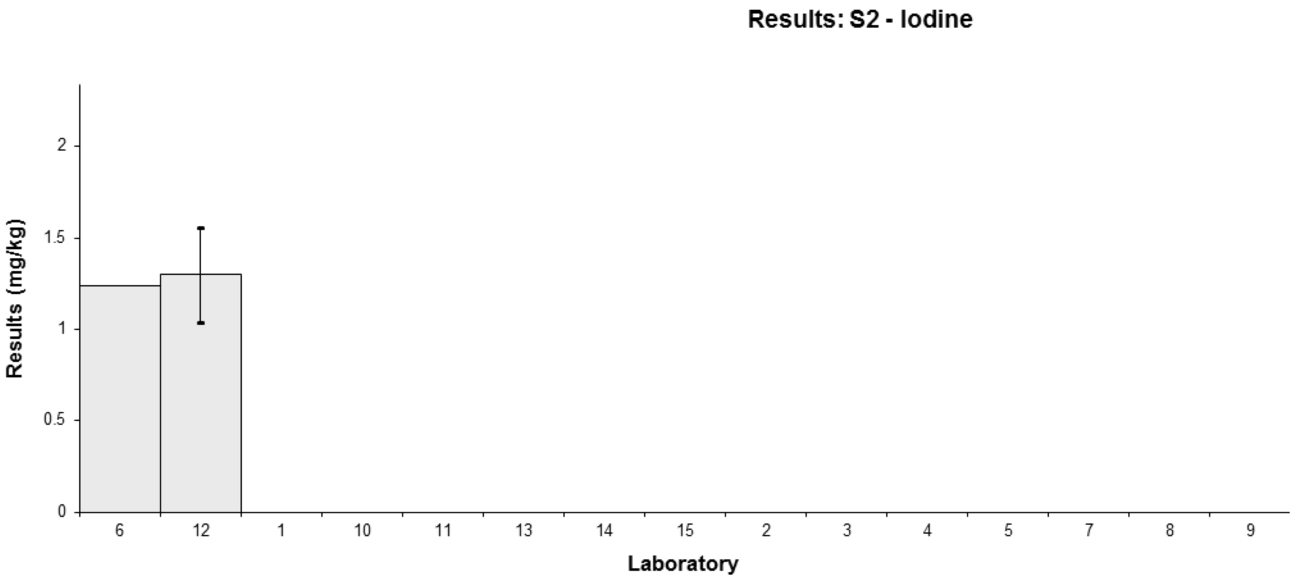

Results: S2 - Iodine
Results (mg/kg)
0
0.5
1
1.5
2
6
12
1
10
11
13
14
15
2
3
4
5
7
8
9
Laboratory

Figure 44

Table 50

Sample Details

Sample No.	S2
Matrix.	Infant formula
Analyte.	K
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	7600	400	-1.03	-1.56
2	8686	88	0.26	0.54
3	NT	NT		
4	NT	NT		
5	9060	1360	0.70	0.42
6	8810	1230	0.40	0.26
7	8760	510	0.34	0.45
8	NT	NT		
9	NT	NT		
10	8170	817	-0.35	-0.33
11	8240	1110	-0.27	-0.20
12	8130	1220	-0.40	-0.27
13	NT	NT		
14	NT	NT		
15	8570	1200	0.12	0.08

Statistics

Assigned Value	8470	390
Spike	Not Spiked	
Homogeneity Value	7800	1200
Robust Average	8470	390
Median	8570	380
Mean	8447	
N	9	
Max.	9060	
Min.	7600	
Robust SD	470	
Robust CV	5.5%	

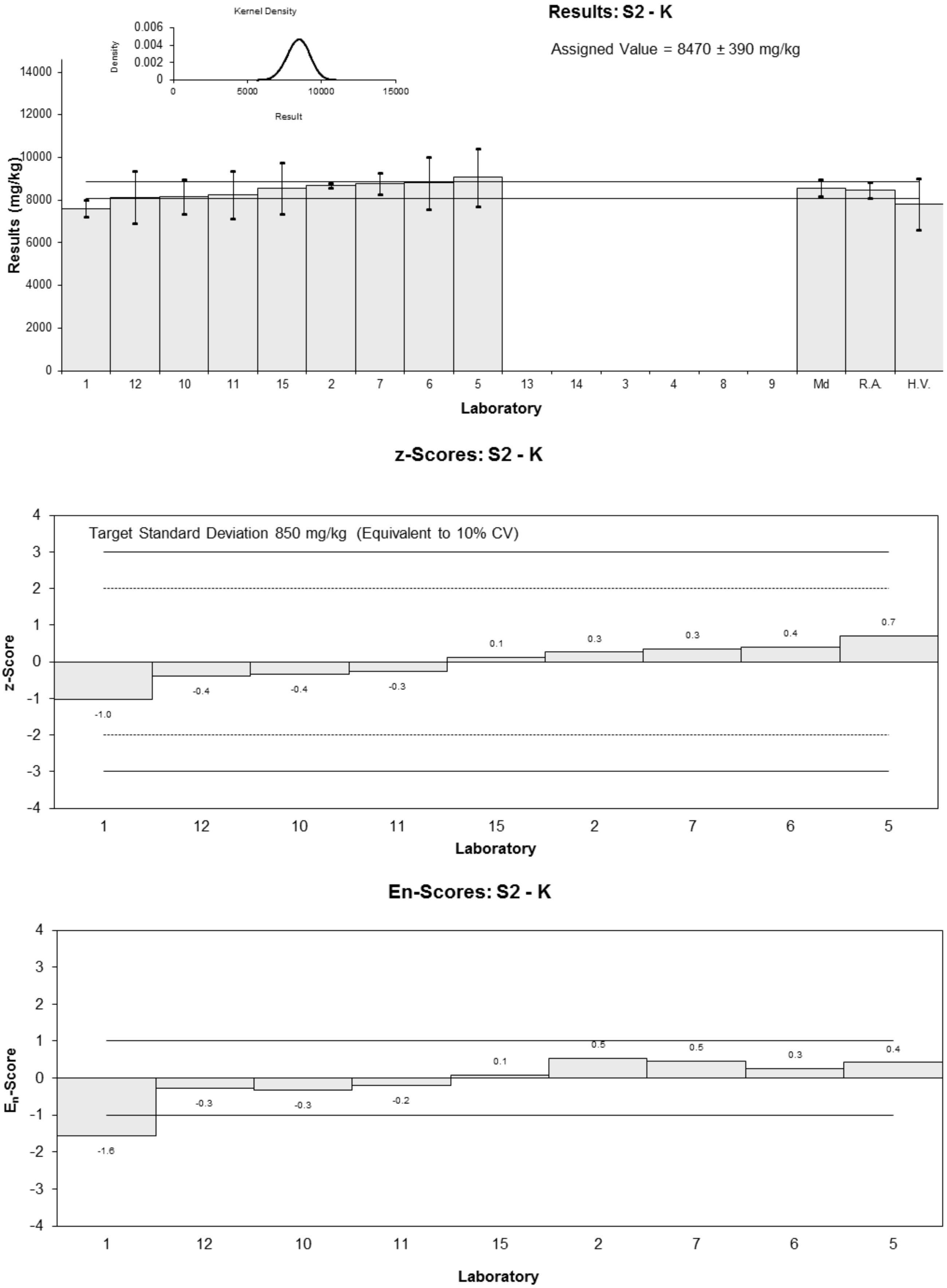

Results: S2 - K
Assigned Value = 8470 ± 390 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
1 12 10 11 15 2 7 6 5 13 14 3 4 8 9 Md R.A. H.V.
z-Scores: S2 - K
Target Standard Deviation 850 mg/kg (Equivalent to 10% CV)
z-Score
-1.0 -0.4 -0.4 -0.3 0.1 0.3 0.3 0.4 0.7
Laboratory
En-Scores: S2 - K
En-Score
-1.6 -0.3 -0.3 -0.2 0.1 0.5 0.5 0.3 0.4
Laboratory

Figure 45

Table 51

Sample Details

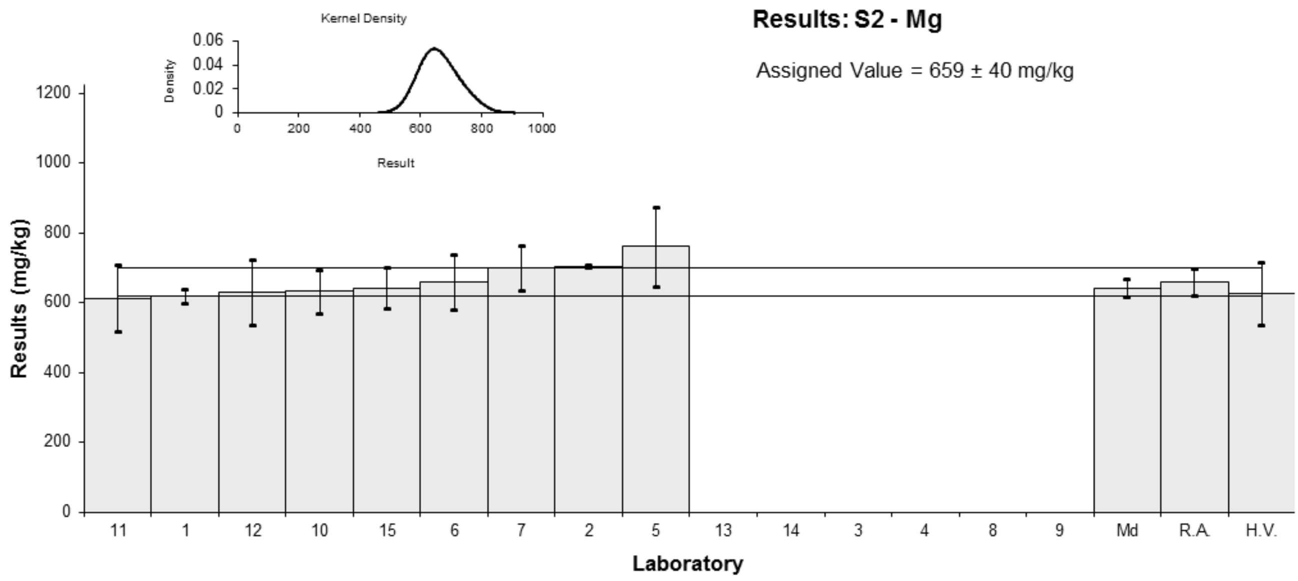
Sample No.	S2
Matrix.	Infant formula
Analyte.	Mg
Units	mg/kg

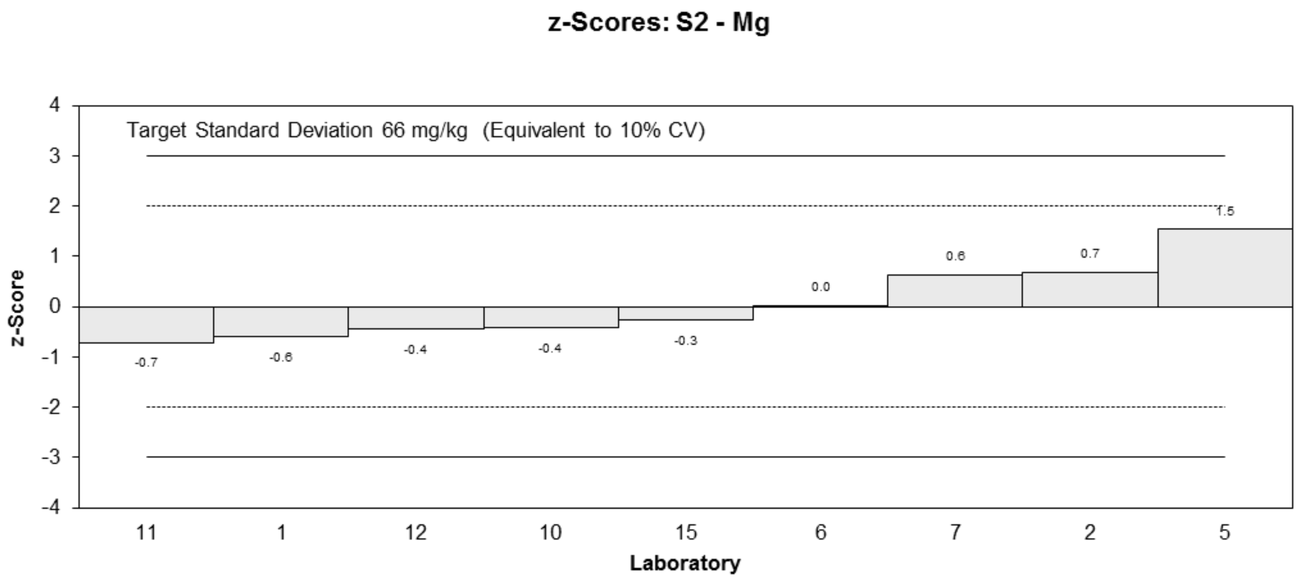
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	620	20	-0.59	-0.87
2	704.2	4.67	0.69	1.12
3	NT	NT		
4	NT	NT		
5	760	114	1.53	0.84
6	660	79	0.02	0.01
7	700	64	0.62	0.54
8	NT	NT		
9	NT	NT		
10	632	63.2	-0.41	-0.36
11	612	95.5	-0.71	-0.45
12	630	94.5	-0.44	-0.28
13	NT	NT		
14	NT	NT		
15	642	57.8	-0.26	-0.24

Statistics

Assigned Value	659	40
Spike	Not Spiked	
Homogeneity Value	627	90
Robust Average	659	40
Median	642	25
Mean	662	
N	9	
Max.	760	
Min.	612	
Robust SD	48	
Robust CV	7.4%	


Results: S2 - Mg
Assigned Value = 659 ± 40 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
11 1 12 10 15 6 7 2 5 13 14 3 4 8 9 Md R.A. H.V.


z-Scores: S2 - Mg
Target Standard Deviation 66 mg/kg (Equivalent to 10% CV)
z-Score
-0.7 -0.6 -0.4 -0.4 -0.3 0.0 0.6 0.7 1.5
11 1 12 10 15 6 7 2 5
Laboratory

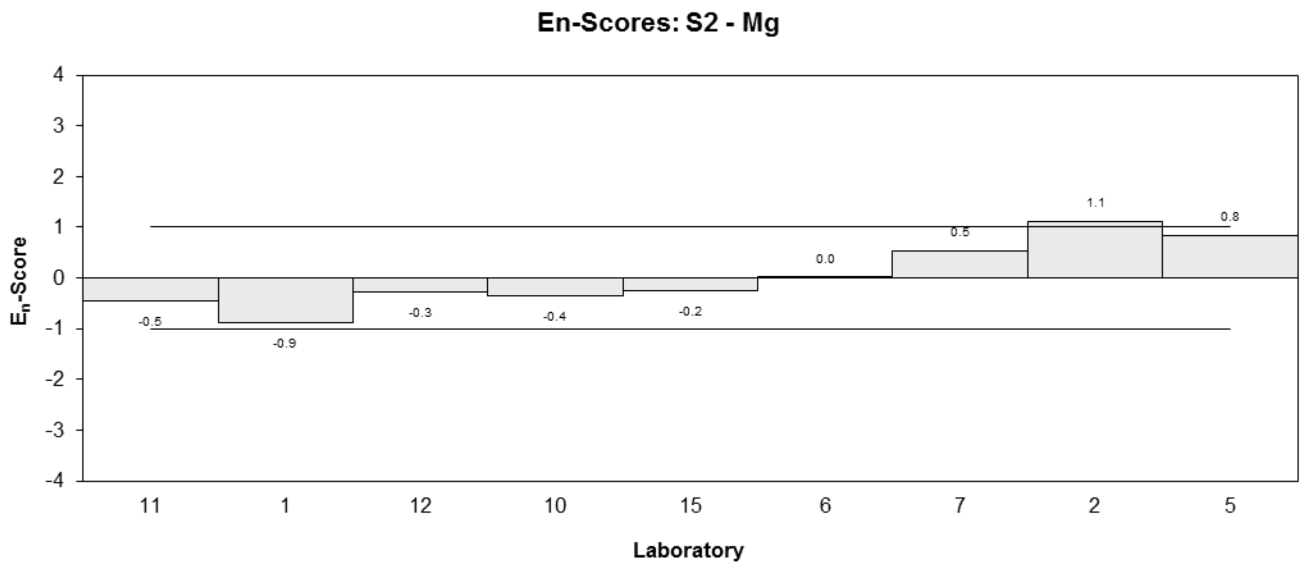

En-Scores: S2 - Mg
E_n-Score
-0.5 -0.9 -0.3 -0.4 -0.2 0.0 0.5 1.1 0.8
11 1 12 10 15 6 7 2 5
Laboratory

Figure 46

Table 52

Sample Details

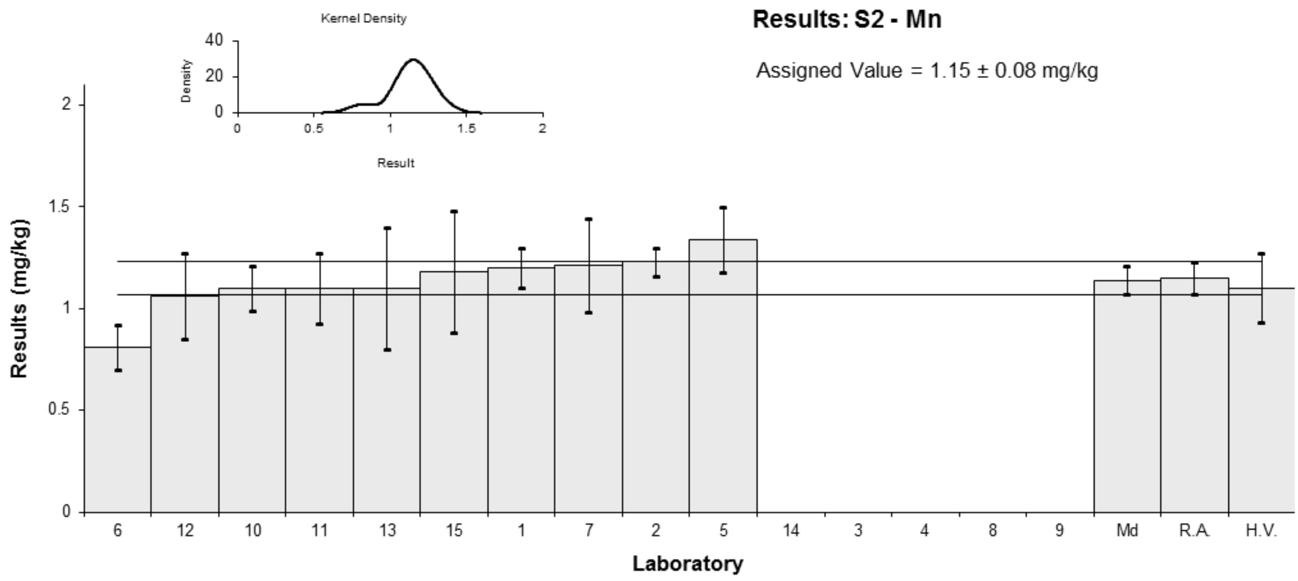
Sample No.	S2
Matrix.	Infant formula
Analyte.	Mn
Units	mg/kg

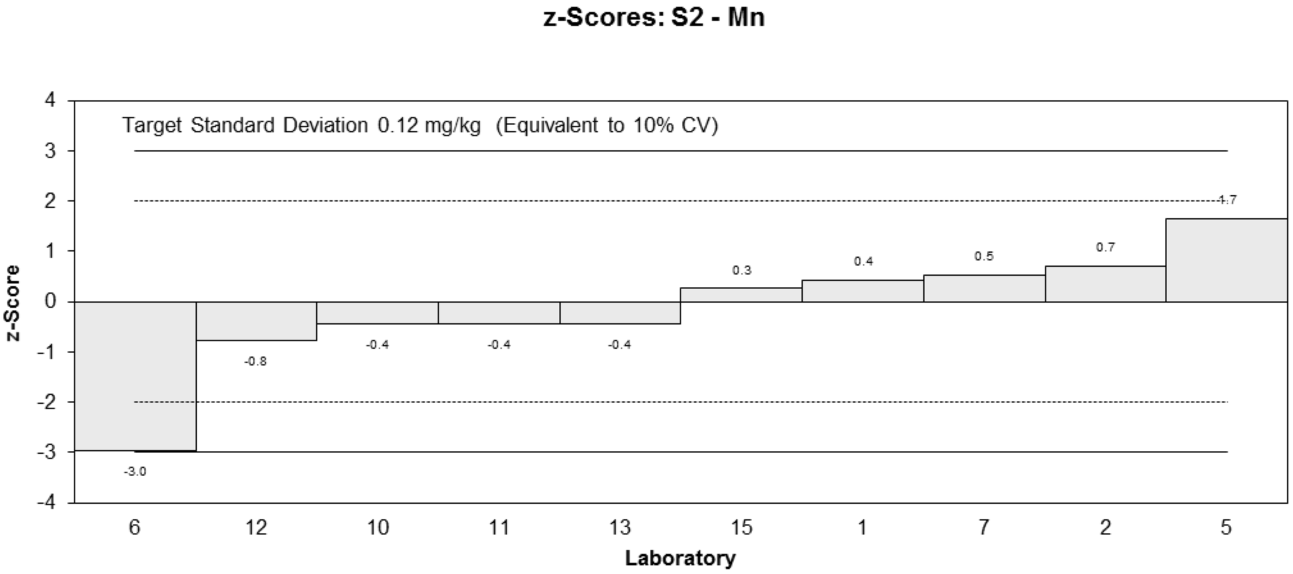
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	1.2	0.1	0.43	0.39
2	1.23	0.07	0.70	0.75
3	NT	NT		
4	NT	NT		
5	1.34	0.16	1.65	1.06
6	0.81	0.11	-2.96	-2.50
7	1.21	0.23	0.52	0.25
8	NT	NT		
9	NT	NT		
10	1.10	0.11	-0.43	-0.37
11	1.10	0.175	-0.43	-0.26
12	1.06	0.212	-0.78	-0.40
13	1.1	0.3	-0.43	-0.16
14	NT	NT		
15	1.18	0.30	0.26	0.10

Statistics

Assigned Value	1.15	0.08
Spike	Not Spiked	
Homogeneity Value	1.10	0.17
Robust Average	1.15	0.08
Median	1.14	0.07
Mean	1.13	
N	10	
Max.	1.34	
Min.	0.81	
Robust SD	0.10	
Robust CV	9.3%	


Results: S2 - Mn
Assigned Value = 1.15 ± 0.08 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
6
12
10
11
13
15
1
7
2
5
14
3
4
8
9
Md
R.A.
H.V.
Laboratory


z-Scores: S2 - Mn
Target Standard Deviation 0.12 mg/kg (Equivalent to 10% CV)
z-Score
-3.0
-0.8
-0.4
-0.4
-0.4
0.3
0.4
0.5
0.7
1.7
6
12
10
11
13
15
1
7
2
5
Laboratory

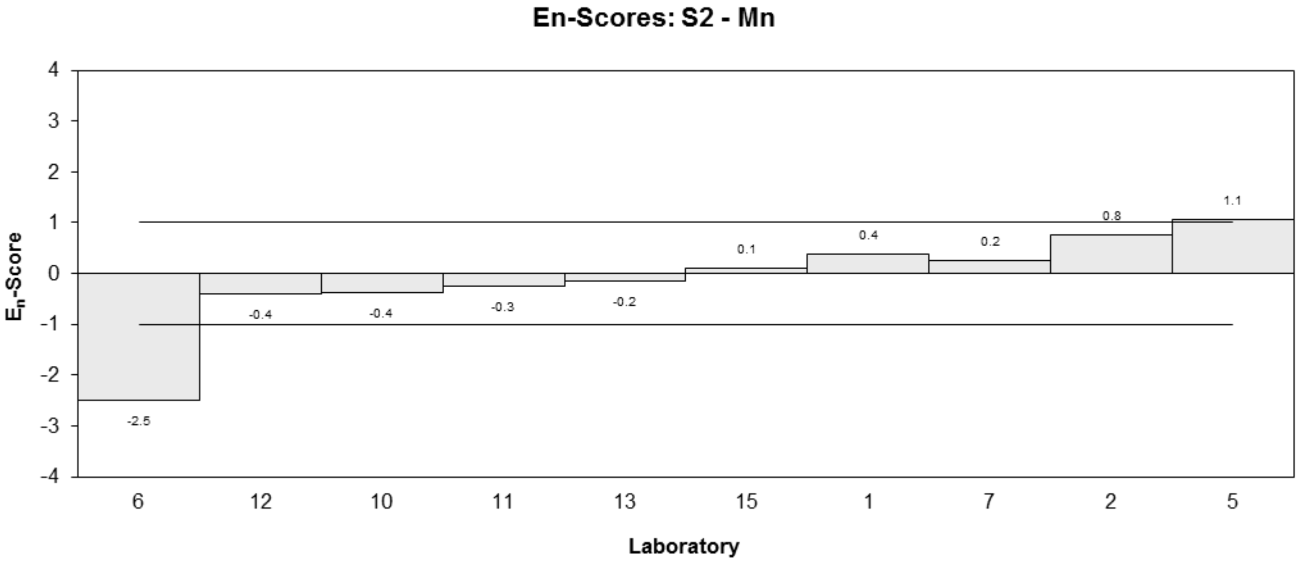

En-Scores: S2 - Mn
En-Score
-2.5
-0.4
-0.4
-0.3
-0.2
0.1
0.4
0.2
0.8
1.1
6
12
10
11
13
15
1
7
2
5
Laboratory

Figure 47

Table 53

Sample Details

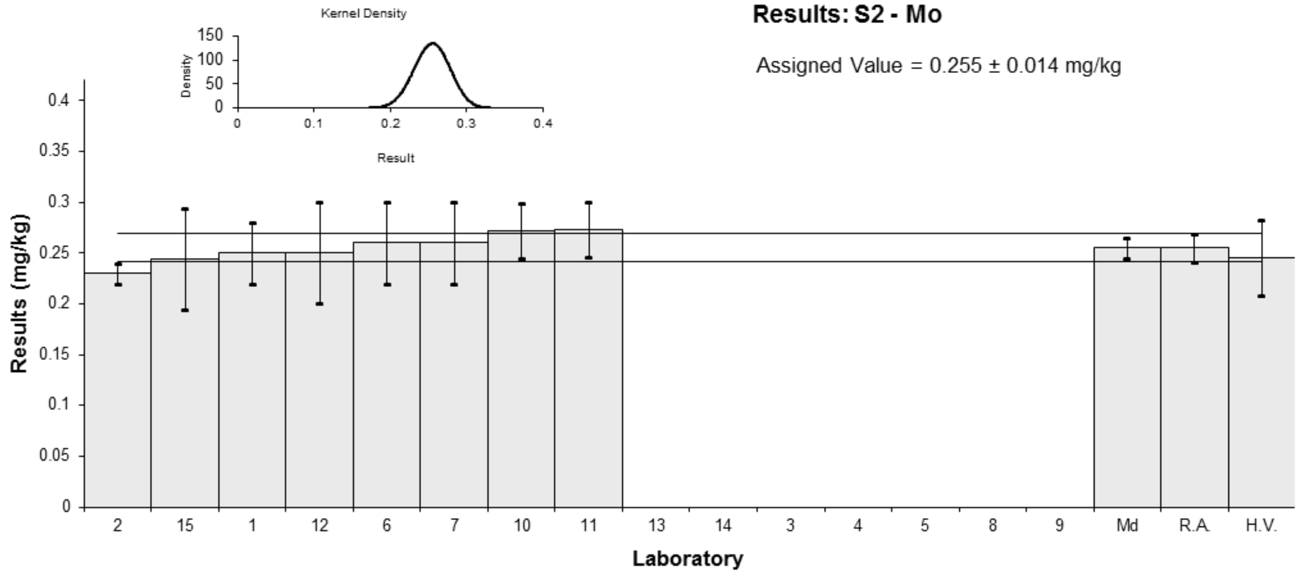
Sample No.	S2
Matrix.	Infant formula
Analyte.	Mo
Units	mg/kg

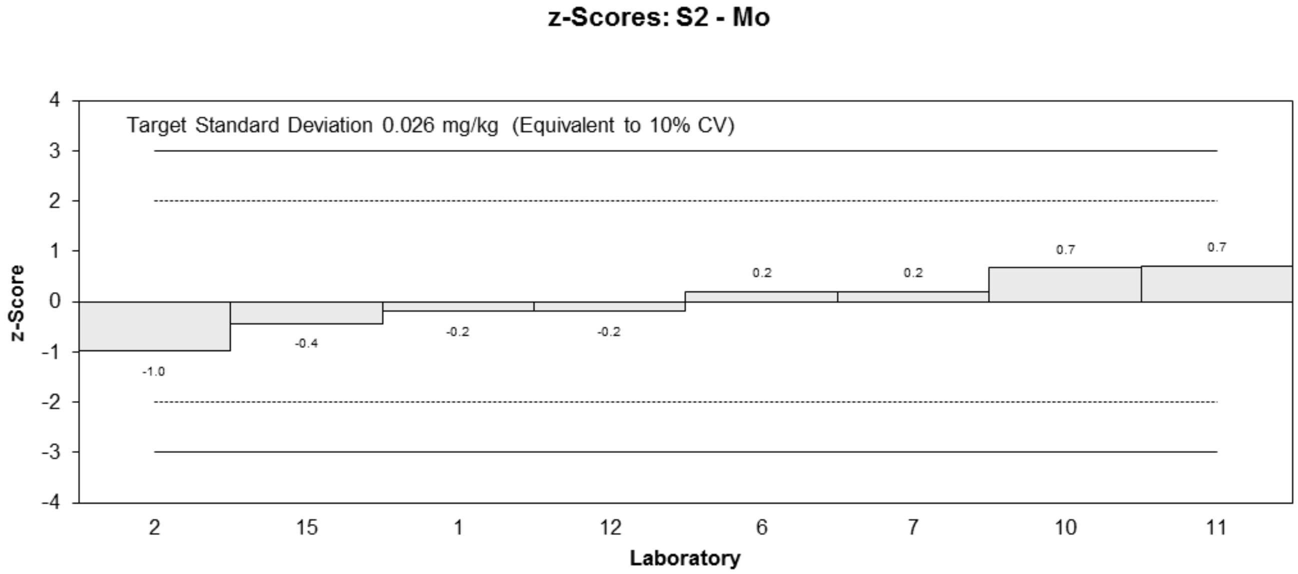
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.25	0.03	-0.20	-0.15
2	0.23	0.01	-0.98	-1.45
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	0.26	0.04	0.20	0.12
7	0.26	0.04	0.20	0.12
8	NT	NT		
9	NT	NT		
10	0.272	0.027	0.67	0.56
11	0.273	0.0274	0.71	0.58
12	0.25	0.05	-0.20	-0.10
13	<1	NR		
14	NT	NT		
15	0.244	0.050	-0.43	-0.21

Statistics

Assigned Value	0.255	0.014
Spike	Not Spiked	
Homogeneity Value	0.245	0.037
Robust Average	0.255	0.014
Median	0.255	0.010
Mean	0.255	
N	8	
Max.	0.273	
Min.	0.23	
Robust SD	0.016	
Robust CV	6.3%	


Results: S2 - Mo
Assigned Value = 0.255 ± 0.014 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
2
15
1
12
6
7
10
11
13
14
3
4
5
8
9
Md
R.A.
H.V.


z-Scores: S2 - Mo
Target Standard Deviation 0.026 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-1.0
-0.4
-0.2
-0.2
0.2
0.2
0.7
0.7

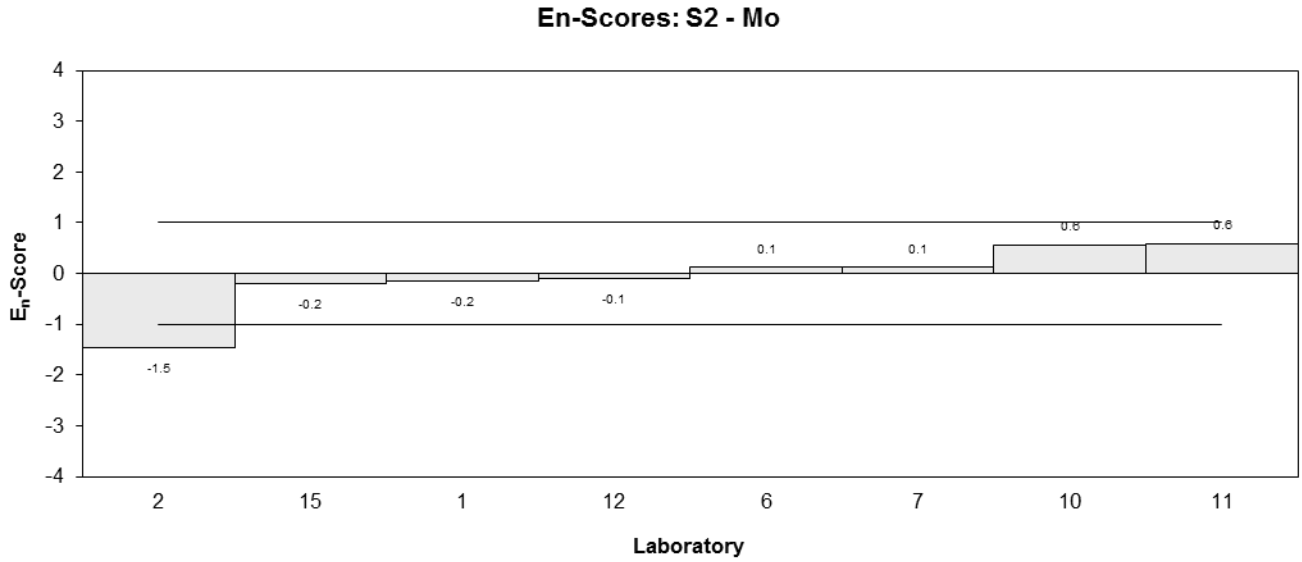

En-Scores: S2 - Mo
En-Score
Laboratory
-1.5
-0.2
-0.2
-0.1
0.1
0.1
0.6
0.6

Figure 48

Table 54

Sample Details

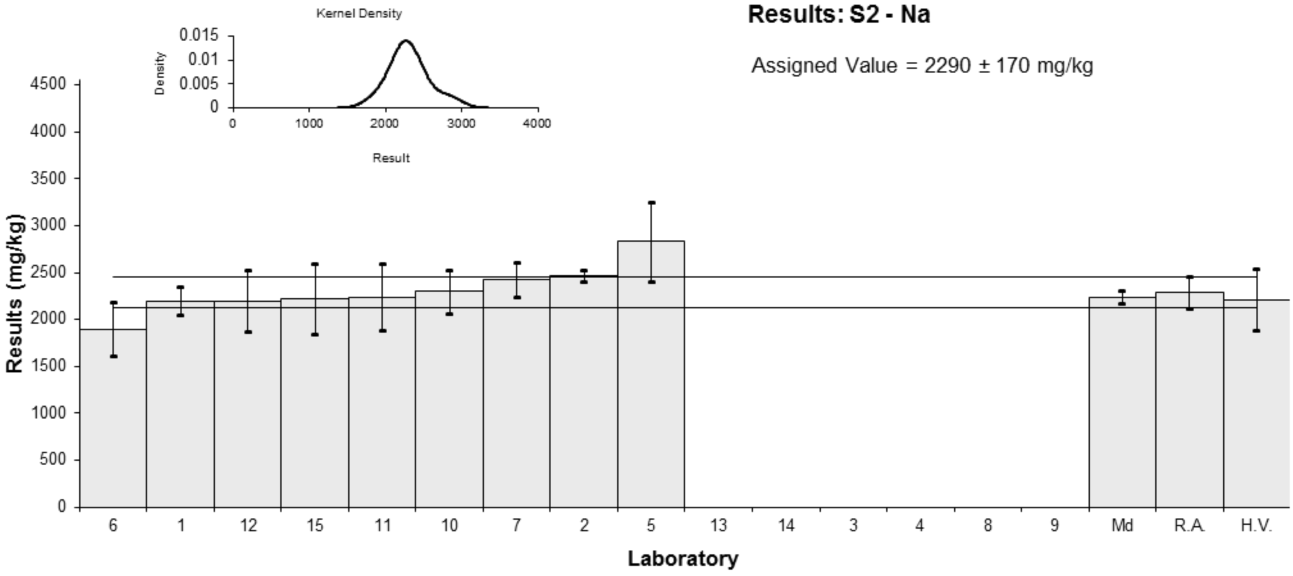
Sample No.	S2
Matrix.	Infant formula
Analyte.	Na
Units	mg/kg

Participant Results

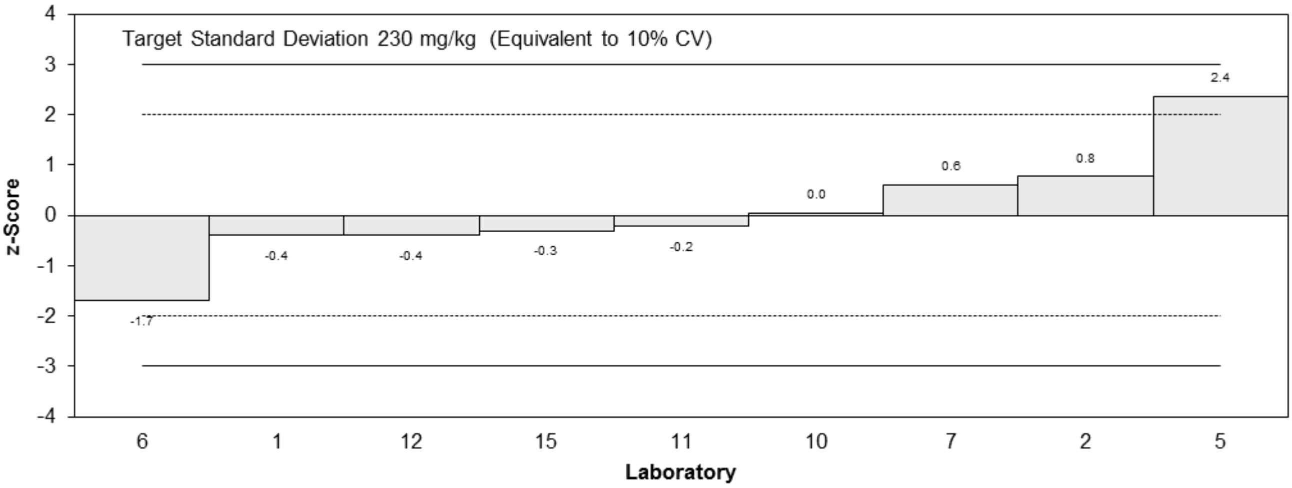
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	2200	150	-0.39	-0.40
2	2466	66	0.77	0.97
3	NT	NT		
4	NT	NT		
5	2830	425	2.36	1.18
6	1900	285	-1.70	-1.18
7	2430	183	0.61	0.56
8	NT	NT		
9	NT	NT		
10	2300	230	0.04	0.03
11	2240	358	-0.22	-0.13
12	2200	330	-0.39	-0.24
13	NT	NT		
14	NT	NT		
15	2220	377	-0.31	-0.17

Statistics

Assigned Value	2290	170
Spike	Not Spiked	
Homogeneity Value	2210	330
Robust Average	2290	170
Median	2240	70
Mean	2310	
N	9	
Max.	2830	
Min.	1900	
Robust SD	210	
Robust CV	9.1%	



z-Scores: S2 - Na



En-Scores: S2 - Na

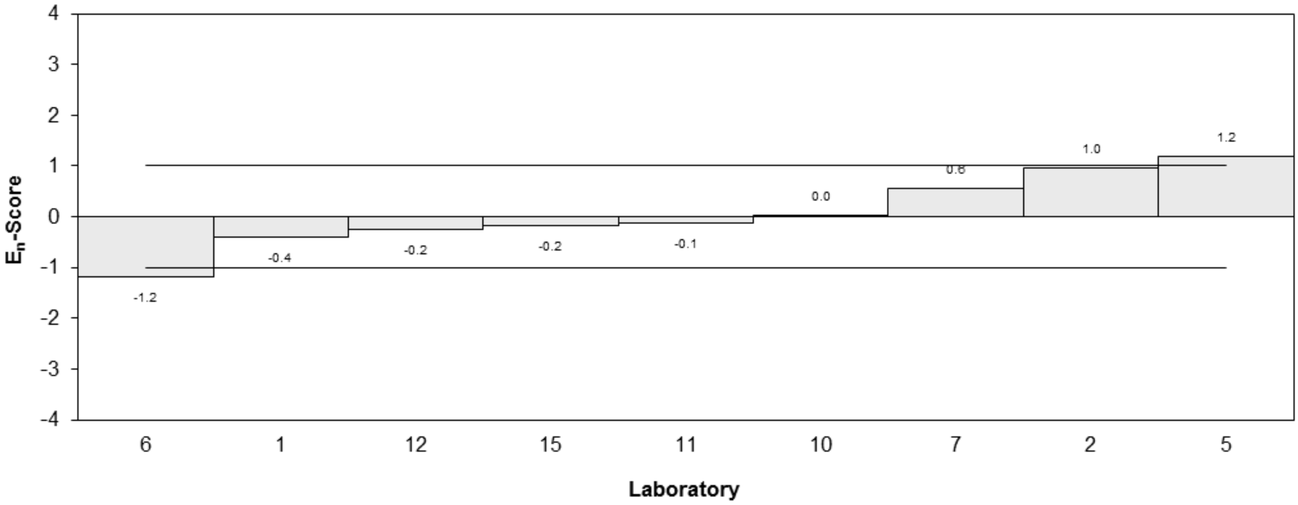


Figure 49

Table 55

Sample Details

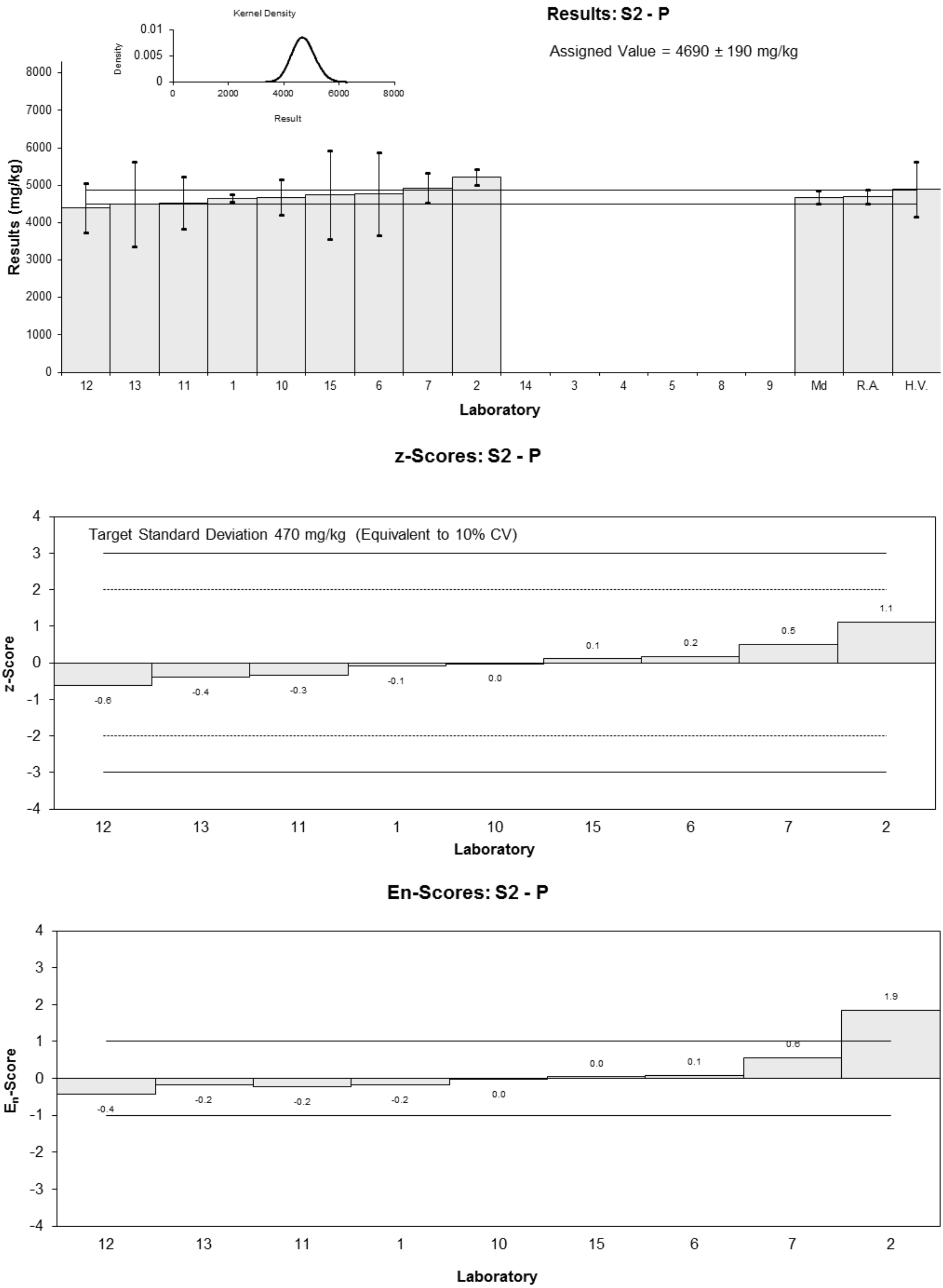
Sample No.	S2
Matrix.	Infant formula
Analyte.	P
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	4650	100	-0.09	-0.19
2	5213	209	1.12	1.85
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	4770	1100	0.17	0.07
7	4930	387	0.51	0.56
8	NT	NT		
9	NT	NT		
10	4680	468	-0.02	-0.02
11	4530	702	-0.34	-0.22
12	4400	660	-0.62	-0.42
13	4500	1130	-0.41	-0.17
14	NT	NT		
15	4740	1190	0.11	0.04

Statistics

Assigned Value	4690	190
Spike	Not Spiked	
Homogeneity Value	4900	730
Robust Average	4690	190
Median	4680	170
Mean	4713	
N	9	
Max.	5213	
Min.	4400	
Robust SD	230	
Robust CV	5%	



Results: S2 - P
Assigned Value = 4690 ± 190 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
12
13
11
1
10
15
6
7
2
14
3
4
5
8
9
Md
R.A.
H.V.
z-Scores: S2 - P
Target Standard Deviation 470 mg/kg (Equivalent to 10% CV)
z-Score
-0.6
-0.4
-0.3
-0.1
0.0
0.1
0.2
0.5
1.1
Laboratory
En-Scores: S2 - P
E_n-Score
-0.4
-0.2
-0.2
-0.2
0.0
0.0
0.1
0.6
1.9
Laboratory

Figure 50

Table 56

Sample Details

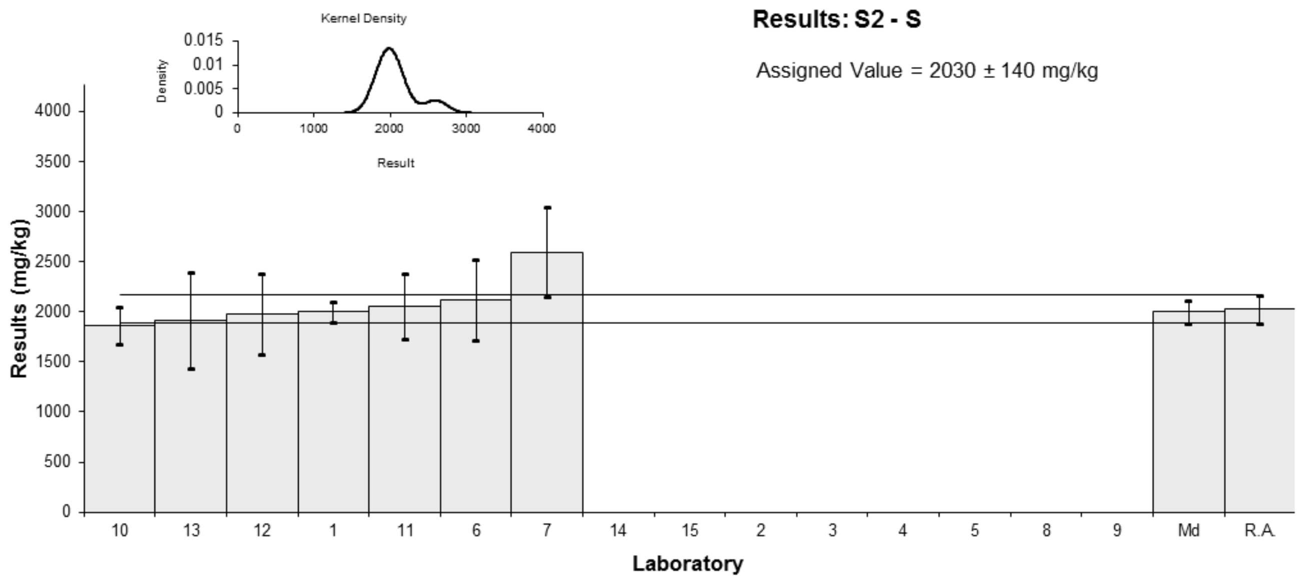
Sample No.	S2
Matrix.	Infant formula
Analyte.	S
Units	mg/kg

Participant Results

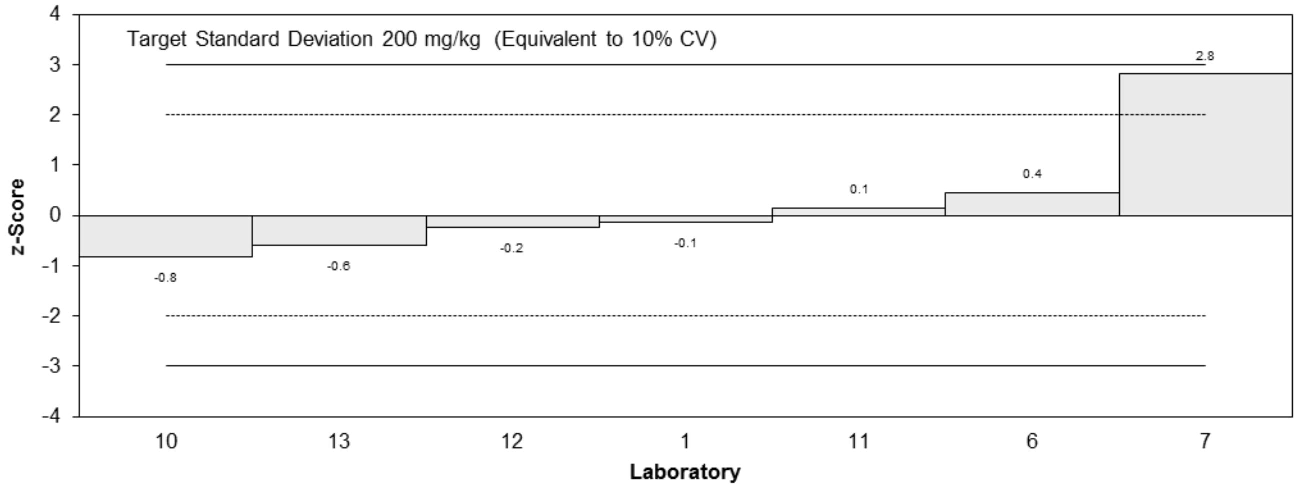
Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	2000	100	-0.15	-0.17
2	NT	NT		
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	2120	400	0.44	0.21
7	2600	451	2.81	1.21
8	NT	NT		
9	NT	NT		
10	1860	186	-0.84	-0.73
11	2060	329	0.15	0.08
12	1980	400	-0.25	-0.12
13	1910	480	-0.59	-0.24
14	NT	NT		
15	NT	NT		

Statistics

Assigned Value	2030	140
Spike	Not Spiked	
Robust Average	2030	140
Median	2000	120
Mean	2080	
N	7	
Max.	2600	
Min.	1860	
Robust SD	150	
Robust CV	7.4%	



z-Scores: S2 - S



En-Scores: S2 - S

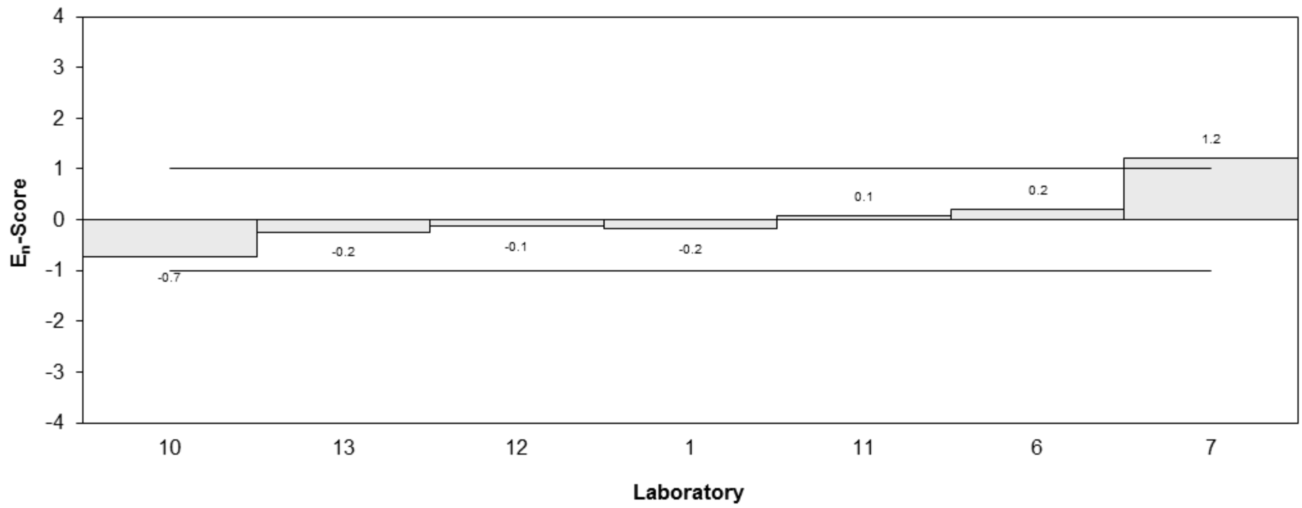


Figure 51

Table 57

Sample Details

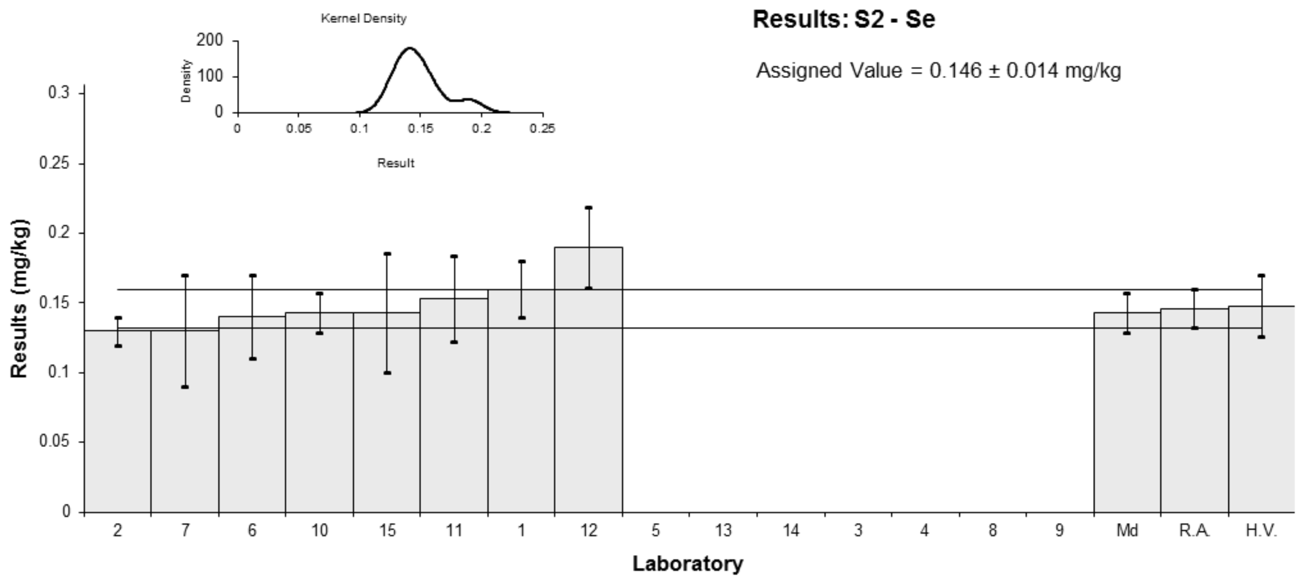
Sample No.	S2
Matrix.	Infant formula
Analyte.	Se
Units	mg/kg

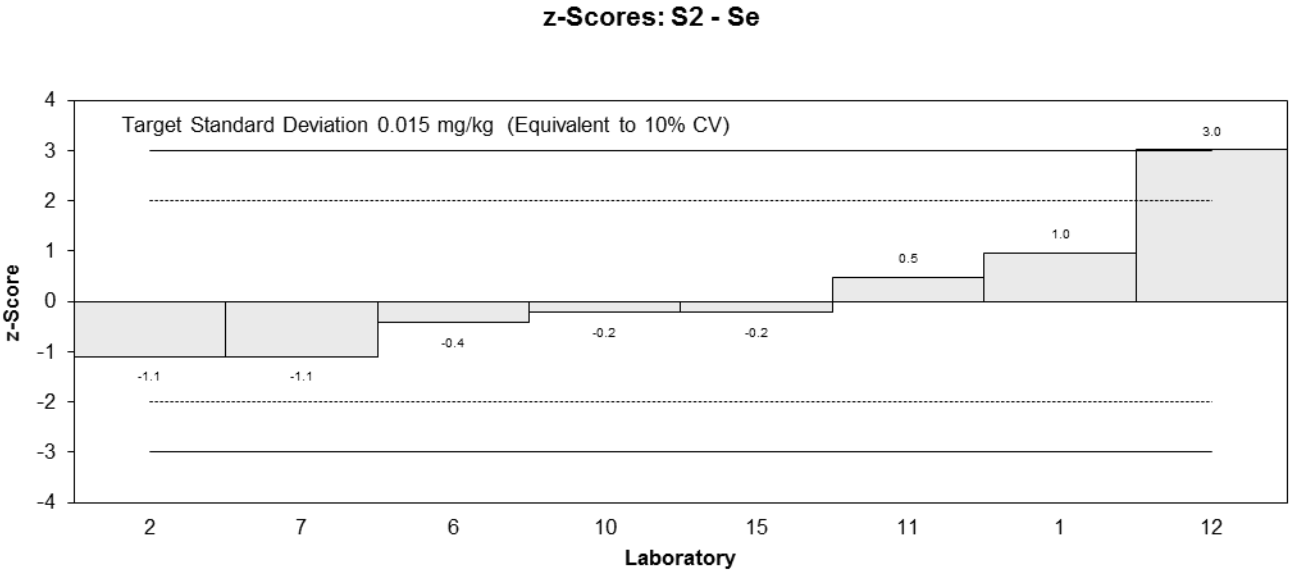
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	0.16	0.02	0.96	0.57
2	0.13	0.01	-1.10	-0.93
3	NT	NT		
4	NT	NT		
5	< 0.5	NR		
6	0.14	0.03	-0.41	-0.18
7	0.13	0.04	-1.10	-0.38
8	NT	NT		
9	NT	NT		
10	0.143	0.014	-0.21	-0.15
11	0.153	0.0306	0.48	0.21
12	0.19	0.029	3.01	1.37
13	<5	NR		
14	NT	NT		
15	0.143	0.043	-0.21	-0.07

Statistics

Assigned Value	0.146	0.014
Spike	Not Spiked	
Homogeneity Value	0.148	0.022
Robust Average	0.146	0.014
Median	0.143	0.014
Mean	0.149	
N	8	
Max.	0.19	
Min.	0.13	
Robust SD	0.016	
Robust CV	11%	


Results: S2 - Se
Assigned Value = 0.146 ± 0.014 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
2
7
6
10
15
11
1
12
5
13
14
3
4
8
9
Md
R.A.
H.V.


z-Scores: S2 - Se
Target Standard Deviation 0.015 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
2
7
6
10
15
11
1
12
-1.1
-1.1
-0.4
-0.2
-0.2
0.5
1.0
3.0

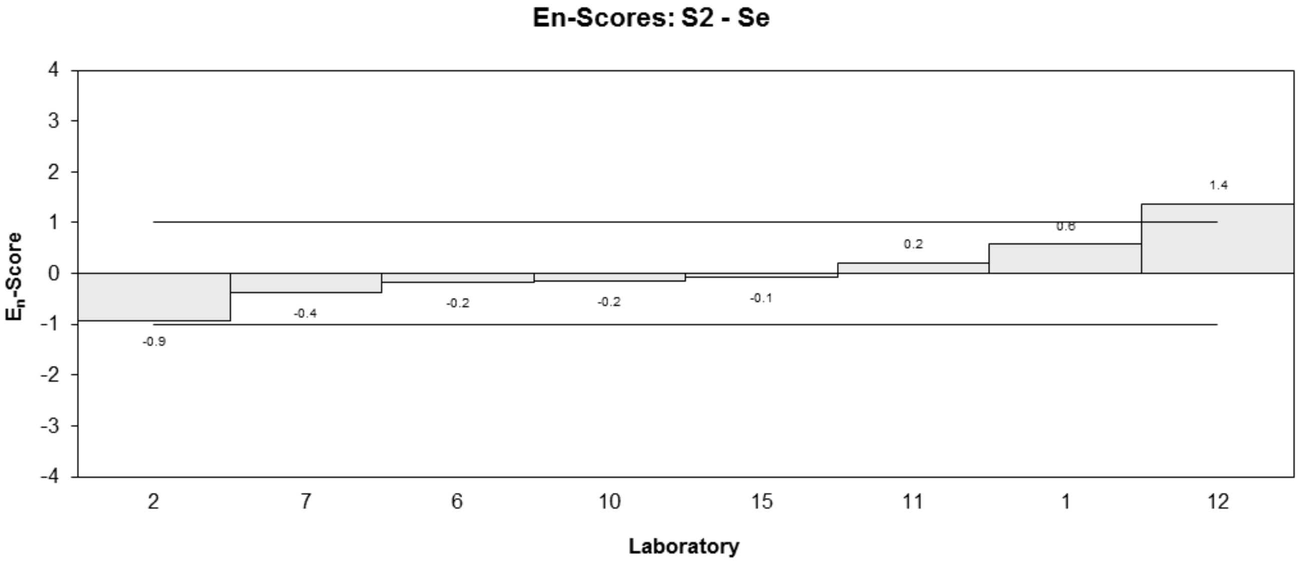

En-Scores: S2 - Se
En-Score
Laboratory
2
7
6
10
15
11
1
12
-0.9
-0.4
-0.2
-0.2
-0.1
0.2
0.6
1.4

Figure 52

Table 58

Sample Details

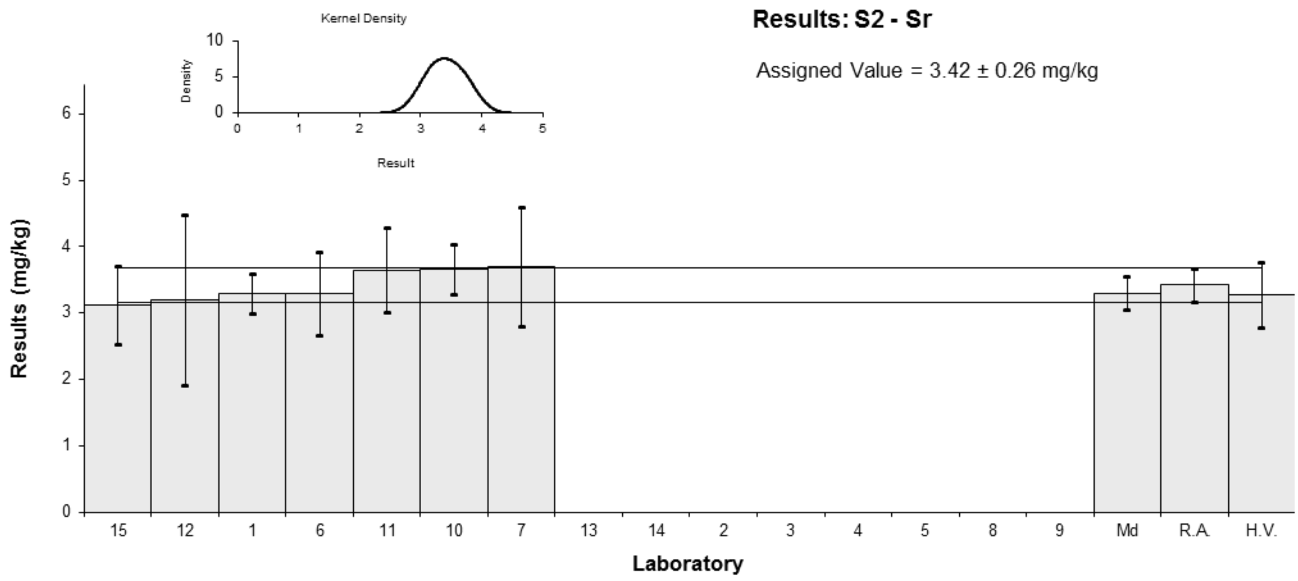
Sample No.	S2
Matrix.	Infant formula
Analyte.	Sr
Units	mg/kg

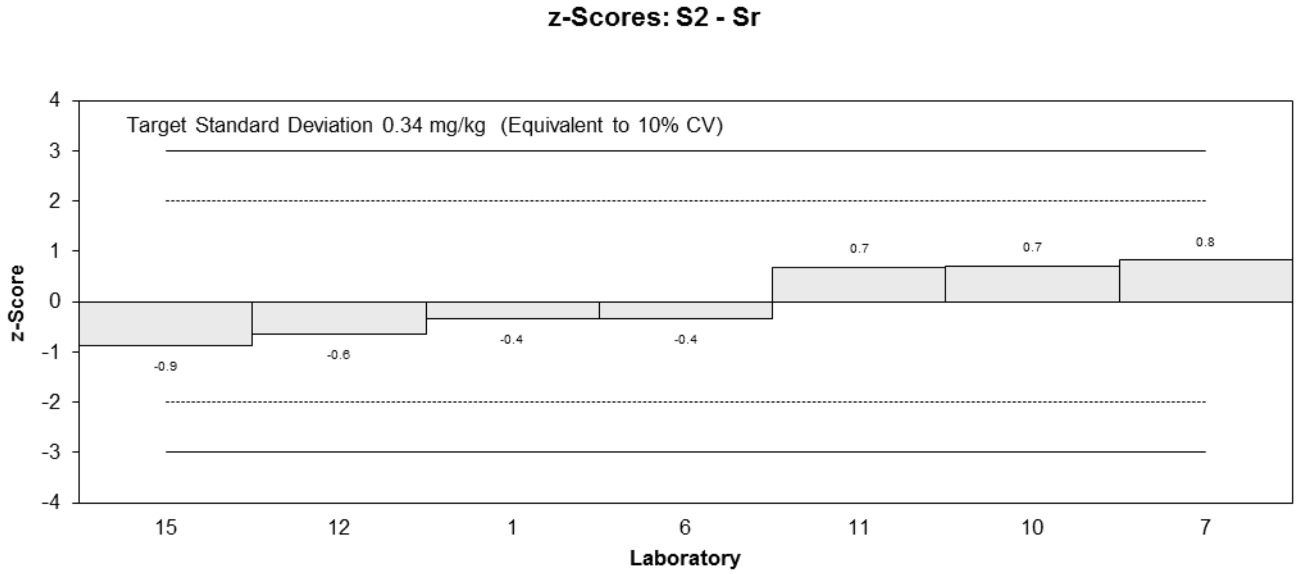
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	3.3	0.3	-0.35	-0.30
2	NT	NT		
3	NT	NT		
4	NT	NT		
5	NT	NT		
6	3.3	0.63	-0.35	-0.18
7	3.7	0.9	0.82	0.30
8	NT	NT		
9	NT	NT		
10	3.66	0.37	0.70	0.53
11	3.65	0.635	0.67	0.34
12	3.2	1.28	-0.64	-0.17
13	NT	NT		
14	NT	NT		
15	3.12	0.59	-0.88	-0.47

Statistics

Assigned Value	3.42	0.26
Spike	Not Spiked	
Homogeneity Value	3.28	0.49
Robust Average	3.42	0.26
Median	3.30	0.25
Mean	3.42	
N	7	
Max.	3.7	
Min.	3.12	
Robust SD	0.28	
Robust CV	8.1%	


Results: S2 - Sr
Assigned Value = 3.42 ± 0.26 mg/kg
Kernel Density
Density
Result
Results (mg/kg)
Laboratory
15
12
1
6
11
10
7
13
14
2
3
4
5
8
9
Md
R.A.
H.V.


z-Scores: S2 - Sr
Target Standard Deviation 0.34 mg/kg (Equivalent to 10% CV)
z-Score
Laboratory
-0.9
-0.6
-0.4
-0.4
0.7
0.7
0.8
15
12
1
6
11
10
7

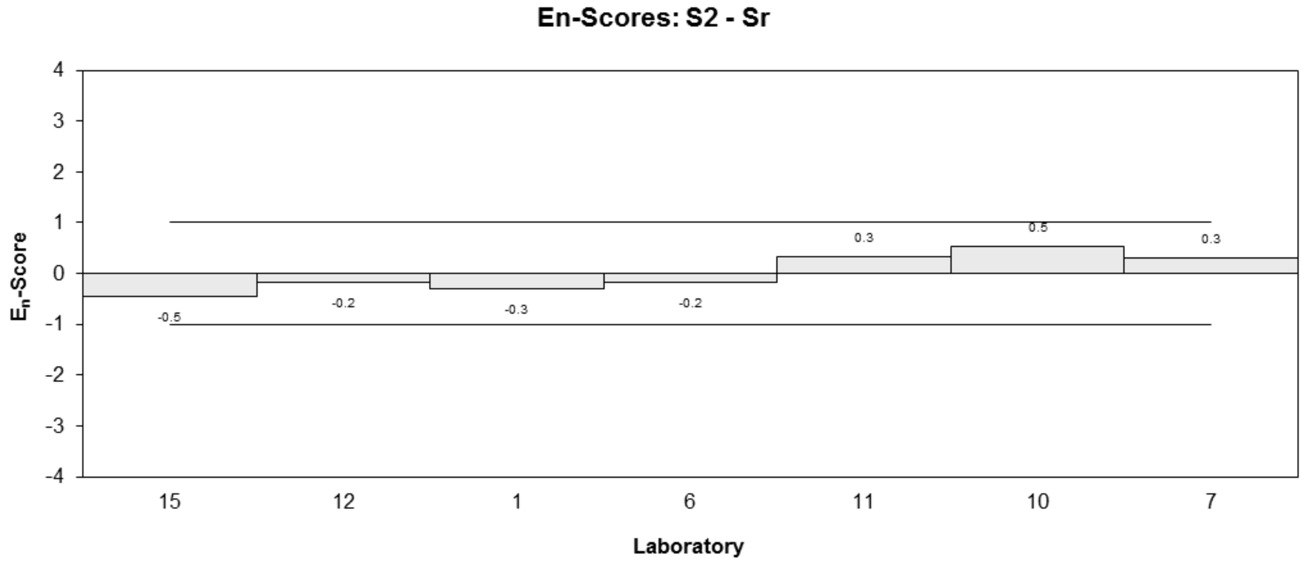

En-Scores: S2 - Sr
En-Score
Laboratory
-0.5
-0.2
-0.3
-0.2
0.3
0.5
0.3
15
12
1
6
11
10
7

Figure 53

Table 59

Sample Details

Sample No.	S2
Matrix.	Infant formula
Analyte.	V
Units	mg/kg

Participant Results

Lab Code	Result	Uncertainty
1	0.049	0.012
2	<0.10	NR
3	NT	NT
4	NT	NT
5	NT	NT
6	0.053	0.006
7	<0.05	NR
8	NT	NT
9	NT	NT
10	0.052	0.005
11	0.061	0.0119
12	<0.10	0.04
13	<1	NR
14	NT	NT
15	0.0575	0.016

Statistics

Assigned Value	Not Set	
Spike	Not Spiked	
Homogeneity Value	0.0443	0.0067
Robust Average	0.0545	0.0060
Median	0.0530	0.0074
Mean	0.0545	
N	5	
Max.	0.061	
Min.	0.049	
Robust SD	0.0054	
Robust CV	9.9%	

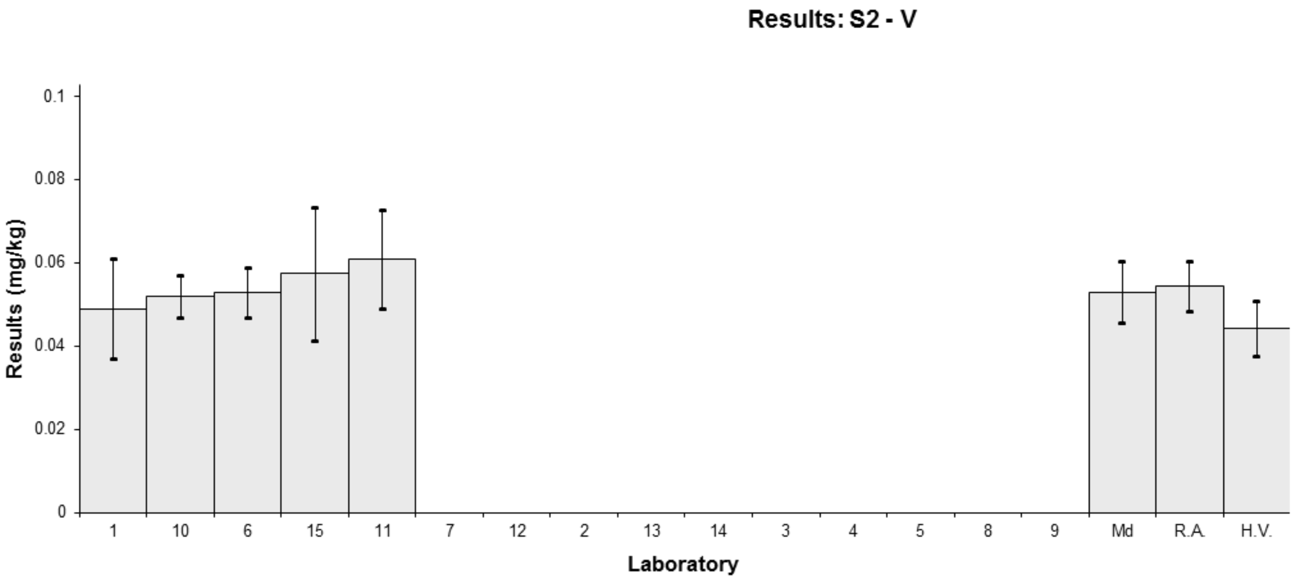

Results: S2 - V
Results (mg/kg)
0.1
0.08
0.06
0.04
0.02
0
1
10
6
15
11
7
12
2
13
14
3
4
5
8
9
Md
R.A.
H.V.
Laboratory

Figure 54

Table 60

Sample Details

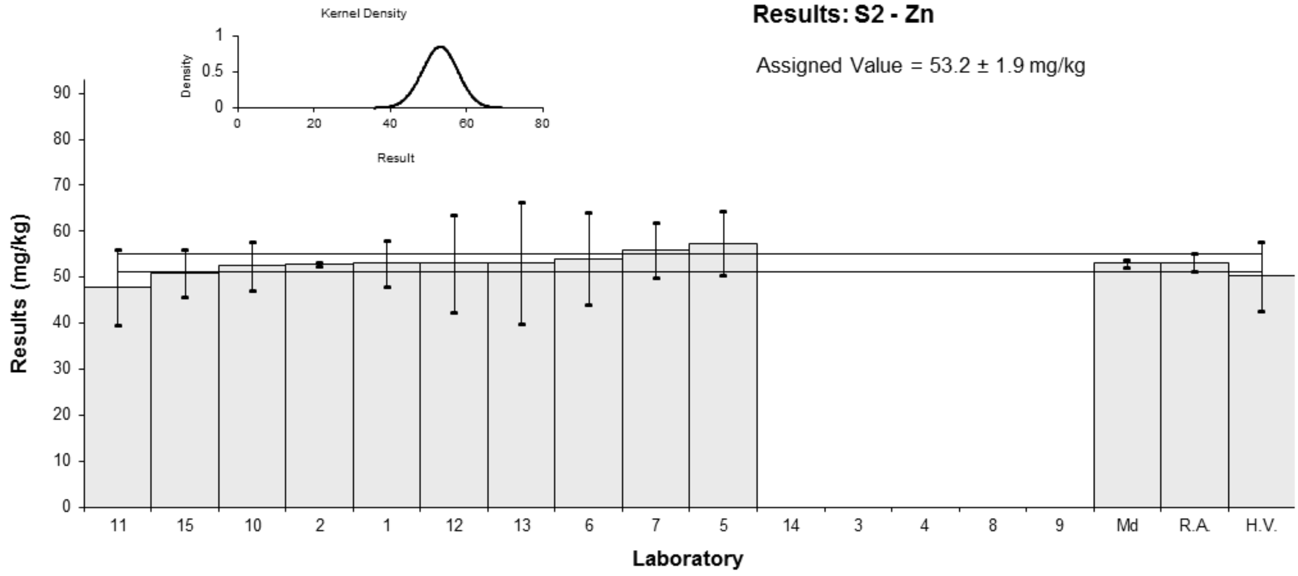
Sample No.	S2
Matrix.	Infant formula
Analyte.	Zn
Units	mg/kg

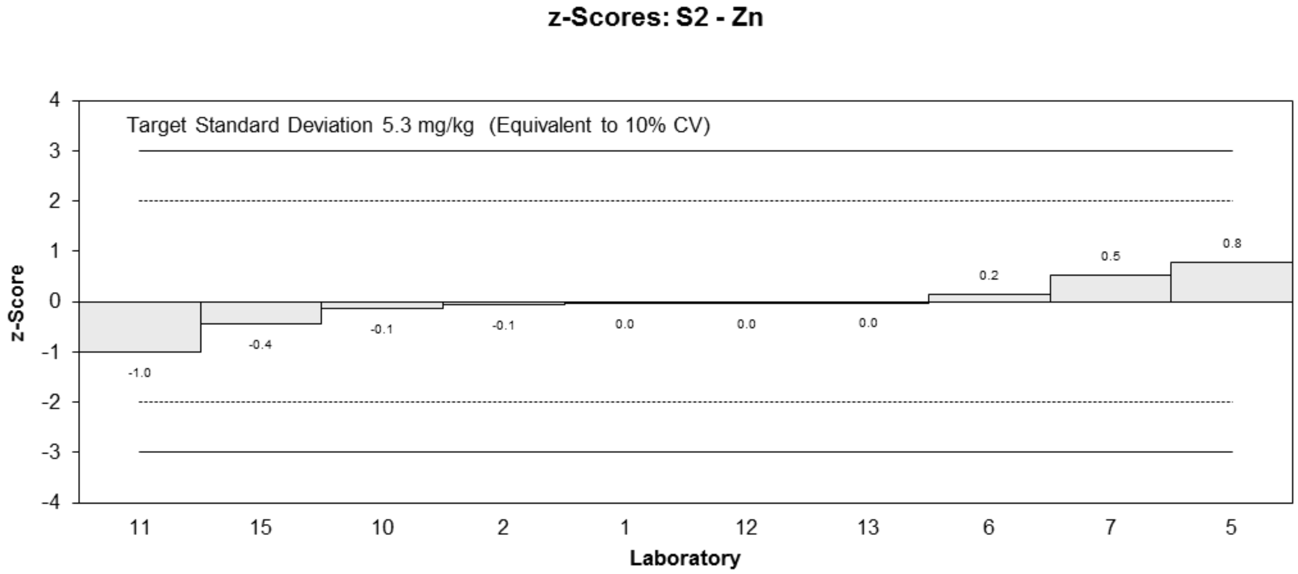
Participant Results

Lab Code	Result	Uncertainty	z-Score	E_n-Score
1	53	5.0	-0.04	-0.04
2	52.86	0.35	-0.06	-0.18
3	NT	NT		
4	NT	NT		
5	57.4	6.89	0.79	0.59
6	54	10	0.15	0.08
7	56	6	0.53	0.44
8	NT	NT		
9	NT	NT		
10	52.5	5.25	-0.13	-0.13
11	47.8	8.13	-1.02	-0.65
12	53.0	10.6	-0.04	-0.02
13	53.1	13.3	-0.02	-0.01
14	NT	NT		
15	50.84	5.084	-0.44	-0.43

Statistics

Assigned Value	53.2	1.9
Spike	Not Spiked	
Homogeneity Value	50.2	7.5
Robust Average	53.2	1.9
Median	53.0	0.8
Mean	53.1	
N	10	
Max.	57.4	
Min.	47.8	
Robust SD	2.4	
Robust CV	4.6%	


Kernel Density
Density
Result
Results: S2 - Zn
Assigned Value = 53.2 ± 1.9 mg/kg
Results (mg/kg)
Laboratory
11 15 10 2 1 12 13 6 7 5 14 3 4 8 9 Md R.A. H.V.


z-Scores: S2 - Zn
Target Standard Deviation 5.3 mg/kg (Equivalent to 10% CV)
z-Score
-1.0 -0.4 -0.1 -0.1 0.0 0.0 0.0 0.2 0.5 0.8
11 15 10 2 1 12 13 6 7 5
Laboratory

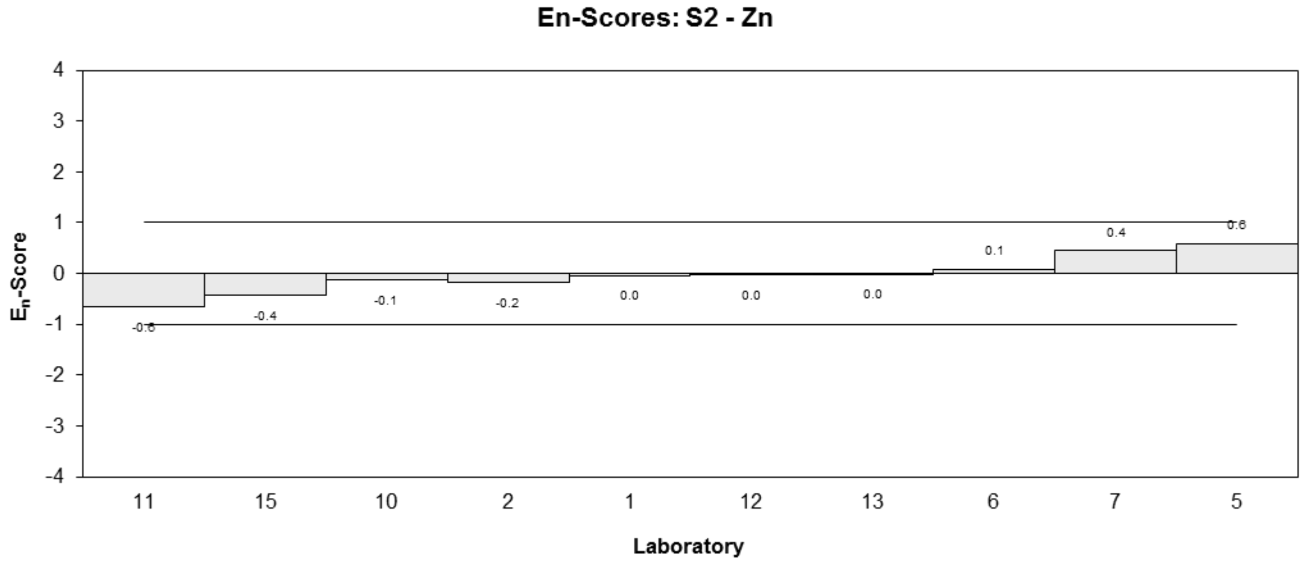

En-Scores: S2 - Zn
En-Score
-0.6 -0.4 -0.1 -0.2 0.0 0.0 0.0 0.1 0.4 0.6
11 15 10 2 1 12 13 6 7 5
Laboratory

Figure 55

7 DISCUSSION OF RESULTS

7.1 Assigned Value

Samples S1 – was freeze dried prawn matter, a certified reference material previously prepared by NMI. Assigned values for As, Cr, Cu, Fe, Mn, Ni and Se in S1 were reference values from measurements made using SA-ICP-MS (Appendix 3). The assigned value for Hg in S1 was the certified value of MX009 for Hg previously measured by NMI using IDMS (Appendix 4).

Assigned values for Ag, B, Ba, Bi, Ca, Cd, Cs, K, Mg, Na, P, Pb, Rb, S, Sn, Sr, U and Zn were the robust average of participants' results. The robust averages and associated expanded uncertainties were calculated using the procedure described in ISO13528:2015(E), Statistical methods for use in proficiency testing by interlaboratory comparisons. Results less than 50% and more than 150% of the robust average were removed before calculation of the assigned value. [8] Appendix 2 sets out the calculation for the robust average of U in Sample S1 and its associated uncertainty.

No assigned values were calculated for Al, La, Li, Sb and V in this sample because the reported results were too few or too variable. No assigned value was set for inorganic-As because this was included as a pilot program.

Sample S2 – was infant formula, prepared specifically for this study. The assigned values were the robust average of participants' results, calculated as for Sample S1.

No assigned value was set for As, B, Cd, Co Pb and V in S2 because the levels were below the reporting level of most participants. No assigned value was set for iodine either as only two results were reported. However, participants may still compare their reported results for these elements with the robust average of participants' results and/or the homogeneity value. Descriptive statistics for these elements are presented in chapter 6. No descriptive statistics were presented for Pb in S2 due to only one result (0.005 mg/kg) being reported.

Traceability of the reference values for As, Cr, Cu, Fe, Mn, Ni and Se 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 by standard addition are traceable to international standards through calibration with certified single element standards. ICP-MS measurements calibrated with isotope dilution are traceable to the SI units for mass (kg) through the primary calibration standard certified by NIST (USA) and the SI unit for amount of substance (mol) through data for isotopic composition and relative atomic mass. Isotopic compositions are traceable to IUPAC published data.

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

7.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded measurement uncertainty associated with their results. Of 460 numerical results, 437 (95%) were reported with an expanded measurement uncertainty, indicating that the majority of laboratories have addressed this requirement of ISO/IEC 17025.[10] The participants used a wide variety of procedures to estimate the expanded measurement uncertainty. These are presented in Table 6.

Approaches to estimating measurement uncertainty include: standard deviation of replicate analysis, Horwitz formula, professional judgement, bottom up approach, top down approach using precision and estimates of method and laboratory bias, and top down approach using only the reproducibility from inter-laboratory comparisons studies.[1,11 – 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 7 for Ag in S1) or under-estimated (e.g. Lab 2 for Cu, Fe, Mg in S2). As a simple rule of thumb, when the uncertainty estimate is either smaller than the uncertainty of the assigned value or larger than the uncertainty of the assigned value plus twice the target standard deviation then this should be reviewed as suspect.

Double counting the precision uncertainty components and overestimation of the laboratory or method bias are the most common errors seen in the laboratories' estimated uncertainty budgets. According to General Accreditation Guidance, Estimating and reporting measurement uncertainty of chemical test results[13] 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.

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

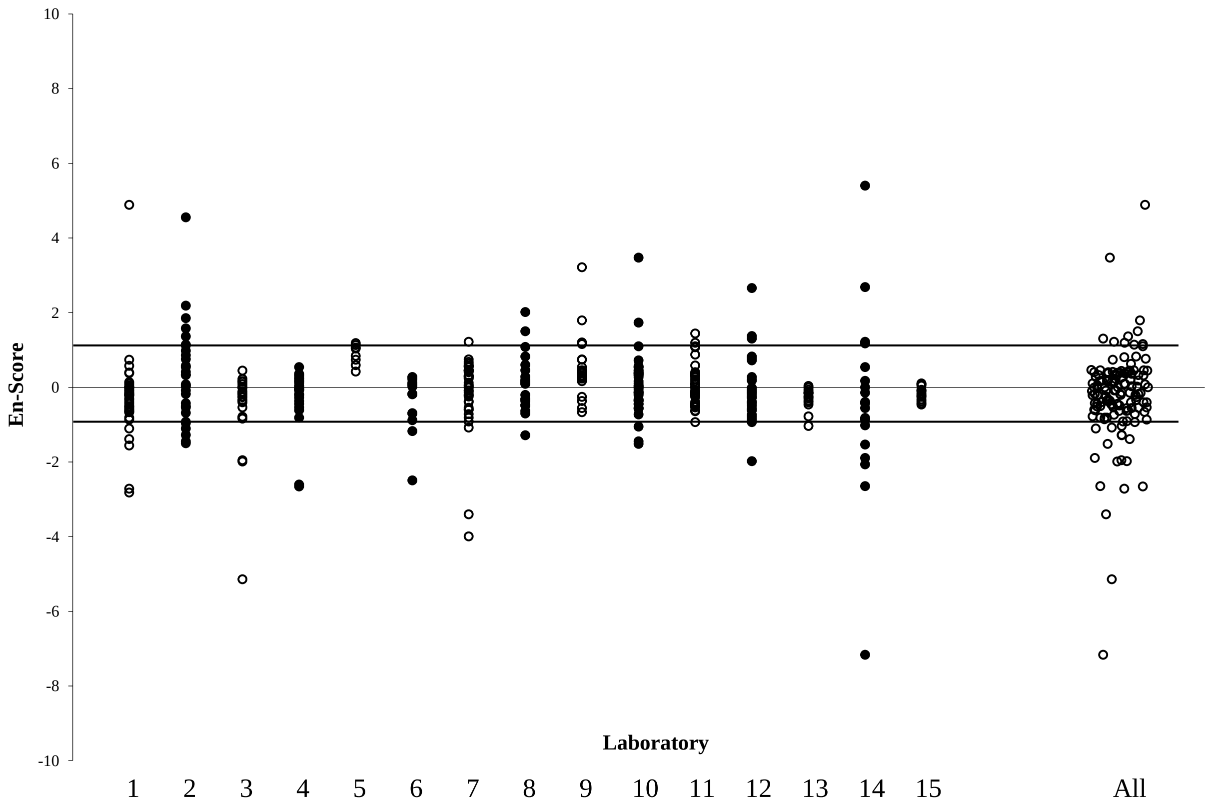
In some cases the results were reported with an inappropriate number of significant figures. The recommended format is to write uncertainty to no more than two significant figures and then to write the result with the corresponding number of decimal places. For example, instead of 0.626 ± 0.21 mg/kg,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

7.3 E_n-score

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

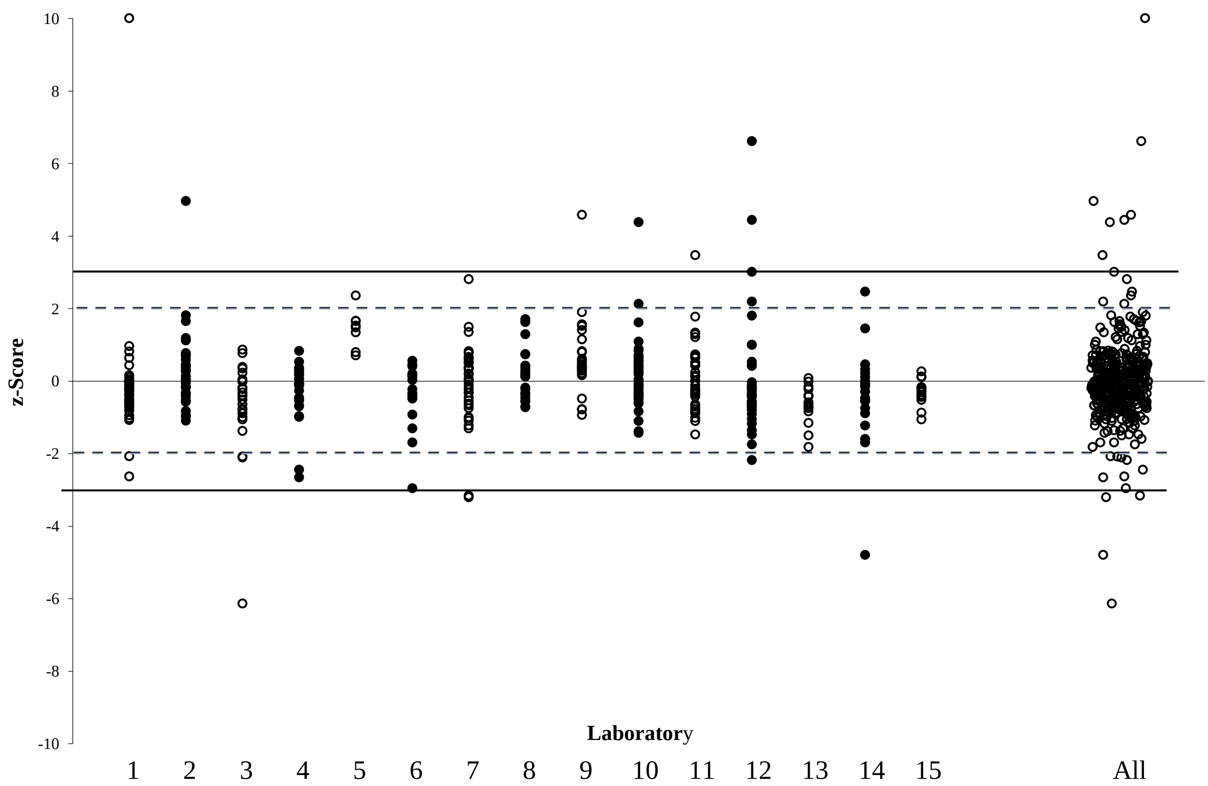
The dispersal of participants' E_n-scores is graphically presented in Figure 56. Where a laboratory did not report an expanded uncertainty with a result, an expanded uncertainty of zero (0) was used to calculate the E_n-score.

Of 401 results for which E_n-scores were calculated, 334 (83%) returned a satisfactory score of $|E_n| \leq 1$ indicating agreement of the participants' results with the assigned values within their respective expanded measurement uncertainties.



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

7.4 z-Score

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

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

The between laboratory coefficient of variation predicted by the Thompson equation[9] and the between laboratory coefficient of variation resulted in this study are presented for comparison in Table 61. The dispersal of participants' z-scores is presented in Figure 57 (by laboratory code) and in Figure 58 (by test). Of 401 results for which z-scores were calculated, 376 (94%) returned a satisfactory score of $|z| \le 2$ and 13 (3%) were questionable of $2 < |z| < 3$. Participants with multiple z-scores larger than 2 or smaller than -2 should check for laboratory bias.

Laboratories **8, 13 and 15** returned satisfactory z-scores for all analytes reported.

Table 61 Between Laboratory CV of this study, Thompson CV and Set Target PCV

Sample	Analyte	Assigned value (mg/kg)	Between Laboratories CV	Thompson CV	Target SD (as PCV)
S1	Ag	0.0154	26%	22%	20%
S1	Al	158*	78%	8%	Not Set
S1	As	3.645	7.1%	13%	10%
S1	B	1.14	9.3%	16%	10%
S1	Ba	1.24	17%	15%	15%
S1	Bi	0.0120	8.9%	22%	15%
S1	Ca	5310	3.4%	4%	10%
S1	Cd	0.128	12%	22%	15%
S1	Cr	0.95	41%	16%	20%
S1	Cs	0.0203	26%	22%	20%
S1	Cu	15.4	5.5%	11%	10%
S1	Fe	104	13%	8%	15%
S1	Hg	0.1345	12%	22%	10%
S1	Inorganic As	Not Set	44%	NA	Not Set
S1	K	1370	4.6%	5%	10%
S1	La	Not Set	31%	NA	Not Set
S1	Li	Not Set	27%	NA	Not Set
S1	Mg	1730	4.3%	5%	10%
S1	Mn	4.98	3.9%	13%	10%
S1	Na	12300	4.2%	4%	10%
S1	Ni	0.462	43%	18%	20%
S1	P	6030	5.5%	4%	10%
S1	Pb	0.305	12%	19%	10%
S1	Rb	0.652	7.7%	17%	15%
S1	S	9600	13%	4%	10%
S1	Sb	Not Set	1%	NA	10%

Table 61 Between Laboratory CV of this study, Thompson CV and Set Target PCV (continued)

Sample	Analyte	Assigned value (mg/kg)	Between Laboratories CV	Thompson CV	Target SD (as PCV)
S1	Se	2.498	11%	14%	10%
S1	Sn	0.275	19%	19%	20%
S1	Sr	63.0	5.4%	9%	10%
S1	U	0.00672	10%	22%	20%
S1	V	0.252*	55%	20%	Not Set
S1	Zn	67.8	5.3%	8%	10%
S2	Al	2.54	14%	14%	15%
S2	As	Not Set	15%	NA	Not Set
S2	B	Not Set	28%	NA	Not Set
S2	Ba	0.398	12%	18%	10%
S2	Ca	7700	8.3%	4%	10%
S2	Cd	Not Set	5.5%	NA	Not Set
S2	Co	Not Set	21%	NA	Not Set
S2	Cr	0.150	16%	21%	20%
S2	Cu	3.72	14%	13%	15%
S2	Fe	79.0	5.4%	8%	10%
S2	Iodine	Not Set	3.8%	NA	Not Set
S2	K	8470	5.5%	4%	10%
S2	Mg	659	7.4%	6%	10%
S2	Mn	1.15	9.3%	16%	10%
S2	Mo	0.255	6.3%	20%	10%
S2	Na	2290	9.1%	5%	10%
S2	P	4690	5%	4%	10%
S2	Pb	Not Set	NA	NA	Not Set
S2	S	2030	7.4%	5%	10%
S2	Se	0.146	11%	21%	10%
S2	Sr	3.42	8.1%	13%	10%
S2	V	Not Set	9.9%	NA	Not Set
S2	Zn	53.2	4.6%	9%	10%

NA = Not Available, *Information Value

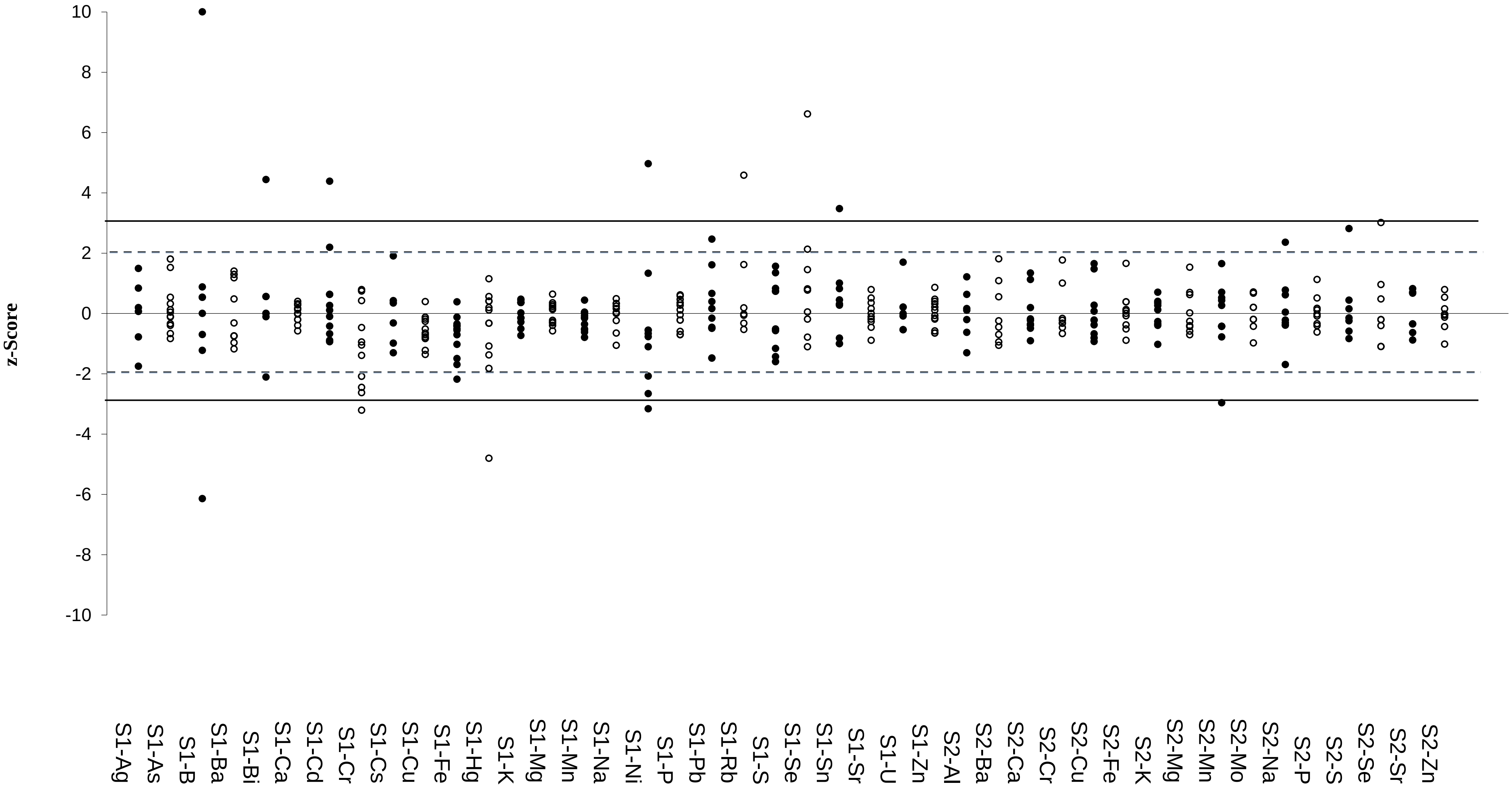
7.5 Participants' Results and Analytical Methods for Total Elements

A summary of participants' performance in the two study samples is presented in Figure 57 score dispersal and Tables 62 and 63.

Measurements of total Al, Cr, Ni and V in the freeze dried prawn sample presented most analytical difficulty.

As, B, Cd, Co Pb and V levels in S2 were below the reporting level of most participants.

The method descriptions provided by participants are presented in Tables 1 to 4; the instrumental conditions are presented in Appendix 6.



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

Figure 58 z-Score Dispersal by Analyte

Table 62 Summary of Participants' Results and Performance in S1

Lab. Code	S1-Ag	S1-Al	S1-As	S1-B	S1-Ba	S1-Bi	S1-Ca	S1-Cd	S1-Cr	S1-Cs	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
H.V./R.V.	0.0127	158	3.645	1.38	1.06	0.0108	4770	0.127	0.95	0.0165	15.4
A.V.	0.0154	Not Set	3.645	1.14	1.24	0.0120	5310	0.128	0.95	0.0203	15.4
1	<0.02	77	3.6	2.7	1.1	0.012	5200	0.14	0.45	NT	15
2	<0.02	NT	3.76	NT	1.46	NT	5312	0.12	0.77	NT	14.12
3	<0.2	50.9	3.66	0.44	1.18	0.0082	5100	0.111	0.552	0.0217	14.4
4	0.0156	42.9	3.84	1.06	1.06	0.0118	5400	0.115	0.484	0.0163	15.2
5	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
6	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
7	0.02	40	3.4	1	1.1	0.012	5300	0.126	0.34	0.015	15.1
8	0.016	117	3.69	NT	1.48	NT	5200	0.133	1.09	0.022	14.3
9	0.013	160	4.2	1.2	1.5	0.013	5460	0.11	1.1	0.028	16
10	0.018	43.2	3.52	1.24	1.33	0.013	5520	0.212	0.685	0.019	14.6
11	<0.050	172	3.34	1.14	1.48	<0.050	5380	0.14	1.03	NT	14.2
12	0.01	29.4	4.3	1.2	1.02	0.02	5000	0.17	0.75	<0.05	13.3
13	NT	NT	3.5	NT	1.1	NT	NT	<1	<1	NT	14.1
14	<0.1	34.2	3.61	<5	1.1	<0.1	5480	0.13	0.86	<0.1	13.5
15	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT

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

Lab. Code	S1-Fe	S1-Hg	S1-Inorganic As	S1-K	S1-La	S1-Li	S1-Mg	S1-Mn	S1-Na	S1-Ni
Units	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg
H.V./R.V.	104	0.1345	0.16	1240	0.031	0.148	1620	4.98	11700	0.462
A.V.	104	0.1345	Not Set	1370	Not Set	Not Set	1730	4.98	12300	0.462
1	93	0.12	0.21	1330	NT	0.14	1630	5	11500	0.27
2	98.58	0.14	NT	1373	NT	<0.20	1752	4.72	12340	0.92
3	97.4	0.116	NR	1300	0.0286	<0.3	1770	4.58	11000	0.391
4	96	0.137	NT	1420	0.0311	0.105	1760	4.71	12700	0.216
5	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
6	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
7	88	0.13	0.23	1420	0.03	0.11	1840	5	12900	0.17
8	95.3	0.13	NT	1270	0.049	0.15	1790	4.8	12500	0.41
9	110	0.15	NR	1420	0.12	0.23	1780	5.2	12700	NR
10	97.6	0.142	NT	1350	NT	0.152	1690	4.67	12300	0.359
11	102	0.136	NT	1435	NT	NT	1675	4.98	12600	0.585
12	70	0.14	0.41	1430	<0.05	0.079	1660	4.91	12000	0.4
13	80.6	0.11	NT	NT	NT	NT	NT	4.9	NT	<1
14	77.5	0.07	NT	1350	<0.1	0.1	1680	4.94	12300	0.41
15	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT

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

Table 62 Summary of Participants' Results and Performance in S1 (continued)

Lab. Code	S1-P	S1-Pb	S1-Rb	S1-S	S1-Sb	S1-Se	S1-Sn	S1-Sr	S1-U	S1-V	S1-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./R.V.	6380	0.276	NA	NA	NA	2.498	0.235	59.8	0.00617	0.252	66.4
A.V.	6030	0.305	0.652	9600	Not Set	2.498	0.275	63.0	0.00672	Not Set	67.8
1	6000	0.29	NT	9100	<0.01	2.7	0.23	63	0.006	0.13	68.5
2	6382	0.3	NT	NT	<0.05	2.45	0.3	NT	NT	0.21	63.87
3	5900	0.317	0.65	NR	0.0513	2.69	0.22	61.8	0.0067	0.0604	73.6
4	6190	0.291	0.646	10400	<0.10	2.51	0.29	61.3	0.0066	0.095	67.3
5	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
6	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
7	6240	0.325	0.67	10900	<0.02	2.69	0.22	68	0.006	0.09	70
8	6100	0.31	0.81	10300	NT	NT	NT	60.1	0.009	0.65	66.5
9	6400	0.29	1.1	11100	0.018	2.3	0.32	64	0.007	0.19	71
10	5670	0.354	NT	8230	0.018	3.03	0.292	65.2	0.007	0.115	70.5
11	5900	0.26	NT	10300	<0.10	2.22	0.466	66.2	NT	0.258	63.4
12	5600	0.26	0.62	9050	<0.01	4.15	0.33	62.3	<0.05	0.22	66.7
13	5610	<1	NT	8490	NT	<5	NT	NT	NT	<1	63.4
14	6310	0.38	0.6	8060	<0.1	2.86	<0.5	57.4	<0.01	<0.5	69.2
15	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT

A.V. = Assigned Value, H.V. = Homogeneity Value, R.V.=Reference 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 S2

Lab. Code	S2-Al	S2-As	S2-B	S2-Ba	S2-Ca	S2-Cd	S2-Co	S2-Cr	S2-Cu	S2-Fe	S2-Iodine
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.	3.06	0.0060	1.10	0.377	6500	0.0020	0.0080	0.135	3.53	76	NA
A.V.	2.54	Not Set	Not Set	0.398	7700	Not Set	Not Set	0.150	3.72	79.0	Not Set
1	2.6	0.005	1.4	0.36	7400	<0.005	<0.01	0.13	3.6	79	NT
2	NT	<0.10	NT	0.47	8564	<0.01	<0.05	0.14	4.64	82.04	NT
3	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
4	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
5	NT	NT	NT	NT	8730	NT	NT	NT	4.54	92.1	NT
6	2.04	<0.01	0.81	0.42	7320	<0.01	<0.01	0.143	3.2	80	1.24
7	2.3	<0.05	<1	0.38	7520	<0.01	<0.02	0.14	3.76	82	NT
8	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
9	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
10	2.78	0.006	1.07	0.441	7850	0.0014	0.007	0.136	3.87	78.4	NT
11	3	<0.10	0.815	0.388	7420	<0.010	<0.010	0.203	3.34	72	NT
12	2.46	<0.025	1.3	0.37	7000	<0.01	0.01	0.18	3.27	76	1.3
13	NT	NT	NT	<1	NT	NT	NT	<1	3.6	79.6	NT
14	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
15	2.58	<0.01	NT	0.356	7560	0.0015	0.00806	0.145	3.51	74.9	NT

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

Table 63 Summary of Participants’ Results and Performance in S2 (continued)

Lab. Code	S2-K	S2-Mg	S2-Mn	S2-Mo	S2-Na	S2-P	S2-Pb	S2-S	S2-Se	S2-Sr	S2-V	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	mg/kg
H.V.	7830	627	1.10	0.245	2210	4900	NA	NA	0.148	3.28	0.0443	50.2
A.V.	8470	659	1.15	0.255	2290	4690	Not Set	2030	0.146	3.42	Not Set	53.2
1	7600	620	1.2	0.25	2200	4650	<0.005	2000	0.16	3.3	0.049	53
2	8686	704.2	1.23	0.23	2466	5213	<0.02	NT	0.13	NT	<0.10	52.86
3	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
4	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
5	9060	760	1.34	NT	2830	NT	< 0.05	NT	< 0.5	NT	NT	57.4
6	8810	660	0.81	0.26	1900	4770	<0.01	2120	0.14	3.3	0.053	54
7	8760	700	1.21	0.26	2430	4930	<0.05	2600	0.13	3.7	<0.05	56
8	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
9	NR	NR	NR	NR	NR	NR	NR	NR	NR	NT	NR	NR
10	8170	632	1.1	0.272	2300	4680	0.005	1860	0.143	3.66	0.052	52.5
11	8240	612	1.1	0.273	2240	4530	<0.030	2060	0.153	3.65	0.061	47.8
12	8130	630	1.06	0.25	2200	4400	<0.01	1980	0.19	3.2	<0.10	53
13	NT	NT	1.1	<1	NT	4500	NT	1910	<5	NT	<1	53.1
14	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT	NT
15	8570	642	1.18	0.244	2220	4740	<0.01	NT	0.143	3.12	0.0575	50.84

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

Extraction Method

Some good evidence was found in the previous and present studies of the importance of using a digestion temperature of 170ºC or higher and/or a digestion regime which involves a high ratio HCl (mL)/sample size (g) when total elements except Al are being analysed.

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] In previous NMI PT studies participants used various extraction methods and the results produced were compatible except for Al, Cr, Ni and V in most types of food. 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 (> 170ºC) and/or hydrofluoric acid) is usually recommended for the complete extraction of these elements.

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

The method descriptions provided by participants are presented in Tables 1 to 4. Seven laboratories reported results in both samples and all used the same extraction regime for both samples.

The amount of sample taken for analysis by most participants was 0.5 g. One reported using 0.1 g. Caution should be exercised when a small sample size is taken for analysis as this might not be representative of the whole sample.

Four participants used a digestion temperature higher than 170°C; two participants digested their samples at 85°C.

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

Laboratory 9 reported: “Sample digested to completion by microwave”.

Aluminium is one of the most difficult elements to analyse in food samples. In previous PT studies, no assigned values could be set in the wheat, oyster tissue, freeze dried liver 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 prawn tissue were variable (CV_{rob}29%) and had a bimodal distribution (Figure 59). The robust average of reported results for Al in S1 was 76 mg/kg while the NMI information value was 158 mg/kg.

As the request was for “digestion until extraction was complete” the values closest to the “true” value are most likely those reported by Laboratories 8, 9 and 11 whose results were 117 mg/kg, 160 mg/kg and 172 mg/kg respectively. These results were also in good agreement with the NMI information value (158 mg/kg). All three laboratories used high digestion temperatures of 200°C and above for Al extraction.

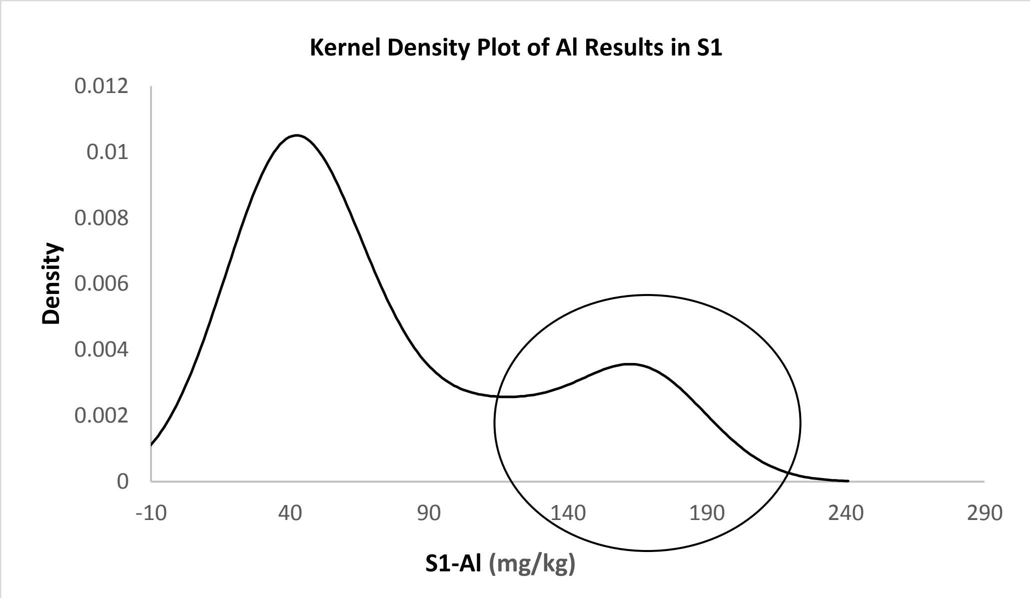


Figure 59 Participants' Results versus Digestion Regime

Lab code	S1 Al Results mg/kg	Digestion Regime					
		Sample Mass (g)	Temp ºC	Time (min.)	HNO_3 (mL)	HCl (mL)	H_2O_2 (mL)
12	29.4	0.5	85	240	5		
14	34.2	1	95	60	2(1:1)	10(1:4)	
7	40	0.5	85	60	2.5	0.5	
4	42.9	0.51	105	60	2.5	0.5	
10	43.2	0.5	110	60	5	1.5	
3	50.9	0.5	95	120	8	1	
1	77	0.5	110-160	45	4		
8	117	0.1	**200**	18	10		2
9	160	0.5	**210**	15	7(1:1)	0.5	
11	172	0.5	**220**	30	5	1	
NMI Information Value	158	0.5	260	30	7	0.5	1

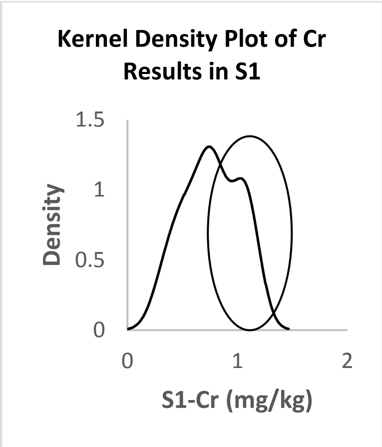
Chromium, Nickel and Vanadium results in S1 had a multimodal distribution, with the laboratories which used more aggressive digestion regimes (high digestion temperature and/or a high ratio HCl/sample size) producing the most satisfactory results.

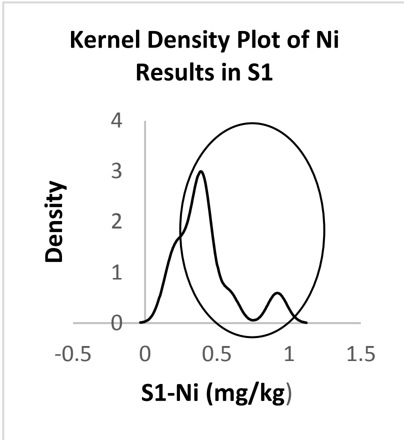
As in previous studies, there was agreement between Cr and Ni results produced by high digestion temperatures and those produced by a high ratio of HCl/sample size (Figure 60).

Table 64 presents the results of measurements of total Cr, Ni and V in S1. The measurements were made by NMI using various mixtures of reagents but the same samples size (0.5 g), the same digestion temperature (95±5ºC) and the same instrumental technique (ICP-MS).

Table 64: NMI Cr, Ni and V Results in S1 vs. Reagents

Reagents	Cr (mg/kg)	Ni (mg/kg)	V (mg/kg)
3 mL HNO_3, 1 mL HCl	0.695	0.362	0.0962
3 mL HNO_3, 3 mL HCl	0.78	0.400	0.0939





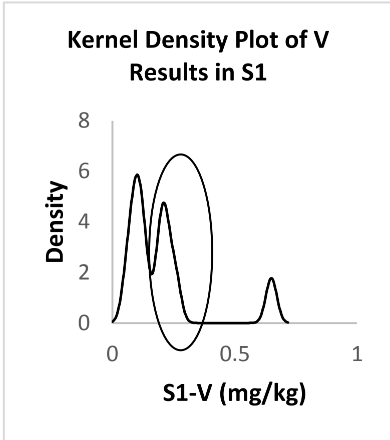


Figure 60 Participants' Results vs. Digestion Regime

Lab. Code	S1 Cr* mg/kg	S1Ni* mg/kg	S1-V* mg/kg	Digestion Regime				
				HCl (mL)/ sample size (g)	Temp (ºC)	Time (min)	Sample Size (g)	Reagents Used
7	0.34	0.17	0.09	1	85	60	0.5	2.5 mL HNO_3, 0.5 mL HCl
1	0.45	**0.27**	0.13	0	110-160	45	0.5	4 mL HNO_3
4	0.484	0.216	0.095	1	105	60	0.51	2.5 mL HNO_3, 0.5 mL HCl, 2 mL H_2O
3	0.552	**0.391**	0.0604	**2**	95	120	0.5	8 mL HNO_3, 1 mL HCl, 2 mL H_2O_2
10	**0.685**	**0.359**	0.115	**3**	110	60	0.5	5 mL HNO_3, 1.5 mL HCl
12	**0.7**	**0.4**	**0.22**	**10**	85	240	0.5	5 mL HNO_3, 5 mL HCl
14	**0.75**	**0.41**	NR	**2**	95	60	1	7 (1:1) mL $HNO_3$10 (1:4) mL HCl, 2 mL H_2O_2
11	**0.86**	**0.585**	**0.258**	2	**220**	30	0.5	5 mL HNO_3, 1 mL HCl
8	**1.03**	**0.41**	0.65	0	**200**	18	0.1	10 mL HNO_3
9	**1.09**	NR	**0.19**	1	**210**	15	0.5	7 (1:1) mL HNO_3, 0.5 mL HCl
R.V./I.V.	**0.95**	**0.462**	**0.252**	1	**270**	60	0.5	3 mL HNO_3, 0.5 mL HCl, 1 mL H_2O_2

R.V.= Reference Value ; I.V.= InformationValue; * The results in bold are satisfatory except for Vanadium, Vanadium results in bold are those which are in good agreement with the indicative value.

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

Instrumental Techniques

Plots of participants' results/ performance with the instrumental technique used are presented in Figure 61.

Aluminium, Chromium, Nickel and Vanadium No association between Al, Cr, Ni and V results in S1 and the instrumental technique used was apparent (Figure 61).

Arsenic level in Sample S2 was below the reporting level of most participating laboratories. Only two results were reported for this test: one from ICP-MS/MS-ORS measurements with O_2 as reaction gas and one from ICP-MS-CRI measurements with high energy He as collision gas. Both results were in good agreement with each other and with a homogeneity value of 0.006 mg/kg.

All the results reported for As in the prawn sample returned satisfactory z-scores (Figure 61).

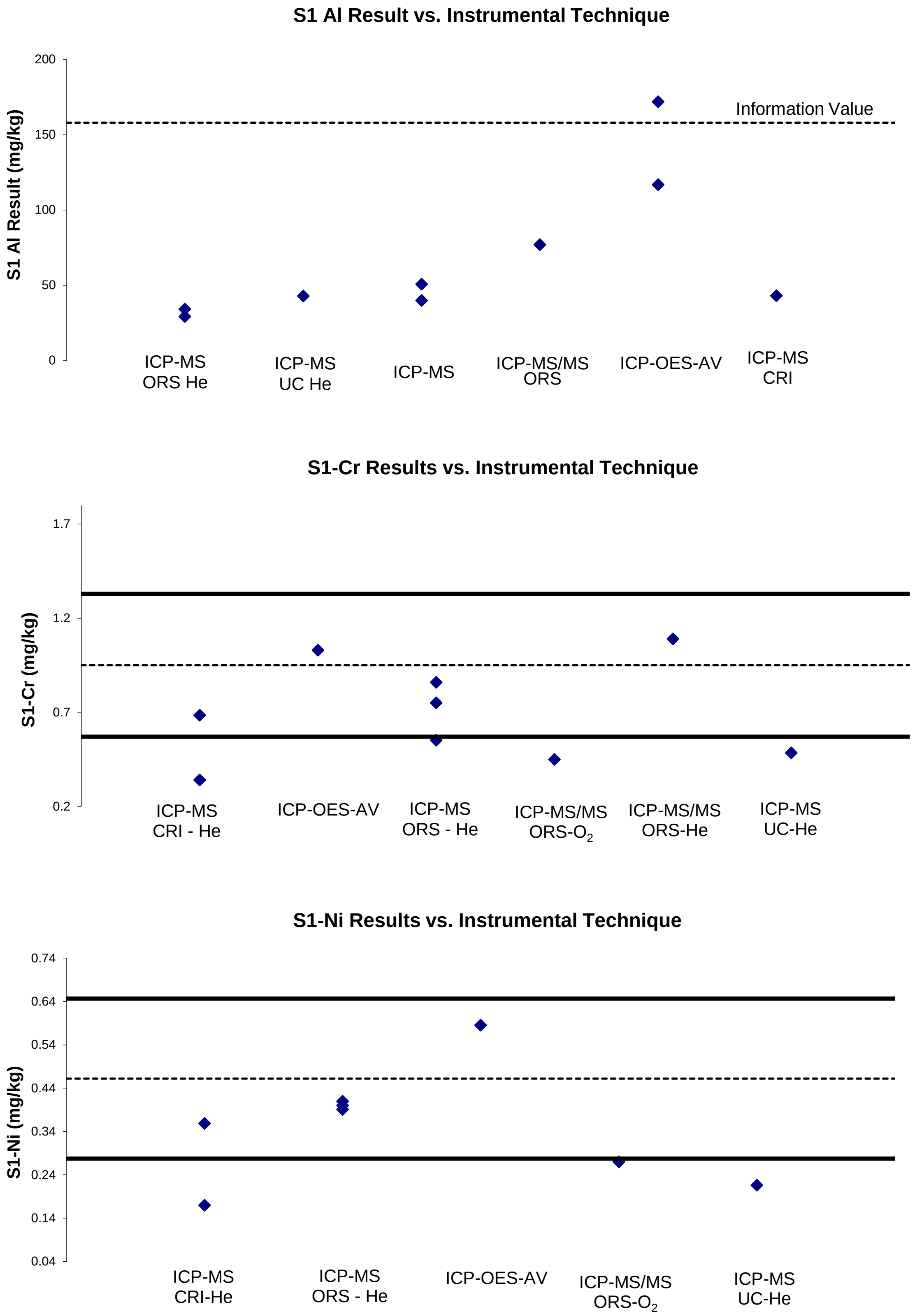
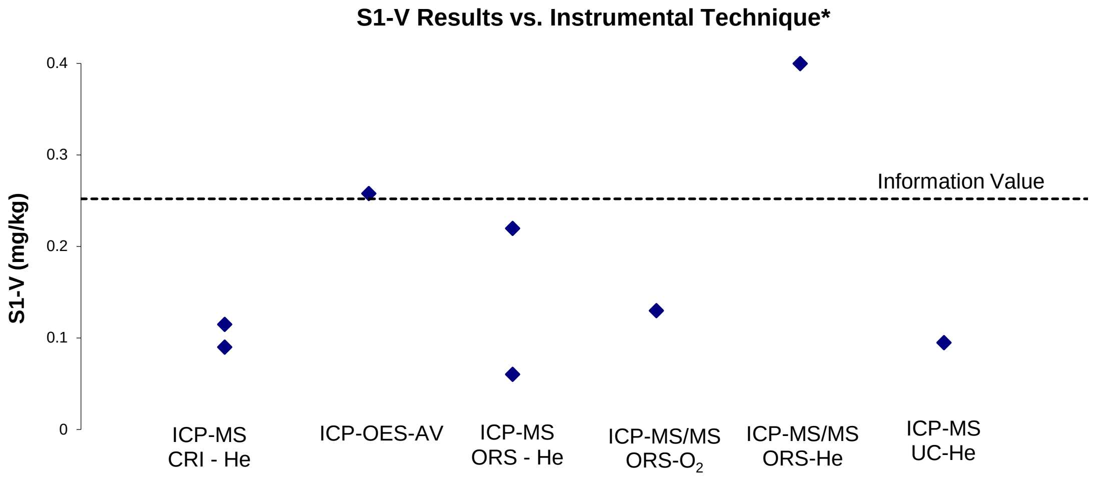
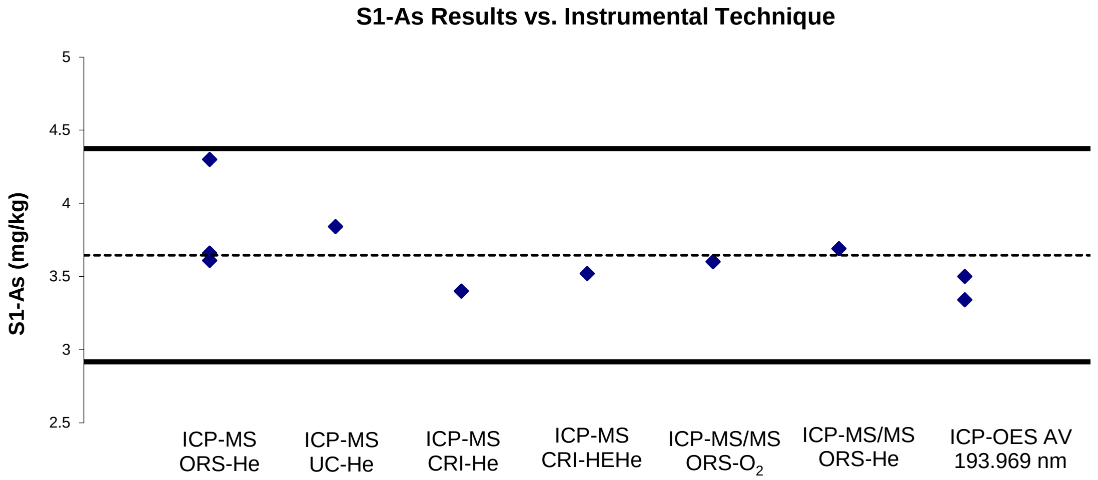


Figure 61: Participants' Results/Performance vs. Instrumental Technique



*Result larger than 0.4 mg/kg has been plotted as 0.4 mg/kg.



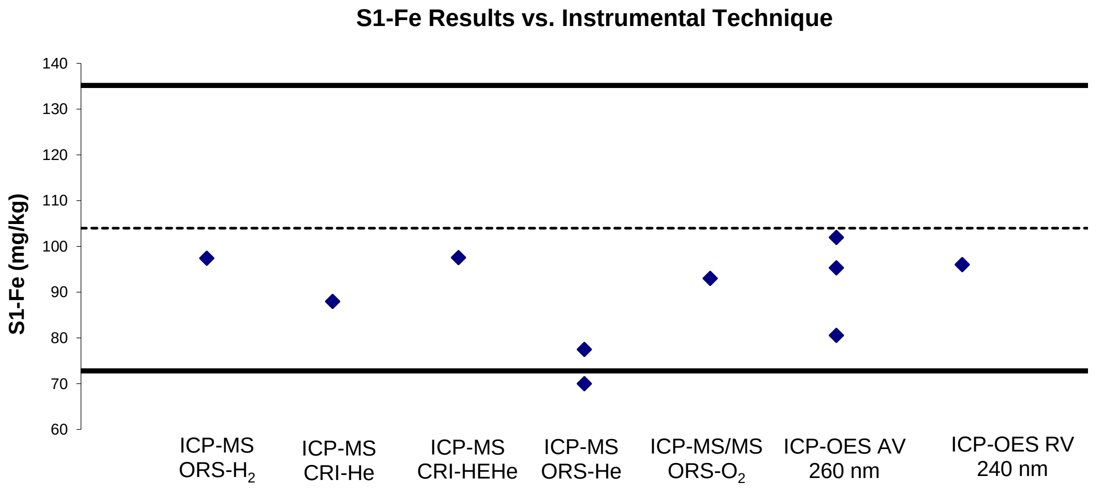
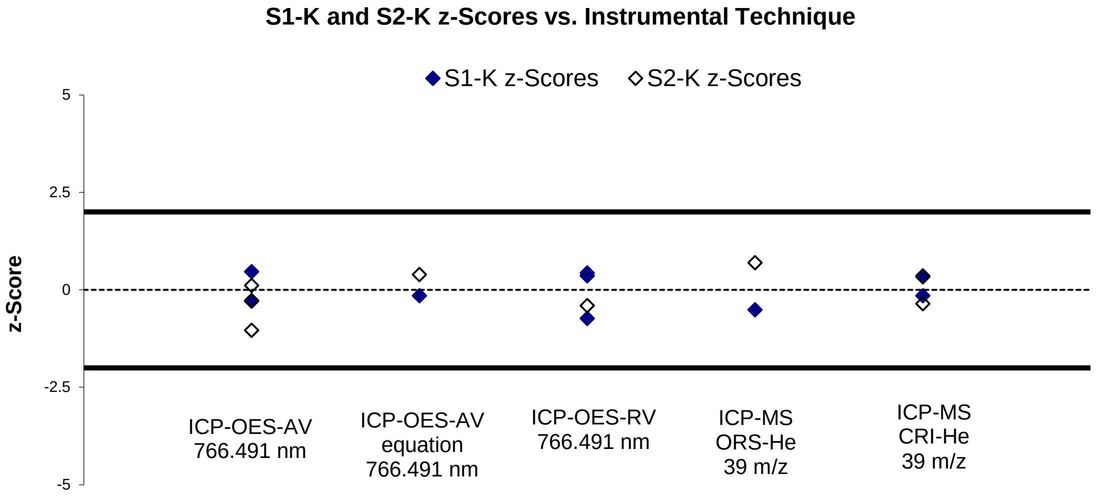


Figure 61: Participants' Results/Performance vs. Instrumental Technique (continued)



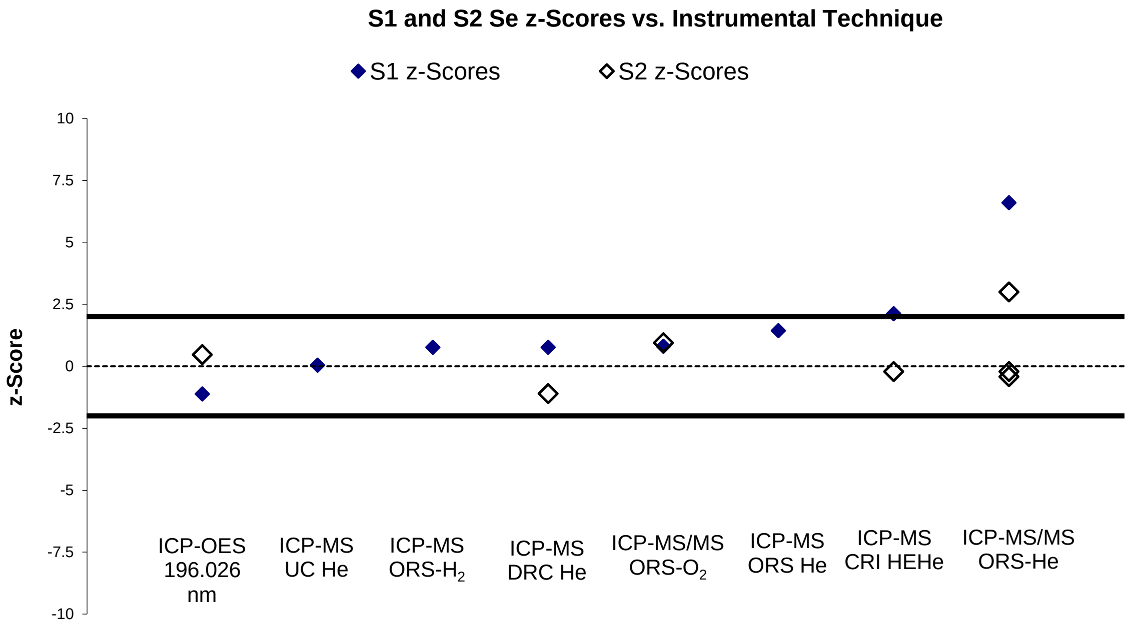


Figure 61: Participants' Results/Performance vs. Instrumental Technique (continued)

Boron Only 5 results were reported in S2 and all were in agreement with each other centred on 1.08 mg/kg. One laboratory used ICP-OES with wavelength 249.678 nm. Boron measured at 249.678 nm can have significant spectral interferences from Fe 249.771 nm.

Cadmium level in S2 was low. Only two participants reported their result. One measured Cd by
ICP-MS/MS-ORS and O_2 as reaction gas and one by ICP-MS. The two results were in relatively good agreement with each other and with the homogeneity value (0.0020 ±0.00030 mg/kg).

Cobalt Three results were reported for Co in S2 and all were in agreement with each other centred on a value of 0.0084 mg/kg. All results were produced using ICP-MS measurements in collision mode.

Iodine Two laboratories reported results for Iodine in the infant formula sample. One reported using TMAH digestion and ICP-MS determination for iodine measurements. The two results were in good agreement with each other.

Iron Most participants used for Fe measurements ICP-MS with various collision or reaction gases (Figure 61).

Potassium Participants used a wide variety of measurement techniques in the two study samples and all produced comparable results (Figure 61).

Selenium Apart from molecular and polyatomic interferences whose effects may be reduced by using a collision reaction cell, matrix effects are another main factor that can hamper accurate measurements of elements in food samples when complete digestion cannot be achieved. Matrix effects are common in food analyses using ICP-MS; they take place in the plasma and consist of signal enhancement caused by charge transfer reactions from charged carbon species to atoms like Se with a lower ionization potential.[19] One participant reported high unsatisfactory z-scores in both study samples, they may have not overcome the interference problem (Figure 61).

Participants' Results and Analytical Methods for Inorganic As

Inorganic Arsenic was included as a pilot program. Figure 62 presents plots of participants' results versus method used.

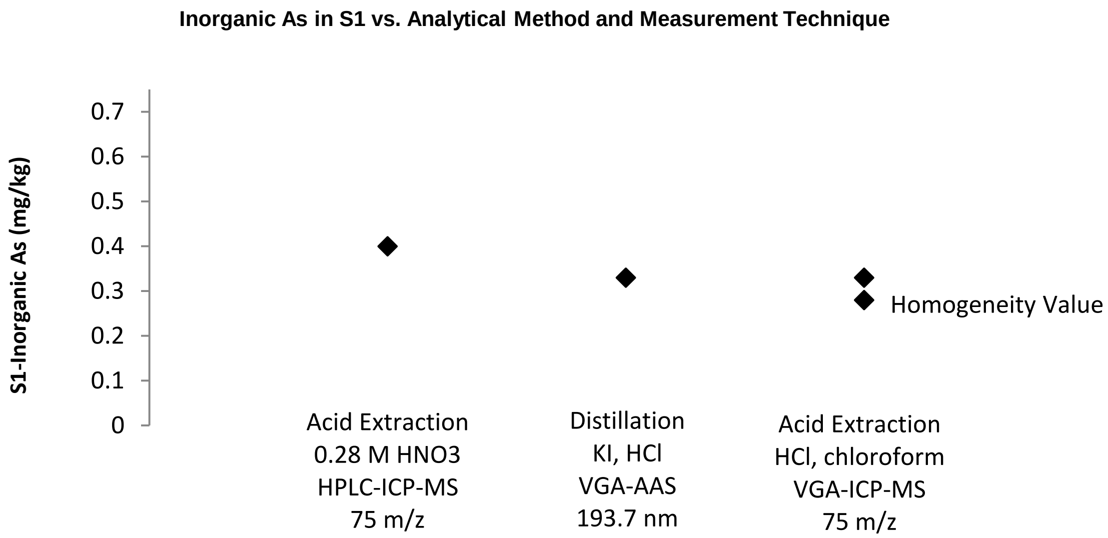


Figure 62: Participants' Results for Inorganic As in S1 vs. Method

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

AQA 19-10 is the thirteenth NMI study of elements in food. The participants' performance in measurements of trace elements in food over time is presented in Figure 63. Despite differences in matrices and laboratories, performance has remained fairly constant.

Over time laboratories should expect at least 95% of its scores to be $|z| \leq 2$. 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 anlyte over time provides much more useful information than a single z-score.

7.7 Reference Materials and Certified Reference Materials

Proficiency testing and matrix matched control samples taken through all steps of the analytical process are highly valuable quality control tools for assessing extraction efficiency. Control samples used by participants in this study are presented in table 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	Apple Leaves NIST 1515, Oyster Tissue NIST 1566b, Rice Flour NIST 1568a, Mushroom CS-M-3, Dairy Proficiency – Micronutrients NRS Metals in Liver, FAPAS MBM and Meat Paste-Global Proficiency
5	SRM 1849a
7	In house reference samples, NIST 2976, NIST 1549
8	DORM-3 (fish protein) and NIST 2976
9	DORM-2
10	NIST SRM 1548a – Typical diet, NIST SRM 1849A – Infant formula, DORM3 – Fish Protein
15	Agal3, agal4, agal6, agal7, pharmtab

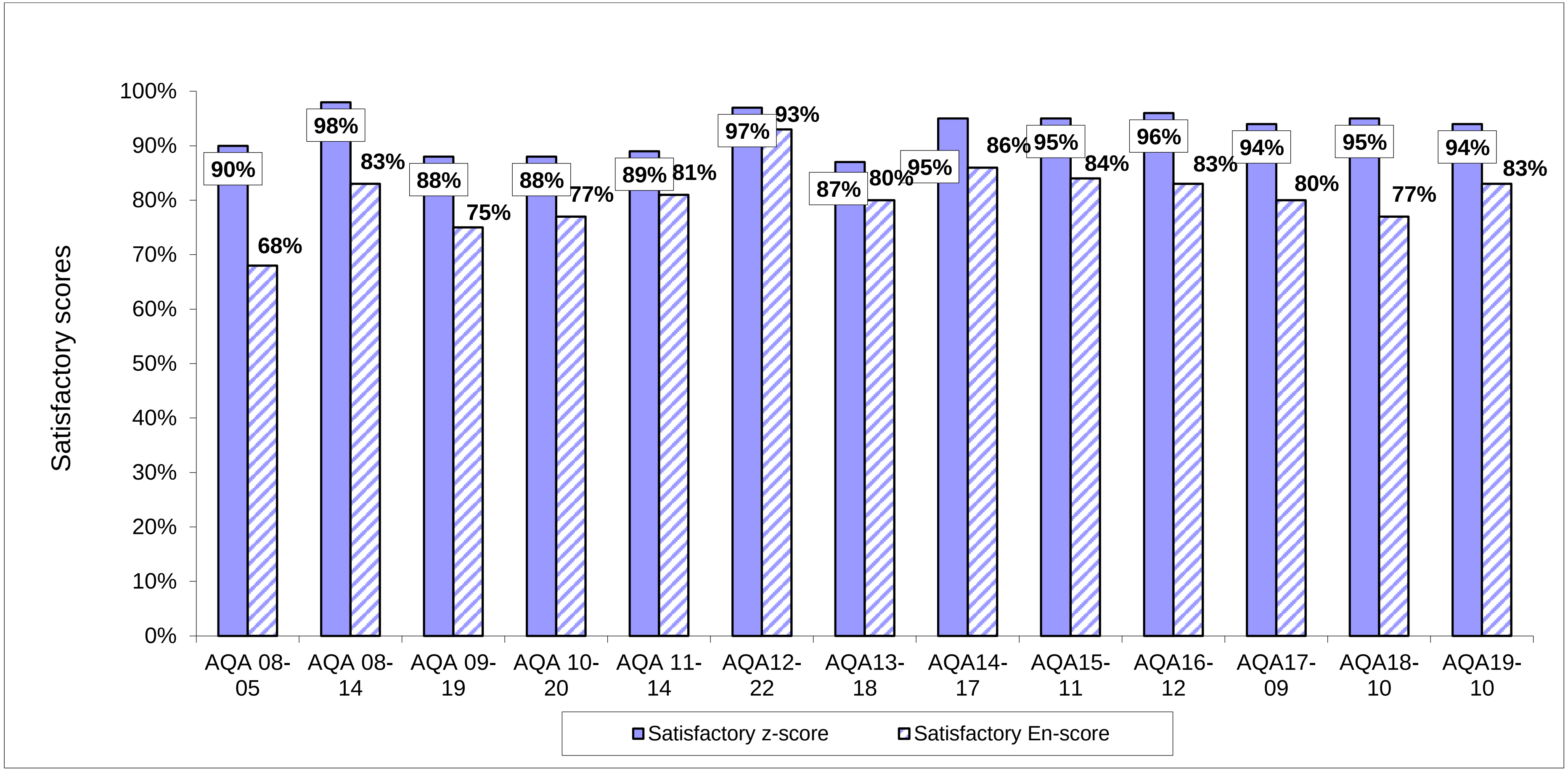


Figure 63 Participants' Performance over Time

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APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING

A1.1 Sample Preparation

Sample S1 – was freeze dried prawn matter, a reference material previously prepared by NMI.

Sample S2 – was infant formula, prepared specifically for this study.

A1.2 Sample Analysis and Homogeneity Testing

Although sample S1 was formerly tested for homogeneity by NMI, a partial homogeneity test was still conducted for both samples. The results from the partial homogeneity test are reported in this study as the homogeneity value. No homogeneity testing was conducted for Rb, S, Sb in S1 and iodine, Pb and S in S2. For Al, As, Cr, Cu, Fe, Hg, Mn, Ni, Se, Sn, V and Zn, homogeneity values are the reference and information values (see Appendix 3).

Sample Analysis for Total Elements in S1 and S2

Approximately 0.5 g of sample was weighed and digested at 100ºC for 2 hours with 3 mL HNO_3 and 1 mL HCl. After digestion, each sample was diluted to 40 mL with ultra-high purity water and then further diluted as necessary for ICP-MS determination. A summary of the instruments used and the ion monitored for each analyte is given in Table 66.

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

Analyte	Instrument	Internal Standard	Reaction/ Collision Cell (if applicable)	Cell Mode/ Gas (if applicable)	S1 Final Dilution Factor	S2 Final Dilution Factor	Ion
Ag	ICP-MS	Rh	ORS	He	400	NA	107 m/z
Al	ICP-MS	Rh	NA	NA	NA	400	27 m/z
As	ICP-MS	Rh	ORS	He	NA	400	75 m/z
B	ICP-MS	Rh	NA	NA	400	400	11 m/z
Ba	ICP-MS	Rh	ORS	He	400	400	137 m/z
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	400	400	114 m/z
Co	ICP-MS	Rh	ORS	He	NA	400	59 m/z
Cr	ICP-MS	Rh	ORS	He	NA	400	53 m/z
Cs	ICP-MS	Rh	ORS	He	400	NA	133 m/z
Cu	ICP-MS	Rh	ORS	He	NA	400	65 m/z
Fe	ICP-MS	Rh	ORS	He	NA	400	56 m/z
K	ICP-MS	Rh	ORS	He	400	400	39 m/z
La	ICP-MS	Rh	ORS	He	400	NA	139 m/z
Li	ICP-MS	Rh	ORS	He	400	NA	7 m/z
Mg	ICP-MS	Rh	ORS	He	400	400	24 m/z
Mn	ICP-MS	Rh	ORS	He	NA	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
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	NA	400	78 m/z
Sn	ICP-MS	Rh	ORS	He	400	NA	118 m/z
Sr	ICP-MS	Rh	ORS	He	400	400	88 m/z
U	ICP-MS	Ir	ORS	He	400	NA	238 m/z
V	ICP-MS	Rh	ORS	He	NA	400	51 m/z

Zn	ICP-MS	Rh	ORS	He	NA	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 'ISO13258:2015(E), Statistical methods for use in proficiency testing by interlaboratory comparisons – Annex C[7] the uncertainty was estimated as:

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

where:

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

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

A worked example is set out below in Table 67.

Table 67 Uncertainty of Assigned Value for K in Sample S1

No. results (p)	7
Robust Average	1370 mg/kg
$S_{rob\ av}$	70 mg/kg
$u_{rob\ av}$	21 mg/kg
k	2.228
$U_{rob\ av}$	50 mg/kg

The assigned value for **K** in Sample S1 is **1370 ± 50 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 10).

A worked example is set out below in Table 68.

Table 68 z-Score and E_n-score for K result reported by Laboratory 9 in S1

K Result mg/kg	Assigned Value mg/kg	Set Target Standard Deviation	z-Score	E_n-Score
1420 ± 104	1370 ± 50	10% as CV or 0.1 x 1370= 137 mg/kg	$z = \frac{(1420 - 1370)}{137}$ z = 0.36	$En = \frac{(1420 - 1370)}{\sqrt{104^2 + 50^2}}$ E_n = 0.43

APPENDIX 3 – REFERENCE VALUES

A.3.1 Description of Method of Analysis

All analytes were quantified by the method of standard additions using ICP-MS. NIST 3100 series primary calibration materials were used, see table below for details, and these were diluted gravimetrically to working concentrations.

Analyte	Standard Name	Lot No.
Al	NIST 3101a	140903
As	NIST 3103a	100818
Cr	NIST 3112a	170630
Cu	NIST 3114	121207
Fe	NIST 3126a	140812
Mn	NIST 3132	050429
Ni	NIST 3136	120619
Se	NIST 3149	100901
V	NIST 3165	160906
Zn	NIST 3168a	120629

Approximately 0.5 g of sample was weighed into a PTFE microwave vessel along with a mixed internal standard and with 3 mL HNO3 (69%) + 1 mL H2O2 (30%) + 0.5 mL HCl (35%) digestion reagents. Samples were then digested in a microwave digester (Ultrawave, 260 °C, 90 bar, 30 min ramp, 30 min hold). Digests were then diluted to 20 mL with UHP water to produce a clear and colourless solution free from particulates. This solution was then gravimetrically separated into 'spiked' and 'unspiked' solutions with gravimetric additions of standards to the 'spiked' solutions. Each experimental batch contained CRMs and method blanks prepared using the same procedures. Isotope ratios, using incurred and spiked internal standards, were measured by ICP-SF-MS (Element 2) using medium resolution (Al, Cr, Fe, Mn, Ni and V) and high resolution (As, Cu, Se and Zn). For all ICP-MS measurements of standard addition samples the 'spiked' solutions were measured bracketed on either side by the 'unspiked' solution.

A.3.2 Reference Values

The reference values and associated measurement uncertainty estimates for AQA 19-10 Sample S1 are presented below. The reference values come from the analysis of 12 bottles in duplicate. Measurement uncertainty is given as a 95% level of confidence. Measurements are based on the sample as received (wet mass), no specific correction or uncertainty for moisture content variation has been made.

Sample	Analyte	Reference Value (mg/kg)	Expanded Uncertainty (95%) (mg/kg)	Relative Expanded Uncertainty	Coverage Factor (95%)
AQA 19-10 S1	As	3.645	0.081	2.2%	2.07
	Cr	0.95	0.16	17%	2.03
	Cu	15.4	1.0	6.5%	2.01
	Fe	104	10	9.6%	2.09

Mn	4.98	0.27	5.4%	2.07
Ni	0.462	0.061	13%	2.12
Se	2.498	0.067	2.7%	2.04

A.3.3 Information Values

Information values and associated measurement uncertainty estimates for Al, V and Zn are also provided below. These information values come from the analysis of 12 bottles in duplicate. Measurement uncertainty is given as a 95% level of confidence. Measurements are based on the sample as received (wet mass), no specific correction or uncertainty for moisture content variation has been made.

Sample	Analyte	Information Value (mg/kg)	Expanded Uncertainty (95%) (mg/kg)	Relative Expanded Uncertainty	Coverage Factor (95%)
AQA 19-10 S1	Al	158	11	7.0%	2.11
	V	0.252	0.021	8.3%	2.13
	Zn	66.4	4.0	6.0%	2.10

Based on matrix, CRM recoveries for these elements, see table below, insufficient evidence is currently available to confidently state that total recoveries have been achieved. The digestion method (see §Description of Method of Analysis) should represent a 'practical' total extraction (i.e. not using hydrofluoric acid). It should be further noted that the low recoveries are for NIST 1573a, tomato leaves, a matrix expected to contain a significantly higher silica content than the AQA 19-10 S1 sample. Further investigation will be conducted on these elements for reference material certification.

Analyte	NIST 1548a Typical Diet		NIST 1573a Tomato Leaves	
	Certified Mass Fraction Value (mg/kg)	Recovery (%)	Certified Mass Fraction Value (mg/kg)	Recovery (%)
Al	72.4 ± 1.52	100.5	598 ± 12	87.1%
V	N/A	N/A	0.835 ± 010	90.4%
Zn	24.6 ± 1.79	96.1	30.9 ± 0.7	88.0%

A.3.4 Homogeneity Assessment

Homogeneity was assessed based on duplicate analysis of 12 bottles and is included as a component of the reported uncertainty.

A.3.5 Stability Assessment

Stability was not assessed.

A.3.6 Reference Value 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 primary calibration material, gravimetric sample preparation, homogeneity, method trueness and method precision. Some variation in moisture content is expected to be covered in the method precision term but has not been specifically considered. Measurement uncertainty is reported as a 95% level of confidence.

A.3.7 Statement of Traceability

The reference values given in this report rely on gravimetric sample preparation and elemental quantification by ICP-MS. Gravimetric measurements were calibrated using Australian standards for mass and are traceable to the SI unit for mass (kg). ICP-MS measurements were calibrated with standard addition and are traceable to (i) the SI unit for mass (kg) through the primary calibration standards certified by NIST (USA) and (ii) the SI unit for amount of substance (mol) through gravimetric measurement and data for isotopic composition and relative atomic mass. Isotopic composition is traceable to IUPAC published data.

APPENDIX 4 – NMIA MX009 CERTIFICATE

Australian Government

National Measurement Institute

Report ID: MX009.2017.04
20 April 2017
Accredited for compliance with ISO Guide 34

NATA
WORLD RECOGNISED ACCREDITATION

CERTIFICATE OF ANALYSIS

FOR CERTIFIED REFERENCE MATERIAL

NMIA MX009

Trace Elements in Freeze-Dried Prawn Tissue

Batch No. 2008.01

(i) Description of the material

Certified reference material MX009 is a freeze-dried prawn tissue powder bottled in units of approximately 10 g. A certified value is provided for the mass fraction of Hg in MX009. Information values are given in the appendix for the mass fractions of As, Ba, Ca, Cd, Co, Cu, Fe, K, Mg, Mn, Na, P, Pb, Se, Zn and for the mass fraction of moisture.

(ii) Certified property values

The certified value for Hg in MX009 is given in Table 1. The mass fraction of Hg is given on a dry mass basis and on an undried basis. Dry mass basis measurements give a more accurate characterization of MX009 as the analyte mass fractions are not affected by moisture content variation over time. Instructions for use of dry mass basis and undried basis certified values are described in Section (v).

Table 1: Certified mass fraction values for MX009.
Measurement uncertainty is reported as a 95% coverage interval.

	Mass fraction (dry mass basis)	Mass fraction (undried basis)
	µg/kg	µg/kg
Hg	148.3 ± 5.1	134.5 ± 9.0

Certified values were determined by isotope dilution ICP-MS analysis and are traceable to SI units. Details of the analytical methodology and measurement uncertainty estimation are provided in Sections (vii) and (x).

(iii) Expiration of certification

The MX009 certified values are valid until **15 March 2022** provided the material is stored and handled in accordance with the instructions given in this certificate. MX009 may be reassessed prior to the expiration date to investigate extension of the certification period.

(iv) Intended use

Certified reference material MX009 is intended to be used to verify and/or validate analytical methods for elemental analysis in prawn tissue, marine biota and similar sample types.

(v) Instructions for use

Storage— MX009 should be stored tightly sealed in the original bottle at 20°C ± 5°C. Dry mass basis measurements are not affected by the humidity of storage conditions, whereas undried basis measurements rely on avoiding extremes of humidity. Storage in a desiccator is therefore not recommended if undried basis results are to be used. MX009 should not be exposed to direct sunlight or intense sources of radiation.

Subsampling— The contents of the MX009 bottle should be mixed thoroughly shortly before subsampling. For trace element analysis, a minimum sample size of 0.5 g (dry mass basis) should be used for MX009 analysis. Trace element certified values may not be valid for sample sizes less than 0.5 g.

Contamination minimisation— MX009 should only be opened to the atmosphere in a clean environment such as a ULPA-filtered laminar flow hood. For trace element analysis, MX009 should be subsampled and handled in such a way as to avoid contamination of both the bulk material and the subsample.

Use of dry mass basis certified values— To remove the effect of moisture content variation on trace element mass fraction measurements, correction for moisture content should be applied. Gravimetric moisture content determination should be performed on a separate portion of MX009 to that used for trace element analysis to avoid contamination and/or loss of analytes through vaporization. A sample size not less than 0.5 g (dry mass basis) should be used for moisture content measurement. The drying protocol for MX009 is oven-drying at 100°C for 24 hours. Longer oven drying should not be used as mass loss will continue but does not correspond to loss of moisture from the reference material. The moisture content measured at time of analysis should then be used to calculate the moisture content correction factor to convert undried basis measurements to dry mass basis measurements.

Use of undried basis certified values— Where correction for MX009 moisture content is not applied to analytical results, comparison can be made to the undried basis certified values. In this case, possible moisture content variation over time is accounted for in the measurement uncertainty estimate of the certified value. Consequently, the relative expanded uncertainty is larger for the undried basis certified values than for the dry mass basis certified values.

(vi) Production of MX009

MX009 was prepared from fresh prawns (approximately 300 kg) collected from the Central Coast, Queensland, Australia. The prawns were cooked and shelled then freeze-dried. The prawn tissue was then ground in an industrial blender fitted with stainless steel blades. The ground prawn tissue was passed through a 212 μm sieve. The sieved powder was thoroughly homogenized in a drum-hoop mixer and then bottled into 10 g portions using a rotary sample divider. The entire production batch was gamma irradiated (25 kGy minimum) to prevent microbial activity. Each unit of MX009 is labelled with a unique bottle number.

(vii) Analytical method

The certified trace element mass fraction of Hg in MX009 was measured by double isotope dilution ICP-MS [1]. ^{201}Hg-enriched mercury was used as the isotope dilution internal standard added prior to sample digestion. IDMS measurements were made using a sample size of 0.5 g. The sample was digested using nitric acid and hydrogen peroxide (1:1) and microwave heating in sealed high-pressure vessels.

Hg isotope ratio measurements were made using ICP-SF-MS. ^{202}Hg/^{201}Hg isotope ratio was used for isotope dilution quantification. Instrumental analysis was optimised to minimize the systematic error from spectral interference, linearity, mass bias and detector deadtime.

Analysis was calibrated using a high purity single element standard solution traceable to SI units (NIST SRM 3133 lot no. 061204). The internal standard used was Oak Ridge National Laboratory Hg201 (batch no. 176506, ^{201}Hg = 98.11%).

(viii) Homogeneity assessment

Homogeneity assessment for MX009 certified values was conducted according to ISO Guide 35 [2]. Fifteen sample bottles of MX009 were selected in a stratified random sampling plan to assess the homogeneity of the reference material batch. Measurements were made on duplicate subsamples taken from these 15 bottles using the method described in Section (vii). Analysis of variance (ANOVA) was used to estimate the measurement uncertainty due to method precision and reference material inhomogeneity.

(ix) Stability assessment

The main source of instability for MX009 is variation of moisture content in transport and/or storage which will directly affect elemental mass fraction values. Applying a moisture content correction and using dry mass basis certified values is recommended to negate the effect of moisture content variation. Undried basis certified values include a measurement uncertainty contribution for variation in the moisture content of MX009 under recommended storage conditions.

The long-term stability of certified values has been demonstrated through periodic analysis of MX009 both at NMIA and by interlaboratory testing. Statistical assessment following ISO Guide 35 [2] showed that the linear regression model for stability assessment data had a gradient not statistically different from zero ($\alpha = 0.05$). The gradient of the Hg linear regression stability model was -0.0005 mg/kg/year.

The long term stability of MX009 was reassessed in January 2014 and March 2017. Based on this long term stability testing this certificate has been re-issued with an extended expiry date.

(x) Measurement uncertainty

Measurement uncertainty was estimated according to international standards [2,3] and NMIA standard procedures. All factors that could reasonably be expected to affect the measurement result were identified and the standard uncertainty of each was estimated from experimental data. The standard uncertainty estimates were combined and expanded to a 95% coverage interval using the coverage factors given in Table 2.

For MX009 certified values, the measurement uncertainty contributors examined were the calibration standard, gravimetric preparation, isotopic composition, moisture content, isotopic equilibration, isotope ratios measurements, method precision, method bias, between-bottle homogeneity and long-term stability.

Table 2: Coverage Factors for MX009 Certified Values.

	Coverage Factor	
	Mass fraction (dry mass basis)	Mass fraction (undried basis)
Hg	1.98	2.36

(xi) Metrological traceability

Certified values are traceable to the SI units for mass (kilogram) and amount of substance (mole). Gravimetric preparation is traceable to the SI kilogram through balance calibrations. Isotope ratio measurements are traceable to the mole through double isotope dilution (a primary ratio method). Isotope dilution mass fraction quantification is traceable to SI units through NIST single element calibration solutions. For dry mass basis certified values, moisture content correction is traceable to the method described in Section (v).

(xii) Legal Notice

Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

References

1. L.G. Mackay, C.P. Taylor, R.B. Myors, R. Hearn and B. King; "High accuracy analysis by isotope dilution mass spectrometry using an iterative exact matching technique"; *Accred. Qual. Assur.* (2003) 8:191-194.
2. *Reference materials — General and statistical principles for certification*; ISO Guide 35, 3rd edition (2006).
3. *Evaluation of measurement data — Guide to the expression of uncertainty in measurement*; JCGM 100, 1st edition (2008).

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APPENDIX 5 - ACRONYMS AND ABBREVIATIONS

CRI	Collison Reaction Interface
CV	Coefficient of Variation
CV_{Rob}	Robust Coefficient of Variation
DRC	Dynamic Reaction Cell
HV	Homogeneity Value
HPLC	High Performance Liquid Chromatography
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
IDMS	Isotope Dilution Mass Spectrometry
ISO/IEC	International Organisation for Standardisation / International Electrotechnical Commission
IUPAC	International Union of Pure and Applied Chemists
Max	Maximum value in a set of results
Md	Median
Min	Minimum value in a set of results
MU	Measurement Uncertainty
N	Number of Participants
NATA	National Association of Testing Authorities
NIST	National Institute of Standards and Technology
NMI	National Measurement Institute (Australia)
NR	Not Reported
NT	Not Tested
ORS	Octopole Reaction System
PCV	Performance Coefficient of Variation
PFAS	Polyfluoroalkyl Substances
PT	Proficiency Test
RA	Robust Average
RM	Reference Material
RV	Reference Value
S	Spiked or formulated concentration of a PT sample
SD_{Rob}	Robust Standard Deviation
SI	The International System of Units
SS	Spiked Sample
s^2_{sam}	Sampling variance
s_a/σ	Analytical standard deviation divided by the target standard deviation
SRM	Standard Reference Material (Trademark of NIST)
Target SD	Target standard deviation (symbol: σ)
UC	Universal Cell

APPENDIX 6 - 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						
3	ICP-MS	Rh	ORS	He	1000	107
4	ICP-MS	Rh	UC	He	98.04	109
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Rh	NA	NA	2000	109
8	ICP-MS/MS	None			1200	107
9						
10	ICP-MS	Rh	CRI		80	
11	ICP-OES-AV	NA	NA	NA	30	328.068
12	ICP-MS	Ge 72	ORS		50	107 m/z
13						
14	ICP-MS	Rh 103	ORS	He	50	107
15	NA	NA	NA	NA	NA	NA

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						
3	ICP-MS	Sc	NA	NA	1000	27
4	ICP-MS	Sc	UC	He	98.04	27
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	NA	NA	2000	27
8	ICP-OES-AV	None			2400	167.019
9						
10	ICP-MS	Sc	CRI		80	
11	ICP-OES-AV	NA	NA	NA	30	396.152
12	ICP-MS	Ge 72	ORS		1000	27 m/z
13						
14	ICP-MS	Sc 45	ORS	He	50	27
15	NA	NA	NA	NA	NA	NA

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						
3	ICP-MS	Ge	ORS	He	1000	75
4	ICP-MS	Te	UC	He	98.04	75
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Rh	CRI	He	2000	75
8	ICP-MS/MS	None	ORS	He	1200	75
9						
10	ICP-MS	Rh	CRI	HEHe	80	
11	ICP-OES-AV	NA	NA	NA	30	193.696
12	ICP-MS	Ge 72	ORS		50	75 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	193.696
14	ICP-MS			He	50	75
15	NA	NA	NA	NA	NA	NA

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						
3	ICP-MS	Y	NA	NA	1000	11
4	ICP-MS	Sc	UC	He	98.04	10
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	NA	NA	2000	10
8	ICP-MS/MS	None			1200	11
9						
10	ICP-MS	Rh	CRI		80	
11	ICP-OES-AV	NA	NA	NA	30	249.678
12	ICP-MS	Ge 72	ORS		50	11 m/z
13						
14	ICP-MS	Sc 45	ORS	He	50	11
15	NA	NA	NA	NA	NA	NA

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						
3	ICP-MS	Tb	ORS	He	1000	137
4	ICP-MS	Rh	UC	He	98.04	137
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Tm	NA	NA	2000	137
8	ICP-MS/MS	None	ORS	He	1200	137
9						
10	ICP-MS	Rh	CRI		80	
11	ICP-OES-AV	NA	NA	NA	30	233.527
12	ICP-MS	RH 103	ORS		50	137 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	233.527
14	ICP-MS	Ir 193	ORS	He	50	137
15	NA	NA	NA	NA	NA	NA

Table 74 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						
3	ICP-MS	Tb	NA	NA	1000	209
4	ICP-MS	Tb	UC	He	98.04	209
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Ir	NA	NA	2000	209
8						
9						
10	ICP-MS	Rh	CRI		80	
11	ICP-OES-AV	NA	NA	NA	30	190.171
12	ICP-MS	RH 103	ORS		50	209 m/z
13						
14	ICP-MS	Ir 193	ORS	He	50	209
15	NA	NA	NA	NA	NA	NA

Table 75 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						
3	ICP-MS	Sc	ORS	H2	1000	70
4	ICP-OES-RV	N/A	NA	NA	98.04	184
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	NA	NA	2000	143
8	ICP-OES-RV	None			2400	315.887
9						
10	ICP-MS	Rh	CRI		800	
11	ICP-OES-AV	NA	NA	NA	30	318.127
12	ICP-OES-RV	Y377			100	317.933 nm
13						
14	ICP-OES-AV-equation	Eu 397.2			50	
15	NA	NA	NA	NA	NA	NA

Table 76 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						
3	ICP-MS	Rh	NA	NA	1000	103
4	ICP-MS	Rh	UC	He	98.04	111
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Rh	NA	NA	2000	111
8	ICP-MS/MS	None			1200	111
9						
10	ICP-MS	Rh	CRI		80	
11	ICP-OES-AV	NA	NA	NA	30	214.439
12	ICP-MS	RH 103	ORS		50	111 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	228.802
14	ICP-MS	Rh 103	ORS	He	50	111
15	NA	NA	NA	NA	NA	NA

Table 77 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						
3	ICP-MS	Ge	ORS	He	1000	52
4	ICP-MS	Sc	UC	He	98.04	52
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	CRI	He	2000	52
8	ICP-MS/MS	None	ORS	He	1200	52
9						
10	ICP-MS	Rh	CRI	He	80	
11	ICP-OES-AV	NA	NA	NA	30	205.56
12	ICP-MS	Ge 72	ORS		50	52 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	267.716
14	ICP-MS	Sc 45	ORS	He	50	52
15	NA	NA	NA	NA	NA	NA

Table 78 Instrument Conditions for Cs in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	NT	NT	NT	NT	NT	NT
2						
3	ICP-MS	Tb	ORS	He	1000	133
4	ICP-MS	Rh	UC	He	98.04	133
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Tm	NA	NA	2000	133
8	ICP-MS/MS	None			1200	133
9						
10	ICP-MS	Rh	CRI		80	
11	NA	NA	NA	NA	30	NA
12	ICP-MS	RH 103	ORS		50	133 m/z
13						
14	ICP-MS	Rh 103	ORS	He	50	133
15	NA	NA	NA	NA	NA	NA

Table 79 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						
3	ICP-MS	Rh	ORS	He	1000	63
4	ICP-MS	Ga	UC	He	98.04	63
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	CRI	He	2000	63
8	ICP-OES-AV	None			2400	324.754
9						
10	ICP-MS	Rh	CRI	He	80	
11	ICP-OES-AV	NA	NA	NA	30	324.754
12	ICP-MS	Ge 72	ORS		1000	63 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	327.395
14	ICP-MS	Sc 45	ORS	He	50	63
15	NA	NA	NA	NA	NA	NA

Table 80 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						
3	ICP-MS	Rh	ORS	H2	1000	56
4	ICP-OES-RV	N/A	NA	NA	98.04	240
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	CRI	He	2000	56
8	ICP-OES-AV	None			2400	258.588
9						
10	ICP-MS	Rh	CRI	HEHe	80	
11	ICP-OES-AV	NA	NA	NA	30	261.187
12	ICP-MS	Ge 72	ORS		1000	56 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	259.94
14	ICP-MS	Sc 45	ORS	He	50	56
15	NA	NA	NA	NA	NA	NA

Table 81 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						
3	ICP-MS	Ir	NA	NA	1000	201
4	ICP-MS	Tb	UC	He	98.04	201
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Ir	NA	NA	2000	102
8	ICP-MS/MS	None				
9						
10	ICP-MS	Te	CRI		80	
11	VGA-AAS	NA	NA	NA	30	253.7
12	ICP-MS	Ir 193	ORS		50	202 m/z
13	CVAFS	NA	NA	NA	500	254
14	AAS	N\A			50	253
15	NA	NA	NA	NA	NA	NA

Table 82 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						
3	ICP-MS	Sc	ORS	He	1000	39
4	ICP-OES-RV	N/A	NA	NA	98.04	766
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	CRI	He	2000	39
8	ICP-OES-RV	None			2400	766.491
9						
10	ICP-MS	Rh	CRI	He	800	
11	ICP-OES-AV	NA	NA	NA	30	766.491
12	ICP-OES-RV	Y377			100	766.491 nm
13						
14	ICP-OES-AV-equation	Eu 397.2			50	
15	NA	NA	NA	NA	NA	NA

Table 83 Instrument Conditions for La in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	NT	NT	NT	NT	NT	NT
2						
3	ICP-MS	Tb	ORS	He	1000	139
4	ICP-MS	Rh	UC	He	98.04	139
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Tm	NA	NA	2000	139
8	ICP-MS/MS	None			1200	139
9						
10						
11	NA	NA	NA	NA	30	NA
12	ICP-MS	RH 103	ORS		50	139 m/z
13						
14	ICP-MS	Rh 103	ORS	He	50	139
15	NA	NA	NA	NA	NA	NA

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						
3	ICP-MS	Sc	ORS	H2	1000	7
4	ICP-MS	Sc	UC	He	98.04	7
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	NA	NA	2000	7
8	ICP-MS/MS	None			1200	7
9						
10	ICP-MS	Rh	CRI		80	
11	NA	NA	NA	NA	30	NA
12	ICP-MS	Ge 72	ORS		50	7 m/z
13						
14	ICP-MS	Sc 45			50	7
15	NA	NA	NA	NA	NA	NA

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						
3	ICP-MS	Y	ORS	H2	1000	24
4	ICP-OES-RV	N/A	NA	NA	98.04	285
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	CRI	He	2000	54
8	ICP-OES-RV	None			2400	279.078
9						
10	ICP-MS	Rh	CRI	He	800	
11	ICP-OES-AV	NA	NA	NA	30	280.27
12	ICP-OES-RV	Y377			100	383.829 nm
13						
14	ICP-OES-AV-equation	Eu 397.2			50	
15	NA	NA	NA	NA	NA	NA

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						
3	ICP-MS	Ge	NA	NA	1000	55
4	ICP-MS	Sc	UC	He	98.04	55
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	CRI	He	2000	55
8	ICP-OES-AV	None			2400	257
9						
10	ICP-MS	Rh	CRI	He	80	
11	ICP-OES-AV	NA	NA	NA	30	293.931
12	ICP-MS	Ge 72	ORS		50	55 m/z
13	ICP-OES-AV	Y 371.069	NA	Na	50	293.931
14	ICP-MS	Sc 45	ORS	He	50	55
15	NA	NA	NA	NA	NA	NA

Table 87 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						
3	ICP-MS	Sc	ORS	H2	1000	23
4	ICP-OES-RV	N/A	NA	NA	98.04	590
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	CRI	He	2000	23
8	ICP-OES-RV	None			2400	589.582
9						
10	ICP-MS	Rh	CRI	He	800	
11	ICP-OES-AV	NA	NA	NA	30	589.592
12	ICP-OES-RV	Y377			100	589.592 nm
13	50	293.931				
14	ICP-OES-AV-equation	Eu 397.2			50	
15	NA	NA	NA	NA	NA	NA

Table 88 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						
3	ICP-MS	Rh	ORS	He	1000	60
4	ICP-MS	Ga	UC	He	98.04	60
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	CRI	He	2000	60
8	ICP-MS/MS	None	CRI	He	1200	60
9						
10	ICP-MS	Rh	CRI	He	80	
11	ICP-OES-AV	NA	NA	NA	30	231.604
12	ICP-MS	Ge 72	ORS		50	60 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	231.604
14	ICP-MS	Sc 45	ORS	He	50	60
15	NA	NA	NA	NA	NA	NA

Table 89 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						
3	ICP-MS	Ge	ORS	He	1000	31
4	ICP-OES-RV	N/A	NA	NA	98.04	186
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	CRI	He	2000	31
8	ICP-OES-AV	None			2400	
9						
10	ICP-OES-AV				80	
11	ICP-OES-AV	NA	NA	NA	30	213.618
12	ICP-OES-AV	Te214			100	185.878 nm
13	ICP-OES-AV	Y 371.029	NA	NA	50	213.618
14	ICP-OES-AV-equation	Eu 397.2			50	
15	NA	NA	NA	NA	NA	NA

Table 90 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						
3	ICP-MS	Tb	NA	NA	1000	208
4	ICP-MS	Tb	UC	He	98.04	206+207+208
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Ir	NA	NA	2000	206+207+208
8	ICP-MS/MS	None			1200	206+206+208
9						
10	ICP-MS	Tb	CRI		80	
11	ICP-OES-AV	NA	NA	NA	30	220.353
12	ICP-MS	Ir 193	ORS		50	208 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	220.353
14	ICP-MS	Ir 193	ORS	He	50	208
15	NA	NA	NA	NA	NA	NA

Table 91 Instrument Conditions for Rb in S1

Lab. Code	Instrument	Internal standard	Reaction/ Collision Cell	Cell Gas	S1 Final Dilution factor	Wavelength (nm)/ Ion(m/z)
1	NT	NT	NT	NT	NT	NT
2						
3	ICP-MS	Y	ORS	He	1000	85
4	ICP-MS	Rh	UC	He	98.04	85
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Rh	NA	NA	2000	85
8	ICP-MS/MS	None			1200	85
9						
10						
11	NA	NA	NA	NA	30	NA
12	ICP-MS	RH 103	ORS		50	85 m/z
13						
14	ICP-MS	Rh 103	ORS	He	50	85
15	NA	NA	NA	NA	NA	NA

Table 92 Instrument Conditions for S 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	181.972 nm
2						
3	ICP-MS				1000	
4	ICP-OES-RV	N/A	NA	NA	98.04	182
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	CRI	He	2000	34
8	ICP-OES-AV	None			2400	180.669
9						
10	ICP-OES-AV				80	
11	ICP-OES-AV	NA	NA	NA	30	182.562
12	ICP-OES-AV	Te214			100	180.669 nm
13	ICP-OES-AV	Y 371.029	NA	NA	50	181.972
14	ICP-OES-AV-equation	Eu 397.2			50	
15	NA	NA	NA	NA	NA	NA

Table 93 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						
3	ICP-MS	Ge	ORS	H2	1000	78
4	ICP-MS	Te	UC	He	98.04	82
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Rh	DRC	He	2000	78
8						
9						
10	ICP-MS	Rh	CRI	HEHe	80	
11	ICP-OES-AV	NA	NA	NA	30	196.026
12	ICP-MS	RH 103	ORS	HEHe	50	78 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	196.026
14	ICP-MS	Sc 45	ORS	He	50	78
15	NA	NA	NA	NA	NA	NA

Table 94 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						
3	ICP-MS	Ir	NA	NA	1000	121
4	ICP-MS	Rh	UC	He	98.04	121
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	In	NA	NA	2000	121
8						
9						
10	ICP-MS	Rh	CRI		80	
11	ICP-OES-AV	NA	NA	NA	30	217.582
12	ICP-MS	RH 103	ORS		50	121 m/z
13						
14	ICP-MS	Rh 103	ORS	He	50	121
15	NA	NA	NA	NA	NA	NA

Table 95 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						
3	ICP-MS	Rh	ORS	He	1000	118
4	ICP-MS	Rh	UC	He	98.04	120
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	In	NA	NA	2000	120
8						
9						
10	ICP-MS	Rh	CRI		80	
11	ICP-OES-AV	NA	NA	NA	30	283.998
12	ICP-MS	RH 103	ORS		50	118 m/z
13						
14	ICP-MS	Rh 103	ORS	He	50	118
15	NA	NA	NA	NA	NA	NA

Table 96 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						
3	ICP-MS	Y	ORS	He	1000	88
4	ICP-MS	Rh	UC	He	98.04	88
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Rh	NA	NA	2000	88
8	ICP-OES-AV	None			2400	
9						
10	ICP-MS	Rh	CRI		800	
11	ICP-OES-AV	NA	NA	NA	30	421.552
12	ICP-MS	Ge 72	ORS		1000	88 m/z
13						
14	ICP-MS	Rh 103	ORS	He	50	88
15	NA	NA	NA	NA	NA	NA

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						
3	ICP-MS	Tb	NA	NA	1000	238
4	ICP-MS	Tb	UC	He	98.04	238
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Ir	NA	NA	2000	238
8	ICP-MS/MS	None			1200	238
9						
10	ICP-MS	Rh	CRI		80	
11	NA	NA	NA	NA	30	NA
12	ICP-MS	Ir 193	ORS		50	238 m/z
13						
14	ICP-MS	Ir 193	ORS	He	50	238
15	NA	NA	NA	NA	NA	NA

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						
3	ICP-MS	Ge	ORS	He	1000	51
4	ICP-MS	Sc	UC	He	98.04	51
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	CRI	He	2000	51
8	ICP-MS/MS	None	ORS	He	1200	51
9						
10	ICP-MS	Rh	CRI	He	80	
11	ICP-OES-AV	NA	NA	NA	30	311.837
12	ICP-MS	Ge 72	ORS		50	51 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	311.837
14	ICP-MS	Sc 45	ORS	He	50	51
15	NA	NA	NA	NA	NA	NA

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						
3	ICP-MS	Ge	ORS	He	1000	66
4	ICP-MS	Te	UC	He	98.04	66
5	NA	NA	NA	NA	NA	NA
6	NA	NA	NA	NA	NA	NA
7	ICP-MS	Sc	CRI	He	2000	66
8	ICP-OES-AV	None			2400	213
9						
10	ICP-MS	Rh	CRI	He	800	
11	ICP-OES-AV	NA	NA	NA	30	213.857
12	ICP-MS	Ge 72	ORS		1000	66 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	213.857
14	ICP-MS	Sc 45	ORS	He	50	66
15	NA	NA	NA	NA	NA	NA

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA
6	ICP-MS	Sc Rh Ir	NA	NA	x2	Al27
7	ICP-MS	Sc	NA	NA	2000	27
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI		40	
11	ICP-OES-AV	NA	NA	NA	30	396.152
12	ICP-MS	Ge 72	ORS		50	27 m/z
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rhodium			70	27

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA
6	ICP-MS	Sc Rh Ir	ORS	He	x2	as75
7	ICP-MS	Rh	CRI	He	2000	75
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI	HEHe	40	
11	ICP-OES-AV	NA	NA	NA	30	193.696
12	ICP-MS	Ge 72	ORS		50	75 m/z
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Germanium	ORS	He	700	75

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA
6	ICP-MS	Sc Rh Ir	NA	NA	x2	B11
7	ICP-MS	Sc	NA	NA	2000	10
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI		40	
11	ICP-OES-AV	NA	NA	NA	30	249.678
12	ICP-MS	Ge 72	ORS		50	11 m/z
13						
14	NA	NA	NA	NA	NA	NA
15	NT					

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA
6	ICP-MS	Sc Rh Ir	ORS	He	x2	Ba137
7	ICP-MS	Tm	NA	NA	2000	137
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI		40	
11	ICP-OES-AV	NA	NA	NA	30	233.527
12	ICP-MS	Rh 103	ORS		50	137 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	233.527
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rhodium			70	136

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Scandium	ORS	H2	100.1602564	40
6	ICP-OES-AV	Y	NA	NA	x10	Ca315.887
7	ICP-MS	Sc	NA	NA	2000	43
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI		4000	
11	ICP-OES-AV	NA	NA	NA	30	318.127
12	ICP-OES-RV	Y377			100	317.933 nm
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-OES-AV	Lutetium			700	315317

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA
6	ICP-MS	Sc Rh Ir	ORS	He	x2	Cd111
7	ICP-MS	Rh	NA	NA	2000	111
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI		40	
11	ICP-OES-AV	NA	NA	NA	30	214.439
12	ICP-MS	Rh 103	ORS		50	111 m/z
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rhodium			70	111114

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA
6	ICP-MS	Sc Rh Ir	ORS	He	x2	Co59
7	ICP-MS	Sc	CRI	He	2000	59
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI	He	40	
11	ICP-OES-AV	NA	NA	NA	30	230.786
12	ICP-MS	Rh 103	ORS		50	59 m/z
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Germanium	ORS	He	70	59

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA
6	ICP-MS	Sc Rh Ir	ORS	He	x2	Cr52
7	ICP-MS	Sc	CRI	He	2000	52
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI	He	40	
11	ICP-OES-AV	NA	NA	NA	30	205.56
12	ICP-MS	Ge 72	ORS		50	52 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	267.716
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Germanium	ORS	He	70	52,53

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Germanium	ORS	He	100.1602564	63
6	ICP-MS	Sc Rh Ir	ORS	He	x2x10	Cu62
7	ICP-MS	Sc	CRI	He	2000	63
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI	He	40	
11	ICP-OES-AV	NA	NA	NA	30	324.754
12	ICP-MS	Ge 72	ORS		50	63 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	327.395
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Germanium	ORS	He	700	63,65

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Yttrium	ORS	He	100.1602564	56
6	ICP-MS	Sc Rh Ir	ORS	He	x10x2	Fe238.204
7	ICP-MS	Sc	CRI	He	2000	56
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI	HEHe	40	
11	ICP-OES-AV	NA	NA	NA	30	261.187
12	ICP-MS	Ge 72	ORS		1000	56 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	259.94
14	NA	NA	NA	NA	NA	NA
15	ICP-OES-AV	Lutetium			70	259

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Scandium	ORS	He	100.1602564	39
6	ICP-OES-AV	Y	NA	NA	x10	K766.491
7	ICP-MS	Sc	CRI	He	2000	39
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI	He	400	
11	ICP-OES-AV	NA	NA	NA	30	766.491
12	ICP-OES-RV	Y377			100	766.491 nm
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-OES-AV	Lutetium			700	766

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Scandium	ORS	He	100.1602564	24
6	ICP-OES-AV	Y	NA	NA	x10	Mg279.800
7	ICP-MS	Sc	CRI	He	2000	24
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI	He	40	
11	ICP-OES-AV	NA	NA	NA	30	280.27
12	ICP-OES-RV	Y377			100	383.829 nm
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-OES-AV	Lutetium			70	285

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Scandium	ORS	He	100.1602564	55
6	ICP-MS	Sc Rh Ir	ORS	He	x2	Mn55
7	ICP-MS	Sc	CRI	He	2000	55
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI	He	40	
11	ICP-OES-AV	NA	NA	NA	30	293.931
12	ICP-MS	Ge 72	ORS		50	55 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	293.931
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rhodium			70	55

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA
6	ICP-MS	Sc Rh Ir	ORS	He	x2	Mo95
7	ICP-MS	Rh	NA	NA	2000	98
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI		40	
11	ICP-OES-AV	NA	NA	NA	30	204.598
12	ICP-MS	Ge 72	ORS		50	95 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	202.032
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rhodium			70	98

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Scandium	ORS	He	100.1602564	23
6	ICP-OES-AV	Y	NA	NA	X10	Na588.995
7	ICP-MS	Sc	CRI	He	2000	23
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI	He	400	
11	ICP-OES-AV	NA	NA	NA	30	589.592
12	ICP-OES-RV	Y377			100	589.592 nm
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-OES-AV	Lutetium			70	589

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA
6	ICP-OES-AV	Y	NA	NA	x10	P213.618
7	ICP-MS	Sc	CRI	He	2000	31
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-OES-AV				40	
11	ICP-OES-AV	NA	NA	NA	30	213.618
12	ICP-OES-AV	Te214			100	185.878 nm
13	ICP-OES-AV	Y 371.029	NA	NA	50	213.618
14	NA	NA	NA	NA	NA	NA
15	ICP-OES-AV	Lutetium			70	178

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Lutetium	ORS	He	100.1602564	208
6	ICP-MS	Sc Rh Ir	ORS	He	x2	Pb207
7	ICP-MS	Ir	NA	NA	2000	206+207+208
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Tb	CRI		40	
11	ICP-OES-AV	NA	NA	NA	30	220.353
12	ICP-MS	Ir 193	ORS		50	208 m/z
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rhodium			70	206207208

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA
6					x10	S181.972
7	ICP-MS	Sc	CRI	He	2000	34
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-OES-AV				40	
11	ICP-OES-AV	NA	NA	NA	30	182.562
12	ICP-OES-AV	Te214			100	180.669 nm
13	ICP-OES-AV	Y 371.029	NA	NA	50	181.972
14	NA	NA	NA	NA	NA	NA
15	NT					

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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Yttrium	ORS	H2	100.1602564	78
6	ICP-MS/MS	Y	ORS	HEHe	x10	Se78
7	ICP-MS	Rh	DRC	He	2000	78
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI	HEHe	80	
11	ICP-OES-AV	NA	NA	NA	30	196.026
12	ICP-MS	Rh 103	ORS	HEHe	50	78 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	196.026
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Germanium	ORS	HeHe	700	78

Table 119 Instrument Conditions for Sr 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	88 m/z
2						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA
6	ICP-MS	Sc Rh Ir	ORS	He	x10	Sr88
7	ICP-MS	Rh	NA	NA	2000	88
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI		40	
11	ICP-OES-AV	NA	NA	NA	30	421.552
12	ICP-MS	Ge 72	ORS		50	88 m/z
13						
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Rhodium			700	88

Table 120 Instrument Conditions for V 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	67 m/z
2						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	NA	NA	NA	NA	NA	NA
6	ICP-MS	Sc Rh Ir	ORS	He	x10	V51
7	ICP-MS	Sc	CRI	He	2000	51
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI	He	40	
11	ICP-OES-AV	NA	NA	NA	30	311.837
12	ICP-MS	Ge 72	ORS		50	51 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	311.837
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Germanium	ORS	He	70	51

Table 121   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						
3	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA
5	ICP-MS	Germanium	ORS	He	100.1602564	66
6	ICP-MS	Sc Rh Ir	ORS	He	x10	Zn66
7	ICP-MS	Sc	CRI	He	2000	66
8	NA	NA	NA	NA	NA	NA
9						
10	ICP-MS	Rh	CRI	He	400	
11	ICP-OES-AV	NA	NA	NA	30	213.857
12	ICP-MS	Ge 72	ORS		1000	66 m/z
13	ICP-OES-AV	Y 371.029	NA	NA	50	213.857
14	NA	NA	NA	NA	NA	NA
15	ICP-MS	Germanium	ORS	He	700	66

END OF REPORT