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Proficiency Test Final Report AQA 21-14 Metals, Nutrients and Anions in Soil

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Luminita Antin

Andrew Evans

Hamish Lenton

Elizabeth Tully

Raluca Iavetz

Manager, Chemical Proficiency Testing



Phone: 61-2-9449 0111
proficiency@measurement.gov.au



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1 SUMMARY

This report presents the results of the proficiency test AQA 21-14 Metals, Nutrients and Anions in Soil. The study covers the measurement of acid extractable elements: Ag, Al, As, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cs, Cu, Fe, Ga, Hg, K, La, Li, Mg, Mn, Mo, Na, Ni, P, Pb, Rb, S, Sb, Se, Sn, Sr, Th, Tl, U, V and Zn. Measurement of pH, electrical conductivity (EC), water soluble bromide (Br^-), chloride (Cl^-), fluoride (F^-), iodide (I^-), orthophosphate-P (PO_4^{3-} -P), and sulphate (SO_4^{2-}), total Kjeldahl nitrogen (TKN), 2M KCl extractable ammonium nitrogen (NH_4^+ -N) and 2M KCl extractable nitrate nitrogen (NO_3^- -N) were also included in the program.

The sample set consisted of one soil sample, one sediment sample and one agricultural soil sample.

Thirty laboratories registered to participate and all submitted results.

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

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

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

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

Of 785 z-scores, 711 (91%) were satisfactory with $|z| \leq 2.0$.

Of 785 E_n -scores, 623 (79%) were satisfactory with $|E_n| \leq 1.0$.

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

Boron, antimony and low level selenium were the tests which presented the most analytical difficulty to participating laboratories.

- iii. evaluate within laboratory reproducibility;*

Sample S1 of the present study was distributed as Sample S2 of AQA 19-12. Although the assigned values set for the two samples were not significantly different, in some cases, the reported results varied greatly.

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

On average participants' performance in the measurement of inorganic analytes in soil over has remained fairly consistent over time.

- v. develop the practical application of traceability and measurement uncertainty and provide participants with information that will be useful in assessing their uncertainty estimates;*

Of 871 numerical results, 825 (95%) were reported with an expanded measurement uncertainty. An example of estimating measurement uncertainty using the proficiency testing data only is given in Appendix 3.

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

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

2 INTRODUCTION

2.1 NMI Proficiency Testing Program

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

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

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

AQA 21-14 is the 29th NMI proficiency study of inorganic analytes in soil.

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

2.3 Study Conduct

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

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

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

3 STUDY INFORMATION

3.1 Selection of Matrices and Inorganic Analytes

The 54 tests were selected from those for which an investigation level is published in the Guideline on the Investigation Levels for Soil and Groundwater, promulgated by the National Environmental Protection Council (NEPC)⁵ and from analytes commonly measured in soil.

3.2 Participation

Thirty laboratories participated and all thirty submitted results.

The timetable of the study was:

Invitation issued: 22 September 2021
Samples dispatched: 12 October 2021
Results due: 17 November 2021
Interim report issued: 18 November 2021

3.3 Test Material Specification

Three samples were provided for analysis:

Sample S1 was 25 g of dried sediment previously distributed as sample S2 of AQA 19-12;

Sample S2 was 25 g of dried soil; and

Sample S3 was 75 g of dried agricultural soil.

3.4 Laboratory Code

All participant laboratories were assigned a confidential code number.

3.5 Sample Preparation, Analysis and Homogeneity Testing

Test samples from previous studies have been demonstrated to be sufficiently homogeneous for the evaluation of participants' performance. Therefore, only a partial homogeneity test was conducted for all analytes with the exception of Sb and Rb in S2 and EC, pH, iodide, 2M KCl extractable nitrate-N and S in S3. The results from the partial homogeneity test for these samples are reported in the present study as the homogeneity value.

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

3.6 Stability of Analytes

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

3.7 Sample Storage, Dispatch and Receipt

The samples were dispatched by courier on 12 October 2021.

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

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

3.8 Instructions to Participants

Participants were instructed as follows:

- Quantitatively analyse the samples using your normal test method.
- For *Sample S3* for 2M KCl Extractable (NO_3^- -N) and (NH_4^+ -N), participants are asked to follow the extraction procedure described below:

"Prepare a 1:10 w/v soil/2M KCl extracting solution. For example, weigh 5 g of soil into a suitable bottle or jar and add 50 mL of 2M KCl extracting solution.

Mechanically shake (end-over-end preferred), at room temperature for 1 h. Allow around 20-30 min for soil to settle and clarify and then take a known aliquot for the measurement technique employed. Further dilution of the aliquot may be required."

Measure the analytes using a colorimetric method; and report results of 1:10 soil/2M KCl extracting solution on as received basis in units of mg/kg for: 2M KCl extractable ammonium-N (NH_4^+ -N) and 2M KCl extractable nitrate-N (NO_3^- -N).

- Report on as received basis in units of mg/kg except for EC and pH. EC results are to be reported in units of $\mu\text{S}/\text{cm}$.

| SAMPLE S1 (sediment) | | SAMPLE S2 (soil) | | SAMPLE S3 (agricultural soil) | |
|-----------------------|-------------------------------|-----------------------|-------------------------------|---|-------------------------------|
| Test Acid Extractable | Approximate Conc. Range mg/kg | Test Acid Extractable | Approximate Conc. Range mg/kg | Test | Approximate Conc. Range*mg/kg |
| Al | 250-10000 | Ag | 0.5-20 | Ca (acid extractable) | 100-4000 |
| As | 5-200 | B | 20-800 | Fe (acid extractable) | 1000-40000 |
| Cd | 5-200 | Ba | 20-800 | K (acid extractable) | 100-4000 |
| Co | 5-200 | Be | 0.5-20 | Mg (acid extractable) | 100-4000 |
| Cr | 5-200 | Bi | 0.5-20 | Na (acid extractable) | 100-4000 |
| Cu | 5-200 | Cd | 0.5-20 | P (acid extractable) | 100-4000 |
| Hg | 5-200 | Cs | 0.5-20 | S (acid extractable) | NA |
| Mn | 50-2000 | Ga | 0.5-20 | Sr (acid extractable) | 10-400 |
| Mo | 0.5-20 | Hg | 0.2-8 | Water soluble Bromide (Br^-) – 1:5 soil/water extract | 0.5-20 |
| Ni | 0.5-20 | La | 5-200 | Water soluble Chloride (Cl^-) – 1:5 soil/water extract | 5-200 |
| Pb | 10-400 | Li | 0.5-20 | Water soluble Fluoride (F^-) – 1:5 soil/water extract | 0.5-20 |
| Sb | 0.5-20 | Ni | 5-200 | Water soluble Iodide (I^-) – 1:5 soil/water extract | 5-200 |
| Se | 5-200 | Rb | 0.5-20 | Water soluble Orthophosphate-P (PO_4^{3-} -P) – 1:5 soil/water extract | 0.5-20 |
| Th | 0.5-20 | Sb | 5-200 | Water soluble Sulphate (SO_4^{2-}) – 1:5 soil/water extract | 50-2000 |
| U | 0.5-20 | Se | 0.5-20 | pH of 1:5 soil/water suspension | <10 |
| V | 5-200 | Sn | 0.5-20 | EC of 1:5 soil/water extract | >500 $\mu\text{S}/\text{cm}$ |
| Zn | 5-200 | Tl | 0.5-20 | Kjeldahl Nitrogen, Total (TKN) | 250-10000 |
| | | Zn | 5-200 | 2M KCl Extractable (Ammonium-N) | NA |
| | | | | 2M KCl Extractable (Nitrate-N) | NA |

NA = Not Available

- Report results using the electronic results sheet emailed to you:
- Report results as you would report to a client. For each analyte, report the expanded measurement uncertainty.
- Please send the requested details regarding the test method and the basis of your uncertainty estimate.

3.9 Interim Report

An interim report was emailed to participants on 18 November 2021.

4 PARTICIPANT LABORATORY INFORMATION

4.1 Test Method Summaries

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

Table 1 Methodology for Acid Extractable Elements

| Lab. Code | Method Reference | Sample Mass (g) | Temp. (°C) | Time (min) | Vol. HNO ₃ (mL) | Vol. HCl (mL) | Vol. HNO ₃ (1:1) (mL) | Vol. HCl (1:1) (mL) | Vol. H ₂ O ₂ (mL) | Other (mL) |
|-----------|--|-------------------|------------|------------|----------------------------|---------------|----------------------------------|---------------------|---|----------------------|
| 1 | AS4479.2-1997 | 2 | 90 | 90 | 2 | 6 | | | | |
| 2 | USEPA Method 200.2 | 1 | 95 | 60 | | | 2 | 10 | | |
| 4 | USEPA method 200.2 Revision 2.8 | 1 | 95 | 60 | | | 2 | 10 | 2 | |
| 5 | In House, US EPA 6020B | 2 | 90-95 | 60 | 4 | 12 | | | | 4 (H ₂ O) |
| 6 | In House S6 - Referencing APHA 3125 | 0.4 | 120 | 60 | 2.5 | 7.5 | | | | |
| 7 | USEPA 200.2 | 1 | 95 | 60 | | | 2 | | 2 | 10 (HCl 1:4) |
| 8 | 200.2 Revision 2.8 | 1 | 95 ± 5 | 60 | 2 | 10 | | | 2 | |
| 10* | EPA 200.2, (1:1 Nitric:Hydrochloric Acid) | 0.5 | 96 | 30 | 1 | 1 | | | | |
| 11 | USEPA Method 3050B and APHA Method 3125 | 0.5 | 95 | 120 | 3 | 3 | | | | |
| 12 | US EPA 3050 B | 1.0006 | 98 | 100 | | | 5.0 mL | 10.0 mL | | |
| 13 | US EPA 3050B | 0.5 | 95 | 120 | 7.5 | 5 | | | | 1.5 |
| 14 | USEPA 3051A (Modification) | 1 | 170 | 15 | | | 8 | 2 | | |
| 15 | ICPOES/ICPMS/AFS In-house methods referenced to USEPA 6010,6020 and 7474 | 1 | 95 | 90 | 2 | 3 | | | | 2 |
| 16* | Soil Chemical Methods - Australasia (Raymod and Lyons) - Method 17B1 | 3 | 95 | 120 | 22.5 | 7.5 | | | | |
| 17 | USEPA 3050B | 1.0000 | 98 | 150 | 5 | 5 | | | | |
| 18 | US EPA Methods 3050B, 3051A and 6020B | 2 | 100 | 60 | 4 | 12 | | | | |
| 19 | | 1 | 90 | 240 | 4 | 1 | | | | |
| 20 | USEPA-6010C (Except Mercury by USEPA-7471B) | ICP=1g, FIMS=0.5g | 95 | | 5 | 5 | | 3 | | |
| 21* | 200.2 | 1 | 98 | 90 | 3 | 3 | | | | |
| 22 | | 1 | 95-100 | 120 | 3 | 3 | | | | |
| 23* | EPA 200.2 | 0.5 | 95 | 45 | 1 | 1 | | | | 5 (H ₂ O) |

Table 1 Methodology for Acid Extractable Elements (continued)

| Lab. Code | Method Reference | Sample Mass (g) | Temp. (°C) | Time (min) | Vol. HNO ₃ (mL) | Vol. HCl (mL) | Vol. HNO ₃ (1:1) (mL) | Vol. HCl (1:1) (mL) | Vol. H ₂ O ₂ (mL) | Other (mL) |
|-----------|---|-----------------|------------|------------|----------------------------|---------------|----------------------------------|---------------------|---|-----------------------|
| 24 | EPA 3052 | 0.50xx | 210 | 30 | 5 | 3 | | | | 0.75 (HF) |
| 25 | APHA 3030E | 1 | 40 | 240 | 10 | 1 | | | | |
| 26 | US-EPA Method 200.2 | 1 | 95 | 50 | 2 | 2 | | | | 10 (H ₂ O) |
| 27 | EPA (Environmental Protection Agency) 1994 Method 200.8 | | 109 | 60 | 800 | 400 | | | | 1200 (Type 1 Water) |
| 28 | USEPA Method 3010 | 5 | 95 | 60 | 10 | 10 | | | | |
| 29 | EPA3050B, 6020B | 2 | 95-105 | 60 | 4 | 12 | | | | |
| 30 | AS 4479.2-1997, AS4479.4-1999 | 0.5 | 95 | 120 | 1 | 3 | | | | |

*Additional information in Table 10

Table 2 Methodology for Total Kjeldahl Nitrogen

| Lab. Code | Method Reference | Digestion | Distillation | Measurement Method | Instrument |
|-----------|--|-----------|--------------|----------------------------------|-----------------|
| 2 | APHA 4500 Norg, A & D | Yes | No | Colorimetric - salicylate method | DA |
| 4 | APHA 4500 - Norg. A & D. | Yes | No | Colorimetric - salicylate method | DA |
| 6* | | | | TKN = TN-NOx (Dumas) | |
| 7 | APHA 4500 | Yes | | Colorimetric - phenate method | DA |
| 8 | APHA 22nd edition 4500 Norg A & D with Jirka Modification- Jirka et al. (1976) and the appropriate Discrete Analyser method. | Yes | No | Colorimetric - phenate method | DA |
| 10 | APHA (online edition) 4500-N org | Yes | | Colorimetric - salicylate method | DA |
| 12 | NSW.AES.072: Based on 7A1 Australian Laboratory Handbook of Soil and Water Chemical Methods (By Rayment and Higginson) | Yes | Yes | | |
| 14 | APHA Method 4500-N Org B and Method 4500-NH ₃ F | Yes | Yes | Colorimetric - phenate method | DA |
| 15 | APHA 4500-Norg D In-house method | Yes | | Colorimetric - salicylate method | FIA |
| 16 | In-house method based on APHA 23rd edition 4500-Norg B | Yes | Yes | Colorimetric - salicylate method | DA |
| 22 | | Yes | Yes | Titrimetric method | Manual Analysis |
| 25 | APHA 4500-Norg C | Yes | Yes | Titrimetric method | Manual Analysis |
| 28 | | Yes | No | Colorimetric - salicylate method | FIA |

*Additional information in Table 10

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

| Lab. Code | Method Reference | | Sample Mass (g) | Extraction Solution 2M KCl Volume (mL) | Shake time (hours) | Measurement Method | | Measurement Instrument | |
|-----------|---|---|-----------------|--|--------------------|----------------------------------|---|---------------------------------|---------------------------------|
| | NH ₄ ⁺ -N | NO ₃ ⁻ -N | | | | NH ₄ ⁺ -N | NO ₃ ⁻ -N | NH ₄ ⁺ -N | NO ₃ ⁻ -N |
| 6 | In House S37 | In House S37 | 2 | 20 | 1 | Colorimetric - Salicylate method | Colorimetric - vanadium III method | FIA | FIA |
| 9 | 7C2b | 7C2b | 10 | 100 | 1 | Colorimetric Salicylate Method | Colorimetric-Sulfanilamide-NEDD hydrazine reduction | FIA | FIA |
| 10 | APHA (online edition) 4500-NH ₃ H | NT | 5 | 100 | 1 | Colorimetric - Phenate method | NT | FIA | NT |
| 12 | APHA 4500 NH ₃ B/C | | 10.0003 | 100 | 1 | | | | |
| 14* | 7C2b | 7C2b | 3 | 30 | 1 | Colorimetric - Phenate method | Colorimetric-Sulfanilamide-NEDD Cd reduction | FIA | FIA |
| 15* | APHA 4500-NH ₃ H | 4500-NO ₃ I | 3 | 30 | 1 | Colorimetric - Phenate method | Colorimetric-Sulfanilamide-NEDD Cd reduction | FIA | FIA |
| 16 | Soil Chemical Methods, Rayment & Lyons method 7C2 | Soil Chemical Methods, Rayment & Lyons method 7B1 | 5 | 50 | 1 | Colorimetric - Salicylate method | Colorimetric - vanadium III method | DA | DA |
| 17 | R&L 7C2b | R&L 7C2b | 2 | 20 | 1 | | | DA | DA |
| 22 | | apha | 10 | 100 | 1 | Fluorescence is measure at 460 | Colorimetric-Sulfanilamide-NEDD Cd reduction | SFA | |
| 28 | | | 10 | 50 | 16 | Colorimetric - Phenate method | Colorimetric-Sulfanilamide-NEDD Cd reduction | FIA | FIA |
| 29 | APHA 4500-NH ₃ | | 5 | 50 | 1 | Colorimetric - Phenate method | | DA | |

Additional information in Table 10. NT = Not Tested

Table 4 Methodology for Water Soluble Br⁻

| Lab. Code | Method Reference | Sample Mass (g) | Water Volume (mL) | Shake Time (hours) | Measurement Method | Measurement Instrument |
|-----------|------------------|-----------------|-------------------|--------------------|----------------------------|------------------------|
| 7 | APHA 4110 | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 13 | | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 14 | APHA 4110B | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 21 | APHA 4110B and C | 5 | 25 | | Ion Chromatographic Method | IC |
| 22 | apha | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 29 | APHA 4110B | 10 | 50 | 1 | Ion Chromatographic Method | IC |

Table 5 Methodology for Water Soluble Cl⁻

| Lab. Code | Method Reference | Sample Mass (g) | Water Volume (mL) | Shake Time (hours) | Measurement Method | Measurement Instrument |
|-----------|--|-----------------|-------------------|--------------------|----------------------------------|------------------------|
| 2 | | 10 | 50 | 1 | Mercuric Nitrate Titration | DA |
| 4 | APAH 4500-Cl | 10 | 50 | 1 | Ferricyanide Colorimetric Method | DA |
| 7 | APHA 4110 | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 9 | 5 | 5 | 40 | 1 | Ferricyanide Colorimetric Method | FIA |
| 10 | | | | | NT | |
| 12 | NSW.AES.030 | 15.0006 | 75 | 1 | | |
| 13 | | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 14 | APHA 4110B | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 15 | Inhouse with reference to APHA4110B | 4 | 20 | 1 | Ion Chromatographic Method | IC |
| 16 | Soil Chemical Methods, Rayment & Lyons | 50 | 250 | 1 | Potentiometric Method | Titration |
| 17 | | 10 | 50 | 1 | | |
| 21 | APHA 4110B and C | 5 | 25 | | Ion Chromatographic Method | IC |
| 22 | apha | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 28 | | 5 | 25 | 16 | Mercuric Thiocyanate | DA |
| 29 | APHA 4110B | 10 | 50 | 1 | Ion Chromatographic Method | IC |

NT = Not Tested

Table 6 Methodology for Water Soluble F⁻

| Lab. Code | Method Reference | Sample Mass (g) | Water Volume (mL) | Shake Time (hours) | Measurement Method | Measurement Instrument |
|-----------|--|-----------------|-------------------|--------------------|--------------------------------|------------------------------|
| 2 | | 10 | 50 | 1 | Potenciometric | Fluoride Selective Electrode |
| 4 | In house | 10 | 50 | 1 | Ion Selective Electrode Method | Fluoride Selective Electrode |
| 7 | APHA 4110 | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 10 | | | | | NT | |
| 14 | APHA 4500-F C | 10 | 50 | 1 | Ion Selective Electrode Method | Fluoride Selective Electrode |
| 16 | Soil Chemical Methods, Rayment & Lyons | 50 | 250 | 1 | Ion Selective Electrode Method | Fluoride Selective Electrode |
| 21 | APHA 4110B and C | 5 | 25 | | Ion Chromatographic Method | IC |
| 22 | apha | 10 | 50 | 1 | Ion Selective Electrode Method | IC |
| 28 | | 5 | 25 | 16 | Ion Selective Electrode Method | Fluoride Selective Electrode |

NT = Not Tested

Table 7 Methodology for Water Soluble I⁻

| Lab. Code | Method Reference | Sample Mass (g) | Water Volume (mL) | Shake Time (hours) | Measurement Method | Measurement Instrument |
|-----------|------------------|-----------------|-------------------|--------------------|----------------------------|------------------------|
| 7 | APHA 4110 | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 14 | APHA 4110B | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 22 | apha | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 29 | APHA 4110B | 10 | 50 | 1 | Ion Chromatographic Method | IC |

NT = Not Tested

Table 8 Methodology for Water Soluble Orthophosphate-P

| Lab. Code | Method Reference | Sample Mass (g) | Water Volume (mL) | Shake Time (hours) | Measurement Method | Measurement Instrument |
|-----------|----------------------------|-----------------|-------------------|--------------------|---|------------------------|
| 2 | | 10 | 50 | 1 | Ascorbic Acid Colorimetric | DA |
| 4 | APHA 4500-P | 10 | 50 | 1 | Vanadomolybdophosphoric Colorimetric Method | DA |
| 6 | | 2 | 10 | 1 | Vanadomolybdophosphoric Colorimetric Method | FIA |
| 7 | APHA 4110 | 10 | 50 | 1 | Ion Chromatographic Method | FIA |
| 10 | | | | | NT | |
| 14 | APHA 4500-P G | 10 | 50 | 1 | Ascorbic Acid Colorimetric Method | FIA |
| 16 | Soil Chemical Methods, R&L | 50 | 250 | 1 | Ascorbic Acid Colorimetric Method | DA |
| 17 | | 10 | 50 | 1 | | DA |
| 21 | APHA 4110B and C | 5 | 25 | | Ion Chromatographic Method | IC |
| 22 | apha | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 28 | | 5 | 25 | 16 | Ascorbic Acid Colorimetric Method | DA |
| 29 | APHA 4500-PE, 4500-P B | 10 | 50 | 1 | Ascorbic Acid Colorimetric Method | DA |

NT = Not Tested

Table 9 Methodology for Water Soluble SO₄²⁻

| Lab. Code | Method Reference | Sample Mass (g) | Water Volume (mL) | Shake Time (hours) | Measurement Method | Measurement Instrument |
|-----------|---------------------------|-----------------|-------------------|--------------------|----------------------------|------------------------|
| 2 | | 10 | 50 | 1 | Turbidimetric Method | DA |
| 4 | In house | 10 | 50 | 1 | ICP-Method | ICP-OES |
| 6 | | 2 | 10 | 1 | ICP-Method | ICP-OES |
| 7 | APHA 4110 | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 12 | NSW.AES.032 | 15.0006 | 75 | 1 | | |
| 14 | APHA 4110B | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 16 | Soil Chemical Methods R&L | 50 | 250 | 1 | Turbidimetric Method | Turbidimeter |
| 17 | | 10 | 50 | 1 | | ICP-OES |
| 21 | APHA 4110B and C | 5 | 25 | | Ion Chromatographic Method | IC |
| 22 | apha | 10 | 50 | 1 | Ion Chromatographic Method | IC |
| 28 | | 5 | 25 | 16 | Turbidimetric Method | DA |
| 29 | APHA 4110B | 10 | 50 | 1 | Ion Chromatographic Method | IC |

4.2 Instruments Used for Measurements

The instruments and settings used by participants for acid extractable elements are presented in Appendix 4.

4.3 Additional Information

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

Table 10 Additional information

| Lab Code | Additional Information |
|----------|--|
| 6 | Methodology for Total Kjeldahl Nitrogen: TKN - TN done by LECO, NOx by FIA |
| 10 | Instrument Techniques for Acid Extractable Elements: APHA (online edition) 3125B by ICPMS |
| 14 | Methodology for 2M KCl Extractable Ammonium-N and Nitrate-N: Method reference from Rayment & Lyons, 2011. |
| 15 | Methodology for 2M KCl Extractable Ammonium-N and Nitrate-N: Rayment and Lyons(for 2M KCL Extraxtable Ammonium-N and Nitrate-N) 7C2b |
| 16 | Methodology for Acid Extractable Elements: Once digested the sample is made up to 100 mL with DI Water Instrument Techniques for Acid Extractable Elements: Mercury is analysed by in-house method based on USEPA method 245.7 using FIMS Total Phosphorus was analysed by Ascorbic Acid colorimetric using DA |
| 21 | Sample S2: Se not detected for this sample (<LOR) |
| 23 | Methodology for Acid Extractable Elements: After acid addition, samples are left for 5-10 minutes before water added & heated. |

4.4 Basis of Participants' Measurement Uncertainty Estimates

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

Table 11 Basis of Uncertainty Estimate

| Lab. Code | Approach to Estimating MU | Information Sources for MU Estimation ^a | | Guide Document for Estimating MU |
|-----------|---|---|--|---|
| | | Precision | Method Bias | |
| 1 | Bottom Up (ISO/GUM, fish bone/cause and effect diagram) | Control Samples - CRM Duplicate Analysis Instrument Calibration | CRM Instrument Calibration | |
| 2 | Bottom Up (ISO/GUM, fish bone/cause and effect diagram) | Standard deviation from PT studies only | | Eurachem/CITAC Guide |
| | | Control Samples - CRM Duplicate Analysis Instrument Calibration | CRM Instrument Calibration Recoveries of SS | |
| 3 | Standard deviation of replicate analyses multiplied by 2 or 3 | Control Samples - CRM | CRM Laboratory Bias from PT Studies Recoveries of SS | Eurachem/CITAC Guide |
| 4 | Top Down - precision and estimates of the method and laboratory bias | Control Samples - CRM | CRM Recoveries of SS | Eurachem/CITAC Guide |
| 5 | Top Down - precision and estimates of the method and laboratory bias | Control Samples - SS | Recoveries of SS | ISO/GUM |
| 6 | Top Down - precision and estimates of the method and laboratory bias | Control Samples - RM Duplicate Analysis | Instrument Calibration Standard Purity | Eurachem/CITAC Guide |
| 7 | Top Down - precision and estimates of the method and laboratory bias | Control Samples - RM Duplicate Analysis Instrument Calibration | | Eurachem/CITAC Guide |
| 8 | Top Down - precision and estimates of the method and laboratory bias | Control Samples - CRM Duplicate Analysis | CRM | Eurachem/CITAC Guide |
| 9 | Top Down - reproducibility (standard deviation) from PT studies used directly | Control Samples - CRM Duplicate Analysis Instrument Calibration | | Nordtest Report TR537 |
| 10 | Top Down - precision and estimates of the method and laboratory bias | Control Samples - SS Duplicate Analysis Instrument Calibration | CRM Instrument Calibration Recoveries of SS Standard Purity | IANZ Technical Guide |
| 11 | SD of replicate analyses x 2 x 100/85 x 100/mean | Control Samples | CRM Instrument Calibration | |
| 12 | | Control Samples - CRM Duplicate Analysis Instrument Calibration | CRM Instrument Calibration Recoveries of SS Standard Purity | Eurachem/CITAC Guide |
| 13 | Top Down - precision and estimates of the method and laboratory bias | Control Samples - CRM Duplicate Analysis | CRM Recoveries of SS | Top Down approach, John Eames |
| 14 | Top Down - precision and estimates of the method and laboratory bias | Control Samples - CRM Duplicate Analysis | CRM | NMI Uncertainty Course |
| 15 | Bottom Up (ISO/GUM, fish bone/cause and effect diagram) | Control Samples - SS Duplicate Analysis Instrument Calibration | CRM Instrument Calibration Recoveries of SS | Eurachem/CITAC Guide |
| 16 | Top Down - precision and estimates of the method and laboratory bias | Control Samples - SS Duplicate Analysis Instrument Calibration | Recoveries of SS | NATA Estimating and Reporting measurement |

| Lab. Code | Approach to Estimating MU | Information Sources for MU Estimation ^a | | Guide Document for Estimating MU |
|-----------|--|---|--|---|
| | | Precision | Method Bias | |
| | | | | uncertainty of chemical test results - Jan 2018 |
| 17 | Top Down - reproducibility (standard deviation) from PT studies used directly | Standard deviation from PT studies only | | Eurolab Technical Report No1/2007 |
| | | Control Samples | CRM Laboratory Bias from PT Studies | |
| 18* | Top Down - precision and estimates of the method and laboratory bias | Control Samples - CRM Duplicate Analysis Instrument Calibration | CRM Instrument Calibration Laboratory Bias from PT Studies | ASTM E2554-13 |
| 19 | Top Down - precision and estimates of the method and laboratory bias | Control Samples - RM Duplicate Analysis | CRM | NATA General Accreditation, Guidance, Estimating and Reporting MU |
| 20 | Top Down - precision and estimates of the method and laboratory bias | Control Samples - CRM Duplicate Analysis | CRM Instrument Calibration Recoveries of SS | NMI Uncertainty Course |
| 21 | Top Down - precision and estimates of the method and laboratory bias | Standard deviation from PT studies only | | Nordtest Report TR537 |
| | | Control Samples - RM Duplicate Analysis Instrument Calibration | CRM Instrument Calibration Recoveries of SS | |
| 22 | Top Down - precision and estimates of the method and laboratory bias | Control Samples Duplicate Analysis | CRM Variation in Sample Moisture Content Recoveries of SS | Nordtest Report TR537 |
| 23 | Top Down - precision and estimates of the method and laboratory bias | Control Samples Duplicate Analysis | CRM Recoveries of SS | ISO/GUM |
| 24 | | Duplicate Analysis | Instrument Calibration Standard Purity | |
| 25 | Standard deviation of replicate analyses multiplied by 2 or 3 | Standard deviation from PT studies only | | ISO/GUM |
| | | Duplicate Analysis Instrument Calibration | CRM | |
| 26 | Standard deviation of replicate analyses multiplied by 2 or 3 | Control Samples Duplicate Analysis | CRM Instrument Calibration Laboratory Bias from PT Studies | Eurachem/CITAC Guide |
| 27 | Standard deviation of replicate analyses multiplied by 2 or 3 | Control Samples - RM | Recoveries of SS | |
| 28 | Top Down - precision and estimates of the method and laboratory bias | Control Samples - CRM | CRM | ISO/GUM |
| 29 | Estimation of MU from within-laboratory data on bias and precision has been calculated by using the procedures outlined in ASTM E2554-13 Standard Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques | Control Samples Duplicate Analysis Instrument Calibration | CRM | ASTM E2554-13 |

| Lab. Code | Approach to Estimating MU | Information Sources for MU Estimation ^a | | Guide Document for Estimating MU |
|-----------|---|--|--|---|
| | | Precision | Method Bias | |
| 30 | Calculated from Standard deviation and concentration of long term in house QC samples | Control Samples - RM Duplicate Analysis | Instrument Calibration Laboratory Bias from PT Studies | NATA General Accreditation, Guidance, Estimating and Reporting MU (Replace TN 33) |

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

Table 12 Additional Information for Basis of Uncertainty Estimate

| Lab Code | Additional Information |
|----------|--|
| 18 | Estimation of MU from within-laboratory data on bias and precision has been calculated by using the procedures outlined in ASTM E2554-13 Standard Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques |

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

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

Table 13 Participants' Comments

| Lab Code | Participants' Comments | Study Co-ordinator's Response |
|----------|---|---|
| 3 | These work well for us. Maybe provide larger sample size for ongoing us as quality control samples. | The test samples of this study were checked for homogeneity and are well characterised, both by in-house testing and from the results of the proficiency round. Surplus of these test samples is available for purchase from NMI. |

5 PRESENTATION OF RESULTS AND STATISTICAL ANALYSIS

5.1 Results Summary

Participant results are listed in Tables 14 to 67 with resultant summary statistics: robust average, median, maximum, minimum, robust standard deviation (SD_{rob}) and robust coefficient of variation (CV_{rob}). Bar charts of results and performance scores are presented in Figures 2 to 55. An example chart with interpretation guide is shown in Figure 1.

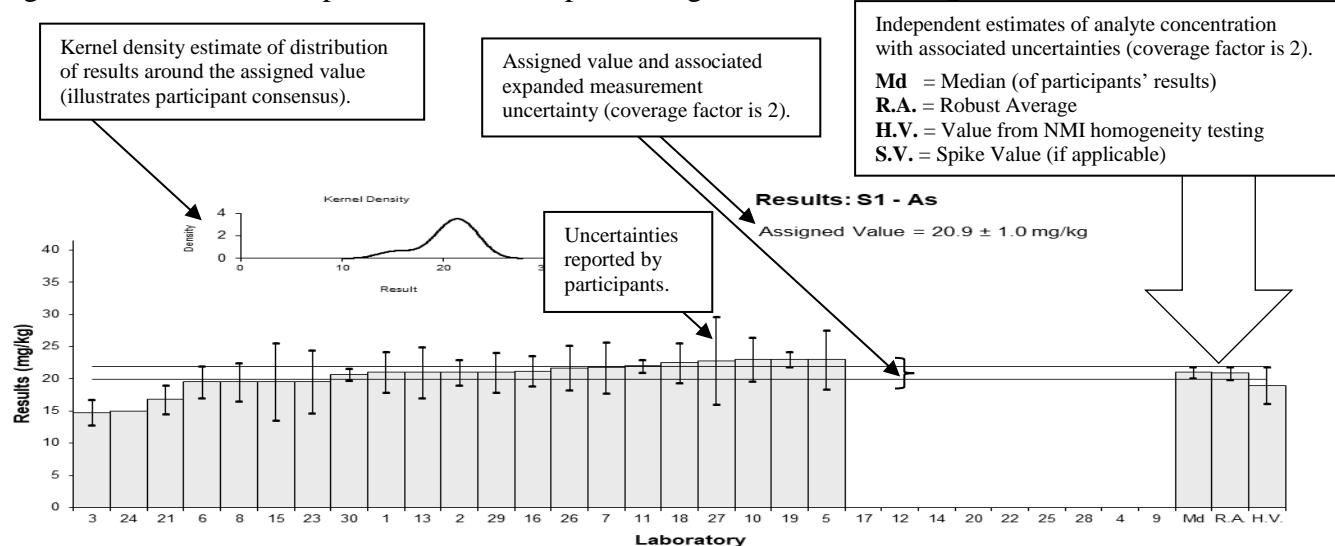


Figure 1 Guide to Presentation of Results

5.2 Outliers and Extreme Outliers

Outliers were results less than 50% and greater than 150% of the robust average and were removed before assigned value calculation. Extreme outliers were obvious blunders, such as those with incorrect units, decimal errors, or results from a different proficiency test item (gross errors) and were removed for calculation of summary statistics.^{3, 4}

5.3 Assigned Value

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

5.4 Robust Average

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

5.5 Robust Between-Laboratory Coefficient of Variation

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

5.6 Target Standard Deviation for Proficiency Assessment

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

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

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

5.7 z-Score

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

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

where:

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

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

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

5.8 E_n-Score

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

The E_n-score is complementary to the z-score in assessment of laboratory performance.

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

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

where:

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

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

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

5.9 Traceability and Measurement Uncertainty

Laboratories accredited to ISO/IEC Standard 17025:2018⁸ must establish and demonstrate the traceability and measurement uncertainty associated with their test results. Guidelines for quantifying uncertainty in analytical measurement are described in the Eurachem/CITAC Guide.⁹

6 TABLES AND FIGURES

Table 14

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Al |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E _n -Score |
|----------|-----------|-------------|---------|-----------------------|
| 1 | 7000 | 1100 | -1.18 | -1.01 |
| 2 | 7900 | 1000 | -0.47 | -0.42 |
| 3 | 7463.05 | 100 | -0.81 | -1.03 |
| 4 | NT | NT | | |
| 5 | 11000 | 2700 | 1.96 | 0.87 |
| 6 | 9280 | 900 | 0.61 | 0.58 |
| 7 | 8546 | 1680 | 0.04 | 0.02 |
| 8 | 6390 | 736 | -1.65 | -1.70 |
| 9 | NT | NT | | |
| 10 | 6500 | 2200 | -1.57 | -0.83 |
| 11 | 10200 | 2040 | 1.33 | 0.75 |
| 12 | NT | NT | | |
| 13 | 8700 | 1100 | 0.16 | 0.13 |
| 14 | NT | NT | | |
| 15 | 11700 | 1230 | 2.51 | 2.02 |
| 16 | 14349 | 1578 | 4.59 | 3.13 |
| 17 | 9820 | 1500 | 1.04 | 0.73 |
| 18 | 7830 | 1140 | -0.53 | -0.44 |
| 19 | 9530 | 953 | 0.81 | 0.75 |
| 20 | NT | NT | | |
| 21 | 4660 | 490 | -3.01 | -3.45 |
| 22 | NT | NT | | |
| 23 | 6080 | 1520 | -1.90 | -1.33 |
| 24 | 23607.181 | NR | 11.85 | 15.11 |
| 25 | NT | NT | | |
| 26 | 8700 | 1100 | 0.16 | 0.13 |
| 27 | 9841 | 2952 | 1.05 | 0.43 |
| 28 | NT | NT | | |
| 29 | 9800 | 1500 | 1.02 | 0.72 |
| 30 | 9200 | 370 | 0.55 | 0.66 |

Statistics

| | | |
|--------------------------|------------|------|
| Assigned Value* | 8500 | 1000 |
| Spike | Not Spiked | |
| Homogeneity Value | 8000 | 1200 |
| Robust Average | 8900 | 1200 |
| Median | 8950 | 780 |
| Mean | 9460 | |
| N | 22 | |
| Max. | 23607.181 | |
| Min. | 4660 | |
| Robust SD | 2200 | |
| Robust CV | 24% | |

*Robust Average excluding laboratories 16 and 24.

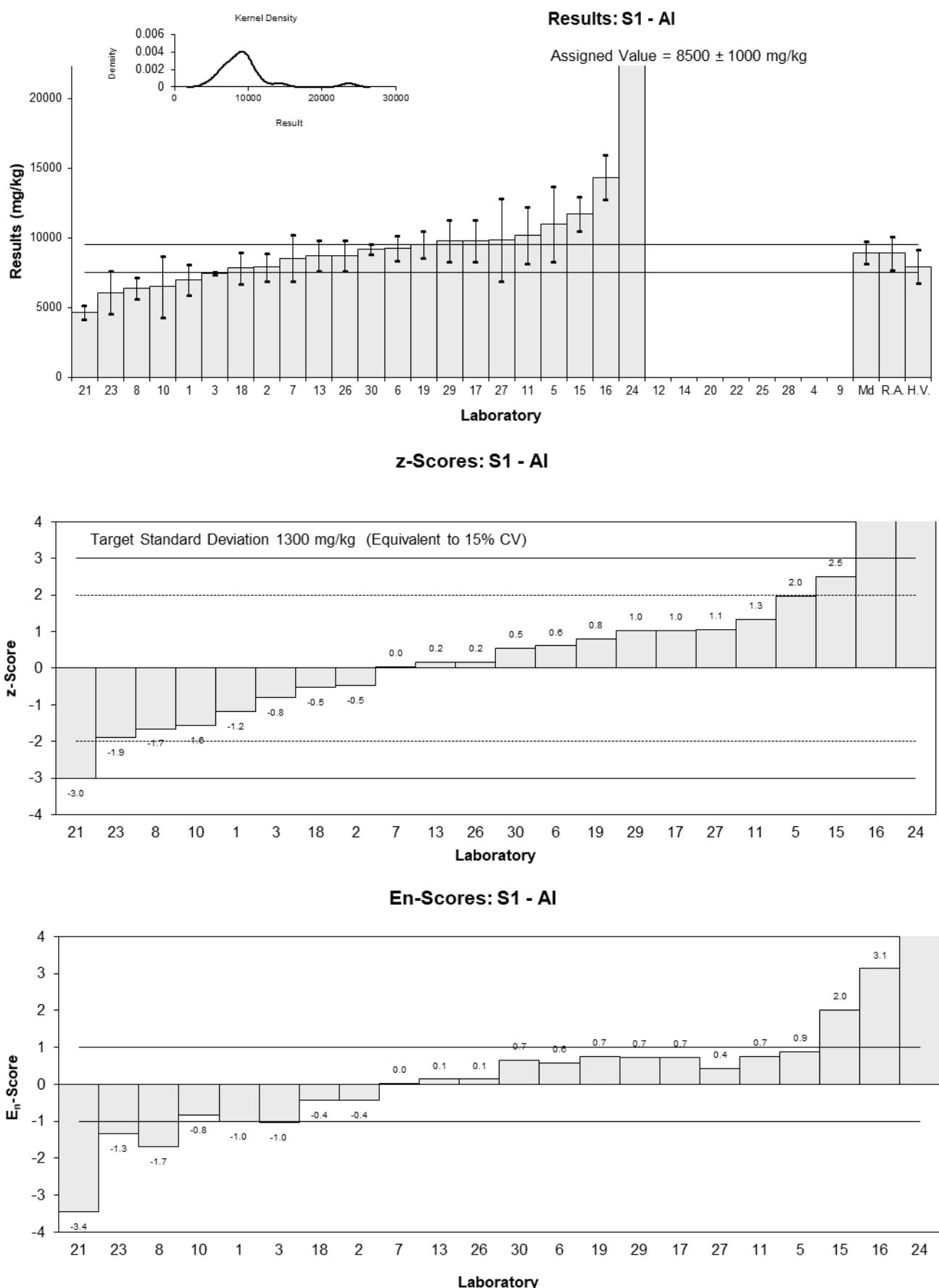


Figure 2

Table 15

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | As |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 21 | 3.15 | 0.05 | 0.03 |
| 2 | 21 | 2 | 0.05 | 0.04 |
| 3 | 14.75 | 2 | -2.94 | -2.75 |
| 4 | NT | NT | | |
| 5 | 23 | 4.6 | 1.00 | 0.45 |
| 6 | 19.5 | 2.5 | -0.67 | -0.52 |
| 7 | 21.74 | 4 | 0.40 | 0.20 |
| 8 | 19.5 | 3.0 | -0.67 | -0.44 |
| 9 | NT | NT | | |
| 10 | 23 | 3.4 | 1.00 | 0.59 |
| 11 | 22 | 1 | 0.53 | 0.78 |
| 12 | NT | NT | | |
| 13 | 21 | 4 | 0.05 | 0.02 |
| 14 | NT | NT | | |
| 15 | 19.6 | 6.0 | -0.62 | -0.21 |
| 16 | 21.2 | 2.33 | 0.14 | 0.12 |
| 17 | <25 | NR | | |
| 18 | 22.5 | 3.1 | 0.77 | 0.49 |
| 19 | 23 | 1.15 | 1.00 | 1.38 |
| 20 | NT | NT | | |
| 21 | 16.8 | 2.2 | -1.96 | -1.70 |
| 22 | NT | NT | | |
| 23 | 19.6 | 4.9 | -0.62 | -0.26 |
| 24 | 14.949 | NR | -2.85 | -5.95 |
| 25 | NT | NT | | |
| 26 | 21.7 | 3.5 | 0.38 | 0.22 |
| 27 | 22.8 | 6.8 | 0.91 | 0.28 |
| 28 | NT | NT | | |
| 29 | 21 | 3.1 | 0.05 | 0.03 |
| 30 | 20.7 | 0.9 | -0.10 | -0.15 |

Statistics

| | | |
|--------------------------|------------|-----|
| Assigned Value | 20.9 | 1.0 |
| Spike | Not Spiked | |
| Homogeneity Value | 19.0 | 2.9 |
| Robust Average | 20.9 | 1.0 |
| Median | 21.0 | 0.9 |
| Mean | 20.5 | |
| N | 21 | |
| Max. | 23 | |
| Min. | 14.75 | |
| Robust SD | 1.9 | |
| Robust CV | 8.9% | |

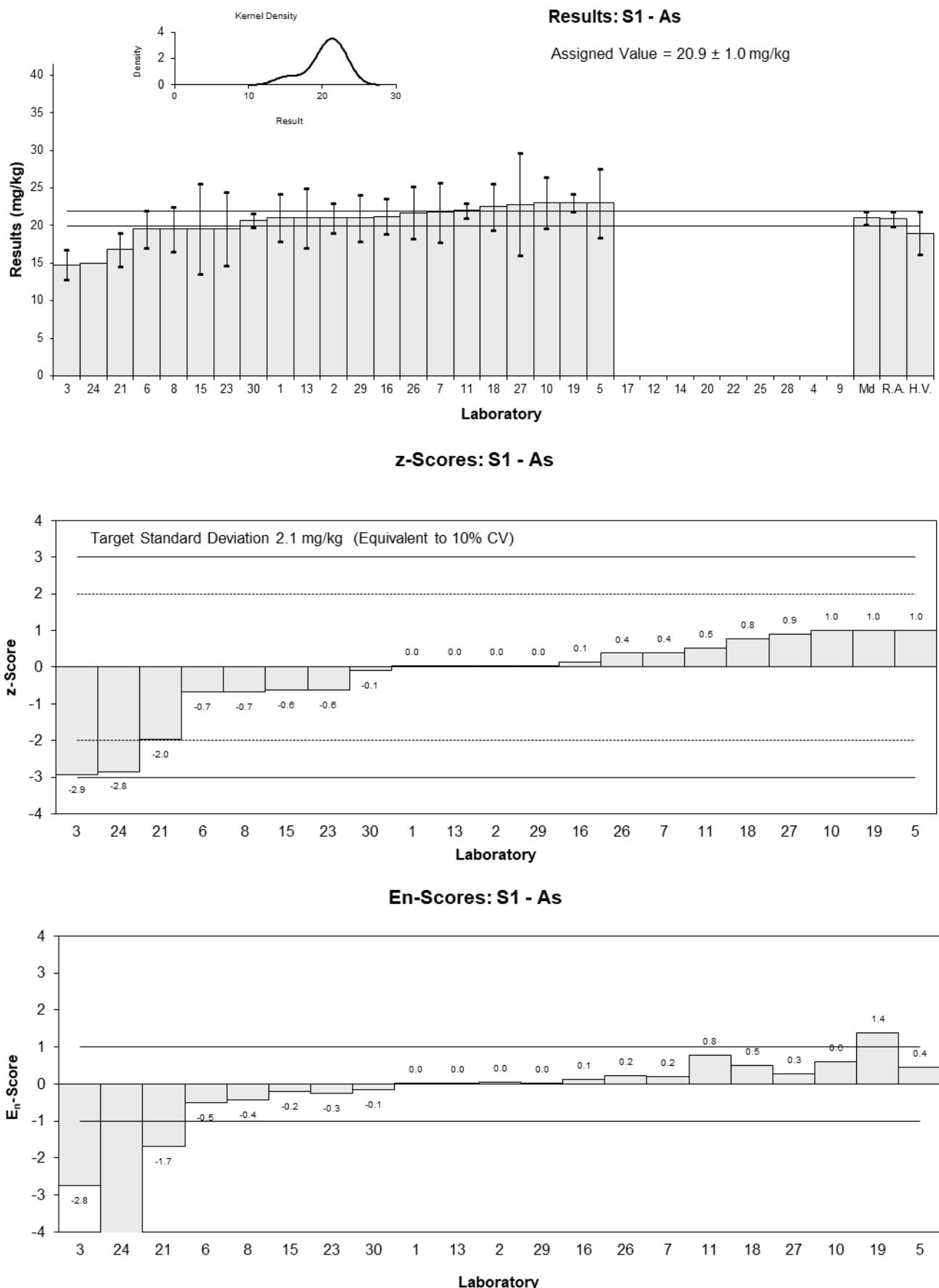


Figure 3

Table 16

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Cd |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 8.0 | 1.2 | -0.62 | -0.43 |
| 2 | 8 | 1.6 | -0.62 | -0.32 |
| 3 | 7.55 | 1 | -1.15 | -0.93 |
| 4 | NT | NT | | |
| 5 | 9.1 | 1.4 | 0.67 | 0.40 |
| 6 | 8.48 | 1.0 | -0.06 | -0.05 |
| 7 | 8.1 | 1 | -0.50 | -0.41 |
| 8 | 8.7 | 1.1 | 0.20 | 0.15 |
| 9 | NT | NT | | |
| 10 | 8.2 | 1.6 | -0.39 | -0.20 |
| 11 | 9 | 0.5 | 0.55 | 0.78 |
| 12 | NT | NT | | |
| 13 | 9.0 | 2.0 | 0.55 | 0.23 |
| 14 | NT | NT | | |
| 15 | 8.09 | 2.43 | -0.52 | -0.18 |
| 16 | 6.99 | 0.70 | -1.81 | -1.99 |
| 17 | 9.55 | 1.9 | 1.20 | 0.53 |
| 18 | 8.36 | 1.0 | -0.20 | -0.16 |
| 19 | 9 | 0.45 | 0.55 | 0.84 |
| 20 | NT | NT | | |
| 21 | 9.3 | 0.9 | 0.90 | 0.80 |
| 22 | NT | NT | | |
| 23 | 8.58 | 2.15 | 0.06 | 0.02 |
| 24 | 3.326 | NR | -6.10 | -15.77 |
| 25 | NT | NT | | |
| 26 | 8.9 | 1.3 | 0.43 | 0.28 |
| 27 | 9.0 | 2.7 | 0.55 | 0.17 |
| 28 | NT | NT | | |
| 29 | 8.1 | 1.2 | -0.50 | -0.35 |
| 30 | 8.61 | 1.3 | 0.09 | 0.06 |

Statistics

| | | |
|--------------------------|------------|------|
| Assigned Value* | 8.53 | 0.33 |
| Spike | Not Spiked | |
| Homogeneity Value | 7.9 | 1.2 |
| Robust Average | 8.48 | 0.36 |
| Median | 8.53 | 0.31 |
| Mean | 8.27 | |
| N | 22 | |
| Max. | 9.55 | |
| Min. | 3.326 | |
| Robust SD | 0.67 | |
| Robust CV | 7.9% | |

*Robust Average excluding laboratory 24.

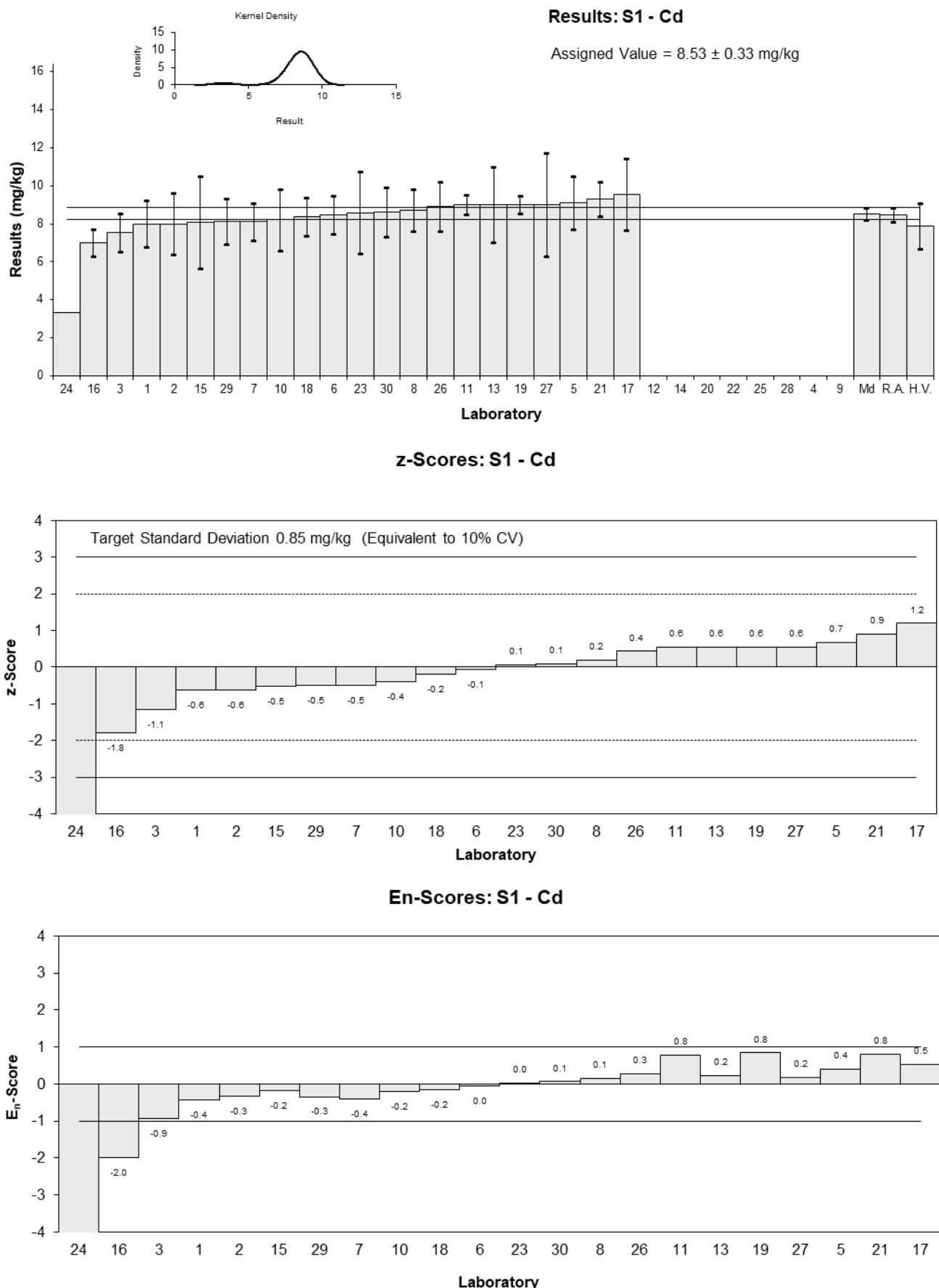


Figure 4

Table 17

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Co |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 8.3 | 1.245 | -0.65 | -0.44 |
| 2 | 9 | 0.8 | 0.14 | 0.13 |
| 3 | 6.98 | 1 | -2.14 | -1.77 |
| 4 | NT | NT | | |
| 5 | 10 | 2.4 | 1.26 | 0.46 |
| 6 | 9.10 | 1.0 | 0.25 | 0.20 |
| 7 | 8.2 | 1 | -0.77 | -0.63 |
| 8 | 8.4 | 1.3 | -0.54 | -0.35 |
| 9 | NT | NT | | |
| 10 | 7.7 | 1.1 | -1.33 | -1.01 |
| 11 | 9 | 1 | 0.14 | 0.11 |
| 12 | NT | NT | | |
| 13 | 9.0 | 2.3 | 0.14 | 0.05 |
| 14 | NT | NT | | |
| 15 | 9.24 | 3.00 | 0.41 | 0.12 |
| 16 | 8.35 | 0.84 | -0.60 | -0.57 |
| 17 | 12.0 | 3 | 3.51 | 1.03 |
| 18 | 8.78 | 1.2 | -0.11 | -0.08 |
| 19 | 9 | NR | 0.14 | 0.31 |
| 20 | NT | NT | | |
| 21 | 8.4 | 1.7 | -0.54 | -0.28 |
| 22 | NT | NT | | |
| 23 | 8.3 | 2.1 | -0.65 | -0.27 |
| 24 | 10.278 | NR | 1.57 | 3.58 |
| 25 | NT | NT | | |
| 26 | 9.3 | 1.4 | 0.47 | 0.29 |
| 27 | 9.4 | 2.8 | 0.59 | 0.18 |
| 28 | NT | NT | | |
| 29 | 8.9 | 1.3 | 0.02 | 0.01 |
| 30 | 9.25 | 0.3 | 0.42 | 0.75 |

Statistics

| | | |
|--------------------------|------------|------|
| Assigned Value | 8.88 | 0.39 |
| Spike | Not Spiked | |
| Homogeneity Value | 8.9 | 1.3 |
| Robust Average | 8.88 | 0.39 |
| Median | 9.00 | 0.33 |
| Mean | 8.95 | |
| N | 22 | |
| Max. | 12 | |
| Min. | 6.98 | |
| Robust SD | 0.73 | |
| Robust CV | 8.2% | |

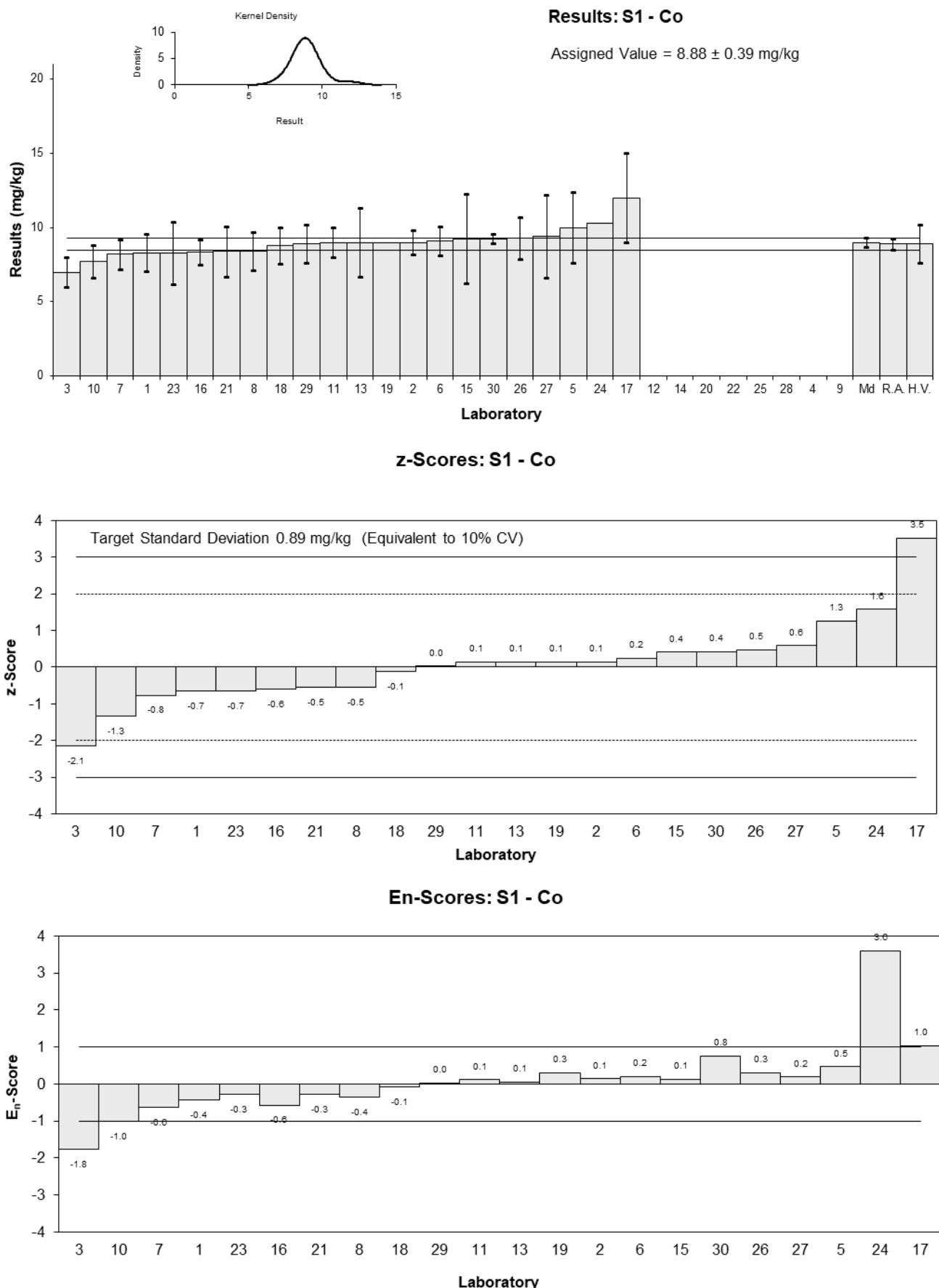


Figure 5

Table 18

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Cr |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 21 | 3.15 | 0.20 | 0.16 |
| 2 | 17 | 3.4 | -1.11 | -0.86 |
| 3 | 16.7 | 2 | -1.21 | -1.31 |
| 4 | NT | NT | | |
| 5 | 25 | 3.8 | 1.50 | 1.07 |
| 6 | 22.2 | 2.5 | 0.59 | 0.56 |
| 7 | 19.26 | 6 | -0.37 | -0.18 |
| 8 | 16.6 | 3.9 | -1.24 | -0.87 |
| 9 | NT | NT | | |
| 10 | 16 | 2.4 | -1.44 | -1.41 |
| 11 | 24 | 1 | 1.18 | 1.61 |
| 12 | NT | NT | | |
| 13 | 21 | 4 | 0.20 | 0.13 |
| 14 | NT | NT | | |
| 15 | 24.3 | 3.3 | 1.27 | 1.01 |
| 16 | 10.9 | 1.09 | -3.10 | -4.17 |
| 17 | 28.0 | 8.4 | 2.48 | 0.88 |
| 18 | 20.9 | 2.9 | 0.16 | 0.14 |
| 19 | 22 | 1.32 | 0.52 | 0.67 |
| 20 | NT | NT | | |
| 21 | 17.5 | 1.8 | -0.95 | -1.08 |
| 22 | NT | NT | | |
| 23 | 17.6 | 4.4 | -0.92 | -0.58 |
| 24 | 40.385 | NR | 6.53 | 9.99 |
| 25 | NT | NT | | |
| 26 | 18.4 | 3.2 | -0.65 | -0.53 |
| 27 | 22.5 | 6.7 | 0.69 | 0.30 |
| 28 | NT | NT | | |
| 29 | 23 | 3.4 | 0.85 | 0.66 |
| 30 | 22.68 | 1.4 | 0.75 | 0.93 |

Statistics

| | | |
|--------------------------|------------|-----|
| Assigned Value* | 20.4 | 2.0 |
| Spike | Not Spiked | |
| Homogeneity Value | 22.0 | 3.3 |
| Robust Average | 20.7 | 2.1 |
| Median | 21.0 | 2.1 |
| Mean | 21.2 | |
| N | 22 | |
| Max. | 40.385 | |
| Min. | 10.9 | |
| Robust SD | 4.0 | |
| Robust CV | 19% | |

*Robust Average excluding laboratory 24.

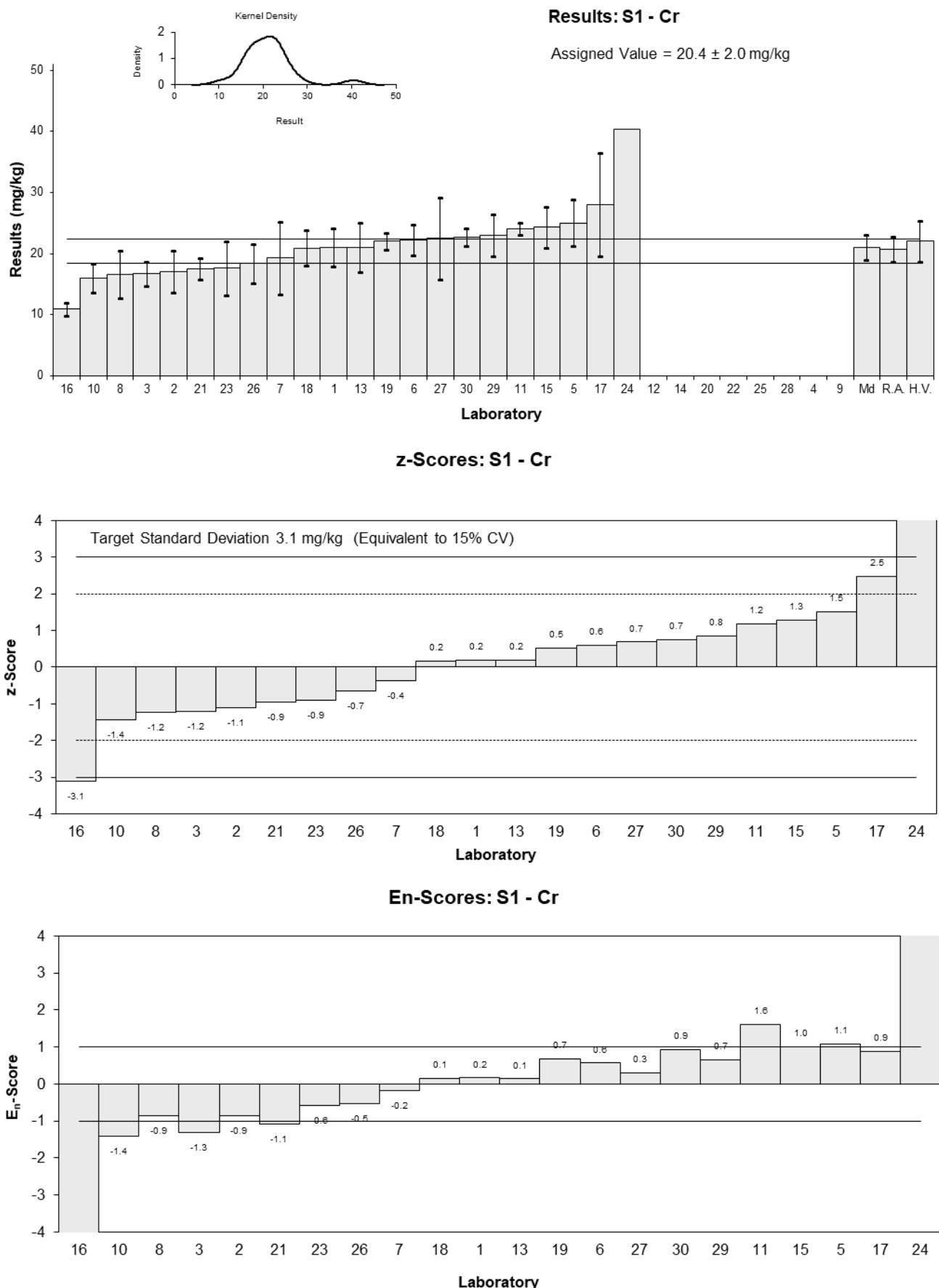


Figure 6

Table 19

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Cu |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 21 | 3.15 | -1.39 | -1.04 |
| 2 | 24 | 2 | -0.16 | -0.18 |
| 3 | 25 | 2 | 0.25 | 0.27 |
| 4 | NT | NT | | |
| 5 | 27 | 5.4 | 1.07 | 0.47 |
| 6 | 25.4 | 2.6 | 0.41 | 0.36 |
| 7 | 25 | 4 | 0.25 | 0.15 |
| 8 | 23.7 | 3.8 | -0.29 | -0.18 |
| 9 | NT | NT | | |
| 10 | 23 | 3.5 | -0.57 | -0.39 |
| 11 | 24 | 1 | -0.16 | -0.30 |
| 12 | NT | NT | | |
| 13 | 25 | 4 | 0.25 | 0.15 |
| 14 | NT | NT | | |
| 15 | 25.6 | 2.7 | 0.49 | 0.42 |
| 16 | 24.2 | 2.9 | -0.08 | -0.07 |
| 17 | 29.0 | 8.7 | 1.89 | 0.53 |
| 18 | 23.3 | 3.1 | -0.45 | -0.34 |
| 19 | 27 | 1.62 | 1.07 | 1.40 |
| 20 | NT | NT | | |
| 21 | 22.1 | 3.7 | -0.94 | -0.60 |
| 22 | NT | NT | | |
| 23 | 22.1 | 5.5 | -0.94 | -0.41 |
| 24 | 23.302 | NR | -0.45 | -1.22 |
| 25 | NT | NT | | |
| 26 | 24.4 | 3.6 | 0.00 | 0.00 |
| 27 | 25.6 | 7.7 | 0.49 | 0.15 |
| 28 | NT | NT | | |
| 29 | 23 | 3.7 | -0.57 | -0.37 |
| 30 | 24.77 | 2.5 | 0.15 | 0.14 |

Statistics

| | | |
|--------------------------|------------|-----|
| Assigned Value | 24.4 | 0.9 |
| Spike | Not Spiked | |
| Homogeneity Value | 25.3 | 3.8 |
| Robust Average | 24.4 | 0.9 |
| Median | 24.3 | 0.7 |
| Mean | 24.4 | |
| N | 22 | |
| Max. | 29 | |
| Min. | 21 | |
| Robust SD | 1.7 | |
| Robust CV | 7.2% | |

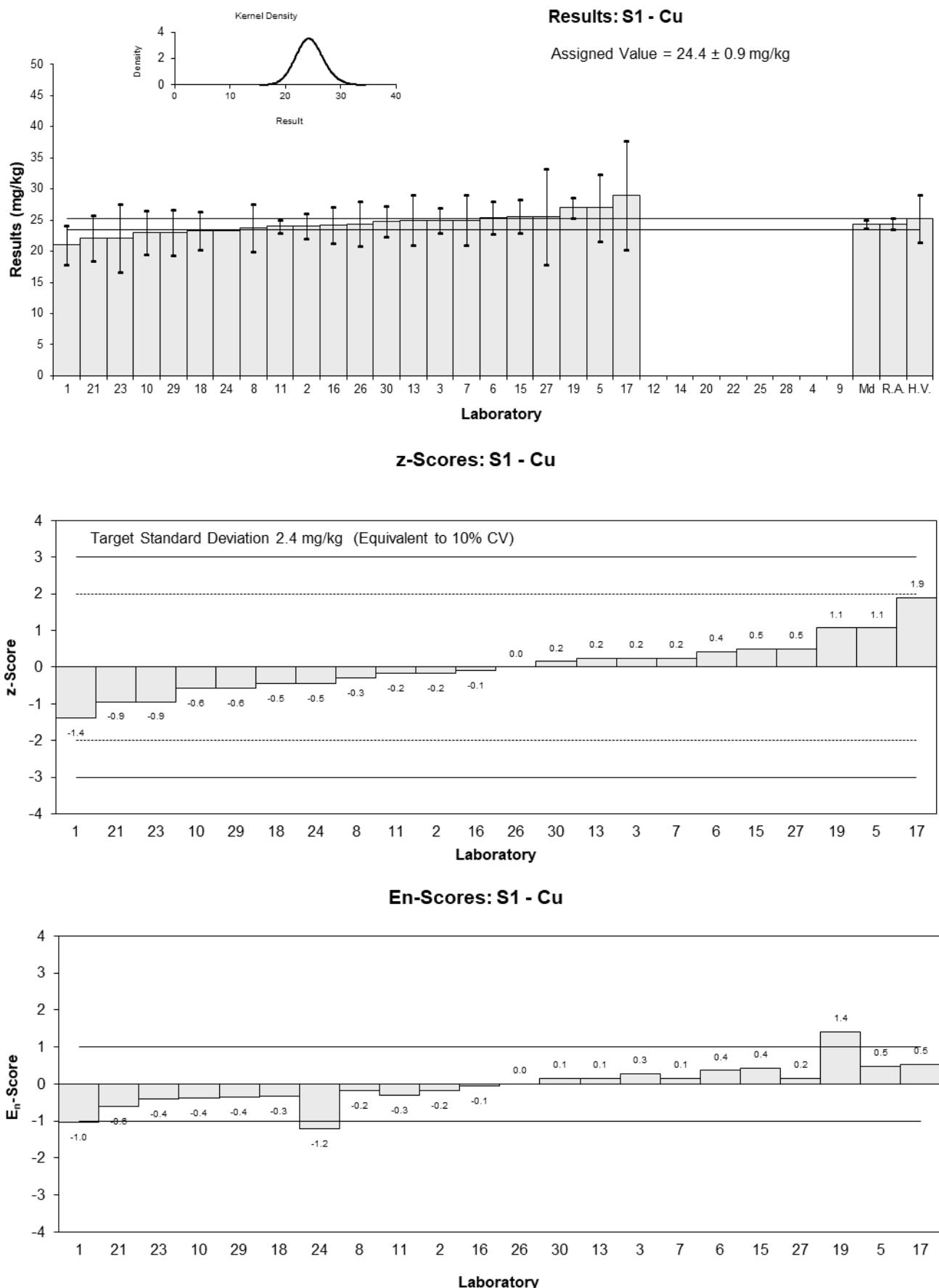


Figure 7

Table 20

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Hg |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 9.7 | 1.455 | 0.53 | 0.32 |
| 2 | 9.8 | 1.3 | 0.64 | 0.43 |
| 3 | 6.59 | 1 | -2.84 | -2.42 |
| 4 | NT | NT | | |
| 5 | 8.6 | 1.3 | -0.66 | -0.45 |
| 6 | 9.39 | 1.2 | 0.20 | 0.14 |
| 7 | 9.3 | NR | 0.10 | 0.22 |
| 8 | 8.9 | 2.2 | -0.34 | -0.14 |
| 9 | NT | NT | | |
| 10 | 8.2 | 1.4 | -1.10 | -0.69 |
| 11 | 10 | 1 | 0.86 | 0.73 |
| 12 | NT | NT | | |
| 13 | 9.31 | 2 | 0.11 | 0.05 |
| 14 | NT | NT | | |
| 15 | 8.39 | 0.48 | -0.89 | -1.30 |
| 16 | 8.8 | 1.5 | -0.45 | -0.26 |
| 17 | 11.0 | 3.3 | 1.94 | 0.54 |
| 18 | 9.61 | 1.1 | 0.43 | 0.34 |
| 19 | 9.2 | 0.46 | -0.01 | -0.02 |
| 20 | 8.81075 | 1.101 | -0.43 | -0.34 |
| 21 | 10.3 | 1.0 | 1.18 | 1.01 |
| 22 | NT | NT | | |
| 23 | 7.01 | 1.75 | -2.39 | -1.22 |
| 24 | 2.950 | NR | -6.80 | -15.27 |
| 25 | NT | NT | | |
| 26 | 9.6 | 1.6 | 0.42 | 0.24 |
| 27 | 9.8 | 2.9 | 0.64 | 0.20 |
| 28 | NT | NT | | |
| 29 | 9.5 | 1.5 | 0.31 | 0.19 |
| 30 | 8.93 | 0.7 | -0.30 | -0.35 |

Statistics

| | | |
|--------------------------|------------|------|
| Assigned Value* | 9.21 | 0.41 |
| Spike | Not Spiked | |
| Homogeneity Value | 8.9 | 1.3 |
| Robust Average | 9.13 | 0.45 |
| Median | 9.30 | 0.32 |
| Mean | 8.86 | |
| N | 23 | |
| Max. | 11 | |
| Min. | 2.95 | |
| Robust SD | 0.86 | |
| Robust CV | 9.5% | |

*Robust Average excluding Laboratory 24.

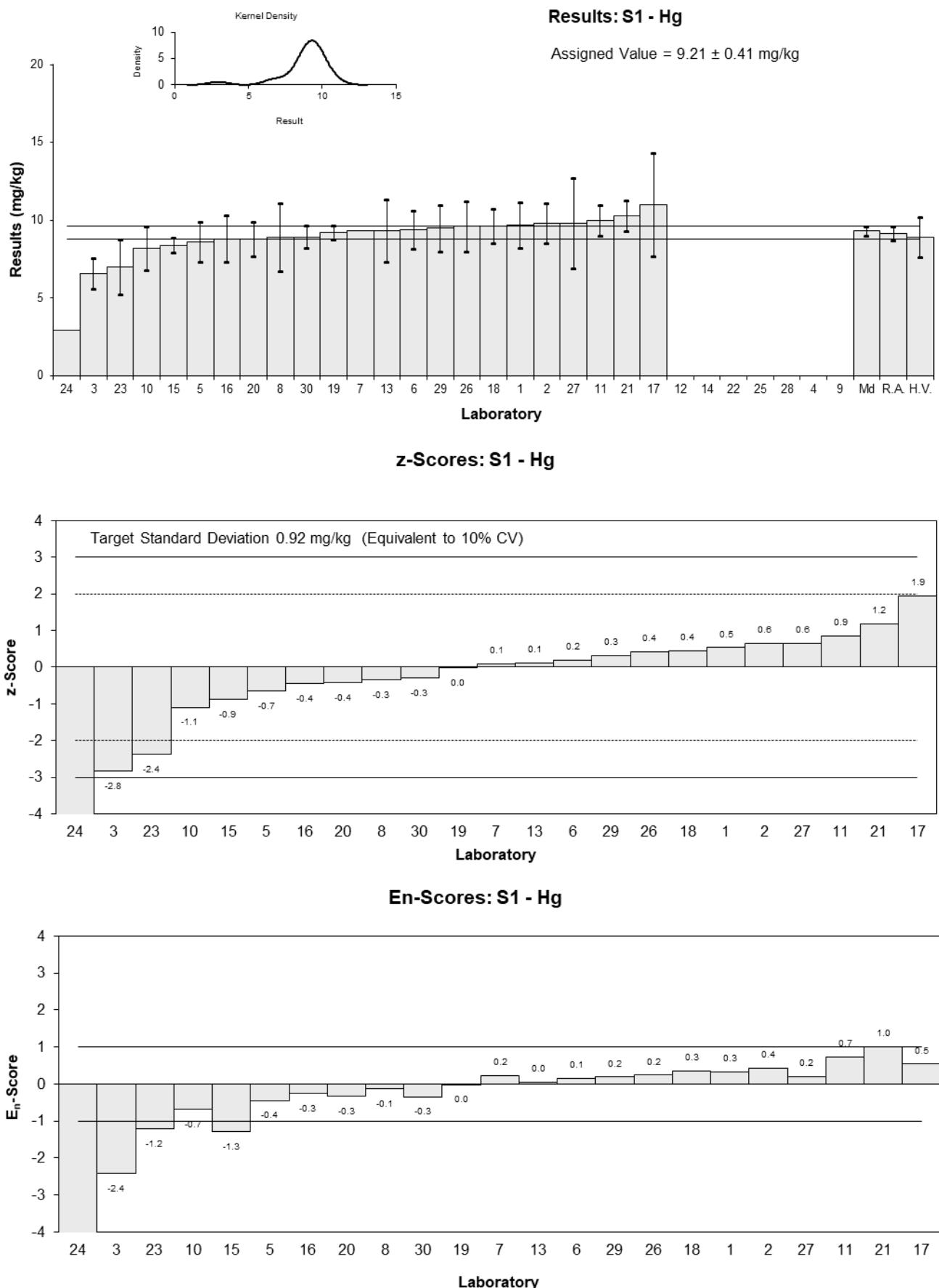


Figure 8

Table 21

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Mn |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 310 | 46.5 | -1.29 | -0.96 |
| 2 | 340 | 52 | -0.45 | -0.30 |
| 3 | 369.75 | 40 | 0.39 | 0.33 |
| 4 | NT | NT | | |
| 5 | 370 | 74 | 0.39 | 0.19 |
| 6 | 356 | 35 | 0.00 | 0.00 |
| 7 | 342 | 52 | -0.39 | -0.26 |
| 8 | 353 | 60 | -0.08 | -0.05 |
| 9 | NT | NT | | |
| 10 | 350 | 77 | -0.17 | -0.08 |
| 11 | 371 | 74 | 0.42 | 0.20 |
| 12 | NT | NT | | |
| 13 | 370 | 50 | 0.39 | 0.27 |
| 14 | NT | NT | | |
| 15 | 365 | 60 | 0.25 | 0.15 |
| 16 | 358 | 35.8 | 0.06 | 0.05 |
| 17 | 402 | 60 | 1.29 | 0.75 |
| 18 | 330 | 35.9 | -0.73 | -0.69 |
| 19 | 380 | NR | 0.67 | 2.00 |
| 20 | NT | NT | | |
| 21 | 337 | 33 | -0.53 | -0.54 |
| 22 | NT | NT | | |
| 23 | 352 | 87.9 | -0.11 | -0.05 |
| 24 | 328.349 | NR | -0.78 | -2.30 |
| 25 | NT | NT | | |
| 26 | 388 | 39 | 0.90 | 0.78 |
| 27 | 360 | 108 | 0.11 | 0.04 |
| 28 | NT | NT | | |
| 29 | 330 | 45 | -0.73 | -0.56 |
| 30 | 367.1 | 12 | 0.31 | 0.65 |

Statistics

| | | |
|--------------------------|------------|----|
| Assigned Value | 356 | 12 |
| Spike | Not Spiked | |
| Homogeneity Value | 340 | 50 |
| Robust Average | 356 | 12 |
| Median | 357 | 9 |
| Mean | 356 | |
| N | 22 | |
| Max. | 402 | |
| Min. | 310 | |
| Robust SD | 22 | |
| Robust CV | 6.1% | |

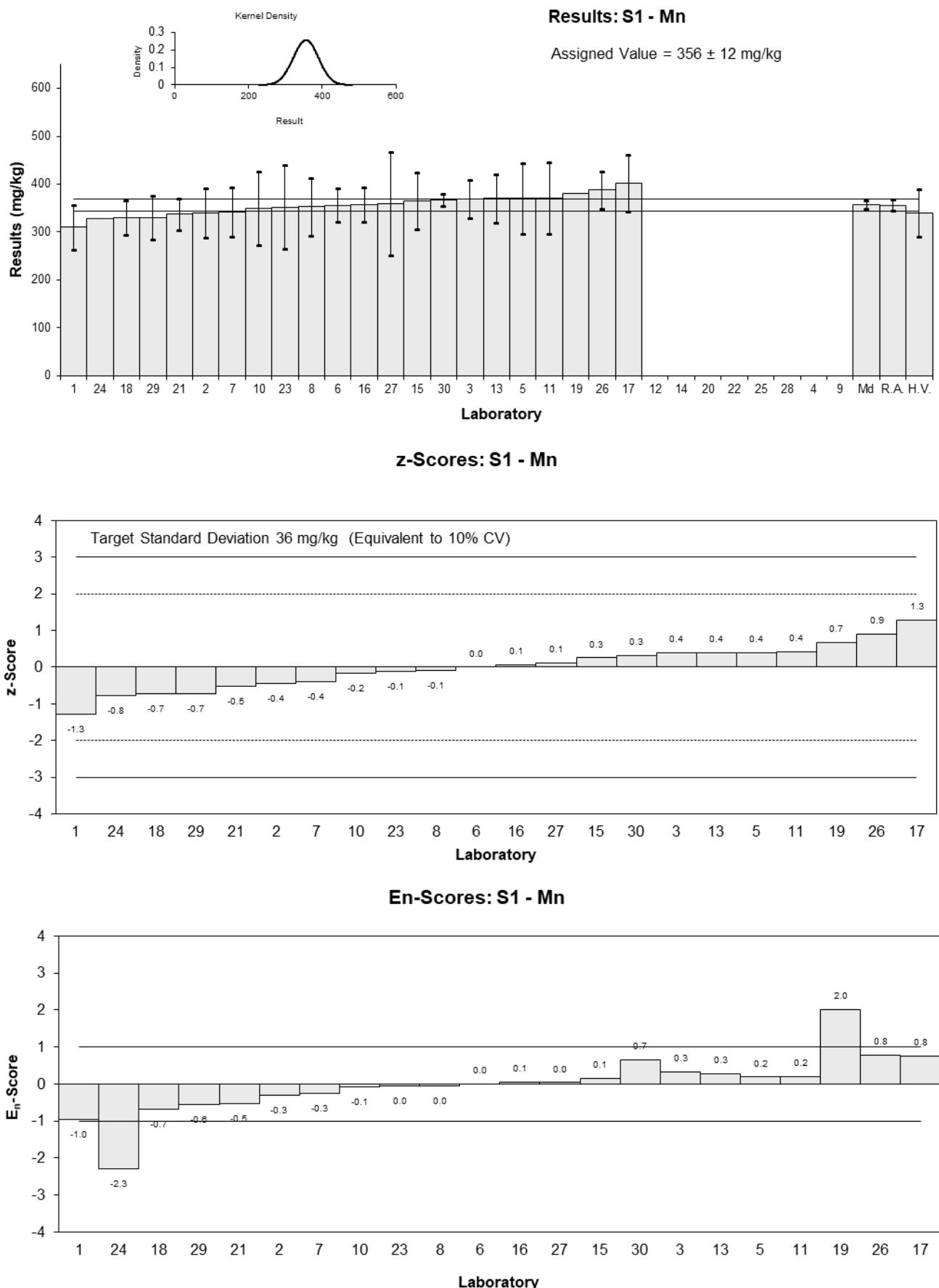


Figure 9

Table 22

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Mo |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 8.3 | 1.245 | -0.19 | -0.11 |
| 2 | 8 | 1.6 | -0.54 | -0.27 |
| 3 | 6.45 | 1 | -2.38 | -1.70 |
| 4 | NT | NT | | |
| 5 | 10 | 2.0 | 1.82 | 0.73 |
| 6 | 9.28 | 1.0 | 0.97 | 0.69 |
| 7 | 9.7 | NR | 1.47 | 1.97 |
| 8 | 8.0 | 1.5 | -0.54 | -0.28 |
| 9 | NT | NT | | |
| 10 | 8.0 | 1.20 | -0.54 | -0.34 |
| 11 | NT | NT | | |
| 12 | NT | NT | | |
| 13 | 9.1 | 1.8 | 0.76 | 0.34 |
| 14 | NT | NT | | |
| 15 | 5.89 | 1.80 | -3.04 | -1.35 |
| 16 | 8.34 | 1.0 | -0.14 | -0.10 |
| 17 | 7.80 | 2.7 | -0.78 | -0.24 |
| 18 | 8.99 | 1.2 | 0.63 | 0.39 |
| 19 | 9 | NR | 0.64 | 0.86 |
| 20 | NT | NT | | |
| 21 | 7.4 | 1.5 | -1.25 | -0.65 |
| 22 | NT | NT | | |
| 23 | 8.1 | 2.0 | -0.43 | -0.17 |
| 24 | 6.480 | NR | -2.34 | -3.14 |
| 25 | NT | NT | | |
| 26 | 9.2 | 1.7 | 0.87 | 0.41 |
| 27 | 9.8 | 2.9 | 1.58 | 0.45 |
| 28 | NT | NT | | |
| 29 | 9.4 | 1.5 | 1.11 | 0.58 |
| 30 | 9.01 | 0.5 | 0.65 | 0.68 |

Statistics

| | | |
|--------------------------|------------|------|
| Assigned Value | 8.46 | 0.63 |
| Spike | Not Spiked | |
| Homogeneity Value | 9.4 | 1.4 |
| Robust Average | 8.46 | 0.63 |
| Median | 8.34 | 0.51 |
| Mean | 8.39 | |
| N | 21 | |
| Max. | 10 | |
| Min. | 5.89 | |
| Robust SD | 1.1 | |
| Robust CV | 14% | |

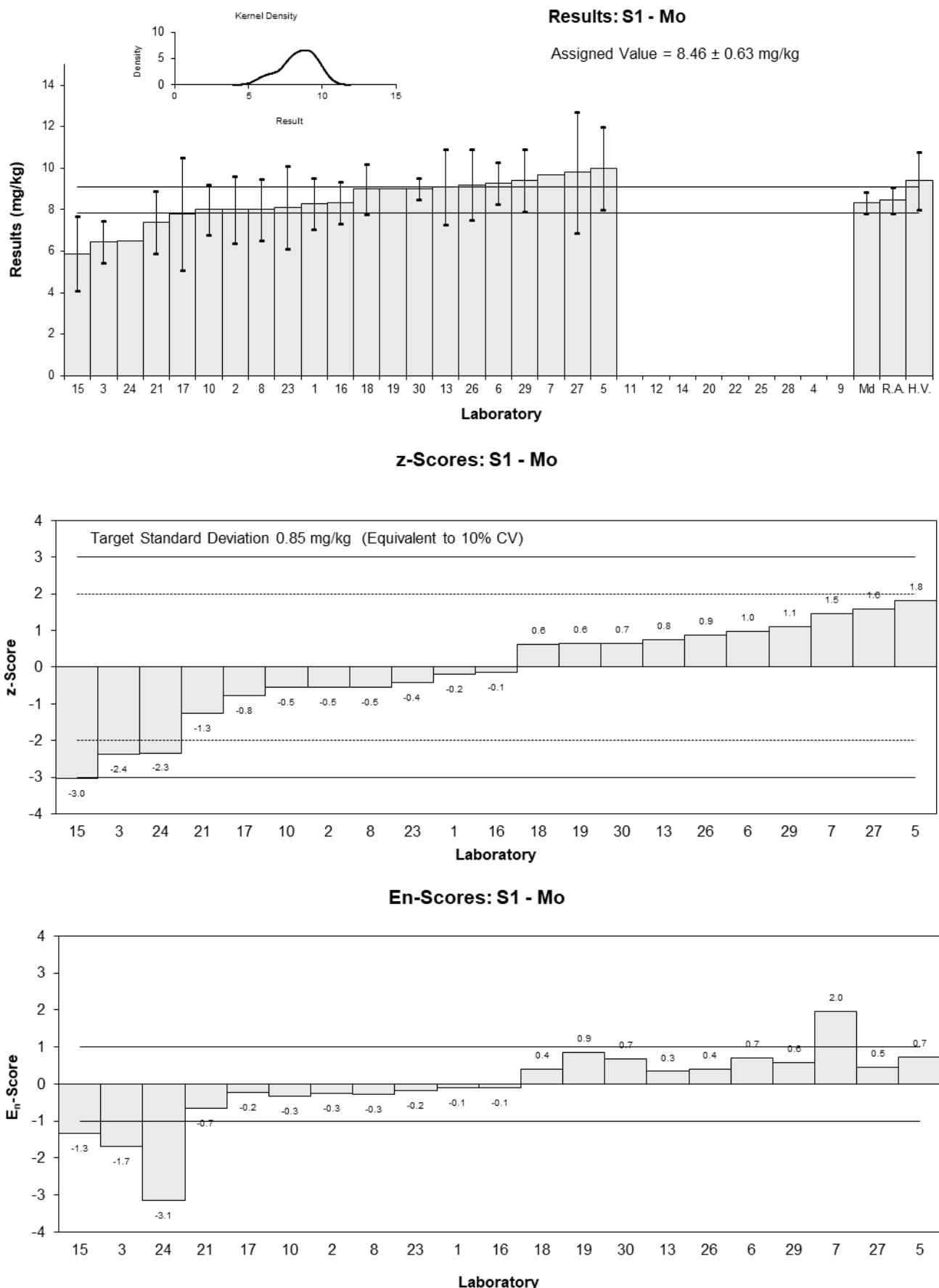


Figure 10

Table 23

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Ni |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 13 | 1.95 | -0.43 | -0.43 |
| 2 | 12 | 1 | -0.91 | -1.56 |
| 3 | 10.2 | 1 | -1.77 | -3.03 |
| 4 | NT | NT | | |
| 5 | 16 | 3.2 | 1.01 | 0.64 |
| 6 | 14.6 | 1.5 | 0.34 | 0.42 |
| 7 | 13.2 | 2 | -0.34 | -0.33 |
| 8 | 12.3 | 2.0 | -0.77 | -0.76 |
| 9 | NT | NT | | |
| 10 | 13 | 3.1 | -0.43 | -0.28 |
| 11 | 15 | 1 | 0.53 | 0.90 |
| 12 | NT | NT | | |
| 13 | 14 | 4 | 0.05 | 0.02 |
| 14 | NT | NT | | |
| 15 | 13.7 | 3.5 | -0.10 | -0.06 |
| 16 | 13.9 | 1.39 | 0.00 | 0.00 |
| 17 | 18.0 | 7 | 1.97 | 0.58 |
| 18 | 14.0 | 1.8 | 0.05 | 0.05 |
| 19 | 15 | 1.5 | 0.53 | 0.66 |
| 20 | NT | NT | | |
| 21 | 12.1 | 1.1 | -0.86 | -1.38 |
| 22 | NT | NT | | |
| 23 | 13.0 | 3.2 | -0.43 | -0.27 |
| 24 | 14.495 | NR | 0.29 | 0.85 |
| 25 | NT | NT | | |
| 26 | 13.6 | 2.2 | -0.14 | -0.13 |
| 27 | 14.8 | 4.4 | 0.43 | 0.20 |
| 28 | NT | NT | | |
| 29 | 15 | 2.4 | 0.53 | 0.44 |
| 30 | 14.67 | 0.9 | 0.37 | 0.68 |

Statistics

| | | |
|--------------------------|------------|-----|
| Assigned Value | 13.9 | 0.7 |
| Spike | Not Spiked | |
| Homogeneity Value | 15.3 | 2.3 |
| Robust Average | 13.9 | 0.7 |
| Median | 14.0 | 0.6 |
| Mean | 13.9 | |
| N | 22 | |
| Max. | 18 | |
| Min. | 10.2 | |
| Robust SD | 1.4 | |
| Robust CV | 10% | |

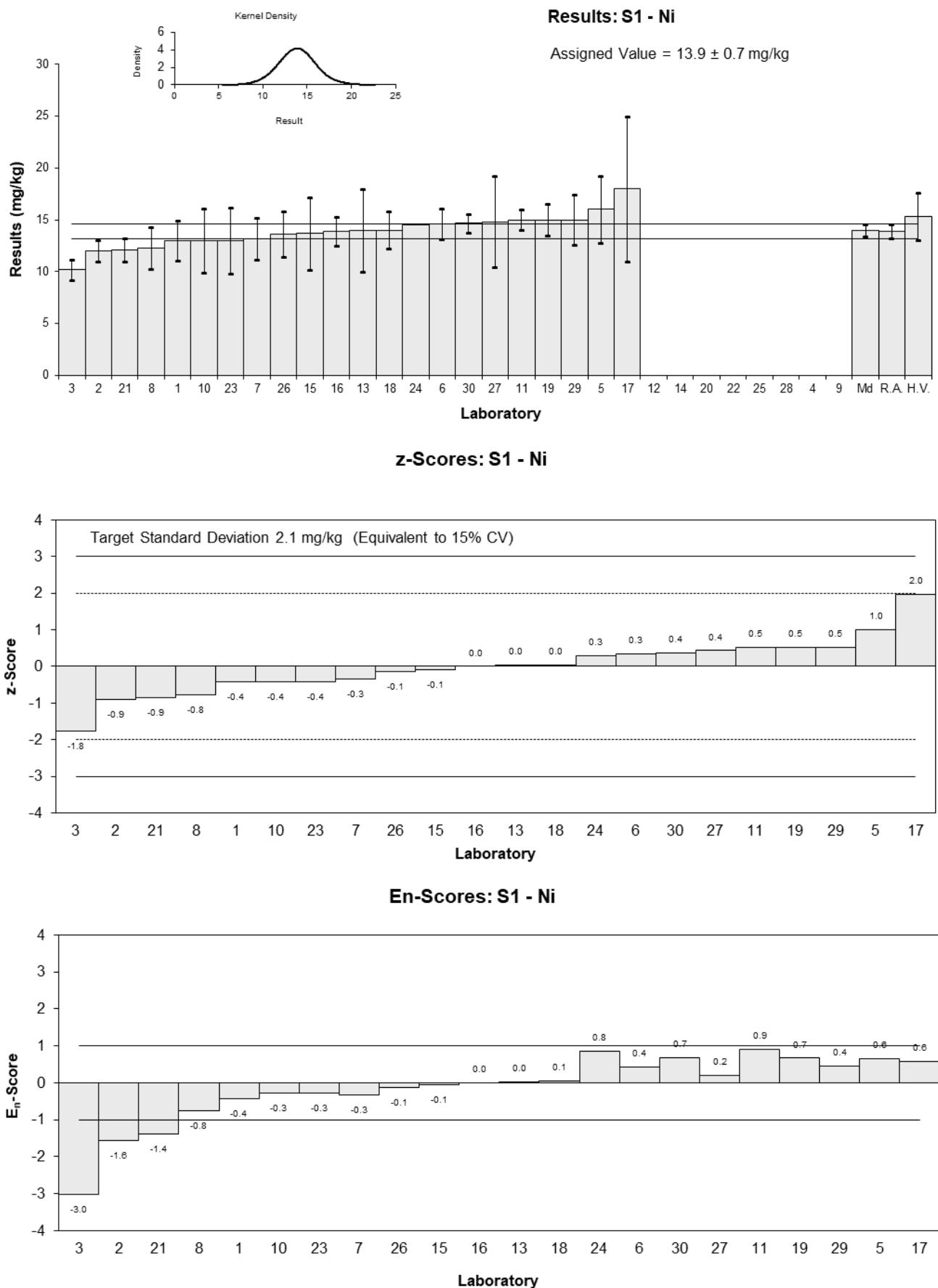


Figure 11

Table 24

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Pb |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 27 | 4.05 | -0.59 | -0.40 |
| 2 | 27 | 5.4 | -0.59 | -0.31 |
| 3 | 23.55 | 2 | -1.79 | -2.16 |
| 4 | NT | NT | | |
| 5 | 30 | 4.5 | 0.45 | 0.28 |
| 6 | 29.0 | 3.5 | 0.10 | 0.08 |
| 7 | 27.7 | 5 | -0.35 | -0.19 |
| 8 | 27.1 | 5.2 | -0.56 | -0.30 |
| 9 | NT | NT | | |
| 10 | 27 | 4.4 | -0.59 | -0.37 |
| 11 | 29 | 1 | 0.10 | 0.18 |
| 12 | NT | NT | | |
| 13 | 29 | 6 | 0.10 | 0.05 |
| 14 | NT | NT | | |
| 15 | 29.7 | 8.2 | 0.35 | 0.12 |
| 16 | 25.7 | 2.31 | -1.05 | -1.13 |
| 17 | 37.0 | 9.2 | 2.89 | 0.89 |
| 18 | 28.3 | 3.6 | -0.14 | -0.10 |
| 19 | 31 | 3.1 | 0.80 | 0.68 |
| 20 | NT | NT | | |
| 21 | 32.4 | 3.5 | 1.29 | 0.99 |
| 22 | NT | NT | | |
| 23 | 22.4 | 5.6 | -2.20 | -1.10 |
| 24 | 30.892 | NR | 0.76 | 1.69 |
| 25 | NT | NT | | |
| 26 | 31.2 | 4.7 | 0.87 | 0.51 |
| 27 | 30.1 | 9.0 | 0.49 | 0.15 |
| 28 | NT | NT | | |
| 29 | 28 | 4.3 | -0.24 | -0.16 |
| 30 | 28.87 | 1.5 | 0.06 | 0.09 |

Statistics

| | | |
|--------------------------|------------|-----|
| Assigned Value | 28.7 | 1.3 |
| Spike | Not Spiked | |
| Homogeneity Value | 29.3 | 4.4 |
| Robust Average | 28.7 | 1.3 |
| Median | 28.9 | 1.2 |
| Mean | 28.7 | |
| N | 22 | |
| Max. | 37 | |
| Min. | 22.4 | |
| Robust SD | 2.4 | |
| Robust CV | 8.5% | |

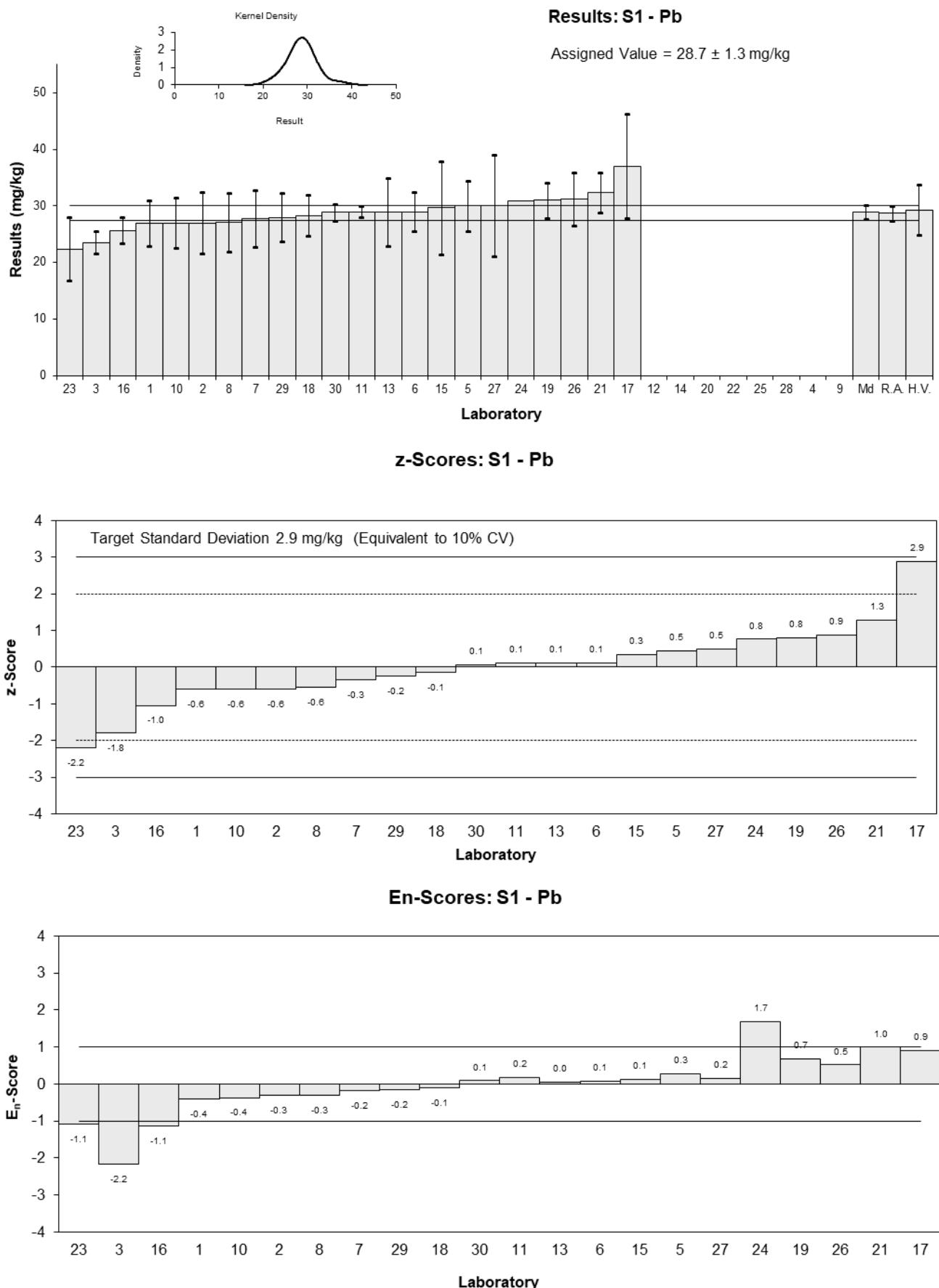


Figure 12

Table 25

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Sb |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty |
|-----------------|---------------|--------------------|
| 1 | 7 | 1.1 |
| 2 | 4.5 | 0.9 |
| 3 | 7.6 | 1 |
| 4 | NT | NT |
| 5 | <10 | NR |
| 6 | 11.4 | 1.2 |
| 7 | 5.9 | NR |
| 8 | 4.3 | 1.1 |
| 9 | NT | NT |
| 10 | 5.1 | 1.7 |
| 11 | NT | NT |
| 12 | NT | NT |
| 13 | NT | NT |
| 14 | NT | NT |
| 15 | 4.94 | 1.40 |
| 16 | 3.35 | 0.37 |
| 17 | <100 | NR |
| 18 | 8.50 | 1.2 |
| 19 | NT | NT |
| 20 | NT | NT |
| 21 | 4.1 | 0.4 |
| 22 | NT | NT |
| 23 | 9.8 | 2.4 |
| 24 | 7.762 | NR |
| 25 | NT | NT |
| 26 | 5.22 | 0.98 |
| 27 | 8.5 | 2.5 |
| 28 | NT | NT |
| 29 | <10 | NR |
| 30 | 9.08 | 0.8 |

Statistics

| | | |
|--------------------------|------------|------|
| Assigned Value | Not Set | |
| Spike | Not Spiked | |
| Homogeneity Value | 5.40 | 0.80 |
| Robust Average | 6.6 | 1.6 |
| Median | 6.5 | 1.6 |
| Mean | 6.7 | |
| N | 16 | |
| Max. | 11.4 | |
| Min. | 3.35 | |
| Robust SD | 2.5 | |
| Robust CV | 38% | |

Results: S1 - Sb

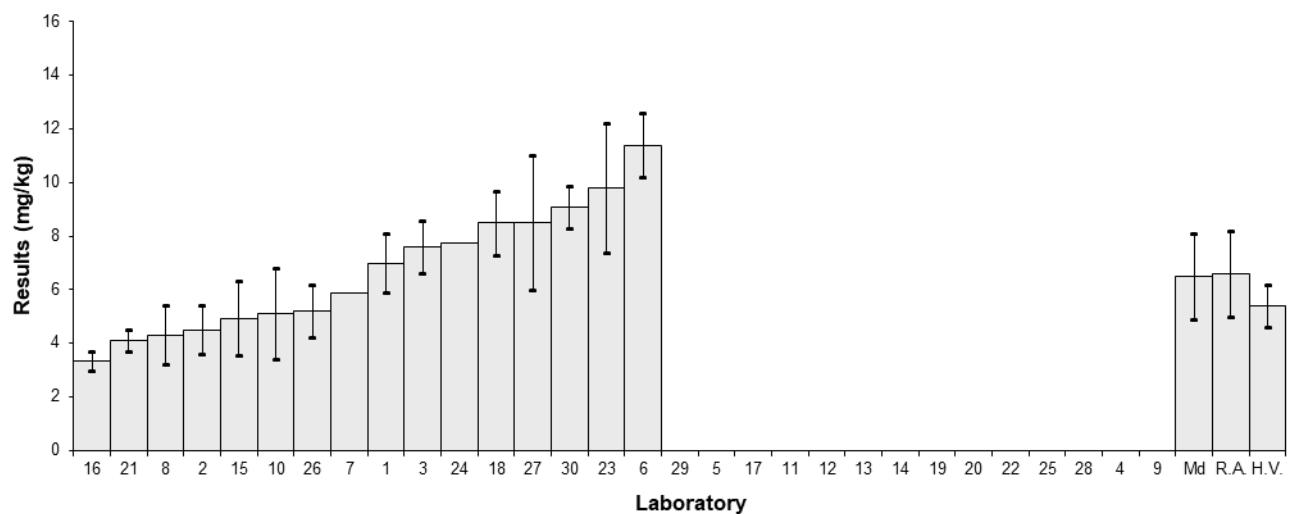


Figure 13

Table 26

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Se |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 9.5 | 1.43 | 0.71 | 0.56 |
| 2 | 9 | 1.8 | 0.33 | 0.21 |
| 3 | 4.7 | 1 | -3.01 | -3.02 |
| 4 | NT | NT | | |
| 5 | 8.8 | 1.8 | 0.17 | 0.11 |
| 6 | 9.12 | 1.1 | 0.42 | 0.40 |
| 7 | 10 | 3 | 1.10 | 0.46 |
| 8 | 7 | 2 | -1.23 | -0.73 |
| 9 | NT | NT | | |
| 10 | 7.2 | 1.1 | -1.07 | -1.01 |
| 11 | NT | NT | | |
| 12 | NT | NT | | |
| 13 | 10.6 | 2.2 | 1.57 | 0.86 |
| 14 | NT | NT | | |
| 15 | 9.40 | 3.62 | 0.64 | 0.22 |
| 16 | 8.08 | 0.97 | -0.39 | -0.40 |
| 17 | <100 | NR | | |
| 18 | 9.47 | 1.1 | 0.69 | 0.65 |
| 19 | 9.7 | 0.485 | 0.87 | 1.19 |
| 20 | NT | NT | | |
| 21 | 7.4 | 1.2 | -0.92 | -0.82 |
| 22 | NT | NT | | |
| 23 | 6.6 | 1.7 | -1.54 | -1.05 |
| 24 | 4.006 | NR | -3.55 | -5.65 |
| 25 | NT | NT | | |
| 26 | < 20 | 14 | | |
| 27 | 9.2 | 2.8 | 0.48 | 0.21 |
| 28 | NT | NT | | |
| 29 | 8.6 | 1.4 | 0.02 | 0.01 |
| 30 | 8.17 | 0.4 | -0.32 | -0.45 |

Statistics

| | | |
|--------------------------|------------|------|
| Assigned Value* | 8.58 | 0.81 |
| Spike | Not Spiked | |
| Homogeneity Value | 9.0 | 1.4 |
| Robust Average | 8.43 | 0.87 |
| Median | 8.80 | 0.51 |
| Mean | 8.24 | |
| N | 19 | |
| Max. | 10.6 | |
| Min. | 4.006 | |
| Robust SD | 1.5 | |
| Robust CV | 18% | |

*Robust Average excluding laboratory 24.

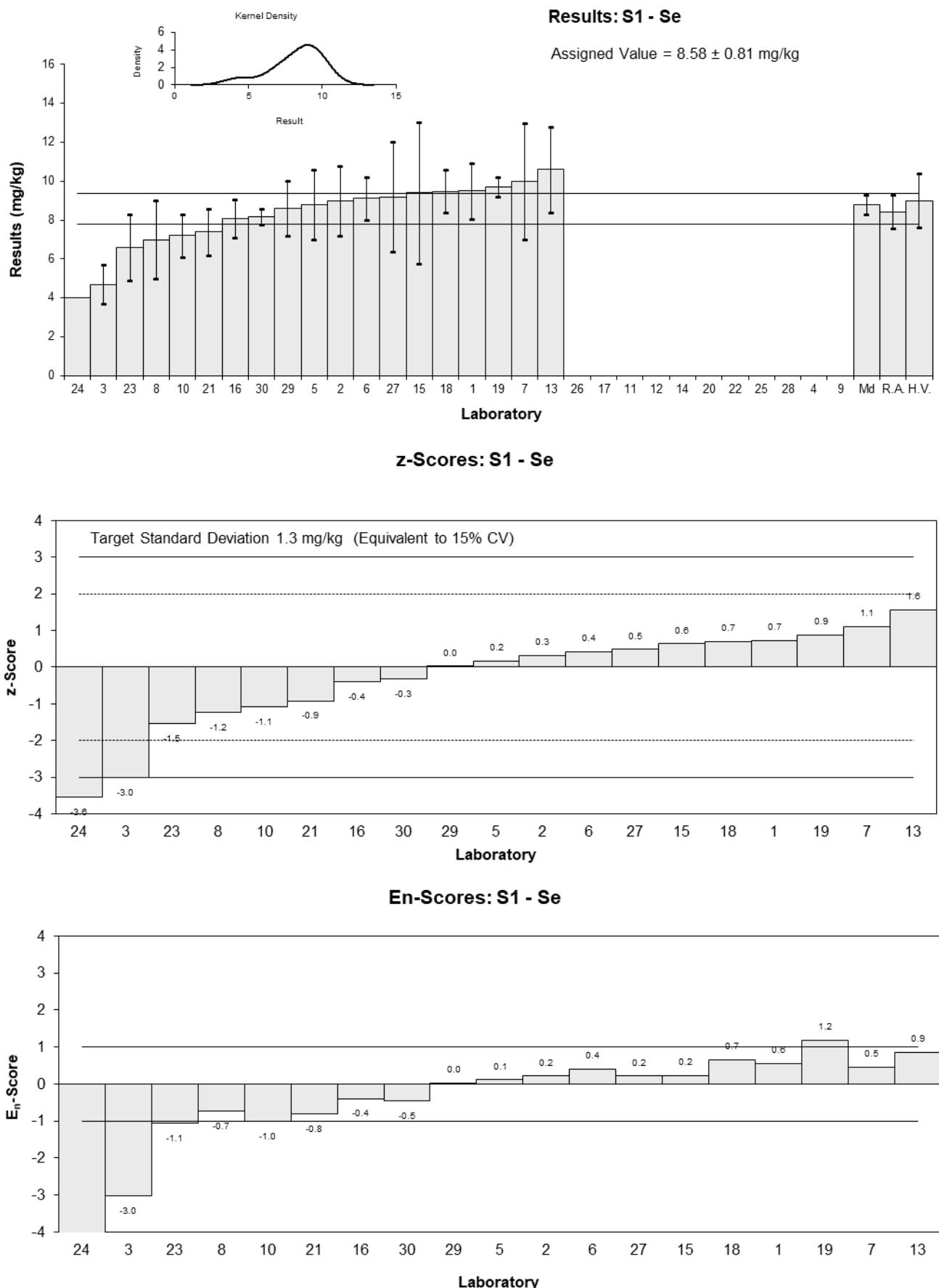


Figure 14

Table 27

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Th |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 4.6 | 0.69 | 1.02 | 0.88 |
| 2 | 3.7 | 0.74 | -0.16 | -0.13 |
| 3 | NT | NT | | |
| 4 | NT | NT | | |
| 5 | NT | NT | | |
| 6 | 3.72 | 0.5 | -0.13 | -0.13 |
| 7 | 3.6 | NR | -0.29 | -0.39 |
| 8 | 2.2 | 0.6 | -2.12 | -1.97 |
| 9 | NT | NT | | |
| 10 | NT | NT | | |
| 11 | NT | NT | | |
| 12 | NT | NT | | |
| 13 | NT | NT | | |
| 14 | NT | NT | | |
| 15 | 3.99 | 1.39 | 0.22 | 0.11 |
| 16 | NT | NT | | |
| 17 | NT | NT | | |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | NT | NT | | |
| 22 | NT | NT | | |
| 23 | NR | NR | | |
| 24 | NR | NR | | |
| 25 | NT | NT | | |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | NT | NT | | |
| 29 | NT | NT | | |
| 30 | 4.21 | 0.7 | 0.51 | 0.44 |

Statistics

| | | |
|--------------------------|------------|------|
| Assigned Value | 3.82 | 0.56 |
| Spike | Not Spiked | |
| Homogeneity Value | 4.60 | 0.70 |
| Robust Average | 3.82 | 0.56 |
| Median | 3.72 | 0.37 |
| Mean | 3.72 | |
| N | 7 | |
| Max. | 4.6 | |
| Min. | 2.2 | |
| Robust SD | 0.60 | |
| Robust CV | 16% | |

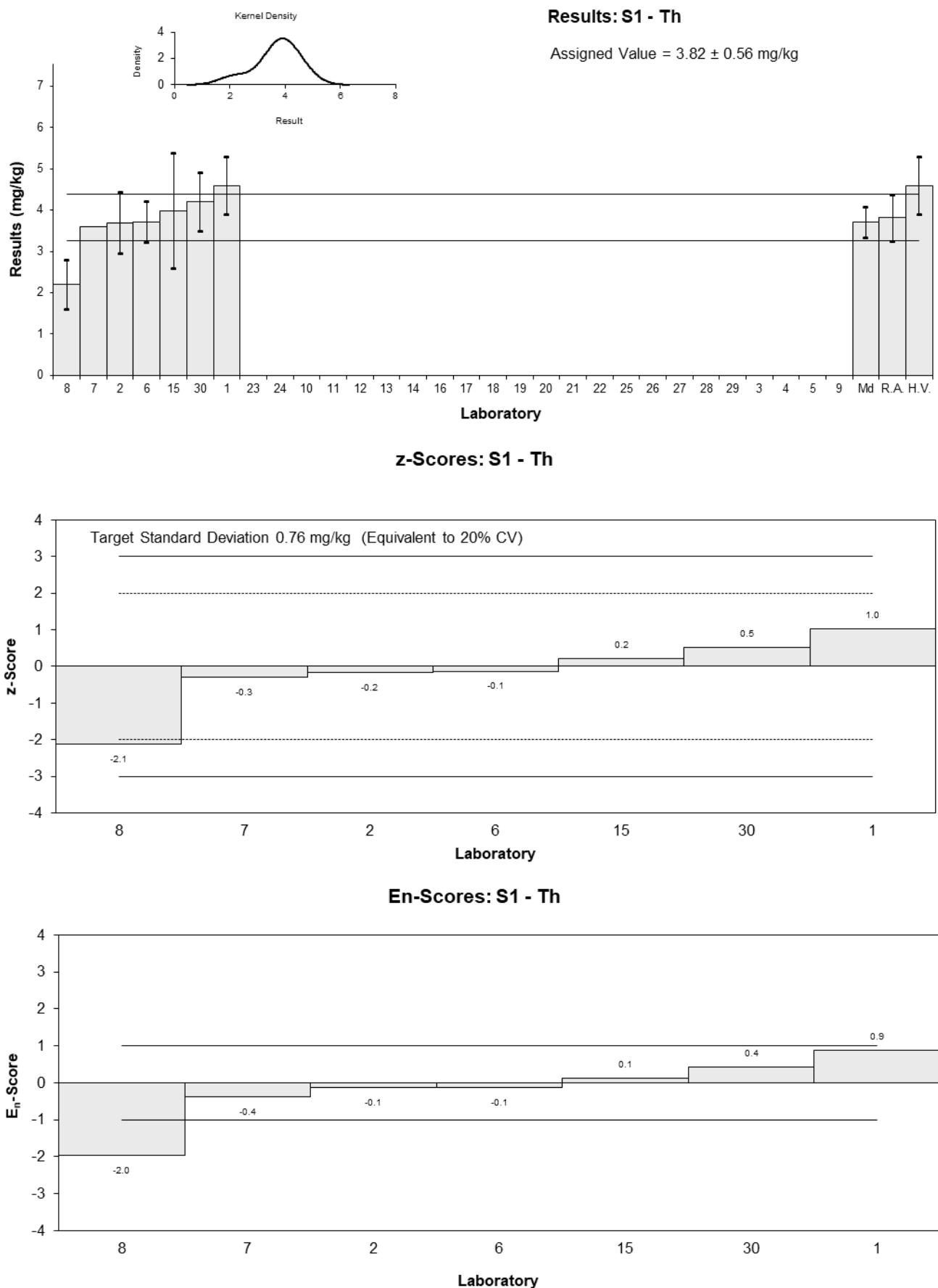


Figure 15

Table 28

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | U |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 0.8 | 0.12 | -0.77 | -0.49 |
| 2 | 0.8 | 0.16 | -0.77 | -0.39 |
| 3 | NT | NT | | |
| 4 | NT | NT | | |
| 5 | <10 | NR | | |
| 6 | 0.90 | 0.1 | 0.38 | 0.28 |
| 7 | 0.8 | NR | -0.77 | -1.06 |
| 8 | 0.8 | 0.2 | -0.77 | -0.32 |
| 9 | NT | NT | | |
| 10 | 0.73 | 0.11 | -1.58 | -1.08 |
| 11 | NT | NT | | |
| 12 | NT | NT | | |
| 13 | 0.94 | 0.2 | 0.84 | 0.35 |
| 14 | NT | NT | | |
| 15 | 0.86 | 0.26 | -0.08 | -0.03 |
| 16 | NT | NT | | |
| 17 | NT | NT | | |
| 18 | 0.81 | 0.1 | -0.66 | -0.48 |
| 19 | 1.1 | NR | 2.69 | 3.70 |
| 20 | NT | NT | | |
| 21 | 1.10 | 0.12 | 2.69 | 1.72 |
| 22 | NT | NT | | |
| 23 | NR | NR | | |
| 24 | NR | NR | | |
| 25 | NT | NT | | |
| 26 | 0.869 | 0.085 | 0.02 | 0.02 |
| 27 | 0.93 | 0.28 | 0.73 | 0.22 |
| 28 | NT | NT | | |
| 29 | <10 | NR | | |
| 30 | 0.876 | 0.05 | 0.10 | 0.11 |

Statistics

| | | |
|--------------------------|------------|-------|
| Assigned Value | 0.867 | 0.063 |
| Spike | Not Spiked | |
| Homogeneity Value | 0.89 | 0.13 |
| Robust Average | 0.867 | 0.063 |
| Median | 0.865 | 0.055 |
| Mean | 0.880 | |
| N | 14 | |
| Max. | 1.1 | |
| Min. | 0.73 | |
| Robust SD | 0.095 | |
| Robust CV | 11% | |

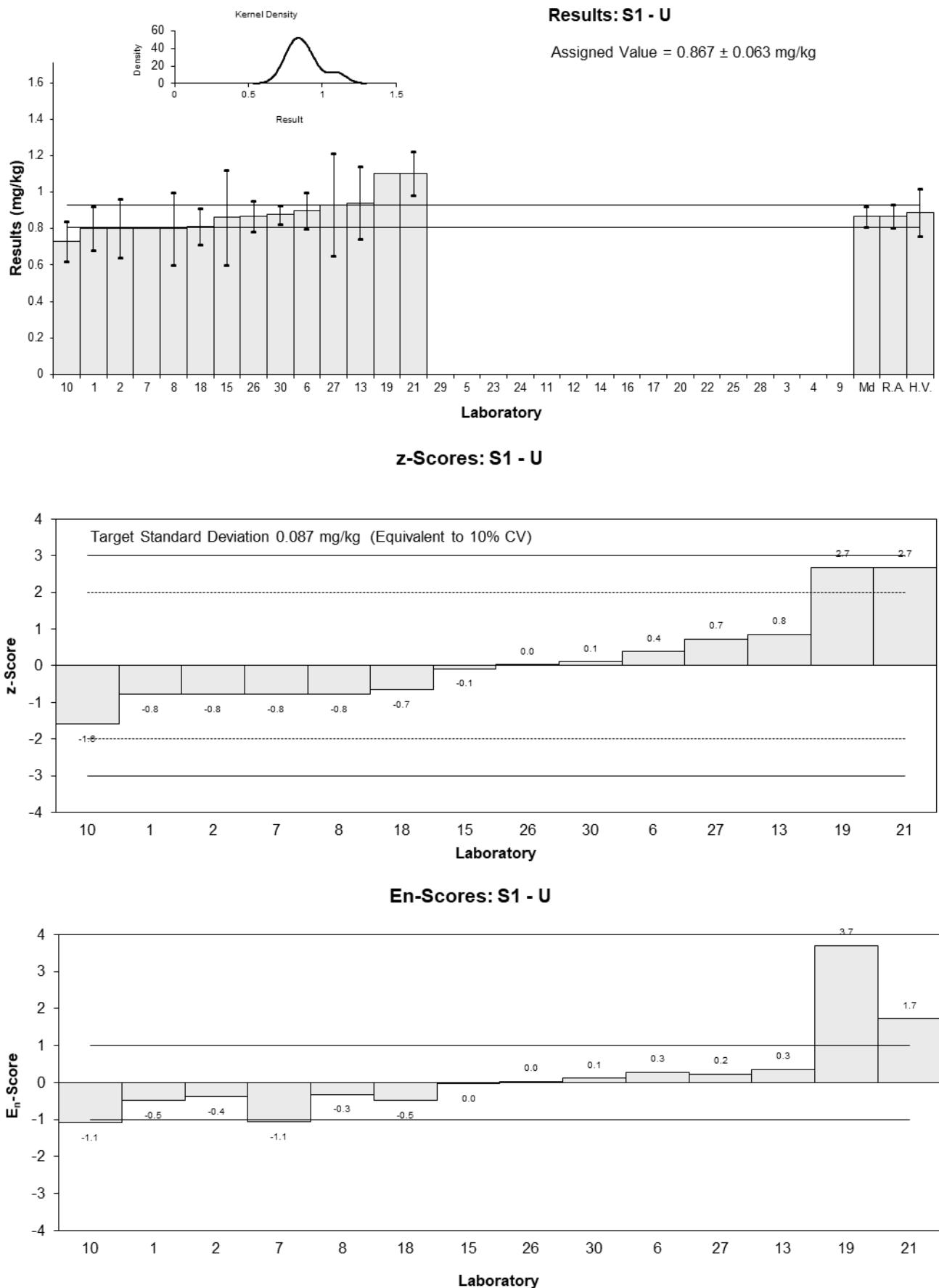


Figure 16

Table 29

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | V |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 34 | 5.1 | 0.30 | 0.18 |
| 2 | 30 | 3 | -0.91 | -0.82 |
| 3 | 25.85 | 3 | -2.17 | -1.95 |
| 4 | NT | NT | | |
| 5 | 35 | 5.3 | 0.61 | 0.35 |
| 6 | 32.8 | 3.5 | -0.06 | -0.05 |
| 7 | 34 | 5 | 0.30 | 0.18 |
| 8 | 30 | 6 | -0.91 | -0.47 |
| 9 | NT | NT | | |
| 10 | 29 | 7.8 | -1.21 | -0.50 |
| 11 | NT | NT | | |
| 12 | NT | NT | | |
| 13 | 38 | 10 | 1.52 | 0.49 |
| 14 | NT | NT | | |
| 15 | 36.0 | 4.8 | 0.91 | 0.57 |
| 16 | 31.1 | 3.89 | -0.58 | -0.43 |
| 17 | 40.5 | 12.1 | 2.27 | 0.61 |
| 18 | 32.2 | 4.4 | -0.24 | -0.16 |
| 19 | 33 | NR | 0.00 | 0.00 |
| 20 | NT | NT | | |
| 21 | NT | NT | | |
| 22 | NT | NT | | |
| 23 | 29.0 | 7.2 | -1.21 | -0.53 |
| 24 | 73.066 | NR | 12.14 | 19.08 |
| 25 | NT | NT | | |
| 26 | < 100 | 67 | | |
| 27 | 36.2 | 10.9 | 0.97 | 0.29 |
| 28 | NT | NT | | |
| 29 | 34 | 4.6 | 0.30 | 0.20 |
| 30 | 34.44 | 1.8 | 0.44 | 0.52 |

Statistics

| | | |
|--------------------------|------------|-----|
| Assigned Value* | 33.0 | 2.1 |
| Spike | Not Spiked | |
| Homogeneity Value | 34.0 | 5.1 |
| Robust Average | 33.4 | 2.2 |
| Median | 34.0 | 1.6 |
| Mean | 35.2 | |
| N | 19 | |
| Max. | 73.066 | |
| Min. | 25.85 | |
| Robust SD | 3.9 | |
| Robust CV | 12% | |

*Robust Average excluding laboratory 24.

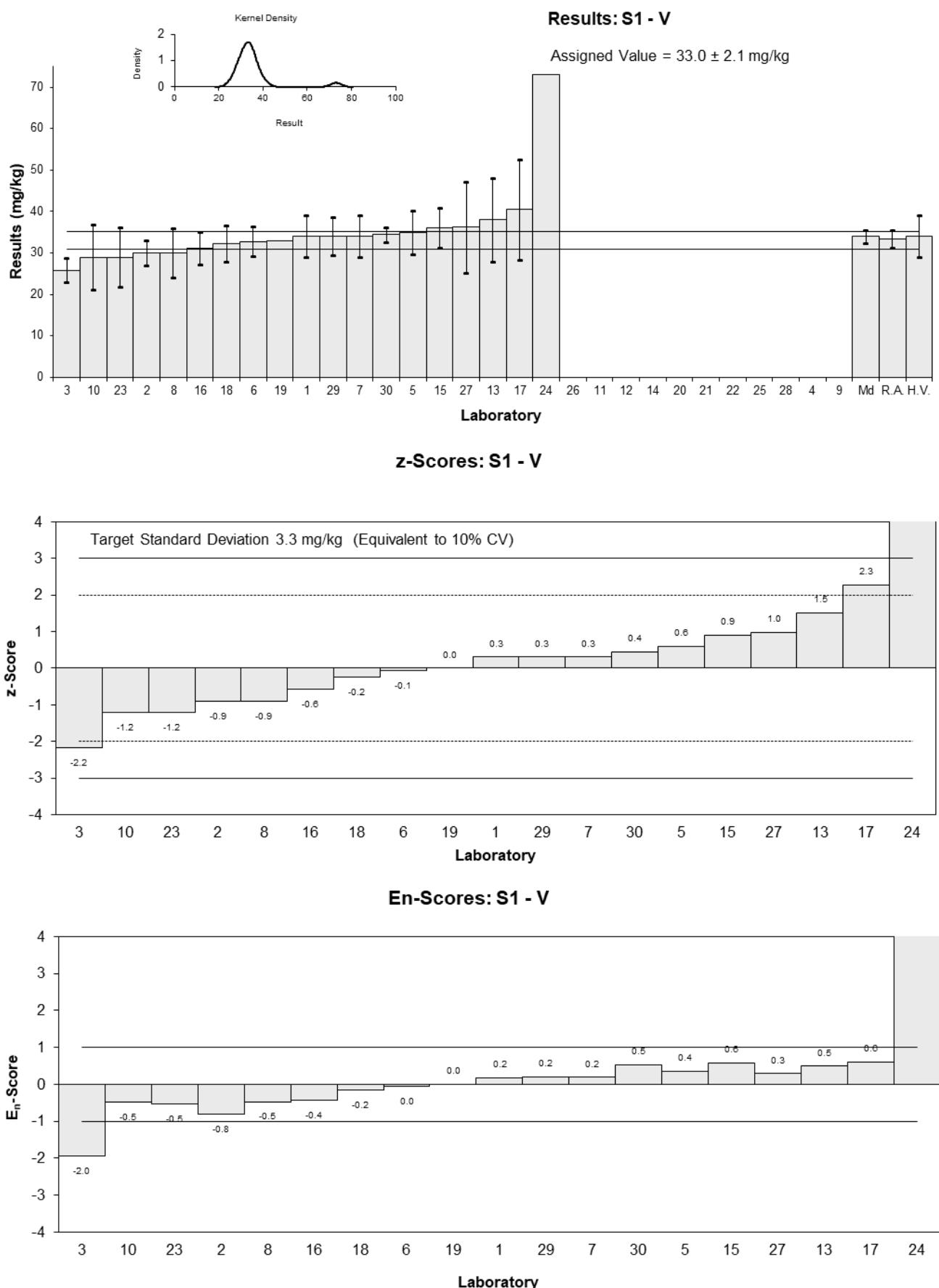


Figure 17

Table 30

Sample Details

| | |
|-------------------|----------|
| Sample No. | S1 |
| Matrix. | Sediment |
| Analyte. | Zn |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 64 | 9.6 | -1.35 | -0.98 |
| 2 | 69 | 7 | -0.68 | -0.64 |
| 3 | 22.6 | 3 | -6.95 | -11.15 |
| 4 | NT | NT | | |
| 5 | 72 | 14 | -0.27 | -0.14 |
| 6 | 73.7 | 8.0 | -0.04 | -0.03 |
| 7 | 72 | 12 | -0.27 | -0.16 |
| 8 | 68.1 | 11.5 | -0.80 | -0.49 |
| 9 | NT | NT | | |
| 10 | 69 | 10 | -0.68 | -0.47 |
| 11 | 82 | 4 | 1.08 | 1.51 |
| 12 | NT | NT | | |
| 13 | 78 | 10 | 0.54 | 0.38 |
| 14 | NT | NT | | |
| 15 | 74.5 | 8.8 | 0.07 | 0.05 |
| 16 | 67.9 | 8.15 | -0.82 | -0.69 |
| 17 | 85.5 | 17 | 1.55 | 0.66 |
| 18 | 74.2 | 11.3 | 0.03 | 0.02 |
| 19 | 82 | 8.2 | 1.08 | 0.90 |
| 20 | NT | NT | | |
| 21 | 80.7 | 11.6 | 0.91 | 0.55 |
| 22 | NT | NT | | |
| 23 | 75.9 | 19.0 | 0.26 | 0.10 |
| 24 | 66.482 | NR | -1.02 | -2.15 |
| 25 | NT | NT | | |
| 26 | 76.7 | 6.1 | 0.36 | 0.38 |
| 27 | 77.6 | 23.3 | 0.49 | 0.15 |
| 28 | NT | NT | | |
| 29 | 33 | 5.0 | -5.54 | -6.72 |
| 30 | 72.72 | 5.9 | -0.17 | -0.19 |

Statistics

| | | |
|--------------------------|------------|-----|
| Assigned Value* | 74.0 | 3.5 |
| Spike | Not Spiked | |
| Homogeneity Value | 66 | 10 |
| Robust Average | 72.9 | 3.9 |
| Median | 73.2 | 3.0 |
| Mean | 69.9 | |
| N | 22 | |
| Max. | 85.5 | |
| Min. | 22.6 | |
| Robust SD | 7.2 | |
| Robust CV | 9.9% | |

*Robust Average excluding laboratories 3 and 29.

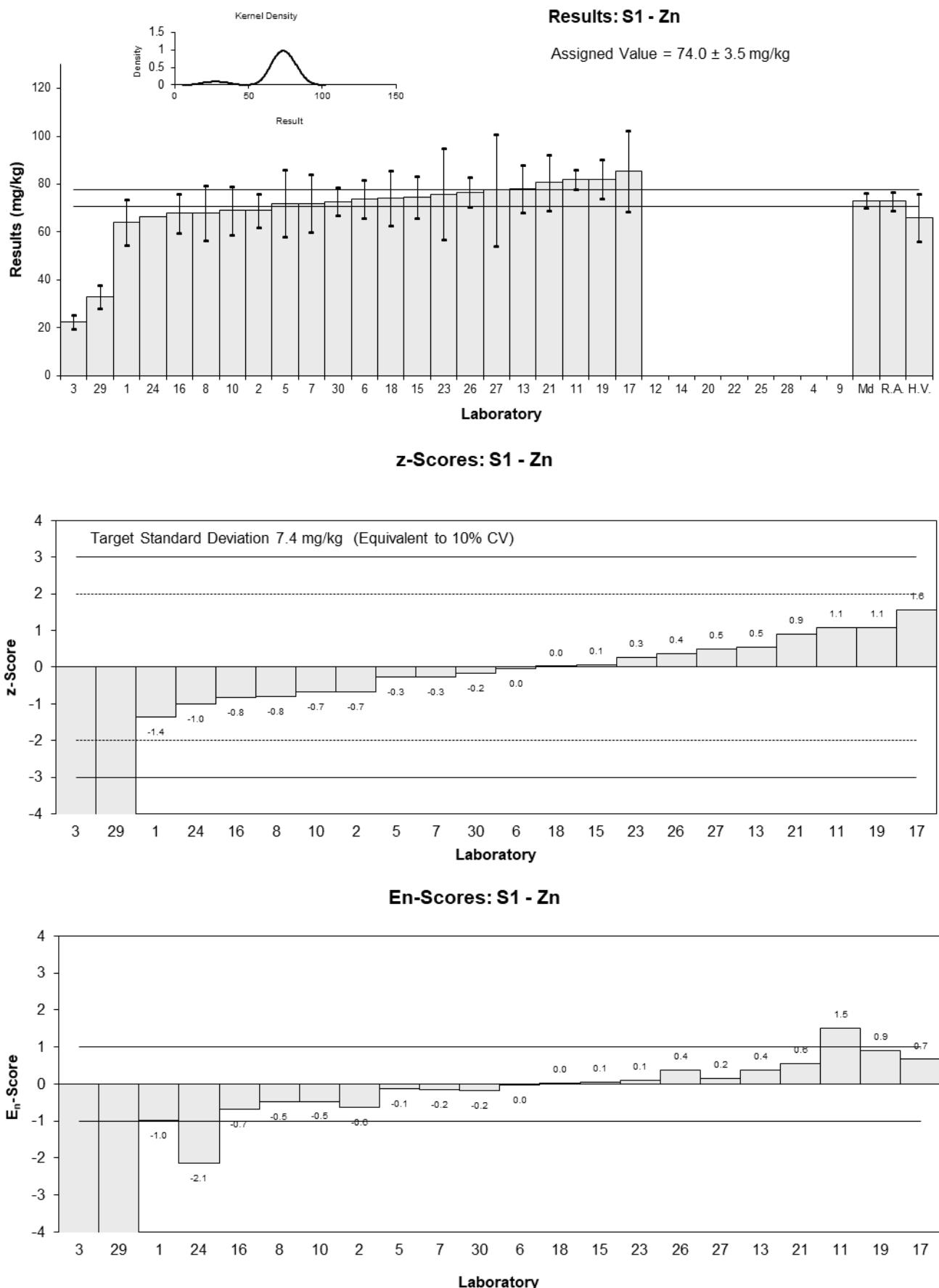


Figure 18

Table 31

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Ag |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 3 | 0.45 | 0.83 | 0.48 |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | 2.09 | 0.38 | -2.45 | -1.66 |
| 5 | 2.9 | 0.58 | 0.47 | 0.22 |
| 6 | 2.72 | 0.4 | -0.18 | -0.12 |
| 7 | NT | NT | | |
| 8 | 3.0 | 0.6 | 0.83 | 0.37 |
| 9 | NT | NT | | |
| 10 | 2.5 | 0.87 | -0.97 | -0.31 |
| 11 | NT | NT | | |
| 12 | NT | NT | | |
| 13 | 2.75 | 1.4 | -0.07 | -0.01 |
| 14 | NT | NT | | |
| 15 | 2.66 | 0.83 | -0.40 | -0.13 |
| 16 | 2.51 | 0.28 | -0.94 | -0.82 |
| 17 | <1 | NR | | |
| 18 | 2.68 | 0.28 | -0.32 | -0.28 |
| 19 | 59 | NR | 203.00 | 374.87 |
| 20 | NT | NT | | |
| 21 | 3.0 | 0.3 | 0.83 | 0.69 |
| 22 | 2.8 | 0.56 | 0.11 | 0.05 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 2.2 | 0.59 | -2.06 | -0.94 |
| 26 | 2.96 | 0.42 | 0.69 | 0.43 |
| 27 | 45.5 | 13.6 | 154.26 | 3.14 |
| 28 | 2.9 | 0.58 | 0.47 | 0.22 |
| 29 | 3.0 | 0.41 | 0.83 | 0.53 |
| 30 | 2.92 | 0.4 | 0.54 | 0.35 |

Statistics*

| | | |
|--------------------------|------|------|
| Assigned Value | 2.77 | 0.15 |
| Spike | 2.84 | 0.06 |
| Homogeneity Value | 2.90 | 0.35 |
| Robust Average | 2.77 | 0.15 |
| Median | 2.80 | 0.12 |
| Mean | 2.74 | |
| N | 17 | |
| Max. | 3 | |
| Min. | 2.09 | |
| Robust SD | 0.24 | |
| Robust CV | 8.7% | |

*Laboratories 19 and 27 were excluded from statistical calculation (extreme outlier).

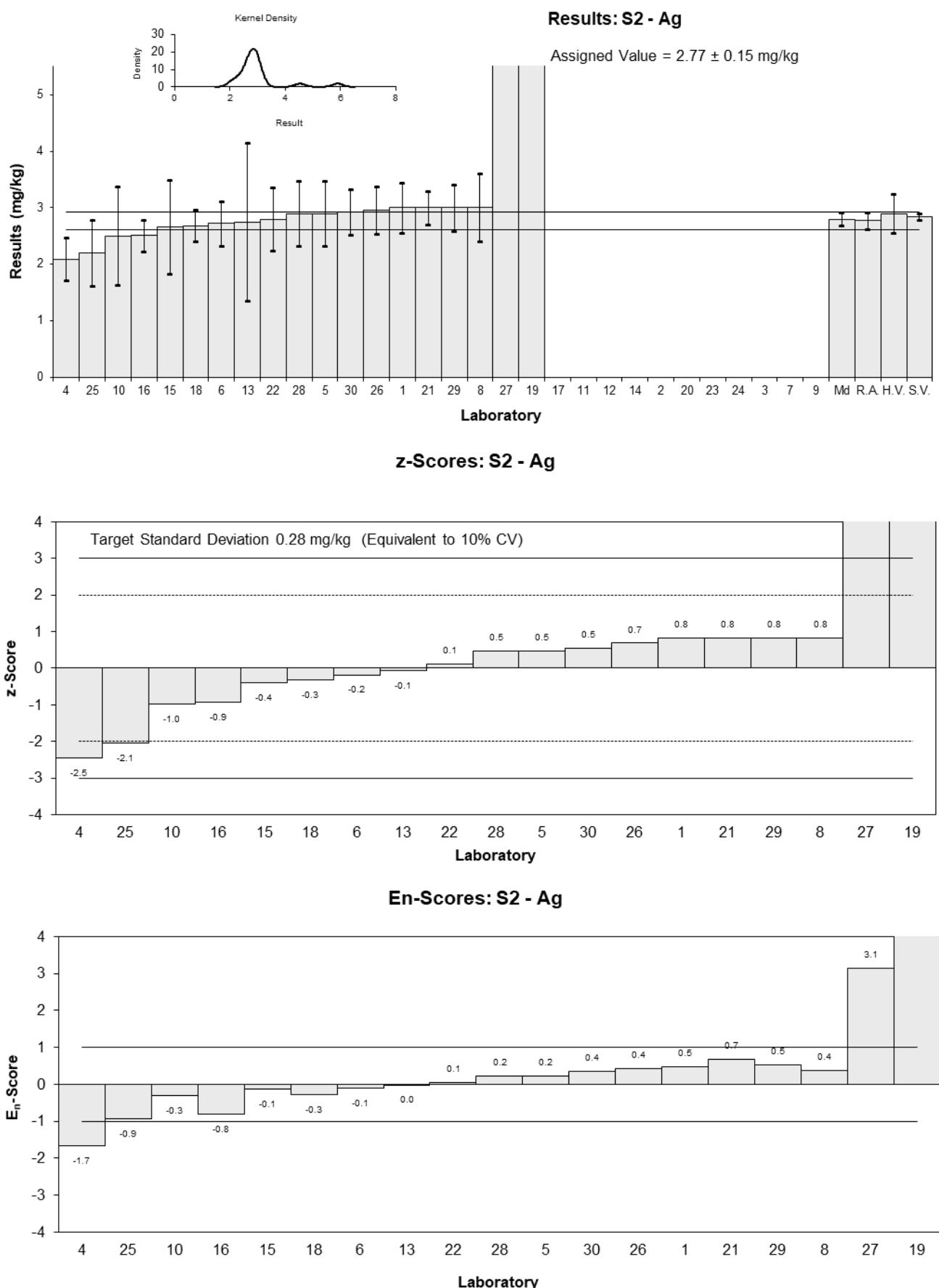


Figure 19

Table 32

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | B |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty |
|-----------------|---------------|--------------------|
| 1 | 28 | 4.2 |
| 2 | NT | NT |
| 3 | 8.6 | 1 |
| 4 | <50 | NR |
| 5 | 37 | 7.4 |
| 6 | 33.1 | 3.5 |
| 7 | NT | NT |
| 8 | <50 | NR |
| 9 | NT | NT |
| 10 | 19 | 4.8 |
| 11 | NT | NT |
| 12 | NT | NT |
| 13 | 32 | 3 |
| 14 | NT | NT |
| 15 | 11.9 | 3.5 |
| 16 | 50.4 | 7.41 |
| 17 | 32.0 | 8 |
| 18 | 20.5 | 3 |
| 19 | 17 | NR |
| 20 | NT | NT |
| 21 | 10.7 | 1.8 |
| 22 | 33 | 6.6 |
| 23 | NT | NT |
| 24 | NT | NT |
| 25 | 5 | 1.3 |
| 26 | 25.7 | 3.9 |
| 27 | 24.7 | 7.4 |
| 28 | 36 | 7.2 |
| 29 | 30 | 5.8 |
| 30 | 27.97 | 3.7 |

Statistics

| | | |
|--------------------------|---------|-----|
| Assigned Value | Not Set | |
| Spike | 38.9 | 0.9 |
| Homogeneity Value | 27.0 | 3.2 |
| Robust Average | 25.1 | 6.7 |
| Median | 28.0 | 5.3 |
| Mean | 25.4 | |
| N | 19 | |
| Max. | 50.4 | |
| Min. | 5 | |
| Robust SD | 12 | |
| Robust CV | 47% | |

Results: S2 - B

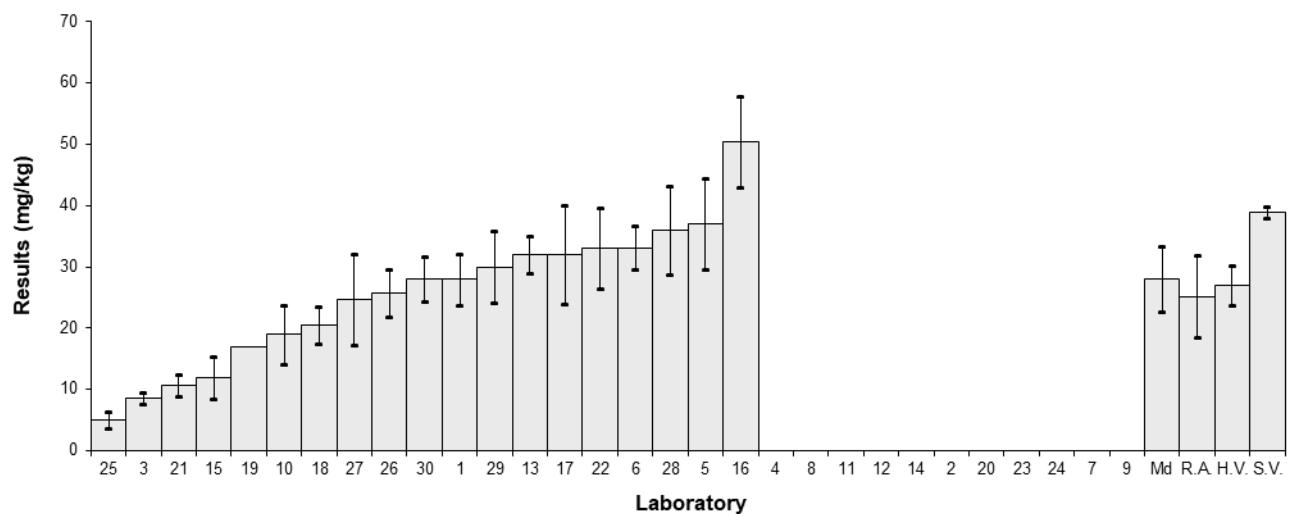


Figure 20

Table 33

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Ba |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 210 | 31.5 | -1.53 | -1.12 |
| 2 | NT | NT | | |
| 3 | 253.1 | 25 | 0.21 | 0.18 |
| 4 | 219 | 24.3 | -1.17 | -1.05 |
| 5 | 250 | 50 | 0.08 | 0.04 |
| 6 | 259 | 26 | 0.44 | 0.38 |
| 7 | NT | NT | | |
| 8 | 213 | 36 | -1.41 | -0.91 |
| 9 | NT | NT | | |
| 10 | 240 | 57 | -0.32 | -0.14 |
| 11 | NT | NT | | |
| 12 | NT | NT | | |
| 13 | 260 | 20 | 0.48 | 0.50 |
| 14 | NT | NT | | |
| 15 | 263 | 28 | 0.60 | 0.49 |
| 16 | 266 | 22.9 | 0.73 | 0.68 |
| 17 | 280 | 70 | 1.29 | 0.45 |
| 18 | 234 | 32 | -0.56 | -0.41 |
| 19 | 240 | NR | -0.32 | -0.62 |
| 20 | NT | NT | | |
| 21 | 239 | 28 | -0.36 | -0.29 |
| 22 | 270 | 54 | 0.89 | 0.40 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 206 | 55.0 | -1.69 | -0.74 |
| 26 | 238 | 39 | -0.40 | -0.24 |
| 27 | 258.3 | 77.5 | 0.42 | 0.13 |
| 28 | 280 | 56 | 1.29 | 0.56 |
| 29 | 260 | 41 | 0.48 | 0.28 |
| 30 | 251.6 | 11 | 0.15 | 0.21 |

Statistics

| | | |
|--------------------------|------|----|
| Assigned Value | 248 | 13 |
| Spike | 235 | 14 |
| Homogeneity Value | 222 | 27 |
| Robust Average | 248 | 13 |
| Median | 252 | 9 |
| Mean | 247 | |
| N | 21 | |
| Max. | 280 | |
| Min. | 206 | |
| Robust SD | 24 | |
| Robust CV | 9.6% | |

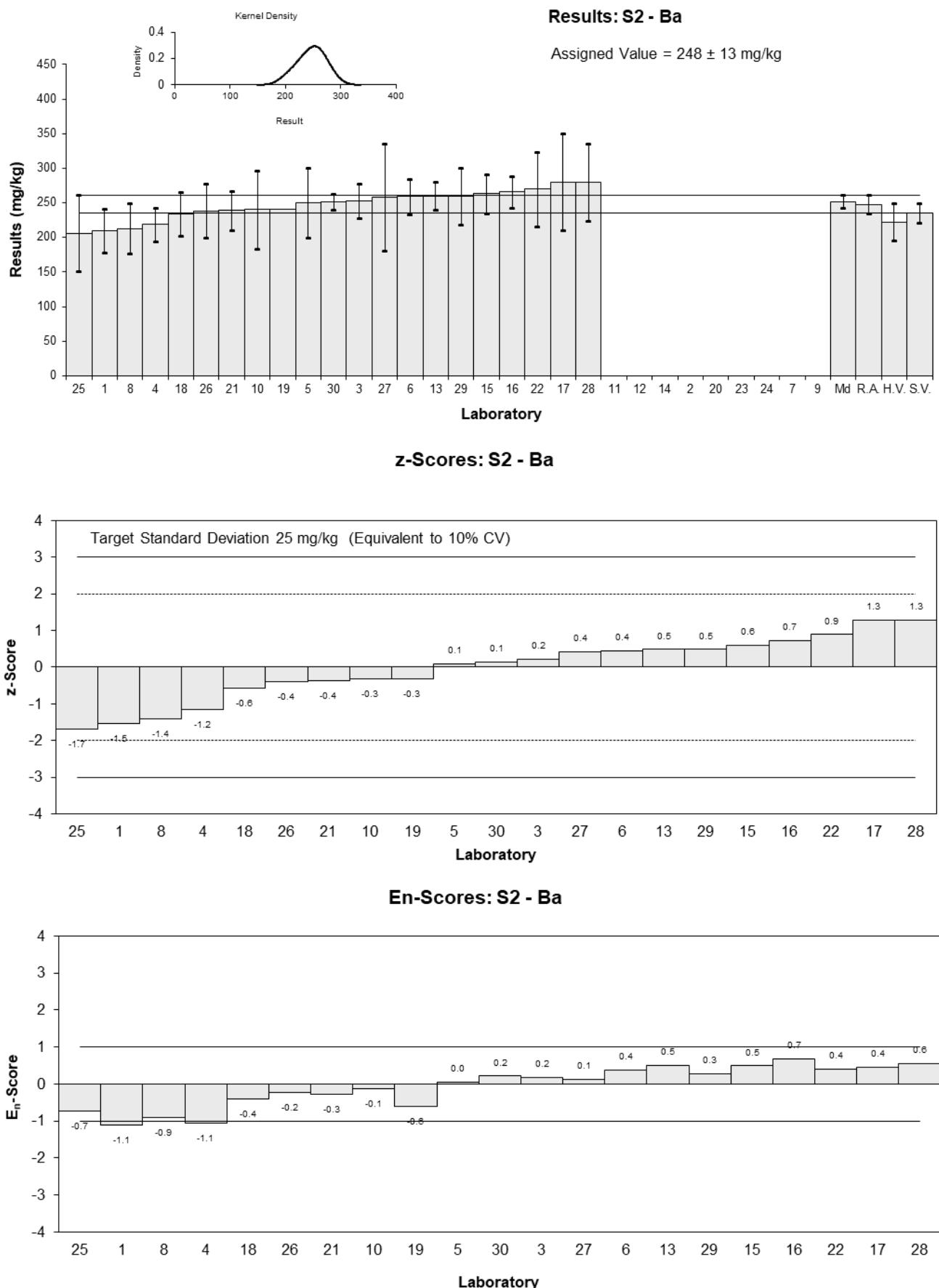


Figure 21

Table 34

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Be |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 1.6 | 0.24 | -0.36 | -0.29 |
| 2 | NT | NT | | |
| 3 | 1.36 | 0.5 | -1.30 | -0.62 |
| 4 | 1.74 | 0.35 | 0.20 | 0.13 |
| 5 | 2.2 | 0.55 | 2.01 | 0.88 |
| 6 | 1.81 | 0.2 | 0.47 | 0.43 |
| 7 | NT | NT | | |
| 8 | 1.2 | 0.3 | -1.93 | -1.38 |
| 9 | NT | NT | | |
| 10 | 1.5 | 0.51 | -0.75 | -0.35 |
| 11 | NT | NT | | |
| 12 | NT | NT | | |
| 13 | 1.80 | 1 | 0.43 | 0.11 |
| 14 | NT | NT | | |
| 15 | 1.86 | 0.42 | 0.67 | 0.37 |
| 16 | 1.25 | 0.13 | -1.74 | -1.91 |
| 17 | <1 | NR | | |
| 18 | 1.63 | 0.26 | -0.24 | -0.19 |
| 19 | 1.82 | NR | 0.51 | 0.68 |
| 20 | NT | NT | | |
| 21 | NT | NT | | |
| 22 | 1.8 | 0.36 | 0.43 | 0.27 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 0.9 | 0.24 | -3.12 | -2.58 |
| 26 | 1.85 | 0.34 | 0.63 | 0.41 |
| 27 | 1.9 | 0.58 | 0.83 | 0.34 |
| 28 | 2.1 | 0.42 | 1.62 | 0.89 |
| 29 | <2 | NR | | |
| 30 | 1.80 | 0.1 | 0.43 | 0.51 |

Statistics

| | | |
|--------------------------|------|------|
| Assigned Value | 1.69 | 0.19 |
| Spike | 1.79 | 0.11 |
| Homogeneity Value | 1.56 | 0.19 |
| Robust Average | 1.69 | 0.19 |
| Median | 1.80 | 0.10 |
| Mean | 1.67 | |
| N | 18 | |
| Max. | 2.2 | |
| Min. | 0.9 | |
| Robust SD | 0.33 | |
| Robust CV | 19% | |

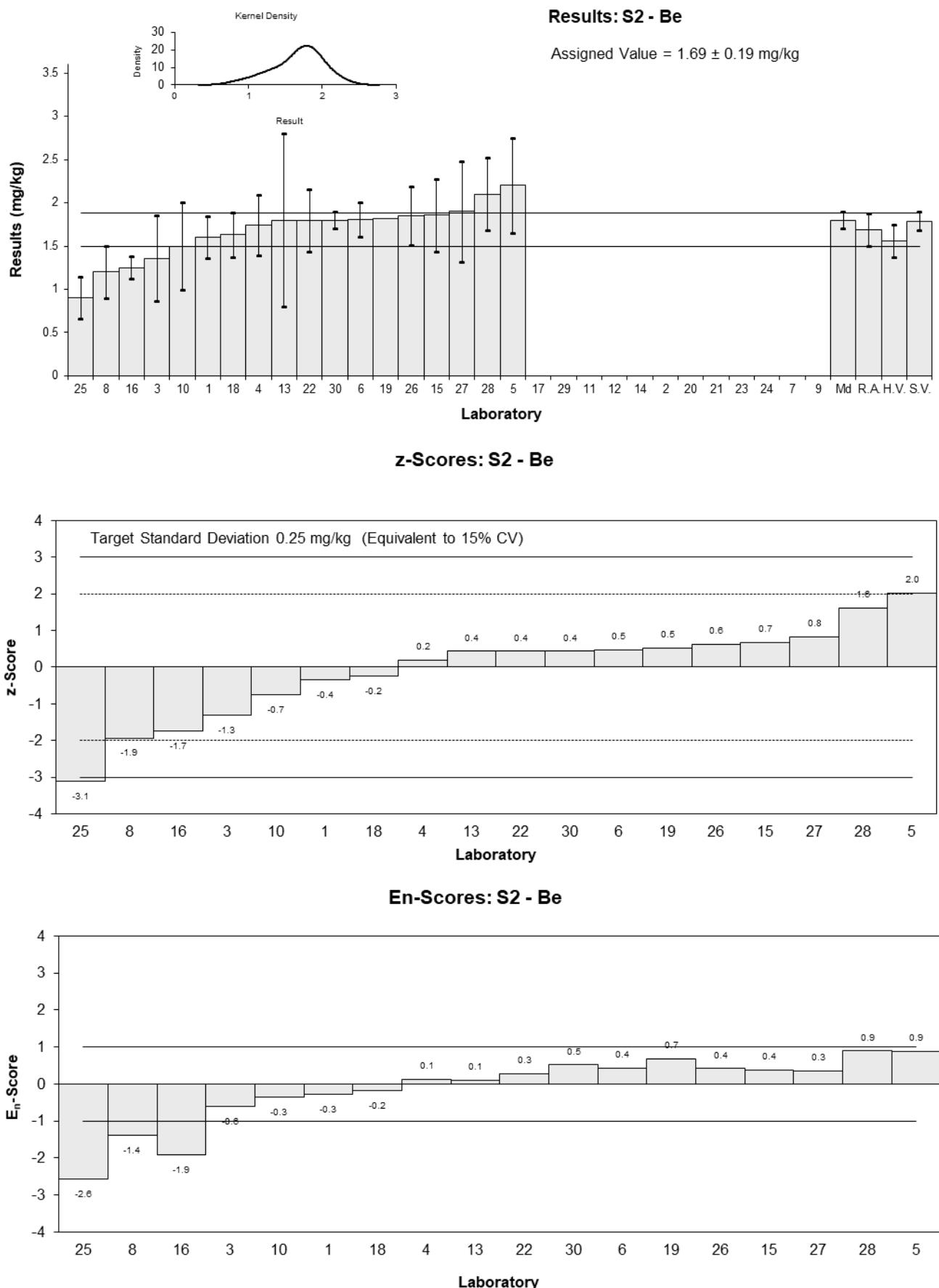


Figure 22

Table 35

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Bi |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E _n -Score |
|----------|--------|-------------|---------|-----------------------|
| 1 | NT | NT | | |
| 2 | NT | NT | | |
| 3 | 2.025 | 0.5 | 11.89 | 2.19 |
| 4 | 0.956 | 0.085 | 0.34 | 0.30 |
| 5 | <10 | NR | | |
| 6 | NT | NT | | |
| 7 | NT | NT | | |
| 8 | 0.8 | 0.2 | -1.35 | -0.60 |
| 9 | NT | NT | | |
| 10 | 0.98 | 0.30 | 0.59 | 0.18 |
| 11 | NT | NT | | |
| 12 | NT | NT | | |
| 13 | NT | NT | | |
| 14 | NT | NT | | |
| 15 | 0.86 | 0.28 | -0.70 | -0.23 |
| 16 | NT | NT | | |
| 17 | NT | NT | | |
| 18 | 0.884 | 0.11 | -0.44 | -0.33 |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | NT | NT | | |
| 22 | 1.0 | 0.20 | 0.81 | 0.36 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NT | NT | | |
| 26 | 0.90 | 0.13 | -0.27 | -0.18 |
| 27 | 0.96 | 0.29 | 0.38 | 0.12 |
| 28 | <10 | NR | | |
| 29 | <10 | NR | | |
| 30 | 0.962 | 0.09 | 0.40 | 0.35 |

Statistics

| | | |
|--------------------------|-------|-------|
| Assigned Value* | 0.925 | 0.057 |
| Spike | 0.872 | 0.022 |
| Homogeneity Value | 0.95 | 0.11 |
| Robust Average | 0.938 | 0.066 |
| Median | 0.958 | 0.053 |
| Mean | 1.03 | |
| N | 10 | |
| Max. | 2.025 | |
| Min. | 0.8 | |
| Robust SD | 0.083 | |
| Robust CV | 8.9% | |

*Robust Average excluding laboratory 3.

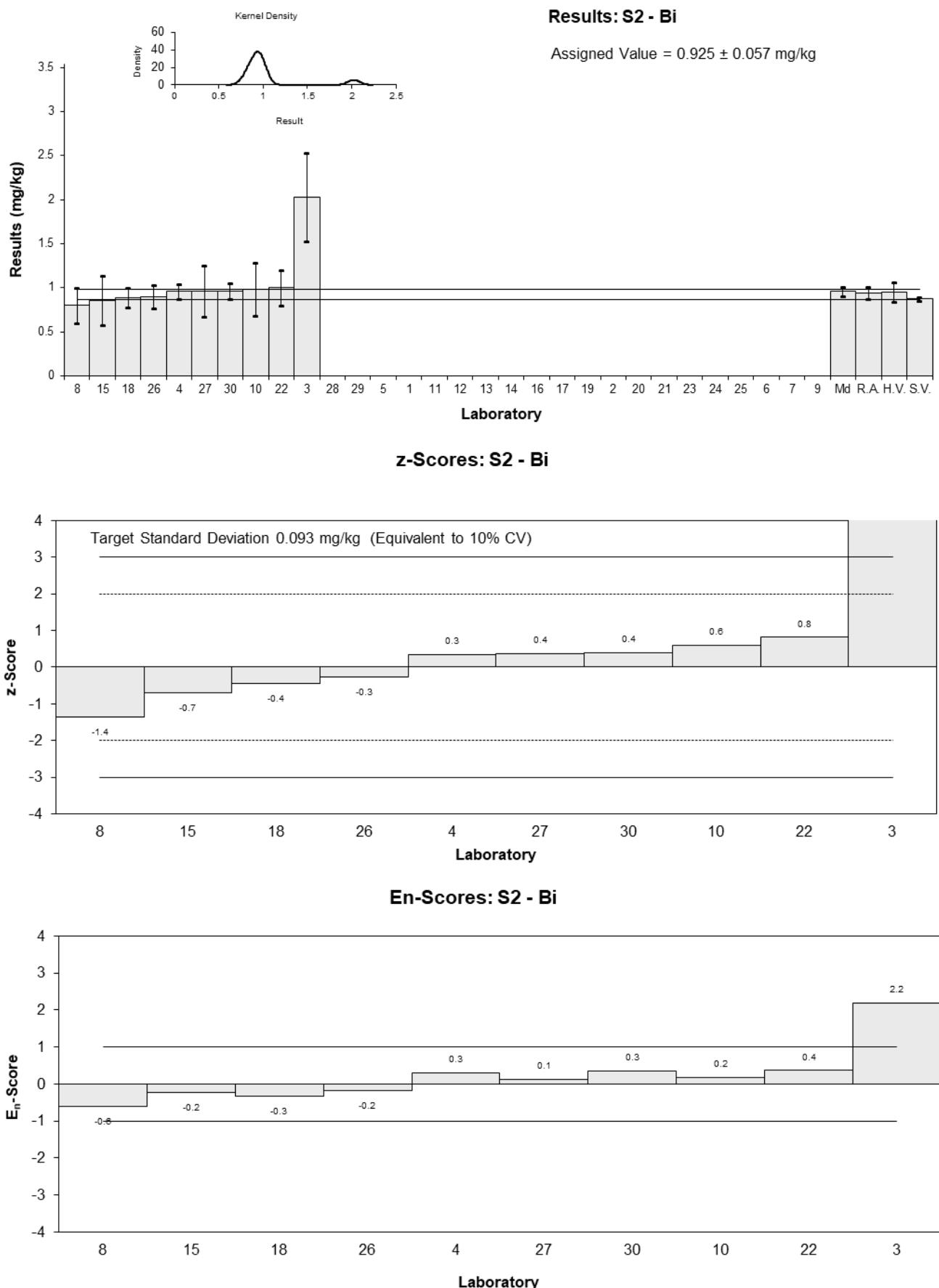


Figure 23

Table 36

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Cd |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 0.49 | 0.074 | -0.98 | -1.04 |
| 2 | NT | NT | | |
| 3 | 0.56 | 0.2 | -0.16 | -0.07 |
| 4 | <1.0 | NR | | |
| 5 | 0.56 | 0.084 | -0.16 | -0.16 |
| 6 | 0.62 | 0.1 | 0.53 | 0.44 |
| 7 | NT | NT | | |
| 8 | 0.5 | 0.1 | -0.86 | -0.70 |
| 9 | NT | NT | | |
| 10 | 0.57 | 0.11 | -0.05 | -0.03 |
| 11 | 0.6 | 0.1 | 0.30 | 0.25 |
| 12 | NT | NT | | |
| 13 | 0.65 | 0.4 | 0.88 | 0.19 |
| 14 | NT | NT | | |
| 15 | 0.544 | 0.16 | -0.35 | -0.18 |
| 16 | NT | NT | | |
| 17 | <1 | NR | | |
| 18 | 0.612 | 0.07 | 0.44 | 0.49 |
| 19 | 0.67 | 0.03 | 1.11 | 2.15 |
| 20 | NT | NT | | |
| 21 | 0.50 | 0.04 | -0.86 | -1.43 |
| 22 | 0.59 | 0.11 | 0.19 | 0.14 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 0.4 | 0.09 | -2.02 | -1.82 |
| 26 | 0.596 | 0.072 | 0.26 | 0.28 |
| 27 | 0.6 | 0.2 | 0.30 | 0.13 |
| 28 | 0.58 | 0.116 | 0.07 | 0.05 |
| 29 | 0.60 | 0.088 | 0.30 | 0.28 |
| 30 | 0.587 | 0.08 | 0.15 | 0.15 |

Statistics

| | | |
|--------------------------|-------|-------|
| Assigned Value | 0.574 | 0.033 |
| Spike | 0.608 | 0.016 |
| Homogeneity Value | 0.583 | 0.070 |
| Robust Average | 0.574 | 0.033 |
| Median | 0.587 | 0.019 |
| Mean | 0.570 | |
| N | 19 | |
| Max. | 0.67 | |
| Min. | 0.4 | |
| Robust SD | 0.058 | |
| Robust CV | 10% | |

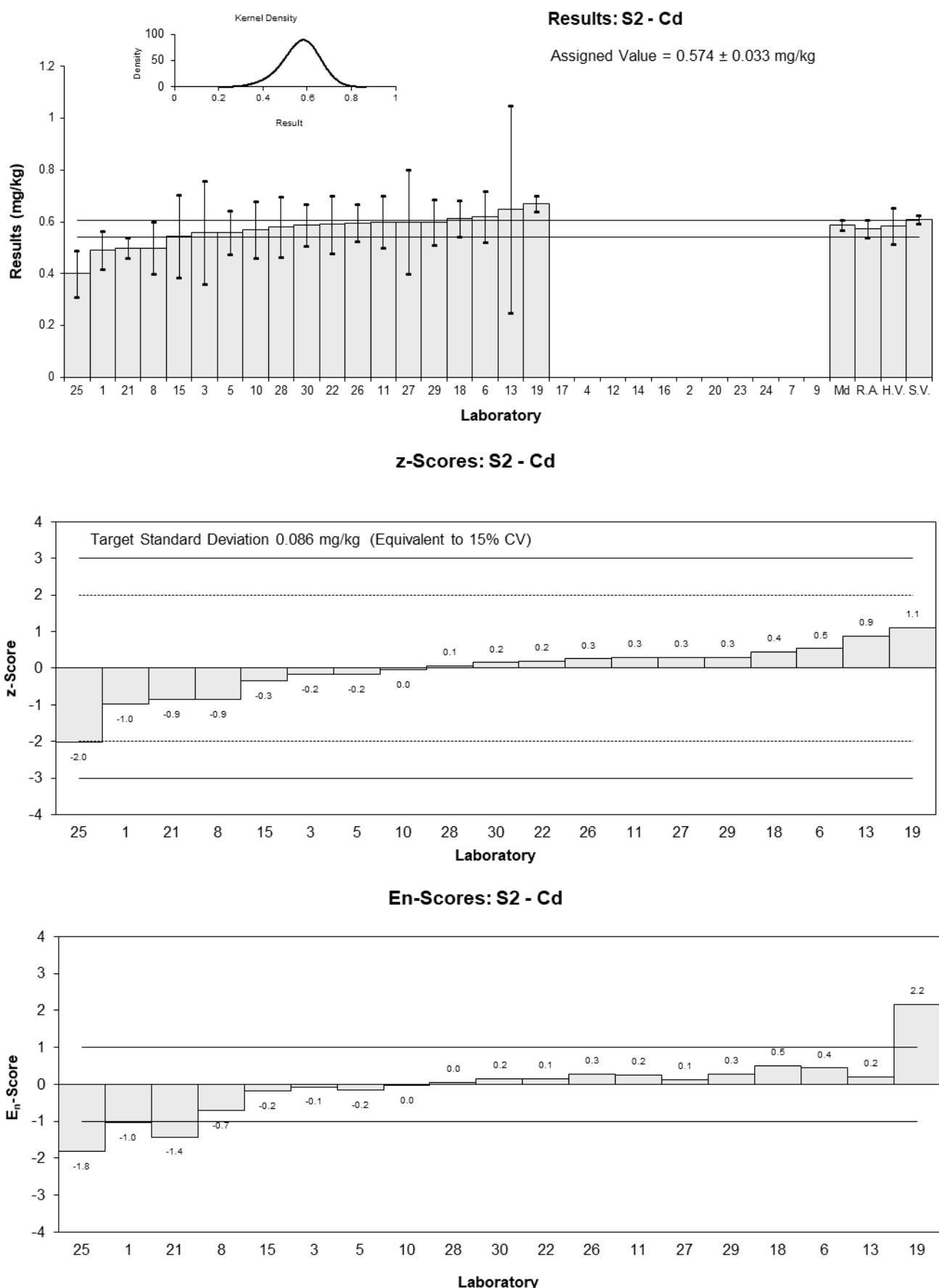


Figure 24

Table 37

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Cs |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty |
|-----------------|---------------|--------------------|
| 1 | NT | NT |
| 2 | NT | NT |
| 3 | NT | NT |
| 4 | 2.5 | 0.23 |
| 5 | NT | NT |
| 6 | NT | NT |
| 7 | NT | NT |
| 8 | 1.3 | 0.3 |
| 9 | NT | NT |
| 10 | 1.6 | 0.25 |
| 11 | NT | NT |
| 12 | NT | NT |
| 13 | NT | NT |
| 14 | NT | NT |
| 15 | NR | NR |
| 16 | NT | NT |
| 17 | NT | NT |
| 18 | NT | NT |
| 19 | NT | NT |
| 20 | NT | NT |
| 21 | NT | NT |
| 22 | 3.6 | 0.72 |
| 23 | NT | NT |
| 24 | NT | NT |
| 25 | NT | NT |
| 26 | 2.42 | 0.30 |
| 27 | 3.1 | 0.9 |
| 28 | NT | NT |
| 29 | NT | NT |
| 30 | NT | NT |

Statistics

| | | |
|--------------------------|---------|------|
| Assigned Value | Not Set | |
| Spike | 3.58 | 0.16 |
| Homogeneity Value | 2.88 | 0.35 |
| Robust Average | 2.4 | 1.0 |
| Median | 2.5 | 1.2 |
| Mean | 2.4 | |
| N | 6 | |
| Max. | 3.6 | |
| Min. | 1.3 | |
| Robust SD | 0.99 | |
| Robust CV | 41% | |

Results: S2 - Cs

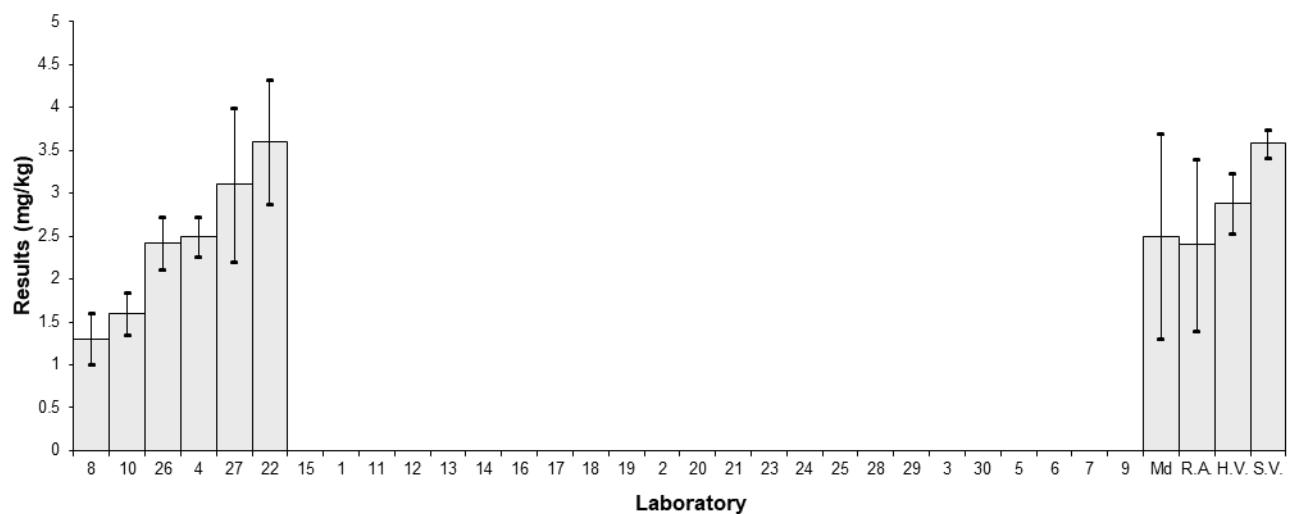


Figure 25

Table 38

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Ga |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty |
|-----------------|---------------|--------------------|
| 1 | NT | NT |
| 2 | NT | NT |
| 3 | NT | NT |
| 4 | 10.8 | 1.12 |
| 5 | NT | NT |
| 6 | NT | NT |
| 7 | NT | NT |
| 8 | 6.7 | 1.1 |
| 9 | NT | NT |
| 10 | NT | NT |
| 11 | NT | NT |
| 12 | NT | NT |
| 13 | NT | NT |
| 14 | NT | NT |
| 15 | NR | NR |
| 16 | NT | NT |
| 17 | NT | NT |
| 18 | NT | NT |
| 19 | NT | NT |
| 20 | NT | NT |
| 21 | NT | NT |
| 22 | 13 | 2.6 |
| 23 | NT | NT |
| 24 | NT | NT |
| 25 | NT | NT |
| 26 | NT | NT |
| 27 | NT | NT |
| 28 | NT | NT |
| 29 | NT | NT |
| 30 | NT | NT |

Statistics*

| | | |
|--------------------------|---------|-----|
| Assigned Value | Not Set | |
| Spike | 13.1 | 1.0 |
| Homogeneity Value | 11.2 | 1.3 |
| Mean | 10.2 | |
| N | 3 | |
| Max. | 13 | |
| Min. | 6.7 | |

*Insufficient data to calculate statistics.

Results: S2 - Ga

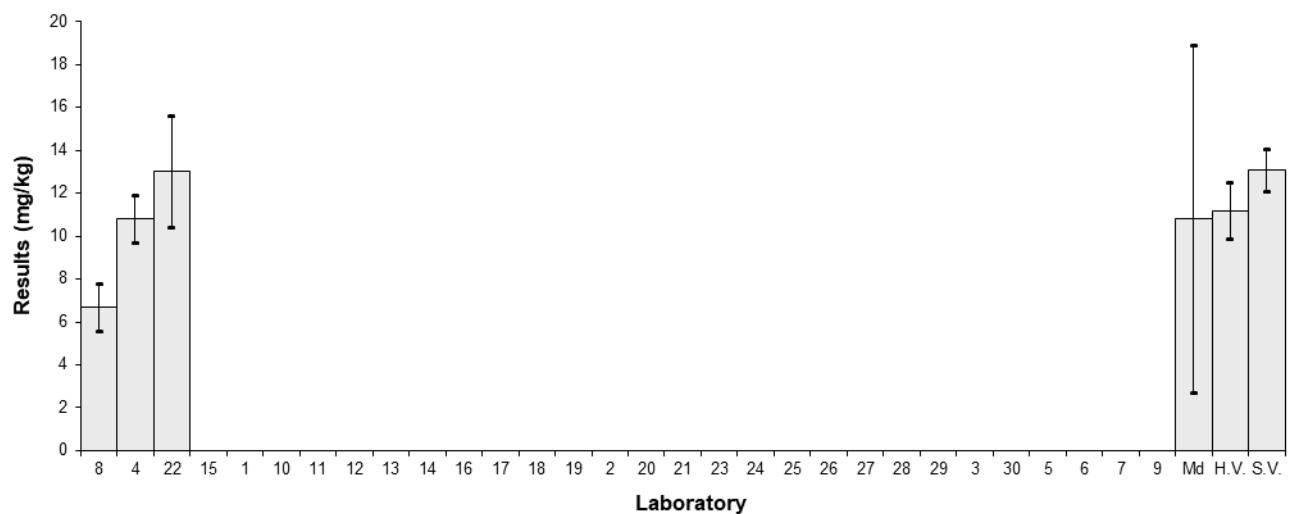


Figure 26

Table 39

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Hg |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 0.51 | 0.077 | 0.83 | 0.48 |
| 2 | NT | NT | | |
| 3 | 0.38 | 0.2 | -1.93 | -0.45 |
| 4 | 0.524 | 0.09 | 1.13 | 0.56 |
| 5 | 0.54 | 0.081 | 1.46 | 0.81 |
| 6 | 0.49 | 0.08 | 0.40 | 0.22 |
| 7 | NT | NT | | |
| 8 | 0.4 | 0.1 | -1.51 | -0.68 |
| 9 | NT | NT | | |
| 10 | 0.44 | 0.075 | -0.66 | -0.39 |
| 11 | 0.5 | 0.1 | 0.62 | 0.28 |
| 12 | NT | NT | | |
| 13 | 0.447 | 0.1 | -0.51 | -0.23 |
| 14 | NT | NT | | |
| 15 | 0.48 | 0.05 | 0.19 | 0.16 |
| 16 | 0.44 | NR | -0.66 | -1.11 |
| 17 | 0.45 | 0.13 | -0.45 | -0.16 |
| 18 | 0.502 | 0.06 | 0.66 | 0.47 |
| 19 | 0.38 | 0.02 | -1.93 | -2.64 |
| 20 | 0.424 | 0.053 | -1.00 | -0.78 |
| 21 | 0.49 | 0.09 | 0.40 | 0.20 |
| 22 | 0.63 | 0.13 | 3.38 | 1.20 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NT | NT | | |
| 26 | 0.444 | 0.054 | -0.57 | -0.44 |
| 27 | 0.51 | 0.2 | 0.83 | 0.19 |
| 28 | 0.46 | 0.092 | -0.23 | -0.11 |
| 29 | 0.52 | 0.084 | 1.04 | 0.55 |
| 30 | 0.460 | 0.04 | -0.23 | -0.23 |

Statistics

| | | |
|--------------------------|-------|-------|
| Assigned Value | 0.471 | 0.028 |
| Spike | 0.541 | 0.051 |
| Homogeneity Value | 0.482 | 0.058 |
| Robust Average | 0.471 | 0.028 |
| Median | 0.470 | 0.020 |
| Mean | 0.474 | |
| N | 22 | |
| Max. | 0.63 | |
| Min. | 0.38 | |
| Robust SD | 0.053 | |
| Robust CV | 11% | |

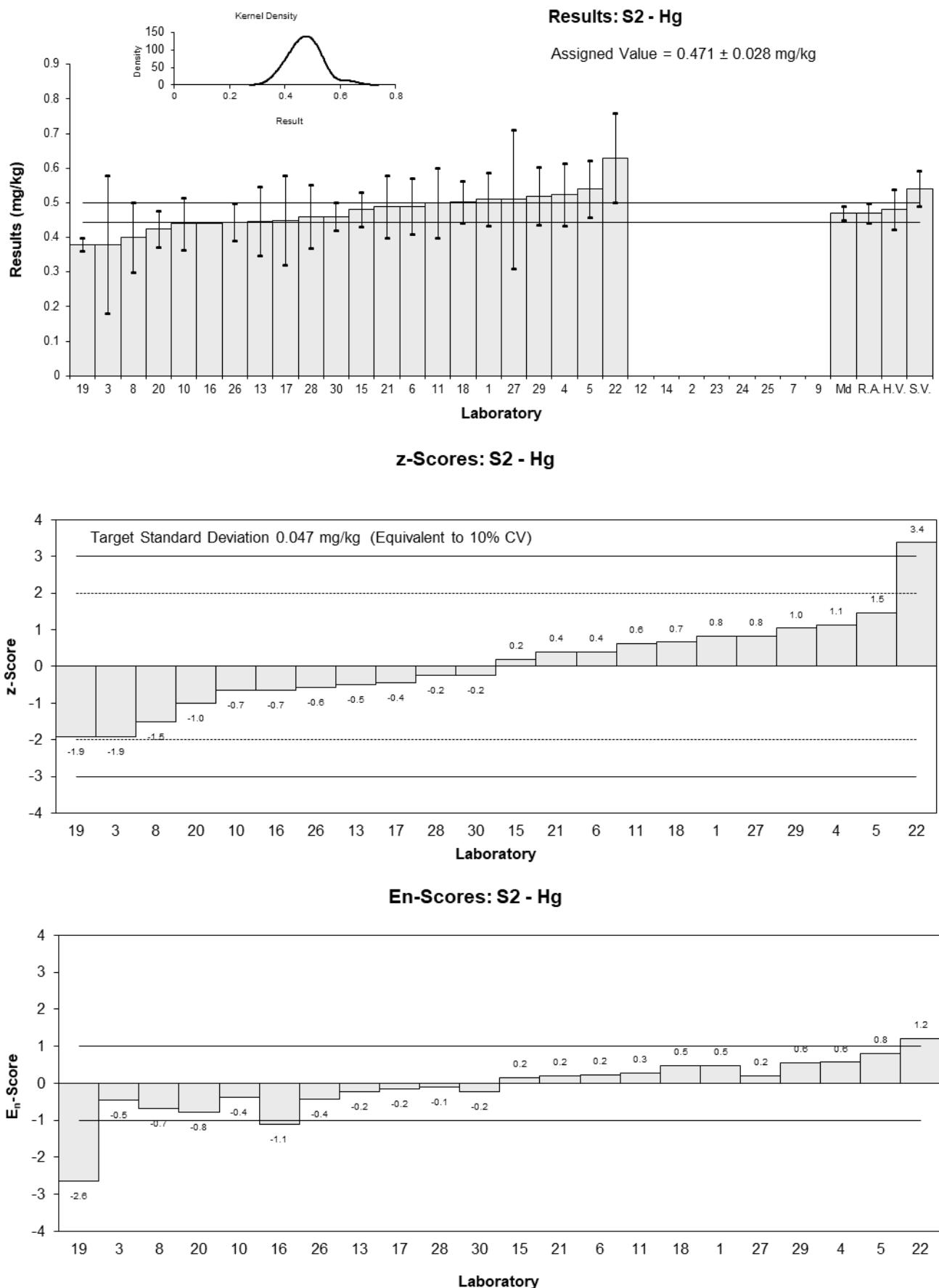


Figure 27

Table 40

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | La |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | NT | NT | | |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | 26.2 | 3.42 | 0.40 | 0.29 |
| 5 | NT | NT | | |
| 6 | NT | NT | | |
| 7 | NT | NT | | |
| 8 | 18.7 | 2.8 | -1.62 | -1.23 |
| 9 | NT | NT | | |
| 10 | 23 | 3.4 | -0.46 | -0.32 |
| 11 | NT | NT | | |
| 12 | NT | NT | | |
| 13 | NT | NT | | |
| 14 | NT | NT | | |
| 15 | 27.8 | 8.3 | 0.84 | 0.34 |
| 16 | NT | NT | | |
| 17 | NT | NT | | |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | NT | NT | | |
| 22 | 28 | 5.6 | 0.89 | 0.48 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NT | NT | | |
| 26 | 24.5 | 3.5 | -0.05 | -0.04 |
| 27 | NT | NT | | |
| 28 | NT | NT | | |
| 29 | NT | NT | | |
| 30 | NT | NT | | |

Statistics

| | | |
|--------------------------|------|-----|
| Assigned Value | 24.7 | 4.0 |
| Spike | 29.3 | 2.9 |
| Homogeneity Value | 26.4 | 3.2 |
| Robust Average | 24.7 | 4.0 |
| Median | 25.4 | 3.7 |
| Mean | 24.7 | |
| N | 6 | |
| Max. | 28 | |
| Min. | 18.7 | |
| Robust SD | 3.9 | |
| Robust CV | 16% | |

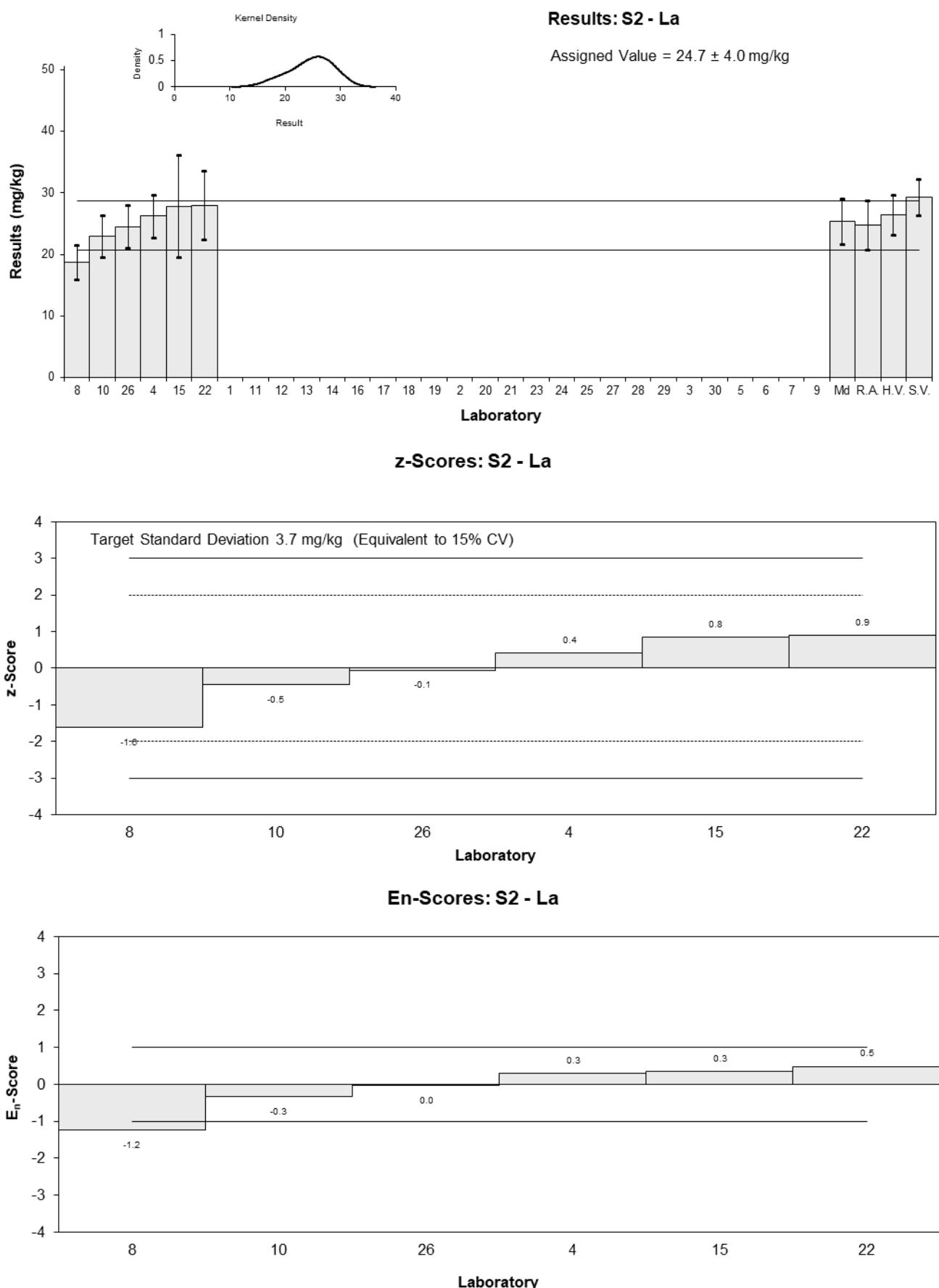


Figure 28

Table 41

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Li |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 8.6 | 1.29 | -1.15 | -0.95 |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | 8.33 | 0.89 | -1.33 | -1.25 |
| 5 | 13 | 3.3 | 1.67 | 0.73 |
| 6 | 9.96 | 1.2 | -0.28 | -0.24 |
| 7 | NT | NT | | |
| 8 | 3.4 | 0.7 | -4.49 | -4.47 |
| 9 | NT | NT | | |
| 10 | 5.6 | 1.1 | -3.08 | -2.70 |
| 11 | NT | NT | | |
| 12 | NT | NT | | |
| 13 | 9.1 | 2.3 | -0.83 | -0.48 |
| 14 | NT | NT | | |
| 15 | 11.9 | 1.4 | 0.96 | 0.76 |
| 16 | NT | NT | | |
| 17 | 12.0 | 5 | 1.03 | 0.31 |
| 18 | 10.0 | 1.5 | -0.26 | -0.19 |
| 19 | 7.7 | NR | -1.73 | -1.93 |
| 20 | NT | NT | | |
| 21 | 10.7 | 1.1 | 0.19 | 0.17 |
| 22 | NT | NT | | |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NT | NT | | |
| 26 | 9.6 | 1.2 | -0.51 | -0.43 |
| 27 | 11.1 | 3.3 | 0.45 | 0.20 |
| 28 | 14 | 2.8 | 2.31 | 1.15 |
| 29 | 13 | 2.6 | 1.67 | 0.88 |
| 30 | 10.63 | 0.5 | 0.15 | 0.15 |

Statistics

| | | |
|--------------------------|------|-----|
| Assigned Value* | 10.4 | 1.4 |
| Spike | 11.3 | 1.0 |
| Homogeneity Value | 11.0 | 1.3 |
| Robust Average | 10.1 | 1.6 |
| Median | 10.0 | 1.3 |
| Mean | 9.92 | |
| N | 17 | |
| Max. | 14 | |
| Min. | 3.4 | |
| Robust SD | 2.6 | |
| Robust CV | 25% | |

*Robust Average excluding laboratory 8.

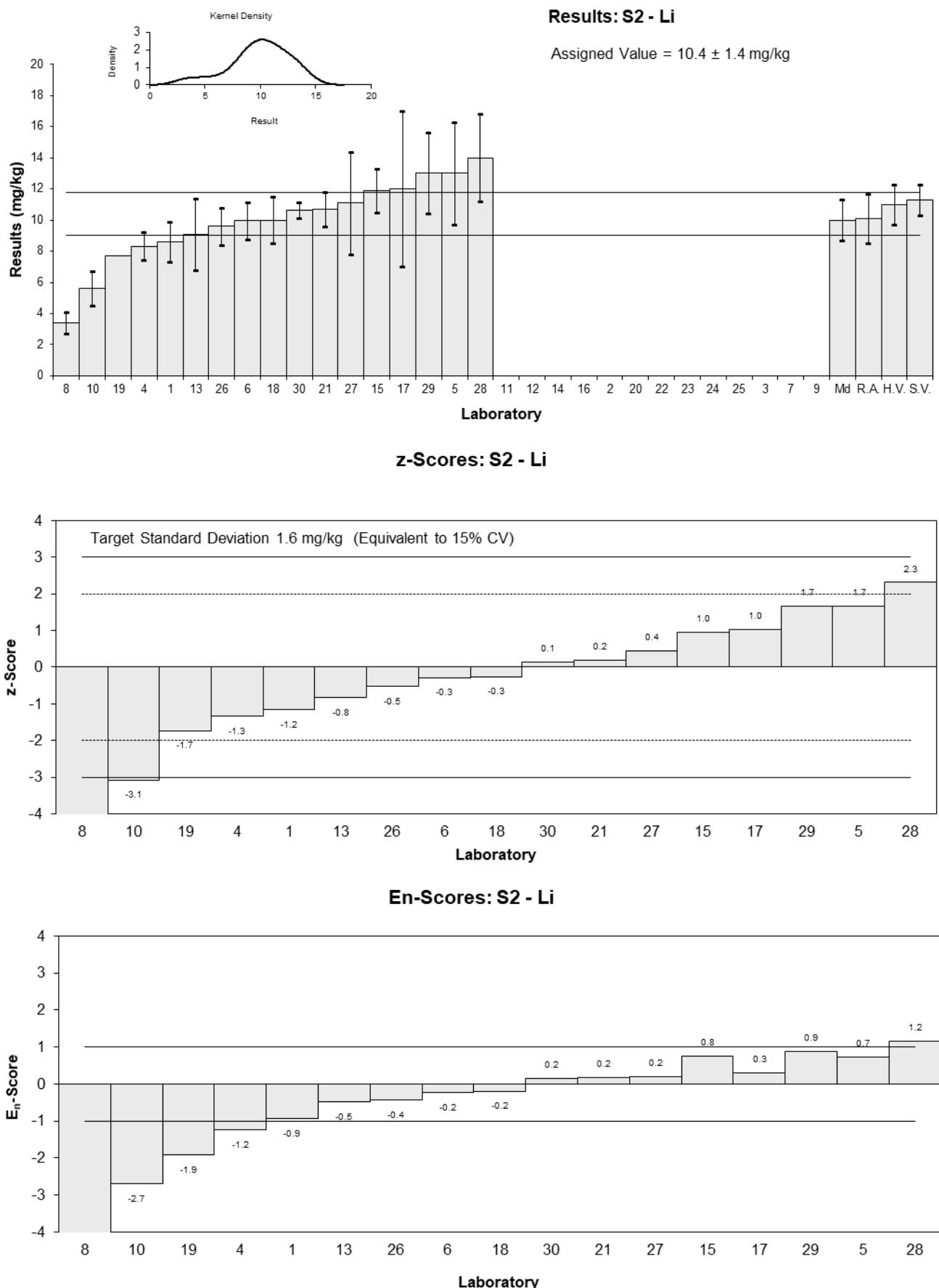


Figure 29

Table 42

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Ni |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 21 | 3.3 | -0.95 | -0.87 |
| 2 | NT | NT | | |
| 3 | 18.25 | 2 | -1.70 | -2.05 |
| 4 | 20.99 | 2.16 | -0.96 | -1.11 |
| 5 | 29 | 5.8 | 1.22 | 0.72 |
| 6 | 26.7 | 3.0 | 0.60 | 0.58 |
| 7 | NT | NT | | |
| 8 | 19.1 | 3.1 | -1.47 | -1.40 |
| 9 | NT | NT | | |
| 10 | 19 | 4.7 | -1.50 | -1.05 |
| 11 | 28 | 1 | 0.95 | 1.40 |
| 12 | NT | NT | | |
| 13 | 27 | 6 | 0.68 | 0.39 |
| 14 | NT | NT | | |
| 15 | 25.2 | 6.5 | 0.19 | 0.10 |
| 16 | 24 | 2.4 | -0.14 | -0.15 |
| 17 | 29.5 | 11.6 | 1.36 | 0.42 |
| 18 | 24.9 | 3.2 | 0.11 | 0.10 |
| 19 | 28 | 2.8 | 0.95 | 0.97 |
| 20 | NT | NT | | |
| 21 | 17.4 | 1.6 | -1.93 | -2.53 |
| 22 | 28 | 5.6 | 0.95 | 0.58 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 21 | 4.0 | -0.95 | -0.76 |
| 26 | 21.9 | 2.2 | -0.71 | -0.82 |
| 27 | 25.8 | 7.7 | 0.35 | 0.16 |
| 28 | 29 | 5.8 | 1.22 | 0.72 |
| 29 | 28 | 4.5 | 0.95 | 0.69 |
| 30 | 26.3 | 1.6 | 0.49 | 0.64 |

Statistics

| | | |
|--------------------------|------|-----|
| Assigned Value | 24.5 | 2.3 |
| Spike | 26.0 | 2.0 |
| Homogeneity Value | 26.6 | 3.2 |
| Robust Average | 24.5 | 2.3 |
| Median | 25.5 | 2.0 |
| Mean | 24.5 | |
| N | 22 | |
| Max. | 29.5 | |
| Min. | 17.4 | |
| Robust SD | 4.4 | |
| Robust CV | 18% | |

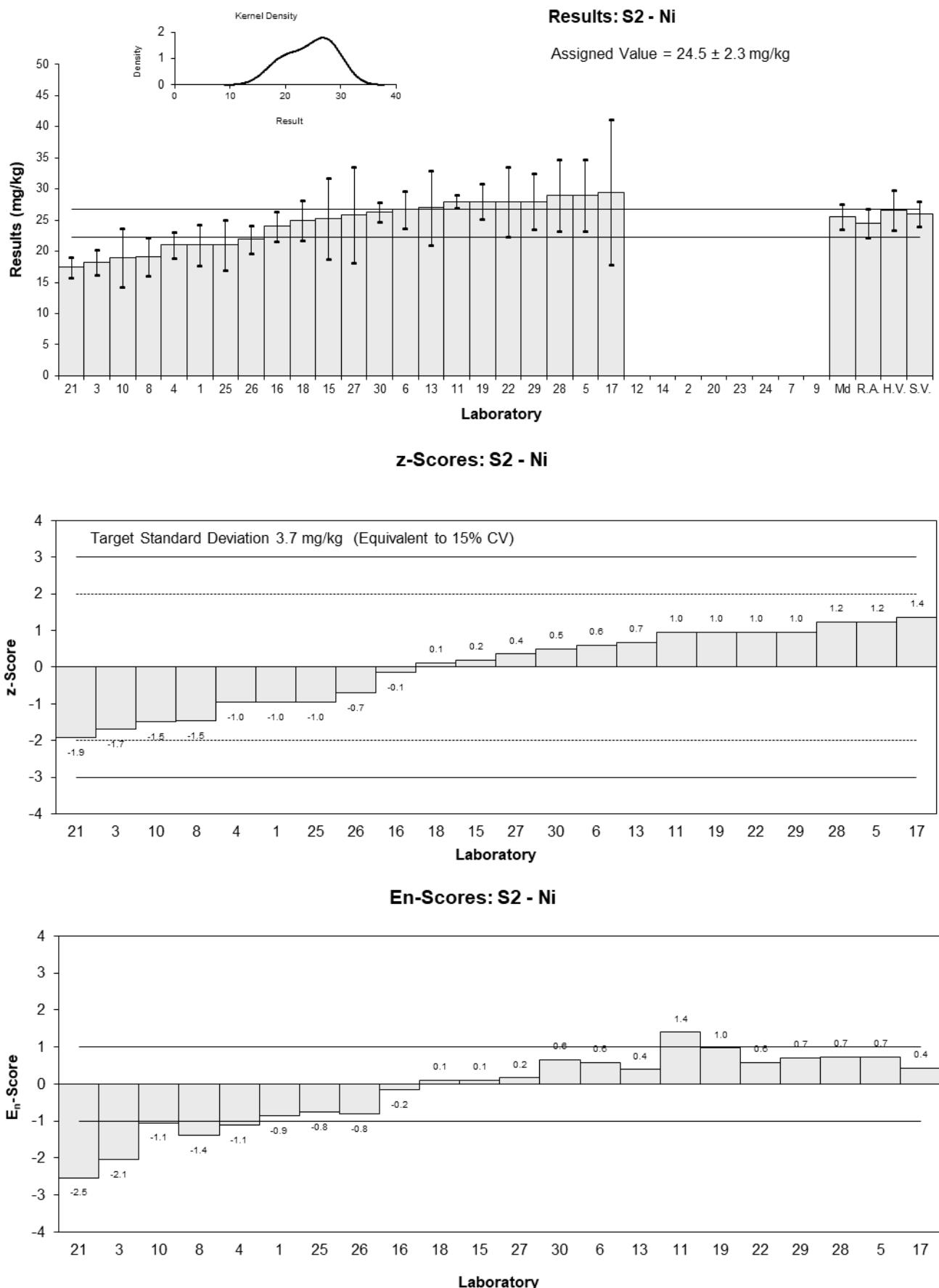


Figure 30

Table 43

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Rb |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty |
|-----------------|---------------|--------------------|
| 1 | NT | NT |
| 2 | NT | NT |
| 3 | NT | NT |
| 4 | 24 | 2.05 |
| 5 | NT | NT |
| 6 | NT | NT |
| 7 | NT | NT |
| 8 | 13.4 | 1.5 |
| 9 | NT | NT |
| 10 | 16 | 5.1 |
| 11 | NT | NT |
| 12 | NT | NT |
| 13 | NT | NT |
| 14 | NT | NT |
| 15 | NR | NR |
| 16 | NT | NT |
| 17 | NT | NT |
| 18 | NT | NT |
| 19 | NT | NT |
| 20 | NT | NT |
| 21 | NT | NT |
| 22 | NT | NT |
| 23 | NT | NT |
| 24 | NT | NT |
| 25 | NT | NT |
| 26 | 21.7 | 4.4 |
| 27 | 25.7 | 7.7 |
| 28 | NT | NT |
| 29 | NT | NT |
| 30 | NT | NT |

Statistics*

| | | |
|-----------------------|---------|------|
| Assigned Value | Not Set | |
| Spike | 7.01 | 0.14 |
| Median | 21.7 | 7.4 |
| Mean | 20.2 | |
| N | 5 | |
| Max. | 25.7 | |
| Min. | 13.4 | |

*Insufficient data to calculate statistics.

Results: S2 - Rb

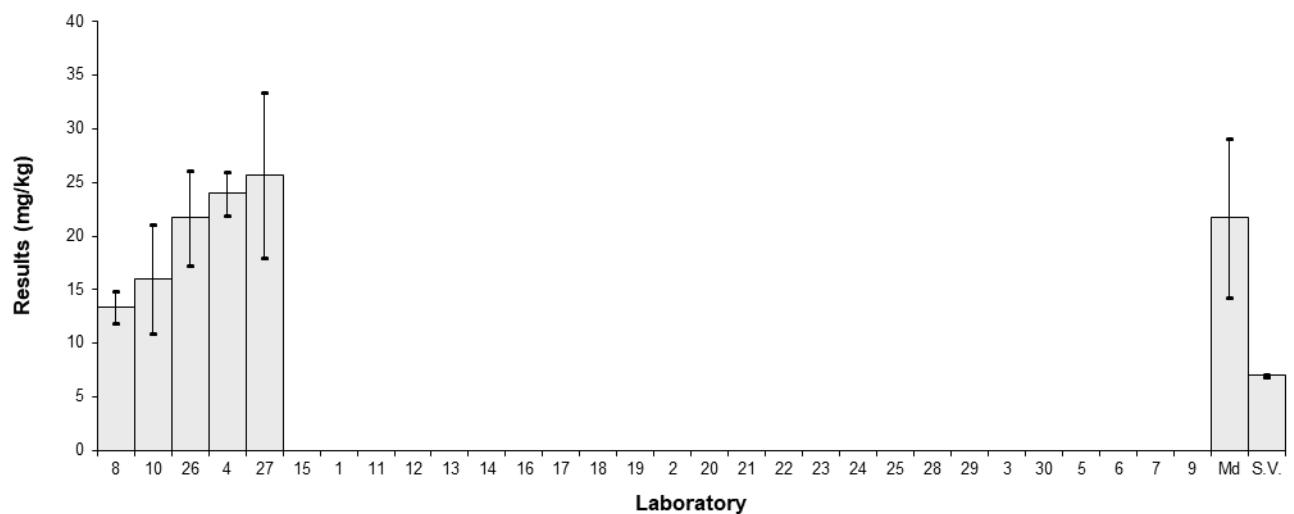


Figure 31

Table 44

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Sb |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty |
|-----------------|---------------|--------------------|
| 1 | 9.5 | 1.4 |
| 2 | NT | NT |
| 3 | 12.4 | 2 |
| 4 | 6.23 | 2.12 |
| 5 | 18 | 3.6 |
| 6 | 26.7 | 3.1 |
| 7 | NT | NT |
| 8 | 5.0 | 1.3 |
| 9 | NT | NT |
| 10 | 8.0 | 2.6 |
| 11 | NT | NT |
| 12 | NT | NT |
| 13 | NT | NT |
| 14 | NT | NT |
| 15 | 8.83 | 2.50 |
| 16 | 12.0 | 1.32 |
| 17 | <100 | NR |
| 18 | 19.2 | 2.6 |
| 19 | NT | NT |
| 20 | NT | NT |
| 21 | 4.8 | 0.5 |
| 22 | 11 | 2.2 |
| 23 | NT | NT |
| 24 | NT | NT |
| 25 | NT | NT |
| 26 | 7.0 | 1.5 |
| 27 | 15.0 | 4.5 |
| 28 | 21 | 4.2 |
| 29 | 18 | 2.6 |
| 30 | 20.40 | 1.7 |

Statistics

| | | |
|-----------------------|---------|-----|
| Assigned Value | Not Set | |
| Spike | 22.2 | 0.4 |
| Robust Average | 12.9 | 4.2 |
| Median | 12.0 | 4.4 |
| Mean | 13.1 | |
| N | 17 | |
| Max. | 26.7 | |
| Min. | 4.8 | |
| Robust SD | 6.9 | |
| Robust CV | 53% | |

Results: S2 - Sb

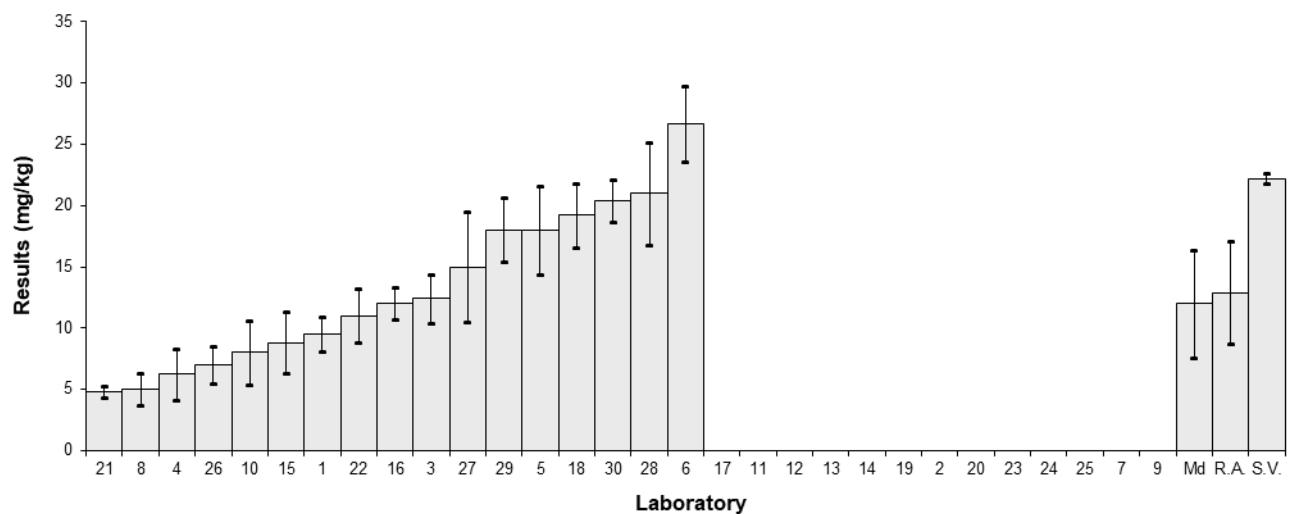


Figure 32

Table 45

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Se |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty |
|-----------------|---------------|--------------------|
| 1 | 9.6 | 1.44 |
| 2 | NT | NT |
| 3 | 0.93 | 0.5 |
| 4 | <5 | NR |
| 5 | <2 | NR |
| 6 | 1.74 | 0.2 |
| 7 | NT | NT |
| 8 | 1 | 0.5 |
| 9 | NT | NT |
| 10 | 0.95 | 0.17 |
| 11 | NT | NT |
| 12 | NT | NT |
| 13 | <5 | NR |
| 14 | NT | NT |
| 15 | 1.10 | 0.42 |
| 16 | 1.08 | 0.13 |
| 17 | <100 | NR |
| 18 | 1.43 | 0.17 |
| 19 | 2.4 | 0.12 |
| 20 | <5 | NR |
| 21 | NR | NR |
| 22 | 1.8 | 0.36 |
| 23 | NT | NT |
| 24 | NT | NT |
| 25 | NT | NT |
| 26 | < 2 | 1.4 |
| 27 | 2.00 | 0.6 |
| 28 | 2.1 | 0.42 |
| 29 | <2 | NR |
| 30 | 1.22 | 0.05 |

Statistics

| | | |
|--------------------------|---------|------|
| Assigned Value | Not Set | |
| Spike | 1.98 | 0.20 |
| Homogeneity Value | 1.60 | 0.19 |
| Robust Average | 1.56 | 0.45 |
| Median | 1.43 | 0.39 |
| Mean | 2.10 | |
| N | 13 | |
| Max. | 9.6 | |
| Min. | 0.93 | |
| Robust SD | 0.64 | |
| Robust CV | 41% | |

Results: S2 - Se

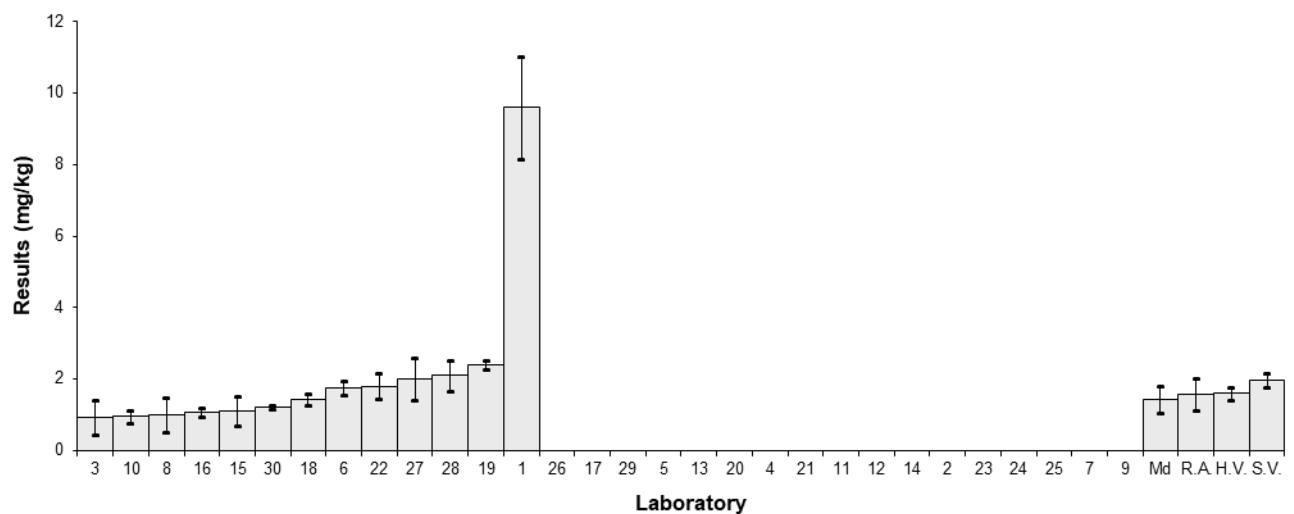


Figure 33

Table 46

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Sn |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 14 | 2.1 | -1.25 | -0.84 |
| 2 | NT | NT | | |
| 3 | 12 | 1 | -2.50 | -2.69 |
| 4 | 15.9 | 4.18 | -0.06 | -0.02 |
| 5 | 17 | 3.4 | 0.62 | 0.28 |
| 6 | NT | NT | | |
| 7 | NT | NT | | |
| 8 | 14.9 | 4.5 | -0.69 | -0.24 |
| 9 | NT | NT | | |
| 10 | 16 | 5.4 | 0.00 | 0.00 |
| 11 | NT | NT | | |
| 12 | NT | NT | | |
| 13 | 15 | 4 | -0.62 | -0.24 |
| 14 | NT | NT | | |
| 15 | 15.9 | 2.9 | -0.06 | -0.03 |
| 16 | 14.2 | 1.99 | -1.13 | -0.79 |
| 17 | <50 | NR | | |
| 18 | 14.6 | 3.1 | -0.88 | -0.43 |
| 19 | 25 | NR | 5.62 | 8.18 |
| 20 | NT | NT | | |
| 21 | NT | NT | | |
| 22 | 18 | 3.6 | 1.25 | 0.53 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NT | NT | | |
| 26 | 17.0 | 2.4 | 0.62 | 0.38 |
| 27 | 17.0 | 5.1 | 0.62 | 0.19 |
| 28 | 19 | 3.8 | 1.88 | 0.76 |
| 29 | 18 | 2.7 | 1.25 | 0.69 |
| 30 | 16.37 | 1.2 | 0.23 | 0.23 |

Statistics

| | | |
|--------------------------|------|-----|
| Assigned Value* | 16.0 | 1.1 |
| Spike | 16.9 | 0.3 |
| Homogeneity Value | 16.8 | 2.0 |
| Robust Average | 16.2 | 1.2 |
| Median | 16.0 | 0.8 |
| Mean | 16.5 | |
| N | 17 | |
| Max. | 25 | |
| Min. | 12 | |
| Robust SD | 2.0 | |
| Robust CV | 12% | |

*Robust Average excluding laboratory 19.

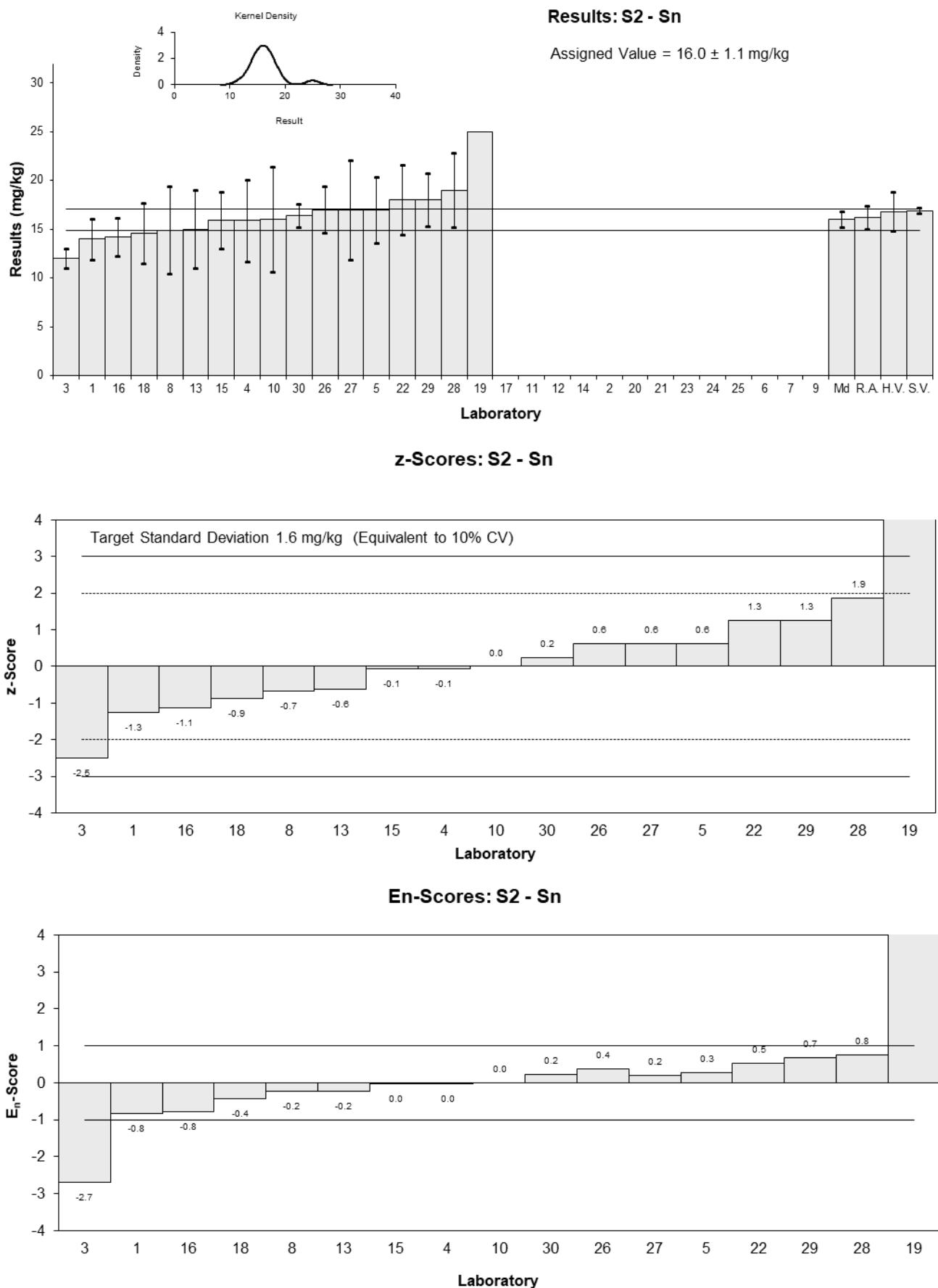


Figure 34

Table 47

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Tl |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 1.2 | 0.18 | -0.32 | -0.25 |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | 126.9 | 9.92 | 664.76 | 12.66 |
| 5 | <10 | NR | | |
| 6 | 1.46 | 0.2 | 1.06 | 0.78 |
| 7 | NT | NT | | |
| 8 | 1.2 | 0.1 | -0.32 | -0.32 |
| 9 | NT | NT | | |
| 10 | <1.8 | NR | | |
| 11 | NT | NT | | |
| 12 | NT | NT | | |
| 13 | 1.28 | 1.1 | 0.11 | 0.02 |
| 14 | NT | NT | | |
| 15 | 0.92 | 0.32 | -1.80 | -0.95 |
| 16 | 0.96 | 0.12 | -1.59 | -1.50 |
| 17 | <2 | NR | | |
| 18 | 1.19 | 0.11 | -0.37 | -0.36 |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | NT | NT | | |
| 22 | 1.5 | 0.30 | 1.27 | 0.71 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NT | NT | | |
| 26 | 1.35 | 0.19 | 0.48 | 0.36 |
| 27 | 1.4 | 4.3 | 0.74 | 0.03 |
| 28 | <10 | NR | | |
| 29 | <10 | NR | | |
| 30 | 1.40 | 0.2 | 0.74 | 0.55 |

Statistics*

| | | |
|--------------------------|------|------|
| Assigned Value | 1.26 | 0.16 |
| Spike | 1.44 | 0.03 |
| Homogeneity Value | 1.40 | 0.17 |
| Robust Average | 1.26 | 0.16 |
| Median | 1.28 | 0.12 |
| Mean | 1.26 | |
| N | 11 | |
| Max. | 1.5 | |
| Min. | 0.92 | |
| Robust SD | 0.21 | |
| Robust CV | 17% | |

*Laboratory 4 was excluded from statistical calculation (extreme outlier).

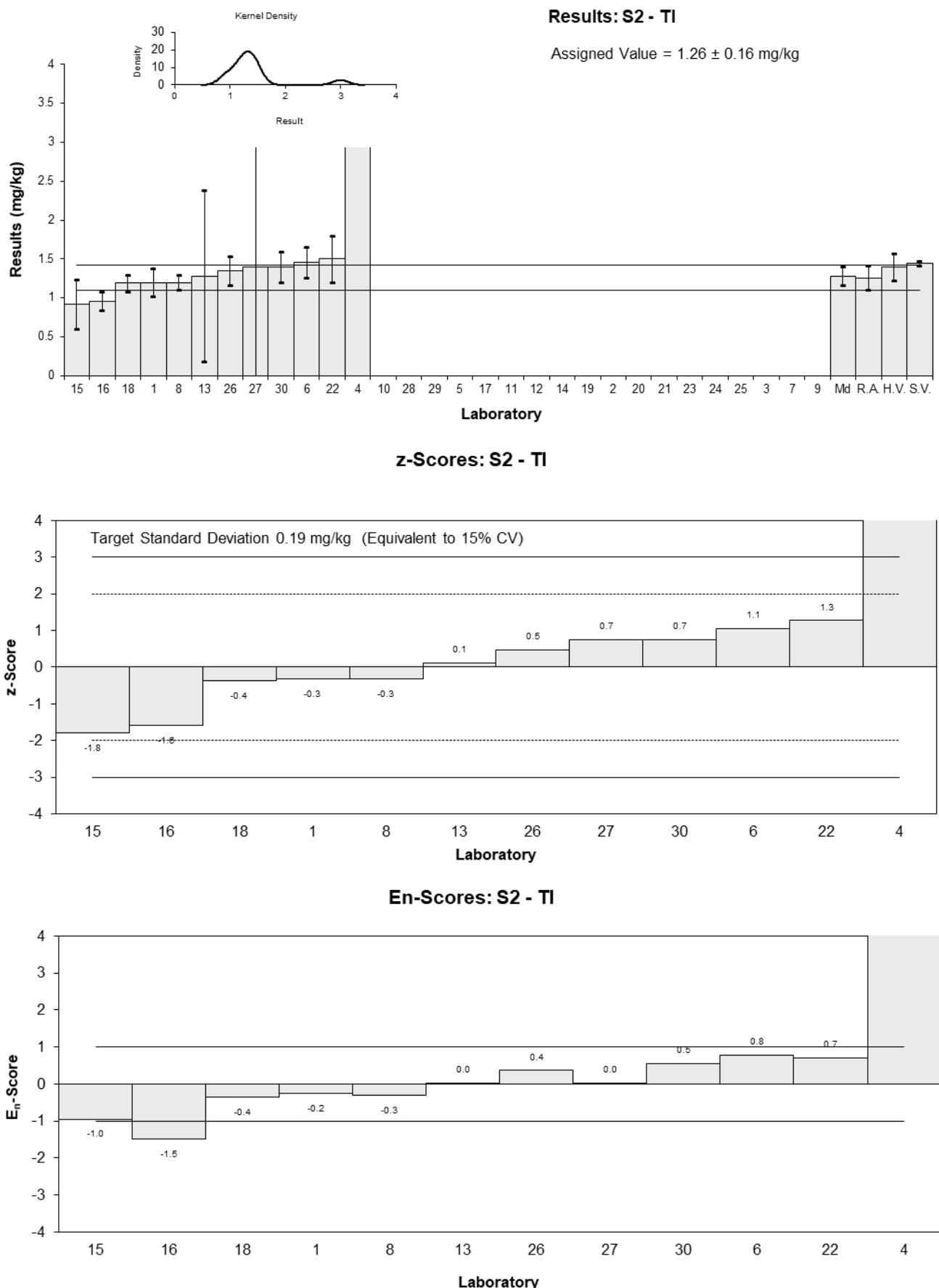


Figure 35

Table 48

Sample Details

| | |
|-------------------|-------|
| Sample No. | S2 |
| Matrix. | Soil |
| Analyte. | Zn |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 22 | 3.9 | -1.92 | -1.85 |
| 2 | NT | NT | | |
| 3 | 15 | 2 | -3.43 | -4.62 |
| 4 | 26.6 | 3.09 | -0.93 | -1.03 |
| 5 | 33 | 6.6 | 0.45 | 0.29 |
| 6 | 30.5 | 3.2 | -0.09 | -0.09 |
| 7 | NT | NT | | |
| 8 | 17.4 | 3.0 | -2.91 | -3.29 |
| 9 | NT | NT | | |
| 10 | 21 | 3.2 | -2.14 | -2.33 |
| 11 | 38 | 2 | 1.53 | 2.06 |
| 12 | NT | NT | | |
| 13 | 32 | 6 | 0.24 | 0.17 |
| 14 | NT | NT | | |
| 15 | 32.7 | 3.9 | 0.39 | 0.37 |
| 16 | 36.1 | 4.33 | 1.12 | 1.01 |
| 17 | 34.0 | 7 | 0.67 | 0.41 |
| 18 | 29.0 | 4.4 | -0.41 | -0.36 |
| 19 | 34 | 3.4 | 0.67 | 0.70 |
| 20 | NT | NT | | |
| 21 | 32.1 | 4.1 | 0.26 | 0.24 |
| 22 | 36 | 7.2 | 1.10 | 0.66 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 26.8 | 4.7 | -0.88 | -0.75 |
| 26 | 26.8 | 4.4 | -0.88 | -0.79 |
| 27 | 31 | 9.3 | 0.02 | 0.01 |
| 28 | 35 | 7 | 0.88 | 0.54 |
| 29 | 36 | 5.5 | 1.10 | 0.83 |
| 30 | 29.82 | 2.4 | -0.23 | -0.29 |

Statistics

| | | |
|--------------------------|------------|-----|
| Assigned Value* | 30.9 | 2.8 |
| Spike | Not Spiked | |
| Homogeneity Value | 29.2 | 3.5 |
| Robust Average | 30.2 | 3.2 |
| Median | 31.5 | 2.6 |
| Mean | 29.8 | |
| N | 22 | |
| Max. | 38 | |
| Min. | 15 | |
| Robust SD | 5.9 | |
| Robust CV | 20% | |

*Robust Average excluding laboratory 3.

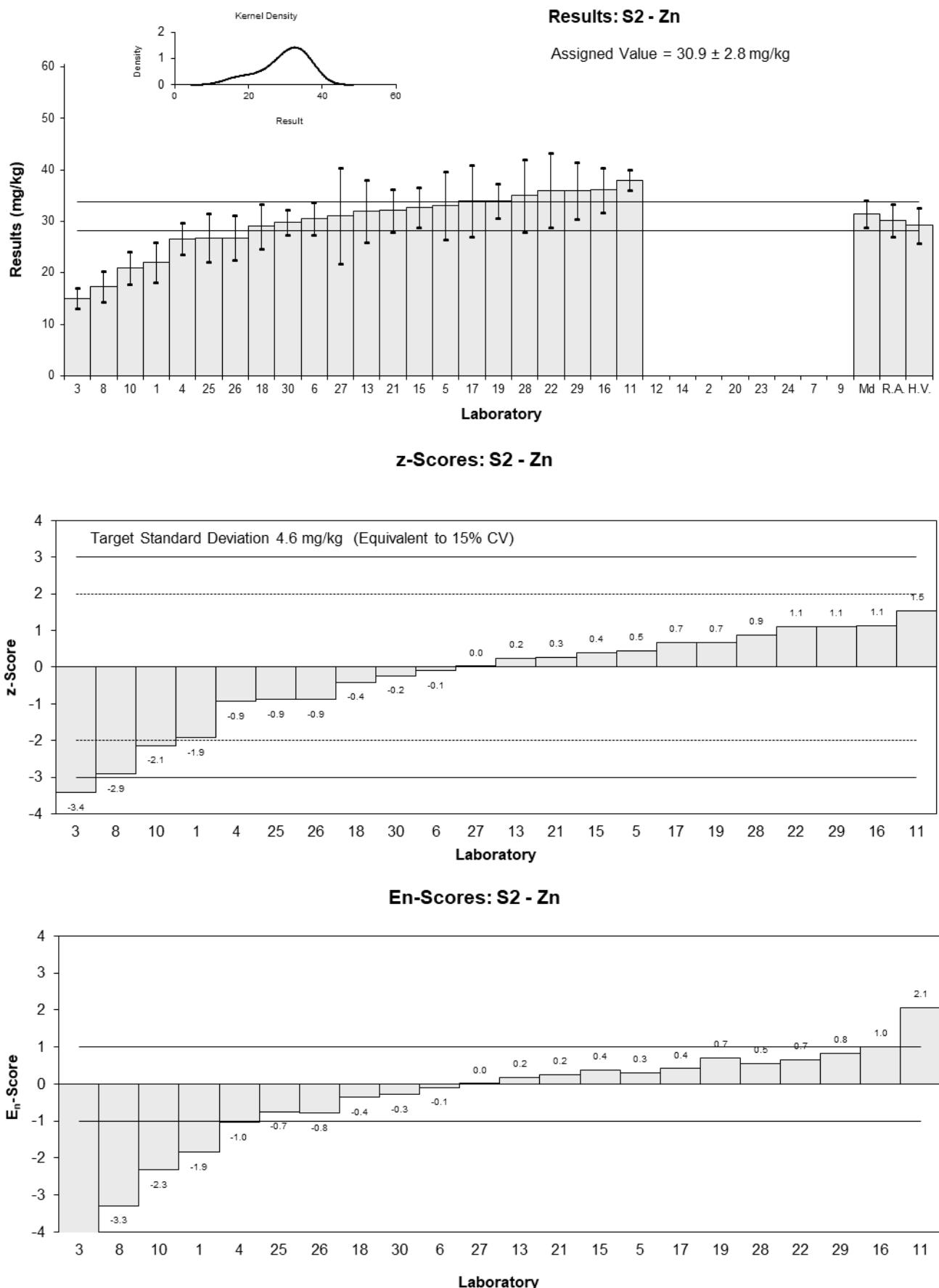


Figure 36

Table 49

Sample Details

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

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E _n -Score |
|----------|--------|-------------|---------|-----------------------|
| 1 | NT | NT | | |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | NT | NT | | |
| 5 | NT | NT | | |
| 6 | 44.6 | 4.8 | 1.21 | 0.85 |
| 7 | NT | NT | | |
| 8 | NT | NT | | |
| 9 | 35.6 | NR | -1.06 | -1.40 |
| 10 | 81 | 12 | 10.35 | 3.33 |
| 11 | NT | NT | | |
| 12 | 41 | 4.0 | 0.30 | 0.24 |
| 13 | NT | NT | | |
| 14 | 39 | 6.3 | -0.20 | -0.11 |
| 15 | 40.05 | 8.01 | 0.06 | 0.03 |
| 16 | 420 | 68 | 95.53 | 5.59 |
| 17 | 42.0 | 12 | 0.55 | 0.18 |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | NT | NT | | |
| 22 | 38 | 8 | -0.45 | -0.21 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NT | NT | | |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 43 | 0.8 | 0.80 | 1.03 |
| 29 | 35.24 | 7.56 | -1.15 | -0.56 |
| 30 | NT | NT | | |

Statistics*

| | | |
|--------------------------|------------|-----|
| Assigned Value** | 39.8 | 3.0 |
| Spike | Not Spiked | |
| Homogeneity Value | 36.5 | 7.3 |
| Robust Average | 40.5 | 3.4 |
| Median | 40.5 | 2.7 |
| Mean | 43.9 | |
| N | 10 | |
| Max. | 81 | |
| Min. | 35.24 | |
| Robust SD | 4.3 | |
| Robust CV | 11% | |

*Laboratory 16 was excluded from statistical calculation (extreme outlier)

**Robust Average excluding laboratory 10.

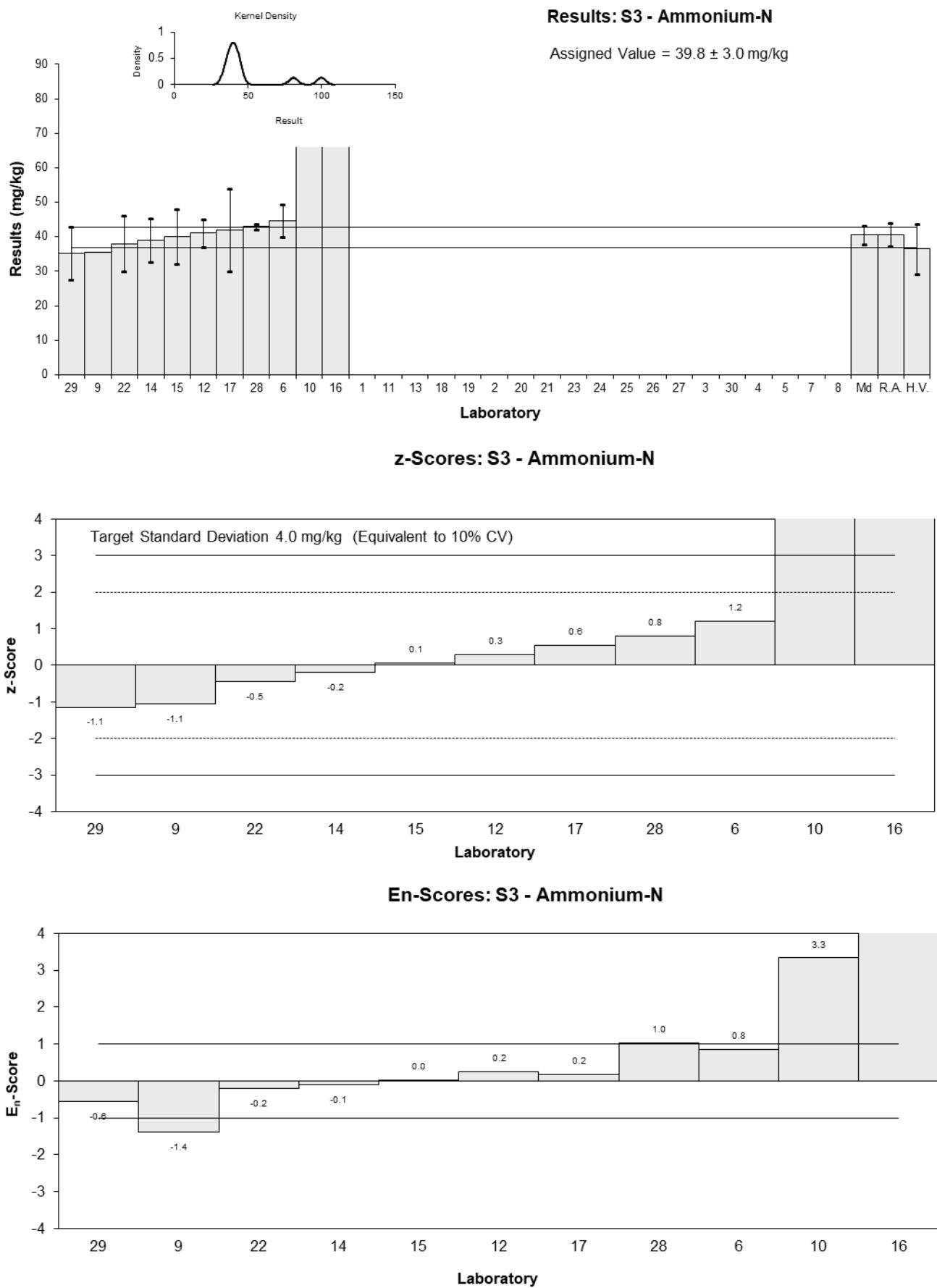


Figure 37

Table 50

Sample Details

| | |
|-------------------|---------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | Bromide |
| 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 | NT | NT |
| 7 | 1.58 | 0.14 |
| 8 | 1.92 | 0.19 |
| 9 | NR | NR |
| 10 | NT | NT |
| 11 | NT | NT |
| 12 | NR | NR |
| 13 | <5 | NR |
| 14 | <5 | NR |
| 15 | NR | NR |
| 16 | NT | NT |
| 17 | NT | NT |
| 18 | NT | NT |
| 19 | NT | NT |
| 20 | NT | NT |
| 21 | 2.41 | 0.3 |
| 22 | 2.0 | 0.4 |
| 23 | NT | NT |
| 24 | NT | NT |
| 25 | NT | NT |
| 26 | NT | NT |
| 27 | NT | NT |
| 28 | NT | NT |
| 29 | 1.92 | 0.49 |
| 30 | NT | NT |

Statistics*

| | | |
|--------------------------|------------|------|
| Assigned Value | Not Set | |
| Spike | Not Spiked | |
| Homogeneity Value | 2.00 | 0.30 |
| Median | 1.92 | 0.15 |
| Mean | 1.97 | |
| N | 5 | |
| Max. | 2.41 | |
| Min. | 1.58 | |

*Insufficient data to calculate statistics.

Results: S3 - Bromide

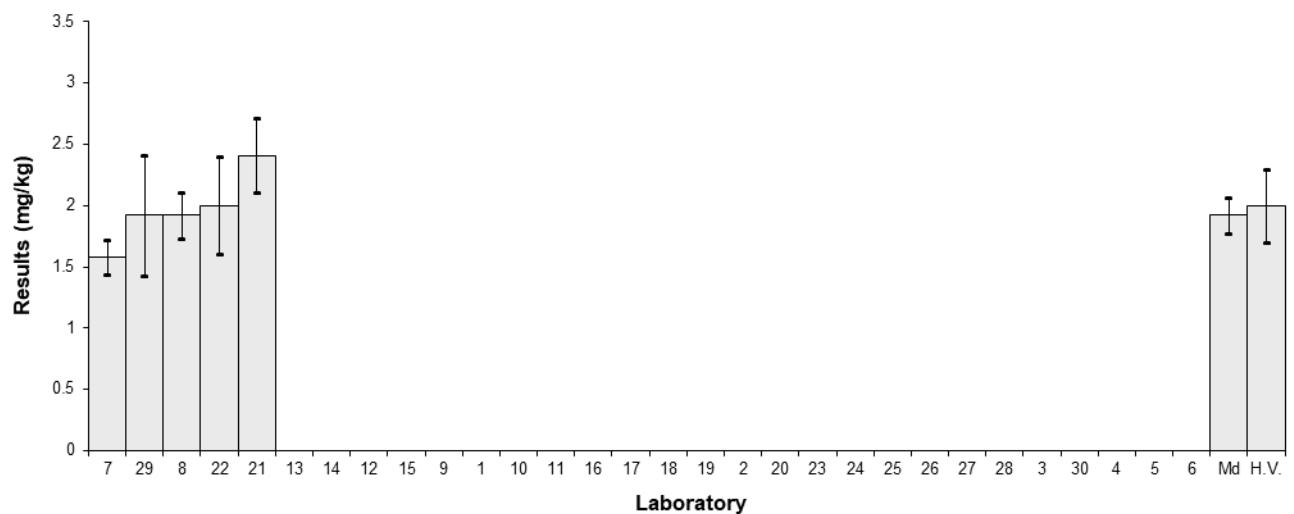


Figure 38

Table 51

Sample Details

| | |
|-------------------|-------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | Ca |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 1100 | 165 | -1.73 | -1.19 |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | 1492 | 162.1 | 1.22 | 0.85 |
| 5 | NT | NT | | |
| 6 | 1550 | 150 | 1.65 | 1.22 |
| 7 | 1430 | 280 | 0.75 | 0.34 |
| 8 | 1440 | 285 | 0.83 | 0.36 |
| 9 | NR | NR | | |
| 10 | 1300 | 280 | -0.23 | -0.10 |
| 11 | NT | NT | | |
| 12 | 1260 | 125 | -0.53 | -0.44 |
| 13 | 1400 | 200 | 0.53 | 0.31 |
| 14 | 1200 | 120 | -0.98 | -0.83 |
| 15 | 1270 | 210 | -0.45 | -0.26 |
| 16 | 1200 | 138 | -0.98 | -0.76 |
| 17 | 1665 | 1300 | 2.52 | 0.26 |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | 1350 | 96 | 0.15 | 0.14 |
| 22 | 1100 | 220 | -1.73 | -0.95 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 1250 | 214 | -0.60 | -0.34 |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 1400 | 28 | 0.53 | 0.67 |
| 29 | NT | NT | | |
| 30 | NT | NT | | |

Statistics

| | | |
|--------------------------|------------|-----|
| Assigned Value | 1330 | 100 |
| Spike | Not Spiked | |
| Homogeneity Value | 1390 | 170 |
| Robust Average | 1330 | 100 |
| Median | 1330 | 90 |
| Mean | 1340 | |
| N | 16 | |
| Max. | 1665 | |
| Min. | 1100 | |
| Robust SD | 170 | |
| Robust CV | 13% | |

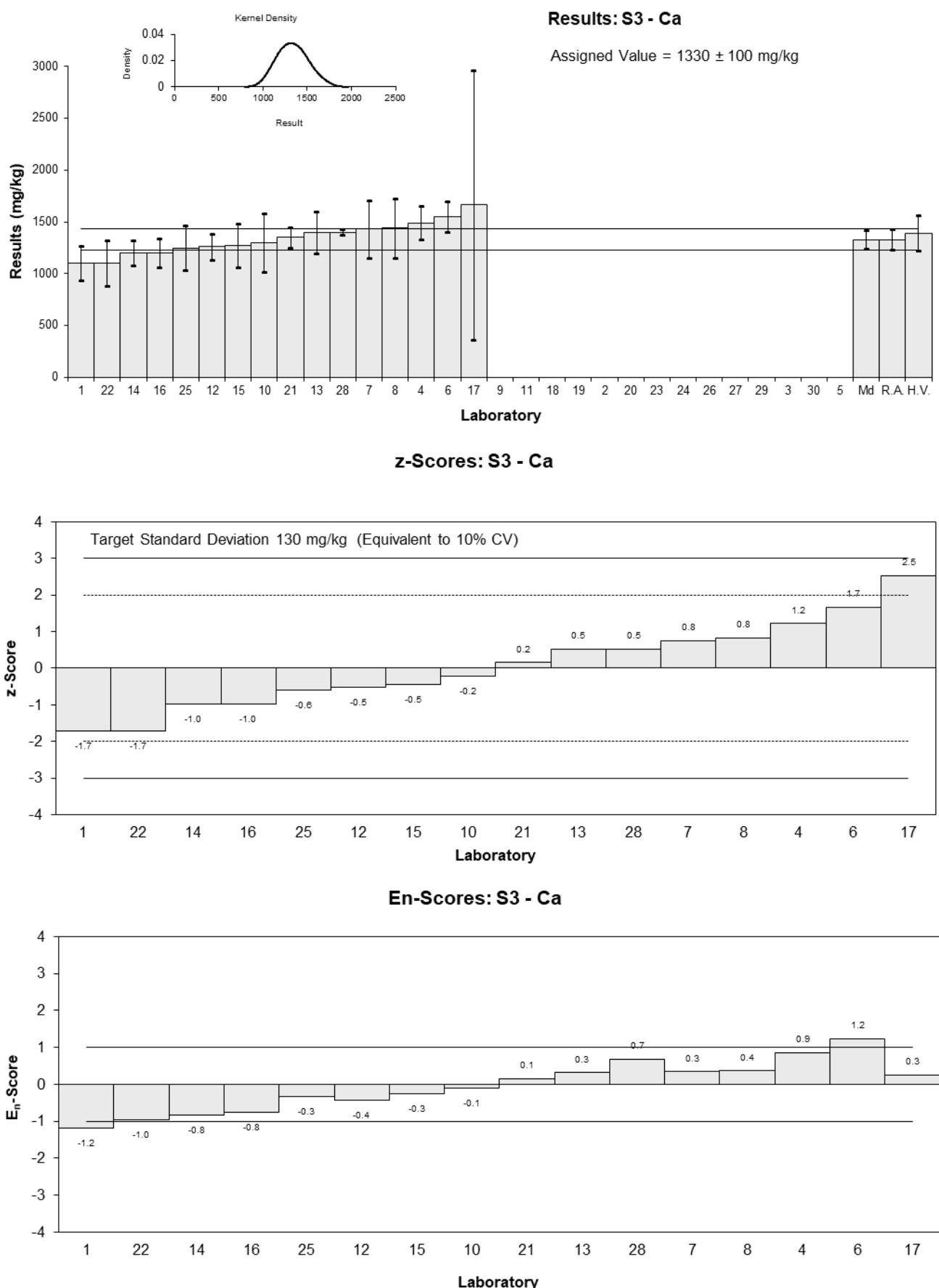


Figure 39

Table 52

Sample Details

| | |
|-------------------|----------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | Chloride |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E _n -Score |
|----------|--------|-------------|---------|-----------------------|
| 1 | NT | NT | | |
| 2 | 100 | 9 | 1.93 | 1.29 |
| 3 | NT | NT | | |
| 4 | 110 | 13.5 | 3.13 | 1.63 |
| 5 | NT | NT | | |
| 6 | NT | NT | | |
| 7 | 85.7 | 10 | 0.23 | 0.14 |
| 8 | 105 | 10 | 2.53 | 1.60 |
| 9 | 76.2 | NR | -0.91 | -0.87 |
| 10 | NT | NT | | |
| 11 | NT | NT | | |
| 12 | 160 | 16 | 9.09 | 4.18 |
| 13 | 77 | 8 | -0.81 | -0.58 |
| 14 | 865 | 196 | 93.22 | 3.98 |
| 15 | 78.3 | 18.8 | -0.66 | -0.27 |
| 16 | 78.5 | 6.67 | -0.63 | -0.48 |
| 17 | 75 | 25 | -1.05 | -0.33 |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | 87.1 | 7.8 | 0.39 | 0.28 |
| 22 | 81 | 16 | -0.33 | -0.15 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NT | NT | | |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 73.3 | 14.66 | -1.25 | -0.62 |
| 29 | 72.74 | 9.20 | -1.32 | -0.87 |
| 30 | NT | NT | | |

Statistics*

| | | |
|--------------------------|------------|-----|
| Assigned Value** | 83.8 | 8.7 |
| Spike | Not Spiked | |
| Homogeneity Value | 80 | 10 |
| Robust Average | 86 | 10 |
| Median | 79.8 | 5.3 |
| Mean | 90.0 | |
| N | 14 | |
| Max. | 160 | |
| Min. | 72.74 | |
| Robust SD | 16 | |
| Robust CV | 18% | |

*Laboratory 14 was excluded from statistical calculation (extreme outlier).

**Robust Average excluding laboratory 12.

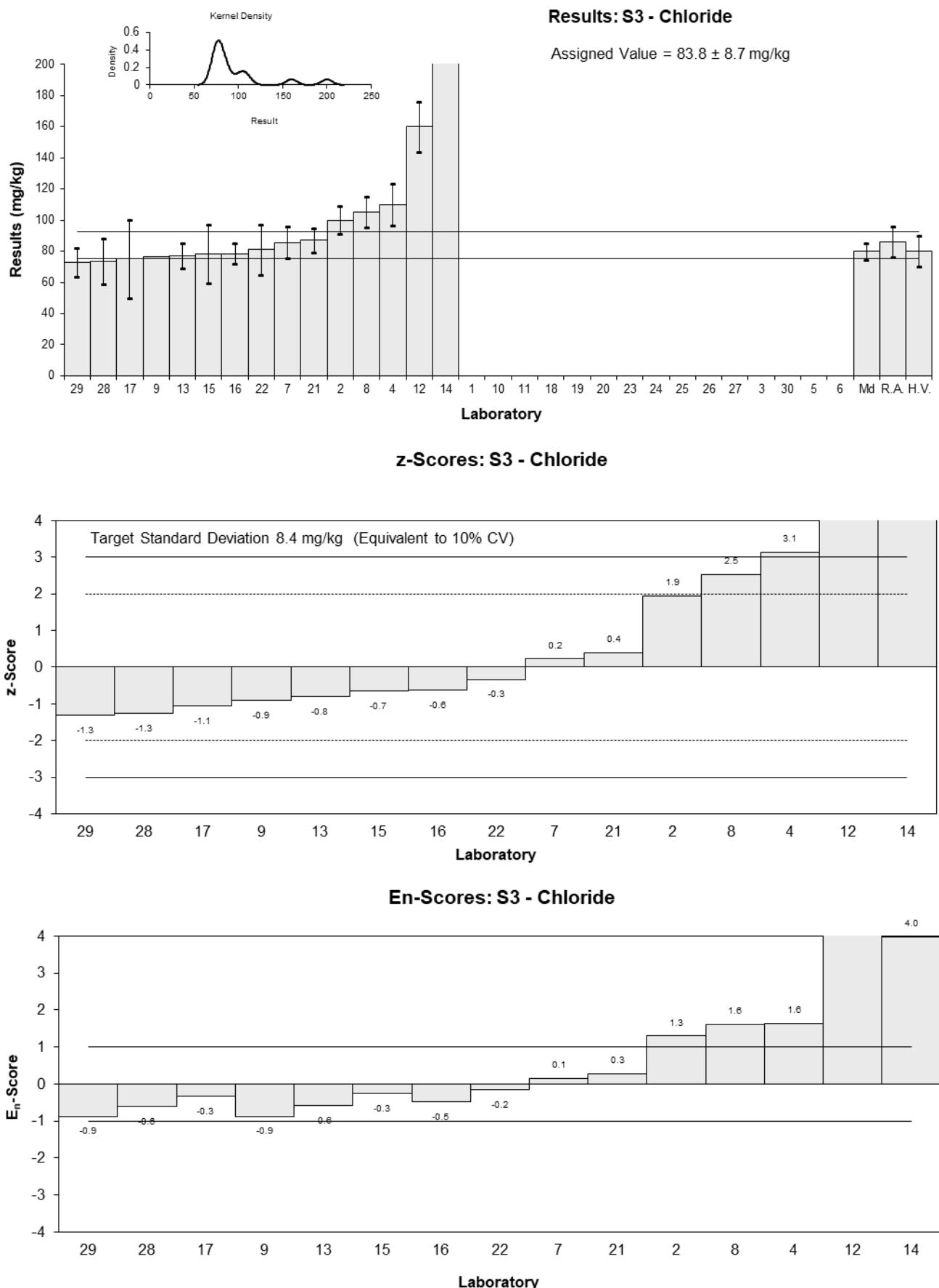


Figure 40

Table 53

Sample Details

| | |
|-------------------|-------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | EC |
| Units | µS/cm |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 890 | 130 | 1.20 | 0.27 |
| 2 | 864 | 21 | 0.33 | 0.26 |
| 3 | NT | NT | | |
| 4 | 595 | 12.1 | -8.67 | -7.57 |
| 5 | 801 | 40 | -1.77 | -1.03 |
| 6 | 890 | 45 | 1.20 | 0.65 |
| 7 | 843 | 76 | -0.37 | -0.13 |
| 8 | 809 | 18 | -1.51 | -1.23 |
| 9 | 857.3 | 6 | 0.11 | 0.10 |
| 10 | 79 | 3.9 | -25.93 | -24.04 |
| 11 | 887.20 | 160 | 1.11 | 0.20 |
| 12 | 915 | 90 | 2.04 | 0.64 |
| 13 | 900 | 20 | 1.54 | 1.22 |
| 14 | 846 | 85 | -0.27 | -0.09 |
| 15 | NR | NR | | |
| 16 | 893 | 75 | 1.30 | 0.48 |
| 17 | 780 | NR | -2.48 | -2.31 |
| 18 | 886 | 3.4 | 1.07 | 0.99 |
| 19 | NT | NT | | |
| 20 | 811 | NR | -1.44 | -1.34 |
| 21 | 912 | 23 | 1.94 | 1.47 |
| 22 | 930 | 140 | 2.54 | 0.53 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NR | NR | | |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 794.8 | 15.8 | -1.98 | -1.66 |
| 29 | 797.9 | 114.66 | -1.88 | -0.47 |
| 30 | NT | NT | | |

Statistics*

| | | |
|-----------------------|------------|----|
| Assigned Value | 854 | 32 |
| Spike | Not Spiked | |
| Robust Average | 854 | 32 |
| Median | 861 | 31 |
| Mean | 845 | |
| N | 20 | |
| Max. | 930 | |
| Min. | 595 | |
| Robust SD | 57 | |
| Robust CV | 6.7% | |

*Laboratory 10 was excluded from statistical calculation (extreme outlier).

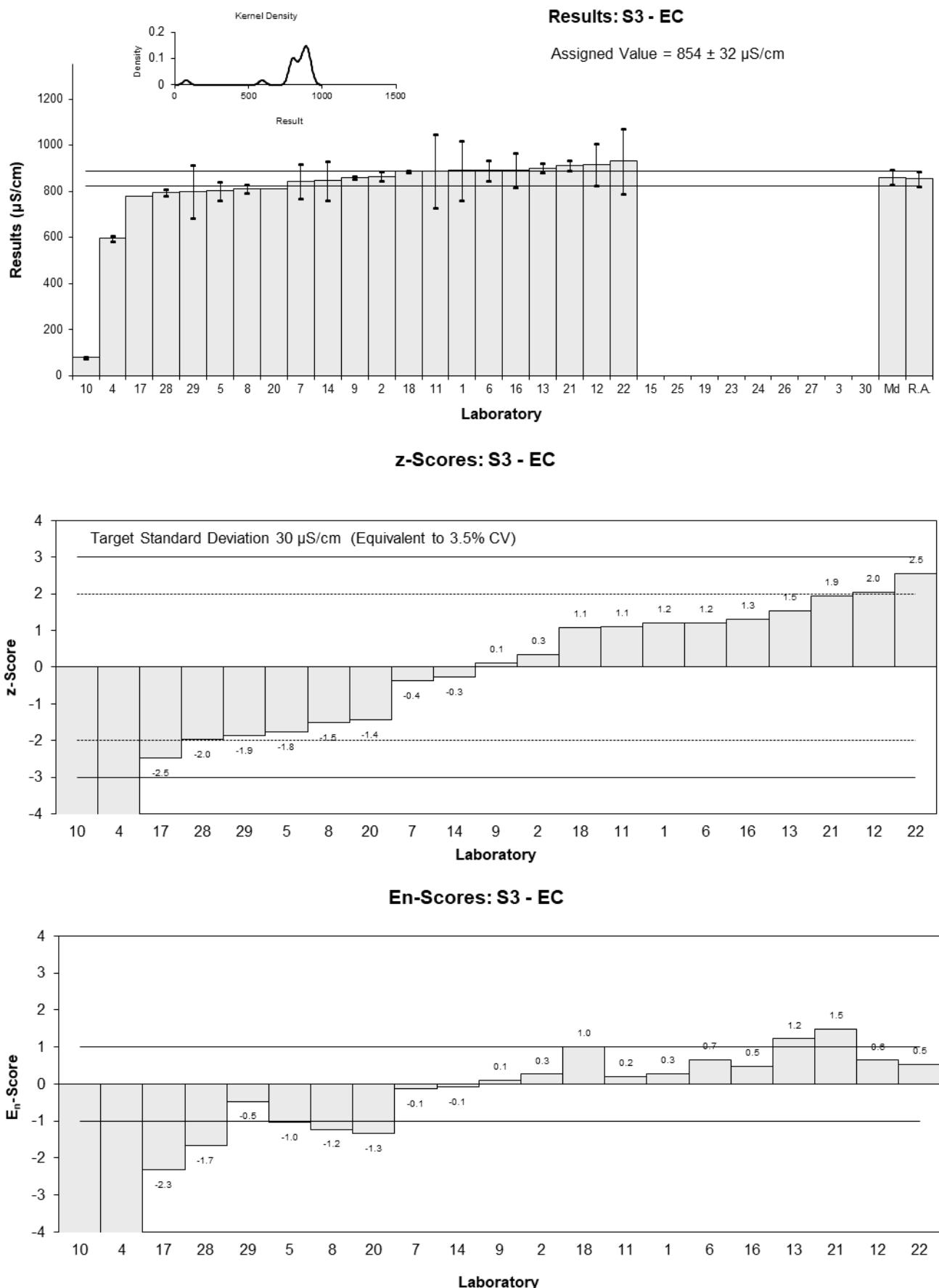


Figure 41

Table 54

Sample Details

| | |
|-------------------|-------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | Fe |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 5800 | 885 | -2.00 | -1.29 |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | 8191 | 770 | 1.30 | 0.90 |
| 5 | 8200 | 1600 | 1.31 | 0.54 |
| 6 | 8130 | 820 | 1.21 | 0.82 |
| 7 | 7010 | 2440 | -0.33 | -0.09 |
| 8 | 5950 | 681 | -1.79 | -1.33 |
| 9 | NR | NR | | |
| 10 | 5800 | 1300 | -2.00 | -0.98 |
| 11 | 7230 | 360 | -0.03 | -0.03 |
| 12 | 6710 | 670 | -0.74 | -0.56 |
| 13 | 7700 | 400 | 0.62 | 0.56 |
| 14 | NR | NR | | |
| 15 | 6530 | 610 | -0.99 | -0.78 |
| 16 | 8324 | 916 | 1.48 | 0.93 |
| 17 | 9065 | 1800 | 2.50 | 0.94 |
| 18 | 7080 | 870 | -0.23 | -0.15 |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | 6750 | 675 | -0.69 | -0.51 |
| 22 | 6900 | 1380 | -0.48 | -0.23 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 5230 | 677 | -2.79 | -2.07 |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 8300 | 166 | 1.45 | 1.46 |
| 29 | 8700 | 1200 | 2.00 | 1.04 |
| 30 | NT | NT | | |

Statistics

| | | |
|--------------------------|------------|-----|
| Assigned Value | 7250 | 700 |
| Spike | Not Spiked | |
| Homogeneity Value | 7570 | 910 |
| Robust Average | 7250 | 700 |
| Median | 7080 | 790 |
| Mean | 7240 | |
| N | 19 | |
| Max. | 9065 | |
| Min. | 5230 | |
| Robust SD | 1200 | |
| Robust CV | 17% | |

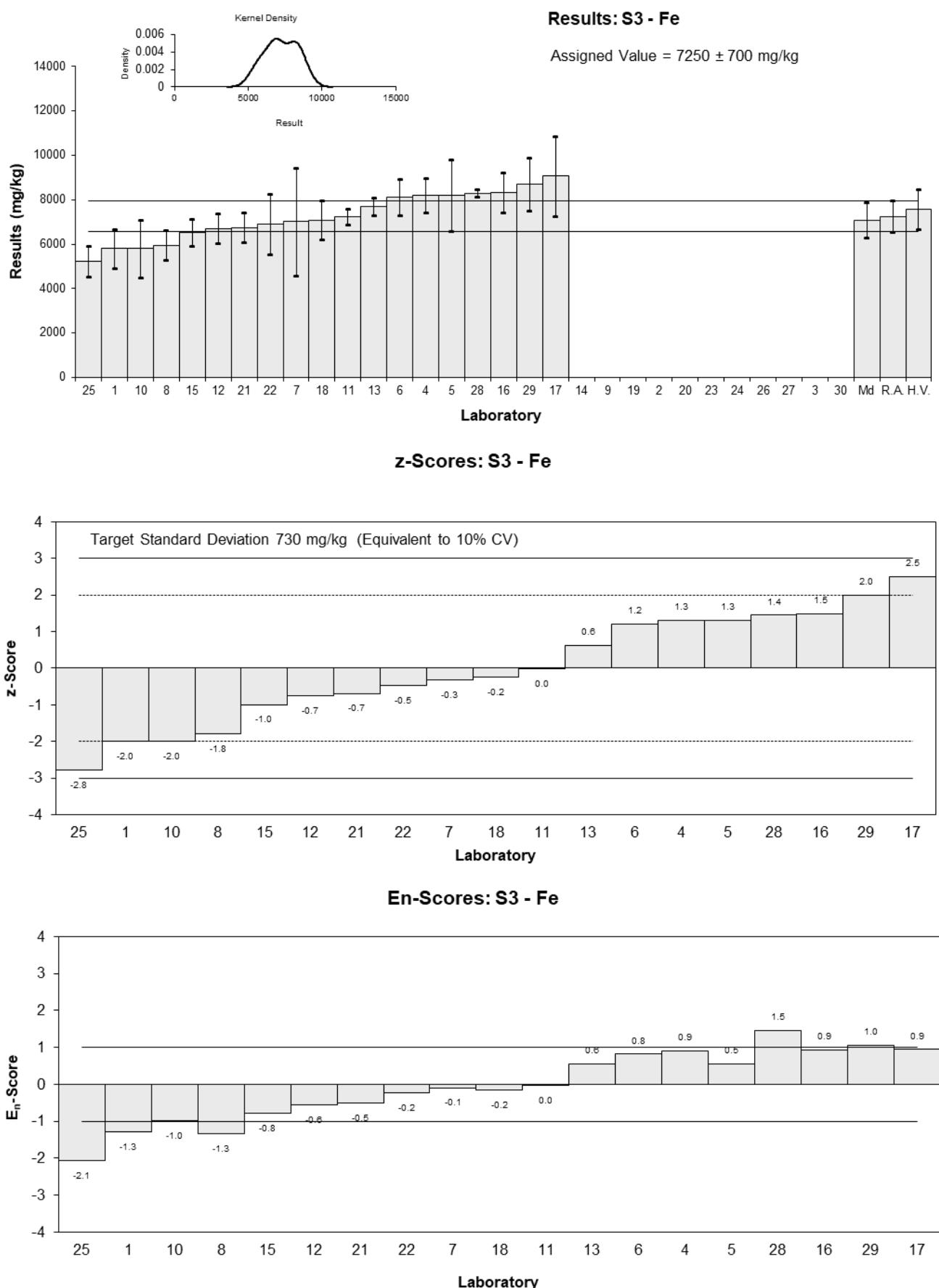


Figure 42

Table 55

Sample Details

| | |
|-------------------|----------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | Fluoride |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | NT | NT | | |
| 2 | 4 | 1 | 1.35 | 0.67 |
| 3 | NT | NT | | |
| 4 | 3.53 | NR | 0.60 | 0.49 |
| 5 | NT | NT | | |
| 6 | NT | NT | | |
| 7 | 29.8 | 2.7 | 42.30 | 9.48 |
| 8 | 4 | 0.7 | 1.35 | 0.81 |
| 9 | NR | NR | | |
| 10 | NT | NT | | |
| 11 | NT | NT | | |
| 12 | NR | NR | | |
| 13 | NT | NT | | |
| 14 | 2.1 | 0.21 | -1.67 | -1.30 |
| 15 | NR | NR | | |
| 16 | 3.0 | 0.26 | -0.24 | -0.18 |
| 17 | NT | NT | | |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | NR | NR | | |
| 22 | 2.6 | 0.5 | -0.87 | -0.59 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NT | NT | | |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 2.8 | 0.56 | -0.56 | -0.36 |
| 29 | NT | NT | | |
| 30 | NT | NT | | |

Statistics*

| | | |
|--------------------------|------------|------|
| Assigned Value | 3.15 | 0.78 |
| Spike | Not Spiked | |
| Homogeneity Value | 4.00 | 0.70 |
| Robust Average | 3.15 | 0.78 |
| Median | 3.00 | 0.73 |
| Mean | 3.15 | |
| N | 7 | |
| Max. | 4 | |
| Min. | 2.1 | |
| Robust SD | 0.82 | |
| Robust CV | 26% | |

*Laboratory 7 was excluded from statistical calculation (extreme outlier).

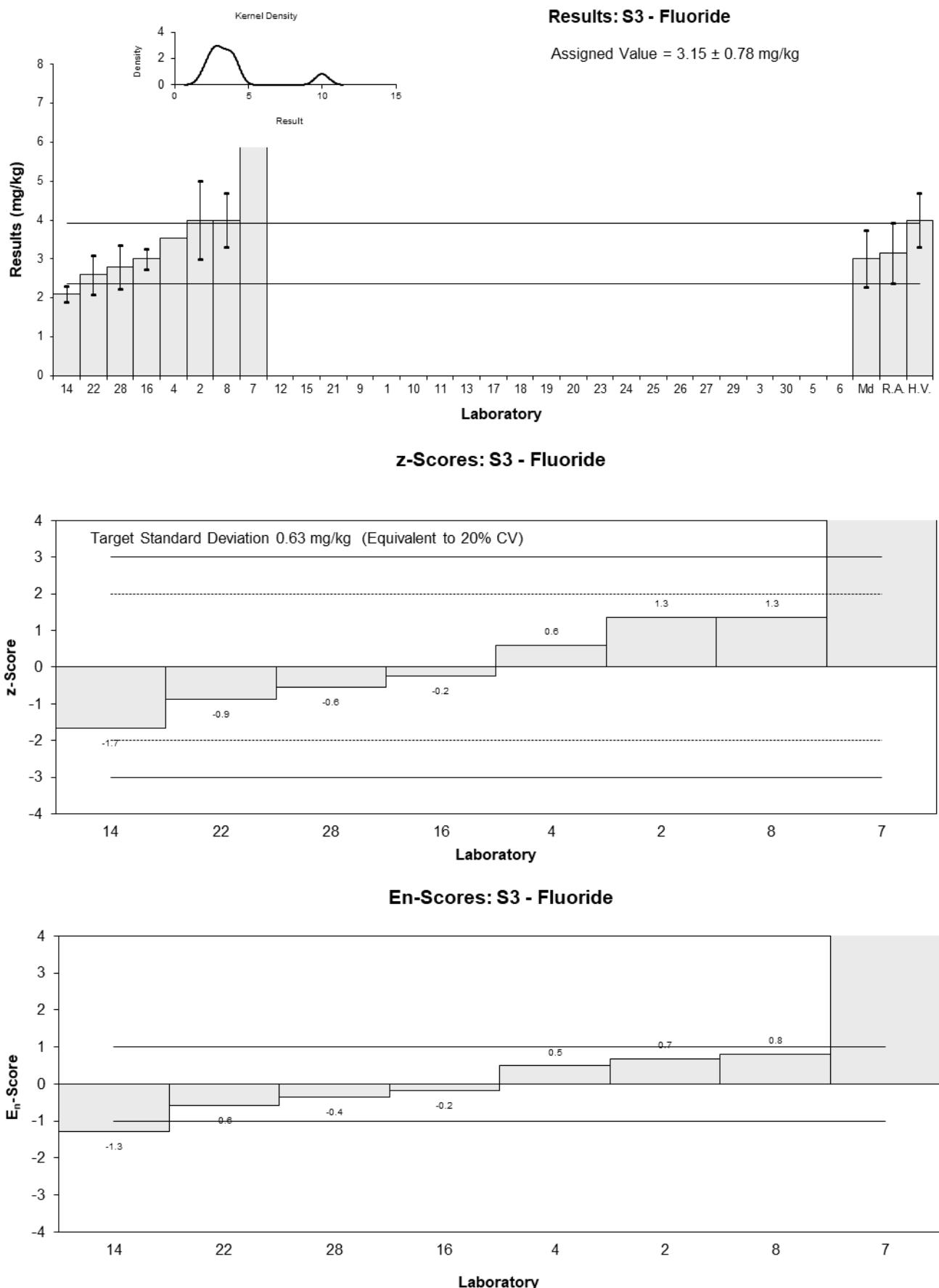


Figure 43

Table 56

Sample Details

| | |
|-------------------|--------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | Iodide |
| 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 | NT | NT |
| 7 | 0.58 | 0.09 |
| 8 | <0.25 | NR |
| 9 | NR | NR |
| 10 | NT | NT |
| 11 | NT | NT |
| 12 | NR | NR |
| 13 | NT | NT |
| 14 | <5 | NR |
| 15 | NR | NR |
| 16 | NT | NT |
| 17 | NT | NT |
| 18 | NT | NT |
| 19 | NT | NT |
| 20 | NT | NT |
| 21 | NT | NT |
| 22 | <1 | 0.2 |
| 23 | NT | NT |
| 24 | NT | NT |
| 25 | NT | NT |
| 26 | NT | NT |
| 27 | NT | NT |
| 28 | NT | NT |
| 29 | 4.00 | 0.84 |
| 30 | NT | NT |

Statistics*

| | | |
|-----------------------|------------|--|
| Assigned Value | Not Set | |
| Spike | Not Spiked | |
| Mean | 2.29 | |
| N | 2 | |
| Max. | 4 | |
| Min. | 0.58 | |

*Insufficient data to calculate statistics.

Results: S3 - Iodide

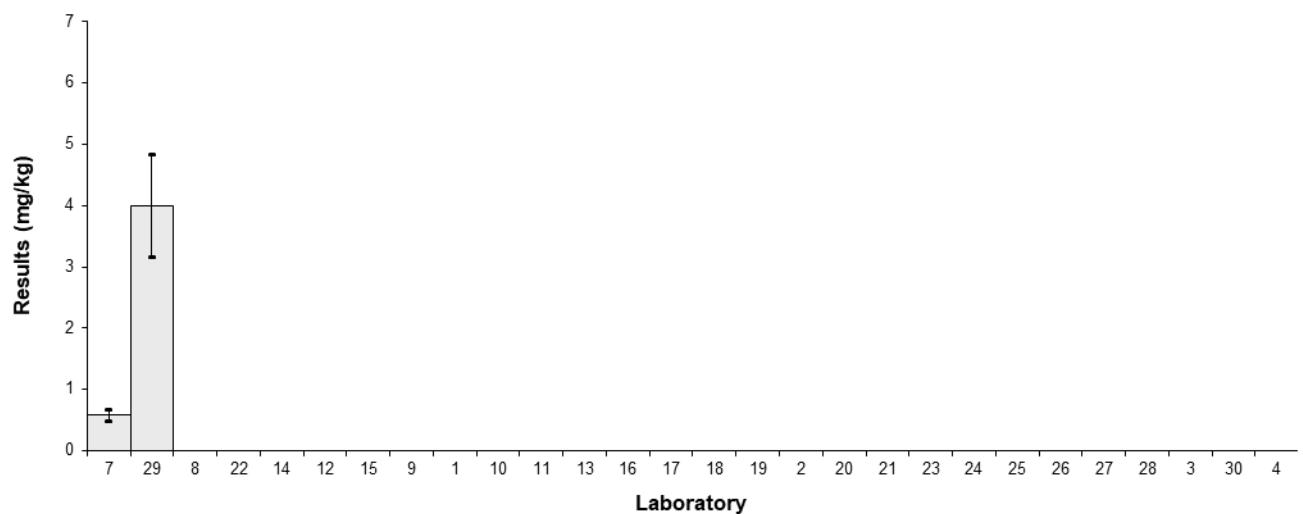


Figure 44

Table 57

Sample Details

| | |
|-------------------|-------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | K |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E _n -Score |
|----------|--------|-------------|---------|-----------------------|
| 1 | 190 | 28.5 | -1.22 | -1.37 |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | 302.3 | 40.6 | 1.02 | 0.97 |
| 5 | NT | NT | | |
| 6 | 248 | 25 | -0.06 | -0.07 |
| 7 | 250 | 40 | -0.02 | -0.02 |
| 8 | 200 | 24 | -1.02 | -1.23 |
| 9 | NR | NR | | |
| 10 | 190 | 46 | -1.22 | -1.07 |
| 11 | NT | NT | | |
| 12 | 270 | 27 | 0.38 | 0.44 |
| 13 | 260 | 120 | 0.18 | 0.07 |
| 14 | NR | NR | | |
| 15 | 270 | 47 | 0.38 | 0.33 |
| 16 | 412 | 20.2 | 3.21 | 4.07 |
| 17 | 284 | 85 | 0.66 | 0.36 |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | 179 | NR | -1.43 | -2.12 |
| 22 | 260 | 52 | 0.18 | 0.14 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 368 | 88 | 2.33 | 1.24 |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 280 | 5.6 | 0.58 | 0.84 |
| 29 | NT | NT | | |
| 30 | NT | NT | | |

Statistics

| | | |
|--------------------------|------------|----|
| Assigned Value* | 251 | 34 |
| Spike | Not Spiked | |
| Homogeneity Value | 223 | 27 |
| Robust Average | 259 | 38 |
| Median | 260 | 20 |
| Mean | 264 | |
| N | 15 | |
| Max. | 412 | |
| Min. | 179 | |
| Robust SD | 59 | |
| Robust CV | 23% | |

*Robust Average excluding laboratory 16.

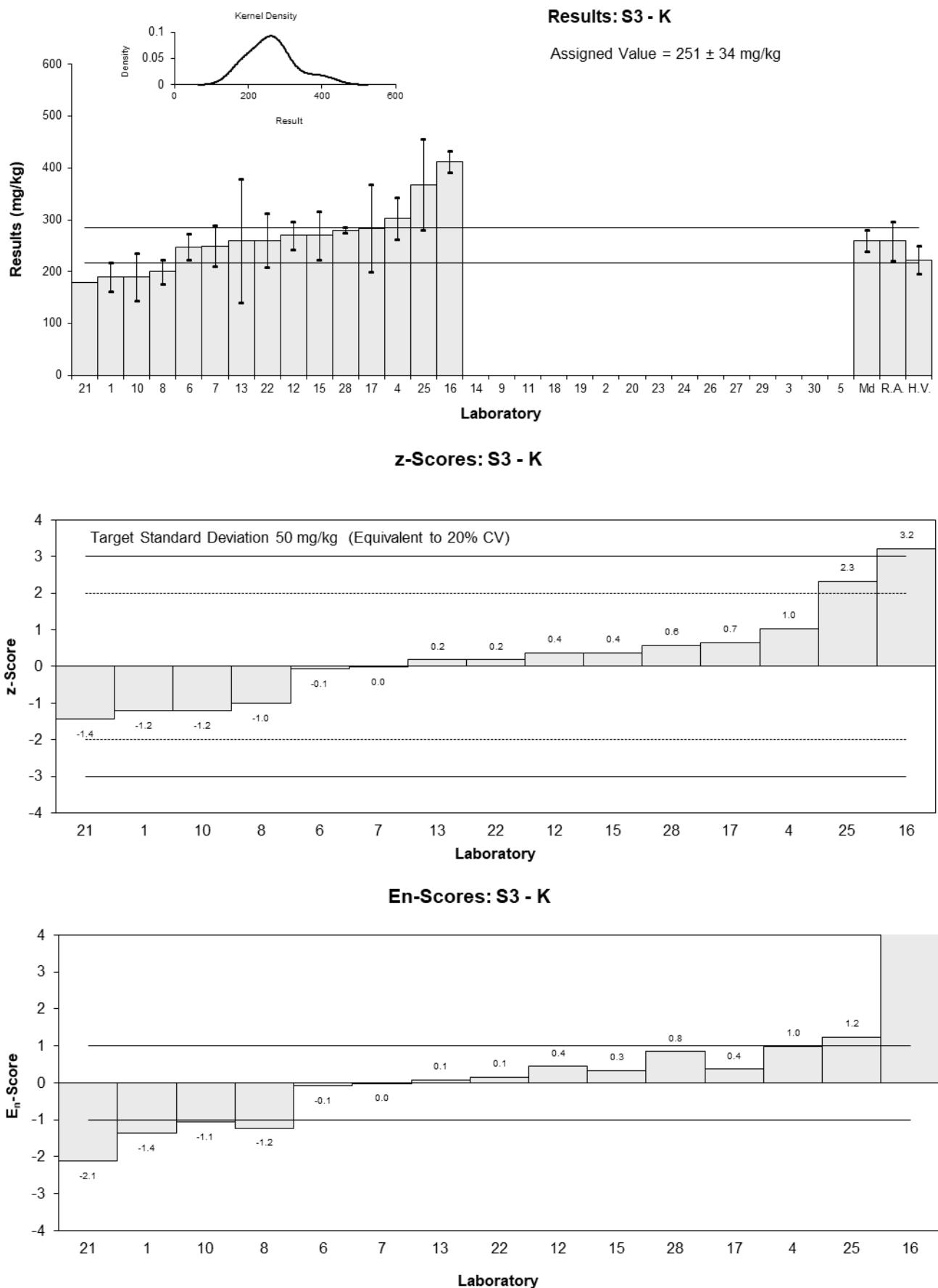


Figure 45

Table 58

Sample Details

| | |
|-------------------|-------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | Mg |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 300 | 49.5 | -1.95 | -1.82 |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | 406 | 50.2 | -0.28 | -0.26 |
| 5 | NT | NT | | |
| 6 | 478 | 50 | 0.85 | 0.79 |
| 7 | 450 | 80 | 0.41 | 0.28 |
| 8 | 360 | 51 | -1.01 | -0.92 |
| 9 | NR | NR | | |
| 10 | 320 | 80 | -1.64 | -1.12 |
| 11 | NT | NT | | |
| 12 | 350 | 35 | -1.16 | -1.26 |
| 13 | 470 | 80 | 0.72 | 0.50 |
| 14 | 403 | 28 | -0.33 | -0.38 |
| 15 | 440 | 60 | 0.25 | 0.21 |
| 16 | 553 | 31.5 | 2.03 | 2.28 |
| 17 | 521 | 200 | 1.53 | 0.47 |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | 389 | 56 | -0.55 | -0.48 |
| 22 | 430 | 86 | 0.09 | 0.06 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 473 | 113 | 0.77 | 0.40 |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 450 | 9 | 0.41 | 0.54 |
| 29 | NT | NT | | |
| 30 | NT | NT | | |

Statistics

| | | |
|--------------------------|------------|----|
| Assigned Value | 424 | 47 |
| Spike | Not Spiked | |
| Homogeneity Value | 418 | 50 |
| Robust Average | 424 | 47 |
| Median | 435 | 32 |
| Mean | 425 | |
| N | 16 | |
| Max. | 553 | |
| Min. | 300 | |
| Robust SD | 76 | |
| Robust CV | 18% | |

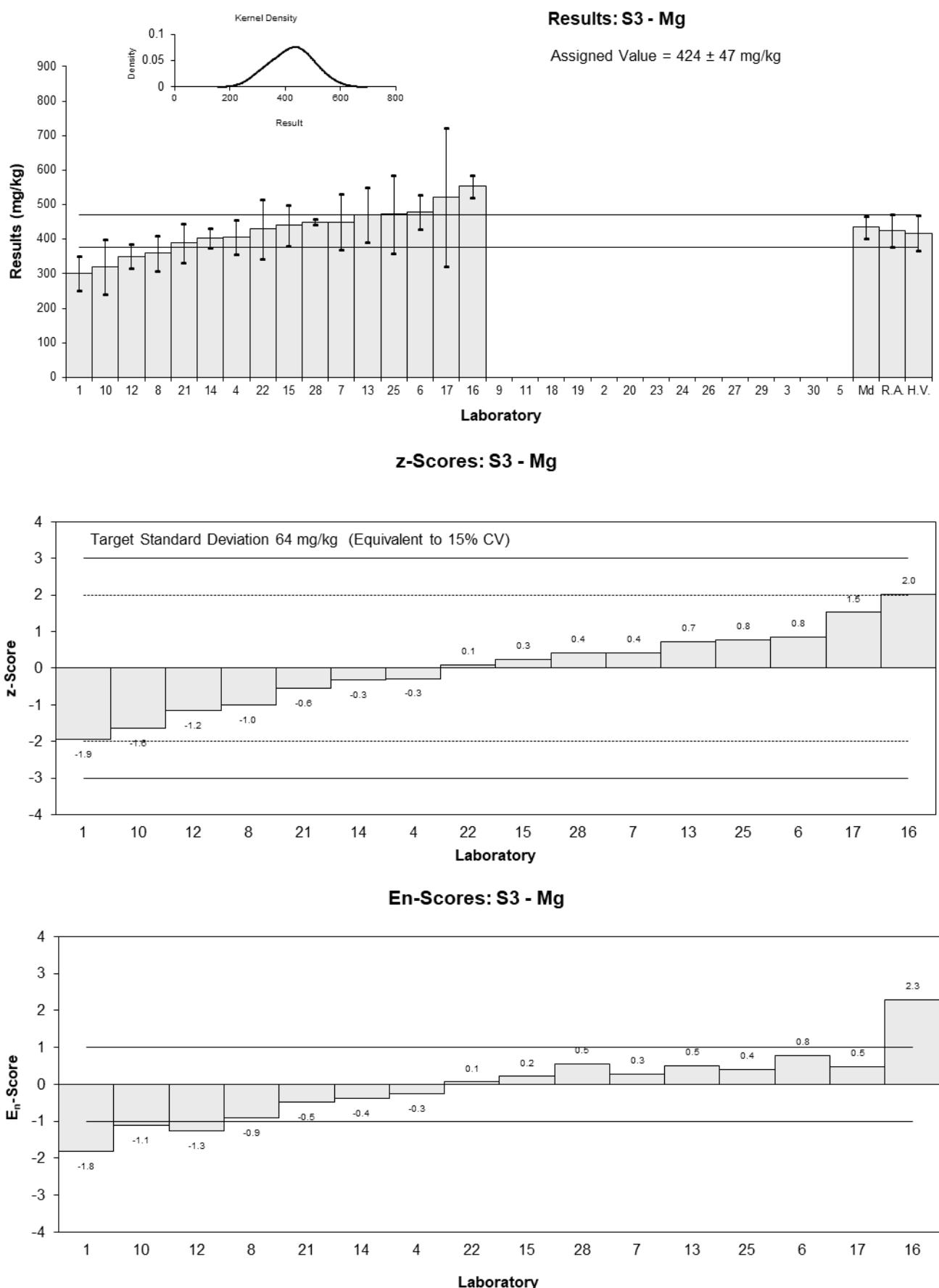


Figure 46

Table 59

Sample Details

| | |
|-------------------|-------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | Na |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 280 | 42 | 6.57 | 2.57 |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | 234.9 | 29.2 | 3.90 | 2.14 |
| 5 | NT | NT | | |
| 6 | 174 | 20 | 0.30 | 0.22 |
| 7 | 180 | 20 | 0.65 | 0.49 |
| 8 | 180 | 27 | 0.65 | 0.38 |
| 9 | NR | NR | | |
| 10 | 150 | 24 | -1.12 | -0.73 |
| 11 | NT | NT | | |
| 12 | 195 | 20 | 1.54 | 1.16 |
| 13 | 160 | 40 | -0.53 | -0.22 |
| 14 | 153 | 25 | -0.95 | -0.59 |
| 15 | 164 | 18 | -0.30 | -0.24 |
| 16 | 164 | 11 | -0.30 | -0.34 |
| 17 | 180 | 50 | 0.65 | 0.22 |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | 167.6 | 26.574 | -0.08 | -0.05 |
| 21 | 174 | 23 | 0.30 | 0.20 |
| 22 | 150 | 30 | -1.12 | -0.60 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 148 | 35.3 | -1.24 | -0.57 |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 170 | 3.4 | 0.06 | 0.09 |
| 29 | NT | NT | | |
| 30 | NT | NT | | |

Statistics

| | | |
|--------------------------|------------|----|
| Assigned Value* | 169 | 10 |
| Spike | Not Spiked | |
| Homogeneity Value | 159 | 19 |
| Robust Average | 171 | 11 |
| Median | 170 | 8 |
| Mean | 178 | |
| N | 17 | |
| Max. | 280 | |
| Min. | 148 | |
| Robust SD | 19 | |
| Robust CV | 11% | |

*Robust Average excluding laboratory 1.

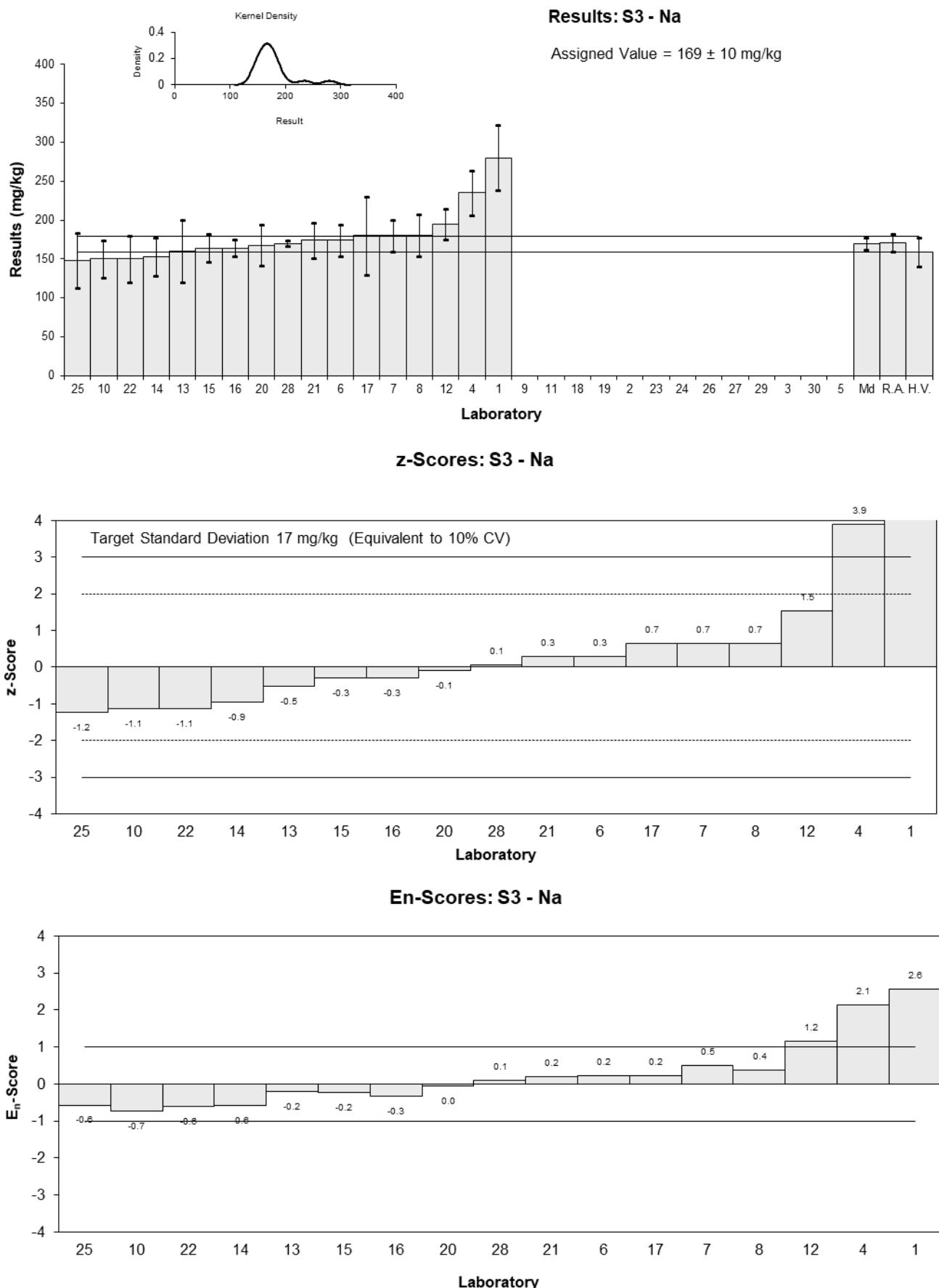


Figure 47

Table 60

Sample Details

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

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E _n -Score |
|----------|--------|-------------|---------|-----------------------|
| 1 | 340 | 51 | 0.86 | 0.45 |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | NT | NT | | |
| 5 | NT | NT | | |
| 6 | 364 | 40 | 1.63 | 1.00 |
| 7 | NT | NT | | |
| 8 | NT | NT | | |
| 9 | 268.4 | NR | -1.42 | -1.39 |
| 10 | NT | NT | | |
| 11 | NT | NT | | |
| 12 | NR | NR | | |
| 13 | NT | NT | | |
| 14 | 305 | 30 | -0.26 | -0.18 |
| 15 | 301.61 | 45.24 | -0.36 | -0.21 |
| 16 | 280 | 25.8 | -1.05 | -0.80 |
| 17 | 306 | 92 | -0.22 | -0.07 |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | NT | NT | | |
| 22 | 28 | 5.6 | -9.11 | -8.77 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NT | NT | | |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 336 | 6.72 | 0.73 | 0.70 |
| 29 | NT | NT | | |
| 30 | NT | NT | | |

Statistics*

| | | |
|-----------------------|------------|----|
| Assigned Value | 313 | 32 |
| Spike | Not Spiked | |
| Robust Average | 313 | 32 |
| Median | 306 | 35 |
| Mean | 313 | |
| N | 8 | |
| Max. | 364 | |
| Min. | 268.4 | |
| Robust SD | 36 | |
| Robust CV | 12% | |

Laboratory 22 was excluded from statistical calculation (extreme outlier).

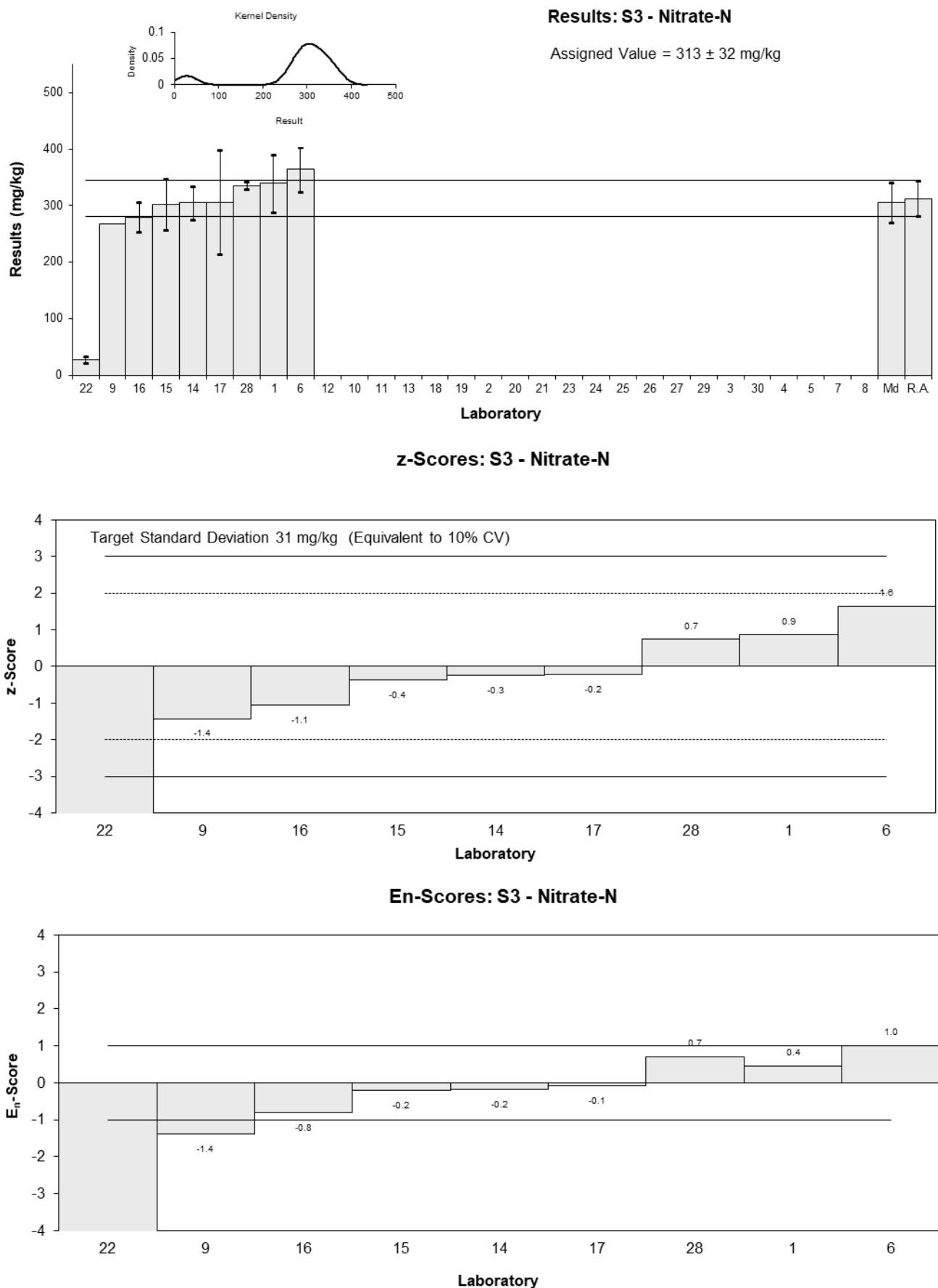


Figure 48

Table 61

Sample Details

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

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E _n -Score |
|----------|--------|-------------|---------|-----------------------|
| 1 | NT | NT | | |
| 2 | 4.8 | 0.2 | 0.64 | 0.79 |
| 3 | NT | NT | | |
| 4 | 4.46 | 0.6 | 0.12 | 0.10 |
| 5 | NT | NT | | |
| 6 | 5.5 | 0.6 | 1.70 | 1.45 |
| 7 | 4.7 | 0.5 | 0.49 | 0.46 |
| 8 | 4.8 | 0.4 | 0.64 | 0.66 |
| 9 | NR | NR | | |
| 10 | NT | NT | | |
| 11 | NT | NT | | |
| 12 | NR | NR | | |
| 13 | NT | NT | | |
| 14 | 4.3 | 0.4 | -0.12 | -0.13 |
| 15 | NR | NR | | |
| 16 | 4.28 | 0.36 | -0.15 | -0.16 |
| 17 | 3.52 | 1.5 | -1.31 | -0.54 |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | 4.6 | NR | 0.33 | 0.45 |
| 22 | 3.8 | 0.8 | -0.88 | -0.62 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NT | NT | | |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 7.2 | 1.44 | 4.29 | 1.85 |
| 29 | 3.52 | 0.37 | -1.31 | -1.40 |
| 30 | NT | NT | | |

Statistics

| | | |
|--------------------------|------------|------|
| Assigned Value* | 4.38 | 0.49 |
| Spike | Not Spiked | |
| Homogeneity Value | 4.40 | 0.66 |
| Robust Average | 4.49 | 0.55 |
| Median | 4.53 | 0.25 |
| Mean | 4.62 | |
| N | 12 | |
| Max. | 7.2 | |
| Min. | 3.52 | |
| Robust SD | 0.77 | |
| Robust CV | 17% | |

*Robust Average excluding laboratory 28.

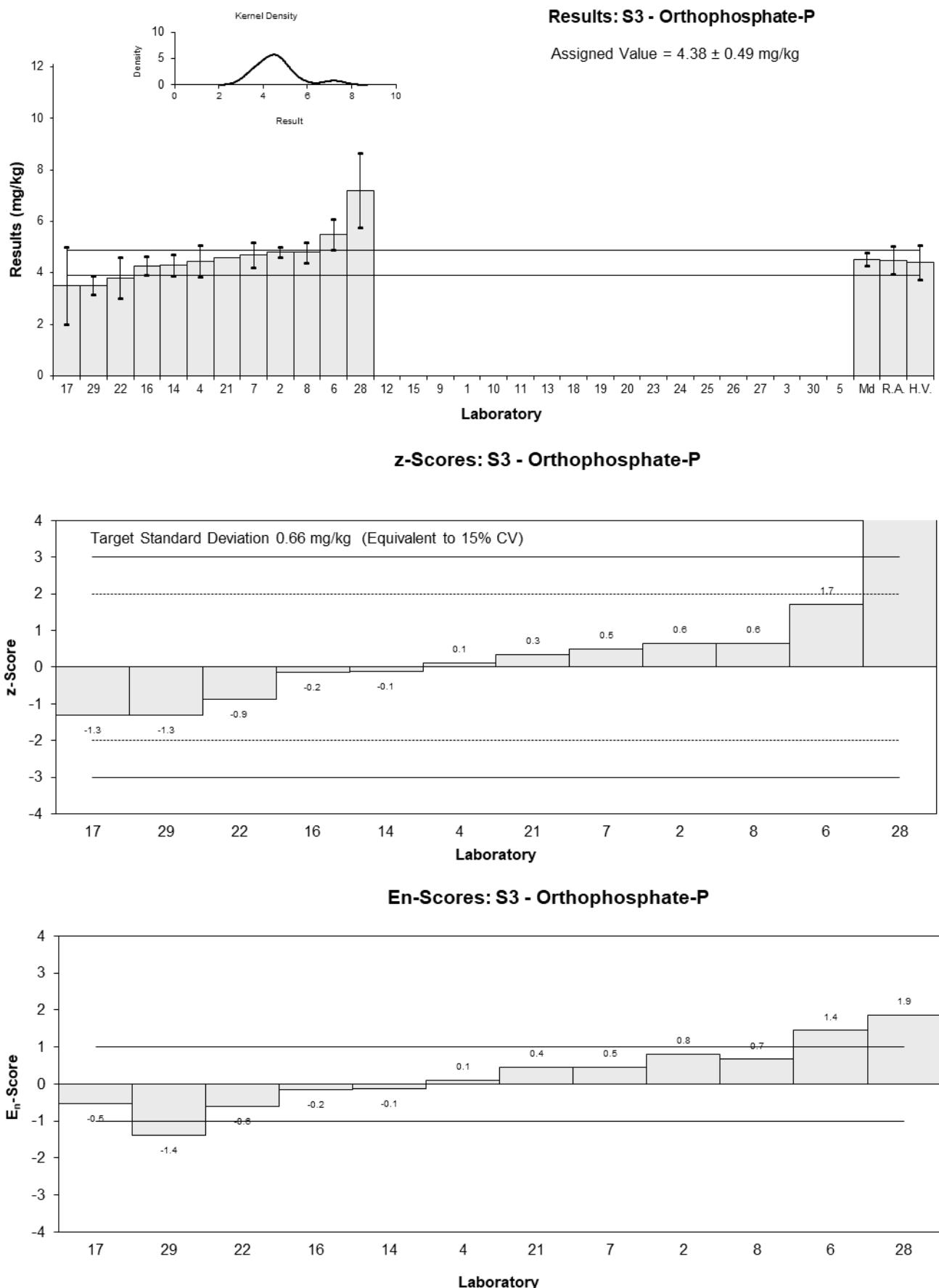


Figure 49

Table 62

Sample Details

| | |
|-------------------|-------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | P |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 720 | 108 | -0.19 | -0.12 |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | 743.9 | NR | 0.13 | 0.23 |
| 5 | NT | NT | | |
| 6 | 800 | 80 | 0.90 | 0.73 |
| 7 | 840 | NR | 1.44 | 2.47 |
| 8 | 690 | 165 | -0.60 | -0.26 |
| 9 | NR | NR | | |
| 10 | 730 | 110 | -0.05 | -0.03 |
| 11 | NT | NT | | |
| 12 | NR | NR | | |
| 13 | 710 | 100 | -0.33 | -0.22 |
| 14 | 660 | 86 | -1.01 | -0.77 |
| 15 | 707 | 160 | -0.37 | -0.16 |
| 16 | 742 | 59.4 | 0.11 | 0.11 |
| 17 | 927 | 275 | 2.63 | 0.69 |
| 18 | 783 | 121 | 0.67 | 0.38 |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | 770 | NR | 0.49 | 0.84 |
| 22 | 630 | 126 | -1.42 | -0.78 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 622 | 149 | -1.53 | -0.72 |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 750 | 15 | 0.22 | 0.35 |
| 29 | 730 | 150 | -0.05 | -0.03 |
| 30 | NT | NT | | |

Statistics

| | | |
|--------------------------|------------|----|
| Assigned Value | 734 | 43 |
| Spike | Not Spiked | |
| Homogeneity Value | 800 | 90 |
| Robust Average | 734 | 43 |
| Median | 730 | 30 |
| Mean | 739 | |
| N | 17 | |
| Max. | 927 | |
| Min. | 622 | |
| Robust SD | 70 | |
| Robust CV | 9.6% | |

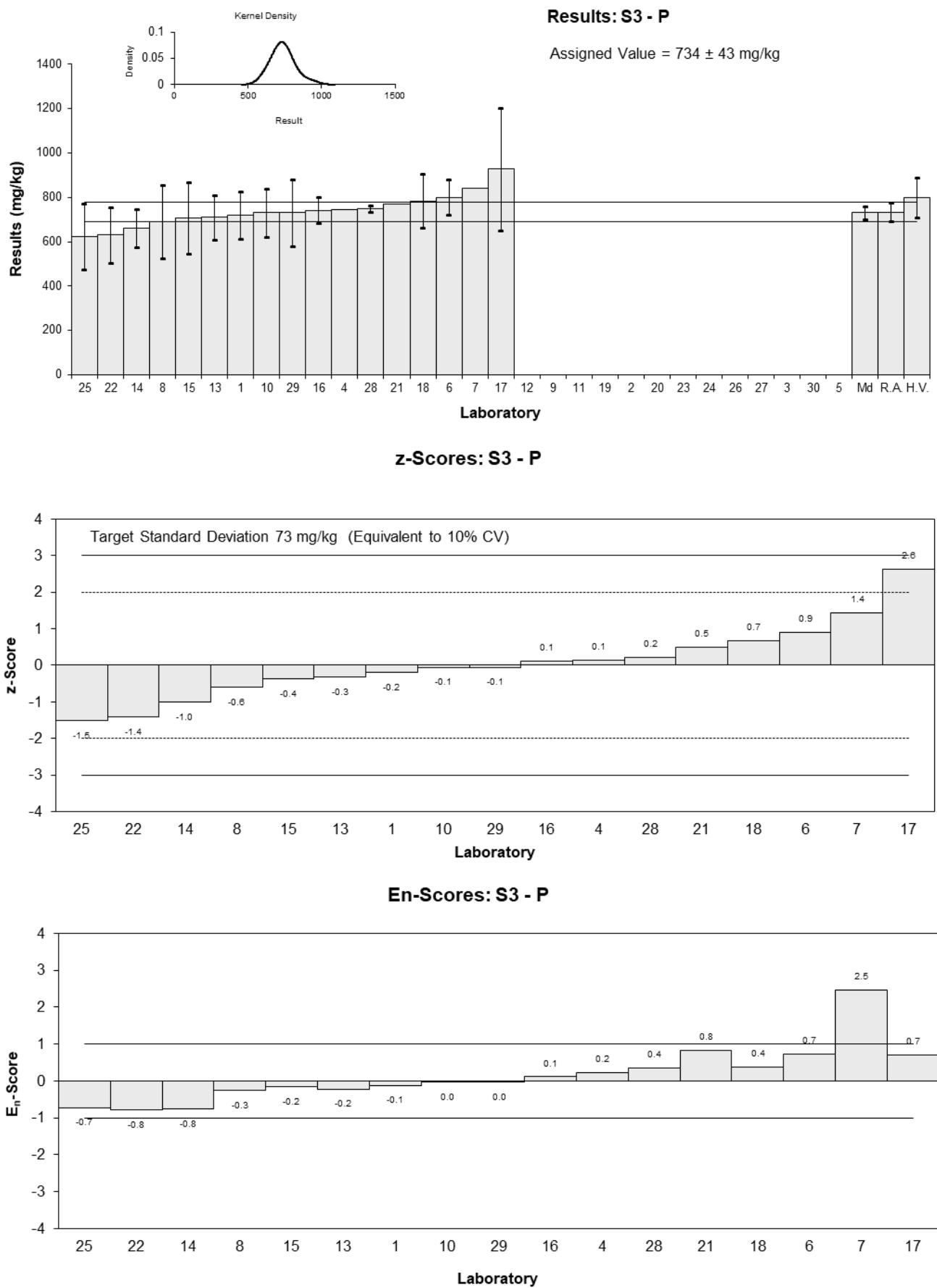


Figure 50

Table 63

Sample Details

| | |
|-------------------|-------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | pH |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 4.5 | 0.675 | 1.40 | 0.31 |
| 2 | 4.2 | 0.04 | -0.60 | -1.41 |
| 3 | NT | NT | | |
| 4 | 4.1 | 0.07 | -1.27 | -2.21 |
| 5 | 4.3 | 0.215 | 0.07 | 0.05 |
| 6 | 4.34 | 0.2 | 0.33 | 0.24 |
| 7 | 4.3 | 0.12 | 0.07 | 0.08 |
| 8 | 4.2 | 0.1 | -0.60 | -0.80 |
| 9 | 4.23 | 0.10 | -0.40 | -0.54 |
| 10 | 4.4 | 0.22 | 0.73 | 0.49 |
| 11 | 4.5 | 0.1 | 1.40 | 1.88 |
| 12 | 4.2 | 0.1 | -0.60 | -0.80 |
| 13 | 4.3 | 0.1 | 0.07 | 0.09 |
| 14 | 4.3 | 0.4 | 0.07 | 0.02 |
| 15 | NR | NR | | |
| 16 | 4.30 | 0.1 | 0.07 | 0.09 |
| 17 | 4.22 | NR | -0.47 | -1.40 |
| 18 | 4.31 | 0.04 | 0.13 | 0.31 |
| 19 | NT | NT | | |
| 20 | 4.4 | 0.1 | 0.73 | 0.98 |
| 21 | 4.27 | 0.08 | -0.13 | -0.21 |
| 22 | 4.3 | 0.2 | 0.07 | 0.05 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NR | NR | | |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 4.2 | 0.1 | -0.60 | -0.80 |
| 29 | 4.36 | 0.2 | 0.47 | 0.34 |
| 30 | NT | NT | | |

Statistics

| | | |
|-----------------------|------------|------|
| Assigned Value | 4.29 | 0.05 |
| Spike | Not Spiked | |
| Robust Average | 4.29 | 0.05 |
| Median | 4.30 | 0.05 |
| Mean | 4.30 | |
| N | 21 | |
| Max. | 4.5 | |
| Min. | 4.1 | |
| Robust SD | 0.092 | |
| Robust CV | 2.1% | |

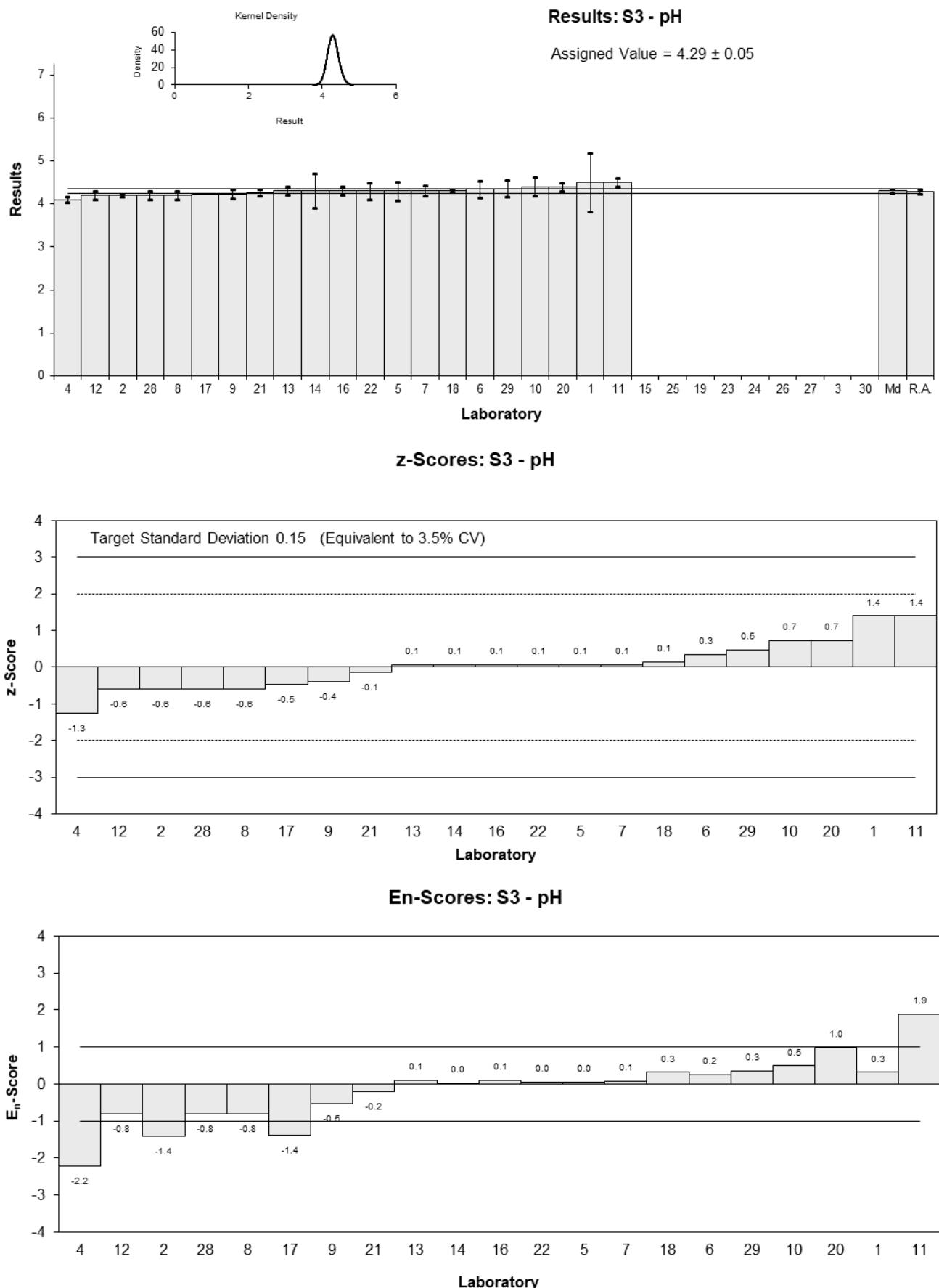


Figure 51

Table 64

Sample Details

| | |
|-------------------|-------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | S |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 560 | 84 | -0.60 | -0.40 |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | 622 | 67.8 | 0.44 | 0.35 |
| 5 | NT | NT | | |
| 6 | 640 | 64 | 0.74 | 0.61 |
| 7 | 580 | NR | -0.27 | -0.50 |
| 8 | 510 | 163 | -1.44 | -0.52 |
| 9 | NR | NR | | |
| 10 | 570 | 114 | -0.44 | -0.22 |
| 11 | NT | NT | | |
| 12 | NR | NR | | |
| 13 | 580 | 40 | -0.27 | -0.31 |
| 14 | 564 | 56 | -0.54 | -0.50 |
| 15 | 654 | 90 | 0.97 | 0.61 |
| 16 | NT | NT | | |
| 17 | 715 | 100 | 2.00 | 1.13 |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | NT | NT | | |
| 22 | 620 | 124 | 0.40 | 0.19 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 580 | 138 | -0.27 | -0.11 |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 590 | 11.8 | -0.10 | -0.18 |
| 29 | NT | NT | | |
| 30 | NT | NT | | |

Statistics

| | | |
|-----------------------|------------|----|
| Assigned Value | 596 | 32 |
| Spike | Not Spiked | |
| Robust Average | 596 | 32 |
| Median | 580 | 18 |
| Mean | 599 | |
| N | 13 | |
| Max. | 715 | |
| Min. | 510 | |
| Robust SD | 46 | |
| Robust CV | 7.8% | |

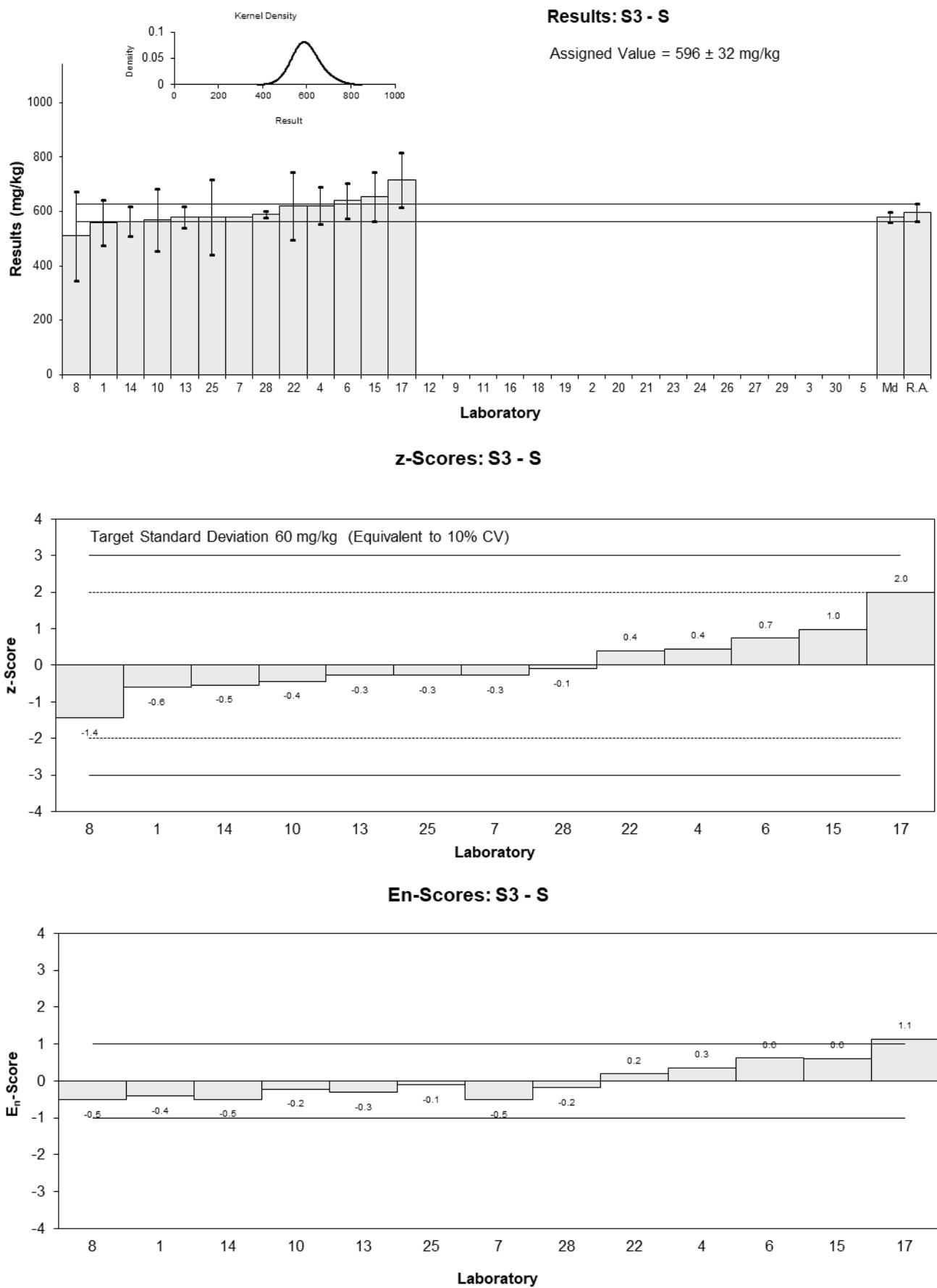


Figure 52

Table 65

Sample Details

| | |
|-------------------|-------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | Sr |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 10 | 1.5 | -1.38 | -0.94 |
| 2 | NT | NT | | |
| 3 | NT | NT | | |
| 4 | 11.12 | 0.93 | -0.41 | -0.39 |
| 5 | 13 | 2.6 | 1.21 | 0.51 |
| 6 | 10.8 | 1.2 | -0.69 | -0.55 |
| 7 | 10.1 | NR | -1.29 | -1.88 |
| 8 | 9 | 1 | -2.24 | -2.03 |
| 9 | NR | NR | | |
| 10 | 10 | 1.8 | -1.38 | -0.81 |
| 11 | NT | NT | | |
| 12 | NR | NR | | |
| 13 | 12 | 6 | 0.34 | 0.07 |
| 14 | 10.5 | 0.72 | -0.95 | -1.02 |
| 15 | 11.8 | 1.9 | 0.17 | 0.10 |
| 16 | 12.4 | 1.41 | 0.69 | 0.49 |
| 17 | 13.0 | 2.5 | 1.21 | 0.53 |
| 18 | 11.4 | 1.5 | -0.17 | -0.12 |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | 12.6 | 1.8 | 0.86 | 0.51 |
| 22 | 11 | 2.2 | -0.52 | -0.26 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 12.8 | 3.1 | 1.03 | 0.37 |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 13 | 0.26 | 1.21 | 1.66 |
| 29 | 13 | 2.1 | 1.21 | 0.62 |
| 30 | NT | NT | | |

Statistics

| | | |
|--------------------------|------------|-----|
| Assigned Value | 11.6 | 0.8 |
| Spike | Not Spiked | |
| Homogeneity Value | 10.0 | 2.0 |
| Robust Average | 11.6 | 0.8 |
| Median | 11.6 | 0.8 |
| Mean | 11.5 | |
| N | 18 | |
| Max. | 13 | |
| Min. | 9 | |
| Robust SD | 1.4 | |
| Robust CV | 12% | |

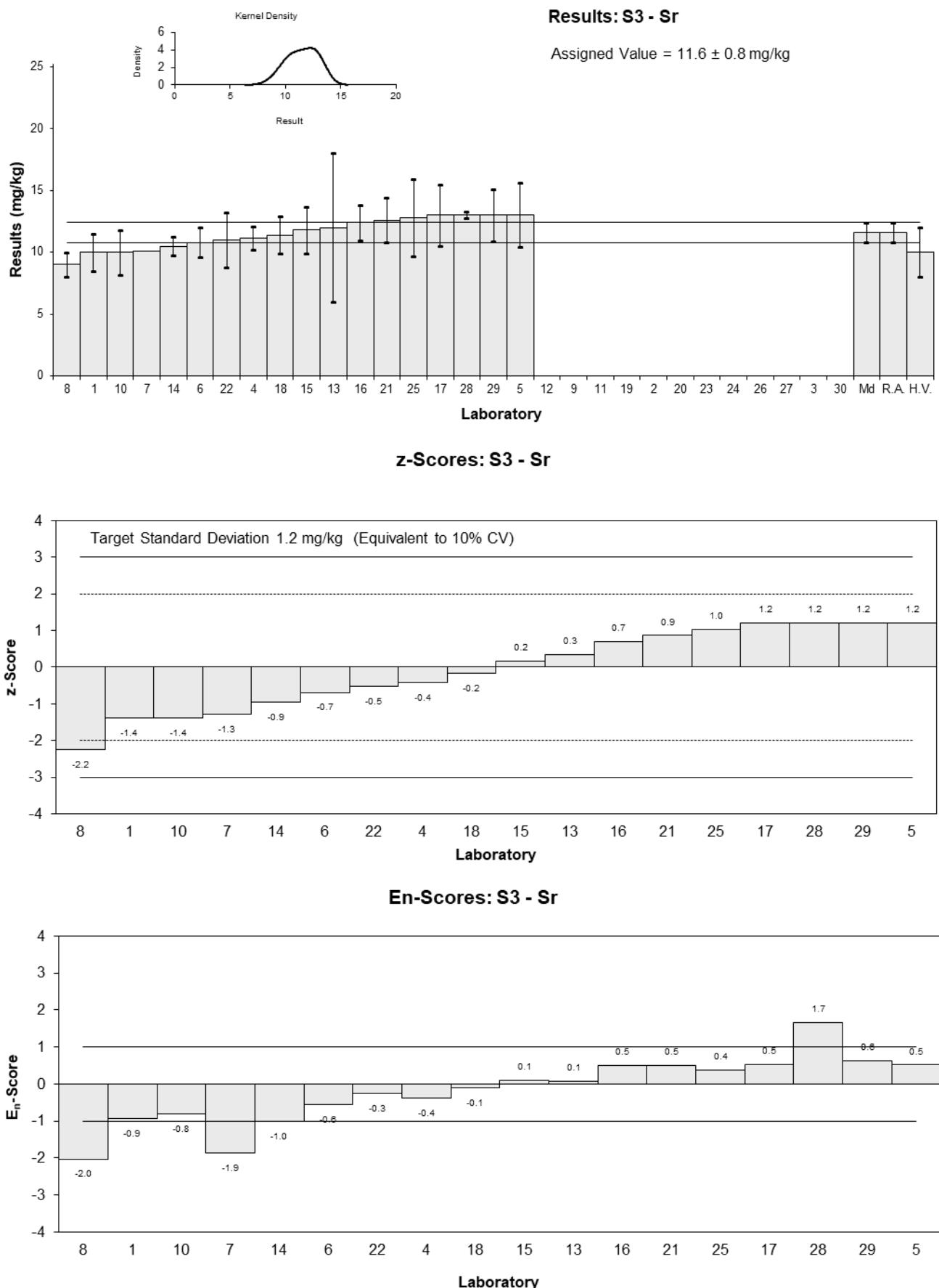


Figure 53

Table 66

Sample Details

| | |
|-------------------|----------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | Sulphate |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 720 | 108 | 0.34 | 0.28 |
| 2 | 710 | 8 | 0.24 | 0.38 |
| 3 | NT | NT | | |
| 4 | 750 | 90.3 | 0.63 | 0.58 |
| 5 | NT | NT | | |
| 6 | 763 | 80 | 0.76 | 0.75 |
| 7 | 666 | 118 | -0.18 | -0.14 |
| 8 | 740 | 43 | 0.54 | 0.70 |
| 9 | NR | NR | | |
| 10 | NT | NT | | |
| 11 | NT | NT | | |
| 12 | 811 | 81 | 1.23 | 1.21 |
| 13 | 540 | 40 | -1.41 | -1.88 |
| 14 | 650 | 65 | -0.34 | -0.38 |
| 15 | NR | NR | | |
| 16 | 454 | 74 | -2.25 | -2.33 |
| 17 | 715 | 280 | 0.29 | 0.10 |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | 652 | 190 | -0.32 | -0.16 |
| 22 | 620 | 120 | -0.63 | -0.47 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | NT | NT | | |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 825 | 165 | 1.36 | 0.79 |
| 29 | 577.67 | 61.97 | -1.04 | -1.19 |
| 30 | NT | NT | | |

Statistics

| | | |
|--------------------------|------------|----|
| Assigned Value | 685 | 66 |
| Spike | Not Spiked | |
| Homogeneity Value | 645 | 97 |
| Robust Average | 685 | 66 |
| Median | 710 | 48 |
| Mean | 680 | |
| N | 15 | |
| Max. | 825 | |
| Min. | 454 | |
| Robust SD | 100 | |
| Robust CV | 15% | |

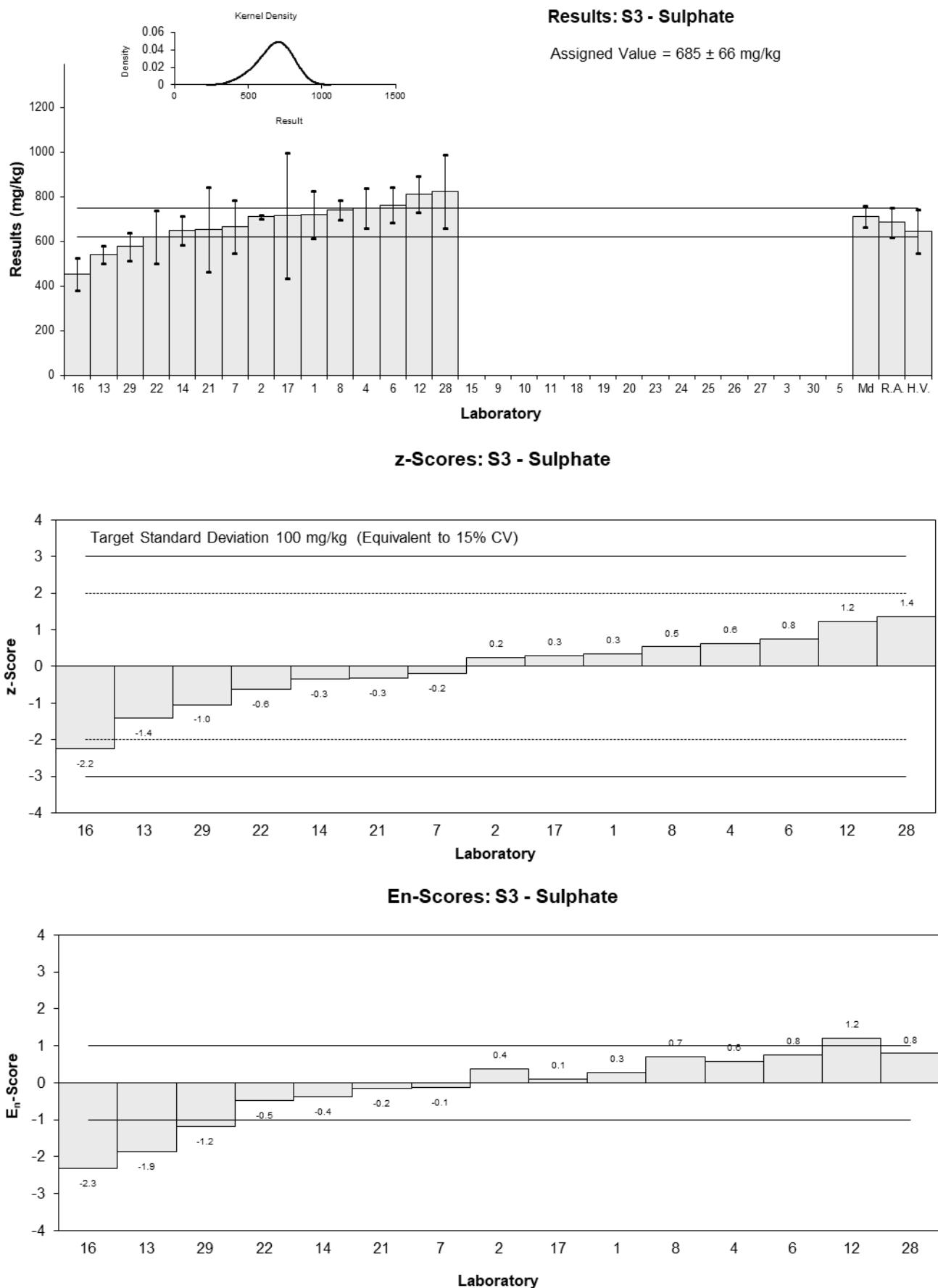


Figure 54

Table 67

Sample Details

| | |
|-------------------|-------|
| Sample No. | S3 |
| Matrix. | Soil |
| Analyte. | TKN |
| Units | mg/kg |

Participant Results

| Lab Code | Result | Uncertainty | z-Score | E_n-Score |
|-----------------|---------------|--------------------|----------------|----------------------------|
| 1 | 2400 | 360 | -0.32 | -0.22 |
| 2 | 2660 | 532 | 0.73 | 0.33 |
| 3 | NT | NT | | |
| 4 | 2480 | 619 | 0.00 | 0.00 |
| 5 | NT | NT | | |
| 6 | 2310 | 230 | -0.69 | -0.70 |
| 7 | 2530 | 480 | 0.20 | 0.10 |
| 8 | 2460 | 436 | -0.08 | -0.05 |
| 9 | NR | NR | | |
| 10 | 3100 | 170 | 2.50 | 3.30 |
| 11 | NT | NT | | |
| 12 | 2490 | 220 | 0.04 | 0.04 |
| 13 | NT | NT | | |
| 14 | 2350 | 630 | -0.52 | -0.20 |
| 15 | 2495.52 | 374.33 | 0.06 | 0.04 |
| 16 | 2300 | 529 | -0.73 | -0.34 |
| 17 | 2564 | 250 | 0.34 | 0.32 |
| 18 | NT | NT | | |
| 19 | NT | NT | | |
| 20 | NT | NT | | |
| 21 | NT | NT | | |
| 22 | 2500 | 380 | 0.08 | 0.05 |
| 23 | NT | NT | | |
| 24 | NT | NT | | |
| 25 | 2490 | 227 | 0.04 | 0.04 |
| 26 | NT | NT | | |
| 27 | NT | NT | | |
| 28 | 2498 | 50 | 0.07 | 0.19 |
| 29 | NT | NT | | |
| 30 | NT | NT | | |

Statistics

| | | |
|--------------------------|------------|-----|
| Assigned Value | 2480 | 80 |
| Spike | Not Spiked | |
| Homogeneity Value | 2600 | 390 |
| Robust Average | 2480 | 80 |
| Median | 2490 | 30 |
| Mean | 2510 | |
| N | 15 | |
| Max. | 3100 | |
| Min. | 2300 | |
| Robust SD | 120 | |
| Robust CV | 4.9% | |

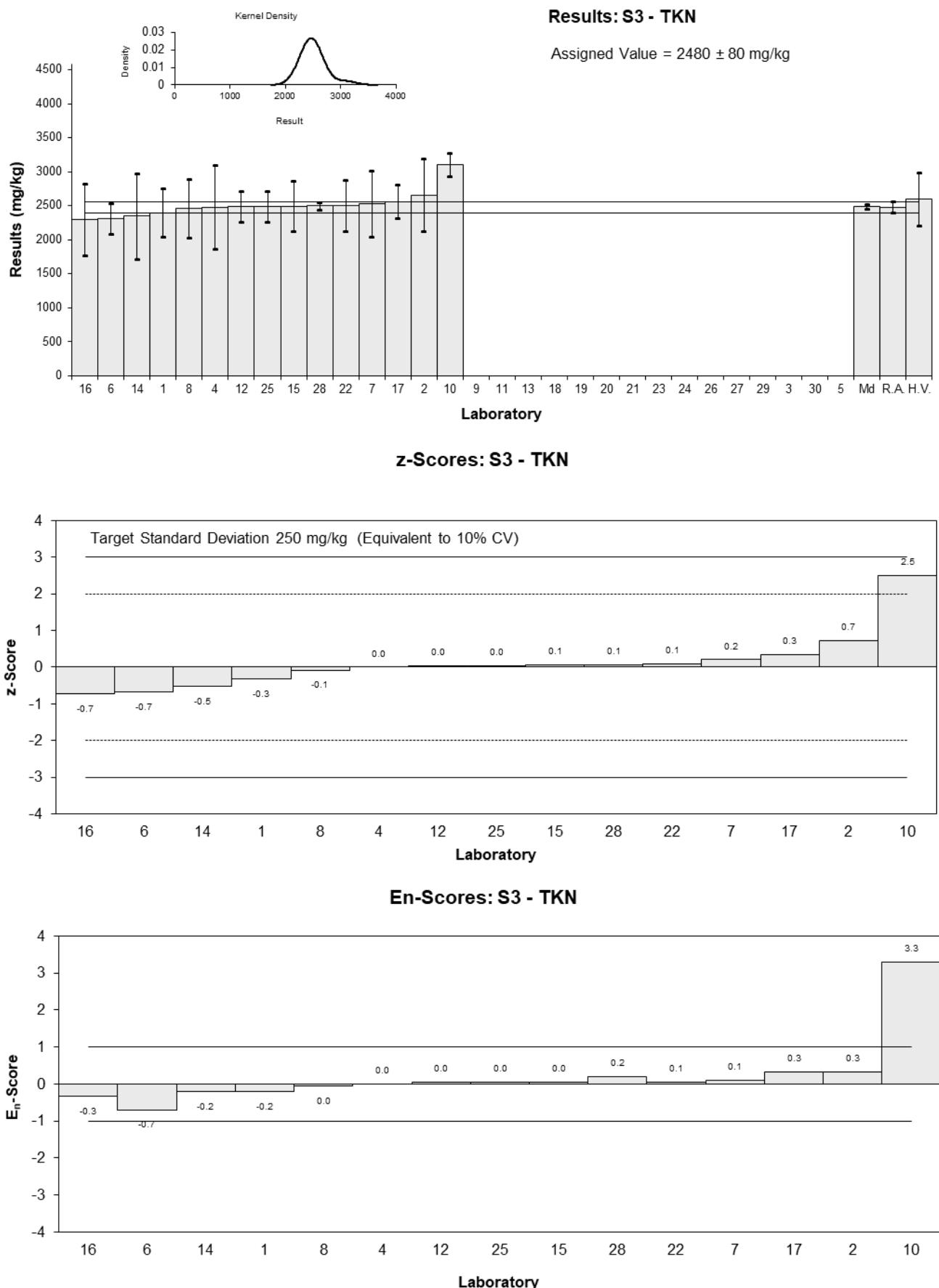


Figure 55

7 DISCUSSION OF RESULTS

7.1 Assigned Value and Traceability

Assigned Values were the robust average of participants' results. The robust averages used as assigned values and their associated expanded uncertainties were calculated using the procedure described in ISO13528:2015(E) 'Statistical methods for use in proficiency testing by interlaboratory comparisons'. Results less than 50% and more than 150% of the robust average were investigated and then removed before calculation of the assigned value.⁶ Appendix 2 sets out the calculation of the robust average of As in Sample S1 and its associated uncertainty.

No assigned value was set for Sb in S1, B, Ga, Rb, Sb and Cs and Se in S2 and bromide and iodide in S3 because the reported results were either too few or too variable.

Traceability The assigned value is not traceable to any external reference; it is traceable to the consensus of participants' results deriving from a variety of measurement methods and (presumably) a variety of calibrators. So although expressed in SI units, the metrological traceability of the assigned values has not been established.

7.2 Measurement Uncertainty Reported by Participants

Participants were asked to report an estimate of the expanded measurement uncertainty associated with their results. Of 871 numerical results, 825 (95%) were reported with an expanded measurement uncertainty. The magnitude of these expanded uncertainties was within the range 0.38% to 307% of the reported value. The participants used a wide variety of procedures to estimate the expanded measurement uncertainty. These are presented in Tables 11 and 12.

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

Participation in proficiency testing programs allows participants to check how reasonable their estimates of uncertainty are. Results and the expanded MU are presented in the bar charts for each analyte (Figure 2 to 55). As a simple rule of thumb, when the uncertainty estimate is smaller than uncertainty of the assigned value, or larger than the uncertainty of the assigned value plus twice the target standard deviation, then this should be reviewed as suspect. For example, 22 laboratories reported results for Al in S1. The uncertainty of the assigned value estimated from the robust standard deviation of the 22 laboratories' results is 1000 mg/kg (see equation 4, Appendix 2). Laboratory 3 might have under-estimated its expanded measurement uncertainties reported for Al in S1 (100 mg/kg) as an uncertainty estimated from one measurement cannot be smaller than the uncertainty estimated from 22 measurements. Alternatively, estimates of uncertainties for Ag in S2 larger than 0.71 mg/kg (the uncertainty of the assigned value, 0.15 mg/kg plus the allowable variation from the assigned value, the target standard deviation of 0.28 mg/kg, multiplied by 2, the coverage factor for a confidence interval of 95%), should also be viewed as suspect. For example, the expanded measurement uncertainties reported by laboratory 13 for Ag in S2 might have been over-estimated.

Laboratory 28 should review their procedure for estimating measurement uncertainty as most of their estimated uncertainties were under-estimated.

Laboratory 13 should also review the procedure they have used for estimating measurement uncertainty as most of their estimated uncertainties were over-estimated.

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

Laboratory 26 attached estimates of the expanded measurement uncertainty to results reported as less than their limit of detection. An estimate of uncertainty expressed as a value cannot be attached to a result expressed as a range.⁹

Laboratory 27 reported an estimate of expanded uncertainty for their Tl in S2 measurement result larger than the result itself.

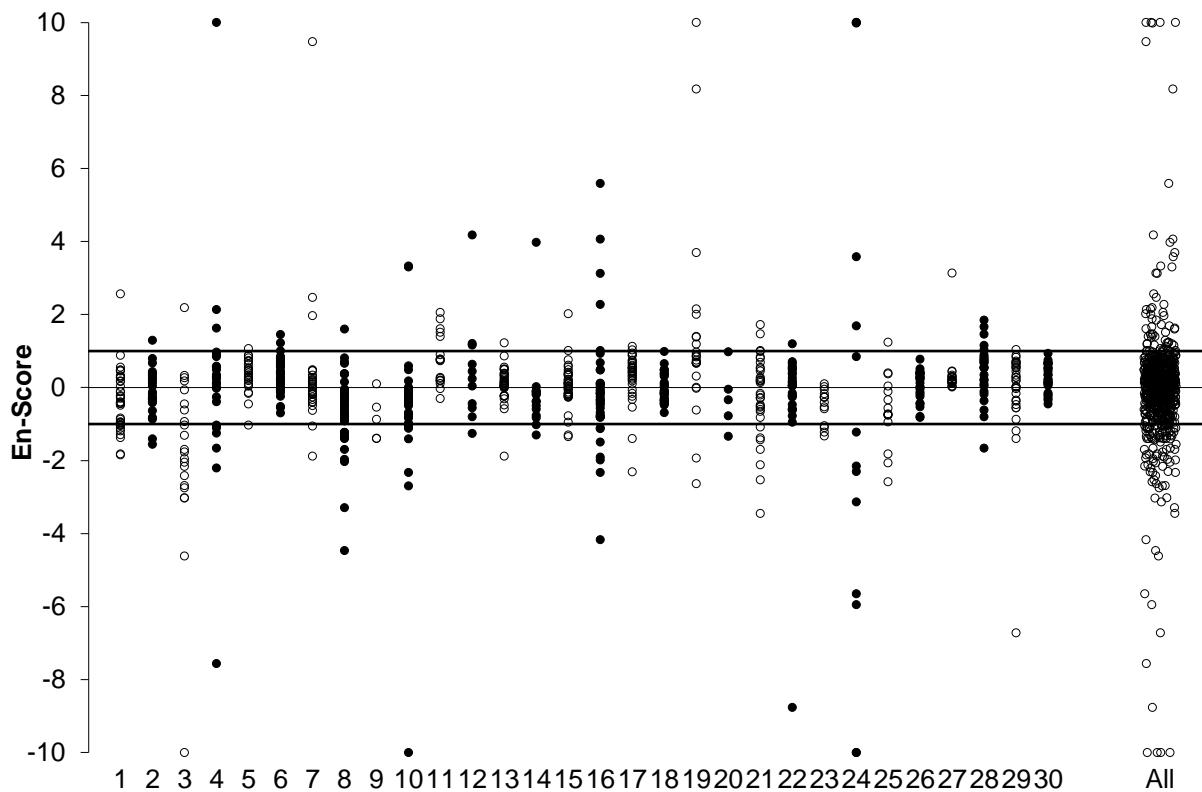
In some cases the results were reported with an inappropriate number of significant figures. The recommended format is to write uncertainty to no more than two significant figures and then to write the result with the corresponding number of decimal places. For example, instead of 2495.52 ± 374.33 mg/kg, it is better to report 2500 ± 370 mg/kg or instead of 9910 ± 1486.50 mg/kg, it is better to report 9910 ± 1500 mg/kg.⁹

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 785 results for which E_n-scores were calculated, 623 (79%) returned a satisfactory score of $|E_n| \leq 1.0$ indicating agreement of the participants' results with the assigned values within their respective expanded measurement uncertainties.



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

Figure 56 E_n-Score Dispersal by Laboratory

7.4 z-Score

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

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

The between laboratory coefficient of variation predicted by the Thompson equation⁷ and the participants' coefficient of variation resulted in this study are presented for comparison in Table 68.

The dispersal of participants' z-scores is presented in Figure 57 (by laboratory code) and in Figure 58 (by test). Of 785 results for which z-scores were calculated, 711 (91%) returned satisfactory score of $|z| \leq 2.0$ and 37 (5%) were questionable of $2.0 < |z| < 3.0$. Participants with multiple z-scores larger than 2.0 or smaller than -2.0 should check for laboratory bias.

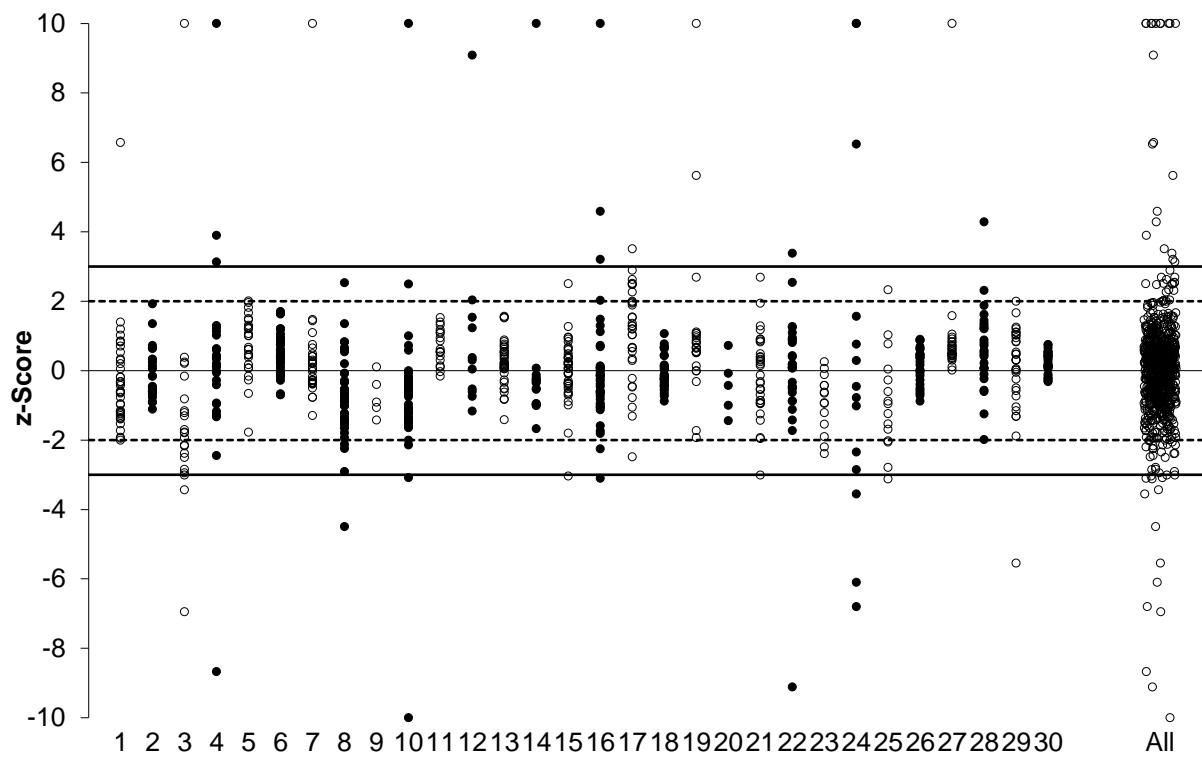


Figure 57 z-Score Dispersal by Laboratory

No Laboratories reported results for all analytes for which a z-score was calculated (45).

Laboratory 6 returned the highest number of satisfactory z scores (40 out of 40 reported). All results reported by **laboratories 13 (37), 18 (31), 30 (27), 26 (25), 2 (23), 11 (18), 9 (5), and 20 (5)** also returned satisfactory z scores.

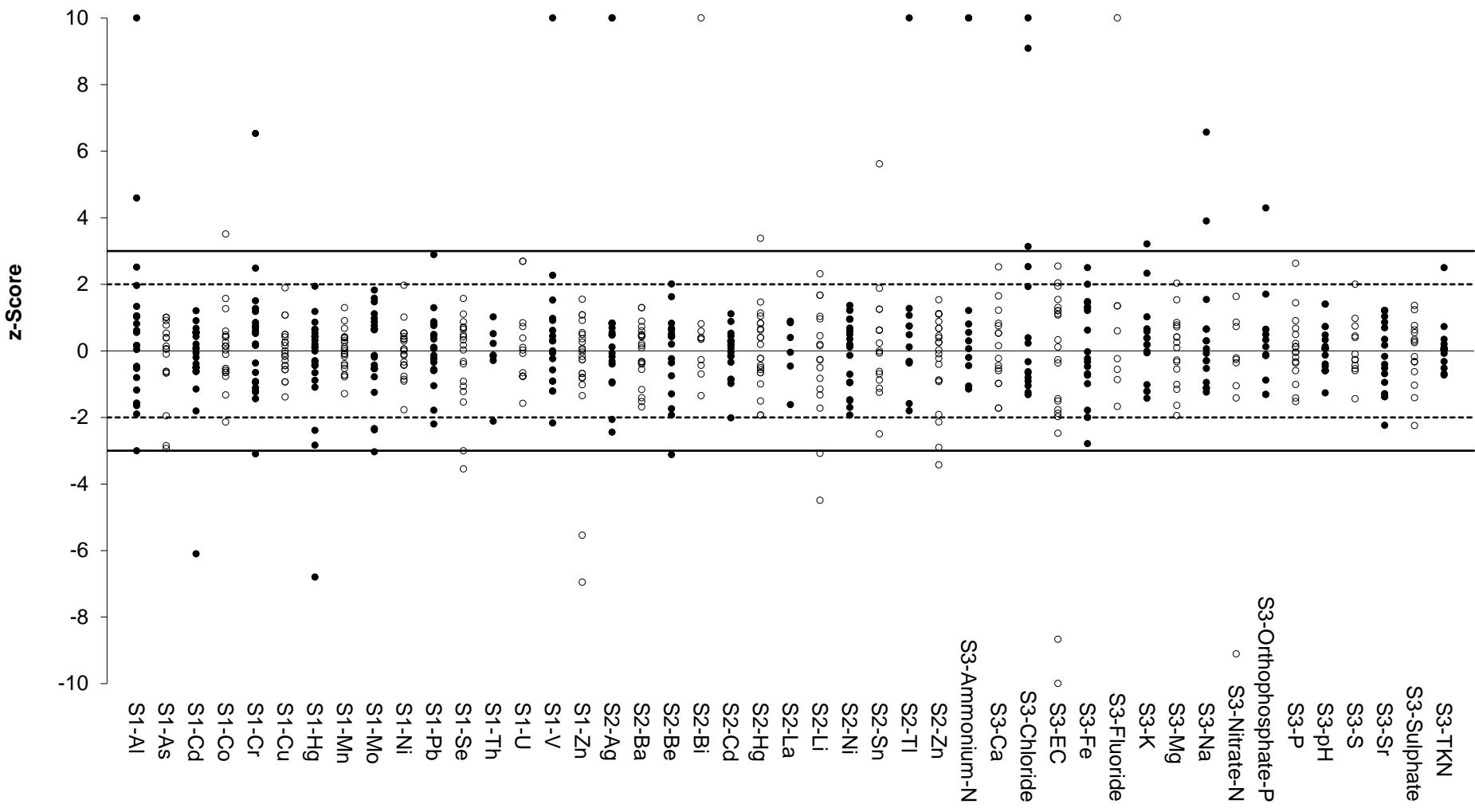
Laboratory 6 returned the highest number of satisfactory En scores (38 out of 40). All results reported by **laboratories 18 (31), 30 (27), and 26 (25)** returned satisfactory En scores.

Table 68 Between Laboratory CV of this Study, Thompson CV and Set Target SD

| Sample | Test | Assigned value (mg/kg) | Between Laboratories CV* | Thompson/Horwitz CV | Target SD (as PCV) |
|--------|------|------------------------|--------------------------|---------------------|--------------------|
| S1 | Al | 8500 | 22% | 4.1% | 15% |
| S1 | As | 20.9 | 8.9% | 10% | 10% |

| Sample | Test | Assigned value (mg/kg) | Between Laboratories CV* | Thompson/ Horwitz CV | Target SD (as PCV) |
|--------|------------------|---------------------------|--------------------------------|-------------------------|-----------------------|
| S1 | Cd | 8.53 | 7.1% | 12% | 10% |
| S1 | Co | 8.88 | 8.2% | 12% | 10% |
| S1 | Cr | 20.4 | 18% | 10% | 15% |
| S1 | Cu | 24.4 | 7.2% | 9.9% | 10% |
| S1 | Hg | 9.21 | 8.4% | 11 | 10% |
| S1 | Mn | 356 | 6.1% | 6.6% | 10% |
| S1 | Mo | 8.46 | 14% | 12% | 10% |
| S1 | Ni | 13.9 | 10% | 11% | 15% |
| S1 | Pb | 28.7 | 8.5% | 9.7% | 10% |
| S1 | Sb | Not Set | 38% | NA | NA |
| S1 | Se | 8.58 | 16% | 12% | 15% |
| S1 | Th | 3.82 | 16% | 13% | 20% |
| S1 | U | 0.867 | 11% | 16% | 10% |
| S1 | V | 33.0 | 11% | 9.5% | 10% |
| S1 | Zn | 74.0 | 8.4% | 8.4% | 10% |
| S2 | Ag | 2.77 | 8.7% | 14% | 10% |
| S2 | B | Not Set | 47% | NA | NA |
| S2 | Ba | 248 | 9.6% | 7% | 10% |
| S2 | Be | 1.69 | 19% | 15% | 15% |
| S2 | Bi | 0.925 | 7.4% | 16% | 10% |
| S2 | Cd | 0.574 | 10% | 17% | 15% |
| S2 | Cs | Not Set | 41% | NA | NA |
| S2 | Ga | Not Set | 36% | NA | NA |
| S2 | Hg | 0.471 | 11% | 18% | 10% |
| S2 | La | 24.7 | 16% | 9.9% | 15% |
| S2 | Li | 10.4 | 22% | 11% | 15% |
| S2 | Ni | 24.5 | 18% | 9.9% | 15% |
| S2 | Rb | Not Set | 30% | NA | NA |
| S2 | Sb | Not Set | 53% | NA | NA |
| S2 | Se | Not Set | 41% | NA | NA |
| S2 | Sn | 16.0 | 11% | 11% | 10% |
| S2 | Tl | 1.26 | 17% | 15% | 15% |
| S2 | Zn | 30.9 | 17% | 9.6% | 15% |
| S3 | Ammonium-N | 39.8 | 9.1% | 9.2% | 10% |
| S3 | Bromide | Not Set | NA | NA | NA |
| S3 | Ca | 1330 | 13% | 5.4% | 10% |
| S3 | Chloride | 83.8 | 15% | 8.2% | 10% |
| S3 | EC | 854 | 6.7% | 5.8% | 3.5% |
| S3 | Fe | 7250 | 17% | 4.2% | 10% |
| S3 | Fluoride | 3.15 | 26% | 13% | 20% |
| S3 | K | 251 | 20% | 7% | 20% |
| S3 | Mg | 424 | 18% | 6.4% | 15% |
| S3 | Na | 169 | 9.8% | 7.4% | 10% |
| S3 | Nitrate-N | 313 | 12% | 6.7% | 10% |
| S3 | Orthophosphate-P | 4.38 | 15% | 13% | 15% |
| S3 | P | 734 | 9.6% | 5.9% | 10% |
| S3 | pH | 4.29 | 2.1% | 13% | 3.5% |
| S3 | S | 596 | 7.8% | 6.1% | 10% |
| S3 | Sr | 11.6 | 12% | 11% | 10% |
| S3 | Sulphate | 685 | 15% | 6% | 15% |
| S3 | TKN | 2480 | 4.9% | 4.9% | 10% |

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



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

Figure 58 z-Score Dispersal by Test

Table 69 Summary of Participants' Results and Performance in Sample S1

| Lab Code | Al (mg/kg) | As (mg/kg) | Cd (mg/kg) | Co (mg/kg) | Cr (mg/kg) | Cu (mg/kg) | Hg (mg/kg) | Mn (mg/kg) | Mo (mg/kg) | Ni (mg/kg) | Pb (mg/kg) | Sb (mg/kg) | Se (mg/kg) | Th (mg/kg) | U (mg/kg) | V (mg/kg) | Zn (mg/kg) |
|----------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|-----------|-----------|------------|
| A.V. | 8500 | 20.9 | 8.53 | 8.88 | 20.4 | 24.4 | 9.21 | 356 | 8.46 | 13.9 | 28.7 | Not Set | 8.58 | 3.82 | 0.867 | 33.0 | 74.0 |
| H.V. | 8000 | 19.0 | 7.9 | 8.9 | 22.0 | 25.3 | 8.9 | 340 | 9.4 | 15.3 | 29.3 | NA | 9.0 | 4.60 | 0.89 | 34.0 | 66 |
| 1 | 7000 | 21 | 8.0 | 8.3 | 21 | 21 | 9.7 | 310 | 8.3 | 13 | 27 | 7 | 9.5 | 4.6 | 0.8 | 34 | 64 |
| 2 | 7900 | 21 | 8 | 9 | 17 | 24 | 9.8 | 340 | 8 | 12 | 27 | 4.5 | 9 | 3.7 | 0.8 | 30 | 69 |
| 3 | 7463.05 | 14.75 | 7.55 | 6.98 | 16.7 | 25 | 6.59 | 369.75 | 6.45 | 10.2 | 23.55 | 7.6 | 4.7 | NT | NT | 25.85 | 22.6 |
| 4 | NT | NT | NT | NT |
| 5 | 11000 | 23 | 9.1 | 10 | 25 | 27 | 8.6 | 370 | 10 | 16 | 30 | <10 | 8.8 | NT | <10 | 35 | 72 |
| 6 | 9280 | 19.5 | 8.48 | 9.10 | 22.2 | 25.4 | 9.39 | 356 | 9.28 | 14.6 | 29.0 | 11.4 | 9.12 | 3.72 | 0.90 | 32.8 | 73.7 |
| 7 | 8546 | 21.74 | 8.1 | 8.2 | 19.26 | 25 | 9.3 | 342 | 9.7 | 13.2 | 27.7 | 5.9 | 10 | 3.6 | 0.8 | 34 | 72 |
| 8 | 6390 | 19.5 | 8.7 | 8.4 | 16.6 | 23.7 | 8.9 | 353 | 8.0 | 12.3 | 27.1 | 4.3 | 7 | 2.2 | 0.8 | 30 | 68.1 |
| 9 | NT | NT | NT | NT |
| 10 | 6500 | 23 | 8.2 | 7.7 | 16 | 23 | 8.2 | 350 | 8.0 | 13 | 27 | 5.1 | 7.2 | NT | 0.73 | 29 | 69 |
| 11 | 10200 | 22 | 9 | 9 | 24 | 24 | 10 | 371 | NT | 15 | 29 | NT | NT | NT | NT | NT | 82 |
| 12 | NT | NT | NT | NT |
| 13 | 8700 | 21 | 9.0 | 9.0 | 21 | 25 | 9.31 | 370 | 9.1 | 14 | 29 | NT | 10.6 | NT | 0.94 | 38 | 78 |
| 14 | NT | NT | NT | NT |
| 15 | 11700 | 19.6 | 8.09 | 9.24 | 24.3 | 25.6 | 8.39 | 365 | 5.89 | 13.7 | 29.7 | 4.94 | 9.40 | 3.99 | 0.86 | 36.0 | 74.5 |
| 16 | 14349 | 21.2 | 6.99 | 8.35 | 10.9 | 24.2 | 8.8 | 358 | 8.34 | 13.9 | 25.7 | 3.35 | 8.08 | NT | NT | 31.1 | 67.9 |
| 17 | 9820 | <25 | 9.55 | 12.0 | 28.0 | 29.0 | 11.0 | 402 | 7.80 | 18.0 | 37.0 | <100 | <100 | NT | NT | 40.5 | 85.5 |
| 18 | 7830 | 22.5 | 8.36 | 8.78 | 20.9 | 23.3 | 9.61 | 330 | 8.99 | 14.0 | 28.3 | 8.50 | 9.47 | NT | 0.81 | 32.2 | 74.2 |
| 19 | 9530 | 23 | 9 | 9 | 22 | 27 | 9.2 | 380 | 9 | 15 | 31 | NT | 9.7 | NT | 1.1 | 33 | 82 |
| 20 | NT | NT | NT | NT | NT | NT | 8.81075 | NT | NT | NT | NT |
| 21 | 4660 | 16.8 | 9.3 | 8.4 | 17.5 | 22.1 | 10.3 | 337 | 7.4 | 12.1 | 32.4 | 4.1 | 7.4 | NT | 1.10 | NT | 80.7 |
| 22 | NT | NT | NT | NT |
| 23 | 6080 | 19.6 | 8.58 | 8.3 | 17.6 | 22.1 | 7.01 | 352 | 8.1 | 13.0 | 22.4 | 9.8 | 6.6 | NR | NR | 29.0 | 75.9 |
| 24 | 23607.181 | 14.949 | 3.326 | 10.278 | 40.385 | 23.302 | 2.950 | 328.349 | 6.480 | 14.495 | 30.892 | 7.762 | 4.006 | NR | NR | 73.066 | 66.482 |
| 25 | NT | NT | NT | NT |
| 26 | 8700 | 21.7 | 8.9 | 9.3 | 18.4 | 24.4 | 9.6 | 388 | 9.2 | 13.6 | 31.2 | 5.22 | <20 | NT | 0.869 | <100 | 76.7 |
| 27 | 9841 | 22.8 | 9.0 | 9.4 | 22.5 | 25.6 | 9.8 | 360 | 9.8 | 14.8 | 30.1 | 8.5 | 9.2 | NT | 0.93 | 36.2 | 77.6 |
| 28 | NT | NT | NT | NT |
| 29 | 9800 | 21 | 8.1 | 8.9 | 23 | 23 | 9.5 | 330 | 9.4 | 15 | 28 | <10 | 8.6 | NT | <10 | 34 | 33 |
| 30 | 9200 | 20.7 | 8.61 | 9.25 | 22.68 | 24.77 | 8.93 | 367.1 | 9.01 | 14.67 | 28.87 | 9.08 | 8.17 | 4.21 | 0.876 | 34.44 | 72.72 |

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

Table 70 Summary of Participants' Results and Performance in Sample S2

| Lab Code | Ag (mg/kg) | B (mg/kg) | Ba (mg/kg) | Be (mg/kg) | Bi (mg/kg) | Cd (mg/kg) | Cs (mg/kg) | Ga (mg/kg) | Hg (mg/kg) | La (mg/kg) | Li (mg/kg) | Ni (mg/kg) | Rb (mg/kg) | Sb (mg/kg) | Se (mg/kg) | Sn (mg/kg) | Tl (mg/kg) | Zn (mg/kg) |
|----------|------------|-----------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|------------|
| A.V. | 2.77 | Not Set | 248 | 1.69 | 0.925 | 0.574 | Not Set | Not Set | 0.471 | 24.7 | 10.4 | 24.5 | Not Set | Not Set | Not Set | 16.0 | 1.26 | 30.9 |
| S.V. | 2.84 | 38.9 | 235 | 1.79 | 0.872 | 0.608 | 3.58 | 13.1 | 0.541 | 29.3 | 11.3 | 26.0 | 7.01 | 22.2 | 1.98 | 16.9 | 1.44 | Not Spiked |
| H.V. | 2.90 | 27.0 | 222 | 1.56 | 0.95 | 0.583 | 2.88 | 11.2 | 0.482 | 26.4 | 11.0 | 26.6 | NA | 12.0 | 1.60 | 16.8 | 1.40 | 29.2 |
| 1 | 3 | 28 | 210 | 1.6 | NT | 0.49 | NT | NT | 0.51 | NT | 8.6 | 21 | NT | 9.5 | 9.6 | 14 | 1.2 | 22 |
| 2 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |
| 3 | NT | 8.6 | 253.1 | 1.36 | 2.025 | 0.56 | NT | NT | 0.38 | NT | NT | 18.25 | NT | 12.4 | 0.93 | 12 | NT | 15 |
| 4 | 2.09 | <50 | 219 | 1.74 | 0.956 | <1.0 | 2.5 | 10.8 | 0.524 | 26.2 | 8.33 | 20.99 | 24 | 6.23 | <5 | 15.9 | 126.9 | 26.6 |
| 5 | 2.9 | 37 | 250 | 2.2 | <10 | 0.56 | NT | NT | 0.54 | NT | 13 | 29 | NT | 18 | <2 | 17 | <10 | 33 |
| 6 | 2.72 | 33.1 | 259 | 1.81 | NT | 0.62 | NT | NT | 0.49 | NT | 9.96 | 26.7 | NT | 26.7 | 1.74 | NT | 1.46 | 30.5 |
| 7 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |
| 8 | 3.0 | <50 | 213 | 1.2 | 0.8 | 0.5 | 1.3 | 6.7 | 0.4 | 18.7 | 3.4 | 19.1 | 13.4 | 5.0 | 1 | 14.9 | 1.2 | 17.4 |
| 9 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |
| 10 | 2.5 | 19 | 240 | 1.5 | 0.98 | 0.57 | 1.6 | NT | 0.44 | 23 | 5.6 | 19 | 16 | 8.0 | 0.95 | 16 | <1.8 | 21 |
| 11 | NT | NT | NT | NT | NT | 0.6 | NT | NT | 0.5 | NT | NT | 28 | NT | NT | NT | NT | NT | 38 |
| 12 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |
| 13 | 2.75 | 32 | 260 | 1.80 | NT | 0.65 | NT | NT | 0.447 | NT | 9.1 | 27 | NT | NT | <5 | 15 | 1.28 | 32 |
| 14 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |
| 15 | 2.66 | 11.9 | 263 | 1.86 | 0.86 | 0.544 | NR | NR | 0.48 | 27.8 | 11.9 | 25.2 | NR | 8.83 | 1.10 | 15.9 | 0.92 | 32.7 |
| 16 | 2.51 | 50.4 | 266 | 1.25 | NT | NT | NT | NT | 0.44 | NT | NT | 24 | NT | 12.0 | 1.08 | 14.2 | 0.96 | 36.1 |
| 17 | <1 | 32.0 | 280 | <1 | NT | <1 | NT | NT | 0.45 | NT | 12.0 | 29.5 | NT | <100 | <100 | <50 | <2 | 34.0 |
| 18 | 2.68 | 20.5 | 234 | 1.63 | 0.884 | 0.612 | NT | NT | 0.502 | NT | 10.0 | 24.9 | NT | 19.2 | 1.43 | 14.6 | 1.19 | 29.0 |
| 19 | 59 | 17 | 240 | 1.82 | NT | 0.67 | NT | NT | 0.38 | NT | 7.7 | 28 | NT | NT | 2.4 | 25 | NT | 34 |
| 20 | NT | NT | NT | NT | NT | NT | NT | NT | 0.424 | NT | NT | NT | NT | <5 | NT | NT | NT | NT |
| 21 | 3.0 | 10.7 | 239 | NT | NT | 0.50 | NT | NT | 0.49 | NT | 10.7 | 17.4 | NT | 4.8 | NR | NT | NT | 32.1 |
| 22 | 2.8 | 33 | 270 | 1.8 | 1.0 | 0.59 | 3.6 | 13 | 0.63 | 28 | NT | 28 | NT | 11 | 1.8 | 18 | 1.5 | 36 |
| 23 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |
| 24 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |
| 25 | 2.2 | 5 | 206 | 0.9 | NT | 0.4 | NT | NT | NT | NT | NT | 21 | NT | NT | NT | NT | NT | 26.8 |
| 26 | 2.96 | 25.7 | 238 | 1.85 | 0.90 | 0.596 | 2.42 | NT | 0.444 | 24.5 | 9.6 | 21.9 | 21.7 | 7.0 | <2 | 17.0 | 1.35 | 26.8 |
| 27 | 45.5 | 24.7 | 258.3 | 1.9 | 0.96 | 0.6 | 3.1 | NT | 0.51 | NT | 11.1 | 25.8 | 25.7 | 15.0 | 2.00 | 17.0 | 1.4 | 31 |
| 28 | 2.9 | 36 | 280 | 2.1 | <10 | 0.58 | NT | NT | 0.46 | NT | 14 | 29 | NT | 21 | 2.1 | 19 | <10 | 35 |
| 29 | 3.0 | 30 | 260 | <2 | <10 | 0.60 | NT | NT | 0.52 | NT | 13 | 28 | NT | 18 | <2 | 18 | <10 | 36 |
| 30 | 2.92 | 27.97 | 251.6 | 1.80 | 0.962 | 0.587 | NT | NT | 0.460 | NT | 10.63 | 26.3 | NT | 20.40 | 1.22 | 16.37 | 1.40 | 29.82 |

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

Table 71 Summary of Participants' Results and Performance for Sample S3

| Lab Code | Ca (mg/kg) | Fe (mg/kg) | K (mg/kg) | Mg (mg/kg) | Na (mg/kg) | P (mg/kg) | S (mg/kg) | Sr (mg/kg) |
|----------|------------|------------|-----------|------------|------------|-----------|-----------|------------|
| A.V. | 1330 | 7250 | 251 | 424 | 169 | 734 | 596 | 11.6 |
| H.V. | 1390 | 7570 | 223 | 418 | 159 | 800 | NA | 10.0 |
| 1 | 1100 | 5800 | 190 | 300 | 280 | 720 | 560 | 10 |
| 2 | NT | NT | NT | NT | NT | NT | NT | NT |
| 3 | NT | NT | NT | NT | NT | NT | NT | NT |
| 4 | 1492 | 8191 | 302.3 | 406 | 234.9 | 743.9 | 622 | 11.12 |
| 5 | NT | 8200 | NT | NT | NT | NT | NT | 13 |
| 6 | 1550 | 8130 | 248 | 478 | 174 | 800 | 640 | 10.8 |
| 7 | 1430 | 7010 | 250 | 450 | 180 | 840 | 580 | 10.1 |
| 8 | 1440 | 5950 | 200 | 360 | 180 | 690 | 510 | 9 |
| 9 | NR | NR | NR | NR | NR | NR | NR | NR |
| 10 | 1300 | 5800 | 190 | 320 | 150 | 730 | 570 | 10 |
| 11 | NT | 7230 | NT | NT | NT | NT | NT | NT |
| 12 | 1260 | 6710 | 270 | 350 | 195 | NR | NR | NR |
| 13 | 1400 | 7700 | 260 | 470 | 160 | 710 | 580 | 12 |
| 14 | 1200 | NR | NR | 403 | 153 | 660 | 564 | 10.5 |
| 15 | 1270 | 6530 | 270 | 440 | 164 | 707 | 654 | 11.8 |
| 16 | 1200 | 8324 | 412 | 553 | 164 | 742 | NT | 12.4 |
| 17 | 1665 | 9065 | 284 | 521 | 180 | 927 | 715 | 13.0 |
| 18 | NT | 7080 | NT | NT | NT | 783 | NT | 11.4 |
| 19 | NT | NT | NT | NT | NT | NT | NT | NT |
| 20 | NT | NT | NT | NT | 167.6 | NT | NT | NT |
| 21 | 1350 | 6750 | 179 | 389 | 174 | 770 | NT | 12.6 |
| 22 | 1100 | 6900 | 260 | 430 | 150 | 630 | 620 | 11 |
| 23 | NT | NT | NT | NT | NT | NT | NT | NT |
| 24 | NT | NT | NT | NT | NT | NT | NT | NT |
| 25 | 1250 | 5230 | 368 | 473 | 148 | 622 | 580 | 12.8 |
| 26 | NT | NT | NT | NT | NT | NT | NT | NT |
| 27 | NT | NT | NT | NT | NT | NT | NT | NT |
| 28 | 1400 | 8300 | 280 | 450 | 170 | 750 | 590 | 13 |
| 29 | NT | 8700 | NT | NT | NT | 730 | NT | 13 |
| 30 | NT | NT | NT | NT | NT | NT | NT | NT |

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

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

| Lab Code | Bromide (mg/kg) | Chloride (mg/kg) | Fluoride (mg/kg) | Iodide (mg/kg) | Orthophosphate-P (mg/kg) | Sulphate (mg/kg) | pH | EC (µS/cm) | TKN (mg/kg) | Ammonium-N (mg/kg) | Nitrate-N (mg/kg) |
|----------|-----------------|------------------|------------------|----------------|--------------------------|------------------|------|------------|-------------|--------------------|-------------------|
| A.V. | Not Set | 83.8 | 3.15 | Not Set | 4.38 | 685 | 429 | 854 | 2480 | 39.8 | 313 |
| H.V. | 2.00 | 80 | 4.00 | NA | 4.40 | 645 | NA | NA | 2600 | 36.5 | NA |
| 1 | NT | NT | NT | NT | NT | 720 | 4.5 | 890 | 2400 | NT | 340 |
| 2 | NT | 100 | 4 | NT | 4.8 | 710 | 4.2 | 864 | 2660 | NT | NT |
| 3 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |
| 4 | NT | 110 | 3.53 | NT | 4.46 | 750 | 4.1 | 595 | 2480 | NT | NT |
| 5 | NT | NT | NT | NT | NT | NT | 4.3 | 801 | NT | NT | NT |
| 6 | NT | NT | NT | NT | 5.5 | 763 | 4.34 | 890 | 2310 | 44.6 | 364 |
| 7 | 1.58 | 85.7 | 29.8 | 0.58 | 4.7 | 666 | 4.3 | 843 | 2530 | NT | NT |
| 8 | 1.92 | 105 | 4 | <0.25 | 4.8 | 740 | 4.2 | 809 | 2460 | NT | NT |
| 9 | NR | 76.2 | NR | NR | NR | NR | 4.23 | 857.3 | NR | 35.6 | 268.4 |
| 10 | NT | NT | NT | NT | NT | NT | 4.4 | 79 | 3100 | 81 | NT |
| 11 | NT | NT | NT | NT | NT | NT | 4.5 | 887.20 | NT | NT | NT |
| 12 | NR | 160 | NR | NR | NR | 811 | 4.2 | 915 | 2490 | 41 | NR |
| 13 | <5 | 77 | NT | NT | NT | 540 | 4.3 | 900 | NT | NT | NT |
| 14 | <5 | 865 | 2.1 | <5 | 4.3 | 650 | 4.3 | 846 | 2350 | 39 | 305 |
| 15 | NR | 78.3 | NR | NR | NR | NR | NR | NR | 2495.52 | 40.05 | 301.61 |
| 16 | NT | 78.5 | 3.0 | NT | 4.28 | 454 | 4.30 | 893 | 2300 | 420 | 280 |
| 17 | NT | 75 | NT | NT | 3.52 | 715 | 4.22 | 780 | 2564 | 42.0 | 306 |
| 18 | NT | NT | NT | NT | NT | NT | 4.31 | 886 | NT | NT | NT |
| 19 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |
| 20 | NT | NT | NT | NT | NT | NT | 4.4 | 811 | NT | NT | NT |
| 21 | 2.41 | 87.1 | NR | NT | 4.6 | 652 | 4.27 | 912 | NT | NT | NT |
| 22 | 2.0 | 81 | 2.6 | <1 | 3.8 | 620 | 4.3 | 930 | 2500 | 38 | 28 |
| 23 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |
| 24 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |
| 25 | NT | NT | NT | NT | NT | NT | NR | NR | 2490 | NT | NT |
| 26 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |
| 27 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |
| 28 | NT | 73.3 | 2.8 | NT | 7.2 | 825 | 4.2 | 794.8 | 2498 | 43 | 336 |
| 29 | 1.92 | 72.74 | NT | 4.00 | 3.52 | 577.67 | 4.36 | 797.9 | NT | 35.24 | NT |
| 30 | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT | NT |

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

7.5 Participants' Results and Analytical Methods for Acid Extractable Elements

A summary of participants' results and performance is presented in Tables 69 to 72 and in Figures 57 and 58.

Boron and antimony were elements which presented the most analytical difficulty. No agreement was found between the results reported by participants for these elements.

Measurement of low level Se in S2 also presented analytical difficulty to participants.

Manganese in S1 was the test which presented the least analytical difficulty to participating laboratories, with a between laboratory CV of 6.1 %.

All unsatisfactory results reported by laboratory 3 were lower than the assigned value and most were lower by the same factor of approximately 0.75. This laboratory should check their dilution and/or standard preparation procedure. The results from this laboratory were not included in the analyses of extraction methods and of instrumental techniques employed by participants.

All unsatisfactory results reported by laboratory 17 were higher than the assigned value. This is an indication of method or laboratory bias.

The method descriptions provided by participants for acid extractable elements are presented in Tables 1 and 10 while instrumental conditions are presented in Appendix 4.

Extraction Methods

The request was for acid extractable elements; NMI PT studies of metals in soil focus on 'pseudo-total' analyses of elements in soil rather than on true total metal content because when an assessment of the anthropogenic impact of the metal content in a soil sample is made, aggressive digestion regimes (HF, high digestion temperature) can lead to misleading conclusions – since metals can be extracted from the fraction naturally present in the soil matrix.^{5, 15-18} While an aggressive digestion regime can produce high, misleading results, weak digestion regimes (low digestion temperature, reduced digestion time, diluted acids and/or a low ratio of acid to sample size) may extract just a fraction of the contaminants from the soil. There is no standardisation of methods for acid extractable elements. In general methods are conventionally defined by procedures involving extractions: with aqua regia or with various amounts of HNO₃, HCl, in combination or alone and most of these methods produce comparable results.¹⁹⁻²¹

Laboratory 10 extracted their sample at 96°C for 30 min only; all results they reported for acid extractable elements were lower than the assigned value with the exception of two. Weak digestion regimes may only extract a fraction of the elements from soil.

Laboratory 24 used an extraction regime involving HF and a digestion temperature of 210°C, which may explain the high, unsatisfactory results they reported for the method dependent elements Al, Cr and V. The high digestion temperature and HF which they employed for sample extraction might have facilitated extraction of these elements from silica lattice. This digestion regime is more appropriate for assessing the samples' geological content (total elements) and not the contamination introduced in the environment by anthropogenic activities (acid extractable elements).

According to Eurachem/CITAC Guide CG4, laboratories should consider using matrix matched control samples to assess their digestion regime (the bias of their analytical methods). Bias can be expressed as recovery and should be corrected for or included in the uncertainty estimate.⁹

Individual Element Commentary

Aluminium is an element which is strongly dependent on digestion regime. The between-laboratory coefficient of variation for Al in Sample S1 was high (22%), larger than that predicted by Thomson (4.1%).⁹ A high extraction temperature (210°C) and the use of HF may explain the high result reported by Laboratory 24.

Plots of Al participants' performance versus instrumental technique used are presented in Figure 59.

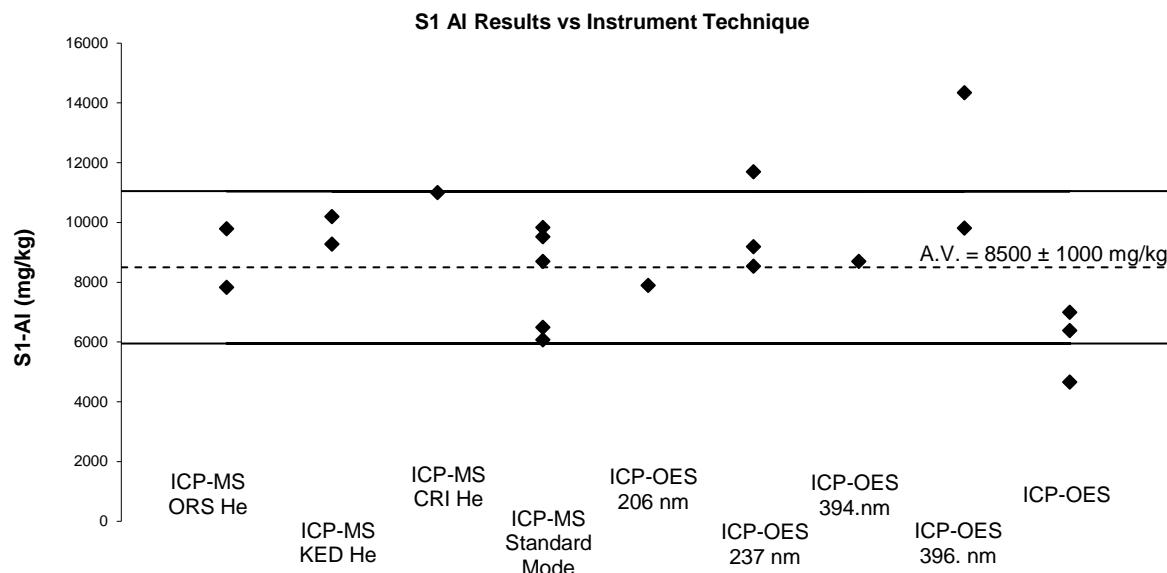


Figure 59 S1-Al z-Results vs. Instrumental Technique

Arsenic most participants used for As measurements ICP-MS with various collision/reaction cells and He as collision gas.

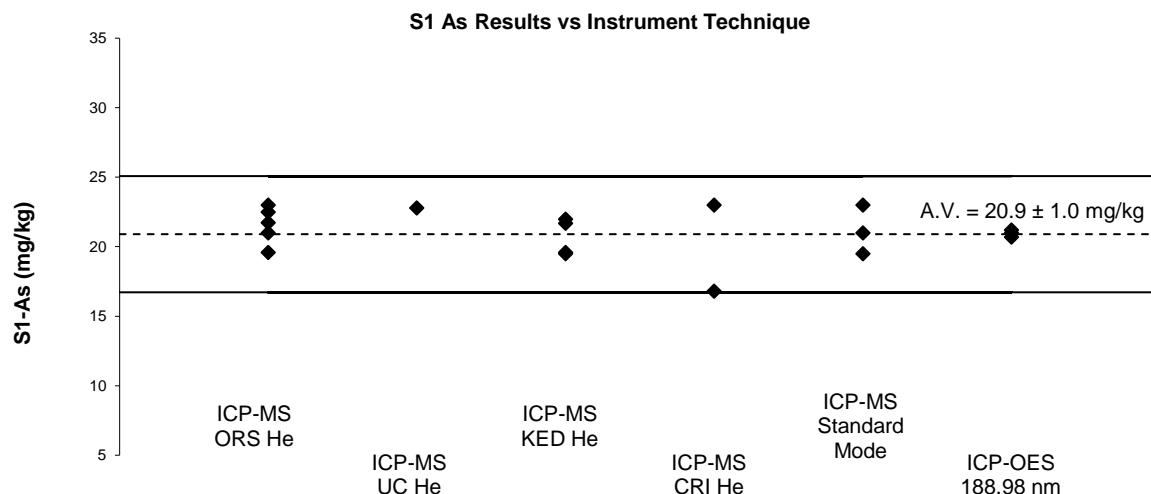


Figure 60 S1-As Results vs. Instrumental Technique

Antimony in the soil sample S1 was one of the most difficult elements to analyse.

Participants used a wide variety of digestion methods; no agreement was found between the results they produced. Antimony is an element whose recovery strongly depends on the acids employed for digestion. It is known that in nitric acid only, Sb is transformed in a mixture of insoluble oxides (Sb_2O_3 , Sb_2O_5 , $\text{Sb}_4\text{O}_4(\text{OH})_2(\text{NO}_3)_2$) but when hydrochloric acid is also involved it changes into chloro-complexes (SbCl_6^-). In aqueous solution, sufficient hydrogen ion concentration must be maintained in order to prevent SbCl_6^- hydrolysis.²²⁻²⁴

Laboratories should consider using matrix matched control samples to assess their digestion regime and increase their estimates of uncertainty for Sb measurements in soil.

Boron level in Sample S2 was low and this may have presented difficulty to some laboratories. The between laboratory coefficient of variation was high (47%) no assigned value was set for this element. Boron is an element prone to contamination. The sampling system should be cleaned prior to low level B determination.

Caution should be exercised when ICP-OES with wavelength 249.7 nm is used for B measurement. Iron line 249.771 nm has direct overlap interference on B line 249.7 nm.

Plots of participants' results versus instrumental technique are presented in Figure 61.

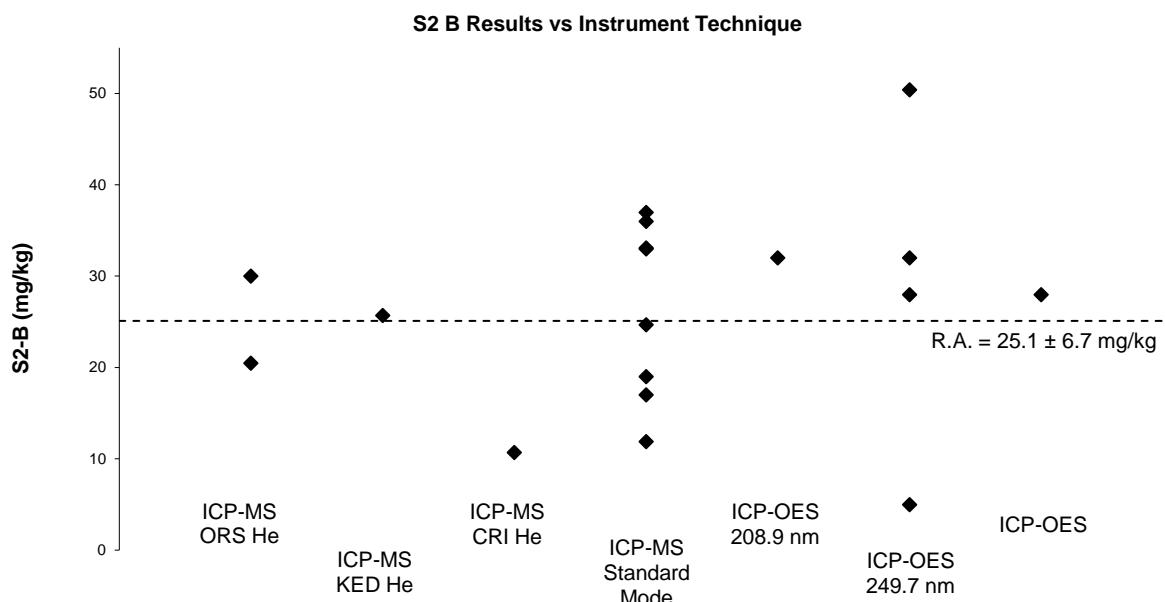


Figure 61 B z-Results vs. Instrumental Technique

Mercury level in S1 was 9.21 mg/kg while in S2 was almost 20 times lower at, 0.471 mg/kg. 22 laboratories reported results in both study samples and all used the same instrumental technique. ICP-MS in standard mode followed by CVAAS were the most popular instrumental techniques used for Hg measurement in S1 and S2 (Figure 62).

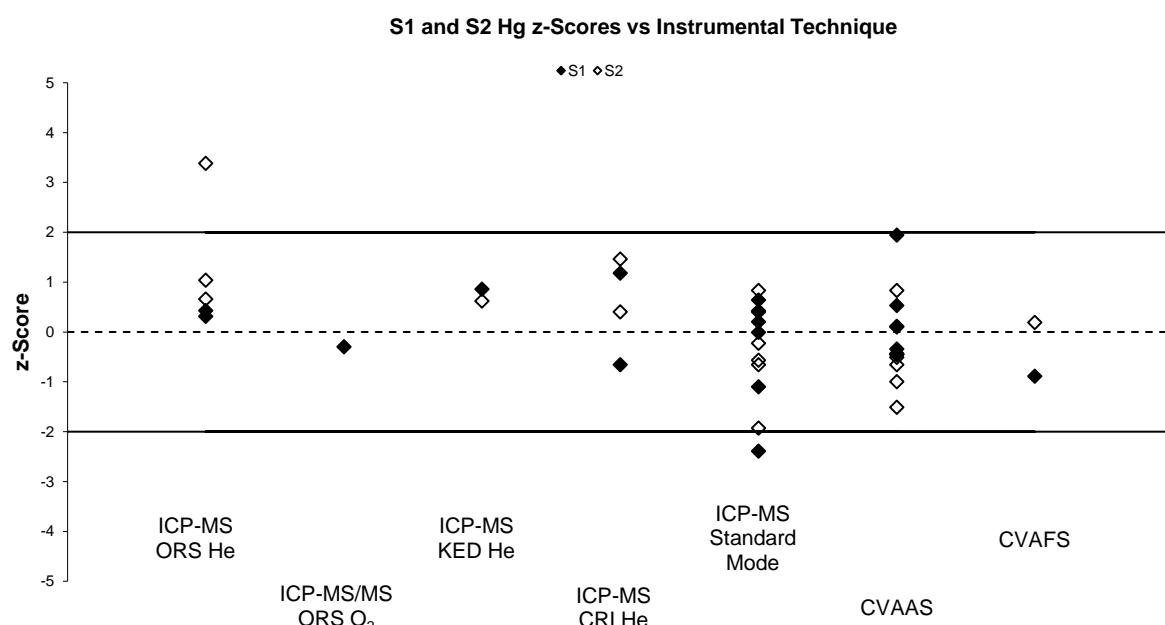


Figure 62 S1 and S2 Hg z-Scores vs. Instrumental Technique

Potassium and Sodium Plots of K and Na results versus instrumental technique are presented in Figures 63 and 64. ICP-OES was the preferred analytical technique.

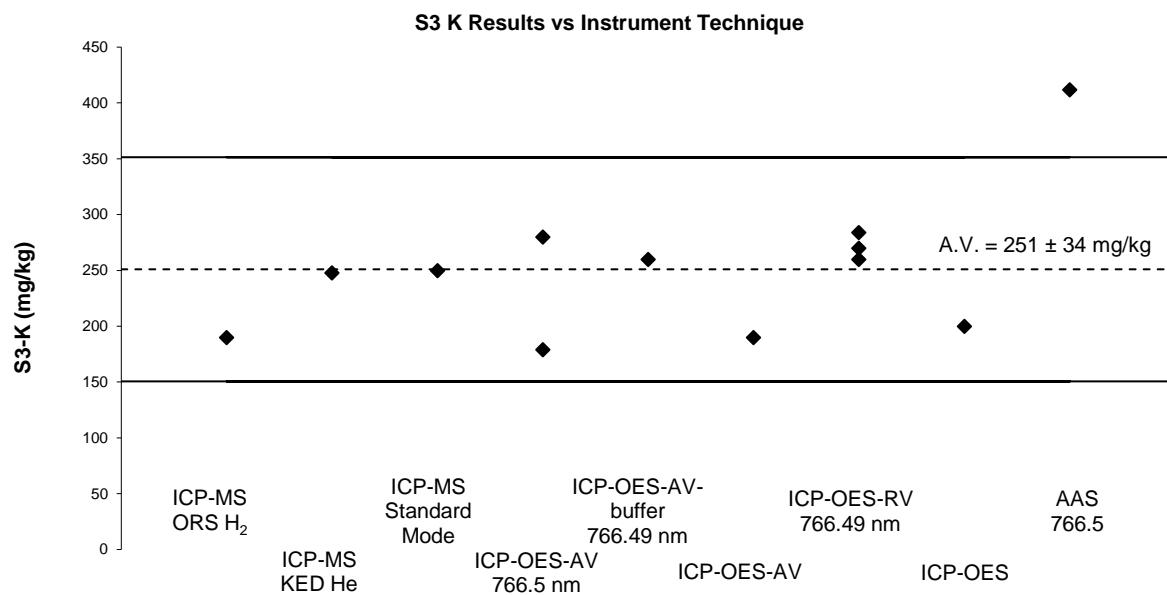


Figure 63 S3 K Results vs. Instrumental Technique

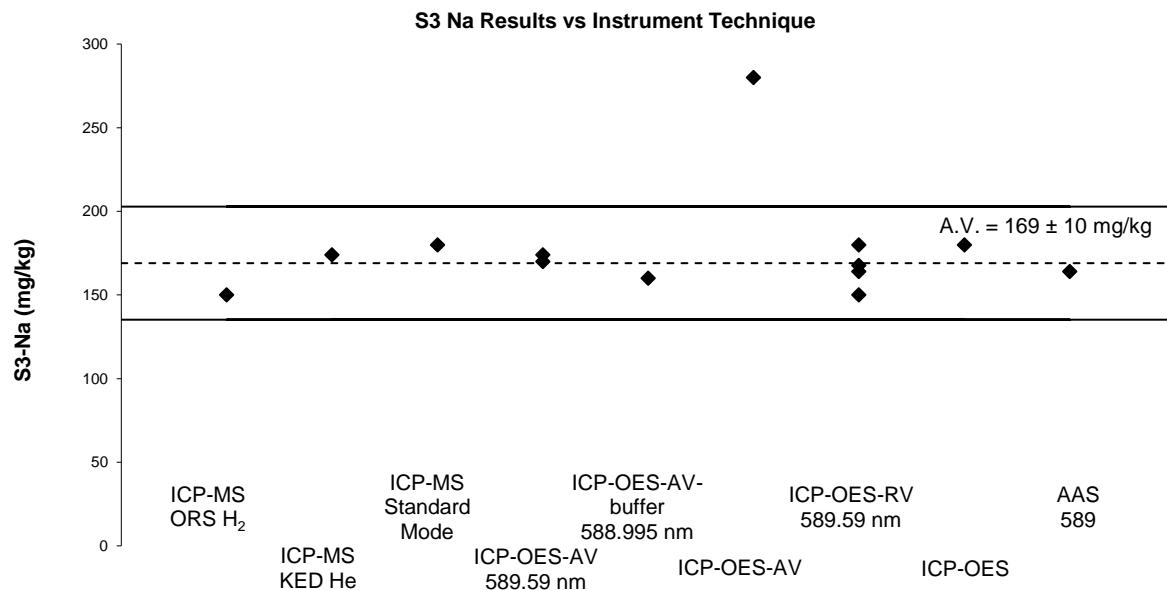
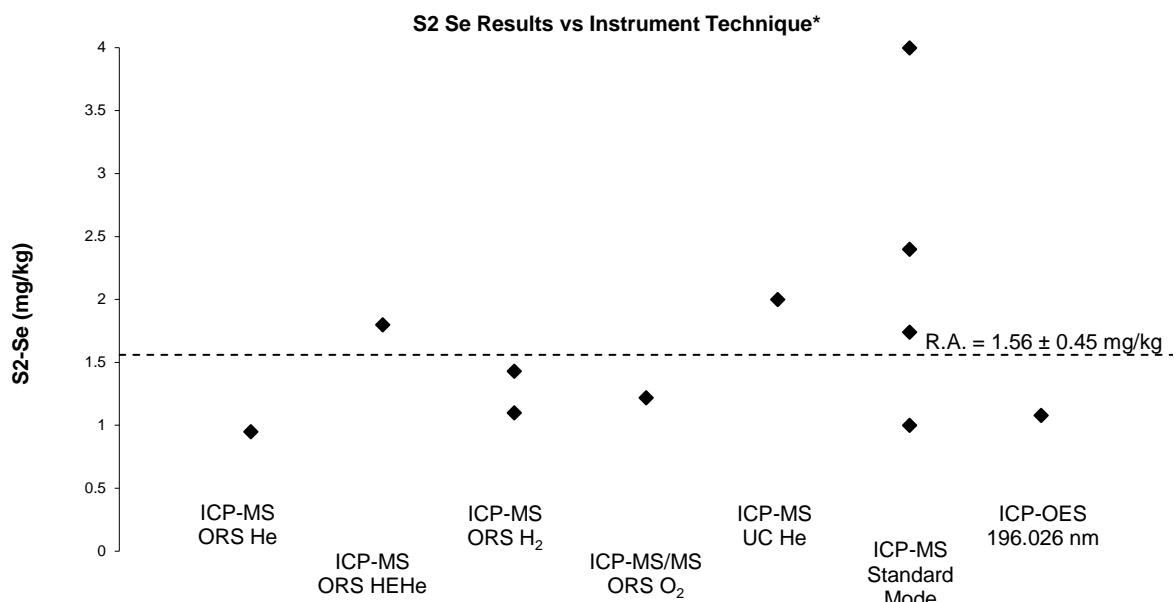


Figure 64 S3 Na Results vs. Instrumental Technique

Lithium The between-laboratory CV for this element was larger than predicted by Thomson and Harwitz (22%). ICP-MS has low sensitivity for light elements due to space-charge effects. An internal standard with similar behaviour may overcome this problem.

Selenium level in S2 was low and challenged participants' instrumental techniques, returning a between-laboratory CV of 41%. When ICP-MS with collision/reaction cell cannot be used, ICP-MS and isotope ⁸²Se may not be the best choice for Se measurements. Consider using ⁷⁷Se or ⁷⁸Se as these isotopes have less interferences.

Plots of participants results versus instrumental techniques used are presented in Figure 65.



*Laboratory 1 results of 9.6 mg/kg has been plotted as 4 mg/kg.

Figure 65 S2 Se Results vs. Instrumental Technique

Selenium level in S2 may be too low for accurate determination by ICP-OES.

7.6 Participants' Within-Laboratory Reproducibility

Sample S1 was a soil sample previously distributed as S2 of AQA 19-12.

Of 23 laboratories who reported results in the present study in S1, nine reported results in AQA 19-12 (Laboratories 3, 6, 7, 13, 16, 17, 26, 27 and 30).

Bar charts of laboratories' results in the two studies are presented in Figure 66. In some cases the participants' results reported in the two studies are significantly different.

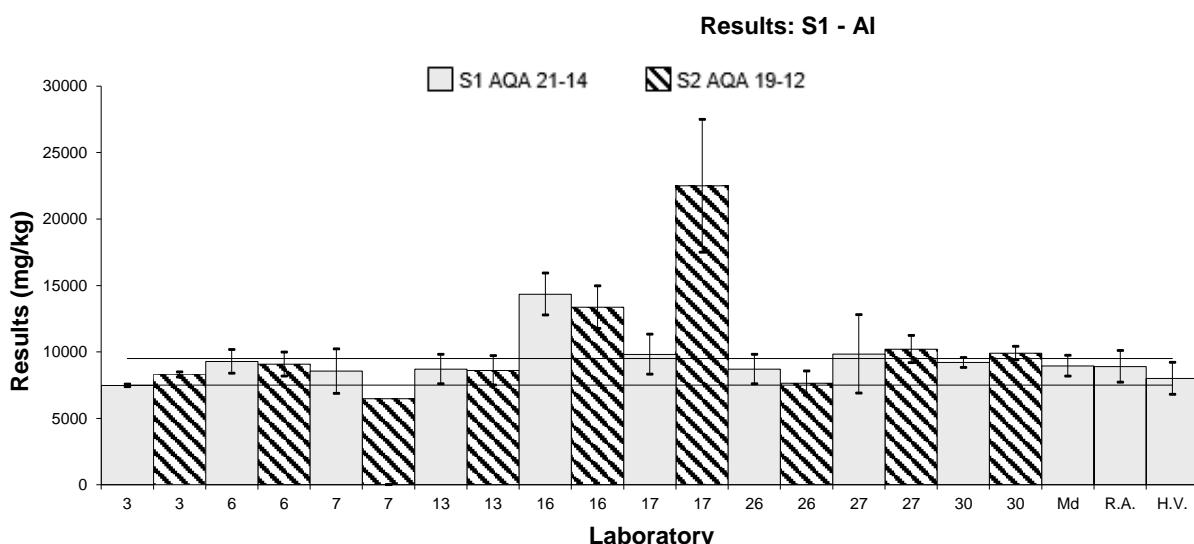
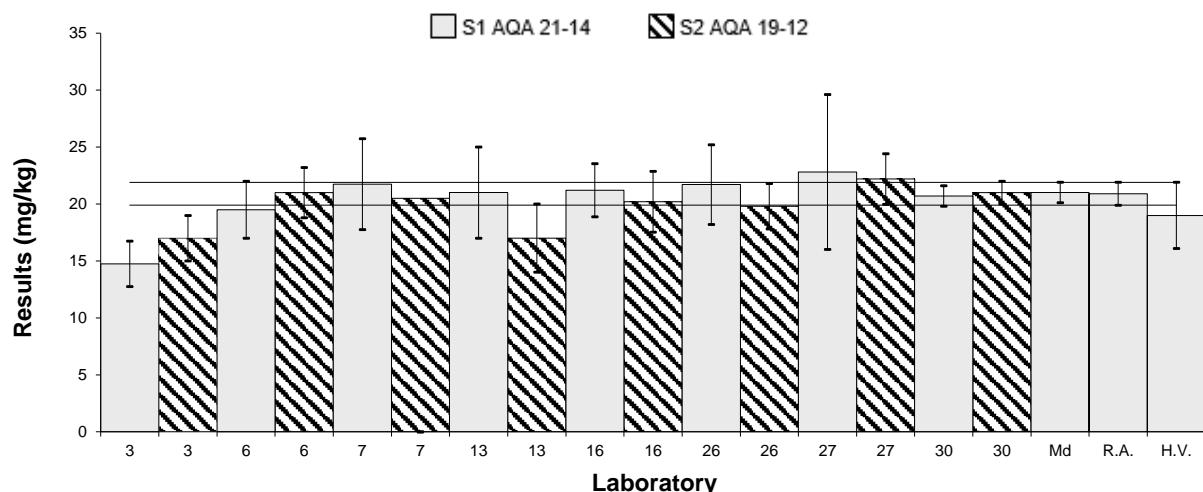
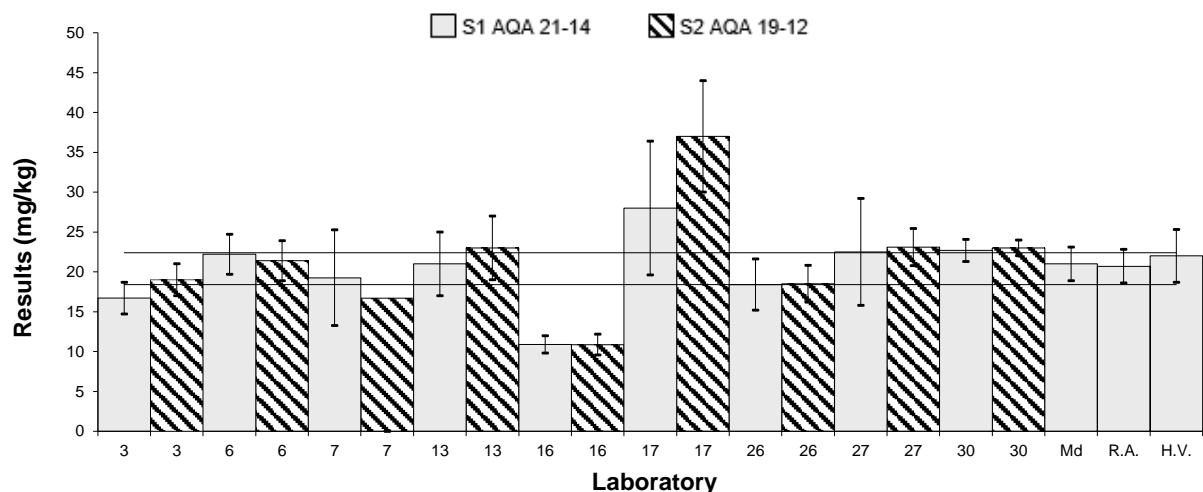


Figure 66 Bar charts of Results in S1 of AQA 21-14 and S2 of AQA 19-12

Results: S1 - As



Results: S1 - Cr



Results: S1 - Hg

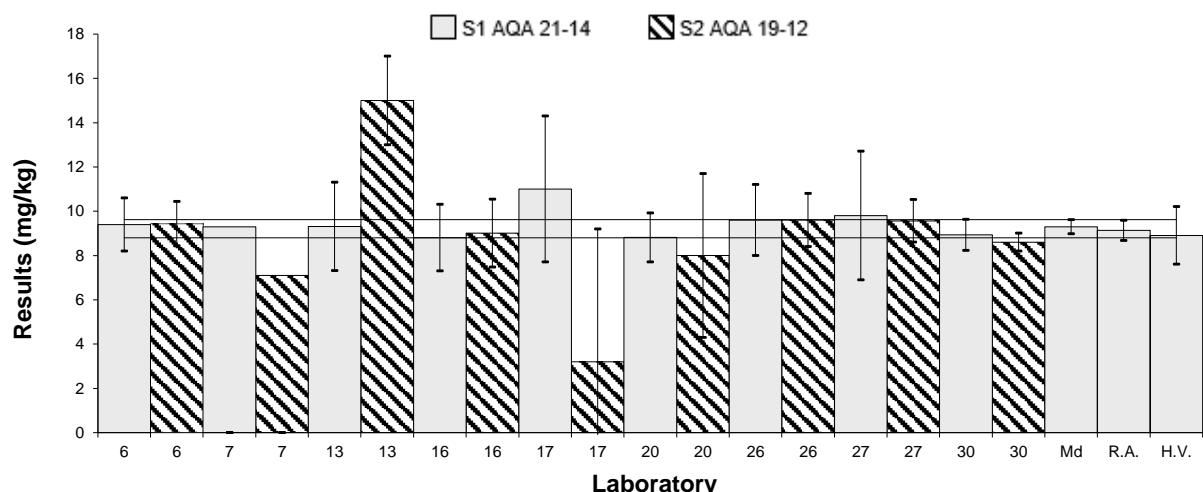
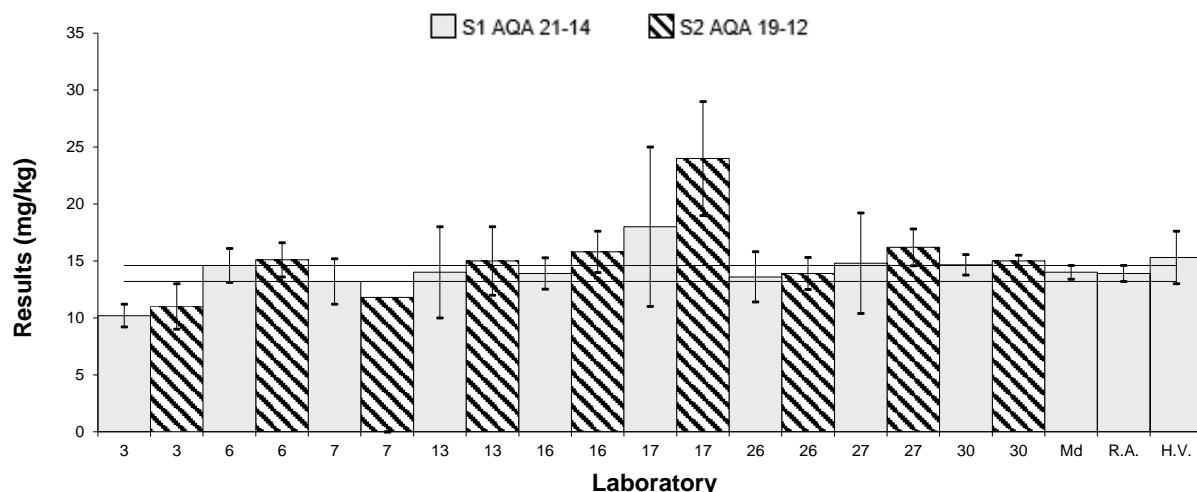
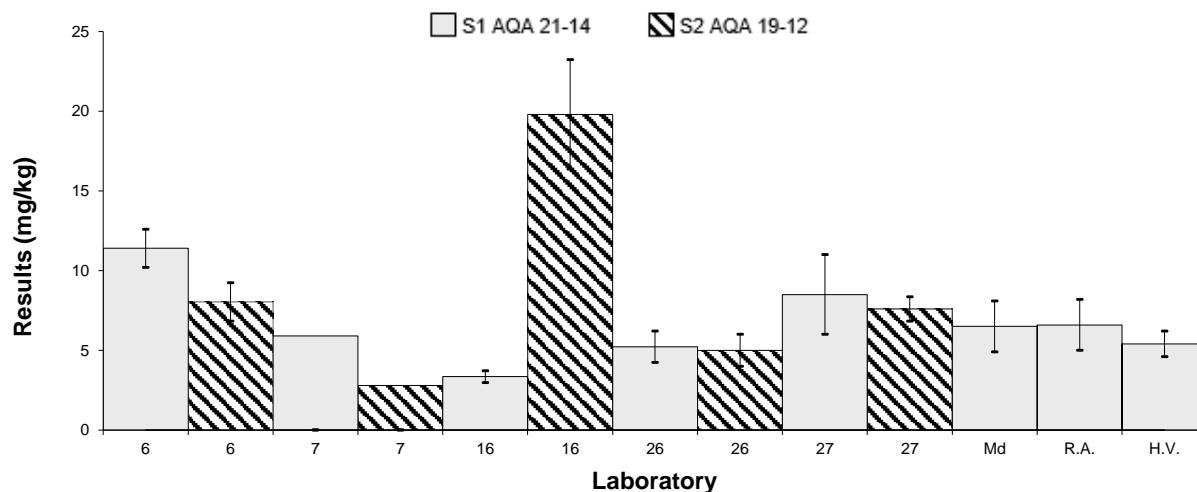


Figure 66 Bar charts of Results in S1 of AQA 21-14 and S2 of AQA 19-12 (continued)

Results: S1 - Ni



Results: S1 - Sb



Results: S1 - Se

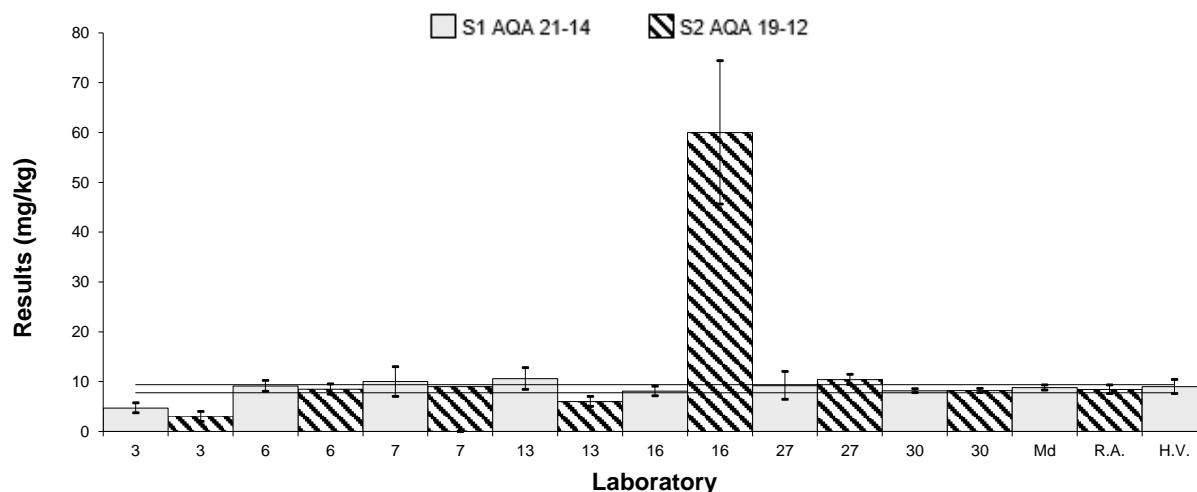


Figure 66 Bar Charts of Results in S1 of AQA 21-14 and S2 of AQA 19-12 (continued)

Figure 67 presents scatter plots of z-scores in Sample S1 of AQA 21-14 and S3 of AQA 19-12. Points close to the diagonal axis represent excellent reproducibility and points close to zero represent excellent reproducibility and accuracy.

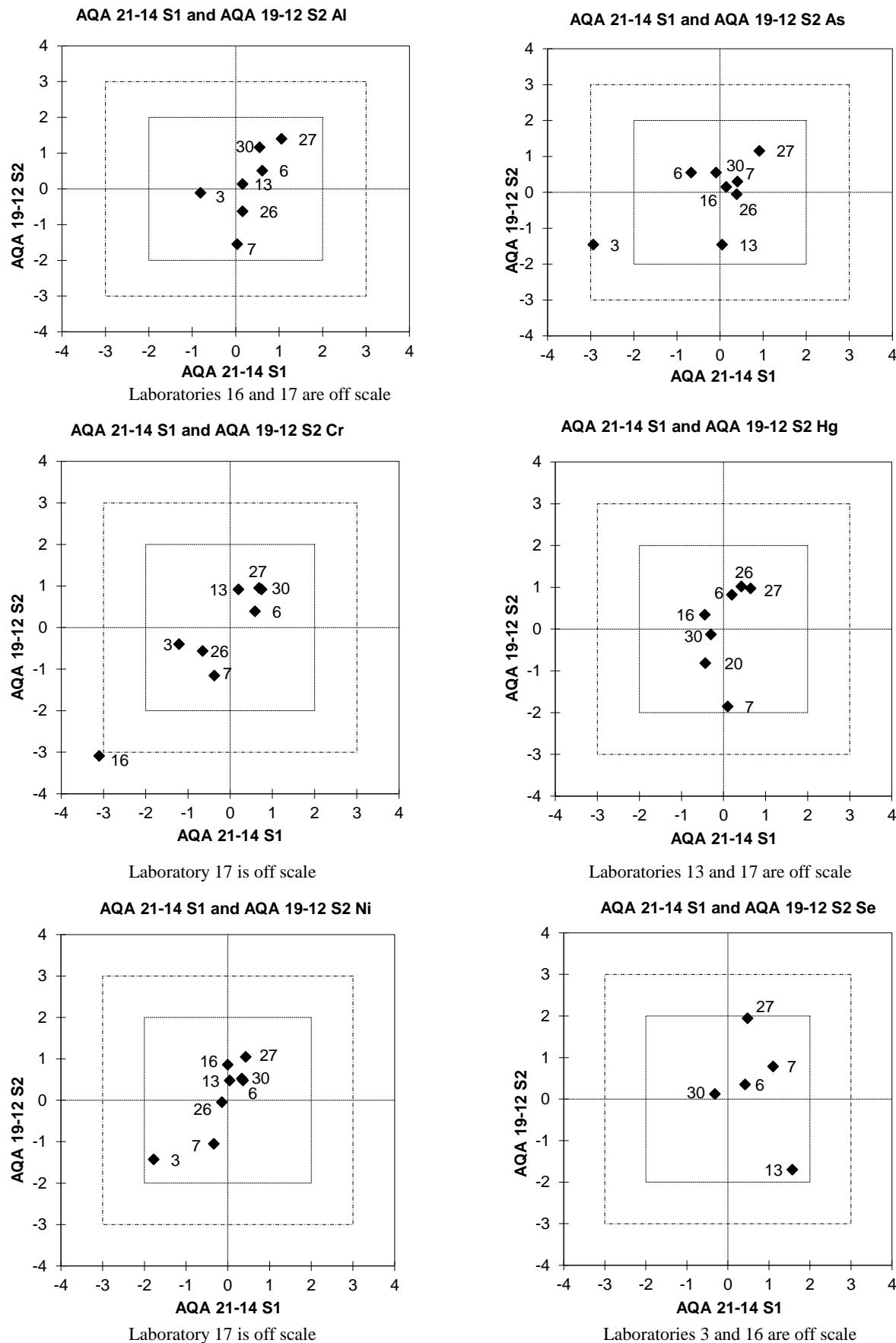


Figure 67 Scatter Plots of: z-Score in S1 of AQA 21-14 and S2 of AQA 19-12

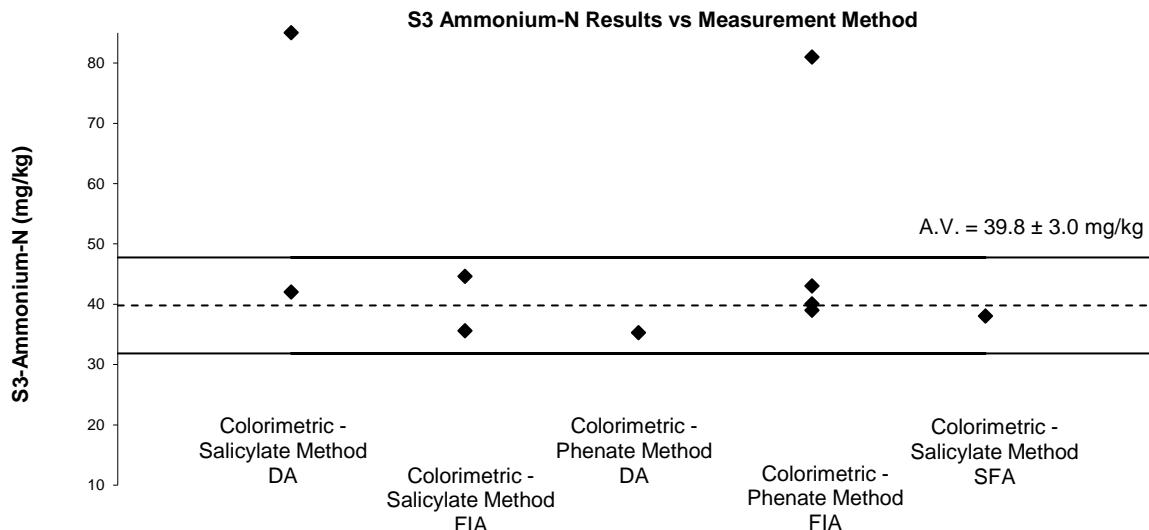
7.7 Participants' Results and Analytical Methods for 2M KCl Extractable Ammonium-N and Nitrate-N

Mineral nitrogen components, ammonium (NH_4^+), nitrite (NO_2^-) and nitrate (NO_3^-), are of particular interest when soil fertility is assessed. While water can extract NO_3^- -N and NO_2^- -N from a majority of soils, NH_4^+ -N has to be displaced by another cation when the surface soil colloids are negatively charged.²⁵ The participating laboratories were asked to analyse the sample using their normal measurement technique but to follow the preparation procedure for the soil extract which involved: a soil/2M KCl ratio of 1:10 and a mixing time of one hour.

The method descriptions provided by participants are presented in Table 3. All but two participants used a soil/2M KCl ratio of 1:10.

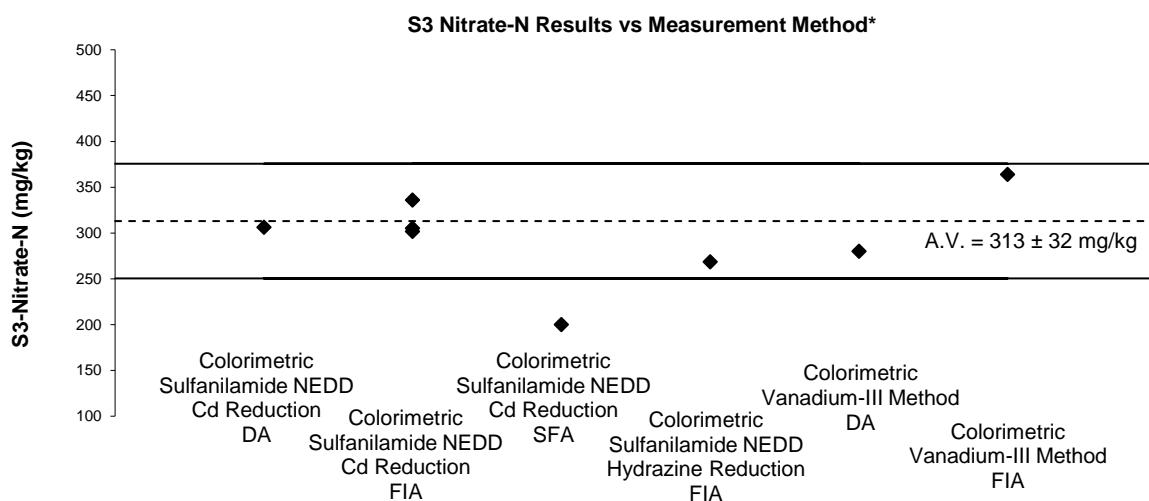
2M KCl Extractable Ammonium-Nitrogen Plots of participants' results versus the analytical methods and instrumental technique used are presented in Figure 68.

Typing errors and/or incorrect dilution factors may explain some of the high unsatisfactory ammonium-N results.



*Laboratory 16 result of 420 mg/kg has been plotted as 85 mg/kg.

Figure 68 S3- NH_4^+ -N Results vs. Analytical Method and Measurement Technique



*Laboratory 22 results of 28 mg/kg has been plotted as 200 mg/kg.

Figure 69: S3- NO_3^- -N Results vs. Measurement Technique

2M KCl Extractable Nitrate-Nitrogen The measurement method used by most laboratories involved NO_3^- -N reduction to NO_2^- -N by passage of the clarified soil extract through a Cd-Cu reduction column followed by NO_x (the reduced NO_2^- -N plus original NO_2^- -N) measurements. NO_x was determined colorimetrically based on Griess-Ilosvay reaction and NO_3^- -N calculated by subtracting NO_2^- -N value (obtained by analysis without passing the sample through the Cd-Cu reduction column), from the NO_x value.

Two laboratories used trivalent V for NO_3^- -N reduction to NO_2^- -N (Figure 69).

7.8 Participants' Results and Analytical Methods for Total Kjeldahl Nitrogen

TKN assigned value was 2480 mg/kg. Of 15 results reported for TKN in S3 only one returned an unsatisfactory z-score. Plots of participants' results versus analytical method and measurement technique are presented in Figure 70.

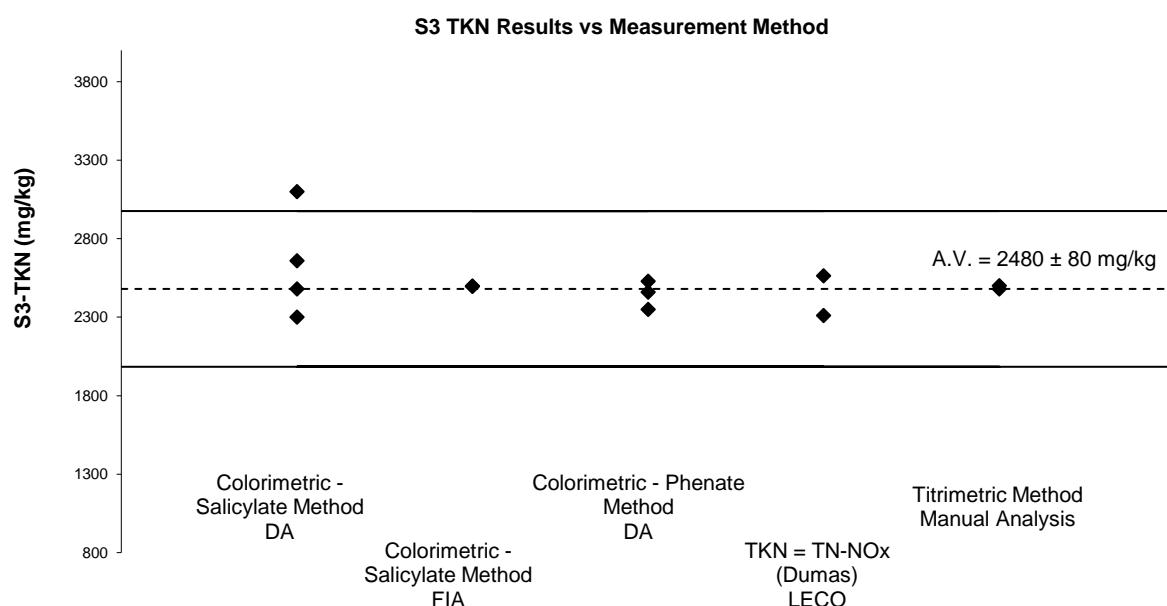


Figure 70 S3-TKN Results vs. Measurement Technique

Interferences from the coloured species in the extract might be the cause of the high, unsatisfactory TKN z-scores.

7.9 Participants' Results and Analytical Methods for Water Soluble Anions

Measurement of water soluble anions in soil is an empirical measurement – where the method of extraction defines the measurand.^{25, 26} With testing laboratories using different methods, each could be considered to be measuring a different measurand that is their version of ‘water soluble anions in soil’. This lack of uniformity in the procedures can make the comparison of participants’ results difficult.

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

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

The method descriptions and instrumental techniques provided by participants are presented in Tables 4 to 9. All, but one laboratory, used a soil/water ratio of 1:5.

Individual Water Soluble Anion Commentary

Bromide. Only 5 participants reported results for bromide in S3. The results were in relatively good agreement with each other (CV17%) centred on a value of 1.97 mg/kg.

All participants used the Ion Chromatographic Method to measure bromide.

Chloride Fifteen participants reported results for chloride and 11 performed satisfactorily. Figure 71 presents a plot of participants' results versus measurement method and instrumental technique used for chloride analysis in S3.

Of 4 unsatisfactory results, 2 were from the ferricyanide colorimetric method by DA.

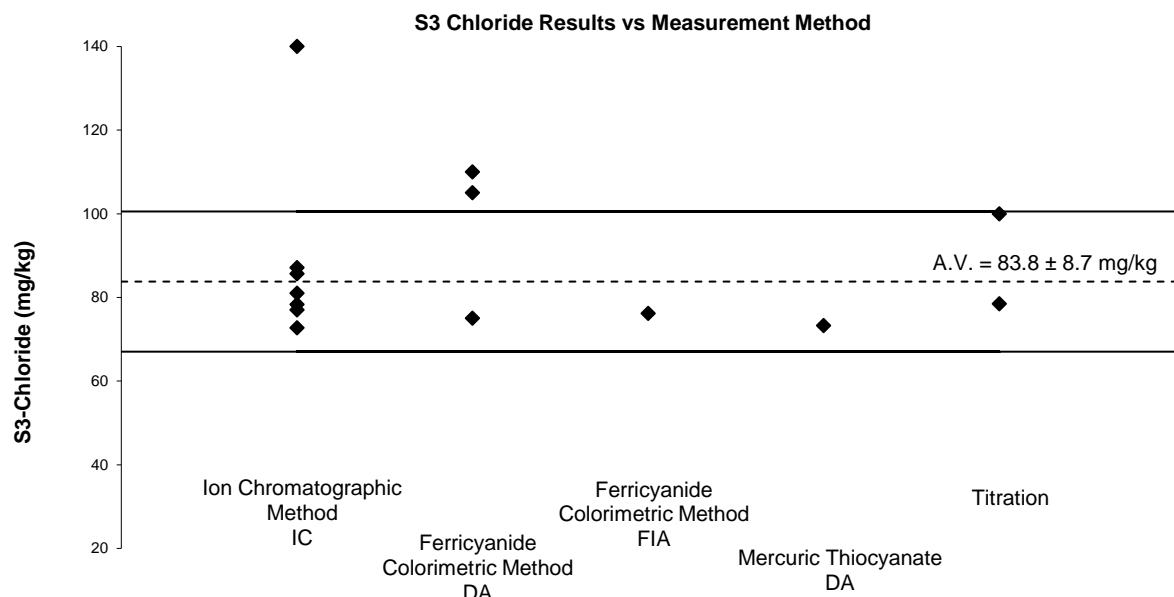


Figure 71 S3-Chloride Results vs. Measurement Method

Fluoride All but one participant who reported results for fluoride in S3 performed satisfactorily. Participants' results versus the methods used are presented in Figure 72.

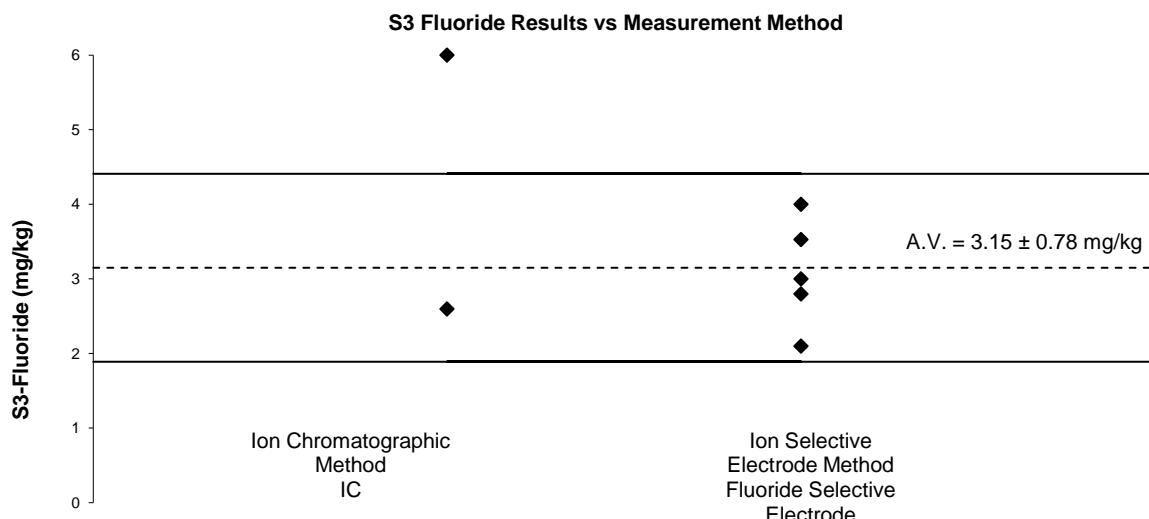


Figure 72 S3-Fluoride Results vs. Measurement Method

The high fluoride results from IC measurements may be explained by interferences from simple organic acids, bromates and chlorates found in soil samples.

Iodide Only two participants reported results for iodide in S3, both used ion chromatographic method.

Orthophosphate-P Participants used a wide variety of measurement methods and instrumental techniques (Figure 73). Ascorbic acid colorimetric method was the most popular method used by participants for the measurement of orthophosphate-P.

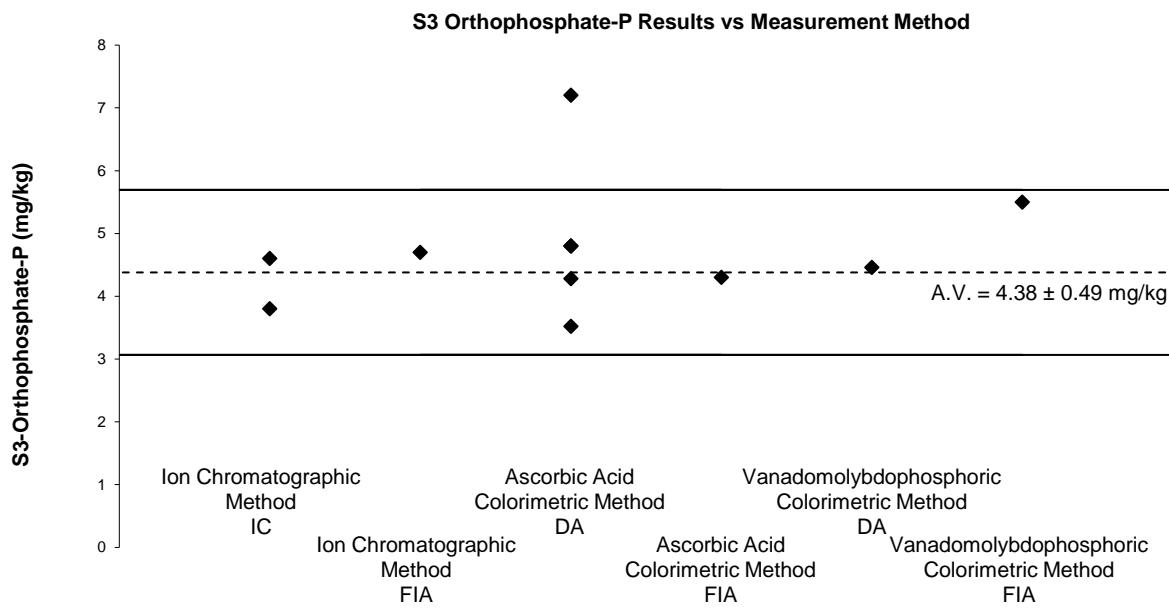


Figure 73 S3-Orthophosphate-P Results vs. Method

Sulphate Plots of participants' results versus the instrumental technique used are presented in Figure 74. Although most of the S in soil samples is from sulphate compounds, false positive results can be produced when this is measured by ICP-OES: this technique measures total S and not only S from sulphate compounds.

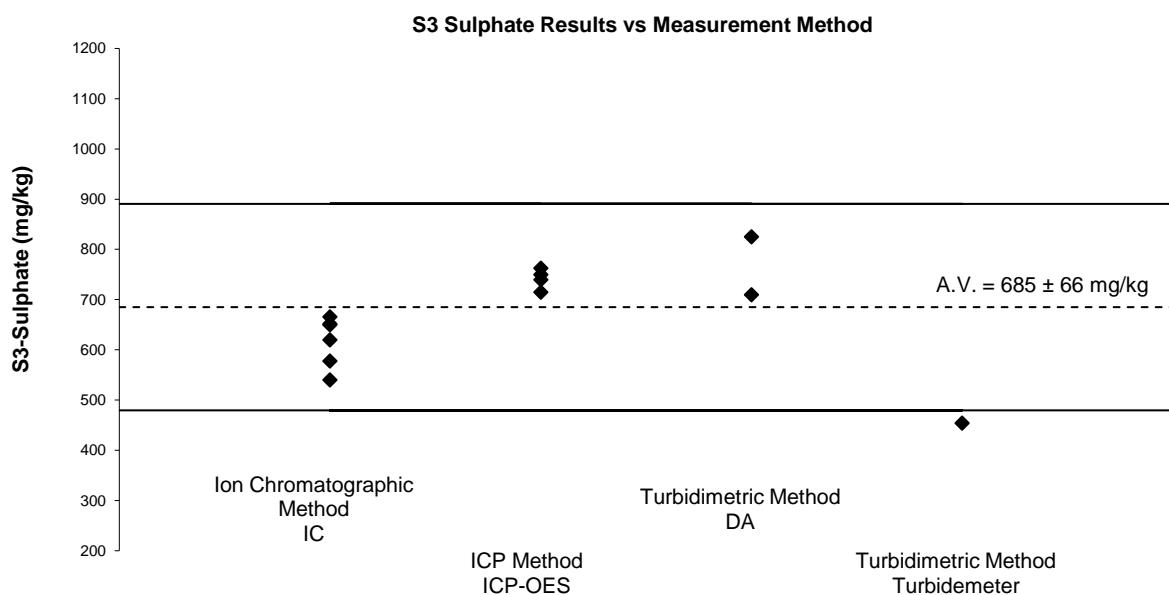


Figure 74 S3-Sulphate Results vs. Measurement Method and Instrumental Technique

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

AQA 21-14 is the twenty-ninth NMI proficiency test of metals in soil. For most of the analytes the same fixed target standard deviation was used in the present study as in the previous studies of metals in soil. This allowed a comparison of participants' performance (z-score) over time and provided a benchmark for progressive improvement.

Participants' performance in the measurement of inorganic analytes in soil over time is presented in Figure 75. On average, participants' performance has remained fairly consistent over time.

Over time laboratories should expect at least 95% of its scores to lay within the range $|z| \leq 2.0$. Scores in the range $2.0 < |z| < 3.0$ occasionally can occur, however these should be interpreted in conjunction with the other scores obtained by that laboratory. For example, a trend of z-scores on one side of the zero line are an indication of method or laboratory bias.

Individual performance history reports are emailed to each participant at the end of the study; the consideration of z-scores for an analyte over time provides much more useful information than a single z-score.

7.11 Reference Materials and Certified Reference Materials

Participants reported whether control samples (spiked samples, certified reference materials-CRMs or matrix specific reference materials-RMs) had been used (Table 73).

Table 73 Control Samples Used by Participants

| Lab. Code | Description of Control Samples |
|-----------|--|
| 1 | CRM |
| 2 | CRM |
| 3 | CRM |
| 4 | CRM |
| 5 | SS |
| 6 | RM – AGAL 12, In House AG reference |
| 7 | RM |
| 8 | CRM – Choice Analytical CRMs |
| 9 | CRM – ASPAC 7098 and ASPAC 6052 |
| 10 | SS |
| 11 | Trace Metals - Sandy Loam 10. Lot# LRAA7890 |
| 12 | CRM – SQC014: Nutrients in Soil for TKN and Ammonia as N |
| 13 | CRM – CRM036 |
| 14 | CRM – PACS2 Marine Sediment |
| 15 | SS – Novachem SQC001S |
| 16 | SS |
| 17 | AGAL 12 & LOAM B |
| 18 | CRM – ICV 1, ICV 3, AGAL 10, AGAL 12, CRM540 |
| 19 | RM – VHG-SL1-50g LOT#711217475 |
| 20 | CRM – AGAL-10 |
| 23 | AGAL 10 / AGAL 12 |
| 25 | CRM |
| 26 | Agal-12 Biosoil |

| Lab. Code | Description of Control Samples |
|-----------|---|
| 27 | RM |
| 28 | CRM |
| 29 | AGAL 10 & AGAL 12 |
| 30 | RM – AQA 20-13 S1,S2 and In house QC soil samples |

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

Some laboratories reported using certified reference materials. These materials may not meet the internationally recognised definition of a Certified Reference Material:

'a reference material, accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceabilities, using valid procedures'²⁷

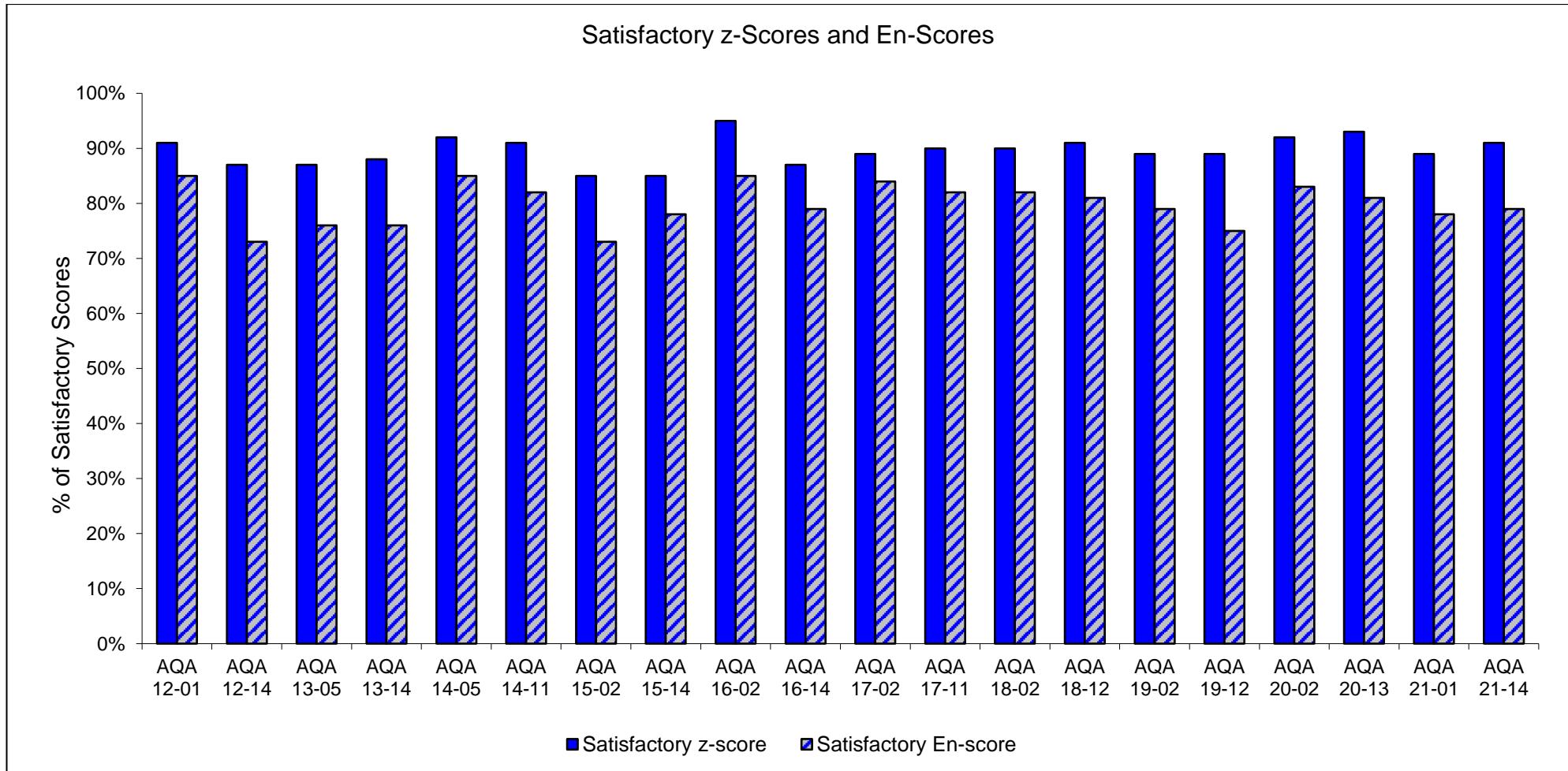


Figure 75 Participants' Performance over Time

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APPENDIX 1 - SAMPLE PREPARATION, ANALYSIS AND HOMOGENEITY TESTING

Sample Preparation

Samples S1 was sediment material. This sample was previously distributed as sample S2 for AQA 19-12. The preparation procedure of this sample is provided in the report for AQA 19-12.

Samples S2 was a soil sample fortified for 17 elements, dried, ground, and passed through a 350 µm sieve. The fortified soil was further mixed and divided into portions of 25 g each.

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

Sample Analysis and Homogeneity Testing

The same preparation procedure as in previous NMI PT studies for inorganic analytes in soil was followed for Samples S1, S2 and S3. Partial homogeneity testing was conducted for the elements of interest with the exception of Sb and Rb in S2 and EC, pH, 2M KCl extractable nitrate-N and S in S3. Three bottles were analysed in duplicate and the average of these results was reported as the homogeneity value. Measurements were made under repeatability conditions in random order.

Sample Analysis for Acid Extractable Elements

A test portion of approximately 0.5 g of soil was weighed into a 50 mL graduated polypropylene centrifuge tube. The sample was digested using 3 mL of concentrated nitric acid and 3 mL of concentrated hydrochloric acid on a hot block at 95°C ± 5°C. After digestion, each sample was diluted to 40 mL with Milli-Q water and then further diluted as necessary.

The measurement instrument was calibrated using external standards for targeted analytes. A set of quality control samples consisting of blanks, blank matrix spike, and matrix matched reference materials, duplicates and sample matrix spikes, was carried through the same set of procedures and analysed at the same time as the samples. A summary of the instrument conditions used and the ion/wavelength monitored for each analyte is given in Table 74.

Table 74 Instrumental Technique used for Acid Extractable Elements

| Analyte | Instrument | Internal Standard | Reaction/Collision Cell | Cell Mode/Gas | S1/2 Final Dilution Factor | S3 Final Dilution Factor | Ion (m/z)/Wavelength (nm) |
|---------|------------|-------------------|-------------------------|---------------|----------------------------|--------------------------|---------------------------|
| Ag | ICP-MS | Rh | ORS | He | 800 | NA | 107 m/z |
| Al | ICP-MS | Rh | NA | NA | 800 | NA | 27 m/z |
| As | ICP-MS | Rh | ORS | He | 800 | NA | 75 m/z |
| B | ICP-MS | Rh | NA | NA | 800 | NA | 11 m/z |
| Ba | ICP-MS | Rh | ORS | He | 800 | NA | 137 m/z |
| Be | ICP-MS | Rh | NA | NA | 800 | NA | 9 m/z |
| Bi | ICP-MS | Ir | NA | He | 800 | NA | 209 m/z |
| Ca | ICP-MS | Rh | ORS | He | NA | 800 | 43 m/z |
| Cd | ICP-MS | Rh | NA | NA | 800 | NA | 111 m/z |
| Co | ICP-MS | Rh | ORS | He | 800 | NA | 59 m/z |
| Cr | ICP-MS | Rh | ORS | He | 800 | NA | 52 m/z |
| Cs | ICP-MS | Rh | ORS | He | 800 | NA | 113 m/z |
| Cu | ICP-MS | Rh | ORS | He | 800 | NA | 65 m/z |
| Fe | ICP-MS | Rh | NA | NA | NA | 800 | 56 m/z |
| Ga | ICP-MS | Rh | ORS | He | 800 | NA | 71 m/z |
| Hg | ICP-MS | Rh | NA | NA | 800 | NA | 201 m/z |
| K | ICP-MS | Rh | ORS | He | NA | 800 | 39 m/z |

| Analyte | Instrument | Internal Standard | Reaction/Collision Cell | Cell Mode/Gas | S1/2 Final Dilution Factor | S3 Final Dilution Factor | Ion (m/z)/Wavelength (nm) |
|---------|------------|-------------------|-------------------------|---------------|----------------------------|--------------------------|------------------------------|
| La | ICP-MS | Rh | ORS | He | 800 | NA | 139 m/z |
| Li | ICP-MS | Rh | ORS | He | 800 | NA | 7 m/z |
| Mg | ICP-MS | Rh | ORS | He | NA | 800 | 24 m/z |
| Mn | ICP-MS | Rh | ORS | He | 800 | NA | 55 m/z |
| Mo | ICP-MS | Rh | ORS | He | 800 | NA | 95 m/z |
| Na | ICP-MS | Rh | ORS | He | NA | 800 | 23 m/z |
| Ni | ICP-MS | Rh | ORS | He | 800 | NA | 60 m/z |
| P | ICP-MS | Rh | ORS | HEHe | NA | 800 | 31 m/z |
| Pb | ICP-MS | Ir | NA | NA | 800 | NA | Average of 206, 207, 208 m/z |
| Se | ICP-MS | Rh | ORS | HEHe | 800 | NA | 78 m/z |
| Sn | ICP-MS | Rh | NA | NA | 800 | NA | 118 m/z |
| Sr | ICP-MS | Rh | ORS | He | NA | 800 | 88 m/z |
| Th | ICP-MS | Ir | ORS | He | 800 | NA | 232 m/z |
| Tl | ICP-MS | Ir | ORS | He | 800 | NA | 205 m/z |
| U | ICP-MS | Ir | NA | NA | 800 | NA | 238 m/z |
| V | ICP-MS | Rh | ORS | He | 800 | NA | 51 m/z |
| Zn | ICP-MS | Rh | ORS | He | 800 | NA | 66 m/z |

Sample Analysis for Water Soluble Anions

A test portion of 10 g was weighed into a 50 mL polypropylene container. The container was then filled with deionised water. The suspension was shaken, at room temperature for 1 h, centrifuged, and filtered through 0.45 µm filter. A summary of the measurement methods and instrumental techniques is presented in Table 75.

Table 75 Summary of the Measurement Methods and Instrumental Techniques used by NMI

| Anion | Measurement Method | Instrument |
|--------------------------------|---------------------------------------|-----------------|
| Total Kjeldahl Nitrogen | Titrimetric Method | Manual Analysis |
| Water Soluble Bromide | Ion Chromatographic Method | IC |
| Water Soluble Chloride | Ion Chromatographic Method | IC |
| Water Soluble Fluoride | Ion Selective Electrode Method | IC |
| Water Soluble Orthophosphate-P | Colorimetric, Ascorbic Acid Reduction | DA |
| Water Soluble Sulphate | Ion Chromatographic Method | IC |

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

The assigned value was calculated as the robust average using the procedure described in ‘ISO13528:2015(E), Statistical methods for use in proficiency testing by inter-laboratory comparisons – Annex C’.⁶ The uncertainty was estimated as:

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

where:

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

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

A worked example is set out below in Table 76.

Table 76 Uncertainty of Assigned Value for As in Sample S1

| | |
|-----------------|------------|
| No. results (p) | 21 |
| Robust Average | 20.9 mg/kg |
| $S_{rob\ av}$ | 1.9 mg/kg |
| $u_{rob\ av}$ | 0.51 mg/kg |
| k | 2 |
| $U_{rob\ av}$ | 1.0 mg/kg |

The assigned value for As in Sample S1 is **20.9 ± 1.0 mg/kg**.

z-Score and E_n-score

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

A worked example is set out below in Table 77.

Table 77 z-Score and E_n-score for As Result Reported by Laboratory 1 in S1

| As Result mg/kg | Assigned Value mg/kg | Set Target Standard Deviation | z-Score | E _n -Score |
|-----------------|----------------------|--|---|---|
| 21 ± 3.15 | 20.9 ± 1.0 | 10% as CV or 0.10x20.9 = =2.1 mg/kg | $z = \frac{(21 - 20.9)}{2.1}$ $z = 0.05$ | $E_n = \frac{(21 - 20.9)}{\sqrt{3.15^2 + 1.0^2}}$ $E_n = 0.03$ |

APPENDIX 3 - USING PT DATA FOR UNCERTAINTY ESTIMATION

When a laboratory has successfully participated in at least 6 proficiency testing studies, the standard deviation from proficiency testing studies can also be used to estimate the uncertainty of their measurement results.^{10, 12} An example is given. Between 2009 and 2021 NMI carried out 24 proficiency tests of metals in soil. These studies involved analyses of acid-extractable elements at low and high levels in dried soil, moist soil, biosoil, sediment and sludge.

Laboratory X submitted results for As in all of these PTs. All reported results returned satisfactory z-scores. This data can usefully be separated into two ranges of results 0.5 to 10 mg/kg and 10 to 100 mg/kg (Tables 78 and 79).

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

Table 78 Laboratory X Reported Results for As at 0.5 to 10 mg/kg Level.

| Study No. | Sample | Laboratory result mg/kg | Assigned value* mg/kg | Robust CV of all results (%) | Number of Results |
|-----------|-------------------|----------------------------|--------------------------|------------------------------------|----------------------|
| AQA 09-13 | S1 - Biosoil | 4.091 | 3.64 | 16 | 11 |
| | S2 - Soil | 4.29 | 4.57 | 15 | 12 |
| AQA 11-01 | S1 - Biosoil | 3.54 | 3.57 | 19.7 | 18 |
| AQA 13-05 | S1 - Soil | 9.22 | 9.21 | 14 | 22 |
| AQA 14-11 | S1 - Sediment | 7.91 | 7.37 | 11.8 | 21 |
| AQA 15-02 | S1 - Moist Sludge | 8.29 | 7.02 | 13 | 22 |
| | S2 - Moist Sludge | 7.42 | 7.02 | 11.3 | 17 |
| AQA 15-14 | S1 - Sediment | 10 | 9.95 | 6.7 | 17 |
| | S2 - Soil | 4.53 | 4.47 | 6.4 | 14 |
| AQA 16-02 | S2 - Clay | 2.67 | 2.11 | 14 | 20 |
| AQA 16-14 | S1 - Soil | 6.03 | 5.61 | 20 | 17 |
| AQA 17-02 | S2 - Soil | 3.71 | 3.76 | 10 | 13 |
| AQA 18-02 | S1 - Compost | 2.22 | 2.73 | 11 | 17 |
| AQA 19-02 | S1 - Soil | 2.83 | 2.65 | 11 | 24 |
| AQA 19-12 | S1 - Soil | 2.32 | 2.12 | 16 | 16 |
| AQA 20-13 | S1 - Biosoil | 2.85 | 3.29 | 11 | 17 |
| AQA 21-01 | S1 - Sediment | 7.02 | 6.26 | 6.9 | 18 |
| AQA 21-01 | S2 - Moist Sludge | 3.99 | 3.58 | 12.6 | 13 |
| Average | | | | 12.1** | |

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

Table 79 Laboratory X Reported Results for As at 10 to 100 mg/kg Level.

| Study No. | Sample | Laboratory result mg/kg | Assigned value* mg/kg | Robust CV of all results (%) | Number of Results |
|-----------|-------------------|-------------------------|-----------------------|------------------------------|-------------------|
| AQA 10-12 | S1 - Soil | 16.6 | 14.4 | 8.5 | 19 |
| AQA 11-12 | S1 - Moist Sludge | 25 | 21.6 | 15 | 13 |
| AQA 12-01 | S1 - Sediment | 18.4 | 17.3 | 8.1 | 21 |
| AQA 12-14 | S2 - Soil | 16.6 | 14.8 | 11 | 20 |
| AQA 13-14 | S1 - Sandy Soil | 16.6 | 15.1 | 10.4 | 21 |
| AQA 14-05 | S1 - Soil | 13.2 | 12.3 | 7.8 | 25 |
| AQA 17-11 | S1 - Sediment | 18.1 | 17.4 | 11 | 22 |
| AQA 18-12 | S2 - Soil | 10.4 | 9.6 | 8 | 20 |
| AQA 19-12 | S2 - Sediment | 21 | 19.9 | 9 | 19 |
| AQA 20-02 | S1 - Soil | 18.8 | 21.6 | 8.8 | 23 |
| AQA 20-02 | S2 - Moist Soil | 16.5 | 17.8 | 6.7 | 24 |
| AQA 21-14 | S1 - Sediment | 19.5 | 20.9 | 8.9 | 21 |
| Average | | | | 9.4** | |

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

Table 80 sets out the expanded uncertainty for results of the measurement of As in soil, biosoil, clay, sediment, sludge, sandy soil, moist soil, compost and agricultural soil over the ranges 0.5 to 10 mg/kg and 10 to 100 mg/kg.

Table 80 Uncertainty of As Results Estimated Using PT Data.

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

The estimates of 25% and 19% relative passes the test of being reasonable, and the analysis of the 30 different PT samples over twelve years can be assumed to include all the relevant uncertainty components (different matrices, operators, reagents, calibrators etc.), and so complies with ISO 17025:2018.⁸

APPENDIX 4 - INSTRUMENT DETAILS

Table 81 Instrument Conditions Ag

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | NA | NA | NA | NA | NA | NA | NA |
| 3 | | | | | | NA | |
| 4 | ICP-OES-RV | | | | | NA | 328.068 |
| 5 | ICP-MS | Rh | CRI | He | 500 | NA | 107 |
| 6 | ICP-MS | Rh | NA | NA | 625 | NA | 109 |
| 7 | NA | NA | NA | NA | NA | NA | NA |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 328.069nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 103 | ORS | He | 50 | | 107 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-MS | In | NA | | 1000 | NA | 107 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | In | ORS | He | 0.1 | NA | 107 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 328.068 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 328.068 |
| 18 | ICP-MS | Rh | ORS | He | 500 | NA | 107 |
| 19 | ICP-MS | Y 89 | KED | | NA | NA | 107 |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | ICP-MS | Rh | ORS | He | 800 | NA | 107 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | 328.068 |
| 26 | ICP-MS | Rh | NA | standard mode | 400 | NA | 109 |
| 27 | ICP-MS | Rh | NA | | NA | 250 | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Rh | ORS | He | 500 | NA | 107 |
| 30 | ICP-MS/MS | Rh103 | ORS | O2 | 4000 | NA | Ag 107/107(m/z) |

Table 82 Instrument Conditions Al

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-OES-AV | Scandium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | NA | NA | NA | NA | NA | NA | NA |
| 5 | ICP-MS | Sc | CRI | He | 500 | NA | 27 |
| 6 | ICP-MS | Sc | KED | He | 625 | NA | 27 |
| 7 | ICP-OES-AV | Eu | NA | NA | 50 | NA | 237 |
| 8 | ICP-OES | Eu & Cs | NA | NA | 50 | NA | 236.707, 308.215, 396.15nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 72 | | standard mode | 50 | | 27 |
| 11 | ICP-MS | Rhodium | KED | He | 1000 | NA | 26.982 |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | NA | 394.401 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-OES-AV | Lu | | | 0.1 | NA | 237.312 |
| 16 | ICP-OES-AV | NA | NA | | 10 | NA | 396.152 |
| 17 | ICP-OES-RV | Yttrium | | | 250 | NA | 396.153 |
| 18 | ICP-MS | Sc | ORS | He | 500 | NA | 27 |
| 19 | ICP-MS | Sc-2 45 | KED | | 1000 | NA | 27 |
| 20 | | | | | | NA | |
| 21 | ICP-OES-AV | | | | | NA | |
| 22 | NA | NA | NA | NA | NA | NA | NA |
| 23 | ICP-MS | Yes | | | | NA | 27 |
| 24 | | | | | | NA | |
| 25 | NA | NA | NA | NA | NA | NA | NA |
| 26 | ICP-MS | Sc | NA | standard mode | 2000 | NA | 27 |
| 27 | ICP-MS | Sc | NA | | NA | 250 | |
| 28 | NA | NA | NA | NA | NA | NA | NA |
| 29 | ICP-MS | Sc | ORS | He | 5000 | NA | 27 |
| 30 | ICP-OES-AV | In 303.936 | NA | NA | 80 | NA | Al 237.312 |

Table 83 Instrument Conditions As

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | NA | NA | NA | NA | NA | NA | NA |
| 5 | ICP-MS | Rh | CRI | He | 500 | NA | 75 |
| 6 | ICP-MS | Ge | KED | He | 625 | NA | 75 |
| 7 | ICP-MS | Sc | ORS | He | 50 | NA | 75 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 188.89nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 72 | ORS | He | 50 | | 75 |
| 11 | ICP-MS | Rhodium | KED | He | 1000 | NA | 74.922 |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | NA | 188.979 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Ge | ORS | He | 0.1 | NA | 75 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 188.98 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 188.979 |
| 18 | ICP-MS | Y | ORS | He | 500 | NA | 75 |
| 19 | ICP-MS | Ge-1 72 | KED | | 10 | NA | 75 |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | NA | NA | NA | NA | NA | NA | NA |
| 23 | ICP-MS | Yes | KED | He | | NA | 75 |
| 24 | | | | | | NA | |
| 25 | NA | NA | NA | NA | NA | NA | NA |
| 26 | ICP-MS | Rh | KED | He | 1000 | NA | 75 |
| 27 | ICP-MS | Rh | UC | He | 250 | 250 | |
| 28 | NA | NA | NA | NA | NA | NA | NA |
| 29 | ICP-MS | Rh | ORS | He | 500 | NA | 75 |
| 30 | ICP-OES-AV | In 303.936 | NA | NA | 80 | NA | As 188.980 |

Table 84 Instrument Conditions B

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-OES-AV | Scandium | | | | NA | |
| 2 | NA | NA | NA | NA | NA | NA | NA |
| 3 | | | | | | NA | |
| 4 | ICP-OES-RV | | | | | NA | 249.772 |
| 5 | ICP-MS | Sc | CRI | NA | 500 | NA | 11 |
| 6 | ICP-MS | Sc | NA | NA | 625 | NA | 10 |
| 7 | NA | NA | NA | NA | NA | NA | NA |
| 8 | ICP-OES | Eu & Cs | NA | NA | 50 | NA | 249.773nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 89 | | standard mode | 10 | | 11 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | NA | 208.957 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Li6 | NA | standard mode | 0.1 | NA | 11 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 249.772 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 249.677 |
| 18 | ICP-MS | Sc | ORS | He | 500 | NA | 11 |
| 19 | ICP-MS | Y 89 | KED | | 10 | NA | 11 |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | ICP-MS | Rh | ORS | NA | 800 | NA | 11 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | 249.678 |
| 26 | ICP-MS | Sc | KED | He | 400 | NA | 11 |
| 27 | ICP-MS | Sc | NA | | 250 | NA | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Sc | ORS | He | 500 | NA | 11 |
| 30 | ICP-OES-AV | In 303.936 | NA | NA | 80 | NA | B 249.678 |

Table 85 Instrument Conditions Ba

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-OES-AV | Scandium | | | | NA | |
| 2 | NA | NA | NA | NA | NA | NA | NA |
| 3 | | | | | | NA | |
| 4 | ICP-OES-RV | | | | | NA | 585.367 |
| 5 | ICP-MS | Rh | CRI | He | 500 | NA | 135 |
| 6 | ICP-MS | Rh | NA | NA | 625 | NA | 138 |
| 7 | NA | NA | NA | NA | NA | NA | NA |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 585.369nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 159 | ORS | He | 50 | | 137 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | NA | 455.403 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-OES-AV | Lu | | | 0.1 | NA | 455.403 |
| 16 | ICP-OES-AV | NA | NA | | 10 | NA | 455.403 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 249.677 |
| 18 | ICP-MS | Rh | ORS | He | 500 | NA | 135 |
| 19 | ICP-MS | In-115 | KED | | 10 | | 138 |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | ICP-MS | Rh | ORS | He | 800 | NA | 137 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | 455.403 |
| 26 | ICP-MS | Tb | NA | standard mode | 400 | NA | 137 |
| 27 | ICP-MS | In | NA | | 250 | 250 | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Sc | ORS | He | 500 | NA | 135 |
| 30 | ICP-OES-AV | Eu 390.711 | NA | NA | 80 | NA | Ba 455.403 |

Table 86 Instrument Conditions Be

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | NA | NA | NA | NA | NA | NA | NA |
| 3 | | | | | | NA | |
| 4 | ICP-OES-RV | | | | | NA | 585.367 |
| 5 | ICP-MS | Sc | CRI | NA | 500 | NA | 9 |
| 6 | ICP-MS | Sc | NA | NA | 625 | NA | 9 |
| 7 | NA | NA | NA | NA | NA | NA | NA |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 313.042nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 72 | | standard mode | 50 | | 9 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-MS | Sc | NA | | 1000 | NA | 9 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Li6 | ORS | He | 0.1 | NA | 9 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 234.861 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 313.107 |
| 18 | ICP-MS | Sc | ORS | He | 500 | NA | NA |
| 19 | ICP-MS | Sc-2 45 | KED | | 10 | NA | 9 |
| 20 | | | | | | NA | |
| 21 | | | | | | NA | |
| 22 | ICP-MS | Rh | ORS | NA | 800 | NA | 9 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | 234.861 |
| 26 | ICP-MS | Sc | NA | standard mode | 400 | NA | 9 |
| 27 | ICP-MS | Sc | NA | | 250 | NA | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Sc[No Gas] | ORS | He | 500 | NA | 9 |
| 30 | ICP-OES-AV | Lu307.760 | NA | NA | 80 | NA | Be 313.107 |

Table 87 Instrument Conditions Bi

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | | | | | | NA | |
| 2 | NA | NA | NA | NA | NA | NA | NA |
| 3 | | | | | | NA | |
| 4 | ICP-MS | Rh, Sc, Ir | ORC | He | NA | NA | 209 |
| 5 | ICP-MS | Lu | CRI | He | 500 | NA | 209 |
| 6 | | | | | | NA | |
| 7 | NA | NA | NA | NA | NA | NA | NA |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 209 m/z |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 159 | | standard mode | 50 | | 209 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | | | | | | NA | NT |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Ir | ORS | He | 0.1 | NA | 209 |
| 16 | | | | | | NA | |
| 17 | NA | | | | | NA | |
| 18 | ICP-MS | Lu | ORS | He | 500 | NA | 209 |
| 19 | NA | NA | NA | NA | NA | NA | NA |
| 20 | | | | | | NA | |
| 21 | | | | | | NA | |
| 22 | ICP-MS | Ir | ORS | He | 800 | NA | 209 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | NA |
| 26 | ICP-MS | Tb | NA | standard mode | 400 | NA | 209 |
| 27 | ICP-MS | Ir | NA | | NA | 250 | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Lu | ORS | He | 500 | NA | 209 |
| 30 | ICP-MS/MS | Ir 193 | ORS | O2 | 4000 | NA | Bi 209/209(m/z) |

Table 88 Instrument Conditions Ca

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-OES-AV | Scandium | | | NA | | |
| 2 | NA | NA | NA | NA | NA | | NA |
| 3 | NA | NA | NA | NA | NA | NA | NA |
| 4 | NA | NA | NA | NA | NA | | NA |
| 5 | NA | NA | NA | NA | NA | NA | NA |
| 6 | ICP-MS | Sc | KED | He | NA | 625 | 44 |
| 7 | ICP-MS | Sc | ORS | He | NA | 50 | 44 |
| 8 | ICP-OES | Eu & Cs | NA | NA | NA | 50 | 315.887, 370.602nm |
| 9 | | | | | NA | | |
| 10 | ICP-MS | 89 | ORS | H2 | | 10 | 40 |
| 11 | | | | | NA | | |
| 12 | | | | | NA | | |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | | 315.887 |
| 14 | | | | | NA | | |
| 15 | ICP-OES-RV | Lu | | | NA | 0.1 | 422.673 |
| 16 | AAS | NA | NA | | NA | 10 | 422.7 |
| 17 | ICP-OES-RV | Yttrium | | | NA | 250 | 315.887 |
| 18 | NA | NA | NA | NA | NA | NA | NA |
| 19 | NA | NA | NA | NA | NA | NA | NA |
| 20 | | | | | NA | | |
| 21 | ICP-MS | | CRI | He | NA | | |
| 22 | ICP-OES-RV | Y | NA | NA | NA | 800 | 422.673 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | NA | NA | NA | NA | NA | 100 | NA |
| 26 | NA | NA | NA | NA | NA | NA | NA |
| 27 | NA | NA | NA | NA | NA | NA | NA |
| 28 | NA | NA | NA | NA | NA | | NA |
| 29 | NA | NA | NA | NA | NA | 500 | NA |
| 30 | NA | NA | NA | NA | NA | NA | NA |

Table 89 Instrument Conditions Cd

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | ICP-OES-RV | | | | | NA | 226.502 |
| 5 | ICP-MS | Rh | CRI | He | 500 | NA | 111 |
| 6 | ICP-MS | Rh | NA | NA | 625 | NA | 111 |
| 7 | ICP-MS | Rh | ORS | He | 50 | NA | 111 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 226.502nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 103 | ORS | He | 50 | | 111 |
| 11 | ICP-MS | Rhodium | KED | He | 1000 | NA | 110.904 |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-MS | In | NA | | 100 | NA | 111 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | In | ORS | He | 0.1 | NA | 111 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 226.502 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 226.502 |
| 18 | ICP-MS | Rh | DRC | He | 500 | NA | 114 |
| 19 | ICP-MS | In-115 | KED | | 10 | | 111 |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | ICP-MS | Rh | ORS | He | 800 | NA | 111 |
| 23 | ICP-MS | Yes | KED | He | | NA | 63 |
| 24 | | | | | | NA | |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | 228.802 |
| 26 | ICP-MS | Rh | KED | He | 1000/400 | NA | 111 |
| 27 | ICP-MS | Rh | NA | | 250 | 250 | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Rh | ORS | He | 500 | NA | 111 |
| 30 | ICP-OES-AV | Lu219.556 | NA | NA | 80 | NA | Cd 226.502 |

Table 90 Instrument Conditions Co

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | NA | NA | NA | NA | NA | NA | NA |
| 5 | ICP-MS | Sc | CRI | He | 500 | NA | 59 |
| 6 | ICP-MS | Ge | KED | He | 625 | NA | 59 |
| 7 | ICP-MS | Sc | ORS | He | 50 | NA | 59 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 228.616nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 103 | ORS | He | 50 | | 59 |
| 11 | ICP-MS | Rhodium | KED | He | 1000 | NA | 58.933 |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-MS | Sc | KED | He | 100 | NA | 59 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Ge | ORS | He | 0.1 | NA | 59 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 231.16 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 228.616 |
| 18 | ICP-MS | Sc | ORS | He | 500 | NA | 59 |
| 19 | ICP-MS | Sc 45 | KED | | 10 | | 59 |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | NA | NA | NA | NA | NA | NA | NA |
| 23 | ICP-MS | Yes | KED | He | | NA | 59 |
| 24 | | | | | | NA | |
| 25 | NA | NA | NA | NA | NA | NA | NA |
| 26 | ICP-MS | Ga | KED | He | 2000 | NA | 59 |
| 27 | ICP-MS | Rh | UC | He | NA | 250 | |
| 28 | NA | NA | NA | NA | NA | NA | NA |
| 29 | ICP-MS | Sc | ORS | He | 500 | NA | 59 |
| 30 | ICP-OES-AV | Lu219.556 | NA | NA | 80 | NA | Co 228.615 |

Table 91 Instrument Conditions Cr

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | NA | NA | NA | NA | NA | NA | NA |
| 5 | ICP-MS | Sc | CRI | He | 500 | NA | 52 |
| 6 | ICP-MS | Sc | KED | He | 625 | NA | 52 |
| 7 | ICP-MS | Sc | ORS | He | 50 | NA | 51 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 267.716nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 72 | ORS | He | 50 | | 52 |
| 11 | ICP-MS | Rhodium | KED | He | 1000 | NA | 51.941 |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | NA | 205.56 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-OES-AV | Lu | | | 0.1 | NA | 205.56 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 283.563 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 267.716 |
| 18 | ICP-MS | Sc | ORS | He | 500 | NA | 52 |
| 19 | ICP-MS | Sc 45 | KED | | 10 | | 52 |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | NA | NA | NA | NA | NA | NA | NA |
| 23 | ICP-MS | Yes | KED | He | | NA | 52 |
| 24 | | | | | | NA | |
| 25 | NA | NA | NA | NA | NA | NA | NA |
| 26 | ICP-MS | Sc | KED | He | 1000 | NA | 52 |
| 27 | ICP-MS | Sc | UC | He | 250 | 250 | |
| 28 | NA | NA | NA | NA | NA | NA | NA |
| 29 | ICP-MS | Sc | ORS | He | 500 | NA | 63 |
| 30 | ICP-MS/MS | Sc 45/61 | ORS | O2 | 1600 | NA | Cr 52/52(m/z) |

Table 92 Instrument Conditions Cs

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | | | | | | NA | |
| 2 | NA | NA | NA | NA | NA | NA | NA |
| 3 | | | | | | NA | |
| 4 | ICP-MS | Rh, Sc, Ir | ORS | He | NA | NA | |
| 5 | NA | NA | NA | NA | NA | NA | NA |
| 6 | | | | | | NA | |
| 7 | NA | NA | NA | NA | NA | NA | NA |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 133 m/z |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 159 | ORS | He | 50 | | 133 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | | | | | | NA | NT |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | | | | | | NA | |
| 16 | | | | | | NA | |
| 17 | NA | | | | | NA | |
| 18 | NA | NA | NA | NA | NA | NA | NA |
| 19 | NA | NA | NA | NA | NA | | NA |
| 20 | | | | | | NA | |
| 21 | | | | | | NA | |
| 22 | ICP-MS | Rh | ORS | He | 800 | NA | 133 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | NA |
| 26 | ICP-MS | Tb | NA | standard mode | 400 | NA | 133 |
| 27 | ICP-MS | Rh | NA | He | NA | 250 | |
| 28 | | | | | | NA | |
| 29 | NA | NA | NA | NA | 500 | NA | NA |
| 30 | NA | NA | NA | NA | NA | NA | NA |

Table 93 Instrument Conditions Cu

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | NA | NA | NA | NA | NA | NA | NA |
| 5 | ICP-MS | Sc | CRI | He | 500 | NA | 63 |
| 6 | ICP-MS | Ge | KED | He | 625 | NA | 63 |
| 7 | ICP-MS | Sc | ORS | He | 50 | NA | 63 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 327.395nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 103 | ORS | He | 50 | | 63 |
| 11 | ICP-MS | Rhodium | KED | He | 1000 | NA | 62.93 |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | NA | 324.752 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-OES-AV | Lu | | | 0.1 | NA | 324.754 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 324.754 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 324.752 |
| 18 | ICP-MS | Sc | ORS | He | 500 | NA | 63 |
| 19 | ICP-MS | Sc 45 | KED | | 10 | NA | 63 |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | NA | NA | NA | NA | NA | NA | NA |
| 23 | ICP-MS | Yes | KED | He | | NA | 63 |
| 24 | | | | | | NA | |
| 25 | NA | NA | NA | NA | NA | NA | NA |
| 26 | ICP-MS | Ga | KED | He | 1000 | NA | 63 |
| 27 | ICP-MS | Ga | UC | He | 250 | 250 | |
| 28 | NA | NA | NA | NA | NA | NA | NA |
| 29 | ICP-MS | Sc | ORS | He | 500 | NA | 52 |
| 30 | ICP-OES-AV | In 303.936 | NA | NA | 80 | NA | Cu 327.395 |

Table 94 Instrument Conditions Fe

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-OES-AV | Scandium | | | NA | | |
| 2 | NA | NA | NA | NA | NA | | NA |
| 3 | NA | NA | NA | NA | NA | NA | NA |
| 4 | NA | NA | NA | NA | NA | | NA |
| 5 | ICP-MS | Sc | CRI | He | NA | 500 | 56 |
| 6 | ICP-MS | Sc | KED | He | NA | 625 | 56 |
| 7 | ICP-MS | Sc | ORS | He | NA | 50 | 56 |
| 8 | ICP-OES | Eu & Cs | NA | NA | NA | 50 | 238.204, 258.588, 259.940nm |
| 9 | | | | | NA | | |
| 10 | ICP-MS | 103 | ORS | H2 | NA | 50 | 56 |
| 11 | ICP-MS | Rhodium | KED | He | NA | 1000 | 55.935 |
| 12 | | | | | NA | | |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | | 239.562 |
| 14 | | | | | NA | | |
| 15 | ICP-OES-AV | Lu | | | NA | 0.1 | 259.94 |
| 16 | ICP-OES-AV | NA | NA | | NA | 10 | 238.204 |
| 17 | ICP-OES-RV | Yttrium | | | NA | 250 | 259.939 |
| 18 | ICP-MS | Sc | ORS | He | NA | 500 | 56 |
| 19 | NA | NA | NA | NA | NA | NA | NA |
| 20 | | | | | NA | | |
| 21 | ICP-OES-AV | | | | NA | | |
| 22 | ICP-OES-RV | Y | NA | NA | NA | 800 | 238.204 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | NA | NA | NA | NA | NA | 100 | NA |
| 26 | NA | NA | NA | NA | NA | NA | NA |
| 27 | NA | NA | NA | NA | NA | NA | NA |
| 28 | NA | NA | NA | NA | NA | | NA |
| 29 | ICP-MS | Sc | ORS | He | NA | 5000 | 56 |
| 30 | NA | NA | NA | NA | NA | NA | NA |

Table 95 Instrument Conditions Ga

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | | | | | | NA | |
| 2 | NA | NA | NA | NA | NA | NA | NA |
| 3 | | | | | | NA | |
| 4 | ICP-MS | Rh, Sc, Ir | ORS | He | NA | NA | 71 |
| 5 | NA | NA | NA | NA | NA | NA | NA |
| 6 | | | | | | NA | |
| 7 | NA | NA | NA | NA | NA | NA | NA |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 71 m/z |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | NT | | | | | |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | | | | | | NA | NT |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | | | | | | NA | |
| 16 | | | | | | NA | |
| 17 | NA | | | | | NA | |
| 18 | NA | NA | NA | NA | NA | NA | NA |
| 19 | NA | NA | NA | NA | NA | NA | NA |
| 20 | | | | | | NA | |
| 21 | | | | | | NA | |
| 22 | ICP-MS | Rh | ORS | He | 800 | NA | 71 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | NA |
| 26 | NA | NA | NA | NA | NA | NA | NA |
| 27 | NA | NA | NA | | NA | NA | |
| 28 | | | | | | NA | |
| 29 | NA | NA | NA | NA | NA | NA | NA |
| 30 | NA | NA | NA | NA | NA | NA | NA |

Table 96 Instrument Conditions Hg

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | CVAAS | | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | | | | | | NA | |
| 5 | ICP-MS | Lu | CRI | He | 500 | NA | 201 |
| 6 | ICP-MS | Ir | NA | NA | 625 | NA | 201 |
| 7 | CVAAS | NA | NA | NA | 50 | NA | 453 |
| 8 | FIMS-AAS | NA | NA | NA | 50 | NA | 253.7nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 193 | | standard mode | 50 | | 202 |
| 11 | ICP-MS | Iridium | KED | He | 1000 | NA | 201.971 |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | CVAAS | | | | | NA | 253.7 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | CVAFS | | | | 0.1 | NA | 253.7 |
| 16 | Cold Vapour FIMS | NA | | | 1 | NA | 253.7 |
| 17 | CVAAS | Yttrium | | | 250 | NA | 253.7 |
| 18 | ICP-MS | Lu | ORS | He | 500 | NA | 201 |
| 19 | ICP-MS | Ir 193 | KED | | 100 | NA | 202 |
| 20 | FIMS | | | | | NA | 253.7 |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | ICP-MS | Ir | ORS | He | 800 | NA | 201 |
| 23 | ICP-MS | Yes | | | | NA | 202 |
| 24 | | | | | | NA | |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | NA |
| 26 | ICP-MS | Tb | NA | standard mode | 1000/400 | NA | 201 |
| 27 | ICP-MS | Ir | NA | | 250 | 250 | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Lu | ORS | He | 500 | NA | 202 |
| 30 | ICP-MS/MS | Ir 193 | ORS | O2 | 4000 | NA | Hg 202/202(m/z) |

Table 97 Instrument Conditions K

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-OES-AV | Scandium | | | NA | | |
| 2 | NA | NA | NA | NA | NA | | NA |
| 3 | NA | NA | NA | NA | NA | NA | NA |
| 4 | NA | NA | NA | NA | NA | | NA |
| 5 | NA | NA | NA | NA | NA | NA | NA |
| 6 | ICP-MS | Sc | KED | He | NA | 625 | 39 |
| 7 | ICP-MS | Sc | ORS | NA | NA | 50 | 39 |
| 8 | ICP-OES | Eu & Cs | NA | NA | NA | 50 | 404.721nm, 766.491nm |
| 9 | | | | | NA | | |
| 10 | ICP-MS | 89 | ORS | H2 | | 10 | 39 |
| 11 | | | | | NA | | |
| 12 | | | | | NA | | |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | | 766.49 |
| 14 | | | | | NA | | |
| 15 | ICP-OES-RV | Lu | | | NA | 0.1 | 766.491 |
| 16 | AAS | NA | | | NA | 10 | 766.5 |
| 17 | ICP-OES-RV | Yttrium | | | NA | 250 | 766.49 |
| 18 | NA | NA | NA | NA | NA | NA | NA |
| 19 | NA | NA | NA | NA | NA | NA | NA |
| 20 | | | | | NA | | |
| 21 | ICP-OES-AV | | | | NA | | |
| 22 | ICP-OES-RV | Y | NA | NA | NA | 800 | 766.491 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | NA | NA | NA | NA | NA | 100 | NA |
| 26 | NA | NA | NA | NA | NA | NA | NA |
| 27 | NA | NA | NA | NA | NA | NA | NA |
| 28 | NA | NA | NA | NA | NA | | NA |
| 29 | NA | NA | NA | NA | NA | NA | NA |
| 30 | NA | NA | NA | NA | NA | NA | NA |

Table 98 Instrument Conditions La

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | | | | | | NA | |
| 2 | NA | NA | NA | NA | NA | NA | NA |
| 3 | | | | | | NA | |
| 4 | ICP-MS | Rh, Sc, Ir | ORS | He | NA | NA | 139 |
| 5 | NA | NA | NA | NA | NA | NA | NA |
| 6 | | | | | | NA | |
| 7 | NA | NA | NA | NA | NA | NA | NA |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 139 m/z |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 159 | ORS | He | 50 | | 139 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | | | | | | NA | NT |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | In | ORS | | 0.1 | NA | 139 |
| 16 | | | | | | NA | |
| 17 | NA | | | | | NA | |
| 18 | NA | NA | NA | NA | NA | NA | NA |
| 19 | NA | NA | NA | NA | NA | NA | NA |
| 20 | | | | | | NA | |
| 21 | | | | | | NA | |
| 22 | ICP-MS | Rh | ORS | He | 800 | NA | 139 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | NA |
| 26 | ICP-MS | Tb | NA | standard mode | 400 | NA | 139 |
| 27 | ICP-MS | In | NA | | NA | 250 | |
| 28 | | | | | | NA | |
| 29 | NA | NA | NA | NA | NA | NA | NA |
| 30 | NA | NA | NA | NA | NA | NA | NA |

Table 99 Instrument Conditions Li

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | NA | NA | NA | NA | NA | NA | NA |
| 3 | | | | | | NA | |
| 4 | ICP-MS | Rh, Sc, Ir | ORS | NA | NA | NA | 7 |
| 5 | ICP-MS | Sc | CRI | NA | 500 | NA | 7 |
| 6 | ICP-MS | Sc | NA | NA | 625 | NA | 7 |
| 7 | NA | NA | NA | NA | NA | NA | NA |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 7 m/z |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 72 | ORS | H2 | 50 | | 7 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-MS | Sc | NA | | 1000 | NA | 7 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Li6 | NA | standard mode | 0.1 | NA | 7 |
| 16 | | | | | | NA | |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 670.784 |
| 18 | ICP-MS | Sc | ORS | He | 500 | NA | 7 |
| 19 | ICP-MS | Sc - 45 | KED | | 10 | NA | 7 |
| 20 | | | | | | NA | |
| 21 | ICP-OES-AV | | | | | NA | |
| 22 | NA | NA | NA | NA | NA | NA | NA |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | NA |
| 26 | ICP-MS | Sc | NA | standard mode | 2000 | NA | 7 |
| 27 | ICP-MS | Sc | NA | | 250 | NA | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Sc[No Gas] | ORS | He | 500 | NA | 7 |
| 30 | ICP-OES-AV | Cs672.328 | NA | NA | 80 | NA | Li 670.783(nm) |

Table 100 Instrument Conditions Mg

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-OES-AV | Scandium | | | NA | | |
| 2 | NA | NA | NA | NA | NA | | NA |
| 3 | NA | NA | NA | NA | NA | NA | NA |
| 4 | NA | NA | NA | NA | NA | | NA |
| 5 | NA | NA | NA | NA | NA | NA | NA |
| 6 | ICP-MS | Sc | KED | He | NA | 625 | 25 |
| 7 | ICP-MS | Sc | ORS | He | NA | 50 | 24 |
| 8 | ICP-OES | Eu & Cs | NA | NA | NA | 50 | 383.829nm |
| 9 | | | | | NA | | |
| 10 | ICP-MS | 89 | ORS | H2 | | 10 | 24 |
| 11 | | | | | NA | | |
| 12 | | | | | NA | | |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | | 279.077 |
| 14 | | | | | NA | | |
| 15 | ICP-OES-RV | Lu | | | NA | 0.1 | 285.213 |
| 16 | AAS | NA | NA | | NA | 10 | 285.2 |
| 17 | ICP-OES-RV | Yttrium | | | NA | 250 | 279.077 |
| 18 | NA | NA | NA | NA | NA | NA | NA |
| 19 | NA | NA | NA | NA | NA | NA | NA |
| 20 | | | | | NA | | |
| 21 | ICP-OES-AV | | | | NA | | |
| 22 | ICP-OES-RV | Y | NA | NA | NA | 800 | 279.078 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | NA | NA | NA | NA | NA | 100 | NA |
| 26 | NA | NA | NA | NA | NA | NA | NA |
| 27 | NA | NA | NA | NA | NA | NA | NA |
| 28 | NA | NA | NA | NA | NA | | NA |
| 29 | NA | NA | NA | NA | NA | NA | NA |
| 30 | NA | NA | NA | NA | NA | NA | NA |

Table 101 Instrument Conditions Mn

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | NA | NA | NA | NA | NA | NA | NA |
| 5 | ICP-MS | Sc | CRI | He | 500 | NA | 55 |
| 6 | ICP-MS | Sc | KED | He | 625 | NA | 55 |
| 7 | ICP-MS | Sc | ORS | He | 50 | NA | 55 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 261.021nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 72 | | standard mode | 50 | | 55 |
| 11 | ICP-MS | Rhodium | KED | He | 1000 | NA | 54.938 |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-OES-AV-buffer | Y | | He | 100 | NA | 257.61 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-OES-AV | Lu | | | 0.1 | NA | 257.61 |
| 16 | ICP-OES-AV | NA | NA | | 10 | NA | 294.921 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 257.61 |
| 18 | ICP-MS | Sc | ORS | He | 500 | NA | 55 |
| 19 | ICP-MS | Sc 45 | KED | | 10 | NA | 55 |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | NA | NA | NA | NA | NA | NA | NA |
| 23 | ICP-MS | Yes | | | | NA | 55 |
| 24 | | | | | | NA | |
| 25 | NA | NA | NA | NA | NA | NA | NA |
| 26 | ICP-MS | Sc | KED | He | 2000 | NA | 55 |
| 27 | ICP-MS | Rh | UC | He | 250 | NA | |
| 28 | NA | NA | NA | NA | NA | NA | NA |
| 29 | ICP-MS | Sc | ORS | He | 500 | NA | 55 |
| 30 | ICP-OES-AV | Eu 271.700 | NA | NA | 80 | NA | Mn 257.610 |

Table 102 Instrument Conditions Mo

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | NA | NA | NA | NA | NA | NA | NA |
| 5 | ICP-MS | Rh | CRI | He | 500 | NA | 95 |
| 6 | ICP-MS | Rh | NA | NA | 625 | NA | 95 |
| 7 | ICP-MS | Rh | ORS | He | 50 | NA | 95 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 202.032nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 89 | ORS | He | 10 | | 95 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-MS | In | NA | | 1000 | NA | 95 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Rh | ORS | He | 0.1 | NA | 95 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 202.032 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 202.031 |
| 18 | ICP-MS | Rh | ORS | He | 500 | NA | 95 |
| 19 | ICP-MS | Y 89 | KED | | NA | | 98 |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | NA | NA | NA | NA | NA | NA | NA |
| 23 | ICP-MS | Yes | | | | NA | 98 |
| 24 | | | | | | NA | |
| 25 | NA | NA | NA | NA | NA | NA | NA |
| 26 | ICP-MS | Rh | NA | standard mode | 2000 | NA | 98 |
| 27 | ICP-MS | Rh | UC | He | NA | 250 | |
| 28 | NA | NA | NA | NA | NA | NA | NA |
| 29 | ICP-MS | Rh | ORS | He | 500 | NA | 95 |
| 30 | ICP-MS/MS | Rh103 | ORS | O2 | 4000 | NA | Mo 95/95(m/z) |

Table 103 Instrument Conditions Na

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-OES-AV | Scandium | | | NA | | |
| 2 | NA | NA | NA | NA | NA | | NA |
| 3 | NA | NA | NA | NA | NA | NA | NA |
| 4 | NA | NA | NA | NA | NA | | NA |
| 5 | NA | NA | NA | NA | NA | NA | NA |
| 6 | ICP-MS | Sc | KED | He | NA | 625 | 23 |
| 7 | ICP-MS | Sc | ORS | NA | NA | 50 | 23 |
| 8 | ICP-OES | Eu & Cs | NA | NA | NA | 50 | 330.237, 589.592nm |
| 9 | | | | | NA | | |
| 10 | ICP-MS | 89 | ORS | H2 | | 5 | 23 |
| 11 | | | | | NA | | |
| 12 | | | | | NA | | |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | | 588.995 |
| 14 | | | | | NA | | |
| 15 | ICP-OES-RV | Y | | | NA | 0.1 | 589.592 |
| 16 | AAS | NA | NA | | NA | 1 | 589 |
| 17 | ICP-OES-RV | Yttrium | | | NA | 250 | 589.592 |
| 18 | NA | NA | NA | NA | NA | NA | NA |
| 19 | NA | NA | NA | NA | NA | NA | NA |
| 20 | ICP-OES-RV | | | | NA | | 589.592 |
| 21 | ICP-OES-AV | | | | NA | | |
| 22 | ICP-OES-RV | Y | NA | NA | NA | 800 | 589.592 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | NA | NA | NA | NA | NA | 100 | NA |
| 26 | NA | NA | NA | NA | NA | NA | NA |
| 27 | NA | N/A | NA | NA | NA | NA | NA |
| 28 | NA | NA | NA | NA | NA | | NA |
| 29 | NA | NA | NA | NA | NA | NA | NA |
| 30 | NA | NA | NA | NA | NA | NA | NA |

Table 104 Instrument Conditions Ni

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | ICP-OES-RV | | | | | NA | 231.604 |
| 5 | ICP-MS | Sc | CRI | He | 500 | NA | 60 |
| 6 | ICP-MS | Ge | KED | He | 625 | NA | 60 |
| 7 | ICP-MS | Sc | ORS | He | 50 | NA | 60 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 231.604nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 103 | ORS | He | 50 | | 60 |
| 11 | ICP-MS | Rhodium | KED | He | 1000 | NA | 59.933 |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | NA | 231.604 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Ge | ORS | He | 0.1 | NA | 51 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 231.604 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 231.604 |
| 18 | ICP-MS | Sc | ORS | He | 500 | NA | 60 |
| 19 | ICP-MS | Sc 45 | KED | | 10 | NA | 60 |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | ICP-MS | Rh | ORS | He | 800 | NA | 60 |
| 23 | ICP-MS | Yes | KED | He | | NA | 60 |
| 24 | | | | | | NA | |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | 231.604 |
| 26 | ICP-MS | Ga | KED | He | 1000/400 | NA | 60 |
| 27 | ICP-MS | Rh | UC | He | 250 | 250 | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Sc | ORS | He | 500 | NA | 60 |
| 30 | ICP-OES-AV | Lu219.556 | NA | NA | 80 | NA | Ni 231.604 |

Table 105 Instrument Conditions P

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | | | | | NA | | |
| 2 | NA | NA | NA | NA | NA | | NA |
| 3 | NA | NA | NA | NA | NA | NA | NA |
| 4 | NA | NA | NA | NA | NA | | NA |
| 5 | NA | NA | NA | NA | NA | NA | NA |
| 6 | ICP-MS | Sc | KED | He | NA | 625 | 31 |
| 7 | ICP-OES-AV | Eu | NA | NA | NA | 50 | 186 |
| 8 | ICP-OES | Eu & Cs | NA | NA | NA | 50 | 185.827nm |
| 9 | | | | | NA | | |
| 10 | ICP-MS | 72 | ORS | He | | 10 | 31 |
| 11 | | | | | NA | | |
| 12 | | | | | NA | | |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | | 178.221 |
| 14 | | | | | NA | | |
| 15 | ICP-OES-AV | Lu | | | NA | 0.1 | 177.434 |
| 16 | DA | NA | NA | | NA | 1 | 880 |
| 17 | ICP-OES-AV | Yttrium | | | NA | 250 | 178.221 |
| 18 | ICP-MS | Sc | ORS | He | NA | 500 | 31 |
| 19 | NA | NA | NA | NA | NA | NA | NA |
| 20 | | | | | NA | | |
| 21 | ICP-OES-AV | | | | NA | | |
| 22 | ICP-OES-AV | Y | NA | NA | NA | 800 | 213.618 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | NA | NA | NA | NA | NA | 100 | NA |
| 26 | NA | NA | NA | NA | NA | NA | NA |
| 27 | NA | N/A | NA | NA | NA | NA | NA |
| 28 | NA | NA | NA | NA | NA | | NA |
| 29 | NA | NA | NA | NA | NA | 500 | NA |
| 30 | NA | NA | NA | NA | NA | NA | NA |

Table 106 Instrument Conditions Pb

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | NA | NA | NA | NA | NA | NA | NA |
| 5 | ICP-MS | Lu | CRI | He | 500 | NA | 208 |
| 6 | ICP-MS | Ir | NA | NA | 625 | NA | 206+207+208 |
| 7 | ICP-MS | Ir | ORS | He | 50 | NA | 208 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 220.353nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 159 | | standard mode | 50 | | 208 |
| 11 | ICP-MS | Iridium | KED | He | 1000 | NA | 207.977 |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | NA | 220.353 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Ir | ORS | He | 0.1 | NA | 208 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 220.353 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 220.353 |
| 18 | ICP-MS | Lu | ORS | He | 500 | NA | 208 |
| 19 | ICP-MS | Ir 193 | KED | | 10 | NA | 208 |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | NA | NA | NA | NA | NA | NA | NA |
| 23 | ICP-MS | Yes | | | | NA | 208 |
| 24 | | | | | | NA | |
| 25 | NA | NA | NA | NA | NA | NA | NA |
| 26 | ICP-MS | Tb | KED | He | 1000 | NA | 206+207+208 |
| 27 | ICP-MS | Ir | NA | | 250 | 250 | |
| 28 | NA | NA | NA | NA | NA | NA | NA |
| 29 | ICP-MS | Lu | ORS | He | 500 | NA | 208 |
| 30 | ICP-OES-AV | Eu271.70 | NA | NA | 80 | NA | Pb 220.353 |

Table 107 Instrument Conditions Rb

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | | | | | | NA | |
| 2 | NA | NA | NA | NA | NA | NA | NA |
| 3 | | | | | | NA | |
| 4 | ICP-MS | | | | | NA | |
| 5 | NA | NA | NA | NA | NA | NA | NA |
| 6 | | | | | | NA | |
| 7 | NA | NA | NA | NA | NA | NA | NA |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 85 m/z |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 89 | ORS | He | 50 | | 85 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | | | | | | NA | NT |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | | | | | | NA | |
| 16 | | | | | | NA | |
| 17 | NA | | | | | NA | |
| 18 | NA | NA | NA | NA | NA | NA | NA |
| 19 | NA | | | | NA | NA | NA |
| 20 | | | | | | NA | |
| 21 | | | | | | NA | |
| 22 | NA | NA | NA | NA | NA | NA | NA |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | NA |
| 26 | ICP-MS | Rh | NA | standard mode | 400 | NA | 85 |
| 27 | ICP-MS | Rh | NA | | 250 | NA | |
| 28 | | | | | | NA | |
| 29 | NA | NA | NA | NA | NA | NA | NA |
| 30 | NA | NA | NA | NA | NA | NA | NA |

Table 108 Instrument Conditions S

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-OES-AV | Scandium | | | NA | | |
| 2 | NA | NA | NA | NA | NA | | NA |
| 3 | NA | NA | NA | NA | NA | NA | NA |
| 4 | NA | NA | NA | NA | NA | | NA |
| 5 | NA | NA | NA | NA | NA | NA | NA |
| 6 | ICP-OES-AV | Y | NA | NA | NA | 62.5 | 181.975 |
| 7 | ICP-OES-AV | Eu | NA | NA | NA | 50 | 207 |
| 8 | ICP-OES | Eu & Cs | NA | NA | NA | 50 | 178.165,181.972nm |
| 9 | | | | | NA | | |
| 10 | ICP-MS/MS | 89-105 | ORS | O2 | | 50 | 32 |
| 11 | | | | | NA | | |
| 12 | | | | | NA | | |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | | 181.975 |
| 14 | | | | | NA | | |
| 15 | ICP-OES-AV | Lu | | | NA | 0.1 | 181.972 |
| 16 | | | | | NA | | |
| 17 | ICP-OES-AV | Yttrium | | | NA | 250 | 181.975 |
| 18 | NA | NA | NA | NA | NA | NA | NA |
| 19 | NA | NA | NA | NA | NA | NA | NA |
| 20 | | | | | NA | | |
| 21 | NA | | | | NA | | |
| 22 | ICP-OES-AV | Y | NA | NA | NA | 800 | 181.972 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | NA | NA | NA | NA | NA | 100 | NA |
| 26 | NA | NA | NA | NA | NA | NA | NA |
| 27 | NA | NA | NA | NA | NA | NA | NA |
| 28 | NA | NA | NA | NA | NA | | NA |
| 29 | NA | NA | NA | NA | NA | NA | NA |
| 30 | NA | NA | NA | NA | NA | NA | NA |

Table 109 Instrument Conditions Sb

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | ICP-OES-RV | | | | | NA | 206.834 |
| 5 | ICP-MS | Rh | CRI | He | 500 | NA | 123 |
| 6 | ICP-MS | Rh | NA | NA | 625 | NA | 121 |
| 7 | ICP-MS | Sc | ORS | He | 50 | NA | 121 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 206.834nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 193 | | standard mode | 50 | | 121 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | | | | | | NA | NT |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | In | ORS | He | 0.1 | NA | 121 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 206.834 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 206.836 |
| 18 | ICP-MS | Rh | ORS | He | 500 | NA | NA |
| 19 | NA | NA | | | NA | NA | NA |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | ICP-MS | Rh | ORS | He | 800 | NA | 121 |
| 23 | ICP-MS | Yes | | | | NA | 121 |
| 24 | | | | | | NA | |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | 217.582 |
| 26 | ICP-MS | Rh | NA | standard mode | 400 | NA | 121 |
| 27 | ICP-MS | Rh | NA | | 250 | NA | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Rh | ORS | He | 500 | NA | 123 |
| 30 | ICP-MS/MS | Rh103 | ORS | O2 | 4000 | NA | Sb 121/121(m/z) |

Table 110 Instrument Conditions Se

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | ICP-OES-RV | | | | | NA | 196.026 |
| 5 | ICP-MS | Sc | CRI | He | 500 | NA | 78 |
| 6 | ICP-MS | Rh | NA | NA | 625 | NA | 82 |
| 7 | ICP-MS | Sc | ORS | He | 50 | NA | 78 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 196.026nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 72 | ORS | He | 10 | | 78 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-MS | Ga | DRC | NH3 | 1000 | NA | 78 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Ge | ORS | H2 | 0.1 | NA | 78 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 196.026 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 196.026 |
| 18 | ICP-MS | Rh | ORS | H2 | 500 | NA | 78 |
| 19 | ICP-MS | Ge-1 72 | KED | | 10 | NA | 82 |
| 20 | ICP-OES-AV | | | | | NA | 196.026 |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | ICP-MS | Rh | ORS | HEHe | 800 | NA | 78 |
| 23 | ICP-MS | | KED | | | NA | 78 |
| 24 | | | | | | NA | |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | 196.026 |
| 26 | ICP-MS | Te | NA | standard mode | 2000/400 | NA | 82 |
| 27 | ICP-MS | Rh | UC | He | 250 | 250 | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Rh | ORS | H2 | 500 | NA | 78 |
| 30 | ICP-MS/MS | Rh103 | ORS | O2 | 4000 | NA | Se 78/94(m/z) |

Table 111 Instrument Conditions Sn

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-OES-AV | Scandium | | | | NA | |
| 2 | NA | NA | NA | NA | NA | NA | NA |
| 3 | | | | | | NA | |
| 4 | ICP-OES-RV | | | | | NA | 189.925 |
| 5 | ICP-MS | Rh | CRI | He | 500 | NA | 118 |
| 6 | | | | | | NA | |
| 7 | NA | NA | NA | NA | NA | NA | NA |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 189.926nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 103 | ORS | He | 50 | | 120 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | NA | 189.927 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | In | ORS | He | 0.1 | NA | 118 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 189.925 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 189.927 |
| 18 | ICP-MS | Rh | ORS | He | 500 | NA | NA |
| 19 | ICP-MS | In-1 115 | KED | | 1 | NA | 118 |
| 20 | | | | | | NA | |
| 21 | | | | | | NA | |
| 22 | ICP-MS | Rh | ORS | He | 800 | NA | 118 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | NA |
| 26 | ICP-MS | Rh | NA | standard mode | 400 | NA | 120 |
| 27 | ICP-MS | Rh | NA | | 250 | NA | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Rh | ORS | He | 500 | NA | 118 |
| 30 | ICP-OES-AV | Lu219.556 | NA | NA | 80 | NA | Sn 189.927 |

Table 112 Instrument Conditions Sr

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-OES-AV | Scandium | | | NA | | |
| 2 | NA | NA | NA | NA | NA | | NA |
| 3 | NA | NA | NA | NA | NA | NA | NA |
| 4 | NA | NA | NA | NA | NA | | NA |
| 5 | ICP-MS | Rh | CRI | He | NA | 500 | 88 |
| 6 | ICP-MS | Rh | NA | NA | NA | 625 | 88 |
| 7 | NA | NA | ORS | He | NA | 50 | 88 |
| 8 | ICP-OES | Eu & Cs | NA | NA | NA | 50 | 430.545nm |
| 9 | | | | | NA | | |
| 10 | ICP-MS | 89 | ORS | He | | 10 | 88 |
| 11 | | | | | NA | | |
| 12 | | | | | NA | | |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | | 407.771 |
| 14 | | | | | NA | | |
| 15 | ICP-OES-AV | Lu | | | NA | 0.1 | 421.552 |
| 16 | ICP-OES-AV | NA | NA | | NA | 1 | 407.771 |
| 17 | ICP-OES-AV | Yttrium | | | NA | 250 | 407.771 |
| 18 | ICP-MS | Rh | ORS | He | NA | 500 | 88 |
| 19 | NA | NA | NA | NA | NA | NA | NA |
| 20 | | | | | NA | | |
| 21 | ICP-OES-AV | | | | NA | | |
| 22 | ICP-OES-AV | Y | NA | NA | NA | 800 | 430.544 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | NA | NA | NA | NA | NA | 100 | NA |
| 26 | NA | NA | NA | NA | NA | NA | NA |
| 27 | NA | N/A | NA | NA | NA | NA | NA |
| 28 | NA | NA | NA | NA | NA | | NA |
| 29 | ICP-MS | Rh | ORS | He | NA | 500 | 88 |
| 30 | NA | NA | NA | NA | NA | NA | NA |

Table 113 Instrument Conditions Th

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | NA | NA | NA | NA | NA | NA | NA |
| 5 | NA | NA | NA | NA | NA | NA | NA |
| 6 | ICP-MS | Ir | NA | NA | 625 | NA | 232 |
| 7 | ICP-MS | Ir | ORS | He | 50 | NA | 232 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 232 m/z |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | NT | | | | | NA |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | | | | | | NA | NT |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Ir | ORS | He | 0.1 | NA | 232 |
| 16 | | | | | | NA | |
| 17 | NA | | | | | NA | |
| 18 | NA | NA | NA | NA | NA | NA | NA |
| 19 | NA | | | | NA | NA | NA |
| 20 | | | | | | NA | |
| 21 | | | | | | NA | |
| 22 | NA | Ir | NA | NA | NA | NA | NA |
| 23 | | | | | | NA | |
| 24 | | | | | | NA | |
| 25 | NA | NA | NA | NA | NA | NA | NA |
| 26 | NA | NA | NA | NA | NA | NA | NA |
| 27 | NA | N/A | NA | NA | NA | NA | NA |
| 28 | NA | NA | NA | NA | NA | NA | NA |
| 29 | NA | NA | NA | NA | NA | NA | NA |
| 30 | ICP-MS/MS | Ir 193 | ORS | He | 4000 | NA | Th 232/232(m/z) |

Table 114 Instrument Conditions Tl

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | NA | NA | NA | NA | NA | NA | NA |
| 3 | | | | | | NA | |
| 4 | ICP-MS | Rh, Sc, Ir | ORS | He | NA | NA | 205 |
| 5 | ICP-MS | Lu | CRI | He | 500 | NA | 205 |
| 6 | ICP-MS | Ir | NA | NA | 625 | NA | 205 |
| 7 | NA | NA | NA | NA | NA | NA | NA |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 203 m/z |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 159 | | standard mode | 10 | | 205 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-MS | In | | | 1000 | NA | 203 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Ir | ORS | He | 0.1 | NA | 205 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 190.794 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 190.801 |
| 18 | ICP-MS | Lu | ORS | He | 500 | NA | 205 |
| 19 | NA | | | | NA | NA | NA |
| 20 | | | | | | NA | |
| 21 | | | | | | NA | |
| 22 | ICP-MS | Ir | ORS | He | 800 | NA | 205 |
| 23 | NA | NA | NA | NA | NA | NA | NA |
| 24 | NA | NA | NA | NA | NA | NA | NA |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | NA |
| 26 | ICP-MS | Tb | NA | standard mode | 400 | NA | 205 |
| 27 | ICP-MS | Ir | NA | | NA | 250 | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Lu | ORS | He | 500 | NA | 205 |
| 30 | ICP-MS/MS | Ir 193 | ORS | O2 | 4000 | NA | Tl 205/205(m/z) |

Table 115 Instrument Conditions U

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|------------|-------------------|---------------|---------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | NA | NA | NA | NA | NA | NA | NA |
| 5 | ICP-MS | Lu | CRI | He | 500 | NA | 238 |
| 6 | ICP-MS | Ir | NA | NA | 625 | NA | 238 |
| 7 | ICP-MS | Ir | ORS | He | 50 | NA | 238 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 238 m/z |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 159 | | standard mode | 10 | | 238 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-MS | In | | | 1000 | NA | 238 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Ir | ORS | He | 0.1 | NA | 238 |
| 16 | | | | | | NA | |
| 17 | NA | | | | | NA | |
| 18 | ICP-MS | Lu | ORS | He | 500 | NA | 238 |
| 19 | ICP-MS | Ir 193 | KED | | 10 | | 238 |
| 20 | | | | | | NA | |
| 21 | ICP-MS | | CRI | He | | NA | |
| 22 | NA | NA | NA | NA | NA | NA | NA |
| 23 | | | | | | NA | |
| 24 | | | | | | NA | |
| 25 | NA | NA | NA | NA | NA | NA | NA |
| 26 | ICP-MS | Tb | NA | standard mode | 2000 | NA | 238 |
| 27 | ICP-MS | Ir | NA | | NA | 250 | |
| 28 | NA | NA | NA | NA | NA | NA | NA |
| 29 | ICP-MS | Lu | ORS | He | 500 | NA | 238 |
| 30 | ICP-MS/MS | Ir 193 | ORS | He | 4000 | NA | U 238/238(m/z) |

Table 116 Instrument Conditions V

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-MS | Indium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | NA | NA | NA | NA | NA | NA | NA |
| 5 | ICP-MS | Sc | CRI | He | 500 | NA | 51 |
| 6 | ICP-MS | Sc | KED | He | 625 | NA | 51 |
| 7 | ICP-MS | Sc | ORS | He | 50 | NA | 51 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 311.837nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 72 | ORS | He | 50 | | 51 |
| 11 | | | | | | NA | |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | NA | 290.88 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-MS | Ge | ORS | He | 0.1 | NA | 51 |
| 16 | ICP-OES-AV | NA | NA | | 1 | NA | 292.401 |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 292.402 |
| 18 | ICP-MS | Sc | ORS | He | 500 | NA | 51 |
| 19 | ICP-MS | Sc 45 | KED | | 10 | NA | 51 |
| 20 | | | | | | NA | |
| 21 | | | | | | NA | |
| 22 | NA | NA | NA | NA | NA | NA | NA |
| 23 | ICP-MS | Yes | KED | He | | NA | 51 |
| 24 | | | | | | NA | |
| 25 | NA | NA | NA | NA | NA | NA | NA |
| 26 | ICP-MS | Sc | KED | He | 2000 | NA | 51 |
| 27 | ICP-MS | Sc | UC | He | 250 | NA | |
| 28 | NA | NA | NA | NA | NA | NA | NA |
| 29 | ICP-MS | Sc | ORS | He | 500 | NA | 51 |
| 30 | ICP-MS/MS | Sc 45/61 | ORS | O2 | 4000 | NA | V 51/67(m/z) |

Table 117 Instrument Conditions Zn

| Laboratory Code | Instrument | Internal standard | Reaction Cell | Reaction Gas | S1/S2 Final Dilution Factor | S3 Final Dilution Factor | Wavelength (nm)/ Ion(m/z)/ Absorbance(nm) |
|-----------------|-------------------|-------------------|---------------|--------------|-----------------------------|--------------------------|---|
| 1 | ICP-OES-AV | Scandium | | | | NA | |
| 2 | | | | | | NA | |
| 3 | | | | | | NA | |
| 4 | ICP-OES-RV | | | | | NA | 206.2 |
| 5 | ICP-MS | Sc | CRI | He | 500 | NA | 66 |
| 6 | ICP-MS | Ge | KED | He | 625 | NA | 66 |
| 7 | ICP-MS | Sc | ORS | He | 50 | NA | 66 |
| 8 | ICP-MS | Ir, Rh & Sc | NA | NA | 50 | NA | 206.2, 334.502nm |
| 9 | NA | NA | NA | NA | NA | NA | NA |
| 10 | ICP-MS | 72 | ORS | He | 50 | | 66 |
| 11 | ICP-MS | Rhodium | KED | He | 1000 | NA | 65.926 |
| 12 | NA | NA | NA | NA | NA | NA | NA |
| 13 | ICP-OES-AV-buffer | Y | | | 100 | NA | 206.2 |
| 14 | NA | NA | NA | NA | NA | NA | NA |
| 15 | ICP-OES-AV | Lu | | | 0.1 | NA | 206.2 |
| 16 | ICP-OES-AV | NA | NA | | 1 | 206.2 | |
| 17 | ICP-OES-AV | Yttrium | | | 250 | NA | 206.200 |
| 18 | ICP-MS | Sc | ORS | He | 500 | NA | 66 |
| 19 | ICP-MS | Sc 45 | KED | | 10 | | 66 |
| 20 | | | | | | NA | |
| 21 | ICP-OES-AV | | | | | NA | |
| 22 | ICP-MS | Rh | ORS | He | 800 | NA | 66 |
| 23 | ICP-MS | Yes | KED | He | | NA | 66 |
| 24 | | | | | | NA | |
| 25 | ICP-OES-AV | Lu | NA | NA | 100 | NA | 213.857 |
| 26 | ICP-MS | Ga | KED | He | 1000/400 | NA | 66 |
| 27 | ICP-MS | Rh | UC | He | 250 | 250 | |
| 28 | | | | | | NA | |
| 29 | ICP-MS | Sc | ORS | He | 500 | NA | 66 |
| 30 | ICP-OES-AV | Lu219.556 | NA | NA | 80 | NA | Zn 206.200 |

END OF REPORT