

# **GEOCHEMICAL PROFICIENCY TESTING PROGRAM**

## **ROUND 12**

## **MARCH 2017**

## **REPORT NO. 1020**

### **ACKNOWLEDGMENTS**

PTA wishes to gratefully acknowledge the technical assistance that was provided for this program by Ms J Hwende of Bureau Veritas Australia Pty Ltd.

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## 1. Foreword

This report summarises the results of a proficiency testing program on the analysis of geochemical samples. The exercise was conducted in December 2016 / January 2017 by Proficiency Testing Australia (PTA). The Program Coordinator was Mrs K Weller and the Technical Adviser was Ms J Hwende, Bureau Veritas Australia Pty Ltd. This report was authorised by Mrs K Cividin, PTA Quality Coordinator/Senior Scientific Officer. The main aim of the program was to assess laboratories' abilities to competently perform the prescribed analyses.

## 2. Program Features and Design

- 2.1 Each laboratory was randomly allocated a unique code number for the program to ensure confidentiality of results. Reference to each laboratory in this report is by code number only.
- 2.2 Participants were provided with one Iron Ore sample.
- 2.3 Laboratories were provided with the "Instructions to Participants" and "Results Sheet" (see Appendix C) and asked to report total analysis of the elements listed below:

Aluminium	Al	Magnesium	Mg	Sodium	Na
Arsenic	Ar	Manganese	Mn	Sulphur	S
Calcium	Ca	Phosphorous	P	Titanium	Ti
Copper	Cu	Potassium	K	Loss on Ignition	LOI
Iron	Fe	Silicon	Si		

- 2.4 A total of 6 laboratories participated in the program with all laboratories returning results for inclusion in the final report. One laboratory submitted results for an extra sample set; therefore there are 7 sets of results.

All participant laboratories were from Australia.

- 2.5 Results (as reported by participants), and where relevant, corresponding summary statistics (i.e. number of results, median, normalised interquartile range, robust coefficient of variation, minimum, maximum, range and uncertainty of the median) are presented in Appendix A.
- 2.6 A robust statistical approach, using z-scores, was utilised to assess laboratories' testing performance (see Section 3). Robust z-scores and z-score charts, where relevant to each test, are presented in Appendix A.

The document entitled *Guide to Proficiency Testing Australia, 2016* (reference [1]) defines the statistical terms and details the statistical procedures referred to in this report.

- 2.7 Samples dispatched for this round were Standards Australia certified reference material ASCRM-035 Iron Ore (Yilgarn Hematite) and therefore are considered homogeneous and stable for the duration of the round. As such, any results later identified as outliers could not be attributed to any notable sample variability.

### 3. Statistical Format

For each test the following information is given, where appropriate:

- a table of results and calculated z-scores;
- a list of summary statistics; and
- ordered z-score charts.

#### 3.1 Outlier Results and Z-scores

In order to assess laboratories' testing performance, a robust statistical approach, using z-scores, was utilised. Z-scores give a measure of how far a result is from the consensus value (i.e. the median), and gives a "score" to each result relative to the other results in the group.

A z-score close to zero indicates that the result agrees well with those from other laboratories, whereas a z-score with an absolute value greater than or equal to 3.0 is considered to be an outlier and is marked by the symbol "§".

Where relevant, each determination was examined for outliers with all methods pooled. Table B on page 7 summarises the outlier results detected.

### 3.2 Results Tables and Summary Statistics

Each of these tables contains the results returned by each laboratory and where appropriate, the robust z-score calculated for each result.

Results have been entered exactly as reported by participants. That is, laboratories which did not report results to the precision (i.e. number of significant figures) requested on the Results Sheet have **not** been rounded to the requested precision before being included in the statistical analysis.

Where relevant, a list of summary statistics appears at the bottom of each of the tables of results and consists of:

- the number of results for that test/sample (*No. of Results*);
- the median of these results, i.e. the middle value (*Median*);
- the uncertainty of the median; a robust estimate of the standard deviation of the *Median*;
- the normalised interquartile range of the results (*Normalised IQR*);
- the robust coefficient of variation, expressed as a percentage (*Robust CV*) - i.e.  $100 \times \text{Normalised IQR} / \text{Median}$ ;
- the minimum and maximum laboratory results; and
- the range (*Maximum - Minimum*).

Please see reference [1] for further details on these robust summary statistics.

### 3.3 Ordered Z-score Charts

On these charts each laboratory's robust z-score is shown, in order of magnitude, and is marked with its code number. From these charts, each laboratory can readily compare its performance relative to the other laboratories.

These charts contain solid lines at +3.0 and -3.0, so that outliers are clearly identifiable as those laboratories whose "bar" extends beyond these "cut-off" lines. The y-axis of these charts has been limited, so very large z-scores appear to extend beyond the chart boundary.

Further details for the interpretation of these diagrams are given in reference [1]. Please also refer to this document for a glossary of terms.

**TABLE A: SUMMARY STATISTICS**

Analysis	No. of Results	Certified Value <sup>2</sup>	Median / Assigned Value (%)	Normalised IQR / Standard Deviation	Uncertainty (Median) (%)
Aluminium	7	0.72	0.7160	0.0110	0.0052
Arsenic <sup>1</sup>	3	0.0079	0.0079	0.0029	0.0005
Calcium	6	0.117	0.1208	0.0019	0.0010
Copper <sup>1</sup>	3	0.0087	0.0087	0.0012	0.0002
Iron	7	62.35	62.450	1.002	0.475
Magnesium	6	0.150	0.1595	0.0157	0.0081
Manganese	6	0.152	0.1558	0.0154	0.0079
Phosphorus <sup>1</sup>	5	0.078	0.078	0.002	0.0002
Potassium	6	0.022	0.0225	0.0011	0.0005
Silicon	7	1.99	2.000	0.027	0.013
Sodium <sup>1</sup>	5	0.0565	0.0565	0.0186	0.0024
Sulphur	6	0.057	0.0565	0.0009	0.0005
Titanium	6	0.046	0.0478	0.0025	0.0013
Loss on Ignition	5	N/A	Not Calculated <sup>3</sup>		

**Notes:**

1. Only a small number of numerical results (<6) were returned for Arsenic, Copper, Phosphorus and Sodium, however as the sample used for this round is a certified reference material, assigned values and target CVs (based on certified values and standard deviations) have been used to calculate robust z-scores for each of these elements.
2. Except for the elements listed in Note 1; certified values have been tabulated for information purposes only.
3. Statistical analysis has not been performed on Loss on Ignition results as less than 6 numerical results were returned. The certified value for Loss on Ignition was not used as the method stated in the instructions for this round is different to that used to determine the certified value.

#### 4. PTA and Technical Adviser's Comments

For this program, laboratories were encouraged to use a method that gave the best detection limit for the element. The comments presented in this section are general in nature.

##### 4.1 Overall performance

In general, a good performance all round was noted for Round 12.

The robust coefficient of variation for all elements shows satisfactory performance. Generally, all values (excluding outliers) agree well with the expected values.

##### 4.2 Outliers

Lab Code	Results Reported	No. of Outliers
1	14	3
2	12	2
3	12	1
4	3	1
5	14	0
6	12	0
7	12	1

Laboratories are encouraged to investigate and eliminate the cause for the outliers and anomalies.

##### 4.3 Measurement Uncertainty (MU) and Detection Limit

All laboratories quantified the Measurement Uncertainty for the testing performed. It is advisable for laboratories to review their process or calculations for suitability if MU is greater than 5% of the result.

##### 4.4 Z-score (Interlaboratory Performance)

Whilst the z-score gives an indication of where each laboratory stands in comparison to their peers (median), laboratories with absolute z-scores:

- greater than 2.0 for any element - should review the technique and calibration for that element.
- greater than or equal to 3.0 for any element - should seriously review the method for that element (except in the case of a typographical or calculation error).

##### 4.5 Duplicates

Duplicate testing was generally performed well. Laboratories are encouraged to investigate wide differences between duplicates, in particular laboratory code 2 which reported wide differences in their duplicates for Iron and Potassium.

#### 4.6 Technique Bias

Most laboratories in this round used a lithium borate fusion with an XRF finish. Laboratories with outliers are encouraged to investigate the source of error.

#### 4.7 Method Code

All laboratories provided method codes.

#### 4.8 Metrological Traceability and Measurement Uncertainty of Assigned Values

Standards Australia certified reference material ASCRM-035 (Yilgarn Hematite) was used for this program.

Where more than 6 results were received from participants, consensus values (median) derived from the participants' results were used. These values are not metrologically traceable to an external reference.

Where less than 6 results were received from participants (except for Loss on Ignition), the certified values provided with the reference material were used.

Where the assigned value for an analyte in this program is the median of the results submitted by the participants, the uncertainty of the median has been calculated for each analysis (where relevant) and is tabulated in Table A on page 4, and also in the summary statistics tables in Appendix A.

#### 4.9 Analysis of Results by Method Groups

In order for methods to be grouped for analysis, PTA requires at least 11 sets of results from the same method group. As there were less than 11 results submitted for each method, reliable conclusions cannot be drawn from analysing grouped methods on this occasion. Therefore, results from all method groups have been pooled for analysis.



## 5. Outlier Results

Laboratories reporting outlier results are listed in the following table:

**TABLE B: OUTLIER RESULTS**

Element	Lab Code	Bias	Method Code	Dissolution/Digestion Technique
Aluminium	1	+ve	6 - XRF	Fused Bead
Arsenic	No outliers reported			
Calcium	7	+ve	6 - XRF	Fusion using Borate Flux
Copper	1	-ve	6 - XRF	Fused Bead
	2	+ve	6 - XRF	Not reported
Iron	No outliers reported			
Magnesium	1	+ve	6 - XRF	Fused Bead
Manganese	No outliers reported			
Phosphorous	No outliers reported			
Potassium	No outliers reported			
Silicon	4	-ve	6 - XRF	Borate Fusion
Sodium	No outliers reported			
Sulphur	2	-ve	6 - XRF	Not reported
	3	+ve	6 - XRF	Fusion using Borate Flux
Titanium	No outliers reported			

**Note:** Z-scores could not be determined for Loss on Ignition due to the small number of results returned.

## 6. References

- [1] *Guide to Proficiency Testing Australia*, 2016. (This document can be found on the PTA website, [www.pta.asn.au](http://www.pta.asn.au))
- [2] SA TR 2.35:2014 *Certified reference materials - Iron ore - Preparation and certification of ASCRM-035 (Yilgarn Hematite)*

# APPENDIX A

## Results and Data Analysis

Aluminium	A1.1
Arsenic	A2.1
Calcium	A3.1
Copper	A4.1
Iron	A5.1
Magnesium	A6.1
Manganese	A7.1
Phosphorous	A8.1
Potassium	A9.1
Silicon	A10.1
Sodium	A11.1
Sulphur	A12.1
Titanium	A13.1
Loss on Ignition	A14.1

**Aluminium (Al) (%)**

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.829	0.851	0.840	0.022	0.03	0.01	6	Fused Bead	11.24 §
2	0.714	0.704	0.709	0.010	0.02	0.10	6	#	-0.63
3	0.723	0.721	0.722	0.002	0.009	0.053	6	Fusion using Borate Flux	0.54
4	0.668	0.699	0.684	0.031	0.08	0.002	6	Borate Fusion	-2.94
5	0.71	0.71	0.710	0.00	0.005	0.01	6	Li T/Li M flux fused bead	-0.54
6	0.712	0.720	0.716	0.008	0.08	0.01	6	12:22 XRF flux with 4% LiNO <sub>3</sub>	0.00
7	0.720	0.723	0.722	0.003	0.009	0.053	6	Fusion using Borate Flux	0.50

Notes:

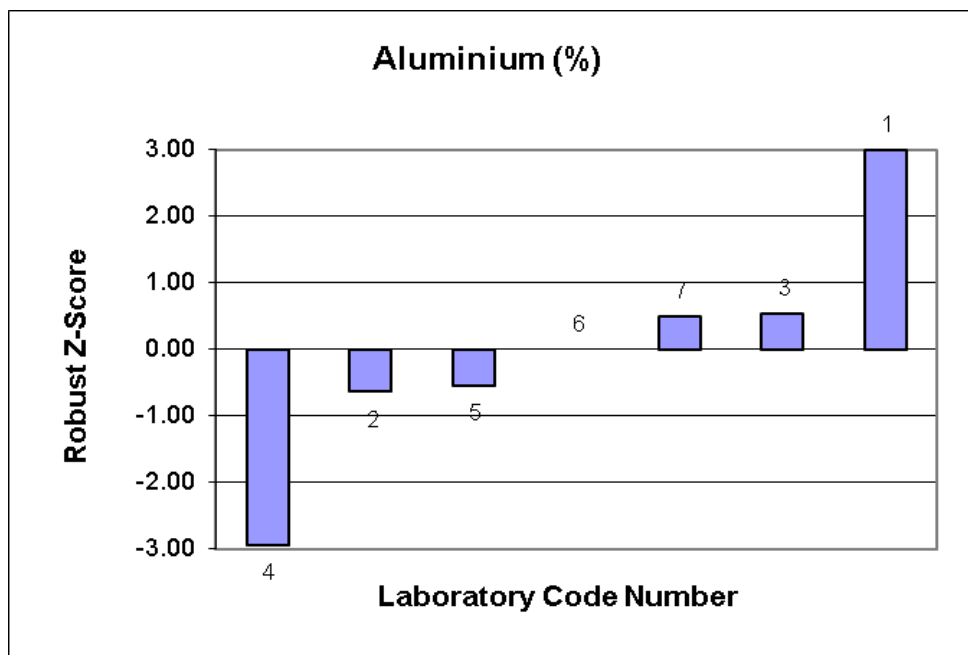
§ denotes an outlier, i.e.  $|z\text{-score}| \geq 3.0$ .

# indicates no result returned.

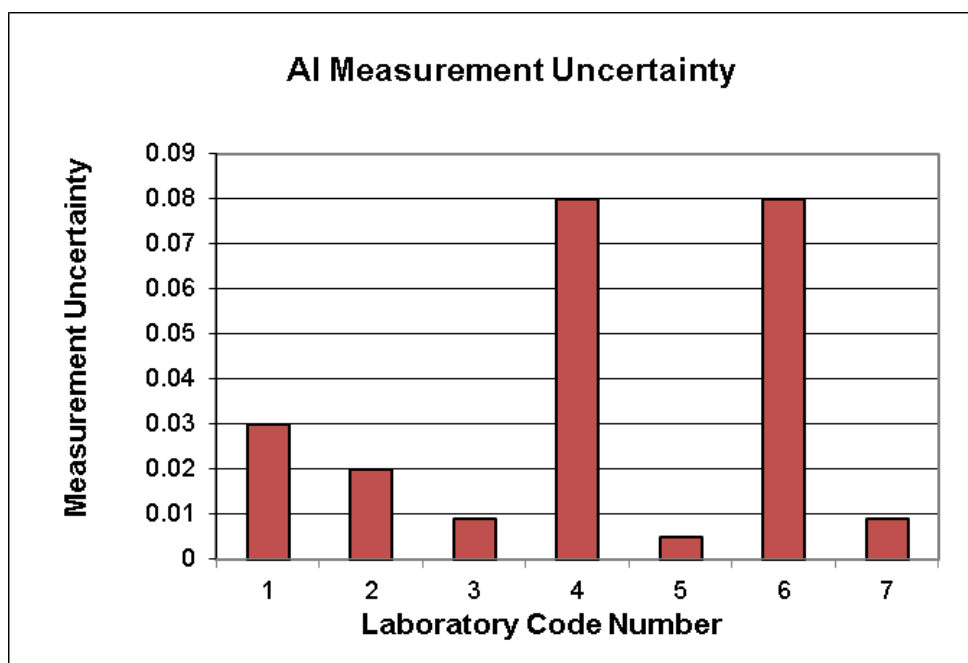
Summary Statistics

No. results	7
Median	0.7160
Norm IQR	0.0110
Robust CV	1.5%
Min	0.684
Max	0.840
Range	0.157
Uncertainty (Median)	0.0052

### Aluminium (Al) (%) Ordered Z-Score Chart



### Aluminium (Al) Measurement Uncertainty by Laboratory



## A2.1

**Arsenic (As) (%)**

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.009	0.009	0.009	0.000	0.01	0.005	6	Fused Bead	0.38
2	0.013	0.009	0.011	0.004	0.01	0.01	6	#	1.07
5	0.008	0.009	0.009	0.001	0.001	0.001	6	Li T/Li M flux fused bead	0.21

<sup>1</sup>Only a small number of numerical results have been returned (3) for this test. As the sample used for this round is a certified reference material, robust z-scores have been calculated using an assigned value and target CV (based on the certified value and standard deviation provided).

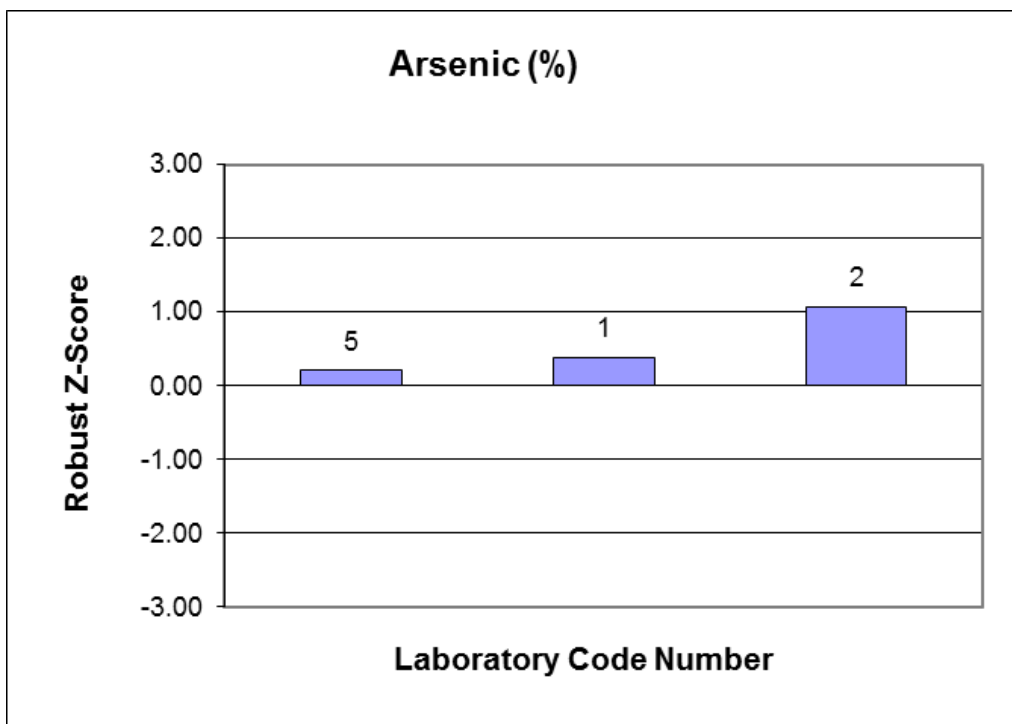
Notes:

# indicates no result returned.

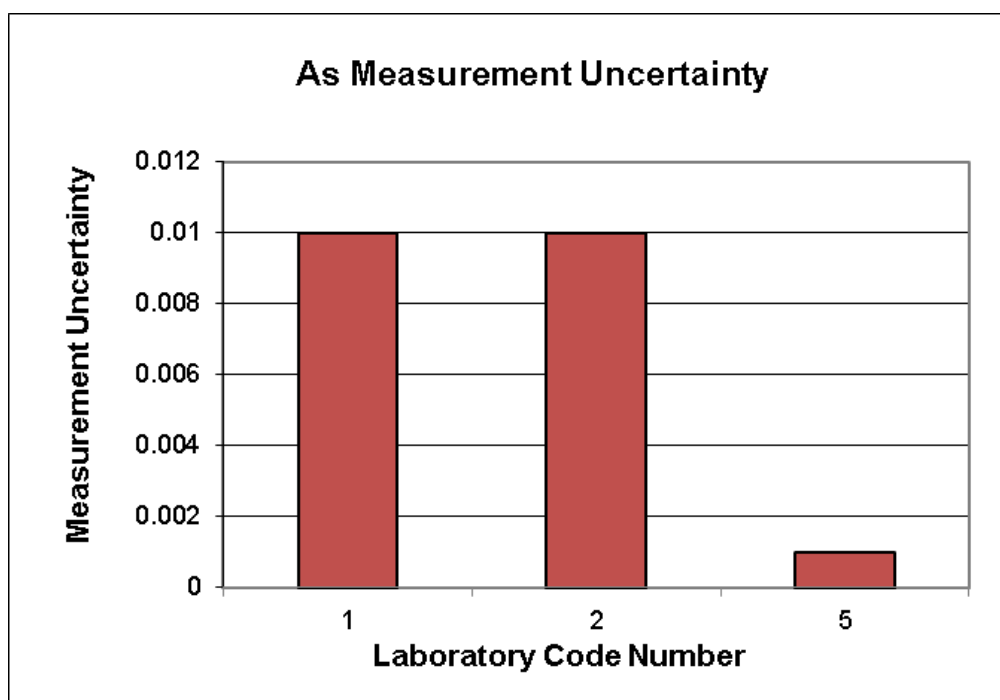
Summary Statistics

No. results	3
Assigned Value <sup>1</sup>	0.0079
Standard Deviation <sup>1</sup>	0.0029
Target CV <sup>1</sup>	36.7%
Min	0.009
Max	0.011
Range	0.003
Uncertainty (Assigned Value) <sup>1</sup>	0.0005

### Arsenic (As) (%) Ordered Z-Score Chart



### Arsenic (As) Measurement Uncertainty by Laboratory



**Calcium (Ca) (%)**

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.129	0.116	0.123	0.013	0.01	0.01	6	Fused Bead	0.93
2	0.119	0.120	0.120	0.001	0.05	0.01	6	#	-0.67
3	0.121	0.121	0.121	0.000	0.009	0.007	6	Fusion using Borate Flux	0.13
5	0.12	0.12	0.120	0.00	0.005	0.01	6	Li T/Li M flux fused bead	-0.40
6	0.121	0.120	0.121	0.001	0.02	0.01	6	12:22 XRF flux with 4% LiNO <sub>3</sub>	-0.13
7	0.142	0.124	0.133	0.018	0.009	0.007	6	Fusion using Borate Flux	6.52 §

Notes:

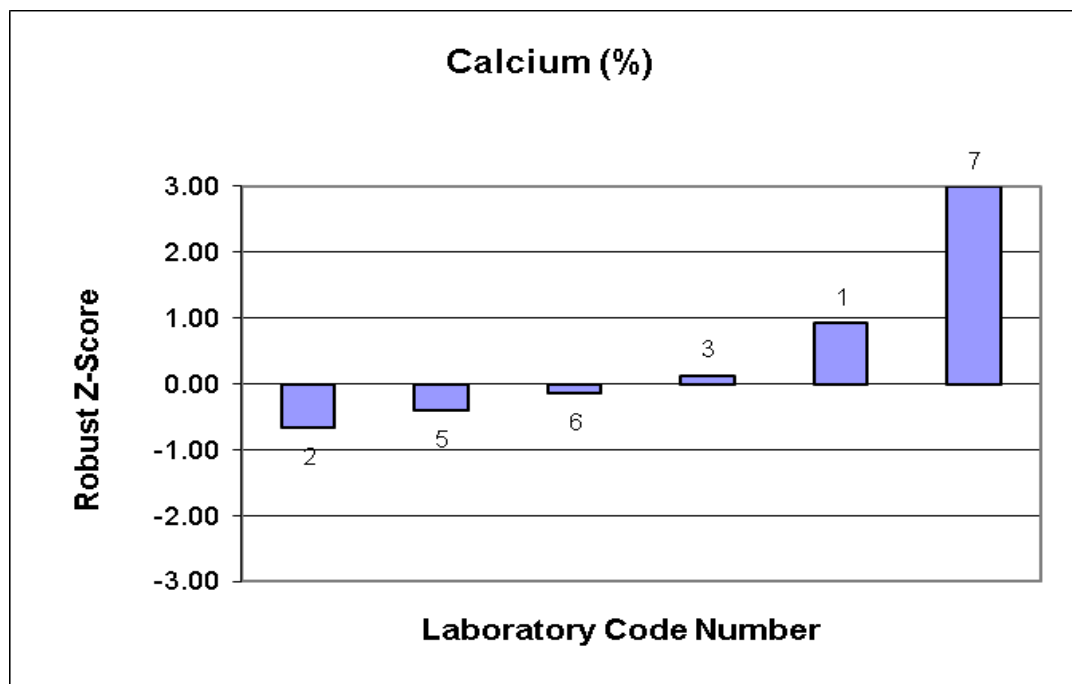
§ denotes an outlier, i.e.  $|z\text{-score}| \geq 3.0$ .

# indicates no result returned.

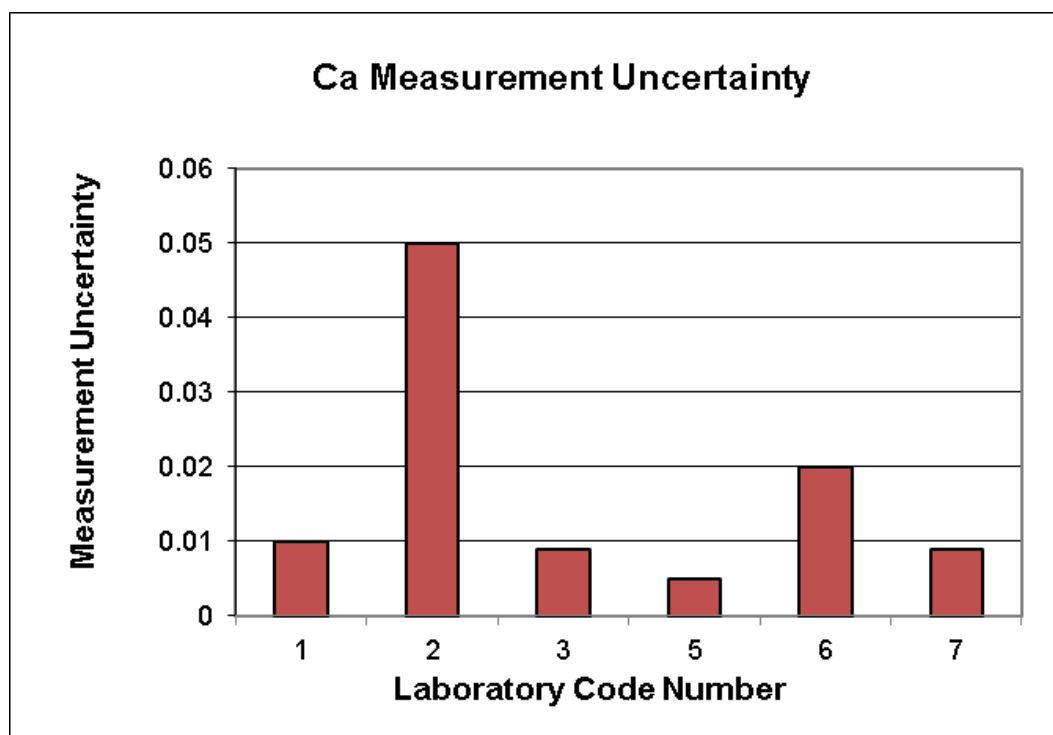
Summary Statistics

No. results	6
Median	0.1208
Norm IQR	0.0019
Robust CV	1.6%
Min	0.120
Max	0.133
Range	0.014
Uncertainty (Median)	0.0010

### Calcium (Ca) (%) Ordered Z-Score Chart



### Calcium (Ca) Measurement Uncertainty by Laboratory





**Copper (Cu) (%)**

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.003	0.003	0.003	0.000	0.01	0.002	6	Fused Bead	-4.75 §
2	0.022	0.024	0.023	0.002	0.05	0.01	6	#	11.92 §
5	0.011	0.011	0.011	0.000	0.001	0.001	6	Li T/Li M flux fused bead	1.92

<sup>1</sup>Only a small number of numerical results have been returned (3) for this test. As the sample used for this round is a certified reference material, robust z-scores have been calculated using an assigned value and target CV (based on the certified value and standard deviation provided).

Notes:

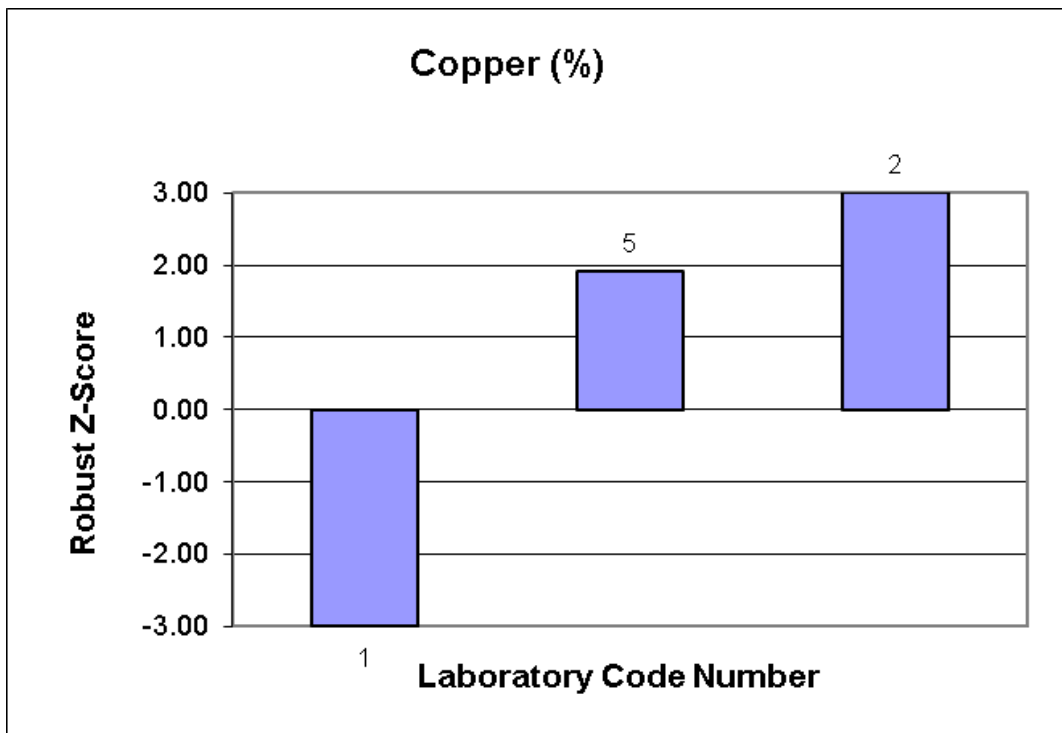
§ denotes an outlier, i.e.  $|z\text{-score}| \geq 3.0$ .

# indicates no result returned.

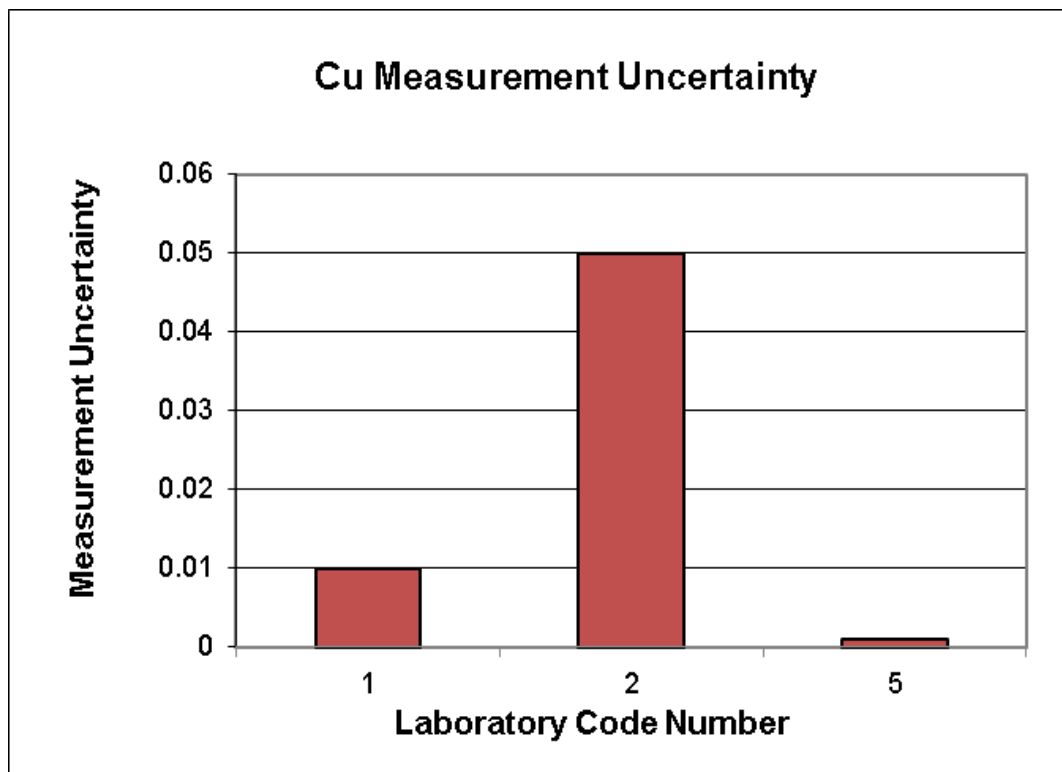
Summary Statistics

No. results	3
Assigned Value <sup>1</sup>	0.0087
Standard Deviation <sup>1</sup>	0.0012
Target CV <sup>1</sup>	13.8%
Min	0.003
Max	0.023
Range	0.020
Uncertainty (Assigned Value) <sup>1</sup>	0.0002

### Copper (Cu) (%) Ordered Z-Score Chart



### Copper (Cu) Measurement Uncertainty by Laboratory



## A5.1

## Iron (Fe) (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	62.28	62.40	62.34	0.12	0.32	0.01	6	Fused Bead	-0.11
2	66.91	62.78	64.85	4.13	0.30	0.01	6	#	2.39
3	63.36	63.39	63.38	0.03	0.02	0.07	6	Fusion using Borate Flux	0.92
4	62.2	62.7	62.45	0.5	1.3	0.05	6	Borate Fusion	0.00
5	62.20	62.23	62.22	0.03	0.04	0.01	6	Li T/Li M flux fused bead	-0.23
6	62.30	62.18	62.24	0.12	0.28	0.10	6	12:22 XRF flux with 4% LiNO <sub>3</sub>	-0.21
7	63.40	63.46	63.43	0.06	0.02	0.07	6	Fusion using Borate Flux	0.98

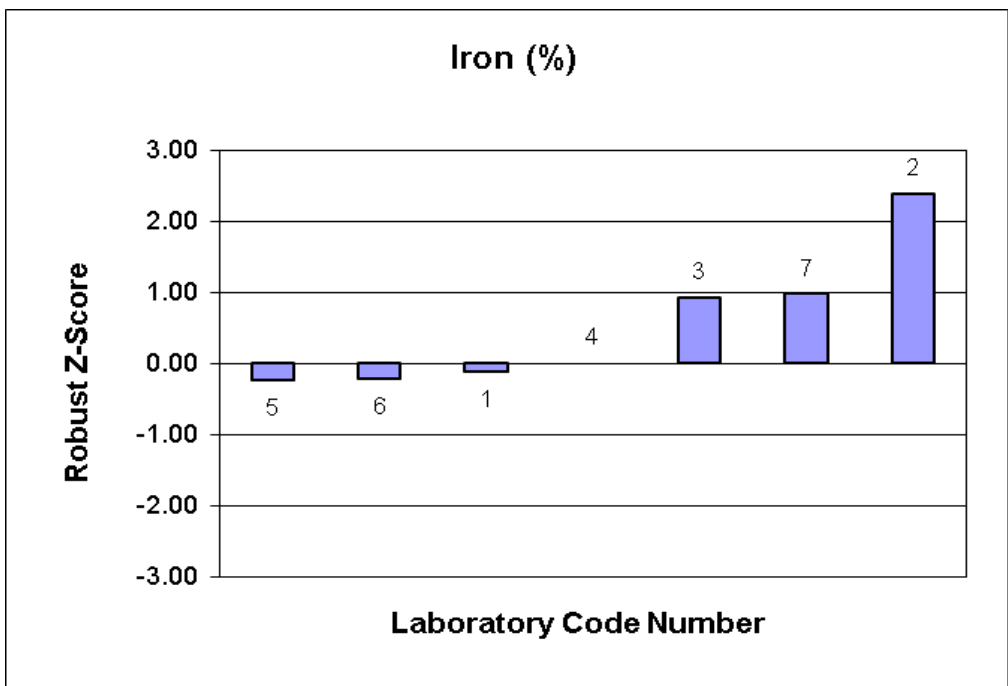
Notes:

# indicates no result returned.

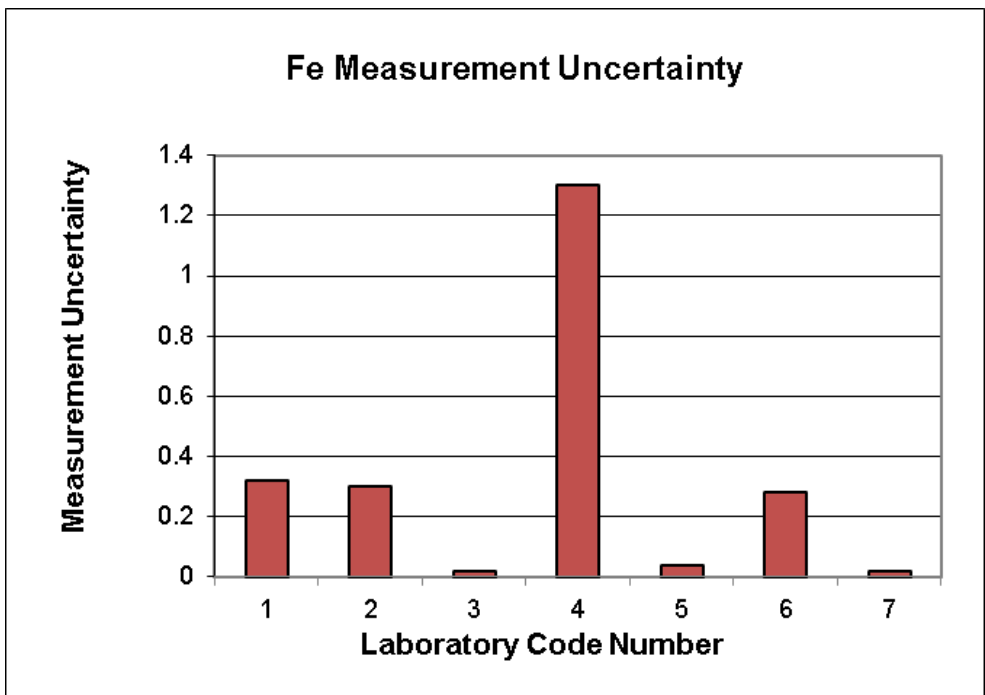
Summary Statistics

No. results	7
Median	62.450
Norm IQR	1.002
Robust CV	1.6%
Min	62.22
Max	64.85
Range	2.63
Uncertainty (Median)	0.475

**Iron (Fe) (%)  
Ordered Z-Score Chart**



**Iron (Fe)  
Measurement Uncertainty by Laboratory**



**Magnesium (Mg) (%)**

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.259	0.267	0.263	0.008	0.02	0.01	6	Fused Bead	6.57 §
2	0.131	0.112	0.122	0.019	0.02	0.01	6	#	-2.41
3	0.163	0.161	0.162	0.002	0.013	0.006	6	Fusion using Borate Flux	0.16
5	0.14	0.14	0.140	0.00	0.005	0.01	6	Li T/Li M flux fused bead	-1.24
6	0.162	0.158	0.160	0.004	0.03	0.01	6	12:22 XRF flux with 4% LiNO <sub>3</sub>	0.03
7	0.160	0.158	0.159	0.002	0.013	0.006	6	Fusion using Borate Flux	-0.03

Notes:

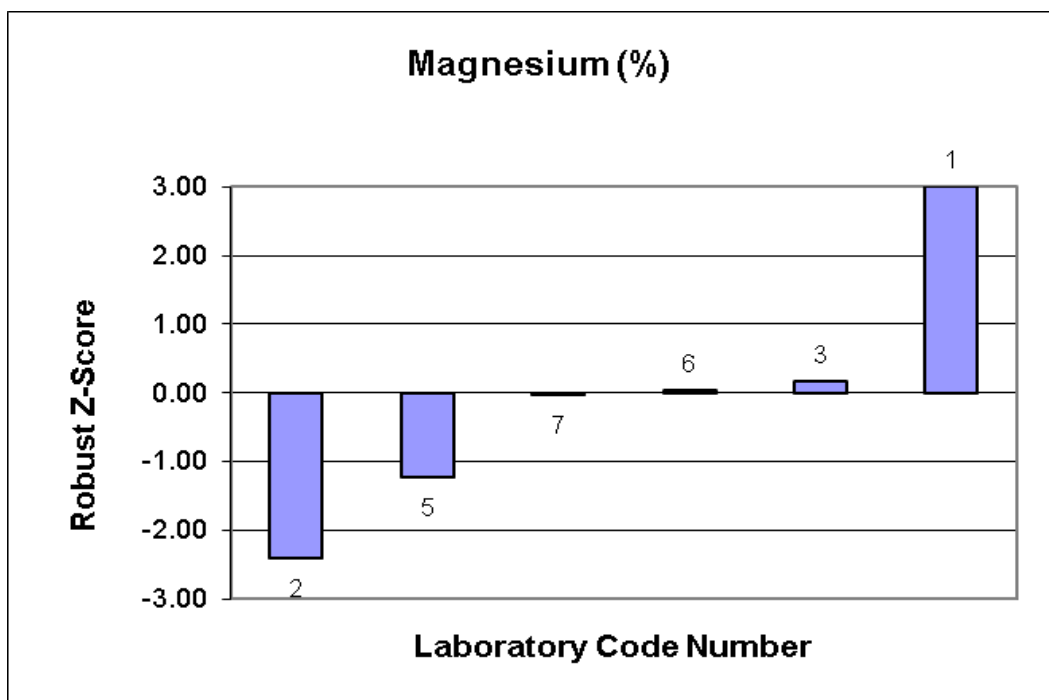
§ denotes an outlier, i.e. |z-score| ≥ 3.0.

# indicates no result returned.

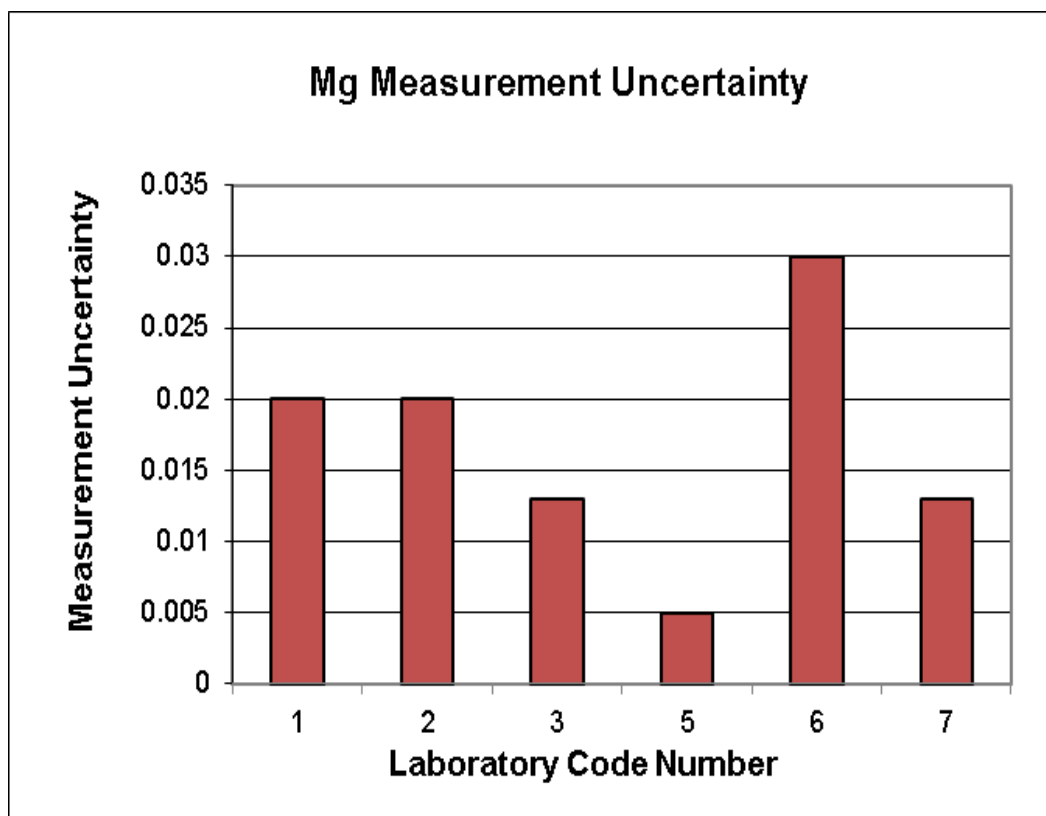
Summary Statistics

No. results	6
Median	0.1595
Norm IQR	0.0157
Robust CV	9.9%
Min	0.122
Max	0.263
Range	0.142
Uncertainty (Median)	0.0081

### Magnesium (Mg) (%) Ordered Z-Score Chart



### Magnesium (Mg) Measurement Uncertainty by Laboratory



**Manganese (Mn) (%)**

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.149	0.149	0.149	0.000	0.01	0.002	6	Fused Bead	-0.44
2	0.161	0.135	0.148	0.026	0.02	0.01	6	#	-0.50
3	0.168	0.167	0.168	0.001	0.006	0.008	6	Fusion using Borate Flux	0.76
5	0.15	0.15	0.150	0.00	0.005	0.01	6	Li T/Li M flux fused bead	-0.37
6	0.165	0.158	0.162	0.007	0.02	0.01	6	12:22 XRF flux with 4% LiNO <sub>3</sub>	0.37
7	0.167	0.167	0.167	0.000	0.006	0.008	6	Fusion using Borate Flux	0.73

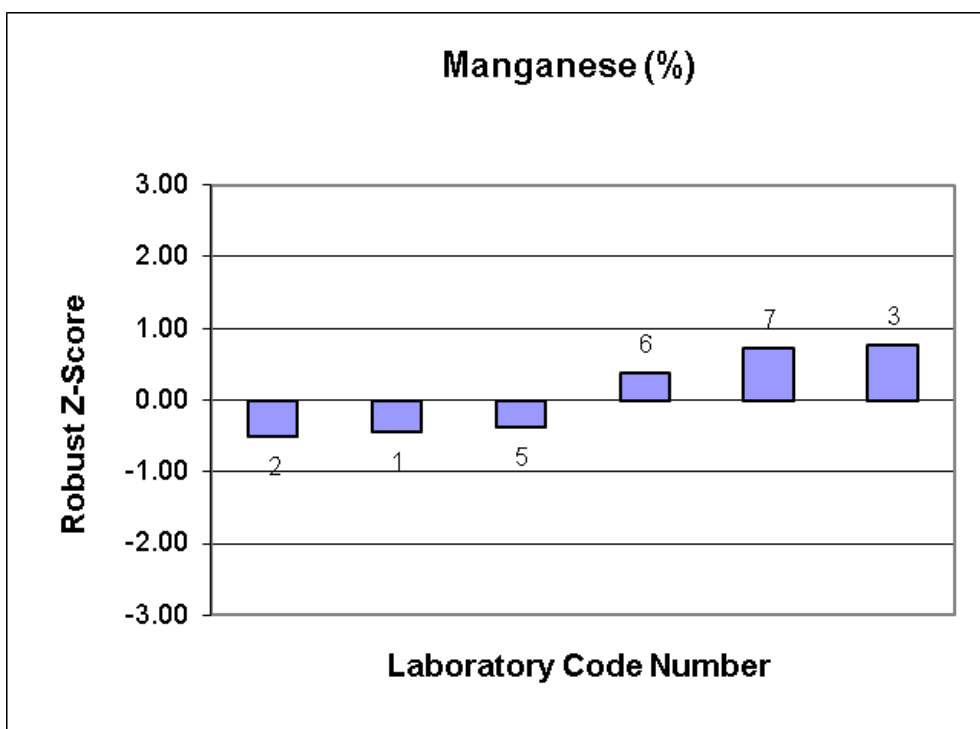
Notes:

# indicates no result returned.

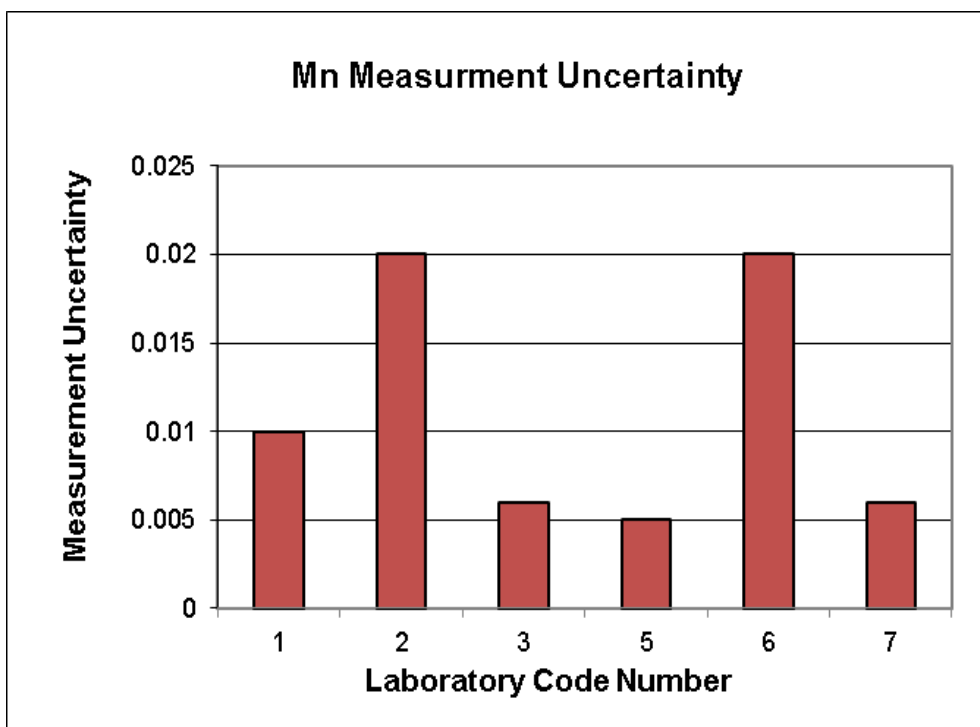
Summary Statistics

No. results	6
Median	0.1558
Norm IQR	0.0154
Robust CV	9.9%
Min	0.148
Max	0.168
Range	0.020
Uncertainty (Median)	0.0079

### Manganese (Mn) (%) Ordered Z-Score Chart



### Manganese (Mn) Measurement Uncertainty by Laboratory





**Phosphorous (P) (%)**

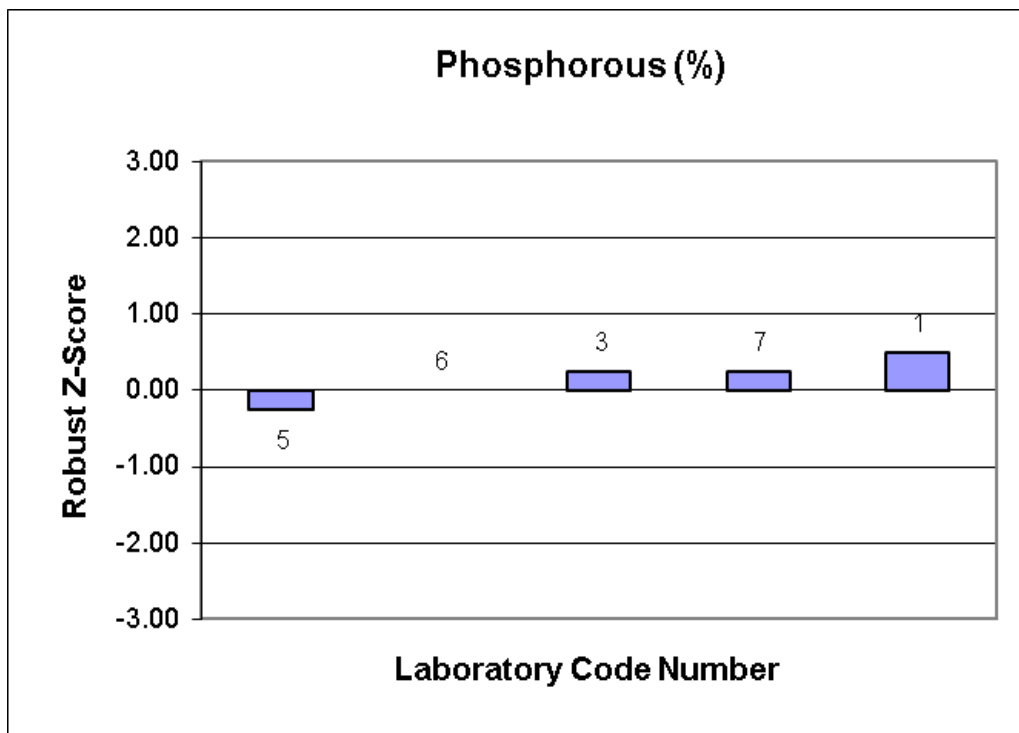
Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.079	0.079	0.079	0.000	0.01	0.001	6	Fused Bead	0.50
3	0.079	0.078	0.079	0.001	0.003	0.004	6	Fusion using Borate Flux	0.25
5	0.077	0.078	0.078	0.001	0.001	0.001	6	Li T/Li M flux fused bead	-0.25
6	0.078	0.078	0.078	0.000	0.0015	0.002	6	12:22 XRF flux with 4% LiNO <sub>3</sub>	0.00
7	0.079	0.078	0.079	0.001	0.003	0.004	6	Fusion using Borate Flux	0.25

<sup>1</sup>Only a small number of numerical results have been returned (5) for this test. As the sample used for this round is a certified reference material, robust z-scores have been calculated using an assigned value and target CV (based on the certified value and standard deviation provided).

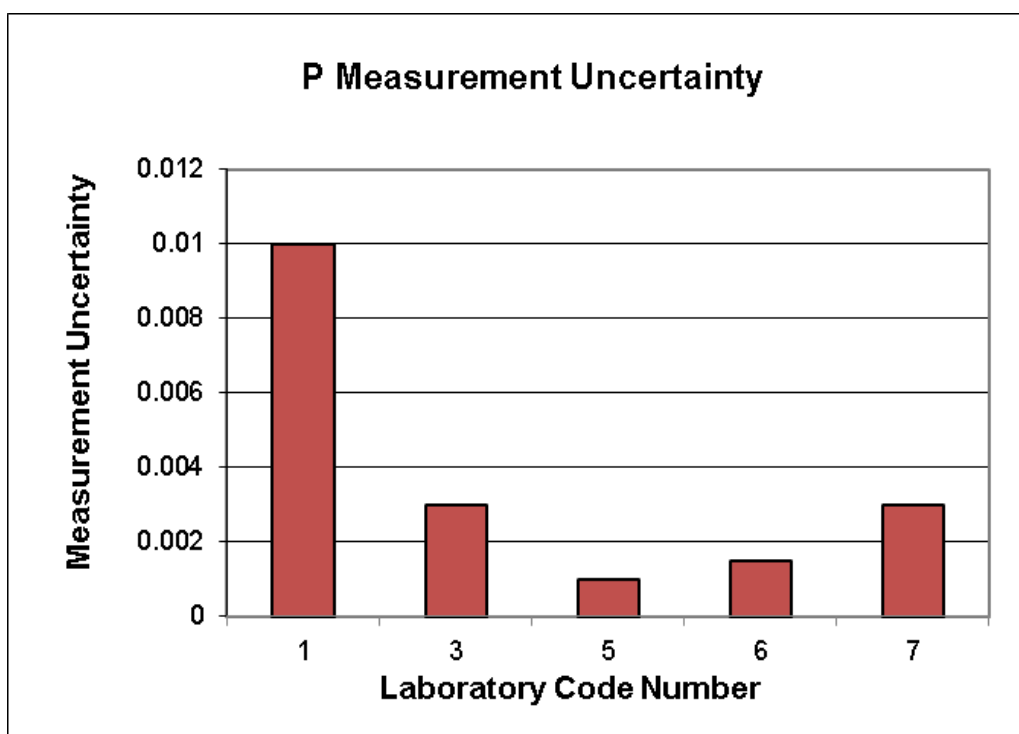
Summary Statistics

No. results	5
Assigned Value <sup>1</sup>	0.078
Standard Deviation <sup>1</sup>	0.002
Target CV <sup>1</sup>	2.6%
Min	0.078
Max	0.079
Range	0.002
Uncertainty (Assigned Value) <sup>1</sup>	0.0002

### Phosphorous (P) (%) Ordered Z-Score Chart



### Phosphorus (P) Measurement Uncertainty by Laboratory



**Potassium (K) (%)**

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.023	0.024	0.024	0.001	0.01	0.002	6	Fused Bead	0.95
2	0.032	0.016	0.024	0.016	0.01	0.01	6	#	1.42
3	0.022	0.022	0.022	0.000	0.008	0.008	6	Fusion using Borate Flux	-0.47
5	0.02	0.02	0.020	0.00	0.005	0.01	6	Li T/Li M flux fused bead	-2.36
6	0.023	0.022	0.023	0.001	0.004	0.002	6	12:22 XRF flux with 4% LiNO <sub>3</sub>	0.00
7	0.022	0.023	0.023	0.001	0.008	0.008	6	Fusion using Borate Flux	0.00

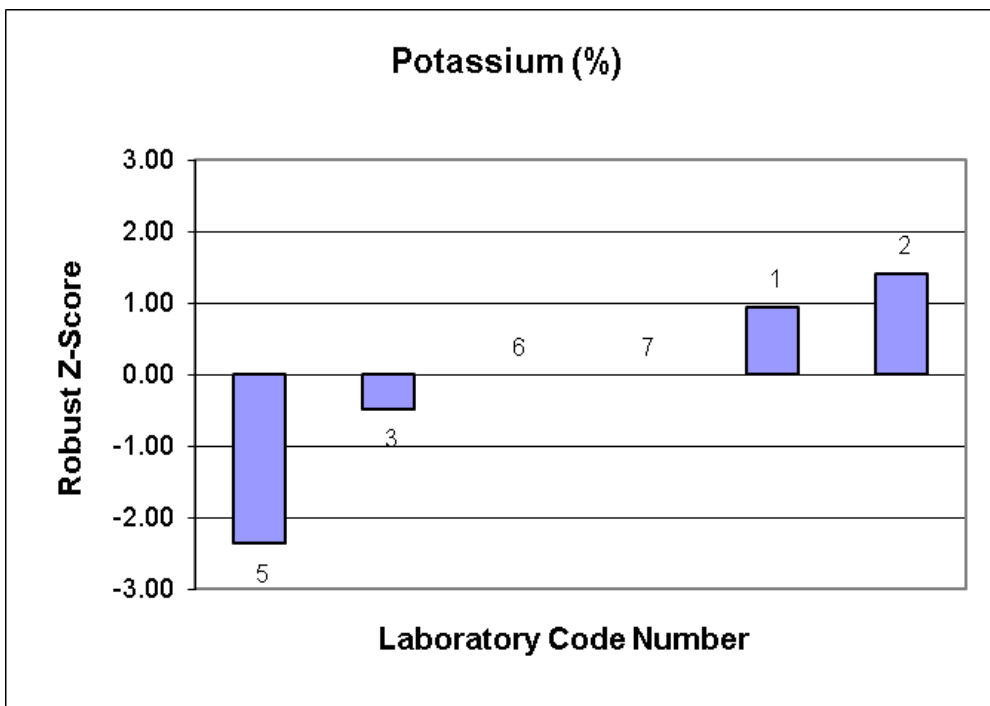
Notes:

# indicates no result returned.

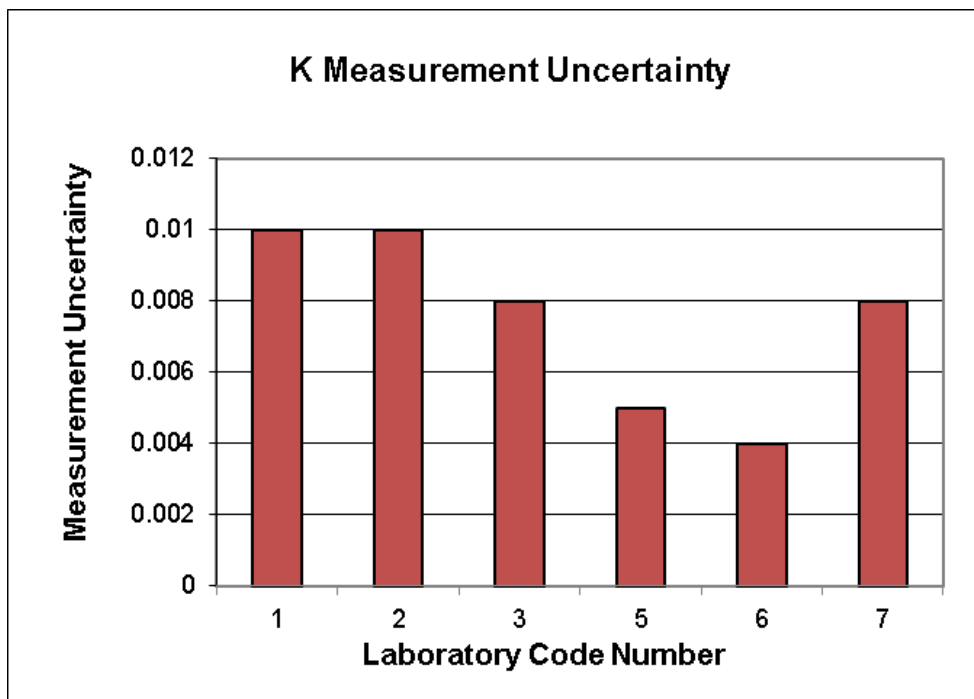
Summary Statistics

No. results	6
Median	0.0225
Norm IQR	0.0011
Robust CV	4.7%
Min	0.020
Max	0.024
Range	0.004
Uncertainty (Median)	0.0005

**Potassium (K) (%)  
Ordered Z-Score Chart**



**Potassium (K)  
Measurement Uncertainty by Laboratory**



**Silicon (Si) (%)**

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	2.036	1.999	2.02	0.037	0.04	0.01	6	Fused Bead	0.65
2	1.97	2.03	2.00	0.06	0.05	0.01	6	#	0.00
3	2.00	2.00	2.00	0.00	0.097	0.05	6	Fusion using Borate Flux	0.00
4	1.51	1.53	1.52	0.02	0.2	0.04	6	Borate Fusion	-17.76 §
5	1.98	1.99	1.99	0.01	0.01	0.01	6	Li T/Li M flux fused bead	-0.56
6	1.96	1.97	1.97	0.01	0.10	0.01	6	12:22 XRF flux with 4% LiNO <sub>3</sub>	-1.30
7	2.01	2.01	2.01	0.00	0.097	0.05	6	Fusion using Borate Flux	0.37

Notes:

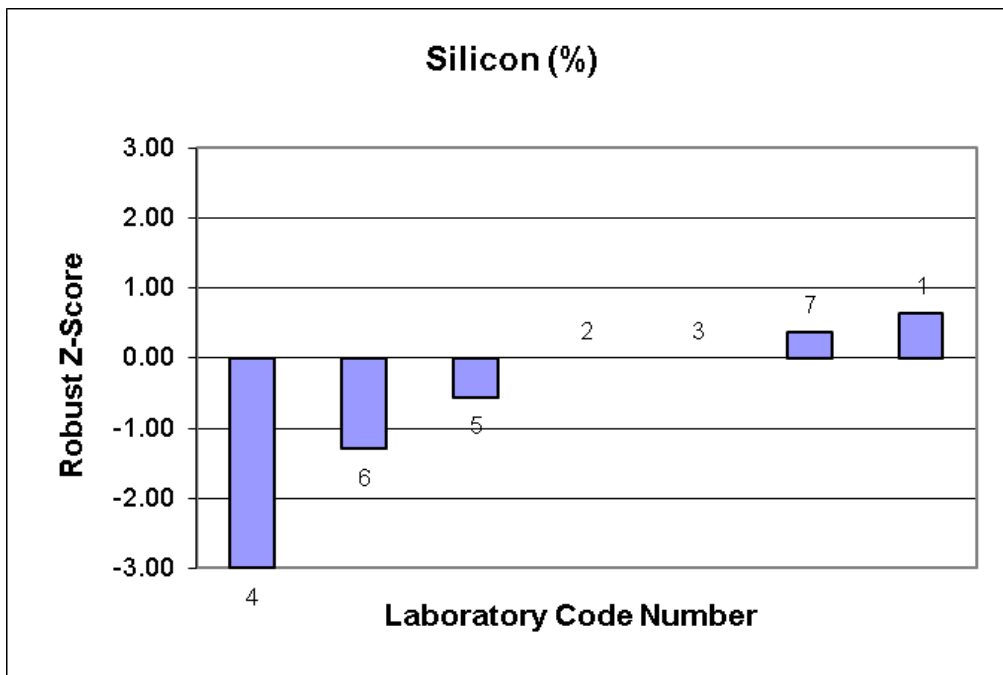
§ denotes an outlier, i.e. |z-score| ≥ 3.0.

# indicates no result returned.

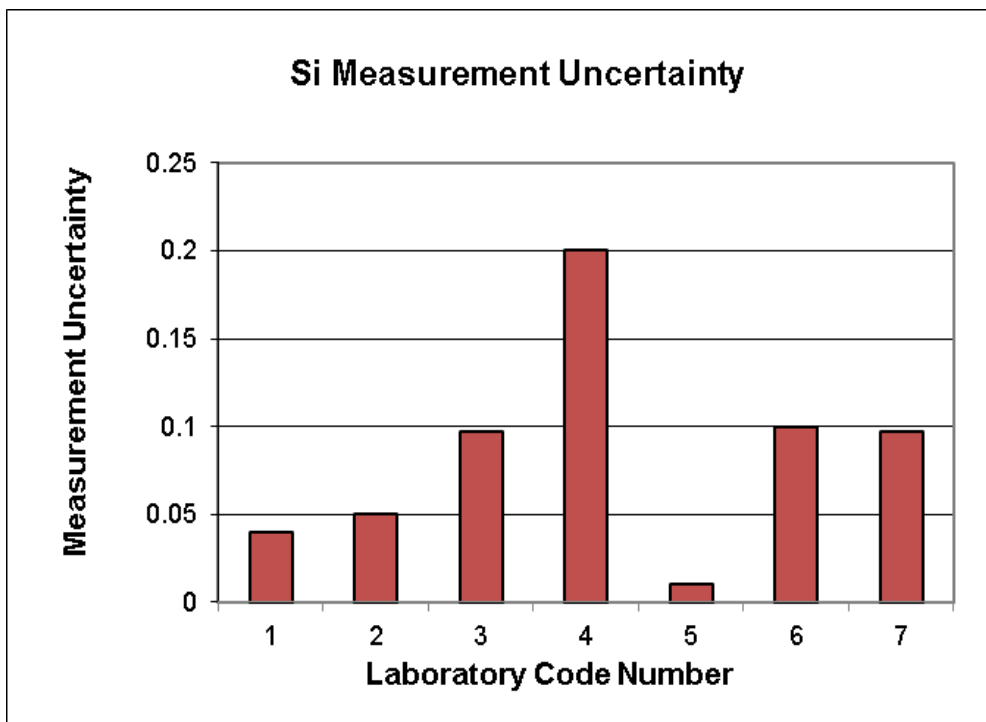
Summary Statistics

No. results	7
Median	2.000
Norm IQR	0.027
Robust CV	1.4%
Min	1.52
Max	2.02
Range	0.50
Uncertainty (Median)	0.013

**Silicon (Si) (%)  
Ordered Z-Score Charts**



**Silicon (Si)  
Measurement Uncertainty by Laboratory**



**Sodium (Na) (%)**

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.074	0.091	0.083	0.017	0.02	0.01	6	Fused Bead	1.40
2	0.047	0.028	0.038	0.019	0.02	0.01	1	#	-1.02
3	0.065	0.074	0.070	0.009	0.008	0.007	6	Fusion using Borate Flux	0.70
5	0.07	0.07	0.070	0.00	0.005	0.01	6	Li T/Li M flux fused bead	0.73
6	<0.050	<0.050	N/A	N/A	0.02	0.05	6	12:22 XRF flux with 4% LiNO <sub>3</sub>	N/A
7	0.070	0.068	0.069	0.002	0.008	0.007	6	Fusion using Borate Flux	0.67

<sup>1</sup>Only a small number of numerical results have been returned (5) for this test. As the sample used for this round is a certified reference material, robust z-scores have been calculated using an assigned value and target CV (based on the certified value and standard deviation provided).

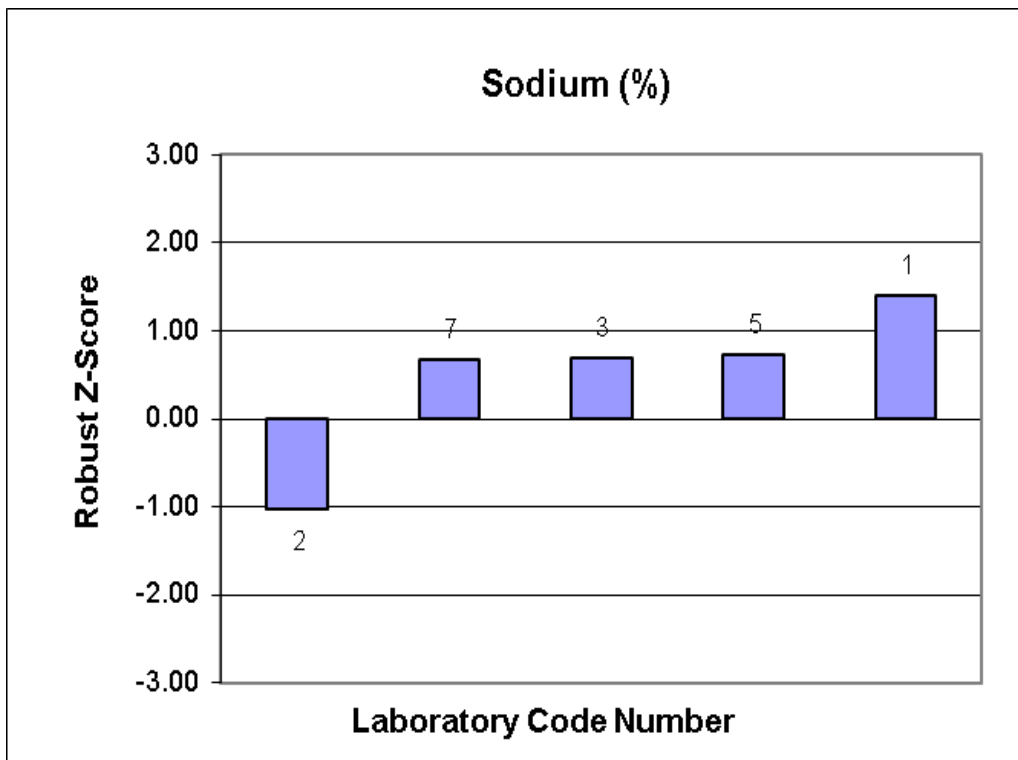
Notes:

# indicates no result returned.

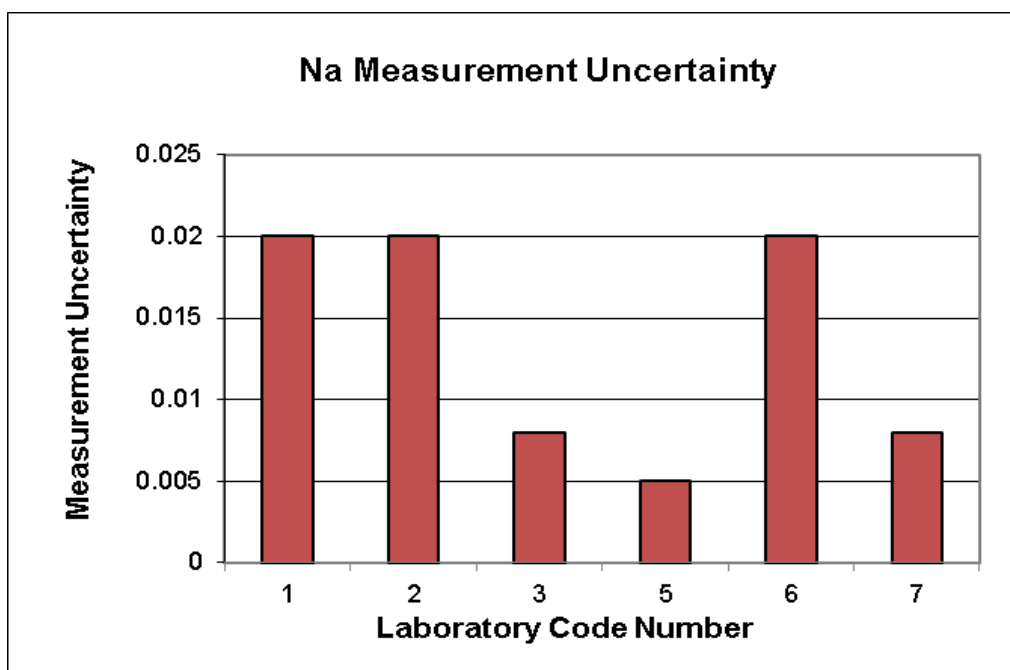
Summary Statistics

No. results	5
Assigned Value <sup>1</sup>	0.0565
Standard Deviation <sup>1</sup>	0.0186
Target CV <sup>1</sup>	32.9%
Min	0.038
Max	0.083
Range	0.045
Uncertainty (Assigned Value) <sup>1</sup>	0.0024

**Sodium (Na) (%)  
Ordered Z-Score Charts**



**Sodium (Na)  
Measurement Uncertainty by Laboratory**





**Sulphur (S) (%)**

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.058	0.054	0.056	0.004	0.01	0.001	6	Fused Bead	-0.53
2	0.049	0.031	0.040	0.018	0.10	0.01	6	#	-17.56 §
3	0.061	0.061	0.061	0.000	0.003	0.004	6	Fusion using Borate Flux	4.79 §
5	0.057	0.057	0.057	0.000	0.001	0.001	6	Li T/Li M flux fused bead	0.53
6	0.056	0.056	0.056	0.000	0.003	0.002	6	12:22 XRF flux with 4% LiNO <sub>3</sub>	-0.53
7	0.056	0.058	0.057	0.002	0.003	0.004	6	Fusion using Borate Flux	0.53

Notes:

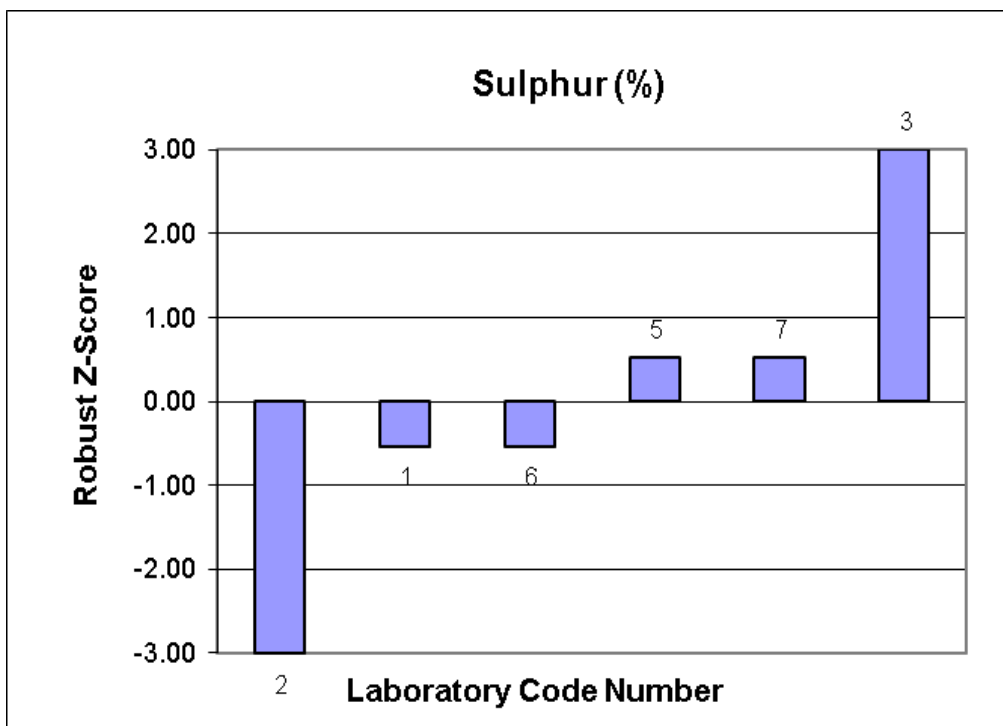
§ denotes an outlier, i.e.  $|z\text{-score}| \geq 3.0$ .

# indicates no result returned.

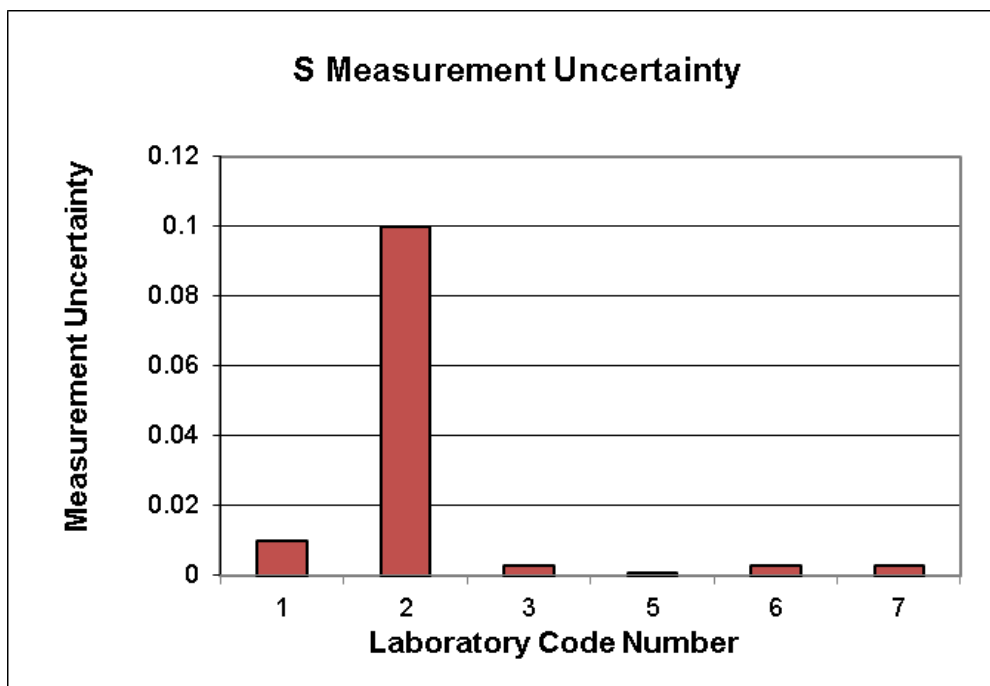
Summary Statistics

No. results	6
Median	0.0565
Norm IQR	0.0009
Robust CV	1.7%
Min	0.040
Max	0.061
Range	0.021
Uncertainty (Median)	0.0005

### Sulphur (S) (%) Ordered Z-Score Charts



### Sulphur (S) Measurement Uncertainty by Laboratory



**Titanium (Ti) (%)**

Lab Code	Result 1 (ppm)	Result 2 (ppm)	Average (ppm)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.045	0.046	0.046	0.001	0.01	0.01	6	Fused Bead	-0.91
2	0.047	0.042	0.045	0.005	0.05	0.01	6	#	-1.32
3	0.049	0.048	0.049	0.001	0.003	0.006	6	Fusion using Borate Flux	0.30
5	0.05	0.05	0.050	0.00	0.005	0.01	6	Li T/Li M flux fused bead	0.91
6	0.048	0.046	0.047	0.002	0.01	0.005	6	12:22 XRF flux with 4% LiNO <sub>3</sub>	-0.30
7	0.048	0.049	0.049	0.001	0.003	0.006	6	Fusion using Borate Flux	0.30

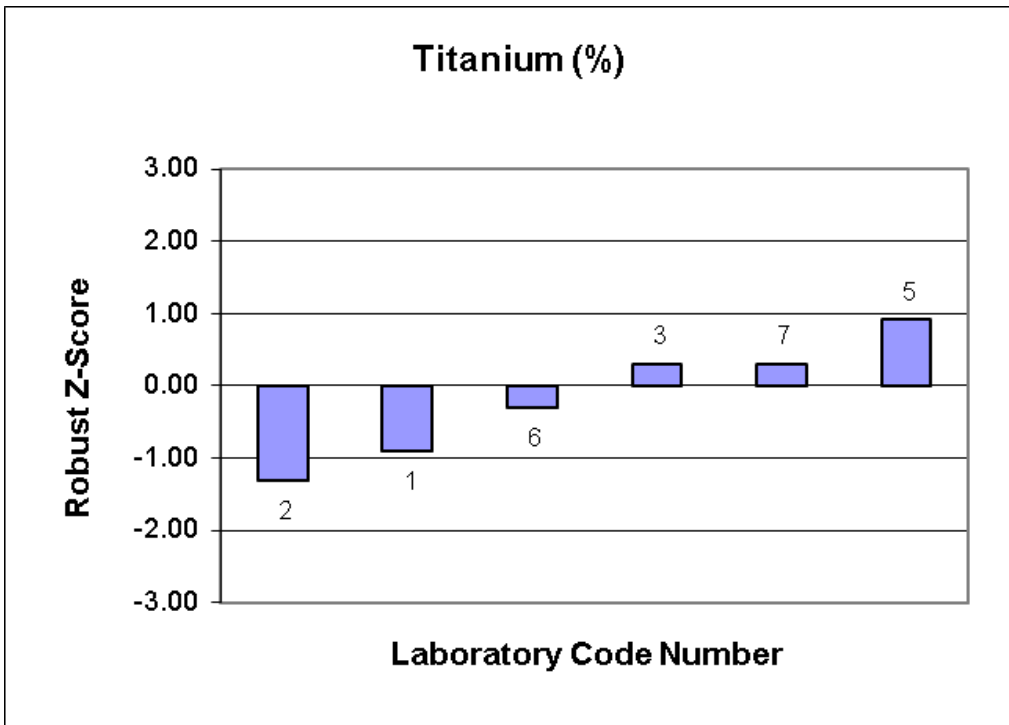
Notes:

# indicates no result returned.

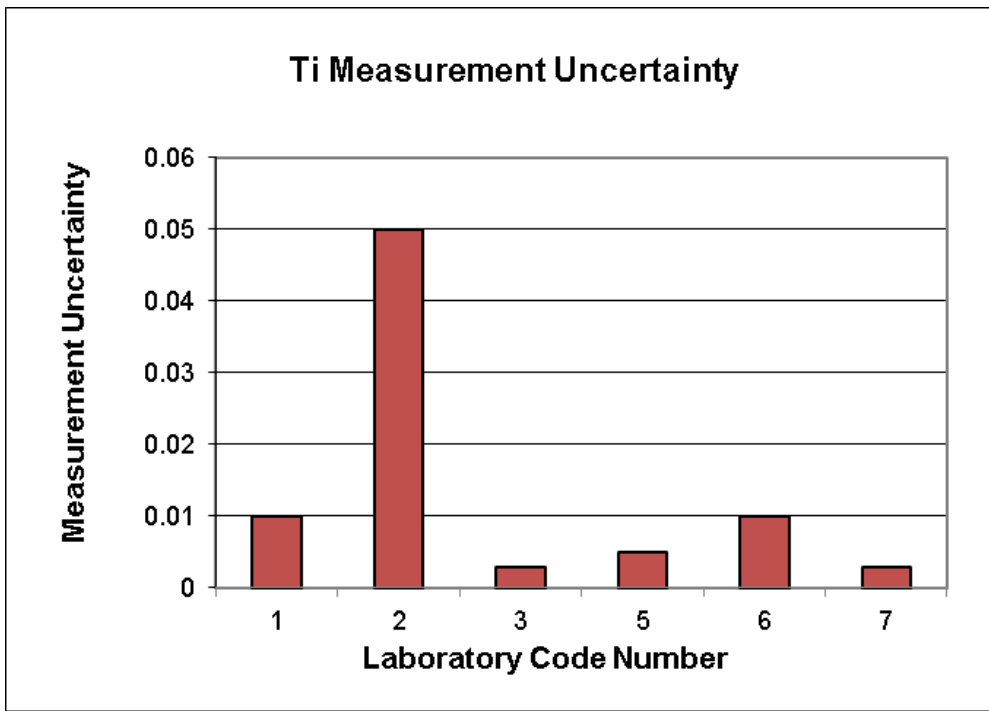
Summary Statistics

No. results	6
Median	0.0478
Norm IQR	0.0025
Robust CV	5.2%
Min	0.045
Max	0.050
Range	0.006
Uncertainty (Median)	0.0013

**Titanium (Ti) (%)  
Ordered Z-Score Charts**



**Titanium (Ti)  
Measurement Uncertainty by Laboratory**



### Loss on Ignition (LOI) (%)

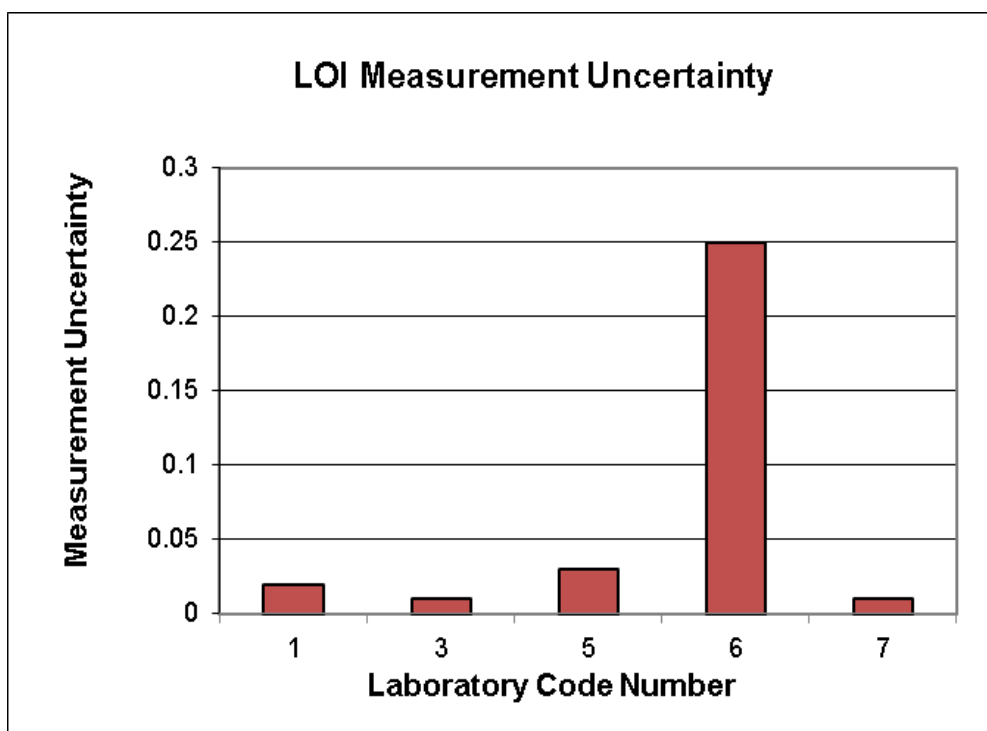
Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique
1	4.27	4.26	4.27	0.01	0.02	0.01	10	Leco TGA 701
3	4.47	4.44	4.46	0.03	0.01	0.005	11	#
5	4.17	4.15	4.16	0.02	0.03	0.01	11-TGA	#
6	4.37	4.35	4.36	0.02	0.25	0.10	11	#
7	4.32	4.29	4.31	0.03	0.01	0.005	11	N/A

Statistical analysis has not been performed as only a small number of numerical results (5) were returned.

#### Notes:

# indicates no result returned.

### Loss on Ignition (LOI) Measurement Uncertainty by Laboratory



# **APPENDIX B**

## **Sample Homogeneity**

## **Homogeneity Testing**

Samples dispatched for this round were Standards Australia certified reference material ASCRM-035 Iron Ore (Yilgarn Hematite) and therefore considered homogeneous and stable for the duration of the round.

# APPENDIX C

## Documentation

Instructions to Participants

C1

Results Sheet

C4



## PROFICIENCY TESTING AUSTRALIA

### Proficiency Testing Program Geochemical Testing – Round 12

#### INSTRUCTIONS TO PARTICIPANTS

Please read instructions carefully **BEFORE** commencing testing.

To ensure that the results of this program can be analysed properly, participants are asked to carefully note the following:

#### 1. Samples

- One **Iron Ore** sample with approximately 10g in a foil sachet has been provided for each laboratory.

**Samples have been thoroughly blended but please make sure your sample is thoroughly mixed before analysis and in between duplicates**

#### 2. Testing

- The testing should commence as soon as possible after receipt of the sample.
- Samples to be analysed for the below elements on a dry basis.

Aluminium	Al	Magnesium	Mg	Sodium	Na
Arsenic	As	Manganese	Mn	Sulphur	S
Calcium	Ca	Phosphorous	P	Titanium	Ti
Copper	Cu	Potassium	K	Loss on Ignition	LOI
Iron	Fe	Silicon	Si		

- Please Note: Where possible, proficiency testing samples should be treated as a routine laboratory sample.

#### 3. Safety

- The samples are for laboratory use only.
- All required safety procedures should be followed.

#### 4. Reporting

- Please submit results on the Results Sheet provided.
- Duplicate results are requested.
- Please specify the dissolution/digestion technique used and record the method of analysis using the attached codes (refer to page 3). Details should be provided of any method techniques that are used that are not specified in the table on page 3.
- Results should be quoted in elemental form as in listed in 2 (on previous page) and on the results sheet.
- Please report each element to the units (% or ppm) indicated on the Results Sheet along with your laboratory's detection limit for that analysis.
- Laboratories are requested to calculate and report an estimate of uncertainty of measurement for each reported measurement result.
- All estimates of uncertainty of measurement must be given as a 95% confidence interval (coverage factor  $k \approx 2$ ). Please note that MU will not be used to evaluate participant performance in this program.
- The following significant figures are recommended for reporting:  
XX.XX%, X.XX%, 0.XXX%, 0.00XX%.

5. Please return results no later than **FRIDAY 6 JANUARY 2017** to:

**Kathy Weller**

**fax: +61 7 3217 1844**

**email: [Kathy.Weller@pta.asn.au](mailto:Kathy.Weller@pta.asn.au)**

6. For this program your laboratory has been allocated the code number shown on the results sheet. All reference to your laboratory in reports associated with this program will be by this code number, thus ensuring confidentiality of results.

**Analysis Method Codes to be used for the Results Sheets**

<b>Method Technique</b>	<b>Method Code</b>
Inductively coupled plasma atomic emission spectrometry	1
Inductively coupled plasma mass spectrometry	2
Atomic absorption spectrometry	
Flame	3
Graphite furnace	4
Hydride generation	5
X-ray fluorescence spectrometry	6
Classical wet chemical analysis	7
Colorimetric	8
Neutron activation analysis	9
Leco combustion analysis	10
Other – please specify	11

Please use a Method Code for each element tested.

**PROFICIENCY TESTING AUSTRALIA**
**Geochemical – Round 12 - Proficiency Testing Program**
**Results Sheet**

Lab Code:

7

Analysis	Result 1	Result 2	Units	Detection Limit	Dissolution/ Digestion Technique (Please Specify)	Method Code	±MU*
Aluminium, Al			%				
Arsenic, As			%				
Calcium Ca			%				
Copper, Cu			%				
Iron, Fe			%				
Magnesium, Mg			%				
Manganese, Mn			%				
Phosphorous, P			%				
Potassium, K			%				
Silicon, Si			%				
Sodium, Na			%				
Sulphur, S			%				
Titanium, Ti			%				
Loss on Ignition (LOI)			%				

**MU\* Laboratories' Uncertainty of Measurement. Please report in the same units as the results for each element.**

Return no later than **FRIDAY 6 JANUARY 2017**, to:

Kathy Weller, Proficiency Testing Australia.

phone: +61 7 3721 7373, fax: +61 7 3217 1844, email: [Kathy.Weller@pta.asn.au](mailto:Kathy.Weller@pta.asn.au)

- *End of Report* -