

GEOCHEMICAL ROUND 8

PROFICIENCY TESTING PROGRAM

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REPORT NO. 800

ACKNOWLEDGMENTS

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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 2012 / January 2013 by Proficiency Testing Australia (PTA). The Program Coordinator was Mrs K Weller. This report was authorised by Ms W Fajloun, Quality Coordinator. 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	Manganese	Mn	Sodium	Na
Calcium	Ca	Phosphorus	P	Sulphur	S
Iron	Fe	Potassium	K	Titanium	Ti
Magnesium	Mg	Silicon	Si	Loss on Ignition	LOI

- 2.4 A total of 11 separate laboratories received samples for the program. All laboratories returned results by the due date for inclusion in the final report. To ensure confidentiality, each laboratory was allocated a random code number for each sample. Reference to each laboratory in this report is by its code number.

Participants included laboratories from Australia, India and Korea.

- 2.5 Results (as reported by participants), with 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 (for each sample and for each of the analyses performed).
- 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 relevant to each test are presented in Appendix A.

The document entitled *Guide to Proficiency Testing Australia, 2011* (reference [1]) defines the statistical terms and details the statistical procedures referred to in this report.

- 2.7 Previously, 10 randomly selected samples were analysed for homogeneity. Based on the results of this testing it was considered that the samples utilised for this program were homogenous. As such, any results later identified as extreme could not be attributed to any notable sample variability. Please refer to Appendix B.

3. Statistical Format

For each test the following information is given:

- 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 "§".

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 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.

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 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;
- the range (*Maximum - Minimum*); and
- the Uncertainty of the Median.

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 and -3, 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 is given in reference [1]. Please also refer to this document for a glossary of terms.

TABLE A: SUMMARY STATISTICS

Analysis	No. of Results	Median (%)	Normalised IQR	Uncertainty (Median) (%)
Aluminium	11	1.157	0.030	0.011
Calcium	9	0.0300	0.0131	0.0055
Iron	11	63.095	0.135	0.051
Magnesium	10	0.0393	0.0030	0.0012
Manganese	10	0.2140	0.0093	0.0037
Phosphorus	9	0.0730	0.0039	0.0016
Potassium	10	0.0113	0.0020	0.0008
Silicon	10	1.563	0.033	0.013
Sodium	9	0.0200	0.0074	0.0031
Sulphur	10	0.0200	0.0037	0.0015
Titanium	9	0.0600	0.0096	0.0040
LOI (1000)	10	3.423	0.164	0.065

4. PTA and Technical Adviser's Comments

This program was aimed at laboratories routinely analysing Iron Ore samples for major elements. The comments presented in this section are general in nature.

4.1 Overall performance:

The general performance was satisfactory and agreement between techniques was generally good. Some variation between laboratories can be seen in the results, detection limit and measurement uncertainty. However, this variation is not method dependent as there are wide ranges of detection limits within methods e.g. XRF. This gives an indication of the differences between the laboratories processes.

Microwave digestion seems to give lower results for some elements than the other techniques.

4.2 Outliers

Seven out of eleven laboratories exhibited outliers. Table B shows outlier results and the techniques used.

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

4.3 Measurement Uncertainty (MU)

Most laboratories made a good effort and quantified MU.

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 z scores:

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

4.5 Duplicates

Duplicate results for most laboratories were reasonable. The permissible tolerance guidelines from ISO 9516-1:2003 [2] for iron ores determination by XRF were used to calculate P (permissible tolerance between duplicates). If the difference between the laboratory's duplicates is greater than P, this is highlighted in the results tables in Appendix A under the "Absolute Difference" column. This guide was applied based on the fact that most analysis in this round was performed by XRF. The guide does not apply for other methods.

4.6 Technique Bias

Based on the z-score analysis, there is no evidence to support technique bias.

4.7 Method Code

All laboratories provided a method code except laboratory 10.

4.8 Metrological Traceability and Measurement Uncertainty of Assigned Values

Consensus values (median) derived from participants' results are used in this program. These values are not metrologically traceable to an external reference.

The sample chosen for this program was provided by Hamersley Iron, WA. The bulk sample was sent to CSIRO, Division of Exploration and Mining, NSW, where the ore was crushed and divided into small plastic containers.

As the assigned value for this program is the median of the results submitted by the participants, the uncertainty of the median has been calculated for each analysis and is tabulated in Table A on page 4, and also in the summary statistics tables for each test in Appendix A.

4.9 Analysis of Results by Method Groups

In order for methods to be grouped for analysis, PTA requires more than 10 sets of results from the same method group. As there were less than 10 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

TABLE B: OUTLIER RESULTS

Laboratories reporting outlier results are listed in the following table:

Element	Lab Code	Bias	Method Code	Dissolution/Digestion Technique
Aluminium	4	-ve	7 – Classical Wet chemistry	Dissolution in HCl medium
	5	+ve	6 - XRF	Glass Fusion
Calcium	1	+ve	6 - XRF	Borate Fusion
	4	+ve	1 - ICPOES	Microwave Digestion
Iron	1	-ve	6 - XRF	Borate Fusion
	3	+ve	6 - XRF	Borate Flux
Magnesium	1	-ve	3 - AAS	Three acid digest - HCl, HF and HNO ₃
	10	+ve	Not reported	Not reported
Manganese	No outliers reported			
Phosphorus	8	-ve	6 - XRF	XRF-fusion
Potassium	3	-ve	6 - XRF	Borate Flux
	8	+ve	6 - XRF	XRF-fusion
	11	+ve	1 - ICPOES	HCl + HNO ₃ + HClO ₄
Silicon	5	+ve	6 - XRF	Glass Fusion
	11	+ve	11 - GRAVIMETRIC	Gravimetric measurement, HCl + HNO ₃ + HClO ₄
Sodium	5	+ve	6 - XRF	Glass Fusion
	8	+ve	6 - XRF	XRF-fusion
Sulphur	8	+ve	6 - XRF	XRF-fusion
Titanium	5	+ve	6 - XRF	Glass Fusion
Loss on Ignition	11	-ve	11 - GRAVIMETRIC	Gravimetric measurement

6. References

- [1] *Guide to Proficiency Testing Australia*, 2011.
- [2] ISO 9516-1:2003 Iron Ores - Determination of various elements by Xray fluorescence spectrometry - Part 1: Comprehensive procedure.

APPENDIX A

Results and Data Analysis

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

Aluminium (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference P = 0.03	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	1.13	1.13	1.13	0.00	0.057	#	6	borate fusion	-0.91
2	1.165	1.165	1.17	0.000	0.033	0.005	6	Fused disc XRF with meta/tetra-borate flux containing LiNO ₃	0.27
3	1.15	1.14	1.15	0.01	0.03	0.005	6	Borate Flux	-0.40
4	1.02	1.08	1.05	0.06	0.170 at 3.03	#	7	Dissolution in HCl Medium	-3.61 §
5	2.13	2.14	2.14	0.01		0.01	6	Glass Fusion	32.98 §
6	1.163	1.151	1.16	0.012	0.03	0.01	6	Li Borate Fused Bead	0.00
7	1.14	1.14	1.14	0.00	0.013	0.01	6	Fusion Lithium Borate	-0.57
8	1.16	1.21	1.19	0.05	0.2	0.1	6	XRF-fusion	0.94
9	1.13	1.15	1.14	0.02	#	#	1	Sodium peroxide fusion	-0.57
10	1.19	1.18	1.19	0.01	#	0.01	#	#	0.94
11	1.17	1.18	1.18	0.01	0.03	0.0009	1	HCl + HNO ₃ + HClO ₄	0.61

Notes

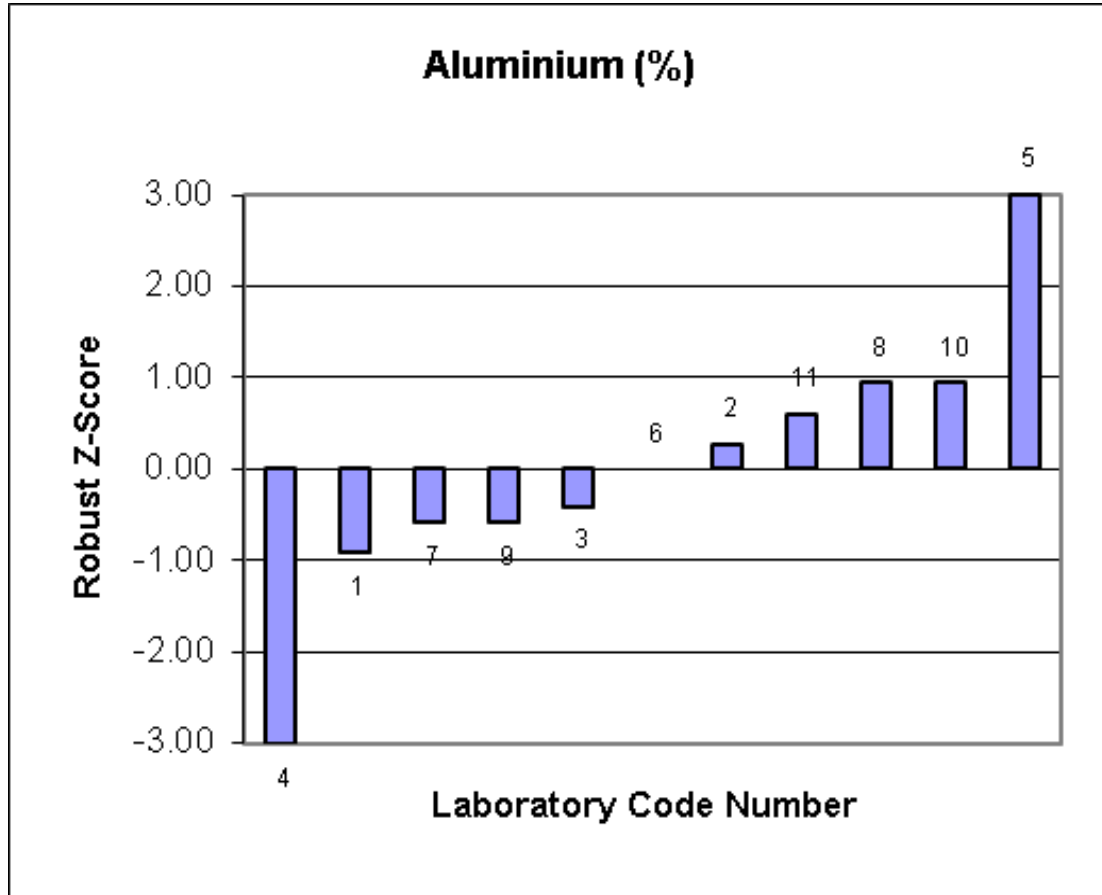
§ denotes an outlier.

indicates no result returned.

P is permissible tolerance between duplicates as calculated from ISO 9516-1:2003 (applicable to Method Code 6 only, i.e. XRF)

Summary Statistics

No. results	11
Median	1.157
Norm IQR	0.030
Robust CV	2.6%
Min	1.05
Max	2.14
Range	1.09
Uncertainty (Median)	0.011



Calcium (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference P = 0.004	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.10	0.10	0.100	0.00	0.005	#	6	borate fusion	5.34 §
2	0.021	0.021	0.021	0.000	0.014	0.007	6	Fused disc XRF with meta/tetra-borate flux containing LiNO ₃	-0.69
3	0.020	0.024	0.022	0.004	0.03	0.005	6	Borate Flux	-0.61
4	0.073	0.074	0.074	0.001	0.0097 at 0.288	0.00029%	1	Microwave Digestion	3.32 §
5	0.040	0.030	0.035	0.010		0.01	6	Glass Fusion	0.38
6	0.019	0.018	0.019	0.001	0.005	0.005	6	Li Borate Fused Bead	-0.88
7	0.03	0.03	0.030	0.00	0.006	0.01	6	Fusion Lithium Borate	0.00
8	<0.0001	<0.0001	*	*	0.2	0.1	6	XRF-fusion	*
9	<0.1	<0.1	*	*	#	#	1	Sodium peroxide fusion	*
10	0.02	0.02	0.020	0.00	#	0.01	#	#	-0.76
11	0.038	0.034	0.036	0.004	0.004	0.0009	1	HCl + HNO ₃ + HClO ₄	0.46

Notes:

* statistics could not be performed for these results (Laboratories 8 and 9).

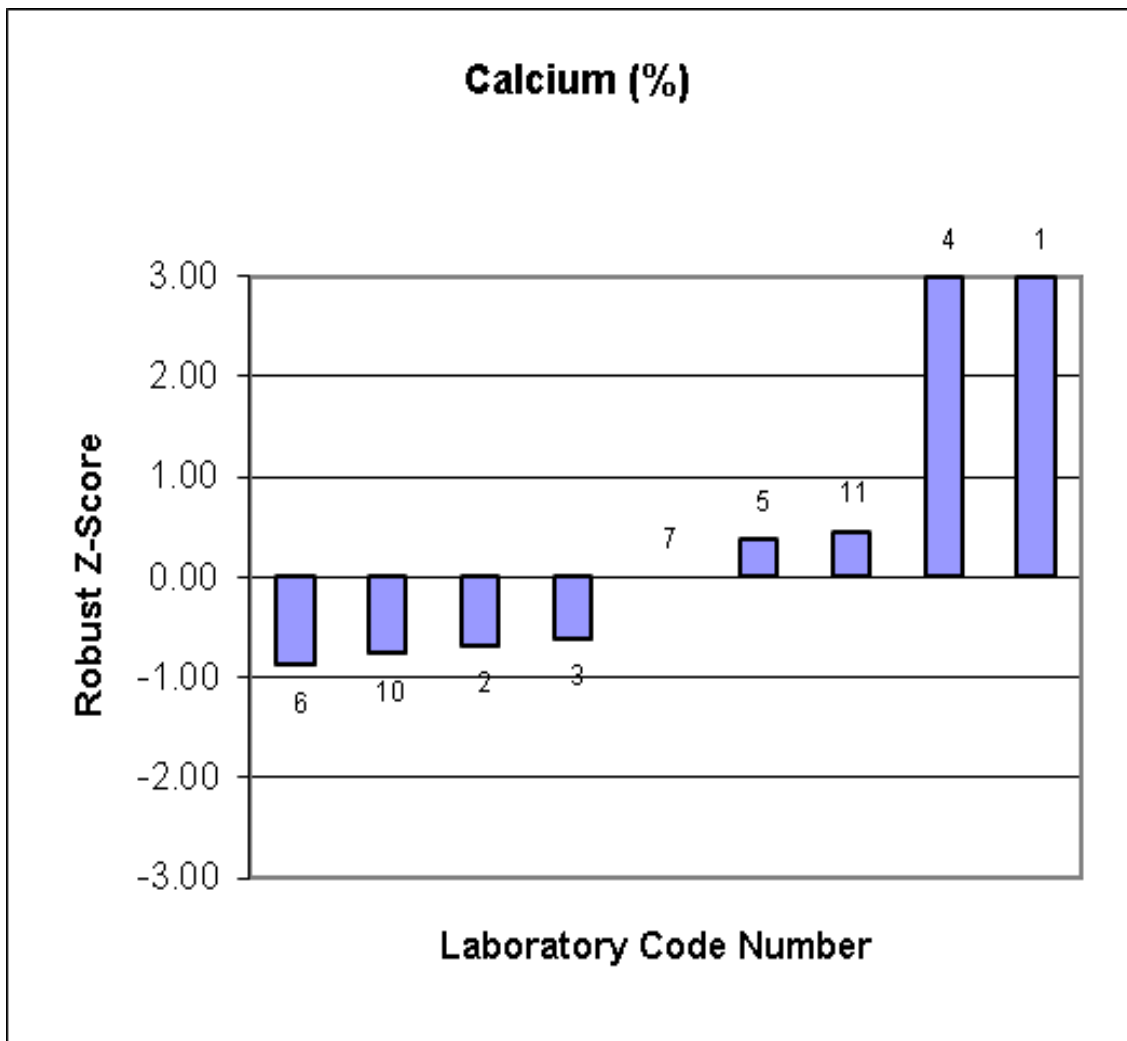
§ denotes an outlier.

indicates no result returned.

P is permissible tolerance between duplicates as calculated from ISO 9516-1:2003 (applicable to Method Code 6 only, i.e. XRF)

Summary Statistics

No. results	9
Median	0.0300
Norm IQR	0.0131
Robust CV	43.7%
Min	0.019
Max	0.100
Range	0.082
Uncertainty (Median)	0.0055



Iron (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference P = 0.3985	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	62.4	62.5	62.45	0.1	3.1	#	6	borate fusion	-4.77 §
2	63.18	63.15	63.17	0.03	0.65	0.01	6	Fused disc XRF with meta/tetra-borate flux containing LiNO ₃	0.52
3	63.89	63.43	63.66	0.43	0.01	0.005	6	Borate Flux	4.18 §
4	63.10	62.92	63.01	0.18	0.210 at 65.56	#	7	Dissolution in HCl Medium	-0.63
5	63.22	63.20	63.21	0.02	#	0.01	6	Glass Fusion	0.85
6	63.06	63.13	63.10	0.07	0.20	0.01	6	Li Borate Fused Bead	0.00
7	63.08	63.16	63.12	0.08	0.194	0.01	6	Fusion Lithium Borate	0.18
8	63.00	63.00	63.00	0.00	0.5	0.1	6	XRF-fusion	-0.70
9	62.87	63.02	62.95	0.15	#	#	11 volumetric	Sodium peroxide fusion	-1.11
10	63.02	63.01	63.02	0.01	#	0.01	#	#	-0.59
11	63.54	63.37	63.46	0.17	0.69	0.005	7	HCl + HNO ₃ + HClO ₄	2.66

Notes:

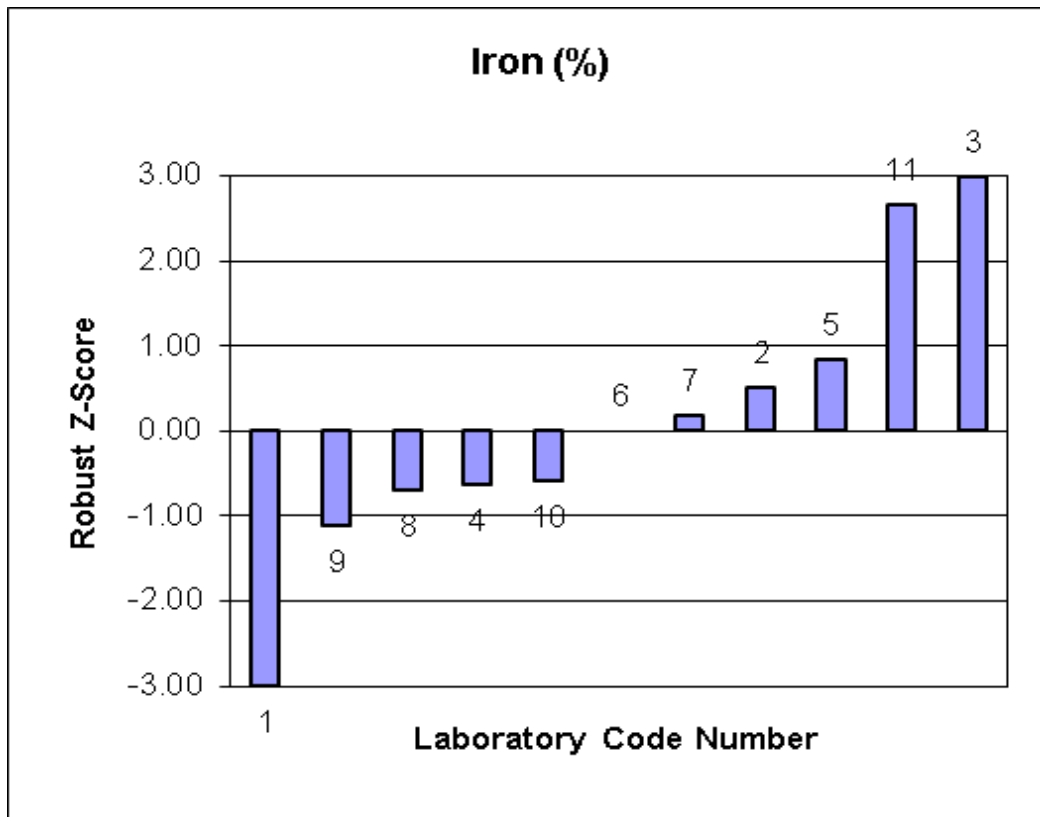
§ denotes an outlier.

indicates no result returned.

P is permissible tolerance between duplicates as calculated from ISO 9516-1:2003 (applicable to Method Code 6 only, i.e. XRF)

Summary Statistics

No. results	11
Median	63.095
Norm IQR	0.135
Robust CV	0.2%
Min	62.45
Max	63.66
Range	1.21
Uncertainty (Median)	0.051



Magnesium (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference P = 0.0204	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.024	0.026	0.025	0.002	0.001	#	3	three acid digest - HCl, HF and HNO ₃	-4.77 §
2	0.036	0.042	0.039	0.006	0.013	0.006	6	Fused disc XRF with meta/tetra-borate flux containing LiNO ₃	-0.08
3	0.038	0.043	0.041	0.005	0.019	0.005	6	Borate Flux	0.42
4	0.045	0.048	0.047	0.003	0.011 at 0.294	0.00004%	1	Microwave Digestion	2.43
5	0.03	0.04	0.035	0.01		0.01	6	Glass Fusion	-1.42
6	0.037	0.036	0.037	0.001	0.01	0.01	6	Li Borate Fused Bead	-0.92
7	0.04	0.04	0.040	0.00	0.010	0.01	6	Fusion Lithium Borate	0.25
8	<0.0001	<0.0001	*	*	0.1	0.1	6	XRF-fusion	*
9	0.039	0.037	0.038	0.002	#	#	1	lithium metaborate / lithium nitrate fusion @ 1000 degrees C	-0.42
10	0.07	0.07	0.070	0.00	#	0.01	#	#	10.30 §
11	0.040	0.039	0.040	0.001	0.003	0.0002	1	HCl + HNO ₃ + HClO ₄	0.08

Notes:

* statistics could not be performed for this result (Laboratory 8).

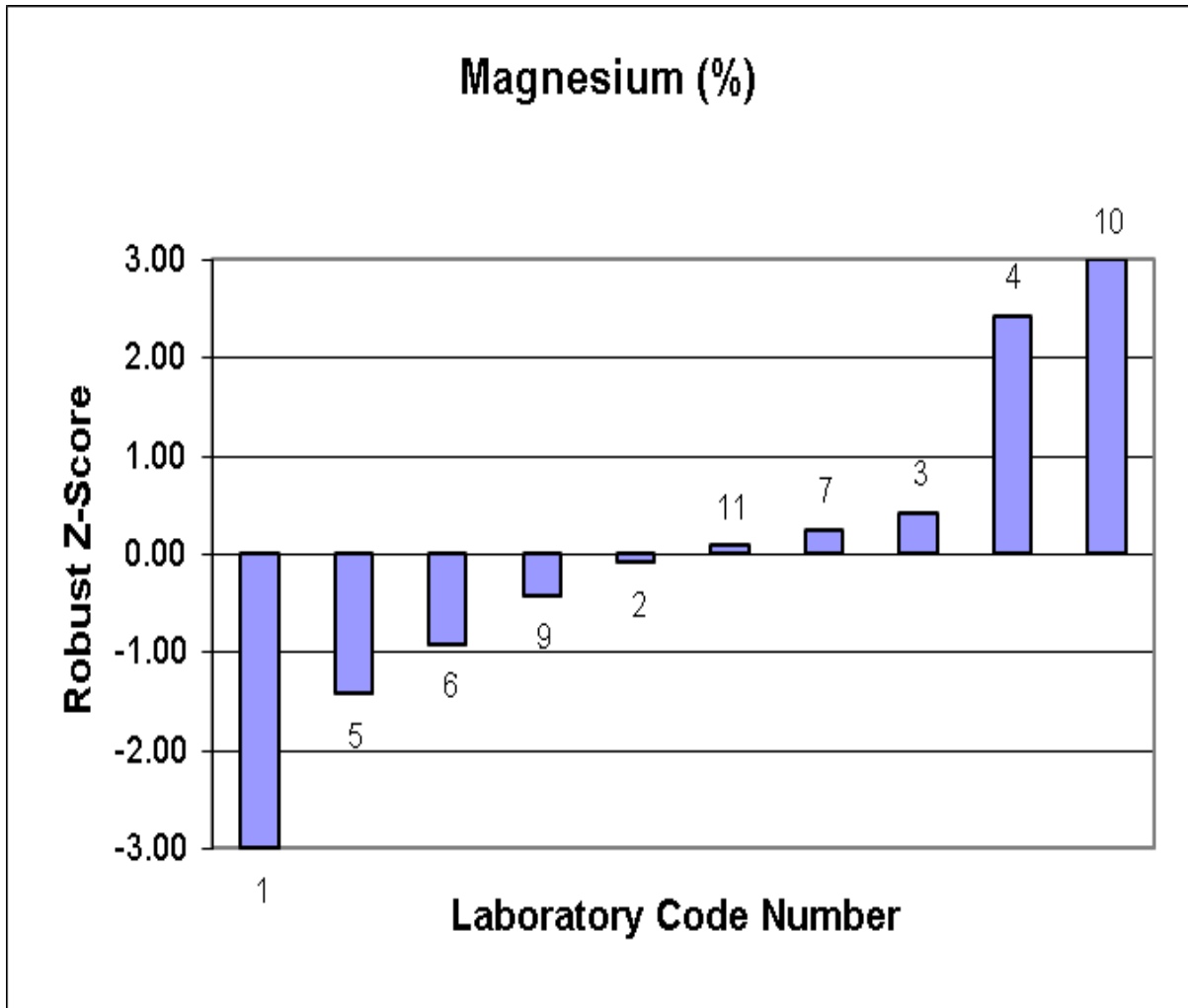
§ denotes an outlier.

indicates no result returned.

P is permissible tolerance between duplicates as calculated from ISO 9516-1:2003 (applicable to Method Code 6 only, i.e. XRF)

Summary Statistics

No. results	10
Median	0.0393
Norm IQR	0.0030
Robust CV	7.6%
Min	0.025
Max	0.070
Range	0.045
Uncertainty (Median)	0.0012



Manganese (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference P = 0.0078	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
2	0.206	0.206	0.206	0.000	0.012	0.001	6	Fused disc XRF with meta/tetra-borate flux containing LiNO ₃	-0.86
3	0.215	0.211	0.213	0.004	0.012	0.002	6	Borate Flux	-0.11
4	0.186	0.192	0.189	0.006	0.0023 at 0.047	0.000006%	1	Microwave Digestion	-2.69
5	0.21	0.21	0.210	0.00	#	0.01	6	Glass Fusion	-0.43
6	0.222	0.216	0.219	0.006	0.01	0.001	6	Li Borate Fused Bead	0.54
7	0.21	0.22	0.215	0.01	0.002	0.01	6	Fusion Lithium Borate	0.11
8	0.21	0.24	0.225	0.003	0.02	0.01	6	XRF-fusion	1.19
9	0.24	0.21	0.225	0.03	#	#	1	Sodium peroxide fusion	1.19
10	0.22	0.22	0.220	0.00	#	0.01	#	#	0.65
11	0.209	0.208	0.209	0.001	0.011	0.0002	1	HCl + HNO ₃ + HClO ₄	-0.59

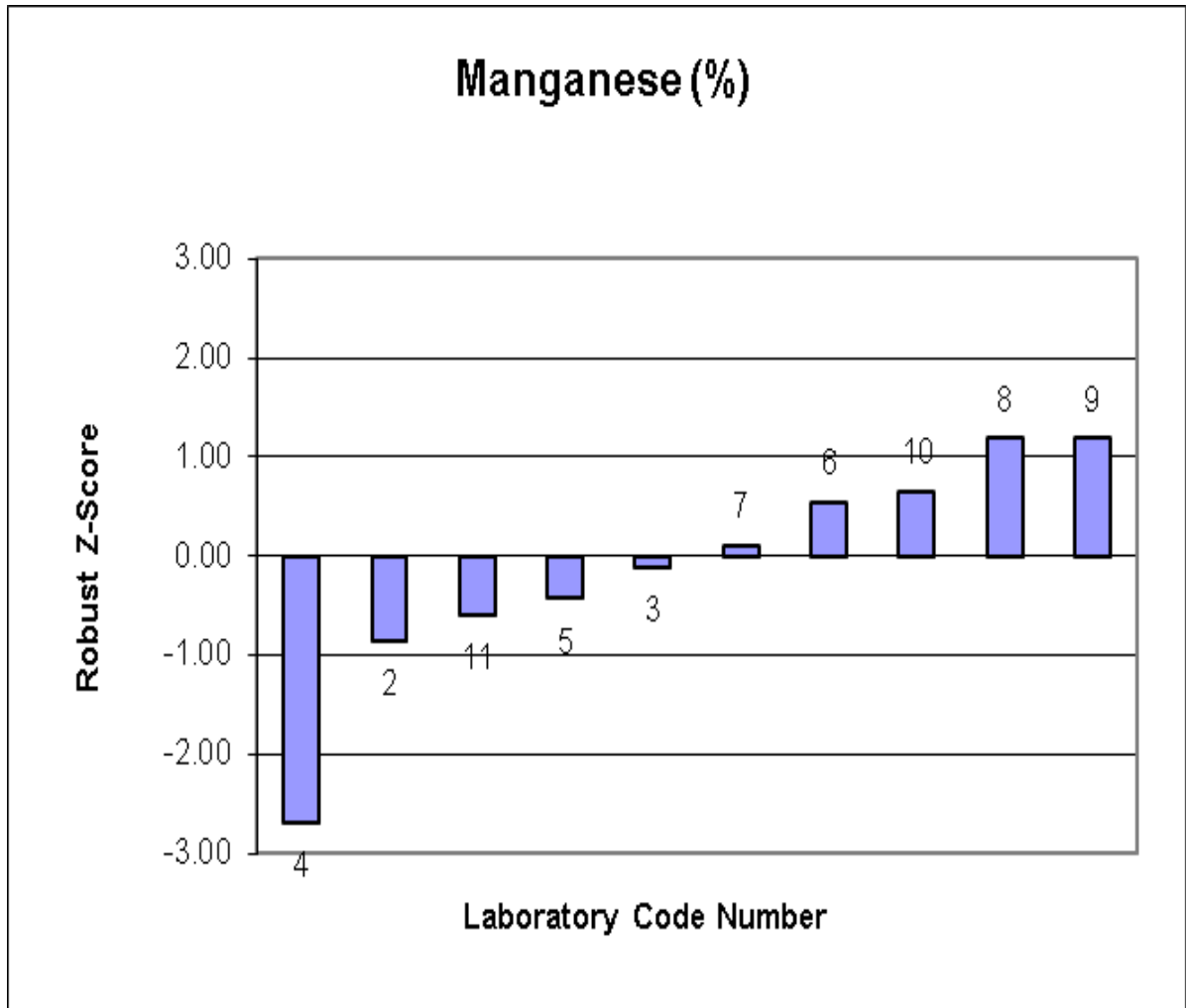
Notes:

indicates no result returned.

P is permissible tolerance between duplicates as calculated from ISO 9516-1:2003 (applicable to Method Code 6 only, i.e. XRF)

Summary Statistics

No. results	10
Median	0.2140
Norm IQR	0.0093
Robust CV	4.3%
Min	0.189
Max	0.225
Range	0.036
Uncertainty (Median)	0.0037



Phosphorus (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference P=0.0026	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
2	0.076	0.073	0.075	0.003	0.006	0.001	6	Fused disc XRF with meta/tetra-borate flux containing LiNO ₃	0.38
3	0.073	0.074	0.074	0.001	0.0061	0.002	6	Borate Flux	0.13
4	0.061	0.066	0.064	0.005	0.0021 at 0.045	0.000008%	1	Microwave Digestion	-2.42
5	0.070	0.071	0.071	0.001	#	0.002	6	Glass Fusion	-0.64
6	0.073	0.075	0.074	0.002	0.003	0.001	6	Li Borate Fused Bead	0.25
7	0.074	0.072	0.073	0.002	0.001	0.001	6	Fusion Lithium Borate	0.00
8	0.04	0.04	0.040	0.00	0.05	0.01	6	XRF-fusion	-8.39 §
10	0.074	0.076	0.075	0.002	#	0.001	#	#	0.51
11	0.069	0.070	0.070	0.001	0.004	0.0002	1	HCl + HNO ₃ + HClO ₄	-0.89

Notes:

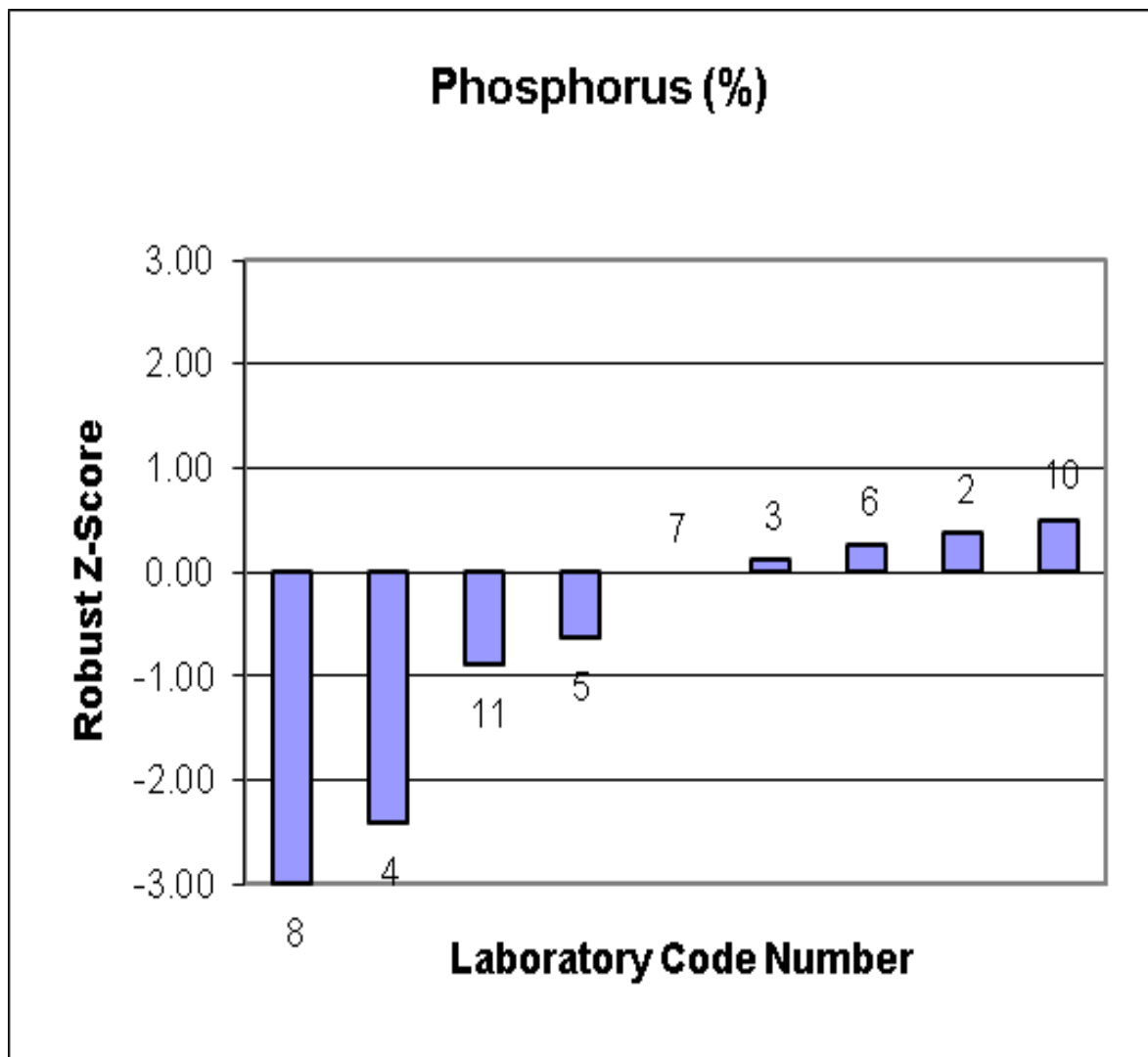
§ denotes an outlier.

indicates no result returned.

P is permissible tolerance between duplicates as calculated from ISO 9516-1:2003 (applicable to Method Code 6 only, i.e. XRF)

Summary Statistics

No. results	9
Median	0.0730
Norm IQR	0.0039
Robust CV	5.4%
Min	0.040
Max	0.075
Range	0.035
Uncertainty (Median)	0.0016



Potassium (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference P=0.0016	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
2	0.0116	0.0116	0.012	0.0000	0.0022	0.0008	6	Fused disc XRF with meta/tetra-borate flux containing LiNO ₃	0.15
3	0.0050	0.0050	0.005	0.0000	0.03	0.005	6	Borate Flux	-3.11 §
4	0.012	0.013	0.013	0.001	0.020 at 0.144	0.00004%	1	Microwave Digestion	0.59
5	0.01	0.01	0.010	0.00		0.002	6	Glass Fusion	-0.64
6	0.011	0.013	0.012	0.002	0.005	0.002	6	Li Borate Fused Bead	0.35
7	0.01	0.01	0.010	0.00	0.002	0.01	6	Fusion Lithium Borate	-0.64
8	0.03	0.03	0.030	0.00	0.05	0.01	6	XRF-fusion	9.23 §
9	0.012	0.010	0.011	0.002	#	#	1	lithium metaborate / lithium nitrate fusion @ 1000 degrees C	-0.15
10	0.01	0.01	0.010	0.00	#	0.01	#	#	-0.64
11	0.027	0.027	0.027	0.000	0.003	0.0038	1	HCl + HNO ₃ + HClO ₄	7.75 §

Notes:

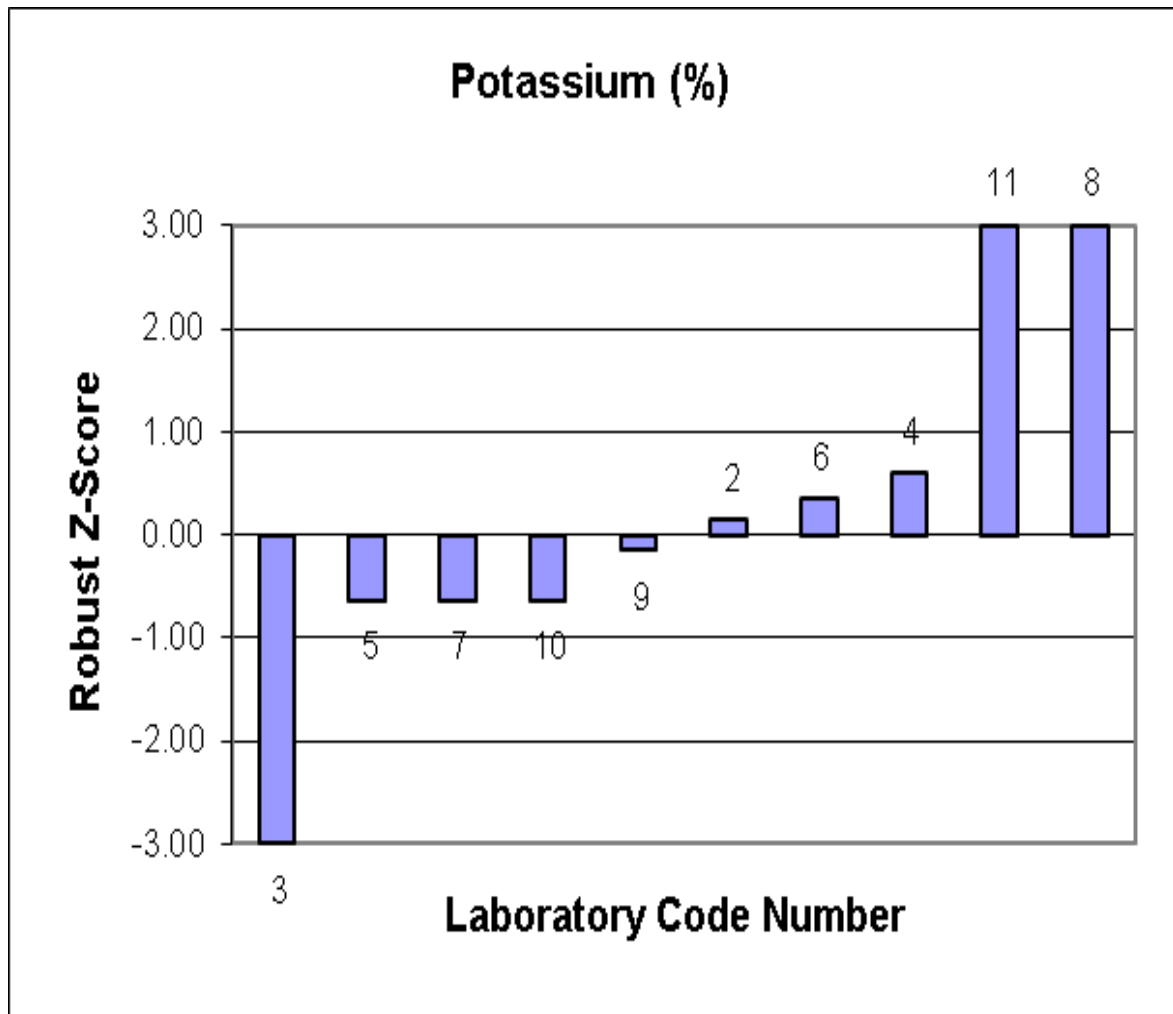
§ denotes an outlier.

indicates no result returned.

P is permissible tolerance between duplicates as calculated from ISO 9516-1:2003 (applicable to Method Code 6 only, i.e. XRF)

Summary Statistics

No. results	10
Median	0.0113
Norm IQR	0.0020
Robust CV	17.9%
Min	0.005
Max	0.030
Range	0.025
Uncertainty (Median)	0.0008



Silicon (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference P=0.0386	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	1.56	1.57	1.57	0.01	0.08	#	6	borate fusion	0.08
2	1.55	1.56	1.56	0.01	0.041	0.005	6	Fused disc XRF with meta/tetra-borate flux containing LiNO ₃	-0.23
3	1.56	1.54	1.55	0.02	0.09	0.005	6	Borate Flux	-0.38
4	1.58	1.61	1.60	0.03	0.180 at 4.31	#	7	Dissolution in HCl Medium	0.98
5	3.23	3.31	3.27	0.08	#	0.01	6	Glass Fusion	51.31 §
6	1.576	1.576	1.58	0.000	0.03	0.01	6	Li Borate Fused Bead	0.41
7	1.54	1.58	1.56	0.04	0.012	0.01	6	Fusion Lithium Borate	-0.08
8	1.52	1.51	1.52	0.01	0.3	0.1	6	XRF-fusion	-1.43
10	1.52	1.57	1.55	0.05	#	0.01	#	#	-0.53
11	1.68	1.75	1.72	0.07	0.08	0.05	11	Gravimetric measurement, HCl + HNO ₃ + HClO ₄	4.58 §

Notes:

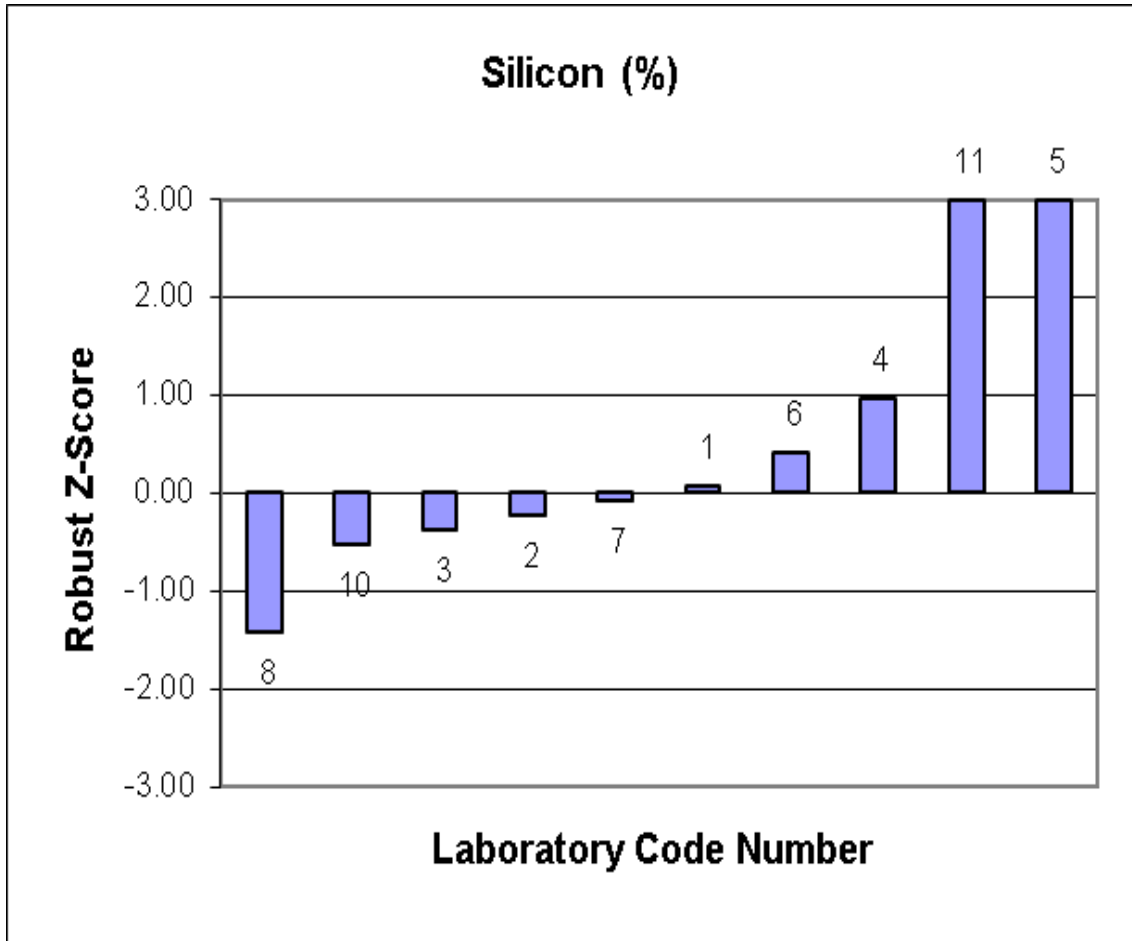
§ denotes an outlier.

indicates no result returned.

P is permissible tolerance between duplicates as calculated from ISO 9516-1:2003 (applicable to Method Code 6 only, i.e. XRF)

Summary Statistics

No. results	10
Median	1.563
Norm IQR	0.033
Robust CV	2.1%
Min	1.52
Max	3.27
Range	1.76
Uncertainty (Median)	0.013



Sodium (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference ^a	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
2	0.023	0.019	0.021	0.004	0.010	0.004	6	Fused disc XRF with meta/tetra-borate flux containing LiNO ₃	0.13
3	0.016	0.013	0.015	0.003	0.03	0.005	6	Borate Flux	-0.74
4	0.017	0.019	0.018	-0.002	0.019 at 0.135	0.00026%	1	Microwave Digestion	-0.27
5	0.049	0.049	0.049	0.000		0.02	6	Glass Fusion	3.90 §
6	0.013	0.009	0.011	0.004	0.02	0.01	6	Li Borate Fused Bead	-1.21
7	0.01	0.01	0.010	0.00	0.008	0.01	6	Fusion Lithium Borate	-1.35
8	0.13	0.16	0.145	0.03	0.05	0.02	6	XRF-fusion	16.82 §
9	0.020	0.026	0.023	0.006	#	#	1	lithium metaborate / lithium nitrate fusion @ 1000 degrees C	0.40
10	<0.01	<0.01	*	*	#	0.01	#	#	*
11	0.019	0.021	0.020	0.002	0.003	0.0015	1	HCl + HNO ₃ + HClO ₄	0.00

Notes:

* statistics could not be performed for this result (Laboratory 10).

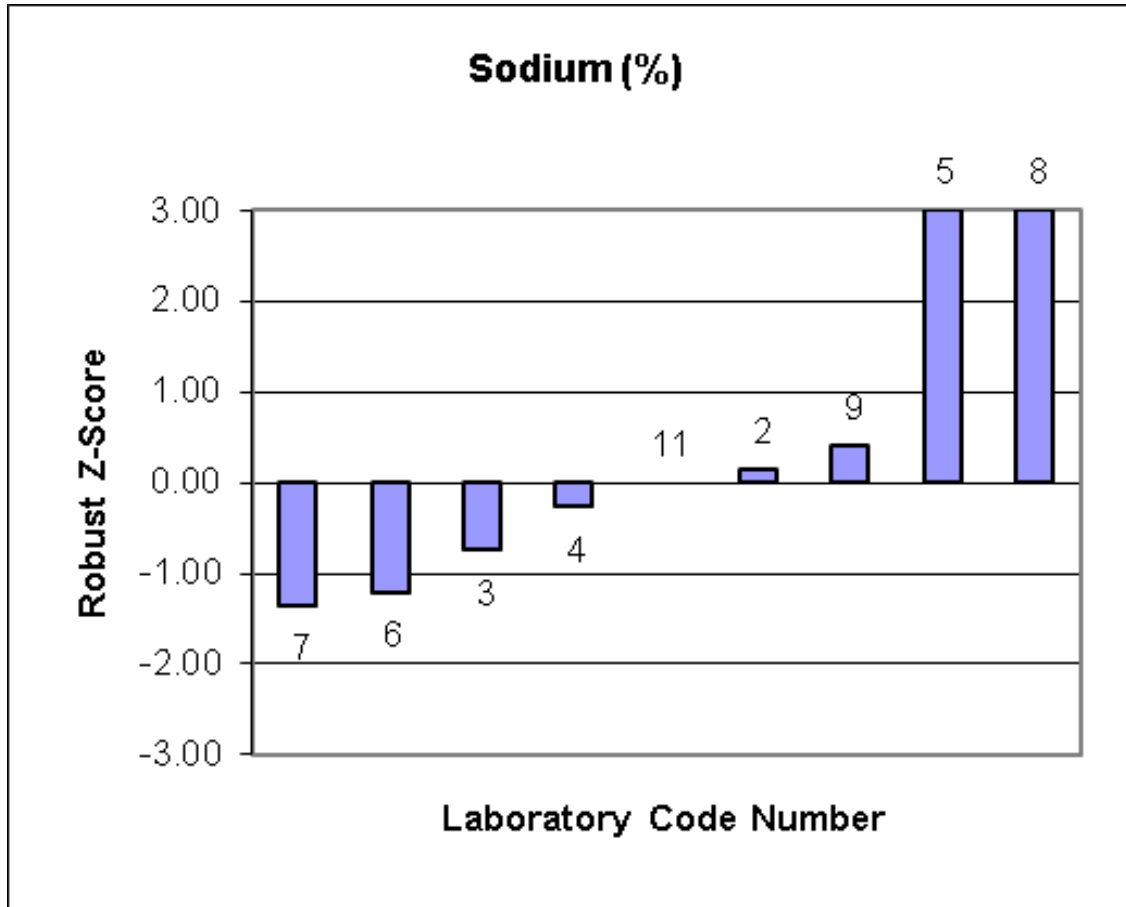
§ denotes an outlier.

indicates no result returned.

^aThere is no guide for permissible tolerance for Sodium stated in ISO 9516-1:2003

Summary Statistics

No. results	9
Median	0.0200
Norm IQR	0.0074
Robust CV	37.2%
Min	0.010
Max	0.145
Range	0.135
Uncertainty (Median)	0.0031



Sulphur (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference P=0.0035	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.032	0.032	0.032	0.000	0.001	#	6	borate fusion	2.68
2	0.02	0.02	0.020	0.00	0.003	0.001	6	Fused disc XRF with meta/tetra-borate flux containing LiNO ₃	-0.54
3	0.020	0.022	0.021	0.002	0.0240	0.002	6	Borate Flux	-0.27
4	0.015	0.016	0.016	0.001	0.0026 at 0.011	0.0004%	10	Combustion / IR Absorption	-1.74
5	0.021	0.020	0.021	0.001	#	0.002	6	Glass Fusion	-0.40
6	0.020	0.020	0.020	0.000	0.002	0.001	6	Li Borate Fused Bead	-0.54
7	0.023	0.023	0.023	0.000	0.002	0.001	6	Fusion Lithium Borate	0.27
8	0.08	0.06	0.070	0.02	0.2	0.1	6	XRF-fusion	12.86 §
9	<0.1	<0.1	*	*	#	#	1	Sodium peroxide fusion	*
10	0.024	0.022	0.023	0.002	#	0.001	#	#	0.27
11	0.026	0.024	0.025	0.002	0.003	0.002	10	#	0.80

Notes:

* statistics could not be performed for this result (Laboratory 9).

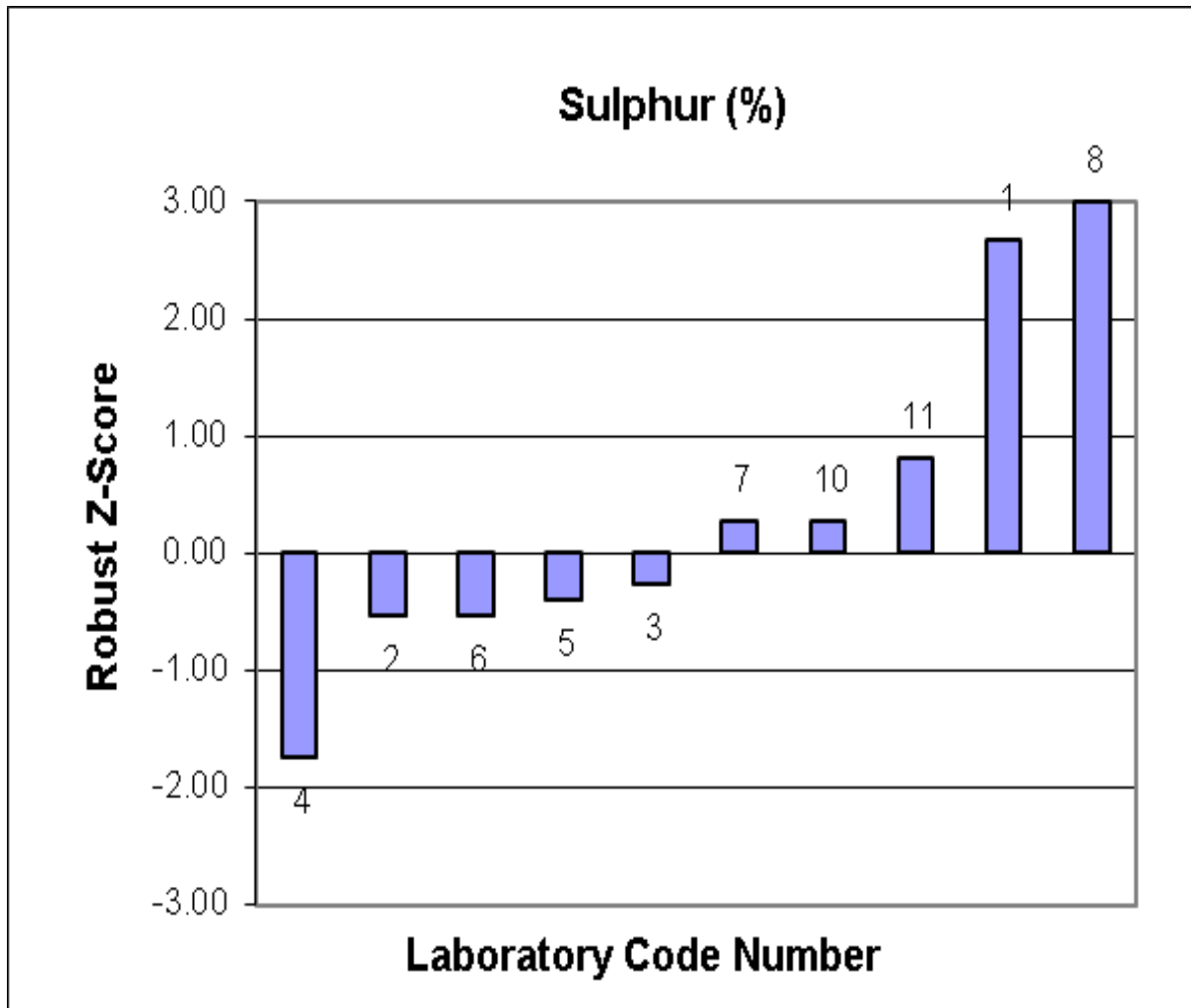
§ denotes an outlier.

indicates no result returned.

P is permissible tolerance between duplicates as calculated from ISO 9516-1:2003 (applicable to Method Code 6 only, i.e. XRF)

Summary Statistics

No. results	10
Median	0.0220
Norm IQR	0.0037
Robust CV	17.0%
Min	0.016
Max	0.070
Range	0.055
Uncertainty (Median)	0.0015



Titanium (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference P=0.0043	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
2	0.06	0.06	0.060	0.00	0.015	0.006	6	Fused disc XRF with meta/tetra-borate flux containing LiNO ₃	0.00
3	0.051	0.051	0.051	0.000	0.003	0.005	6	Borate Flux	-0.94
4	0.046	0.047	0.047	0.001	0.004 at 0.199	0.000007 %	1	Microwave Digestion	-1.40
5	0.109	0.110	0.110	0.001	#	0.005	6	Glass Fusion	5.15 §
6	0.067	0.062	0.065	0.005	0.005	0.005	6	Li Borate Fused Bead	0.47
7	0.06	0.06	0.060	0.00	0.002	0.01	6	Fusion Lithium Borate	0.00
8	0.06	0.06	0.060	0.00	0.05	0.01	6	XRF-fusion	0.00
10	0.062	0.062	0.062	0.000	#	0.001	#	#	0.21
11	0.042	0.044	0.043	0.002	0.003	0.0002	1	HCl + HNO ₃ + HClO ₄	-1.77

Notes:

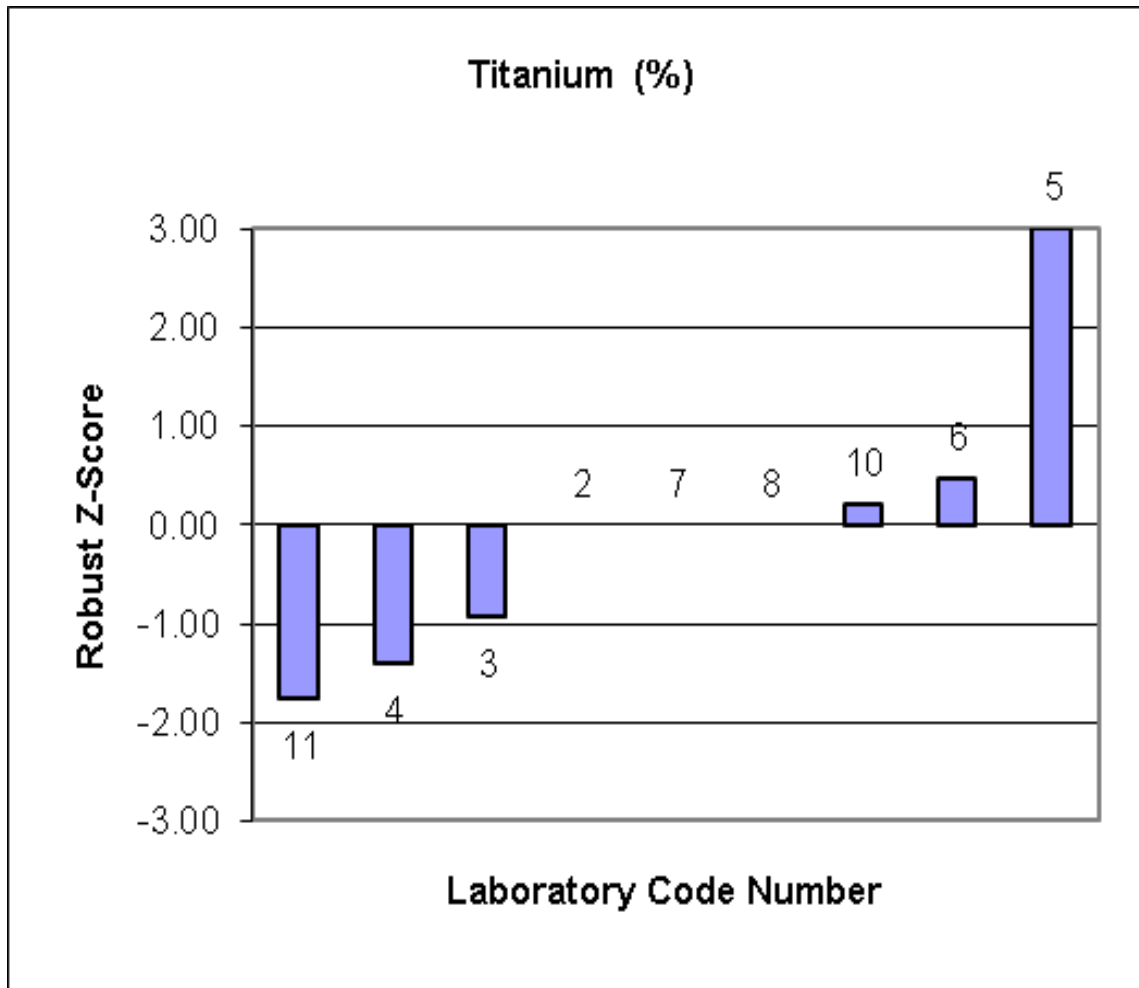
§ denotes an outlier.

indicates no result returned.

P is permissible tolerance between duplicates as calculated from ISO 9516-1:2003 (applicable to Method Code 6 only, i.e. XRF)

Summary Statistics

No. results	9
Median	0.0600
Norm IQR	0.0096
Robust CV	16.0%
Min	0.043
Max	0.110
Range	0.067
Uncertainty (Median)	0.0040



Loss on Ignition (%)

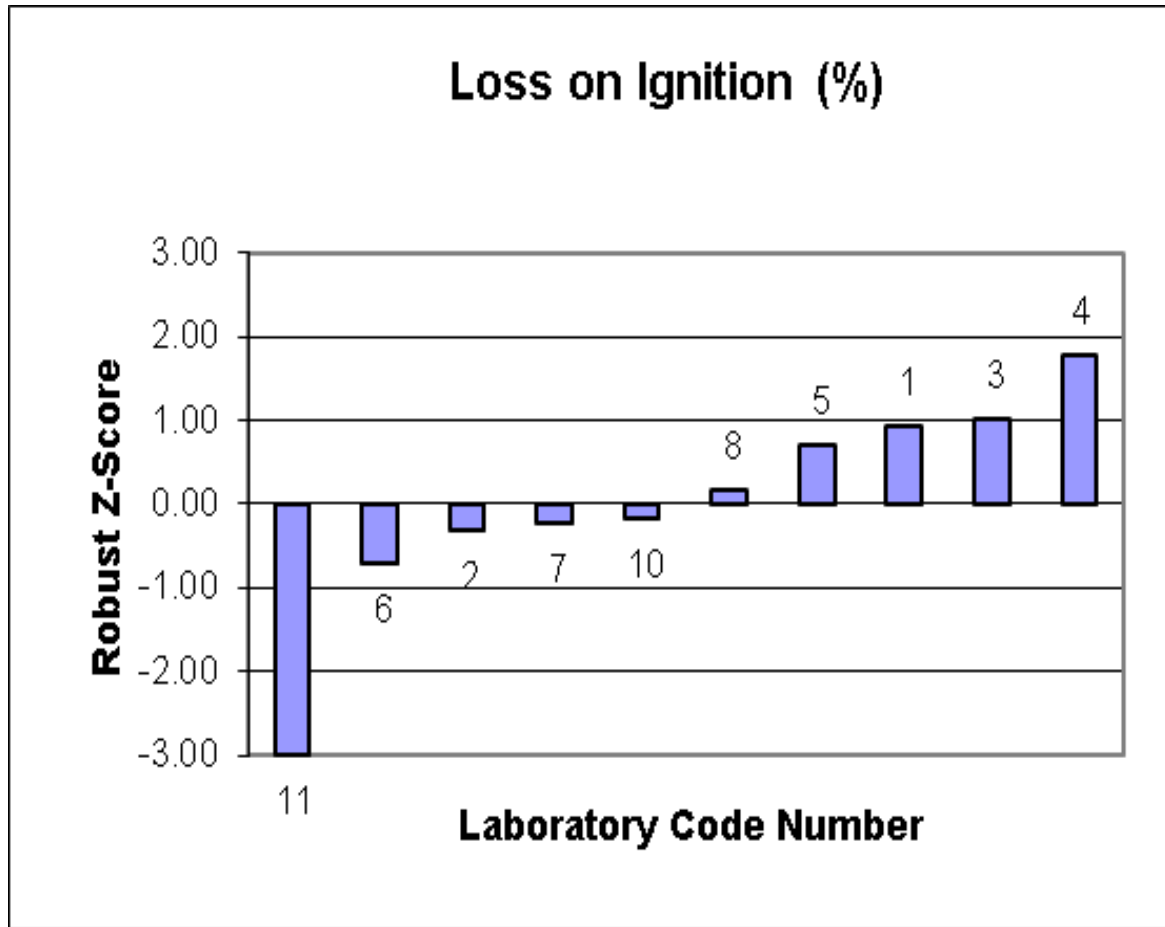
Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	3.57	3.58	3.58	0.01	0.18	#	gravimetric	N/A	0.93
2	3.33	3.41	3.37	0.08	0.12	0.01	10	Gravimetrically determined automatically using a LECO TGA	-0.32
3	3.58	3.60	3.59	0.02	0.02	0.005	7	NA	1.02
4	3.68	3.75	3.72	0.07	0.283 at 5.57	#	11 / Loss in Weight at 950 Degree Celsius	#	1.78
5	3.54	3.54	3.54	0.00	#	#	11 Gravimetric	#	0.72
6	3.32	3.29	3.31	0.03	0.07	0.01	6	Li Borate Fused Bead	-0.72
7	3.37	3.40	3.39	0.03	0.037	0.01	10	Thermo Gravimetric Analysis	-0.23
8	3.45	3.45	3.45	0.00	0.2	0.2	6	XRF-fusion	0.17
10	3.36	3.43	3.40	0.07	#	0.01A	#	#	-0.17
11	2.89	2.95	2.92	0.06	0.14	0.10	11	Gravimetric measurement	-3.06 §

§ denotes an outlier.

indicates no result returned.

Summary Statistics

No. results	10
Median	3.423
Norm IQR	0.164
Robust CV	4.8%
Min	2.92
Max	3.72
Range	0.80
Uncertainty (Median)	0.065



APPENDIX B

Sample Homogeneity

Homogeneity Testing B1

Homogeneity Testing

Previously, ten samples were selected randomly for homogeneity testing by Becquerel Laboratories. Due to the nature of the samples they are considered to remain homogenous provided they were well shaken before analysis (as outlined in the instructions).

Statistical analysis (ANOVA) showed that the samples were sufficiently homogenous so that any results later identified as outliers should not be attributed to any notable sample variability.

Numerous elements were analysed, however only those elements applicable to this round have been reported here.

Sample No.	Iron		Sodium	
	Result 1	Result 2	Result 1	Result 2
1	60.90	61.80	0.012	0.012
2	61.10	61.80	0.012	0.012
3	60.90	62.60	0.011	0.012
4	61.30	61.50	0.012	0.012
5	61.30	62.30	0.012	0.013
6	61.60	60.60	0.011	0.011
7	61.90	62.00	0.012	0.012
8	62.20	61.60	0.014	0.011
9	60.50	63.20	0.011	0.012
10	62.60	61.00	0.013	0.012

APPENDIX C

Documentation

Instructions to Participants C1

Results Sheet C4

PROFICIENCY TESTING AUSTRALIA

Proficiency Testing Program Geochemical Testing – Round 8

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 50g in a glass bottle has been provided for each laboratory.
Please shake sample prior to analysis.

2. Testing

- The testing should commence as soon as possible after receipt of the sample.
- Report total analysis of major and trace elements listed below on a 'dry' basis by the technique that gives the best detection limit for each element.

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

- Test and report Loss on Ignition result.

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.
- In addition to reporting the results, specify the dissolution/digestion technique used. Also 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 rather than oxide basis.
- Please report each element to the units (%) indicated on the Results Sheet along with your laboratory's detection limit for that analysis.
- Laboratories are also 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 report in the same units as the results for each element.**
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 11 JANUARY 2013** to:

Kathy Weller

Proficiency Testing Australia

PO Box 1122

Archerfield BC QLD 4108

phone: +61 7 3721 7373

fax: +61 7 3217 1844

email: 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

Method Technique	Method Code
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

PROFICIENCY TESTING AUSTRALIA
Geochemical – Round 8 - Proficiency Testing Program
Results Sheet

Lab Code:

«Cod
»

Analysis	Result 1	Result 2	Units	Detection Limit	Dissolution/ Digestion Technique (Please Specify)	Method Code	±MU*
Aluminium			%				
Calcium			%				
Iron			%				
Magnesium			%				
Manganese			%				
Phosphorus			%				
Potassium			%				
Silicon			%				
Sodium			%				
Sulphur			%				
Titanium			%				
Loss on Ignition			%				

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

Return no later than **FRIDAY 11 JANUARY 2013**, to:

Kathy Weller, Proficiency Testing Australia.

PO Box 1122, Archerfield BC QLD 4108. Australia.

phone: +61 7 3721 7373, fax: +61 7 3217 1844, email: Kathy.Weller@pta.asn.au

- End of Report -