

# GEOCHEMICAL PROFICIENCY TESTING PROGRAM

# **ROUND 13**

# **FEBRUARY 2018**

# **REPORT NO. 1066**

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 2017 / January 2018 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 F Watton, PTA Quality Manager. 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 Syenite 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	Phosphorous	Ρ	Titanium	Ti
Iron	Fe	Potassium	Κ	Zirconium	Zr
Magnesium	Mg	Silicon	Si		

2.4 A total of 7 laboratories participated in the program with all laboratories returning results for inclusion in the final report.

Participant laboratories were from Australia and Korea.

- 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 Previously, the bulk material was analysed and tested for homogeneity. Based on the results of this testing it was considered that the samples utilised for this program were homogeneous. As such, any results later identified as outliers 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, 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 <u>a z-score with an absolute value greater than or equal to 3.0 is considered to be an outlier</u> 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 <u>number</u> of results for that test/sample (No. of Results);
- the median of these results, i.e. the middle value (Median);
- the <u>uncertainty of the median</u>; a robust estimate of the standard deviation of the *Median*;
- the normalised interguartile range of the results (Normalised IQR);
- the <u>robust coefficient of variation</u>, expressed as a percentage (*Robust CV*) i.e. 100 x *Normalised IQR / 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.

Analysis	Number of Results		Med	Median (%)		ised IQR	Uncertainty (Median) (%)	
Analysis	Round 7	Round 13	Round 7	Round 13	Round 7	<u>Round 13</u>	Round 7	Round 13
Aluminium	10	7	7.848	7.985	0.135	0.120	0.054	0.057
Calcium	10	7	1.978	2.005	0.097	0.052	0.039	0.025
Iron	10	7	7.008	7.065	0.198	0.092	0.079	0.044
Magnesium	10	7	0.1550	0.1470	0.0289	0.0144	0.0115	0.0068
Manganese	9	6	0.1860	0.1910	0.1174	0.0029	0.0031	0.0015
Phosphorous	9	6	0.0830	0.0838	0.0033	0.0022	0.0014	0.0002
Potassium	8	6	4.275	4.284	0.053	0.087	0.023	0.045
Silicon	10	7	26.238	26.390	0.383	0.417	0.152	0.197
Sodium	8	6	3.853	3.885	0.111	0.107	0.049	0.014
Titanium	9	6	0.3100	0.3093	0.0078	0.0095	0.0033	0.0049
Zirconium	7	4	0.0450	NC	0.0169	NC	0.0080	NC

### TABLE A: SUMMARY STATISTICS FOR ROUND 13, INCLUDING A COMPARISON WITH ROUND 7 SUMMARY STATISTICS

### Notes:

- 1. NC Not Calculated.
- 2. Summary Statistics from the Geochemical Round 7 (2011) program are included in the above table as the same sample was used in both Rounds 7 and 13.
- 3. Statistical analysis has not been performed for Zirconium due to the small number of results returned.
- 4. A target CV (1.5%) was used to calculate the z-scores for Aluminium.

### 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 13.

The robust CV for Aluminium was low. This resulted in some laboratories unfairly receiving outliers. On examination of these results, it was decided that not all of the results should be considered outliers and a target CV of 1.5% was considered more appropriate, therefore, z-scores were determined using this value.

Using the normalised interquartile range (NIQR) as a measure of the spread of the data, the comparison between round 7 (which used the same sample in 2011) and round 13 shows a minimal difference between the two programs. Most of the elements returned tighter NIQRs. Round 13 had a smaller data set with 7 laboratories taking part compared to 10 in round 7.

The robust CVs for all elements except Magnesium show satisfactory performance. Magnesium is affected by laboratory 6 whose value is a lot lower than all the other results. Laboratory 6 may need to review both the XRF fusion process and instrument calibration for the cause of the low outliers in Magnesium, Iron and Silicon.

Generally, all values (excluding outliers) agree well with the expected values.

Lab Code	Results Reported	No. of Outliers
1	11	0
2	10	0
3	11	0
4	11	0
5	10	0
6	5	3
7	11	1

### 4.2 Outliers

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 (MU) for the testing performed. It is advisable that laboratories review their processes or calculations for suitability if MU is greater than 5% of the result.

MU reported by laboratories varied significantly for some of the elements despite the same techniques being employed. This might be due to the method used to calculate

the uncertainty as this was not requested to be done the same way for all the laboratories.

#### 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 investigate and 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.

#### 4.6 Technique Bias

Most laboratories in this round used a lithium borate fusion with an XRF finish. One laboratory employed an acid digest and the results were comparable to the fusion indicating that the digest was near total. Laboratories with outliers are encouraged to investigate the source of error.

#### 4.7 Method Code

All laboratories except for one provided method codes.

#### 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 samples chosen for this program were prepared and supplied by the CSIRO Division of Exploration and Mining laboratories at North Ryde, NSW.

As the assigned value for each 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:

Element	Lab Code	Bias	Method Code	Dissolution/Digestion Technique				
Aluminium		No outliers reported						
Calcium		No outliers reported						
Iron	6	-ve	6 - XRF	Fusion				
Magnesium		-ve	6 - XRF	Fusion				
Manganese	No outliers reported							
Phosphorous	No outliers reported							
Potassium			No outliers reporte	ed				
Silicon	Silicon 6		6 - XRF	Fusion				
Sodium 7		-ve	7 - Wet Chemical	Aqua Regia / HF				
Titanium	No outliers reported							

### TABLE B: OUTLIER RESULTS

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

### 6. Reference

[1] *Guide to Proficiency Testing Australia,* 2016. (This document can be found on the PTA website, <u>www.pta.asn.au</u>)

# **APPENDIX A**

# **Results and Data Analysis**

Aluminium	A1.1
Calcium	A2.1
Iron	A3.1
Magnesium	A4.1
Manganese	A5.1
Phosphorous	A6.1
Potassium	A7.1
Silicon	A8.1
Sodium	A9.1
Titanium	A10.1
Zirconium	A11.1

#### A1.1

## Aluminium (AI) (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	8.00	7.97	7.99	0.03	0.09	0.01	6	Borate Fused Bead	0.00
2	7.99	7.98	7.99	0.01	0.08	0.01	6	#	0.00
3	7.97	7.96	7.97	0.01	0.0090	0.053	6	Fusion - Borate flux	-0.17
4	7.99	7.99	7.99	0.00	0.005	0.01	6	LiT/LiM flux fused bead	0.04
5	8.02	8.01	8.02	0.01	#	0.01	6	NA	0.25
6	7.94	7.94	7.94	0.00	±0.07	0.005	6	Fusion	-0.37
7	7.84	7.87	7.86	0.03	0.07	#	1	Aqua regia / HF	-1.08

#### Notes:

# indicates no result returned.

### Summary Statistics

No. results	7
Median	7.985
Norm IQR	0.120
Robust CV	1.5%
Min	7.86
Max	8.02
Range	0.16
Uncertainty (Median)	0.057

Note:

The robust CV achieved for this test was low (robust CV = 0.4%) and using this value would have resulted in some laboratories unfairly receiving outliers. In this case, a target robust CV = 1.5% was considered more appropriate and was used to determine z-scores.

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



Aluminium (AI) Measurement Uncertainty by Laboratory



Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	2.01	2.00	2.01	0.01	0.04	0.01	6	Borate Fused Bead	0.00
2	2.02	2.02	2.02	0.00	0.014	0.01	6	#	0.29
3	1.98	1.98	1.98	0.00	0.0093	0.0071	6	Fusion - Borate flux	-0.48
4	2.10	2.11	2.11	0.01	0.01	0.01	6	LiT/LiM flux fused bead	1.93
5	2.07	2.06	2.07	0.01	#	0.01	6	NA	1.16
6	1.99	1.99	1.99	0.00	±0.05	0.005	6	Fusion	-0.29
7	1.98	1.98	1.98	0.00	0.03	#	1	Aqua regia / HF	-0.48

# Calcium (Ca) (%)

### Notes:

# indicates no result returned.

No. results	7
Median	2.005
Norm IQR	0.052
Robust CV	2.6%
Min	1.98
Max	2.11
Range	0.13
Uncertainty (Median)	0.025

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



Calcium (Ca) Measurement Uncertainty by Laboratory



Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	7.04	7.00	7.02	0.04	0.08	0.01	6	Borate Fused Bead	-0.49
2	7.05	7.08	7.07	0.03	0.25	0.01	6	#	0.00
3	7.01	6.99	7.00	0.03	0.012	0.0070	6	Fusion - Borate flux	-0.70
4	7.09	7.15	7.12	0.06	0.08	0.01	6	LiT/LiM flux fused bead	0.60
5	7.25	7.24	7.25	0.01	#	0.01	6	NA	1.95
6	6.61	6.63	6.62	0.02	±0.29	0.01	6	Fusion	-4.82 <b>§</b>
7	7.11	7.10	7.11	0.01	0.05	#	7	HCI / $H_2SO_4$ SnCl <sub>2</sub> reduction	0.43

# Iron (Fe) (%)

Notes:

§ denotes an outlier, i.e. |z-score $| \ge 3.0$ .

# indicates no result returned.

No. results	7
Median	7.065
Norm IQR	0.092
Robust CV	1.3%
Min	6.62
Max	7.25
Range	0.63
Uncertainty (Median)	0.044

A3.2



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

Iron (Fe) Measurement Uncertainty by Laboratory



Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.144	0.143	0.144	0.001	0.02	0.01	6	Borate Fused Bead	-0.24
2	0.15	0.15	0.150	0.00	0.015	0.01	6	#	0.21
3	0.150	0.144	0.147	0.006	0.013	0.0060	6	Fusion - Borate flux	0.00
4	0.17	0.17	0.170	0.00	0.005	0.01	6	LiT/LiM flux fused bead	1.60
5	0.18	0.17	0.175	0.01	#	0.01	6	NA	1.94
6	0.086	0.074	0.080	0.012	±0.04	0.02	6	Fusion	-4.65 <b>§</b>
7	0.145	0.144	0.145	0.001	0.002	#	1	Aqua regia / HF	-0.17

# Magnesium (Mg) (%)

### Notes:

§ denotes an outlier, i.e.  $|z-score| \ge 3.0$ .

# indicates no result returned.

No. results	7
Median	0.1470
Norm IQR	0.0144
Robust CV	9.8%
Min	0.080
Max	0.175
Range	0.095
Uncertainty (Median)	0.0068

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



Magnesium (Mg) Measurement Uncertainty by Laboratory



Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.190	0.189	0.190	0.001	0.02	0.01	6	Borate Fused Bead	-0.51
2	0.19	0.19	0.190	0.00	0.015	0.01	6	#	-0.34
3	0.193	0.194	0.194	0.001	0.0062	0.0077	6	Fusion - Borate flux	0.85
4	0.19	0.19	0.190	0.00	0.005	0.01	6	LiT/LiM flux fused bead	-0.34
5	0.19	0.20	0.195	0.01	#	0.01	6	NA	1.36
7	0.192	0.192	0.192	0.000	0.003	#	1	Aqua regia / HF	0.34

# Notes:

# indicates no result returned.

## Summary Statistics

No. results	6
Median	0.1910
Norm IQR	0.0029
Robust CV	1.5%
Min	0.1895
Max	0.1950
Range	0.0055
Uncertainty (Median)	0.0015

# Manganese (Mn) (%)

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



Manganese (Mn) Measurement Uncertainty by Laboratory



A6.	1
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# Phosphorous (P) (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.078	0.079	0.079	0.001	0.02	0.005	6	Borate Fused Bead	-2.35
2	0.084	0.083	0.084	0.001	0.002	0.001	6	#	-0.11
3	0.083	0.085	0.084	0.002	0.0035	0.00044	6	Fusion - Borate flux	0.11
4	0.085	0.085	0.085	0.000	0.0005	0.001	6	LiT/LiM flux fused bead	0.56
5	0.09	0.08	0.085	0.01	#	0.01	6	NA	0.56
7	0.082	0.082	0.082	0.000	0.005	#	1	Aqua regia / HF	-0.78

# Notes:

# indicates no result returned.

No. results	6
Median	0.0838
Norm IQR	0.0022
Robust CV	2.7%
Min	0.079
Max	0.085
Range	0.007
Uncertainty (Median)	0.0002

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



Phosphorus (P) Measurement Uncertainty by Laboratory



## A7.1

# Potassium (K) (%)

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	4.34	4.28	4.31	0.06	0.06	0.01	6	Borate Fused Bead	0.30
2	4.277	4.279	4.28	0.002	0.004	0.002	6	#	-0.07
3	4.32	4.31	4.32	0.01	0.0083	0.0083	6	Fusion - Borate flux	0.35
4	4.13	4.17	4.15	0.04	0.055	0.01	6	LiT/LiM flux fused bead	-1.53
5	4.29	4.29	4.29	0.00	#	0.01	6	NA	0.07
7	4.20	4.18	4.19	0.02	0.08	#	1	Aqua regia / HF	-1.08

### Notes:

# indicates no result returned.

No. results	6
Median	4.284
Norm IQR	0.087
Robust CV	2.0%
Min	4.15
Max	4.32
Range	0.16
Uncertainty (Median)	0.045

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



Potassium (K) Measurement Uncertainty by Laboratory



### A8.1

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	26.57	26.49	26.53	0.08	0.25	0.01	6	Borate Fused Bead	0.34
2	26.40	26.38	26.39	0.02	0.13	0.01	6	#	0.00
3	26.56	26.51	26.54	0.05	0.0051	0.047	6	Fusion - Borate flux	0.35
4	26.42	26.40	26.41	0.02	0.03	0.01	6	LiT/LiM flux fused bead	0.05
5	26.08	26.06	26.07	0.02	#	0.01	6	NA	-0.77
6	24.89	24.87	24.88	0.02	±0.28	0.01	6	Fusion	-3.62 <b>§</b>
7	25.93	25.96	25.95	0.03	0.15	#	7	Alkali Fusion	-1.07

# Silicon (Si) (%)

## Notes:

§ denotes an outlier, i.e.  $|z-score| \ge 3.0$ .

# indicates no result returned.

No. results	7
Median	26.390
Norm IQR	0.417
Robust CV	1.6%
Min	24.88
Max	26.54
Range	1.66
Uncertainty (Median)	0.197

A8.2

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



Silicon (Si) Measurement Uncertainty by Laboratory



### A9.1

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	3.87	3.85	3.86	0.02	0.05	0.01	6	Borate Fused Bead	-0.23
2	3.917	3.903	3.91	0.014	0.050	0.02	6	#	0.23
3	3.97	3.98	3.98	0.01	0.0082	0.0074	6	Fusion - Borate flux	0.84
4	3.83	3.85	3.84	0.02	0.03	0.01	6	LiT/LiM flux fused bead	-0.42
5	4.02	4.02	4.02	0.00	#	0.01	6	NA	1.26
7	3.55	3.54	3.55	0.01	0.09	#	2	Aqua regia / HF	-3.18 <b>§</b>

# Sodium (Na) (%)

Notes:

§ denotes an outlier, i.e.  $|z-score| \ge 3.0$ .

# indicates no result returned.

No. results	6
Median	3.885
Norm IQR	0.107
Robust CV	2.8%
Min	3.55
Max	4.02
Range	0.48
Uncertainty (Median)	0.014

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



Sodium (Na) Measurement Uncertainty by Laboratory



A10.1

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique	Robust z-score
1	0.302	0.308	0.305	0.006	0.02	0.01	6	Borate Fused Bead	-0.45
2	0.309	0.308	0.309	0.001	0.010	0.002	6	#	-0.08
3	0.318	0.318	0.318	0.000	0.0030	0.0060	6	Fusion - Borate flux	0.92
4	0.31	0.31	0.310	0.00	0.005	0.01	6	LiT/LiM flux fused bead	0.08
5	0.32	0.32	0.320	0.00	#	0.01	6	NA	1.13
7	0.303	0.299	0.301	0.004	0.002	#	1	Aqua regia / HF	-0.87

# Titanium (Ti) (%)

# Notes:

# indicates no result returned.

No. results	6
Median	0.3093
Norm IQR	0.0095
Robust CV	3.1%
Min	0.301
Max	0.320
Range	0.019
Uncertainty (Median)	0.0049

A10.2

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



Titanium (Ti) Measurement Uncertainty by Laboratory



### A11.1

Lab Code	Result 1 (%)	Result 2 (%)	Average (%)	Absolute Difference	MU	Detection Limit	Method Code	Dissolution/Digestion Technique
1	0.053	0.048	0.051	0.005	0.02	0.01	6	Borate Fused Bead
3	0.056	0.055	0.056	0.001	0.0067	0.0074	6	Fusion - Borate flux
4	0.050	0.050	0.050	0.000	0.0005	0.001	6	LiT/LiM flux fused bead
7	0.049	0.049	0.049	0.000	0.001	#	1	Aqua regia / HF

## Zirconium (Zr) (%)

Statistical analysis has not been performed as only a small number of numerical results (4) were returned.

### Notes:

# indicates no result returned.



Zirconium (Zr) Measurement Uncertainty by Laboratory

# **APPENDIX B**

# **Sample Homogeneity**

Homogeneity Testing

B1

### Homogeneity Testing

Previously, ten samples were selected randomly for homogeneity testing. 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 (i.e.  $F < F_{crit}$  and P > 0.05) so that any results later identified as outliers could 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	Irc	on	Pota	ssium	Sodium		
No.	Result 1	Result 2	Result 1	Result 2	Result 1	Result 2	
1	6.91	6.84	3.80	3.99	6.91	6.84	
2	6.89	6.90	3.59	4.11	6.89	6.90	
3	6.94	6.81	4.48	4.38	6.94	6.81	
4	6.87	6.93	3.87 4.11		6.87	6.93	
5	6.92	6.99	4.25	3.94	6.92	6.99	
6	6.89	6.96	4.30	4.30 4.25 6.89		6.96	
7	6.94	6.88	3.78	4.34	6.94	6.88	
8	7.13	6.91	3.69	3.90	7.13	6.91	
9	6.90	7.03	4.59	2.93	6.90	7.03	
10	7.09	6.90	5.39	3.70	7.09	6.90	
F <sub>crit</sub>		3.020		3.020		3.020	
F		0.721		0.452	_	0.481	
Р		0.683		0.876		0.857	

# **APPENDIX C**

# **Documentation**

Instructions to Participants

**Results Sheet** 

C1

C4



### PROFICIENCY TESTING AUSTRALIA

### **Proficiency Testing Program**

### **Geochemical Testing – Round 13**

### 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 **syenite** sample with approximately 50g in a glass bottle 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	Manganese		Sodium	Na
Calcium	Ca	Phosphorous	Р	Titanium	Ti
Iron	Fe	Potassium	K	Zirconium	Zr
Magnesium	Mg	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 (%) 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 ≈ 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 5 JANUARY 2018 to:

Kathy Weller <u>fax</u>: +61 7 3217 1844 <u>email</u>: 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

Please use a Method Code for each element tested.



### PROFICIENCY TESTING AUSTRALIA

### Geochemical – Round 13 - Proficiency Testing Program

### **Results Sheet**

Lab Code:

Analysis	Result 1	Result 2	Units	Detection Limit	Dissolution/ Digestion Technique (Please Specify)	Method Code	±MU*
Aluminium, Al			%				
Calcium Ca			%				
Iron, Fe			%				
Magnesium, Mg			%				
Manganese, Mn			%				
Phosphorous, P			%				
Potassium, K			%				
Silicon, Si			%				
Sodium, Na			%				
Titanium, Ti			%				
Zirconium, Zr			%				

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

Return no later than **FRIDAY 5 JANUARY 2017**, to: Kathy Weller, Proficiency Testing Australia. <u>phone</u>: +61 7 3721 7373, <u>fax</u>: +61 7 3217 1844, <u>email</u>: Kathy.Weller@pta.asn.au

- End of Report -