Report No. 1052

CEMENT
(ROUND 9)

PROFICIENCY TESTING PROGRAM

December 2017

ACKNOWLEDGMENTS

PTA wishes to gratefully acknowledge the technical assistance provided for this program by Mr P Kidd, Cement Australia Pty Ltd. This assistance included preparation, distribution and homogeneity testing of the samples, in addition to input into the design of the program, technical advice and discussion in the final report.

PO Box 7507, Silverwater NSW 2128, Australia

CONTENTS

1. FOREWORD

This report summarises the results of the ninth round of a proficiency testing program for laboratories testing the chemical and physical properties of cement. The aim of the program was to assess laboratories' ability to competently perform the prescribed analyses.

Proficiency Testing Australia conducted the program in September 2017. The Program Coordinator was Dr M Li and the Technical Adviser was Mr P Kidd from Cement Australia. This report was authorised by Mrs K Cividin, PTA Quality Manager - Senior Scientific Officer.

2. FEATURES OF THE PROGRAM

(a) A total of 18 laboratories (including laboratories from Australia, Iran, Malaysia, Fiji, Pakistan, New Zealand, Sri Lanka, Hong Kong and United Arab Emirates) received samples. Fourteen laboratories returned results for inclusion in the report.

(b) Participating laboratories were each supplied with one cement sample.

(c) The following determinations were to be performed on the samples:

- Chemical composition – SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, SO_3, Na_2O, K_2O, Cl;
- Loss on Ignition;
- Specific Surface Area (Blaine);
- Fineness by the 45µm Sieve;
- Median Particle Size; and
- Particle Size 3 to 32 µm.

(d) Eight randomly selected samples were analysed for homogeneity. Based on the results of this testing, it was concluded that the samples were sufficiently homogeneous, therefore any results later identified as outliers could not be attributed to sample variability (Appendix B).

(e) Laboratories were requested to perform the tests according to the "Instructions to Participants" and to record their results on the accompanying "Results Sheet", all of which were distributed to participants with the samples (Appendix C).

(f) Each laboratory was randomly allocated a unique code number for the program to enable confidentiality of results. Reference to each laboratory in this report is made by its code number.

3. FORMAT OF THE APPENDICES

(a) Appendix A contains the analysis of results reported by laboratories for the samples, and it contains the following for each determinant, where appropriate:

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

(b) Appendix B contains details of the homogeneity and stability testing.

(c) Appendix C contains copies of the *Instructions to Participants* and *Results Sheet*.

4. STATISTICAL DESIGN OF THE PROGRAM

Participating laboratories were each supplied one cement sample. A single result was reported for each test.

For each statistically analysed test, robust statistical procedures were used to generate the z-scores and summary statistics. The list of summary statistics include number of results, median, normalised interquartile range, uncertainty of the median, robust coefficient of variation, minimum, maximum and range.

In order to achieve the program's aim of assessing laboratories' testing performance, a robust statistical approach, which uses z-scores has been utilised. The z-score is a measure of how far the result(s) is from the consensus value - a normalised value which gives a "score" to each result relative to the other results in the group. Therefore a z-score close to zero means that the result agrees well with those from other laboratories. An outlier will be any result(s) which has an absolute z-score value greater than or equal to 3.0. For each laboratory, a single robust z-score was calculated. For further information on the calculation and interpretation of z-scores, please see the *Guide to Proficiency Testing Australia (2016)*[1].

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

The list of summary statistics appears at the bottom of the table of results and consists of:

the number of results for that test / technique (*No. of Results*);

the median of laboratories' 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 x *Normalised IQR* ÷ *Median*;

the minimum and maximum laboratory results;

the range (*Maximum* – *Minimum*); and

the uncertainty of the median; a robust estimate of the standard deviation of the median.

The Median is a measure of the centre of the data and the Normalised IQR is a measure of the spread of the results.

The Normalised IQR is calculated by multiplying the interquartile range (IQR) by a correction factor which converts the IQR to an estimate of the standard deviation. The IQR is the difference between the upper and lower quartiles (i.e. the values above and below which a quarter of the results lie, respectively).

For normally distributed data, the uncertainty of the median is approximated by:

$$\sqrt{\frac{\pi}{2}} \times \frac{normIQR}{\sqrt{n}}$$

n = number of results

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

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.

TABLE A: SUMMARY OF RESULTS

Test	No. of Results	Median	Uncertainty of the Median	Normalised IQR
SiO_2 (%)	12	19.98	0.13	0.35
Al_2O_3 (%)	12	5.80	0.09	0.24
Fe_2O_3 (%)	12	3.40	0.01	0.03
CaO (%)	12	64.75	0.18	0.49
MgO (%)	12	1.00	0.06	0.17
SO_3 (%)	12	2.70	0.03	0.08
Na_2O (%)	11	0.240	0.014	0.037
K_2O (%)	11	0.560	0.014	0.037
Cl (%)	9	0.0100	0.0019	0.0044
Loss on Ignition (%)	13	1.40	0.04	0.12
Specific Surface Area (Blaine) (cm^2/g)	11	4204.0	30.6	81.1
Fineness by the 45 µm Sieve (%)	8	1.10	0.04	0.09
Median Particle Size (µm)	2	N/A	N/A	N/A
Particle Size 3 to 32 µm (%)	2	N/A	N/A	N/A

Note: Summary Statistics for Median Particle Size and Particle Size 3 to 32 µm were not included due to the insufficient number of returned results.

5. OUTLIER RESULTS

Laboratories reporting outlier results are listed in the following table:

TABLE B: STATISTICAL OUTLIER RESULTS (By Laboratory Code)

Test	Laboratory Codes
SiO_2	1
Al_2O_3	-
Fe_2O_3	5, 8, 11, 14 and 17
CaO	2, 11
MgO	1, 2, 3
SO_3	-
Na_2O	4 and 14
K_2O	2, 18
Cl	-
Loss on Ignition	-
Specific Surface Area (Blaine)	6
Fineness by the 45µm Sieve	6, 14 and 15
Median Particle Size	N/A
Particle Size 3 to 32 µm	N/A

Note: Z-scores were not calculated for Median Particle Size and Particle Size 3 to 32 µm due to the insufficient number of returned results.

6. PTA AND TECHNICAL ADVISER'S COMMENTS

Metrological Traceability and Measurement Uncertainty of Assigned Values

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

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 and is presented in Table A.

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.

Chemical Composition and Physical Testing

(a) SiO_2

The robust CV obtained for this test was 1.8%. Laboratory code 1 reported a result that was identified as an outlier for this test.

(b) Al_2O_3

The robust CV obtained was 4.1%. No outliers were identified for this test.

(c) Fe_2O_3

The robust CV obtained was 0.8%. Laboratory codes 5, 8, 11, 14 and 17 reported results that were identified as outliers for this test.

(d) CaO

The robust CV was quite low for this test (0.7%). Laboratory codes 2 and 11 reported results that were identified as outliers for this test.

(e) MgO

The robust CV obtained was 16.7%. Laboratory codes 1, 2 and 3 reported results that were identified as outliers for this test.

(f) SO_3

No outliers were identified for this test. The CV of 0.1% was considered inappropriate to evaluate the participants' results. After reviewing the CVs from past rounds, a target CV of 3.0% was chosen to analyse the results.

(g) Na_2O

The robust CV obtained was 15.4%. Laboratory codes 4 and 14 reported results that were identified as outliers for this test.

(h) K_2O

The robust CV for this test was 6.6%. Laboratory codes 2 and 18 reported results that were identified as outliers for this test.

(i) Cl

The robust CV for this test was 44.5%. No outliers were identified for this test.

(j) Loss on Ignition

The robust CV obtained was 8.5%. No outliers were identified for this test.

(k) Specific Surface Area (Blaine)

The robust CV obtained was 1.9%. Laboratory code 6 reported a result that was identified as an outlier for this test.

(l) Fineness by the 45 µm Sieve

The robust CV obtained was 8.4%. Laboratory codes 6, 14 and 15 reported results that were identified as outliers for this test.

(m) Median Particle Size

Two laboratories returned results for this test.

(n) Particle Size 3 to 32 µm

Two laboratories returned results for this test.

Overall Performance

The overall performance (versus expectations) taking measurement uncertainties into account, was in agreement with the 2016 results and subsequent alignment across the various standards and methodology for analysis of cement. The expected differences across laboratories were relatively low with the chemical oxide analysis being reasonably tight. Perhaps the only difference worth mentioning in the oxide analysis was MgO which may be prone to perturbations in the XRF calibrations and in turn, higher differences within laboratories and between laboratories. SO_3 also had a higher than usual number of outliers (based on z-scores), however the range was only 0.3 overall. Other lighter elements such as Na and Cl gave very good results with a relatively low CV.

There were minimal instances of incorrect units reported in contrast to previous rounds where misalignment was generally due to incorrect reporting units (typically Specific Surface Area and 45 micron Residue); however several instances of incorrect Specific Surface Area units still

persist. In general, participating laboratories reported to the required precision.

The variation within and between laboratories was reasonable and within the expected range as compared to other proficiency testing programs. Oxide analysis and Loss of Ignition were acceptable. Chloride ion analysis was surprisingly good as the broad range of analysis techniques coupled with the relatively low medium value usually gives a much broader range of results. The range of 2.14% for Fineness by 45µm Sieve was exceptionally high and due to an anomalous outlier that was more than double the median. One major shortcoming of sieve testing is the assumption that all screens will have the exact wire diameters. A slight variation in wire diameter can translate to a significant change in opening size. Alternatively, bias in sieve results may be caused by a poor procedure or the use of non-calibrated sieves. An optimum procedure and careful calibration will minimise bias. Dry screening is also thought to produce a higher error than wet screening.

Variation between test methods or procedures was acceptable. Oxide test procedures were comparable and it appears that the most used was X-Ray Fluorescence (XRF), although chloride analysis was undertaken using various methods including both XRF and traditional wet chemistry. Most outliers ($|z\text{-score}| \geq 3.0$) would probably still be within the expected reproducibility limits of nearly all the cement test methods. In this regard, the “z-score” acceptable range may need some adjustment to suit the reproducibility of specific tests.

Sources of error were varied in this round and not solely attributable to reporting errors as in previous rounds. Some oxide results appear to be either affected by a contaminant or otherwise have been slightly skewed by calibration issues; however, overall the oxide results show very good alignment. Fineness by 45µm Sieve appears to be a problematic area in the round and participating laboratories with results denoted as outliers should ideally review their equipment and methodology.

The Measurement Uncertainty (MU) estimates were typically comparable for the oxide and Loss of Ignition analyses. For the physical testing, a larger range was reported, which is to be expected. The 45 micron Residue range was moderate considering the relatively low number; however the Specific Surface Area range was relatively small in view of the inherent variation in the air permeability test.

The acceptance criteria for the homogeneity data was based on Appendix A, Table 1 of AS 2350.2 – 2006, Methods of testing portland, blended and masonry cements; Method 2: Chemical composition. Repeatability (% absolute) is probably a better gauge as compared to the CV. This is because some of the results were relatively low numbers.

Overall, the participating laboratories performed well which reflects well on the cement industry as a whole. Additionally, there appears to be very

good alignment across the various test methods and standards. Chloride testing appears to be one where the most improvement has occurred. The response to the Particle Size section was less than expected but it is recommended to retain this section as particle size distribution technology and accessibility is improving in the industry and will eventually make other particle measurement methods redundant. The main point taken from the particle size distribution analysis is that the wet carrier methods versus the dry carrier produce significantly different results. The dry method appears to give lower medium particle size as well as a lower 3-32µm fraction.

Hexavalent chromium found in Portland cement is coming under increasing scrutiny, which in turn is reflected in the tightening of specifications and standards worldwide. Therefore the determination of Hexavalent chromium will most likely be included in future proficiency programs.

7. REFERENCES

[1]. *Guide to Proficiency Testing Australia, 2016.*
This document can be found on the PTA website at www.pta.asn.au.

[2]. AS 2350.2-2006 *Methods of testing portland, blended and masonry cements; Method 2: Chemical composition*.

APPENDIX A

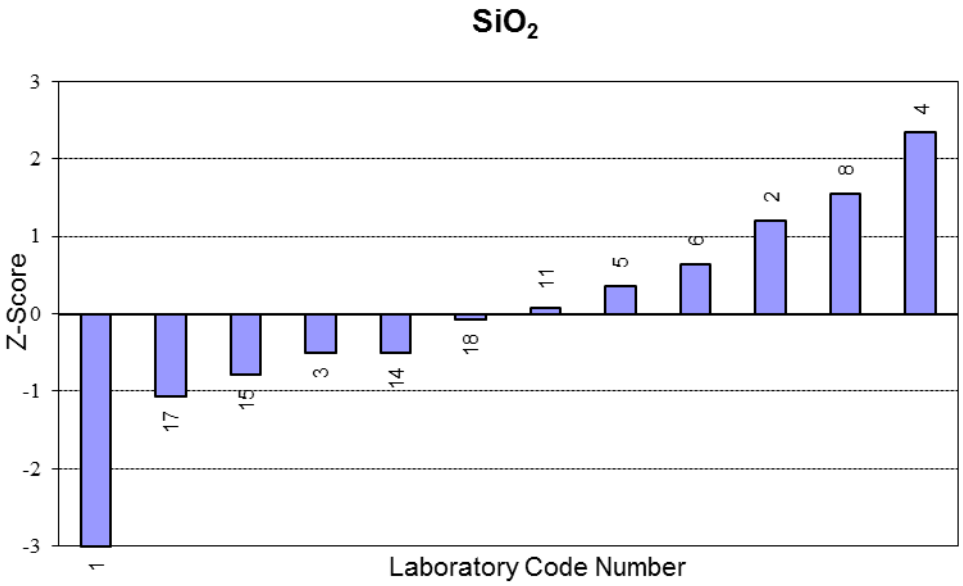
Results and Data Analysis

SiO_2

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	18.8	-3.34 §	0.22	BSEN-196-2
2	20.4	1.21		SLS 107: Part 2:2008
3	19.8	-0.50	2.0	BS EN196-2:2005 Cl. 13.2 to 13.9
4	20.8	2.34		BSEN 196 2 : 2013
5	20.1	0.35	0.50	MS EN 196-2:2007
6	20.2	0.64	0.2	AS2350.2
8	20.52	1.55	0.50	x-ray
11	20.0	0.07		MS EN 196-2
14	19.80	-0.50		ISIRI 1692
15	19.7	-0.78	1.0	MS EN 196-2:2007
17	19.60	-1.06	0.13	LAB - WI 01
18	19.95	-0.07	0.2	ASTM C114

No. Results	12
Median	19.98
Normalised IQR	0.35
Uncertainty (Median)	0.13
Robust CV	1.8%
Minimum	18.8
Maximum	20.8
Range	2.0

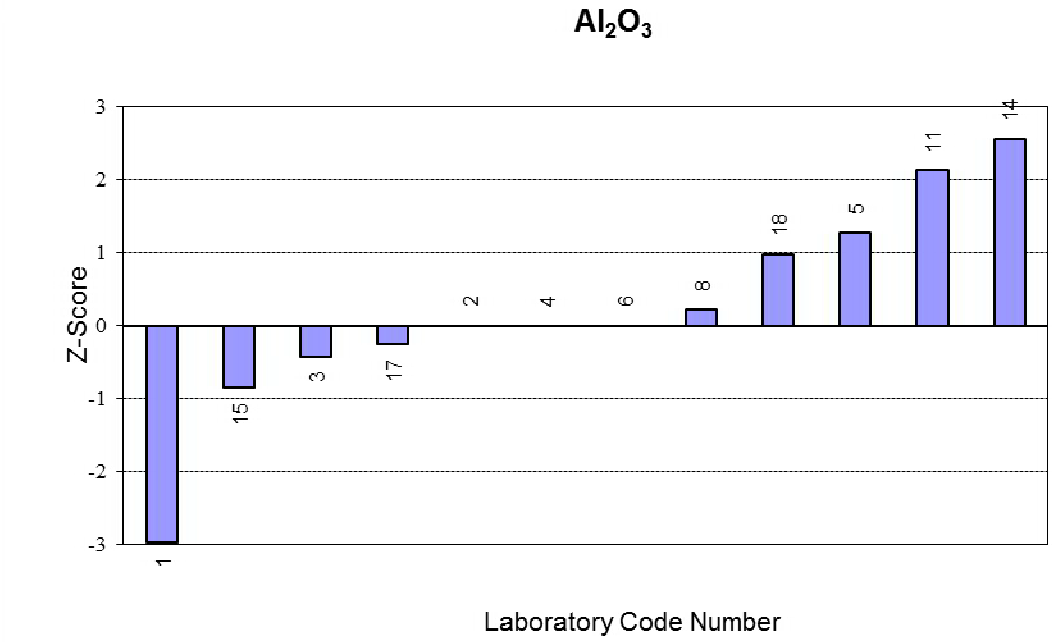
NOTE: § denotes an outlier (i.e. |z-score| ≥ 3.0).



Al_2O_3

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	5.1	-2.97	0.06	BSEN-196-2
2	5.8	0.00		SLS 107: Part 2:2008
3	5.7	-0.42	0.5	BS EN196-2:2005 Cl. 13.11
4	5.8	0.00		BSEN 196 2 : 2013
5	6.1	1.27	0.18	MS EN 196-2:2007
6	5.8	0.00	0.2	AS2350.2
8	5.85	0.21	0.52	x-ray
11	6.3	2.12		MS EN 196-2
14	6.40	2.55		ISIRI 1692
15	5.6	-0.85	0.6	MS EN 196-2:2007
17	5.74	-0.25	0.12	LAB - WI 02
18	6.03	0.98	0.2	ASTM C114

No. Results	12
Median	5.80
Normalised IQR	0.24
Uncertainty (Median)	0.09
Robust CV	4.1%
Minimum	5.1
Maximum	6.40
Range	1.30

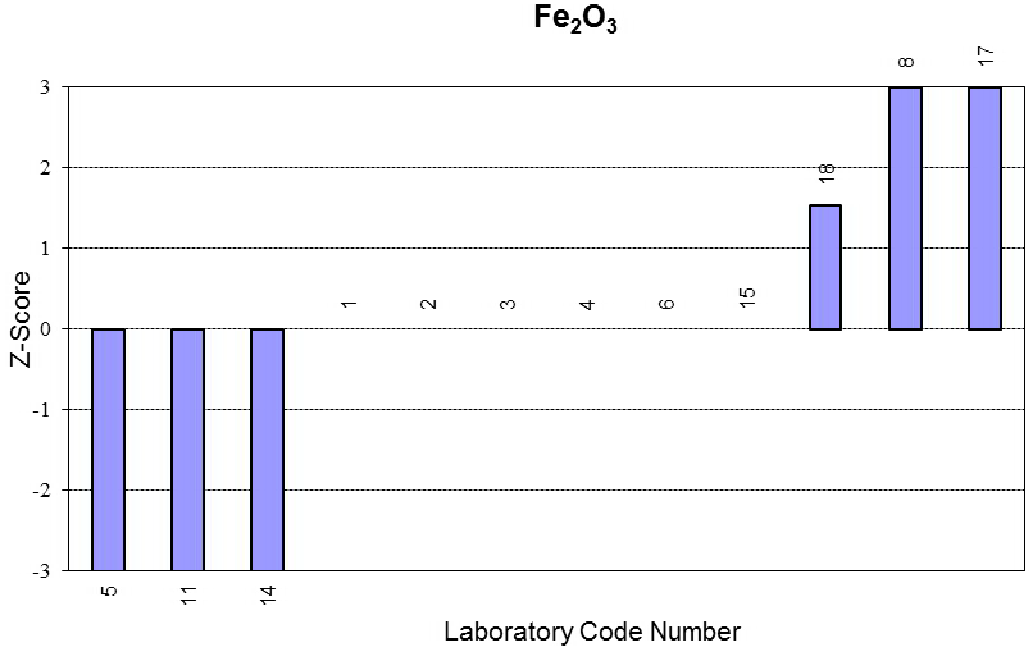


Fe_2O_3

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	3.4	0.00	0.06	BSEN-196-2
2	3.4	0.00		SLS 107: Part 2:2008
3	3.4	0.00	0.3	BS EN196-2:2005 Cl. 13.10
4	3.4	0.00		BSEN 196 2 : 2013
5	3.2	-7.71 §	0.30	MS EN 196-2:2007
6	3.4	0.00	0.1	AS2350.2
8	3.49	3.47 §	0.30	x-ray
11	3.3	-3.85 §		MS EN 196-2
14	3.30	-3.85 §		ISIRI 1692
15	3.4	0.00	0.4	MS EN 196-2:2007
17	3.52	4.63 §	0.02	LAB - WI 03
18	3.44	1.54	0.1	ASTM C114

No. Results	12
Median	3.40
Normalised IQR	0.03
Uncertainty (Median)	0.01
Robust CV	0.8%
Minimum	3.2
Maximum	3.52
Range	0.32

NOTE: **§** denotes an outlier (i.e. |z-score| ≥ 3.0).



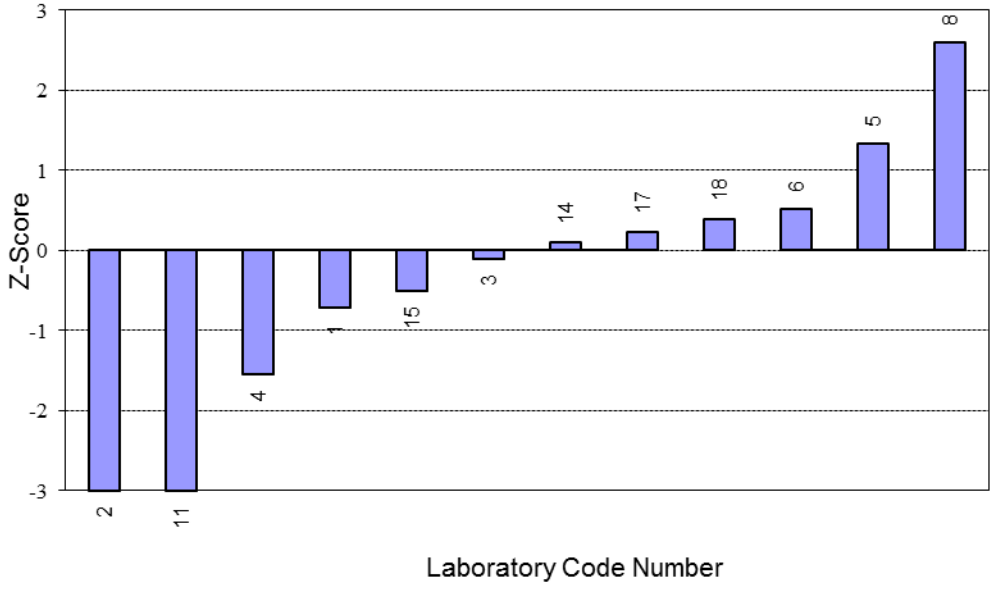
CaO

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	64.4	-0.72	0.1	BSEN-196-2
2	62.6	-4.43 §		SLS 107: Part 2:2008
3	64.7	-0.10	5.8	BS EN196-2:2005 Cl. 13.14
4	64.0	-1.54		BSEN 196 2 : 2013
5	65.4	1.34	0.87	MS EN 196-2:2007
6	65.0	0.51	0.3	AS2350.2
8	66.01	2.59	0.86	x-ray
11	62.8	-4.02 §		MS EN 196-2
14	64.80	0.10		ISIRI 1692
15	64.5	-0.51	1.4	MS EN 196-2:2007
17	64.86	0.23	0.06	LAB - WI 07
18	64.94	0.39	0.3	ASTM C114

No. Results	12
Median	64.75
Normalised IQR	0.49
Uncertainty (Median)	0.18
Robust CV	0.7%
Minimum	62.6
Maximum	66.01
Range	3.4

NOTE: **§** denotes an outlier (i.e. |z-score| ≥ 3.0).

CaO

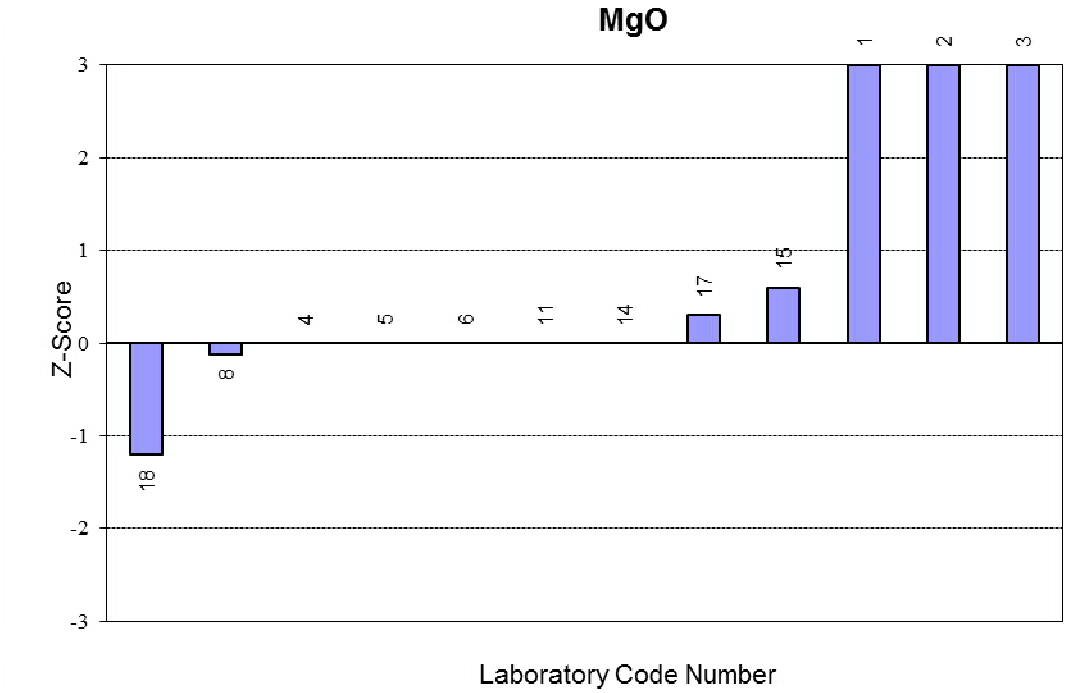


MgO

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	1.6	3.60 §	0.11	BSEN-196-2
2	1.6	3.60 §		SLS 107: Part 2:2008
3	1.8	4.80 §	0.2	BS EN196-2:2005 Cl. 13.15
4	1.0	0.00		BSEN 196 2 : 2013
5	1.0	0.00	0.50	MS EN 196-2:2007
6	1.0	0.00	0.1	AS2350.2
8	0.98	-0.12	0.50	x-ray
11	1.0	0.00		MS EN 196-2
14	1.00	0.00		ISIRI 1692
15	1.1	0.60	0.4	MS EN 196-2:2007
17	1.05	0.30	0.06	LAB - WI 08
18	0.8	-1.20	0.2	ASTM C114

No. Results	12
Median	1.00
Normalised IQR	0.17
Uncertainty (Median)	0.06
Robust CV	16.7%
Minimum	0.8
Maximum	1.8
Range	1.0

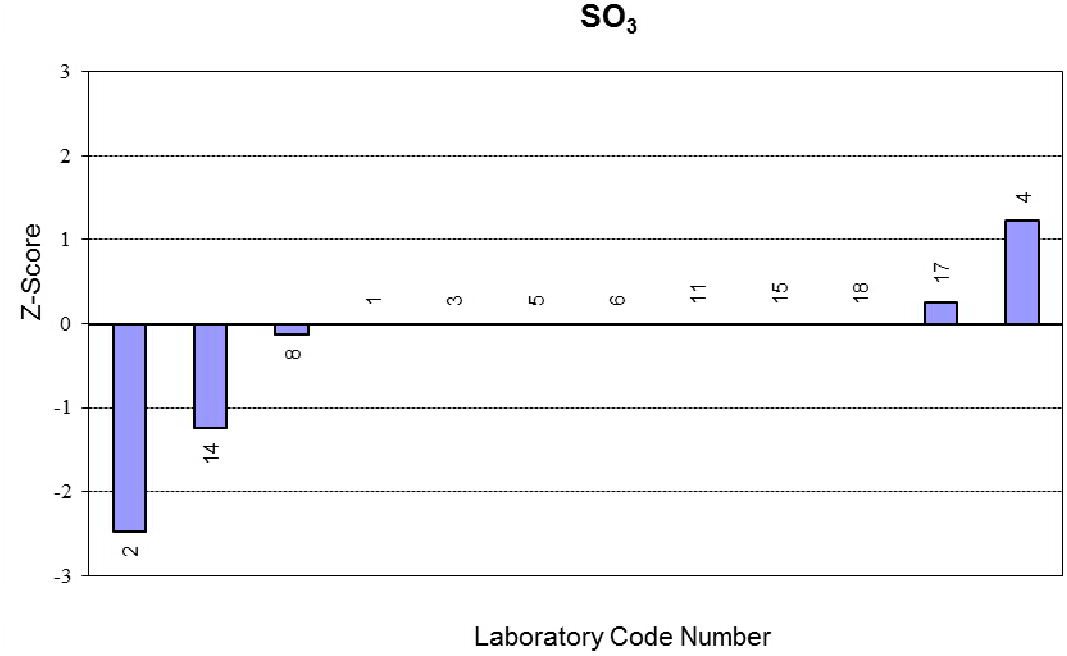
NOTE: **§** denotes an outlier (i.e. |z-score| ≥ 3.0).



SO_3

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	2.7	0.00	0.06	BSEN-196-2
2	2.5	-2.47	0.1%	SLS ISO 29581-1:2011
3	2.7	0.00	0.2	BS EN196-2:2005 Cl. 8
4	2.8	1.23		BSEN 196 2 : 2013
5	2.7	0.00	0.16	MS EN 196-2:2007
6	2.7	0.00	0.2	AS2350.2
8	2.69	-0.12	0.11	MS EN 196-2:2007
11	2.7	0.00		MS EN 196-2
14	2.60	-1.23		ISIRI 1692
15	2.7	0.00	0.4	MS EN 196-2:2007
17	2.72	0.25	0.013	LAB - WI 09
18	2.70	0.00	0.1	ASTM C114

No. Results	12
Median	2.70
Target IQR	0.08
Uncertainty (Median)	0.03
Target CV	3.0%
Minimum	2.5
Maximum	2.8
Range	0.3



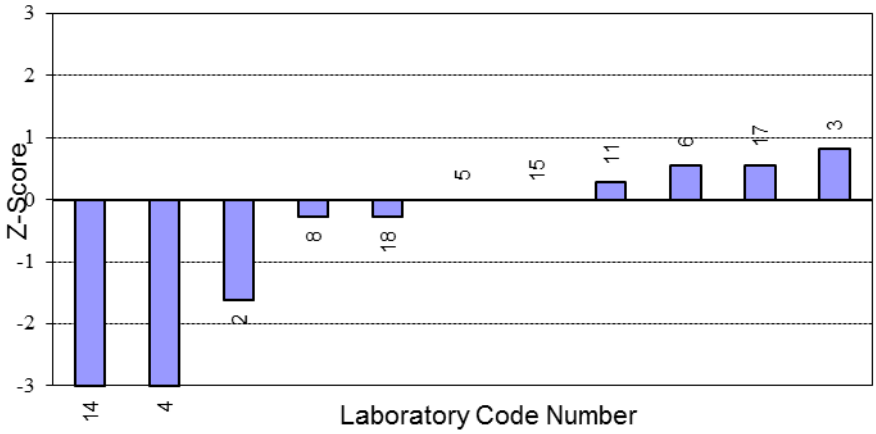
Na_2O

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
2	0.18	-1.62		SLS ISO 29581-1:2011
3	0.27	0.81	0.03	BS EN196-2:2005 Cl. 17
4	0.10	-3.78 §		IHP - 126
5	0.2	0.00	0.04	MS EN 196-2:2007
6	0.26	0.54	0.04	AS2350.2
8	0.23	-0.27		x-ray
11	0.25	0.27		MS EN 196-2
14	0.09	-4.05 §		ISIRI 1695
15	0.24	0.00	0.13	MS EN 196-2:2007
17	0.26	0.54	0.02	LAB - WI 05
18	0.23	-0.27	0.05	ASTM C114

No. Results	11
Median	0.240
Normalised IQR	0.037
Uncertainty (Median)	0.014
Robust CV	15.4%
Minimum	0.09
Maximum	0.27
Range	0.18

NOTE: **§** denotes an outlier (i.e. |z-score| ≥ 3.0).

Na_2O



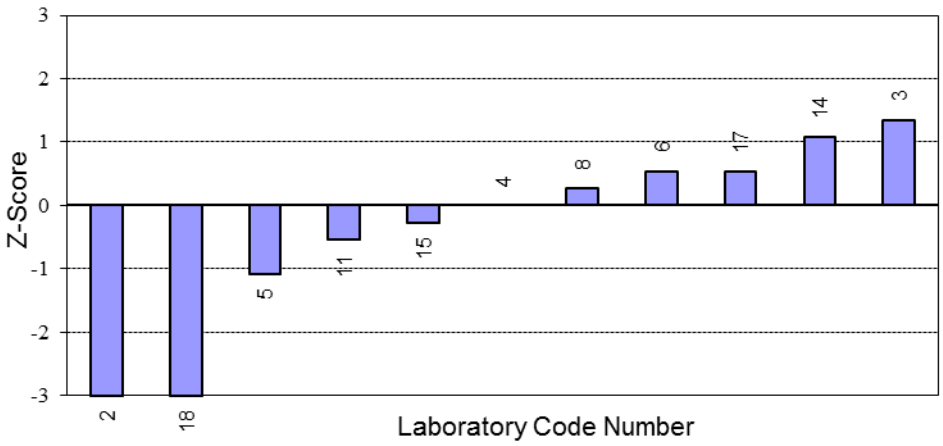
K_2O

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
2	0.36	-5.40 §		SLS ISO 29581-1:2011
3	0.61	1.35	0.06	BS EN196-2:2005 Cl. 17
4	0.56	0.00		IHP - 126
5	0.52	-1.08	0.06	MS EN 196-2:2007
6	0.58	0.54	0.03	AS2350.2
8	0.57	0.27		x-ray
11	0.54	-0.54		MS EN 196-2
14	0.60	1.08		ISIRI 1695
15	0.55	-0.27	0.10	MS EN 196-2:2007
17	0.58	0.54	0.02	LAB - WI 06
18	0.42	-3.78 §	0.05	ASTM C114

No. Results	11
Median	0.560
Normalised IQR	0.037
Uncertainty (Median)	0.014
Robust CV	6.6%
Minimum	0.36
Maximum	0.61
Range	0.25

NOTE: § denotes an outlier (i.e. |z-score| ≥ 3.0).

K_2O

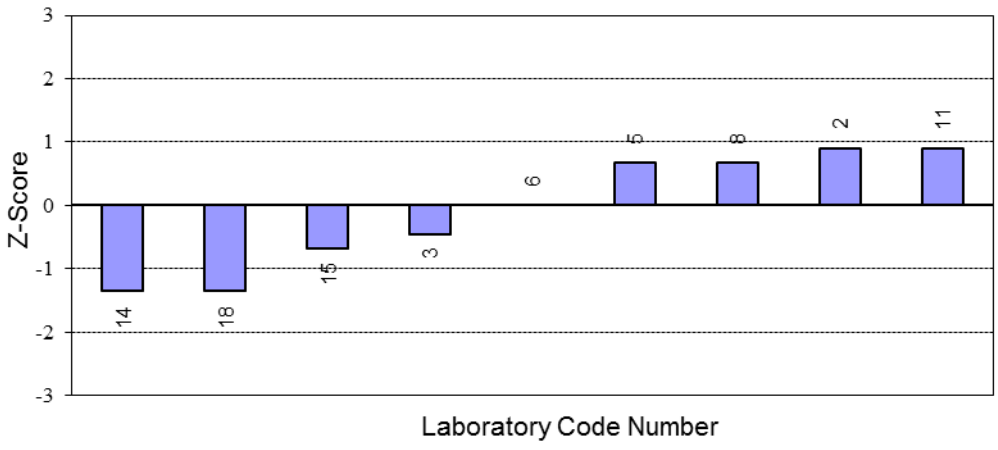


Cl

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
2	0.014	0.90	1%	SLS ISO 29581-1:2011
3	0.008	-0.45	NA	BS EN196-2:2005 Cl. 14
5	0.013	0.67	0.02	MS EN 196-2:2007
6	0.010	0.00	0.005	AS2350.2
8	0.013	0.67	0.020	MS EN 196-2:2007
11	0.014	0.90		MS EN 196-2
14	0.004	-1.35		ISIRI 6443
15	0.007	-0.67	0.005	MS EN 196-2:2007
18	0.004	-1.35	0.003	ASTM C114

No. Results	9
Median	0.0100
Normalised IQR	0.0044
Uncertainty (Median)	0.0019
Robust CV	44.5%
Minimum	0.004
Maximum	0.014
Range	0.010

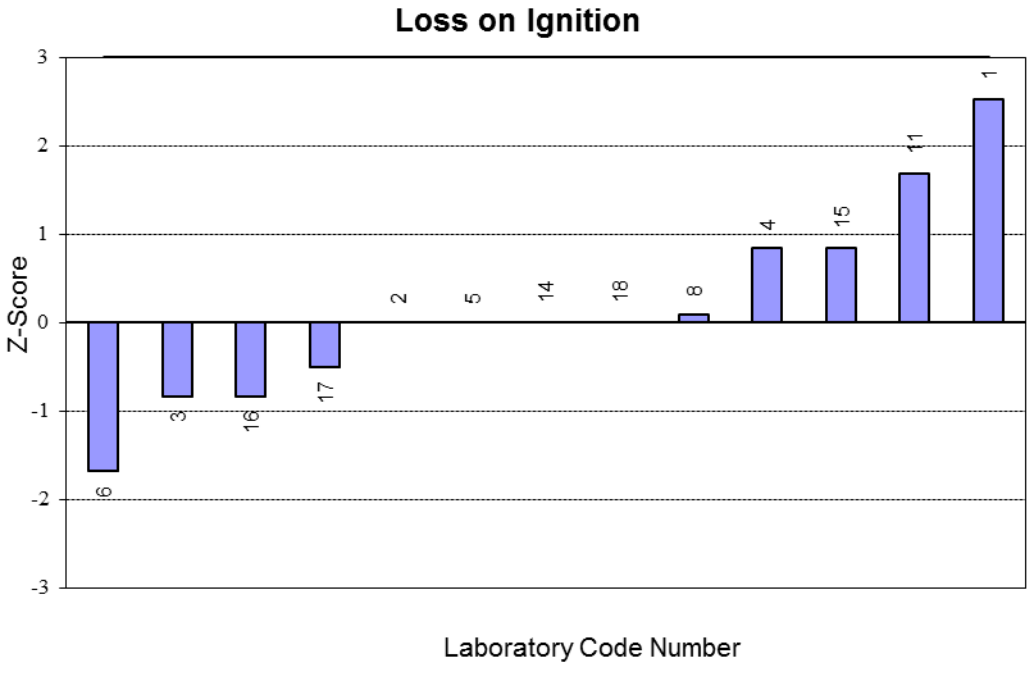
Cl



Loss on Ignition

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	1.7	2.53	0.05	BSEN-196-2
2	1.4	0.00	0.1%	SLS ISO 29581-1:2011
3	1.3	-0.84	0.1	BS EN196-2:2005 Cl. 17
4	1.5	0.84		BSEN 196 2 : 2013
5	1.4	0.00	0.16	MS EN 196-2:2007
6	1.2	-1.69	0.2	AS2350.2
8	1.41	0.08	0.16	MS EN 196-2:2007
11	1.6	1.69		MS EN 196-2
14	1.40	0.00		ISIRI 1692
15	1.5	0.84	0.3	MS EN 196-2:2007
16	1.3	-0.84	0.1	AS2350.2
17	1.34	-0.51	0.06	LAB - WI 10
18	1.4	0.00	0.1	ASTM C114

No. Results	13
Median	1.40
Normalised IQR	0.12
Uncertainty (Median)	0.04
Robust CV	8.5%
Minimum	1.2
Maximum	1.7
Range	0.5

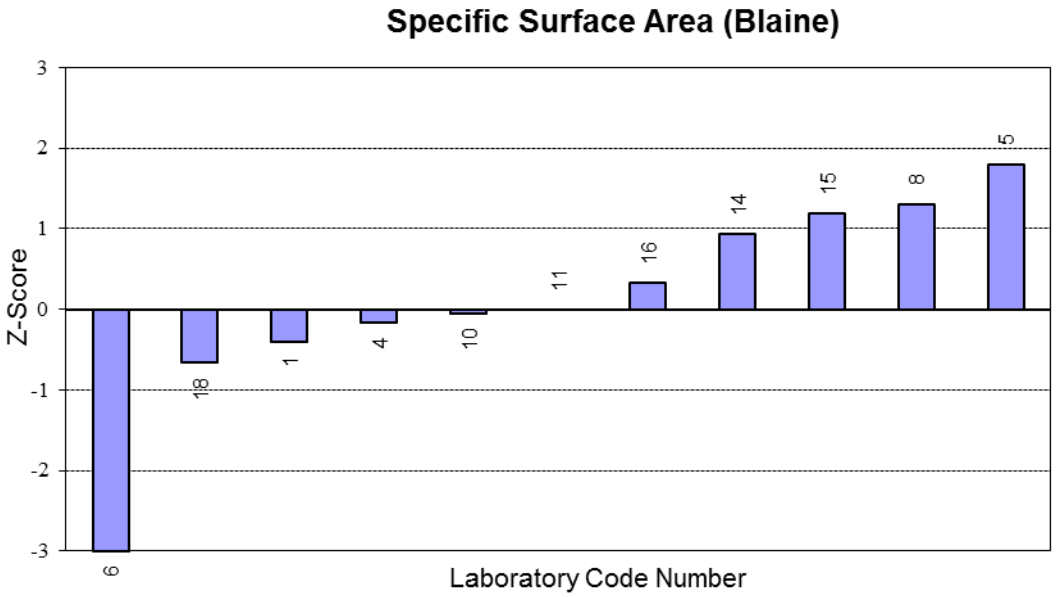


Specific Surface Area (Blaine)

Lab Code	Sample A (cm^2/g)	Z-Score	MU	Method/Technique
1	4171.3	-0.40	96	ASTM C204
4	4190	-0.17		BSEN 196 6 : 2010
5	4350	1.80	7.0	MS EN 196-6:2007
6	410	-46.80 §	50	AS2350.8
8	4310	1.31	200	MS EN 196-6:2007
10	4200	-0.05	13	ASTMC204-2016
11	4204	0.00		MS EN 196-6
14	4280	0.94		ISIRI 390
15	4300	1.18	100	MS EN 196-2:2007
16	4230	0.32	10	AS2350.8
18	4150	-0.67	90	EN196-6

No. Results	11
Median	4204.0
Normalised IQR	81.1
Uncertainty (Median)	30.6
Robust CV	1.9%
Minimum	410
Maximum	4350
Range	3940

NOTE: § denotes an outlier (i.e. |z-score| ≥ 3.0).

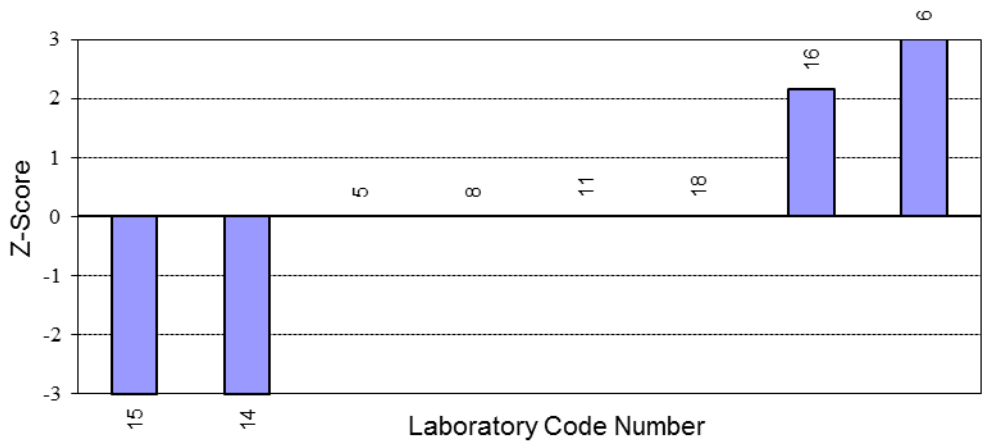


Fineness by the 45 µm Sieve

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
5	1.1	0.00		MS EN 196-6:2007
6	2.5	15.11 §	0.3	AS2350.9
8	1.1	0.00		MS EN 196-6:2007
11	1.1	0.00		in-house method
14	0.80	-3.24 §		ASTM C-430
15	0.36	-7.99 §	0.24	MS EN 196-2:2007
16	1.30	2.16	0.28	AS2350.9
18	1.1	0.00	0.2	EN196-6

No. Results	8
Median	1.10
Normalised IQR	0.09
Uncertainty (Median)	0.04
Robust CV	8.4%
Minimum	0.36
Maximum	2.50
Range	2.14

NOTE: § denotes an outlier (i.e. |z-score| ≥ 3.0).



Median Particle Size

Lab Code	Sample A (µm)	MU	Method/Technique
6	12.0	1.0	Laser Granulometry Malvern (Dry)
15	11.1	0.4	Using Malvern Mastersize 2000 (Dry Method)

No. Results 2

NOTE: Summary statistics were not calculated due to the insufficient number of returned results.

Particle Size 3 to 32 µm

Lab Code	Sample A (%)	MU	Method/Technique
6	69.1	2.0	Laser Granulometry Malvern (Dry)
15	71.4	0.5	Using Malvern Mastersizer 2000 (Dry Method)

No. Results 2

NOTE: Summary statistics were not calculated due to the insufficient number of returned results.

APPENDIX B

Sample Homogeneity

HOMOGENEITY TESTING

The samples utilised in this program were supplied by Cement Australia Pty Ltd. Each participant was provided with one sample.

For this program, 8 samples were randomly selected and tested for homogeneity. Statistical analysis showed that the samples were sufficiently homogeneous so that any results identified as outliers could not be attributed to sample variability.

The results of the homogeneity testing, along with the summary statistics are provided in Table C.

TABLE C: HOMOGENEITY TESTING RESULTS

Test	LOI %	CaO %	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	MgO %	SO_3 %	Na_2O %	K_2O %
Sample 1	1.1	64.9	20.2	5.8	3.4	1.0	2.68	0.27	0.57
Sample 2	1.1	64.9	20.2	5.8	3.4	1.0	2.69	0.28	0.58
Sample 3	1.1	64.9	20.2	5.8	3.4	1.0	2.70	0.25	0.58
Sample 4	1.1	64.9	20.2	5.8	3.3	1.0	2.68	0.26	0.57
Sample 5	1.1	64.9	20.2	5.8	3.4	1.0	2.70	0.27	0.57
Sample 6	1.1	64.9	20.2	5.8	3.4	1.0	2.69	0.25	0.58
Sample 7	1.1	64.9	20.2	5.8	3.4	1.0	2.69	0.27	0.58
Sample 8	1.1	65.0	20.2	5.8	3.4	1.0	2.72	0.27	0.58
Median	**1.13**	**64.92**	**20.18**	**5.79**	**3.38**	**0.97**	**2.690**	**0.270**	**0.580**
Norm IQR	**0.00**	**0.03**	**0.02**	**0.01**	**0.01**	**0.00**	**0.009**	**0.009**	**0.007**
Robust CV	**0.2%**	**0.1%**	**0.1%**	**0.2%**	**0.3%**	**0.4%**	**0.3%**	**3.4%**	**1.3%**

APPENDIX C

Documentation

PROFICIENCY TESTING AUSTRALIA

Proficiency Testing Program

Cement (Round 9)

INSTRUCTIONS TO PARTICIPANTS

Please read the following carefully before commencing testing.

Each participant will be supplied with one 50g sample of OPC Cement (General Purpose Cement with 5% - 7.5% limestone mineral additive).

The proficiency test item should be treated as a routine sample. To ensure the appropriate analysis of results, participants are asked to adhere carefully to the following instructions:

1) Prior to any testing remix the sample thoroughly.

2) The following tests are to be performed on the sample:

   (i) Chemical Composition: SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, SO_3, Na_2O, K_2O, Cl (Chloride)

   (ii) Loss on Ignition (970 ^{0}C)

   (iii) Specific Surface Area (Blaine): Relative Density = 3.11 g/cm^3

   (iv) Fineness by 45µm Sieve (wet)

(v) Laser Granulometry: Report Median Particle Size (µm) and Particle Size 3 to 32 µm (%).

3) For each determination, the test result is to be reported on the Results Sheet to the reporting basis indicated. The method used for each test is to be stated, e.g. AS 2350.8 for Fitness by the 45µm Sieve. Attach additional comments if necessary.

4) Laboratories are also requested to calculate and report an estimate of uncertainty of measurement for each reported result. All estimates of uncertainty of measurement must be given as a 95% confidence interval (coverage factor k = 2). Please note that MU estimates are requested for information purposes only and will not be used for the formal evaluation of results

5) All laboratories are asked to return the Results Sheet by **30 September 2017** to:

Dr Michael Li
Proficiency Testing Australia
PO Box 7507
Silverwater NSW 2128
AUSTRALIA
Phone +61 2 9736 8397
Fax +61 2 9743 6664
Email michael.li@pta.asn.au

PROFICIENCY TESTING AUSTRALIA

Cement (Round 9) - Proficiency Testing Program
Results Sheet

Lab Code:

Test	Results	±MU	Method
SiO_2 (Report to 0.1%)			
Al_2O_3 (Report to 0.1%)			
Fe_2O_3 (Report to 0.1%)			
CaO (Report to 0.1%)			
MgO (Report to 0.1%)			
SO_3 (Report to 0.1%)			
Na_2O (Report to 0.01%)			
K_2O (Report to 0.01%)			
Cl (Report to 0.001%)			
Loss on ignition (970^0) (Report to 0.1%)			
Specific Surface Area (Blaine) (report to cm^2/g - nearest 5)			
Fineness by the 45 µm sieve (report to 0.1%)			
Median Particle size (report to 0.1 µm)			
Particle size 3 to 32 µm (report to 0.1%)			

Signed: _____ **Date:** _____

TO: Dr Michael Li
Proficiency Testing Australia, PO Box 7507, Silverwater NSW 2128, AUSTRALIA
Phone +61 2 9736 8397 Fax +61 2 9743 6664 Email michael.li@pta.asn.au

End of Report