



Report No. 835

**CEMENT
(ROUND 5)**

PROFICIENCY TESTING PROGRAM

December 2013

ACKNOWLEDGMENTS

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

© COPYRIGHT PROFICIENCY TESTING AUSTRALIA 2013
PO Box 7507, Silverwater NSW 2128, Australia

CONTENTS

	Page
1. Foreword	1
2. Statistical Design of the Program	1
3. Features of the Program	1
4. Summary of Results	2
5. Statistical Outlier Results	3
6. PTA and Technical Adviser's Comments	4
7. References	7

APPENDIX A - *Results and Data Analysis*

Chemical Composition:

SiO ₂	A1
Al ₂ O ₃	A2
Fe ₂ O ₃	A3
CaO	A4
MgO	A5
SO ₃	A6
Na ₂ O	A7
K ₂ O	A8
Loss on Ignition	A9
Specific Surface Area (Blaine)	A10
Fineness by the 45 µm Sieve	A11
Median Particle Size	A12
Particle Size 3 to 32 µm	A12

APPENDIX B - *Sample Homogeneity*

Homogeneity Testing	B1
---------------------	----

APPENDIX C - *Documentation*

Instructions to Participants	C1
Results Sheet	C2

1. **FOREWORD**

This report summarises the results of the fifth round of a proficiency testing program for laboratories testing the chemical and physical properties of cement.

Proficiency Testing Australia conducted the program in September 2013. The Program Coordinator was Dr M Li. This report was authorised by Ms W Fajloun, PTA Quality Coordinator. The aim of the program was to assess laboratories' ability to competently perform the prescribed analyses.

2. **STATISTICAL DESIGN OF THE PROGRAM**

Participating laboratories were each supplied one cement sample labelled PTA Sample A. 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 for each test - number of results, median, normalised interquartile range, uncertainty of the median, robust coefficient of variation, minimum, maximum and range.

3. **FEATURES OF THE PROGRAM**

- (a) A total of 33 laboratories (including laboratories from Australia, Fiji, Iran, Malaysia, New Zealand, Pakistan, Sudan, United Arab Emirates, Kuwait, Congo, R. P. China and United Kingdom) received samples. All laboratories except three returned results for inclusion in the final report.
- (b) Participating laboratories were each supplied one cement sample labelled PTA Sample A.
- (c) The following determinations were to be performed on the sample:
 - Chemical composition – SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , MgO , SO_3 , Na_2O , K_2O ;
 - Loss on Ignition;
 - Specific Surface Area (Blaine);
 - Fineness by the 45 μm Sieve;
 - Median Particle Size; and
 - Particle Size 3 to 32 μm .

- (d) Ten 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.

4. **SUMMARY OF RESULTS**

TABLE A: SUMMARY OF RESULTS

Test	No. of Results	Median	Median Uncertainty	Normalised IQR
SiO ₂ (%)	30	20.00	0.03	0.15
Al ₂ O ₃ (%)	30	4.16	0.08	0.37
Fe ₂ O ₃ (%)	30	3.01	0.02	0.07
CaO (%)	30	63.80	0.10	0.42
MgO (%)	30	1.61	0.04	0.18
SO ₃ (%)	32	2.65	0.02	0.07
Na ₂ O (%)	27	0.140	0.008	0.033
K ₂ O (%)	28	0.645	0.016	0.069
Loss on Ignition (%)	31	3.89	0.02	0.09
Specific Surface Area (Blaine) (cm ² /g)	26	3890	36	146
Fineness by the 45 µm Sieve (%)	19	5.70	0.42	1.45
Median Particle Size (µm)	5	N/A	N/A	N/A
Particle Size 3 to 32 µm (%)	5	N/A	N/A	N/A

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

5. STATISTICAL OUTLIER RESULTS

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 (2012)*¹.

TABLE B: STATISTICAL OUTLIER RESULTS (By Laboratory Code)

Test	Laboratory Codes
SiO ₂	1, 7, 8, 29, 30
Al ₂ O ₃	19
Fe ₂ O ₃	19
CaO	1, 7, 30
MgO	29
SO ₃	11, 12, 20
Na ₂ O	12, 19, 31
K ₂ O	12, 30
Loss on Ignition	7, 16, 18
Specific Surface Area (Blaine)	7, 10, 23, 27
Fineness by the 45 µm Sieve	Nil
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 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.

Chemical Composition and Physical Testing

(a) SiO_2

The robust CV obtained for this test was very good (0.7%). Laboratory codes 1, 7, 8, 29 and 30 reported results that were identified as outliers.

(b) Al_2O_3

The robust CV obtained was 8.8%. Laboratory code 19 was identified as reporting an outlier for this test.

(c) Fe_2O_3

The robust CV obtained was quite low for this test (2.5%). Laboratory code 19 reported a result that was identified as an outlier.

(d) CaO

The robust CV was quite low for this test (0.7%). Laboratory codes 1, 7 and 30 reported results that were identified as outliers.

(e) MgO

The robust CV was 11.4%. Laboratory code 29 was identified as having an outlier result.

(f) SO_3

The robust CV obtained was low (2.8%), with three laboratories (laboratory codes 11, 12 and 20) identified as having outlier results.

(g) Na_2O

The range of results reported for this test was much greater than for the analytes previously discussed, with a higher robust CV obtained (23.8%). Laboratory codes 12, 19 and 31 were identified as having outlier results.

(h) K_2O

The robust CV for this test was high (10.6%). Laboratory codes 12 and 30 were identified as having outlier results.

(i) *Loss on Ignition*

The performance of participating laboratories for this test was good with a robust CV of 2.2%. Laboratory codes 7, 16 and 18 were identified as having outlier results.

(j) *Specific Surface Area (Blaine)*

The performance of participating laboratories for this test was good, with quite a low robust CV obtained (3.7%). Four laboratories' results (laboratory codes 7, 10, 23 and 27) were identified as outliers.

(k) *Fineness by the 45 μm Sieve*

A wide range of results were reported for this test with a robust CV of 25.4%. No laboratory reported results identified as outliers.

(l) *Median Particle Size*

Five laboratories returned results for this test.

(m) *Particle Size 3 to 32 μm*

Five laboratories returned results for this test.

Overall Performance

The overall performance (versus expectations) taking measurement uncertainties into account, was satisfactory and there is obviously alignment across the various standards and methodology for analysis of cement. Participating laboratories have generally met expectations regarding expected differences across laboratories. However, there are still several instances of incorrect units reported (typically specific surface area), which has been a reoccurring issue in every cement program to date; although laboratories generally reported to the required precision.

The variation within and between laboratories was reasonable and within the expected range as compared to comparable proficiency testing programs. Oxide analysis and LOI were especially good. The physical testing results (surface area) were skewed due to 4 instances of the wrong unit of measurement used. However, taking the difference in measurement units into account, the actual analysis of the cement physical properties was reasonably close across all laboratories.

Variation between test methods or procedures was acceptable. Oxide test procedures were comparable and it appears that most used X-Ray Fluorescence (XRF). 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 (refer outlier results) were varied in this round and not solely attributable to reporting errors as in previous rounds. The Specific Surface Area tests results may be an exception in this regard. 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.

The measurement uncertainty estimates were typically comparable for the oxide and LOI analysis. For the physical testing, a larger range was reported, which is very similar to the last round. The 45 micron Residue range was acceptable, although the Specific Surface Area range reflected the difference in units as well as 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 Coefficient of Variation. This is because some of the results are 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. The response to the Particle Size section was less than expected but it is recommended to keep this section in as particle size distribution technology and accessibility is improving in the industry and will eventually make other particle measurement methods redundant.

7. **REFERENCES**

[1] *Guide to Proficiency Testing Australia, 2012.*

This document can be found on the PTA website at www.pta.asn.au.

[2] AS 2350.2 - *Methods of Testing Portland, Blended and Masonry Cements - Chemical Composition (2006).*

[3] ASTM C114 - *Standard Test Methods for Chemical Analysis of Hydraulic Cement (2010).*

[4] ASTM C204 - *Standard Test Methods for Fineness of Hydraulic Cement by Air-Permeability Apparatus (2007).*

APPENDIX A

Results and Data Analysis

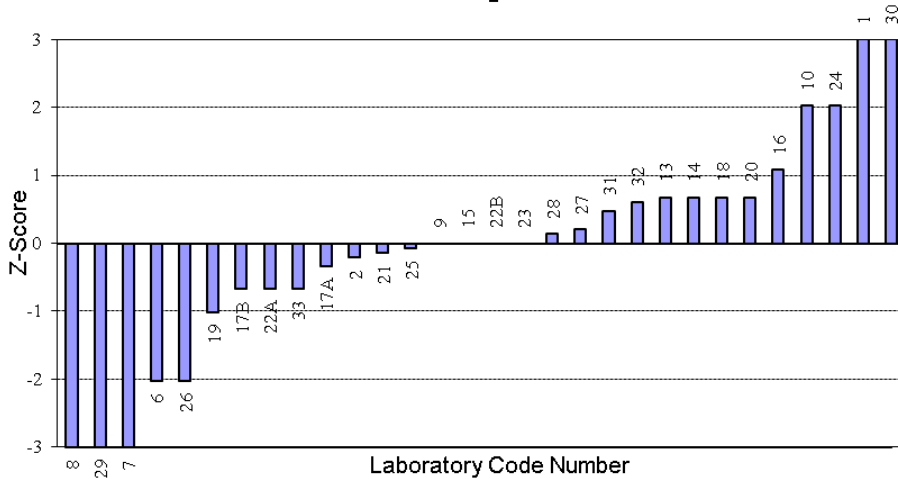
SiO₂	A1
Al₂O₃	A2
Fe₂O₃	A3
CaO	A4
MgO	A5
SO₃	A6
Na₂O	A7
K₂O	A8
Loss on Ignition	A9
Specific Surface Area (Blaine)	A10
Fineness by the 45 µm Sieve	A11
Median Particle Size	A12
Particle Size 3 to 32 µm	A12

SiO₂

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	20.6	4.05 §		XRF
2	19.97	-0.20	0.50	MSEN196-2:2007
6	19.7	-2.02	0.3	MSEN196-2:2007
7	19.51	-3.30 §	0.26	AS2350.2:2006
8	18.9	-7.42 §	0.2	ASTM C114
9	20.0	0.00	0.52	MS EN 196-2
10	20.3	2.02		AS2350.2
13	20.10	0.67		ASTM C114 ISIRI 1692
14	20.1	0.67		
15	20.00	0.00	0.136	ASTM C114
16	20.16	1.08	0.14	ISIRI 1692
17A	19.95	-0.34		ISIRI 1692
17B	19.9	-0.67		ISIRI 1692
18	20.1	0.67	0.2	ISIRI 1692
19	19.85	-1.01		ISIRI 1692
20	20.1	0.67		ISIRI 1692
21	19.98	-0.13	0.024	ASTM C114 (ISIRI 1692)
22A	19.9	-0.67	0.132	CNS1078
22B	20.0	0.00	0.151	ASTM C114
23	20.0	0.00	0.3	xf
24	20.3	2.02		MSEN196-2:2007
25	19.99	-0.07	0.50	EN196:Part-2:2007
26	19.7	-2.02		MSEN196-2:2007 Clause 13.6
27	20.03	0.20	0.20	XRF AS2350.2
28	20.2	1.35	0.14	ASTM C114-2010 ISIRI 1692
29	19.42	-3.91 §		ASTM C114
30	20.6	4.05 §		XRF
31	20.07	0.47	0.18	ISIRI 1692 ASTM C114
32	20.09	0.61	0.13	ISIRI 1692 BSEN 196.2
33	19.9	-0.67		BSEN 196-2:2005 Clause 13.5

No. Results 30
 Median 20.00
 Normalised IQR 0.15
 Uncertainty (Median) 0.03
 Robust CV 0.7%
 Minimum 18.9
 Maximum 20.6
 Range 1.7
 NOTE: § denotes an outlier (i.e. |z-score| ≥ 3.0).

SiO₂



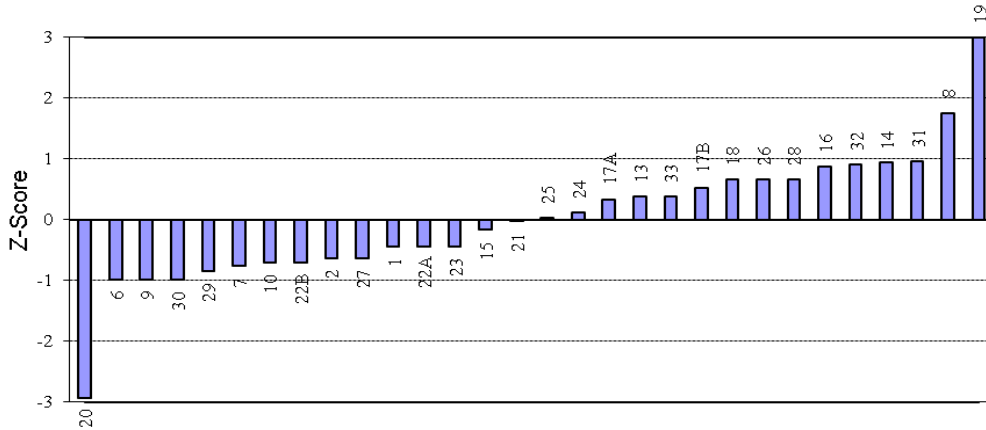
Al₂O₃

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	4.0	-0.44		XRF
2	3.93	-0.63	0.18	MSEN196-2:2007
6	3.8	-0.99	0.1	MSEN196-2:2007
7	3.88	-0.77	0.10	AS2350.2:2006
8	4.8	1.75	0.1	
9	3.8	-0.99	0.03	MS EN 196-2
10	3.9	-0.71		AS2350.2
13	4.30	0.38		ASTMC114 ISRI 1692
14	4.5	0.93		
15	4.10	-0.16	0.136	ASTMC114
16	4.48	0.88	0.06	ISIRI 1692
17A	4.28	0.33		ISIRI 1692
17B	4.35	0.52		ISIRI 1692
18	4.4	0.66	0.2	ISIRI 1692
19	11.79	20.90 §		ISIRI 1692
20	3.09	-2.93		ISIRI 1692
21	4.15	-0.03	0.024	ASTMC114 (ISIRI 1692)
22A	4.0	-0.44	0.180	CNS1078
22B	3.9	-0.71	0.175	ASTMC114
23	4.0	-0.44	0.2	xf
24	4.2	0.11		MSEN196-2:2007
25	4.17	0.03	0.52	EN196:Part-2:2007
26	4.4	0.66	0.20	MSEN196-2:2007 Clause 13.11
27	3.93	-0.63	0.41	XRF AS2350.2
28	4.4	0.66	0.16	ASTMC114-2010 ISIRI 1692
29	3.85	-0.85		ASTMC114
30	3.8	-0.99		XRF
31	4.51	0.96	0.08	ISIRI 1692 ASTM C114
32	4.49	0.90	0.05	ISIRI 1692 BSEN 196.2
33	4.3	0.38		BSEN 196-2:2005 Clause 13.11

No. Results 30
 Median 4.16
 Normalised IQR 0.37
 Uncertainty (Median) 0.08
 Robust CV 8.8%
 Minimum 3.09
 Maximum 11.79
 Range 8.70

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

Al₂O₃



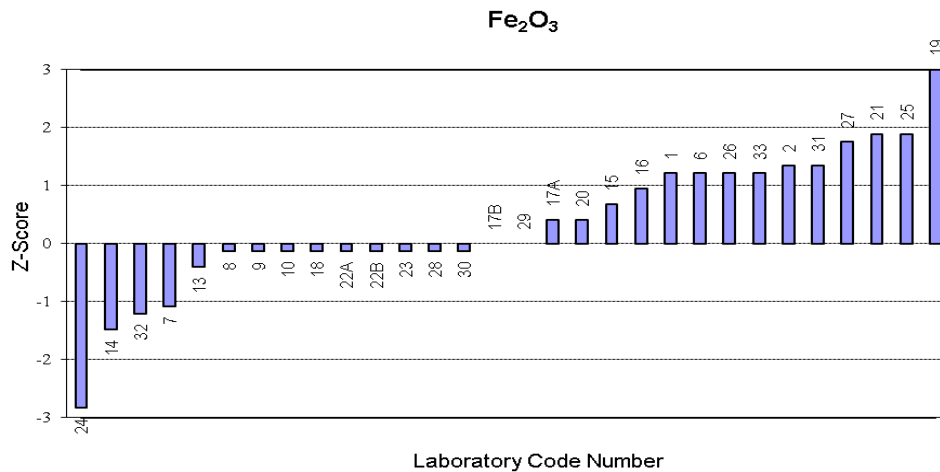
Laboratory Code Number

Fe₂O₃

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	3.1	1.21		XRF
2	3.11	1.35	0.30	MSEN196-2:2007
6	3.1	1.21	0.1	MSEN196-2:2007
7	2.93	-1.08	0.08	AS2350.2:2006
8	3.0	-0.13	0.1	
9	3.0	-0.13	0.21	MS EN 196-2
10	3.0	-0.13		AS2350.2
13	2.98	-0.40		ASTM C114 ISRI 1692
14	2.9	-1.48		
15	3.06	0.67	0.0004	ASTM C114
16	3.08	0.94	0.02	ISIRI 1692
17A	3.04	0.40		ISIRI 1692
17B	3.01	0.00		ISIRI 1692
18	3.0	-0.13	0.1	ISIRI 1692
19	3.36	4.72 §		ISIRI 1692
20	3.04	0.40		ISIRI 1692
21	3.15	1.89	0.024	ASTM C114 (ISIRI 1692)
22A	3.0	-0.13	0.024	CNS1078
22B	3.0	-0.13	0.099	ASTM C114
23	3.0	-0.13	0.5	xrf
24	2.8	-2.83		MSEN196-2:2007
25	3.15	1.89	0.30	EN196:Part-2:2007
26	3.1	1.21	0.22	MSEN196-2:2007 Clause 13.10
27	3.14	1.75	0.39	XRF AS2350.2
28	3.0	-0.13	0.10	ASTM C114-2010 ISIRI 1692
29	3.01	0.00		ASTM C114
30	3.0	-0.13		XRF
31	3.11	1.35	0.09	ISIRI 1692 ASTM C114
32	2.92	-1.21	0.04	ISIRI 1692 BSEN 196.2
33	3.1	1.21		BSEN 196-2:2005 Clause 13.10

No. Results	30
Median	3.01
Normalised IQR	0.07
Uncertainty (Median)	0.02
Robust CV	2.5%
Minimum	2.8
Maximum	3.36
Range	0.56

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



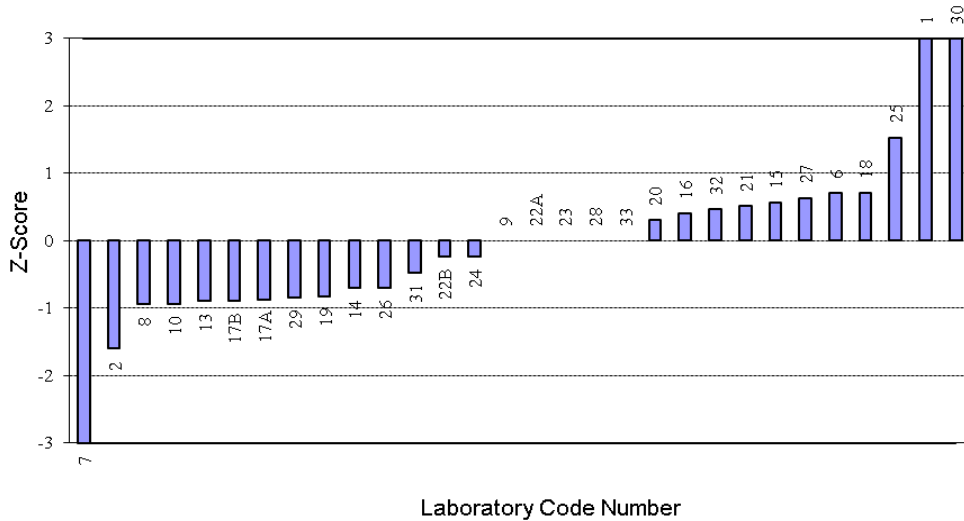
CaO

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	65.3	3.53 §		XRF
2	63.12	-1.60	0.87	MSEN196-2:2007
6	64.1	0.71	0.6	MSEN196-2:2007
7	61.3	-5.89 §	1.06	AS2350.2:2006
8	63.4	-0.94	0.3	
9	63.8	0.00	0.26	MS EN 196-2
10	63.4	-0.94		AS2350.2
13	63.42	-0.90		ASTM C114 ISRI 1692
14	63.5	-0.71		
15	64.04	0.57	0.0061	ASTM C114
16	63.97	0.40	0.18	ISIRI 1692
17A	63.43	-0.87		ISIRI 1692
17B	63.42	-0.90		ISIRI 1692
18	64.1	0.71	0.3	ISIRI 1692
19	63.45	-0.82		ISIRI 1692
20	63.93	0.31		ISIRI 1692
21	64.02	0.52	0.024	ASTM C114 (ISIRI 1692)
22A	63.8	0.00	0.196	CNS1078
22B	63.7	-0.24	0.342	ASTM C114
23	63.8	0.00	0.2	xrf
24	63.7	-0.24		MSEN196-2:2007
25	64.45	1.53	0.86	EN196:Part-2:2007
26	63.5	-0.71	1.57	MSEN196-2:2007 Clause 13.14
27	64.07	0.64	0.31	XRF AS2350.2
28	63.8	0.00	0.14	ASTM C114-2010 ISIRI 1692
29	63.44	-0.85		ASTM C114
30	66.4	6.13 §		XRF
31	63.60	-0.47	0.15	ISIRI 1692 ASTM C114
32	64.00	0.47	0.44	ISIRI 1692 BSEN 196.2
33	63.8	0.00		BSEN 196-2:2005 Clause 13.14

No. Results	30
Median	63.80
Normalised IQR	0.42
Uncertainty (Median)	0.10
Robust CV	0.7%
Minimum	61.3
Maximum	66.4
Range	5.1

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

CaO

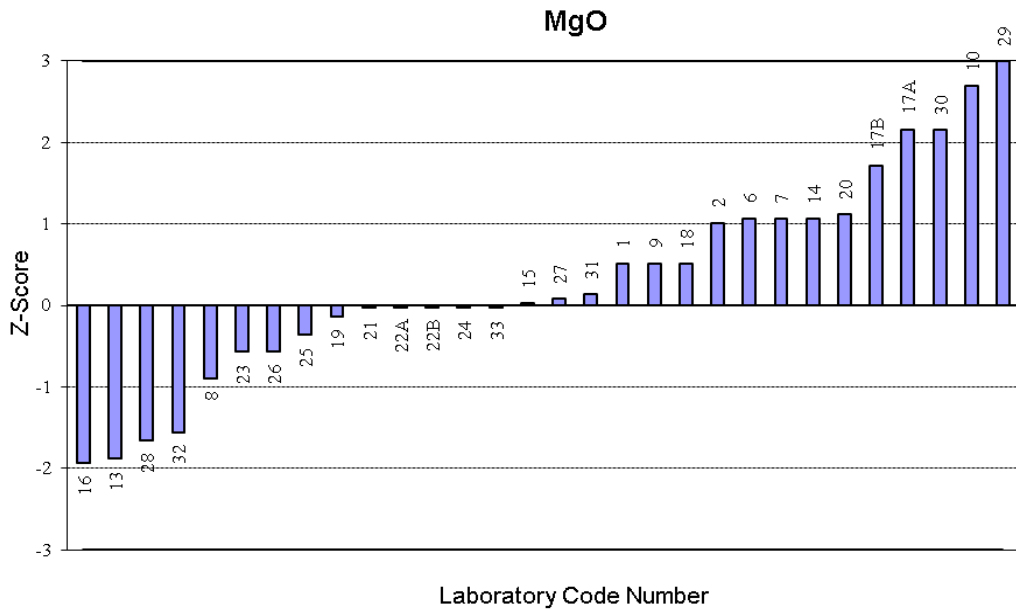


MgO

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	1.7	0.52		XRF
2	1.79	1.01	0.50	MSEN196-2:2007
6	1.8	1.06	0.1	MSEN196-2:2007
7	1.44	1.06	0.18	AS2350.2:2006
8	1.7	-0.90	0.05	
9	2.1	0.52	0.09	MS EN 196-2
10	1.6	2.70		AS2350.2
13	1.26	-1.88		ASTM C114 ISRI 1692
14	1.8	1.06		
15	1.61	0.03	0.0061	ASTM C114
16	1.25	-1.93	0.02	ISIRI 1692
17A	2	2.15		ISIRI 1692
17B	1.92	1.72		ISIRI 1692
18	1.7	0.52	0.2	ISIRI 1692
19	1.58	-0.14		ISIRI 1692
20	1.81	1.12		ISIRI 1692
21	1.60	-0.03	0.024	ASTM C114 (ISIRI 1692)
22A	1.6	-0.03	0.078	CNS1078
22B	1.6	-0.03	0.078	ASTM C114
23	1.5	-0.57	0.1	xf
24	1.6	-0.03		MSEN196-2:2007
25	1.54	-0.35	0.50	EN196:Part-2:2007
26	1.5	-0.57	0.29	MSEN196-2:2007 Clause 13.15
27	1.62	0.08	0.29	XRF AS2350.2
28	1.3	-1.66	0.06	ASTM C114-2010 ISIRI 1692
29	2.55	5.15 §		ASTM C114
30	2.0	2.15		XRF
31	1.63	0.14	0.03	ISIRI 1692 ASTM C114
32	1.32	-1.55	0.02	ISIRI 1962 BSEN 1962
33	1.6	-0.03		BSEN 196-2:2005 Clause 13.15

No. Results 30
 Median 1.61
 Normalised IQR 0.18
 Uncertainty (Median) 0.04
 Robust CV 11.4%
 Minimum 1.3
 Maximum 2.55
 Range 1.30

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

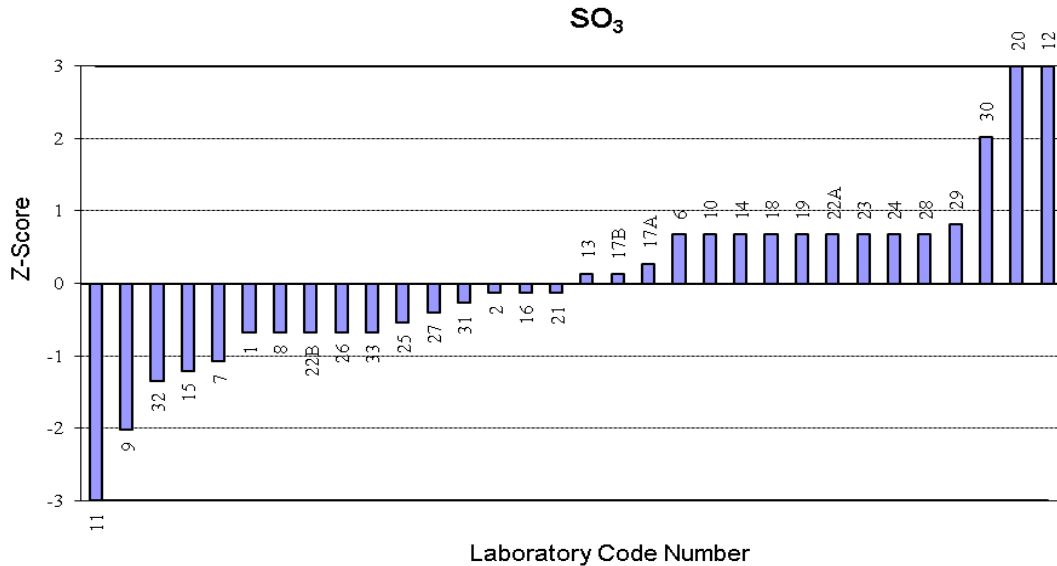


SO₃

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	2.6	-0.67		XRF
2	2.64	-0.13	0.16	MSEN196-2:2007
6	2.7	0.67	0.1	MSEN196-2:2007
7	2.57	-1.08	0.14	AS2350.2:2006
8	2.6	-0.67	0.09	
9	2.5	-2.02	0.09	MS EN 196-2
10	2.7	0.67		AS2350.2
11	2.246	-5.45 §	0.2999	EN-196-2
12	3.1	6.07 §	0.05	EN-196-2:2/2005
13	2.66	0.13		ASTM C114 ISRI 1692
14	2.7	0.67		
15	2.56	-1.21	0.136	ASTM C114
16	2.64	-0.13	0.02	ISIRI 1692
17A	2.67	0.27		ISIRI 1692
17B	2.66	0.13		ISIRI 1692
18	2.7	0.67	0.1	ISIRI 1692
19	2.70	0.67		ISIRI 1692
20	2.95	4.05 §		ISIRI 1692
21	2.64	-0.13	0.024	ASTM C114 (ISIRI 1692)
22A	2.7	0.67	0.050	CNS1078
22B	2.6	-0.67	0.083	ASTM C114
23	2.7	0.67	0.2	xf
24	2.7	0.67		MSEN196-2:2007
25	2.61	-0.54	0.16	EN196:Part-2:2007
26	2.6	-0.67	0.07	MSEN196-2:2007 Clause 8
27	2.62	-0.40	0.27	XRF AS2350.2
28	2.7	0.67	0.02	ASTM C114-2010 ISIRI 1692
29	2.71	0.81		ASTM C114
30	2.8	2.02		XRF
31	2.63	-0.27	0.02	ISIRI 1692 ASTM C114
32	2.55	-1.35	0.1	ISIRI 1692 BSEN 196.2
33	2.6	-0.67		BSEN 196-2:2005 Clause 8.0

No. Results 32
 Median 2.65
 Normalised IQR 0.07
 Uncertainty (Median) 0.02
 Robust CV 2.8%
 Minimum 2.246
 Maximum 3.1
 Range 0.854

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



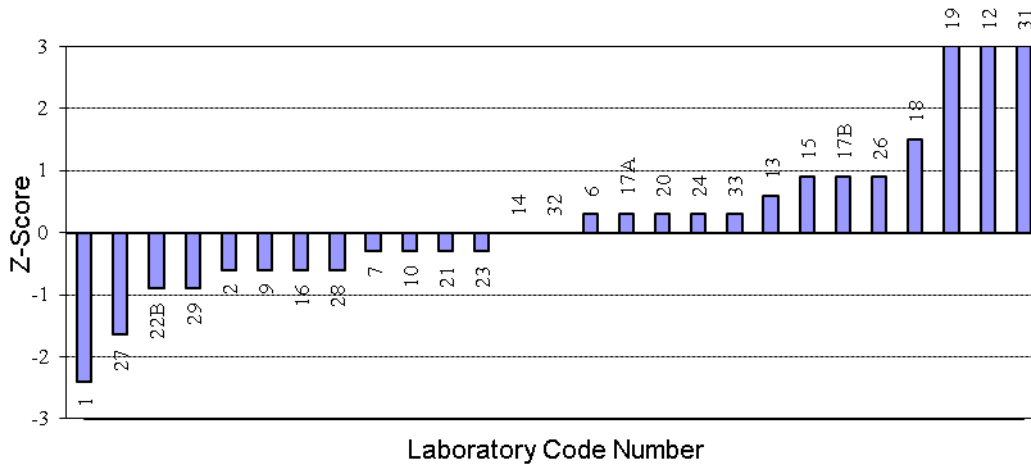
Na₂O

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	0.06	-2.40		XRF
2	0.12	-0.60	0.04	MSEN196-2:2007
6	0.15	0.30	0.06	MSEN196-2:2007
7	0.13	-0.30	0.04	AS2350.2:2006
9	0.12	-0.60	0.04	MS EN 196-2
10	0.13	-0.30		AS2350.2
12	0.40	7.79 §		EN-196-2:2/2005
13	0.16	0.60		EN 196-2 ISIRI 1695
14	0.14	0.00		
15	0.17	0.90	0.015	ASTM C114
16	0.12	-0.60	0.02	ISIRI 1695
17A	0.15	0.30		ISIRI 1695
17B	0.17	0.90		ISIRI 1695
18	0.19	1.50	0.05	ISIRI 1695
19	0.24	3.00 §		ISIRI 1695
20	0.15	0.30		ISIRI 1695
21	0.13	-0.30	0.01	ASTM C114 (ISIRI 1695)
22B	0.11	-0.90	0.023	ASTM C114
23	0.13	-0.30	0.05	xrf
24	0.15	0.30		MSEN196-2:2007
26	0.17	0.90	0.06	MSEN196-2:2007 Clause 17
27	0.085	-1.65	1.30	XRF AS2350.2
28	0.12	-0.60	0.05	ASTM C114-2010 ISIRI 1695
29	0.11	-0.90		ASTM C114
31	0.41	8.09 §	0.01	ISRI 1695 ASTM C114
32	0.14	0.00	0.01	ISIRI 1692 BSEN 196.2
33	0.15	0.30		RHP - 13

No. Results 27
 Median 0.140
 Normalised IQR 0.033
 Uncertainty (Median) 0.008
 Robust CV 23.8%
 Minimum 0.06
 Maximum 0.41
 Range 0.35

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

Na₂O



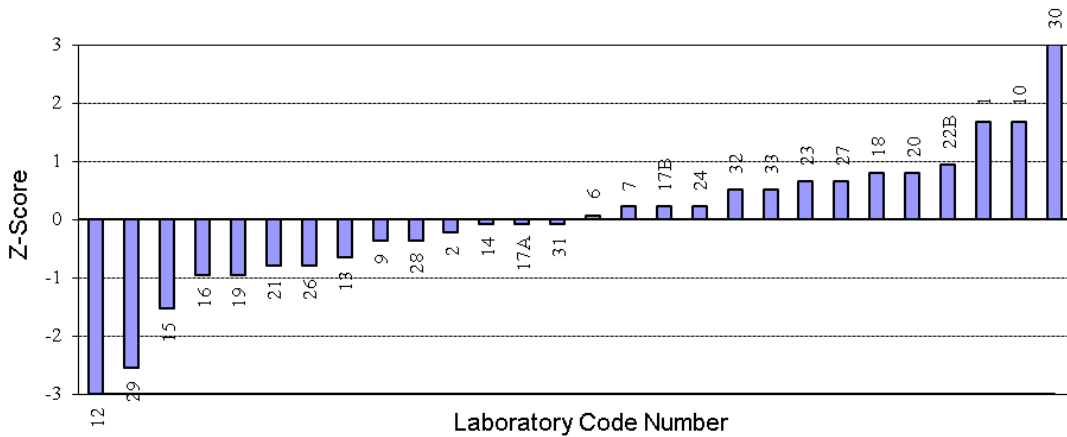
K₂O

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	0.76	1.68		XRF
2	0.63	-0.22	0.06	MSEN196-2:2007
6	0.65	0.07	0.12	MSEN196-2:2007
7	0.66	0.22	0.06	AS2350.2:2006
9	0.62	-0.36	0.01	MS EN 196-2
10	0.76	1.68		AS2350.2
12	0.20	-6.49 §		EN-196-2:2/2005
13	0.60	-0.66		EN 196-2 ISIRI 1695
14	0.64	-0.07		
15	0.54	-1.53	0.013	ASTM C114
16	0.58	-0.95	0.02	ISIRI 1695
17A	0.64	-0.07		ISIRI 1695
17B	0.66	0.22		ISIRI 1695
18	0.70	0.80	0.05	ISIRI 1695
19	0.58	-0.95		ISIRI 1695
20	0.7	0.80		ISIRI 1695
21	0.59	-0.80	0.01	ASTM C114 (ISIRI 1695)
22B	0.71	0.95	0.035	ASTM C114
23	0.69	0.66	0.05	xrf
24	0.66	0.22		MSEN196-2:2007
26	0.59	-0.80	0.04	MSEN196-2:2007 Clause 17
27	0.69	0.66	0.19	XRF AS2350.2
28	0.62	-0.36	0.05	ASTM C114-2010 ISIRI 1695
29	0.47	-2.55		ASTM C114
30	0.90	3.72 §		XRF
31	0.64	-0.07	0.02	ISRI 1695 ASTM C114
32	0.68	0.51	0.02	ISIRI 1692 BSEN 196.2
33	0.68	0.51		RHP - 13

No. Results 28
 Median 0.645
 Normalised IQR 0.069
 Uncertainty (Median) 0.016
 Robust CV 10.6%
 Minimum 0.20
 Maximum 0.90
 Range 0.70

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

K₂O

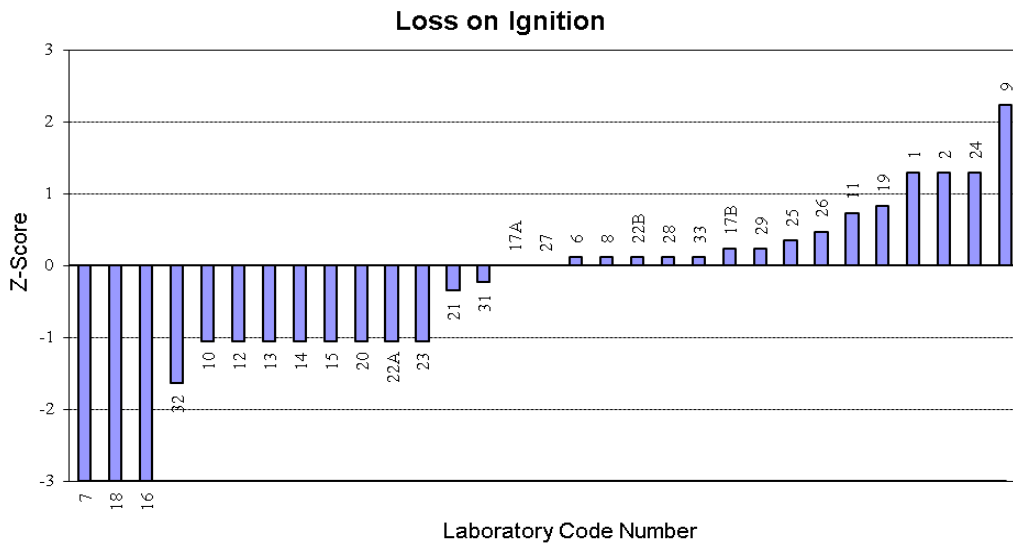


Loss on Ignition

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	4.0	1.29		MSEN196-2:2007
2	4.00	1.29	0.16	MSEN196-2:2007
6	3.9	0.12	0.5	MSEN196-2:2007
7	3.49	-4.69 §	0.16	AS2350.2:2006
8	3.9	0.12	0.07	
9	4.08	2.23	0.05	MS EN 196-2
10	3.8	-1.06		AS2350.2
11	3.952	0.73	0.0837	EN-196-2
12	3.8	-1.06	0.06	EN-196-2:2/2005
13	3.80	-1.06		ASTM C114 ISRI 1692
14	3.8	-1.06		
15	3.8	-1.06	0.136	ASTM C114
16	3.61	-3.28 §	0.38	ISIRI 1692
17A	3.89	0.00		ISIRI 1692
17B	3.91	0.23		ISIRI 1692
18	3.6	-3.40 §	0.1	ISIRI 1692
19	3.96	0.82		ISIRI 1692
20	3.8	-1.06		ISIRI 1692
21	3.86	-0.35	0.024	ASTM C114 (ISIRI 1692)
22A	3.8	-1.06	0.086	CNS1078
22B	3.9	0.12	0.064	ASTM C114
23	3.8	-1.06	0.2	
24	4.0	1.29		MSEN196-2:2007
25	3.92	0.35	0.06	EN196:Part-2:2007
26	3.93	0.47	0.14	MSEN196-2:2007 Clause 2
27	3.89	0.00	2.35	AS2350.2
28	3.9	0.12	0.018	ASTM C114 ISIRI 1692
29	3.91	0.23		ASTM C114
31	3.87	-0.23	0.02	ISRI 1692 ASTM C114
32	3.75	-1.64	0.12	ISIRI 1692 BSEN 196.2
33	3.90	0.12		BSEN 196-2:2005 Clause 17.0

No. Results 31
 Median 3.89
 Normalised IQR 0.09
 Uncertainty (Median) 0.02
 Robust CV 2.2%
 Minimum 3.49
 Maximum 4.08
 Range 0.59

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



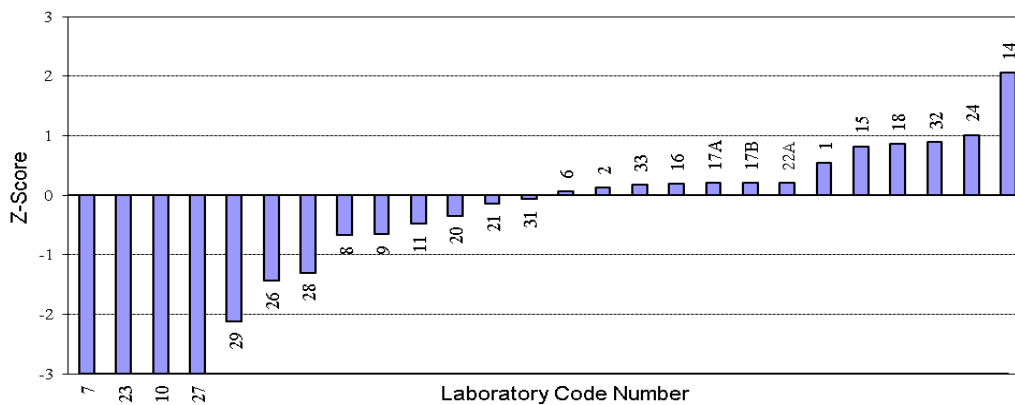
Specific Surface Area (Blaine)

Lab Code	Sample A (cm ² /g)	Z-Score	MU	Method/Technique
1	3970	0.55		MSEN196-6:2007
2	3910	0.14	7.0	MSEN196-6:2007
6	3900	0.07	130	MSEN196-6:2007
7	373	-24.11 §	28	AS2350.8:2006
8	3793	-0.67	1.7	
9	3795	-0.65	159	MS EN 196-6
10	395	-23.96 §		AS2350.8
11	3820	-0.48	87	EN-196-6
14	4190	2.06		RD:3.14
15	4010	0.82	80.08	ASTM C204-92
16	3918	0.19	79	ISIRI 390
17A	3920	0.21		ISIRI 390
17B	3920	0.21		ISIRI 390
18	4015	0.86	60	ISIRI 390
20	3840	-0.34		ISIRI 390
21	3870	-0.14	56	ASTM C204 (ISIRI 390)
22A	3920	0.21	80	CNS2924
23	385	-24.03 §	5	Blaine
24	4038	1.01		MSEN196-6:2007
26	3680	-1.44	62.88	MSEN196-6:2007 Clause 4
27	400	-23.93 §	0.25	AS2350.8
28	3700	-1.30	65	ASTM C204 -2007 ISIRI 390
29	3582	-2.11		C204
31	3880	-0.07	77	ISRI 390 ASTM C204
32	4020	0.89	127	ISIRI 390 BSEN 196.2
33	3915	0.17		BSEN 196-6:1992

No. Results 26
 Median 3890
 Normalised IQR 146
 Uncertainty (Median) 36
 Robust CV 3.7%
 Minimum 373
 Maximum 4190
 Range 3817

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

Specific Surface Area (Blaine)

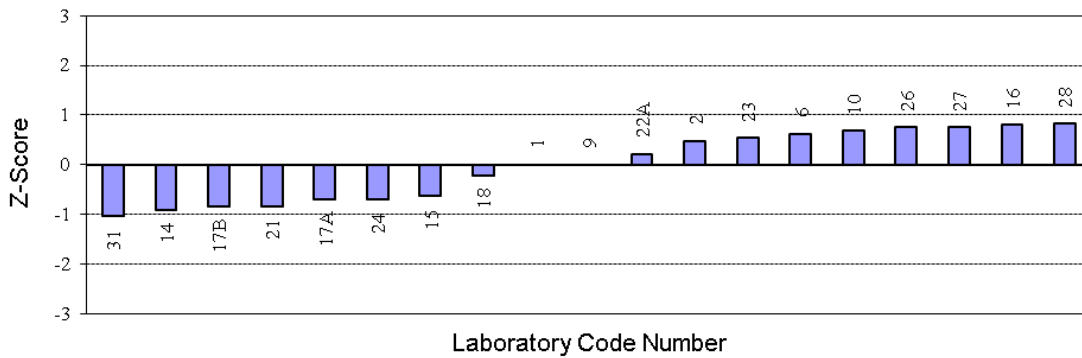


Fineness by the 45 µm Sieve

Lab Code	Sample A (%)	Z-Score	MU	Method/Technique
1	5.7	0.00		MSEN196-6:2007
2	6.4	0.48		MSEN196-6:2007
6	6.6	0.62	0.9	MSEN196-6:2007
9	5.7	0.00	1.22	MS EN 196-2
10	6.7	0.69		AS2350.9
14	4.4	-0.90		
15	4.8	-0.62		
16	6.87	0.81		
17A	4.7	-0.69		ASTM
17B	4.5	-0.83		ASTM
18	5.4	-0.21		ISIRI 1599
21	4.5	-0.83	0.1	In house method (LA-W-12-01/02))
22A	6.0	0.21		CNS11273
23	6.5	0.55	0.5	w et sieve
24	4.7	-0.69		internal procedure
26	6.8	0.76		MSEN196-6:2007 National Annex NA (informative)
27	6.8	0.76	3.94	AS2350.9
28	6.9	0.83	0.016	ASTM C430-96 (Reapproved 2003)
31	4.2	-1.04	1.3	ISRI 390 ASTM C204

No. Results	19
Median	5.70
Normalised IQR	1.45
Uncertainty (Median)	0.42
Robust CV	25.4%
Minimum	4.2
Maximum	6.9
Range	2.7

Fineness by the 45 µm Sieve



Median Particle Size

Lab Code	Sample A (μm)	MU	Method/Technique
1	15.9	0.5	PSD LASER GRANULAOMETRY
6	13.1		Using Malvern Mastersizer 2000 (Dry Method)
9	6.7		PSA (CILAS)
22A	13.5	1.0	Coalter LS 100Q
23	14.0		laser

No. Results 5

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
1	64.4	0.70	PSD LASER GRANULAOMETRY
6	66.0		Using Malvern Mastersizer 2000 (Dry Method)
9	53.9		PSA (CILAS)
22A	73.0	2.00	Coalter LS 100Q
23	66.9		laser

No. Results 5

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, labelled PTA Sample A.

For this program, 10 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 below.

TABLE C: HOMOGENEITY TESTING RESULTS

Test	LOI	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃	Na ₂ O
	%	%	%	%	%	%	%	%
<i>Sample 1</i>	3.8	63.8	20.1	4.0	3.0	1.5	2.64	0.13
<i>Sample 2</i>	3.9	63.8	20.1	4.0	3.0	1.5	2.66	0.13
<i>Sample 3</i>	3.9	63.8	20.0	4.0	3.0	1.5	2.63	0.13
<i>Sample 4</i>	3.8	63.8	20.1	4.0	3.0	1.5	2.66	0.13
<i>Sample 5</i>	3.8	63.9	20.1	4.0	3.0	1.5	2.63	0.13
<i>Sample 6</i>	3.9	63.8	20.1	4.0	3.0	1.5	2.64	0.12
	3.8	63.8	20.0	4.0	3.0	1.5	2.65	0.13
<i>Sample 8</i>	3.8	63.8	20.1	4.0	3.0	1.5	2.65	0.14
<i>Sample 9</i>	3.8	63.8	20.1	4.0	3.0	1.5	2.66	0.14
<i>Sample 10</i>	3.8	63.8	20.1	4.0	3.0	1.5	2.64	0.12
<u>Median</u>	3.8	63.8	20.1	4.0	3.0	1.5	2.65	0.13
<u>Norm IQR</u>	0.022	0.015	0.022	0.015	0.007	0.007	0.015	0.000
<u>Robust CV</u>	0.6%	0.0%	0.1%	0.4%	0.2%	0.5%	0.6%	0.0%

APPENDIX C

Documentation

PROFICIENCY TESTING AUSTRALIA

Proficiency Testing Program Cement (Round 5)

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% limestone mineral additive).

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
 - (ii) Loss on Ignition (970 °C)
 - (iii) Specific Surface Area (Blaine): Relative Density = 3.14 g/cm^3
 - (iv) Fineness by 45 μm sieve (wet)
 - (v) Laser Granulometry: Report median particle size (μm) & 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. AS2350.8 for 45 μm . 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 $k = 2$) and reported as a %. 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 **20 September 2013** 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 5) - Proficiency Testing Program
Results Sheet

Lab Code: « »

Test	Result	±MU	Method
SiO₂ (Report to 0.1%)			
Al₂O₃ (Report to 0.1%)			
Fe₂O₃ (Report to 0.1%)			
CaO (Report to 0.1%)			
MgO (Report to 0.1%)			
SO₃ (Report to 0.1%)			
Na₂O (Report to 0.01%)			
K₂O (Report to 0.01%)			
Loss on ignition (970°C) (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 -