



Report No. 888

Coal Proficiency Testing

Round 32

December 2014

Acknowledgments

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1. FOREWORD

This report summarises the results of a proficiency testing program on the chemical analysis of coal. It constitutes the thirty-second round of an ongoing series of programs.

The program was conducted in October 2014 by Proficiency Testing Australia (PTA). The aim of the program was to assess laboratories' abilities to competently perform the prescribed analyses.

The Program Coordinator was Ms L Galbraith and the Technical Advisers were Mr J Kelly of BHP Billiton Mitsubishi Alliance, QLD and Mr M Preston of ALS-ACTest Gladstone, QLD. This report was authorised by Mrs F Watton, PTA Quality - Business Development Manager.

2. FEATURES OF THE PROGRAM

- (a) Participants were provided with two 125g samples of coal labelled PTA Sample A and PTA Sample B.
- (b) A total of 27 laboratories received samples, comprising:
 - 22 Australian participants; and
 - 5 overseas participants, including:
 - 2 x Philippines;
 - Spain;
 - United Kingdom; and
 - Hong Kong.

Of these 27 laboratories, one was unable to submit results by the due date.

- (c) Laboratories were provided with the *Instructions to Participants* and *Results Sheet* (see Appendix C). Laboratories were requested to perform the tests according to their routine methods and to record their results on the *Results Sheet*.
- (d) Prior to sample distribution, a number of randomly selected samples were analysed for homogeneity. Based on the results of this testing (see Appendix B), the homogeneity of the samples was established.
- (e) 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. Please note that a number of laboratories reported more than one set of results and, therefore, their code numbers (with letter) could appear several times in the same data set.

- (f) Results (as reported by participants) with corresponding summary statistics (i.e. number of results, median, uncertainty of the median, normalised interquartile range, robust coefficient of variation, minimum, maximum and range) are presented in Appendix A (for each sample and for each of the analyses performed). Measurement Uncertainty (MU) is also presented where supplied by participants. Please note that this information is presented for information purposes only and has not been used for the formal evaluation of results.
- (g) 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.
- (h) The document entitled *Guide to Proficiency Testing Australia, 2014* (reference [1]) defines the statistical terms and details the statistical procedures referred to in this report.
- (i) A tabulated listing of laboratories (by code number) identified as having outlier results can be found on page 6.

3. FORMAT OF THE APPENDICES

- (a) Appendix A contains the analysis of results reported by laboratories for the samples. This section 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 testing.
- (c) Appendix C contains copies of the *Instructions to Participants and Results Sheet*.

4. STATISTICAL DESIGN OF THE PROGRAM

- (a) 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 “§”.

The table on page 6 summarises the outlier results detected.

(b) Results Tables and Summary Statistics

Each of these tables contains the results returned by each laboratory, including the code number for the method used, 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 decimal places) 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 uncertainty of the median, a robust estimate of the standard deviation of the Median;
- the normalised interquartile range of the results (*Normalised IQR*);
- the robust coefficient of variation, expressed as a percentage (*Robust CV*) - i.e. $100 \times \text{Normalised IQR} / \text{Median}$;
- the minimum and maximum laboratory results; and
- the range (*Maximum - Minimum*).

The median is a measure of the centre of the data.

The normalised IQR is a measure of the spread of the results. It 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).

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

(c) 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.

The following table summarises the results submitted by participants for the program.

TABLE A: SUMMARY STATISTICS

Test	PTA Sample	No. of Results	Median	Normalised IQR
Ash (0.01%)	A	26	8.153	0.069
	B	26	23.573	0.129
Volatile Matter (0.01%)	A	23	27.885	0.230
	B	23	16.350	0.250
Gross Calorific Value (0.001 MJ/kg)	A	20	32.4993	0.1162
	B	20	26.3088	0.1213
Total Sulfur (0.001%)	A	22	0.4388	0.0184
	B	22	0.3535	0.0127

Note: Statistics for each test are based on the average of the duplicate results reported for the sample. Unless specified, all other results are to a dry basis.

5. PTA AND TECHNICAL ADVISERS' COMMENTS

Of the 27 participating laboratories, one laboratory did not submit results in time for inclusion in the final report. Of the 26 laboratories who submitted results, 7 laboratories have been identified as having reported one or more outlier results. Of the 182 total results for statistical analysis, 20 outliers were reported; therefore 11.00% of the total results have been identified as outlier results.

Table B shows a comparison of past program outlier results.

TABLE B: PREVIOUS OUTLIER RESULTS

	Round 28	Round 29	Round 30	Round 31	Round 32
Total Results	414	654	170	411	182
No. of Outlier Results	11	23	17	28	20
% of Total	2.66%	3.52%	10.00%	6.81%	11.00%

The number of laboratories reporting MU for round 32 is similar to previous rounds. Refer to Table E for Standards Australia Precision Data.

The test moisture has been excluded from z-score analysis. As the moisture result is very much dependent upon the environment of the laboratory atmosphere at time of testing, it is important that it is reported by participants. This may also assist the laboratory to understand why they may have reported an outlier for another parameter.

Table C shows a comparison of past program Robust CV (%) results.

TABLE C: COMPARISON OF ROBUST CVS (%)

Test	Round 30		Round 31		Round 32	
	PTA Sample A	PTA Sample B	PTA Sample A	PTA Sample B	PTA Sample A	PTA Sample B
Ash (0.01%)	1.0	1.2	0.6	0.5	0.8	0.5
Volatile Matter (0.01%)	0.8	1.3	1.5	0.7	0.8	1.5
Gross Calorific Value (0.001 MJ/kg)	0.3	0.5	0.3	0.4	0.4	0.5
Total Sulfur (0.001%)	4.7	4.0	4.3	3.2	4.2	3.6

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.

Samples are selected which are of a coal type that is relatively stable and available. Samples are within a range of test parameters that are suitable for the program. Coal samples were prepared according to AS 4264.1. No certified reference materials were used.

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 tables in Appendix A.

Analysis of Results by Method Groups

Grouped analysis was performed for methods appearing greater than or equal to 11 times in each test. The only test to return greater than or equal to 11 of the same method was AS 1038.6.3.3. PTA Sample A returned 14 results with a Median of 0.442% and an Uncertainty of the Median of 0.003%. PTA Sample B returned 14 results, a Median of 0.354% and an Uncertainty of the Median of 0.002%.

No other methods were so prevalent that reliable conclusions could be drawn from analysing grouped methods.

6. OUTLIER RESULTS

Laboratories reporting outlier results are listed in the following table:

TABLE D: SUMMARY OF STATISTICAL OUTLIERS

Test	PTA Sample	Laboratory Code
Ash (0.01%)	A	36
	B	14, 36
Volatile Matter (0.01%)	A	14, 38, 42, 54
	B	38
Gross Calorific Value (0.001 MJ/kg)	A	42
	B	9
Total Sulfur (0.001%)	A	14, 38, 42, 49
	B	14, 36, 38, 42, 49, 54

7. REFERENCES

- [1] *Guide to Proficiency Testing Australia*, 2014 (This document can be found on the PTA website, www.pta.asn.au)
- [2] *AS 4264.1 - 2009 Coal and coke – Sampling – Coal – Sampling Procedures.*
- [3] *AS 1038.3 - 2000 Coal and coke – Analysis and testing – Proximate analysis of higher rank coal.*
- [4] *AS 1038.6.3.3 - 1997 Coal and coke – Analysis and testing – Higher rank coal – Ultimate analysis – Total sulfur – Infrared method.*

TABLE E: STANDARDS AUSTRALIA PRECISION DATA

Standard	Test	Repeatability	Reproducibility
AS 1038.3 (2000)	Ash	0.15%	0.25%
AS 1038.3 (2000)	Volatile Matter	0.2%	1.0%
AS 1038.5 (1995)	Gross Calorific Value	0.13 MJ/kg	0.30 MJ/kg
AS 1038.6.3.2 (2003)	Total Sulfur	0.03%	0.08%
AS 1038.6.3.3 (1997)	Total Sulfur	0.03%	0.05%

TABLE F: STANDARDS ISO PRECISION DATA

Standard	Test	Repeatability	Reproducibility
ISO 1171 (2010)	Ash	< 10% 0.2% >10 % 2% of mean	<10% 0.3% >10% 3% of mean
ISO 562 (2010)	Volatile Matter	<10% 0.3% >10% 3% of mean	<10% 0.5% >10% 4% of mean
ISO 1928 (2009)	Gross Calorific Value	120 J/g	300 J/g
ISO 19579 (2006)	Total Sulfur	0.02 +0.03 mean	0.02 +0.09 mean

APPENDIX A

Results and Data Analysis

Moisture (air-dry basis) (0.01%).....	A1
Ash (0.01%).....	A3
Volatile Matter (0.01%).....	A7
Gross Calorific Value (0.001 MJ/kg).....	A11
Total Sulfur (0.001%).....	A15

Moisture (air-dry basis) PTA Sample A (0.01%)						
Laboratory code	Result 1	Result 2	Average	MU		Method/Technique
1	2.09	2.04	2.07	±	0.1	AS 1038.3
4	1.77	1.74	1.76	±	0.10	AS 1038.3
5	2.06	2.11	2.09	±	0.1	AS 1038.3
8	1.71	1.67	1.69		#	AS 1038.3
9	1.56	1.57	1.57	±	0.1	In house method
11	1.72	1.74	1.73	±	0.10	AS 1038.3
14	1.88	1.79	1.84		#	a.SL25, b.TGA
18A	1.30	1.28	1.29	±	0.10	ISO 11722
18B	1.55	1.48	1.52	±	0.10	ISO 11722
18C	0.71	0.69	0.70	±	0.10	ISO 11722
18D	1.71	1.74	1.73	±	0.10	ISO 11722
23	1.56	1.55	1.56	±	3.72% relative uncertainty	AS 1038.3 - 2000
25	1.37	1.24	1.31		#	ASTM D3173
29	2.31	2.26	2.29	±	0.31	AS 1038.3.2
30	2.16	2.15	2.16		#	AS 1038.3
33	2.58	2.57	2.58		#	ISO 17246
34	1.40	1.40	1.40	±	0.10	ISO 17246
36	1.47	1.49	1.48	±	0.01	ASTM D3173
38	0.29	0.46	0.38	±	0.89	ISO 589
42	0.50	0.54	0.52		#	DETSC 5005/ BS1016-104.1
44	2.02	2.00	2.01	±	0.10	ISO 17246 - 2010
49	1.27	1.22	1.25	±	0.10	ISO 17246
50	1.63	1.67	1.65		n/a	AS 1038.3
54	1.54	1.56	1.55	±	0.05	a.ISO 11722:2013(E) part 5
56	1.83	1.84	1.84		n/a	AS 1038
60	1.92	1.90	1.91	±	0.1	AS 1038.3

No of Results:	26
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Note: Moisture results are required purely as a correctional value, to enable calculation of other results to dry basis.

A “#” indicates no response was provided.

Moisture (air-dry basis) PTA Sample B (0.01%)						
Laboratory code	Result 1	Result 2	Average	MU		Method/Technique
1	1.45	1.44	1.45	±	0.1	AS 1038.3
4	1.32	1.37	1.35	±	0.10	AS 1038.3
5	1.75	1.76	1.76	±	0.1	AS 1038.3
8	1.44	1.46	1.45		#	AS 1038.3
9	1.92	1.90	1.91	±	0.1	In house method
11	1.17	1.17	1.17	±	0.10	AS 1038.3
14	1.88	1.77	1.83		#	a.SL25, b.TGA
18A	0.96	1.03	1.00	±	0.10	ISO 11722
18B	1.22	1.17	1.20	±	0.10	ISO 11722
18C	0.85	0.82	0.84	±	0.10	ISO 11722
18D	2.02	1.95	1.99	±	0.10	ISO 11722
23	1.25	1.21	1.23	±	3.72% relative uncertainty	AS 1038.3 - 2000
25	1.55	1.49	1.52		#	ASTM D3173
29	0.95	0.92	0.94	±	0.25	AS 1038.3.2
30	1.45	1.41	1.43		#	AS 1038.3
33	1.87	1.86	1.87		#	ISO 17246
34	1.24	1.21	1.23	±	0.10	ISO 17246
36	1.69	1.67	1.68	±	0.01	ASTM D3173
38	0.93	1.06	1.00	±	0.89	ISO 589
42	0.47	0.50	0.49		#	DETSC 5005/ BS1016-104.1
44	1.68	1.61	1.65	±	0.10	ISO 17246 - 2010
49	1.02	1.01	1.02	±	0.10	ISO 17246
50	1.08	1.06	1.07		n/a	AS 1038.3
54	1.76	1.70	1.73	±	0.05	a.ISO 11722:2013(E) part 5
56	1.51	1.47	1.49		n/a	AS 1038
60	1.58	1.57	1.58	±	0.1	AS 1038.3

No of Results:	26
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Note: Moisture results are required purely as a correctional value, to enable calculation of other results to dry basis.

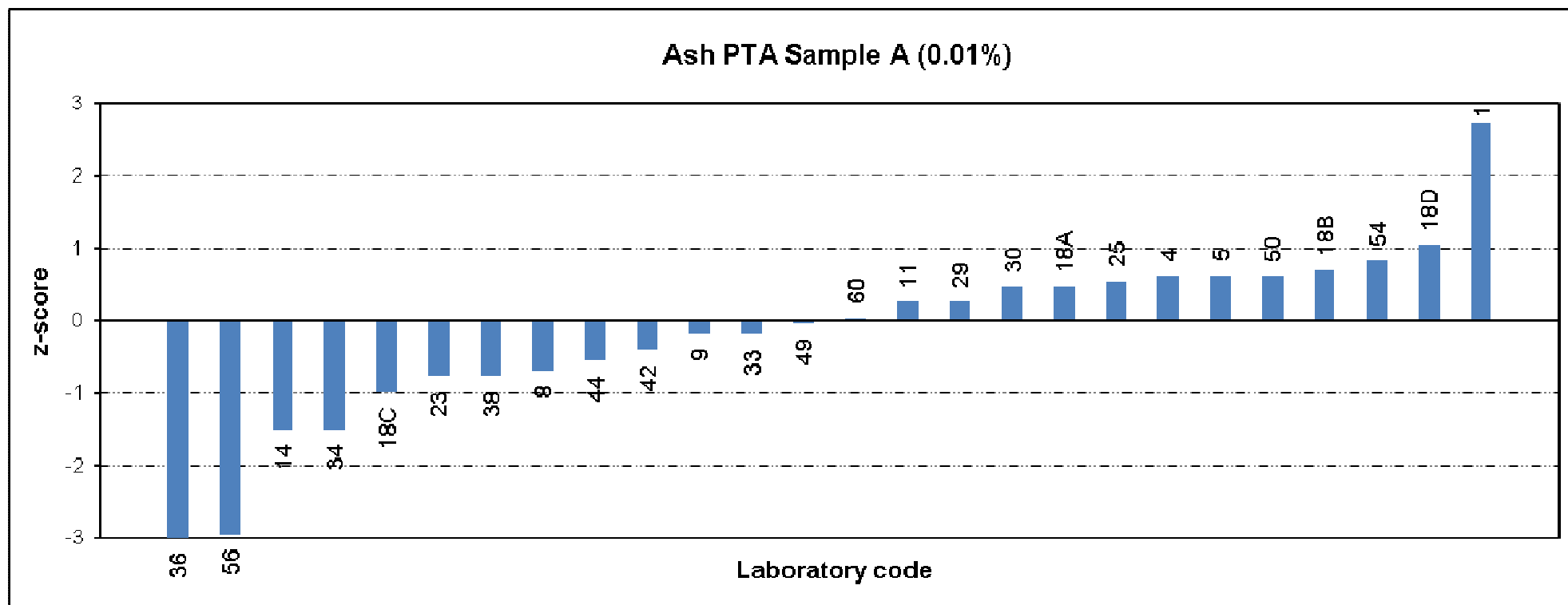
A “#” indicates no response was provided.

Ash PTA Sample A (0.01%)						
Laboratory code	Result 1	Result 2	Average	MU	Robust z-score	Method/Technique
1	8.34	8.34	8.34	± 0.1	2.73	AS 1038.3
4	8.22	8.17	8.20	± 0.10	0.62	AS 1038.3
5	8.23	8.16	8.20	± 0.15	0.62	AS 1038.3
8	8.12	8.09	8.11	#	-0.69	AS 1038.3
9	8.15	8.13	8.14	± 0.1	-0.18	In house method
11	8.19	8.15	8.17	± 0.10	0.26	AS 1038.3
14	8.10	8.00	8.05	#	-1.49	a.SL25, b.TGA
18A	8.18	8.19	8.19	± 0.2	0.47	ISO 1171
18B	8.19	8.21	8.20	± 0.2	0.69	ISO 1171
18C	8.13	8.04	8.09	± 0.2	-0.98	ISO 1171
18D	8.21	8.24	8.23	± 0.2	1.06	ISO 1171
23	8.08	8.12	8.10	± 0.57% relative uncertainty	-0.77	AS 1038.3 - 2000
25	8.20	8.18	8.19	#	0.55	ASTM D3174
29	8.15	8.19	8.17	± 0.15	0.26	AS 1038.3.3
30	8.18	8.19	8.19	#	0.47	AS 1038.3
33	8.13	8.15	8.14	± 0.3	-0.18	ISO 17246
34	8.02	8.08	8.05	± 0.20	-1.49	ISO 17246
36	7.58	7.64	7.61	± 0.03	-7.91	§ ASTM D3174
38	8.1	8.1	8.10	± 0.2	-0.77	ISO 1171
42	8.13	8.12	8.13	± 4.80%	-0.40	DETSC 5001/ BS 1016-104.4
44	8.12	8.11	8.12	± 0.20	-0.55	ISO 17246 - 2010
49	8.17	8.13	8.15	± 0.20	-0.04	ISO 17246
50	8.18	8.21	8.20	± 0.15	0.62	AS 1038.3
54	8.21	8.21	8.21	± 0.05	0.84	a.ISO 1171:2010(E) part 8
56	7.9	8.0	7.95	± 0.60%	-2.95	AS 1038
60	8.15	8.16	8.16	± 0.1	0.04	AS 1038.3

No of Results:	26
Median:	8.153
Uncertainty (median)	0.017
Normalised IQR:	0.069
Robust CV:	0.8%
Minimum:	7.61
Maximum:	8.34
Range:	0.73

Note:

A "§" indicates an outlier i.e. |z-score| ≥ 3.0.
A "#" indicates no response was provided.

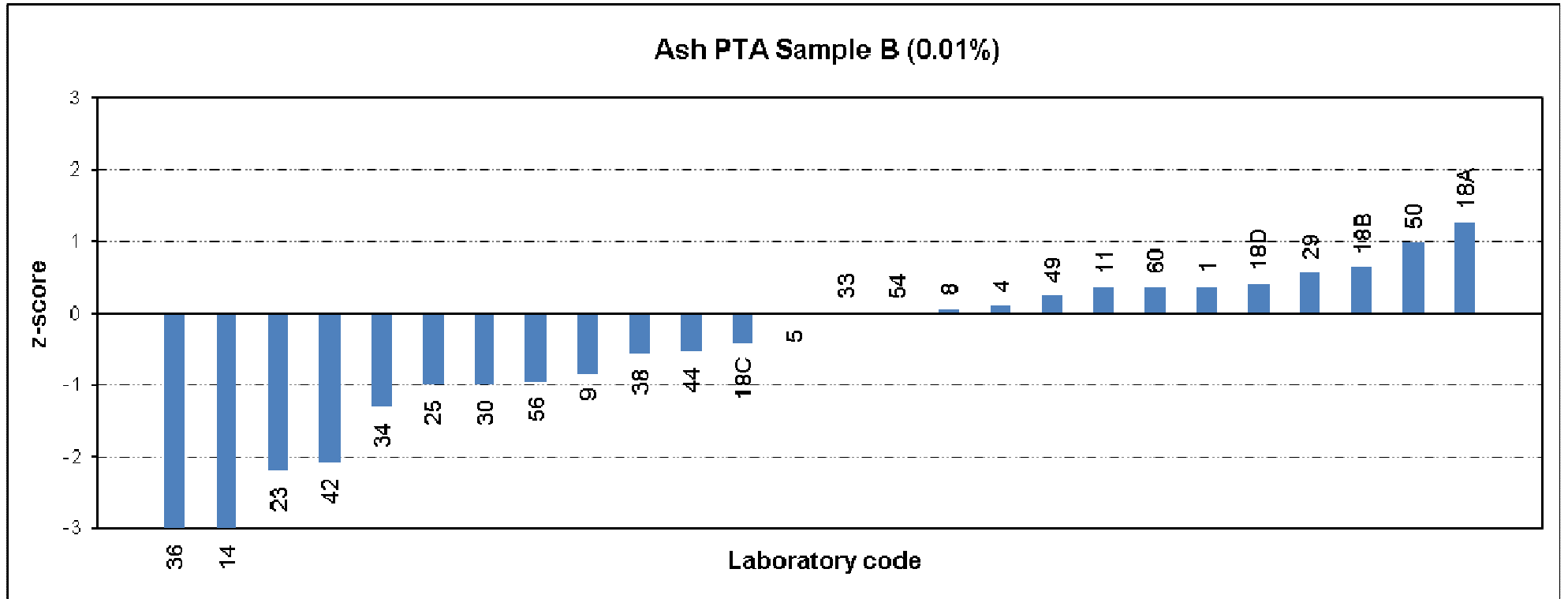


Ash PTA Sample B (0.01%)						
Laboratory code	Result 1	Result 2	Average	MU	Robust z-score	Method/Technique
1	23.60	23.64	23.62	± 0.1	0.37	AS 1038.3
4	23.58	23.59	23.59	± 0.15	0.10	AS 1038.3
5	23.63	23.51	23.57	± 0.25	-0.02	AS 1038.3
8	23.58	23.58	23.58	#	0.06	AS 1038.3
9	23.47	23.46	23.47	± 0.1	-0.83	In house method
11	23.68	23.56	23.62	± 0.15	0.37	AS 1038.3
14	23.00	23.20	23.10	#	-3.67	§ a.SL25, b.TGA
18A	23.74	23.73	23.74	± 2.0% of mean	1.26	ISO 1171
18B	23.66	23.65	23.66	± 2.0% of mean	0.64	ISO 1171
18C	23.53	23.51	23.52	± 2.0% of mean	-0.41	ISO 1171
18D	23.65	23.60	23.63	± 2.0% of mean	0.41	ISO 1171
23	23.33	23.25	23.29	± 1.86% relative uncertainty	-2.19	AS 1038.3 - 2000
25	23.45	23.44	23.45	#	-0.99	ASTM D3174
29	23.64	23.65	23.65	± 0.25	0.56	AS 1038.3.3
30	23.41	23.48	23.45	#	-0.99	AS 1038.3
33	23.63	23.52	23.58	± 3% of mean	0.02	ISO 17246
34	23.43	23.38	23.41	± 0.20	-1.30	ISO 17246
36	22.89	22.93	22.91	± 0.03	-5.14	§ ASTM D3174
38	23.4	23.6	23.50	± 0.5	-0.56	ISO 1171
42	23.38	23.23	23.31	± 4.80%	-2.08	DETSC 5001/ BS 1016-104.4
44	23.50	23.51	23.51	± 0.20	-0.52	ISO 17246 - 2010
49	23.61	23.60	23.61	± 0.20	0.25	ISO 17246
50	23.71	23.69	23.70	± 0.25	0.99	AS 1038.3
54	23.55	23.60	23.58	± 0.05	0.02	a.ISO 1171:2010(E) part 8
56	23.5	23.4	23.45	± 0.60%	-0.95	AS 1038
60	23.59	23.65	23.62	± 0.1	0.37	AS 1038.3

No of Results:	26
Median:	23.573
Uncertainty (median)	0.032
Normalised IQR:	0.129
Robust CV:	0.5%
Minimum:	22.91
Maximum:	23.74
Range:	0.82

Note:

A "§" indicates an outlier i.e. |z-score| ≥ 3.0.
A "#" indicates no response was provided.



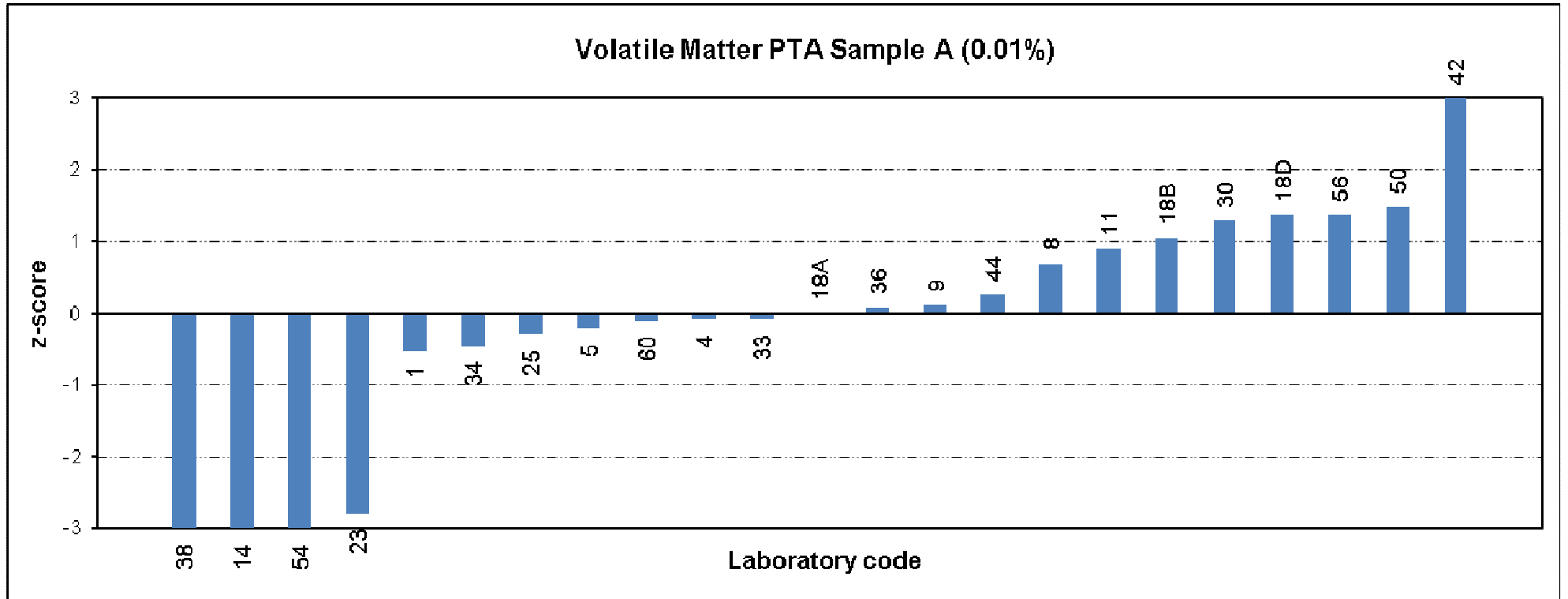
Volatile Matter PTA Sample A (0.01%)							
Laboratory code	Result 1	Result 2	Average	MU	Robust z-score	Method/Technique	
1	27.70	27.83	27.77	± 0.2	-0.52	AS 1038.3	
4	27.89	27.84	27.87	± 0.20	-0.09	AS 1038.3	
5	27.90	27.77	27.84	± 0.2	-0.22	AS 1038.3	
8	28.02	28.06	28.04	#	0.67	AS 1038.3	
9	27.98	27.84	27.91	± 0.2	0.11	AS 1038.3.4	
11	28.10	28.08	28.09	± 0.20	0.89	AS 1038.3	
14	26.40	26.30	26.35	#	-6.68	§ a.SL25, b.TGA	
18A	27.98	27.79	27.89	± 3.0% of mean	0.00	ISO 562	
18B	28.12	28.13	28.13	± 3.0% of mean	1.04	ISO 562	
18D	28.11	28.29	28.20	± 3.0% of mean	1.37	ISO 562	
23	27.23	27.26	27.25	± 4.26% relative uncertainty	-2.78	AS 1038.3 - 2000	
25	27.72	27.91	27.82	#	-0.30	ASTM D3175	
30	28.15	28.21	28.18	#	1.28	AS 1038.3	
33	27.85	27.88	27.87	± 4% of mean	-0.09	ISO 17246	
34	27.73	27.83	27.78	± 3% result	-0.46	ISO 17246	
36	27.90	27.91	27.91	± 0.01	0.09	ASTM D3175	
38	28.60	23.10	25.85	± 0.2	-8.86	§ ASTM D3175	
42	28.66	28.78	28.72	± 5.02%	3.63	§ DETSC 5003/ BS 15402	
44	27.94	27.95	27.95	± 3% of result	0.26	ISO 17246 - 2010	
50	28.32	28.13	28.23	± 0.50	1.48	AS 1038.3	
54	26.93	26.88	26.91	± 0.11	-4.26	§ a.ISO 562:2010(E) part 7	
56	28.1	28.3	28.20	± 1.0%	1.37	AS 1038	
60	27.75	27.97	27.86	± 0.2	-0.11	AS 1038.3	

No of Results:	23
Median:	27.885
Uncertainty (median)	0.060
Normalised IQR:	0.230
Robust CV:	0.8%
Minimum:	25.85
Maximum:	28.72
Range:	2.87

Note:

A "§" indicates an outlier i.e. $|z\text{-score}| \geq 3.0$.

A "#" indicates no response was provided.

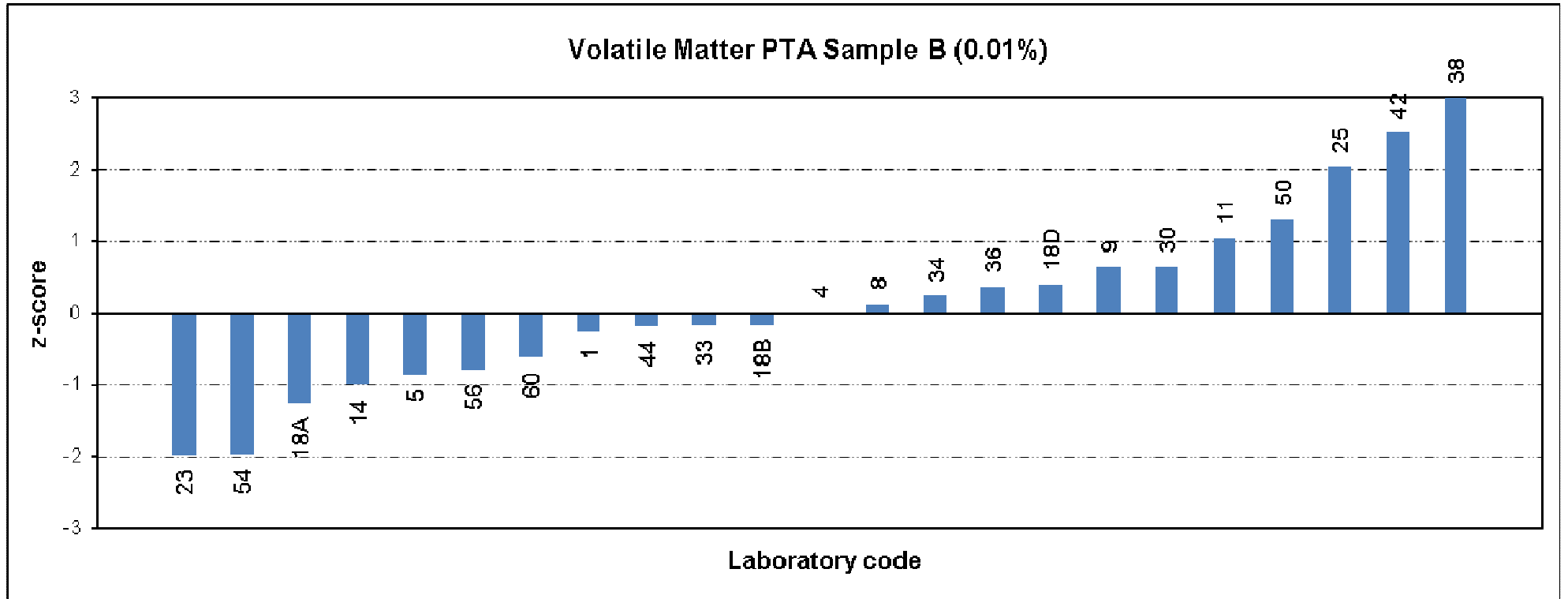


Volatile Matter PTA Sample B (0.01%)							
Laboratory code	Result 1	Result 2	Average	MU	Robust z-score	Method/Technique	
1	16.25	16.32	16.29	± 0.2	-0.26	AS 1038.3	
4	16.33	16.37	16.35	± 0.20	0.00	AS 1038.3	
5	16.12	16.15	16.14	± 0.2	-0.86	AS 1038.3	
8	16.31	16.45	16.38	#	0.12	AS 1038.3	
9	16.47	16.55	16.51	± 0.20	0.64	AS 1038.3.4	
11	16.64	16.58	16.61	± 0.20	1.04	AS 1038.3	
14	16.00	16.20	16.10	#	-1.00	a.SL25, b.TGA	
18A	16.11	15.96	16.04	± 3.0% of mean	-1.26	ISO 562	
18B	16.33	16.29	16.31	± 3.0% of mean	-0.16	ISO 562	
18D	16.50	16.40	16.45	± 3.0% of mean	0.40	ISO 562	
23	15.88	15.83	15.86	± 3.89% relative uncertainty	-1.98	AS 1038.3 - 2000	
25	16.86	16.86	16.86	#	2.04	ASTM D3175	
30	16.49	16.53	16.51	#	0.64	AS 1038.3	
33	16.31	16.31	16.31	± 4% of mean	-0.16	ISO 17246	
34	16.46	16.36	16.41	± 3% result	0.24	ISO 17246	
36	16.43	16.45	16.44	± 0.01	0.36	ASTM D3175	
38	17.30	17.10	17.20	± 0.2	3.40 §	ASTM D3175	
42	17.05	16.91	16.98	± 5.02%	2.52	DETSC 5003/ BS 15402	
44	16.27	16.34	16.31	± 3% of result	-0.18	ISO 17246 - 2010	
50	16.68	16.67	16.68	± 0.50	1.30	AS 1038.3	
54	15.82	15.90	15.86	± 0.11	-1.96	a.ISO 562:2010(E) part 7	
56	16.1	16.2	16.15	± 1.0%	-0.80	AS 1038	
60	16.13	16.26	16.20	± 0.2	-0.62	AS 1038.3	

No of Results:	23
Median:	16.350
Uncertainty (median)	0.065
Normalised IQR:	0.250
Robust CV:	1.5%
Minimum:	15.86
Maximum:	17.20
Range:	1.35

Note:A "§" indicates an outlier i.e. $|z\text{-score}| \geq 3.0$.

A "#" indicates no response was provided.

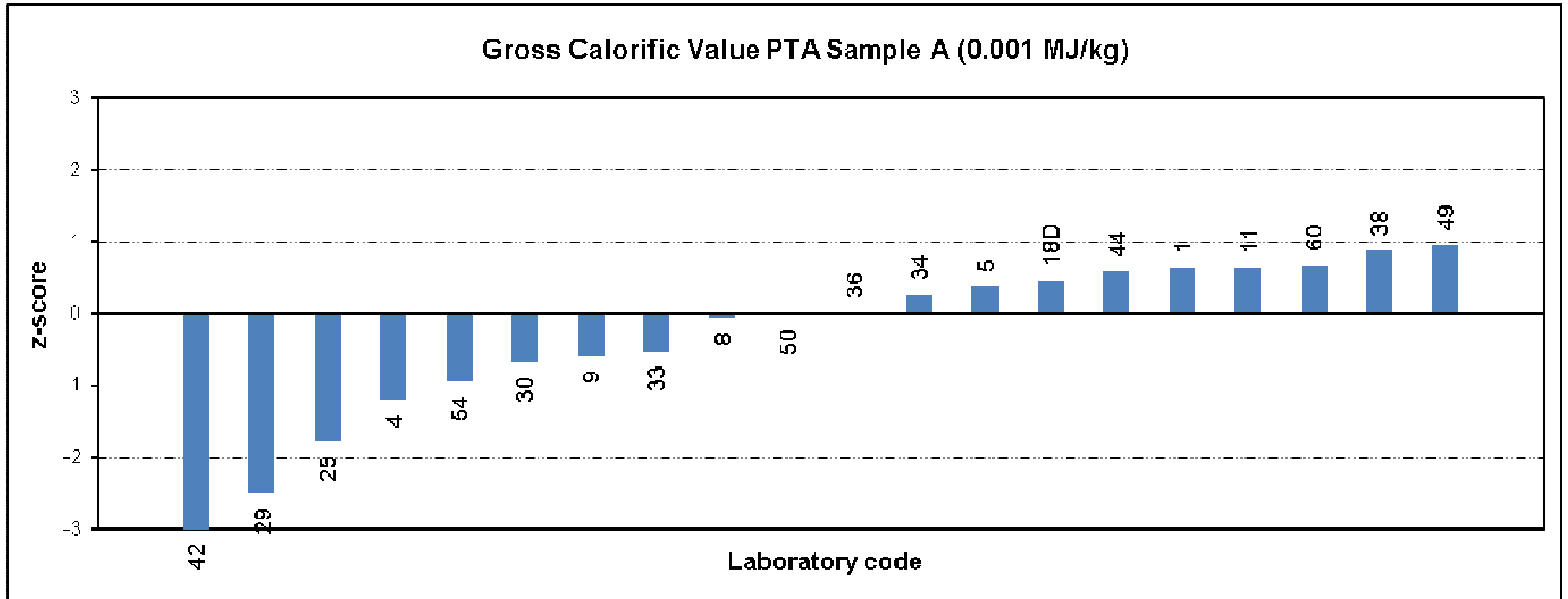


Gross Calorific Value PTA Sample A (0.001 MJ/kg)						
Laboratory code	Result 1	Result 2	Average	MU	Robust z-score	Method/Technique
1	32.584	32.563	32.574	± 0.13	0.64	AS 1038.5
4	32.374	32.343	32.359	± 0.13	-1.21	AS 1038.5
5	32.512	32.573	32.543	± 0.13	0.37	AS 1038.5
8	32.533	32.449	32.491	#	-0.07	AS 1038.5
9	32.40	32.46	32.430	± 0.13	-0.60	AS 1038.5
11	32.583	32.565	32.574	± 0.13	0.64	AS 1038.5
18D	32.574	32.532	32.553	± 0.13	0.46	AS 1038.5
25	32.310	32.276	32.293	#	-1.77	ASTM D5865
29	32.201	32.215	32.208	± 0.3	-2.51	AS 1038.5
30	32.437	32.405	32.421	#	-0.67	AS 1038.5.2
33	32.384	32.490	32.437	± 0.3	-0.54	ISO 1928
34	32.474	32.587	32.531	± 0.12	0.27	ISO 1928
36	32.522	32.481	32.502	± 0.020	0.02	ASTM D5865
38	32.587	32.617	32.602	± 0.188	0.88	ASTM D5865
42	31.966	32.108	32.037	± 2.00%	-3.98 §	DETSC 5007/ BS 15400
44	32.560	32.578	32.569	± 0.12	0.60	ISO 1928 - 2009
49	32.55	32.67	32.610	± 0.13	0.95	AS 1038.5
50	32.505	32.489	32.497	± 0.30	-0.02	AS 1038.5
54	32.381	32.400	32.391	± 0.045	-0.94	a.ISO 1928:2009(E) part 10.2
60	32.580	32.576	32.578	± 0.13	0.68	AS 1038.3

No of Results:	20
Median:	32.4993
Uncertainty (median)	0.0326
Normalised IQR:	0.1162
Robust CV:	0.4%
Minimum:	32.037
Maximum:	32.610
Range:	0.573

Note:A "§" indicates an outlier i.e. $|z\text{-score}| \geq 3.0$.

A "#" indicates no response was provided.

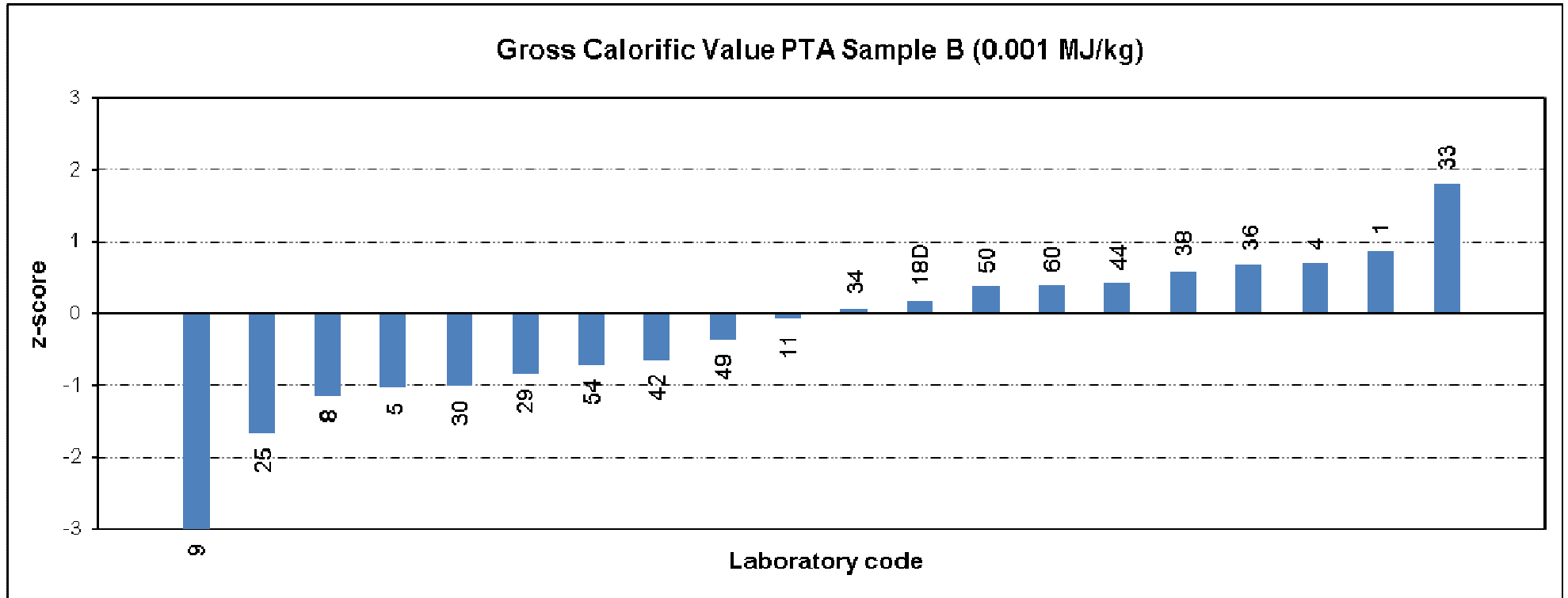


Gross Calorific Value PTA Sample B (0.001 MJ/kg)						
Laboratory code	Result 1	Result 2	Average	MU	Robust z-score	Method/Technique
1	26.373	26.458	26.416	± 0.13	0.88	AS 1038.5
4	26.353	26.432	26.393	± 0.13	0.69	AS 1038.5
5	26.194	26.171	26.183	± 0.13	-1.04	AS 1038.5
8	26.182	26.161	26.172	#	-1.13	AS 1038.5
9	25.69	25.72	25.705	± 0.13	-4.98 §	AS 1038.5
11	26.335	26.265	26.300	± 0.13	-0.07	AS 1038.5
18D	26.333	26.329	26.331	± 0.13	0.18	AS 1038.5
25	26.070	26.144	26.107	#	-1.66	ASTM D5865
29	26.220	26.193	26.207	± 0.3	-0.84	AS 1038.5
30	26.157	26.217	26.187	#	-1.00	AS 1038.5.2
33	26.566	26.490	26.528	± 0.3	1.81	ISO 1928
34	26.265	26.370	26.318	± 0.12	0.07	ISO 1928
36	26.391	26.392	26.392	± 0.020	0.68	ASTM D5865
38	26.356	26.406	26.381	± 0.188	0.60	ASTM D5865
42	26.138	26.323	26.231	± 2.00%	-0.65	DETSC 5007/ BS 15400
44	26.348	26.372	26.360	± 0.12	0.42	ISO 1928 - 2009
49	26.31	26.22	26.265	± 0.13	-0.36	AS 1038.5
50	26.378	26.331	26.355	± 0.30	0.38	AS 1038.5
54	26.210	26.235	26.223	± 0.045	-0.71	a.ISO 1928:2009(E) part 10.2
60	26.347	26.366	26.357	± 0.13	0.39	AS 1038.3

No of Results:	20
Median:	26.3088
Uncertainty (median)	0.0340
Normalised IQR:	0.1213
Robust CV:	0.5%
Minimum:	25.705
Maximum:	26.528
Range:	0.823

Note:A "§" indicates an outlier i.e. $|z\text{-score}| \geq 3.0$.

A "#" indicates no response was provided.

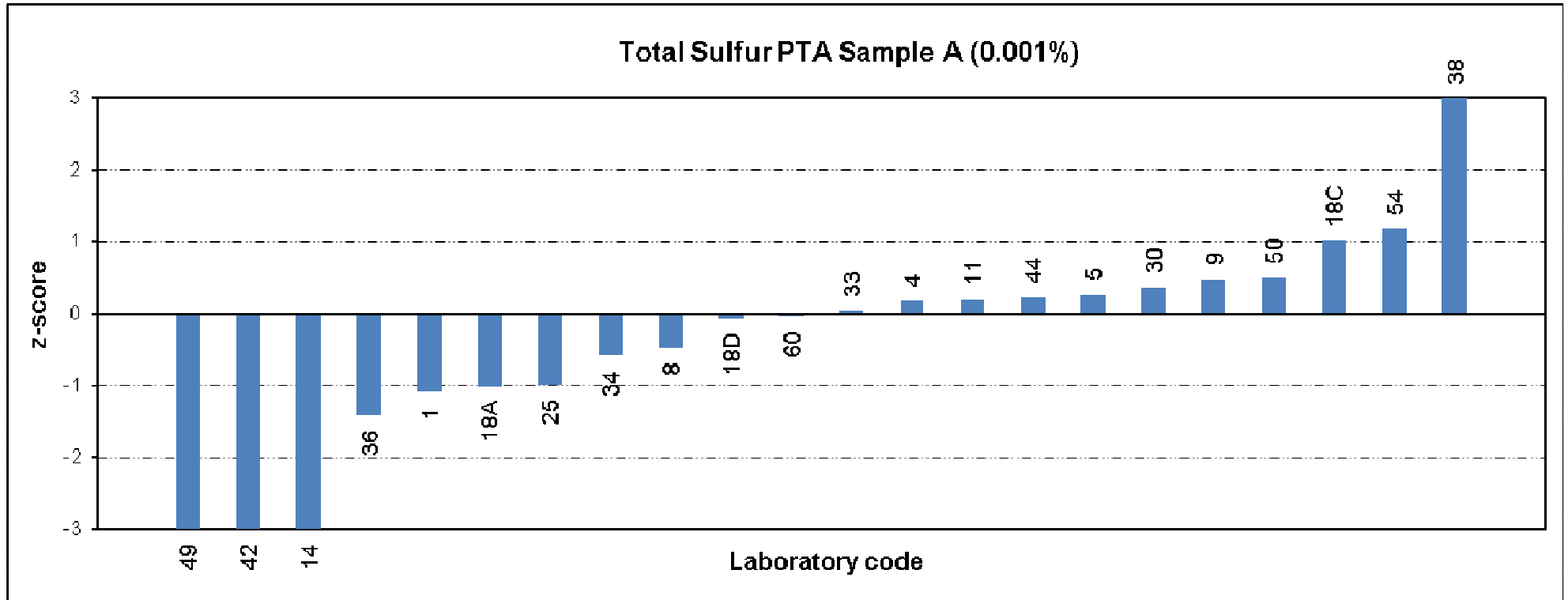


Total Sulfur PTA Sample A (0.001%)						
Laboratory code	Result 1	Result 2	Average	MU	Robust z-score	Method/Technique
1	0.413	0.424	0.419	± 0.03	-1.10	AS 1038.6.3.3
4	0.444	0.440	0.442	± 0.03	0.18	AS 1038.6.3.3
5	0.452	0.435	0.444	± 0.03	0.26	AS 1038.6.3.3
8	0.431	0.429	0.430	#	-0.47	AS 1038.6.3.3
9	0.456	0.439	0.448	± 0.2	0.47	AS 1038.6.3.3
11	0.446	0.439	0.443	± 0.03	0.20	AS 1038.6.3.3
14	0.259	0.250	0.255	#	-9.99	§ a.SL25, b.cls analyser
18A	0.419	0.421	0.420	± 0.03	-1.02	AS 1038.6.3.3
18C	0.465	0.450	0.458	± 0.03	1.02	AS 1038.6.3.3
18D	0.443	0.432	0.438	± 0.03	-0.07	AS 1038.6.3.3
25	0.416	0.425	0.421	#	-0.99	ASTM D3177
30	0.444	0.447	0.446	#	0.37	AS 1038.6.3.3
33	0.438	0.441	0.440	± 0.1	0.04	ISO 19579
34	0.431	0.425	0.428	± 0.03	-0.58	AS 1038.6.3.3
36	0.410	0.415	0.413	± 0.004	-1.42	ASTM D3177
38	0.490	0.500	0.495	± 0.040	3.05	§ ASTM D4239 DETSC 5016
42	0.168	0.160	0.164	± 37.76%	-14.90	§ Microwave acid digest ICPOES analysis
44	0.444	0.442	0.443	± 0.03	0.23	AS 1038.6.3.3
49	0.045	0.048	0.047	± 0.10	-21.27	§ XRF in house method validated against AS 1038.9.2
50	0.452	0.444	0.448	± 0.05	0.50	AS 1038.6.3.3
54	0.462	0.459	0.461	± 0.021	1.18	a.ASTM D4239-14 part 8.2.1 b.Method A: Calibration with coal reference materials
60	0.438	0.438	0.438	± 0.03	-0.04	AS 1038.6.3.3

No of Results:	22
Median:	0.4388
Uncertainty (median)	0.0049
Normalised IQR:	0.0184
Robust CV:	4.2%
Minimum:	0.047
Maximum:	0.495
Range:	0.449

Note:

A "§" indicates an outlier i.e. $|z\text{-score}| \geq 3.0$.
A "#" indicates no response was provided.

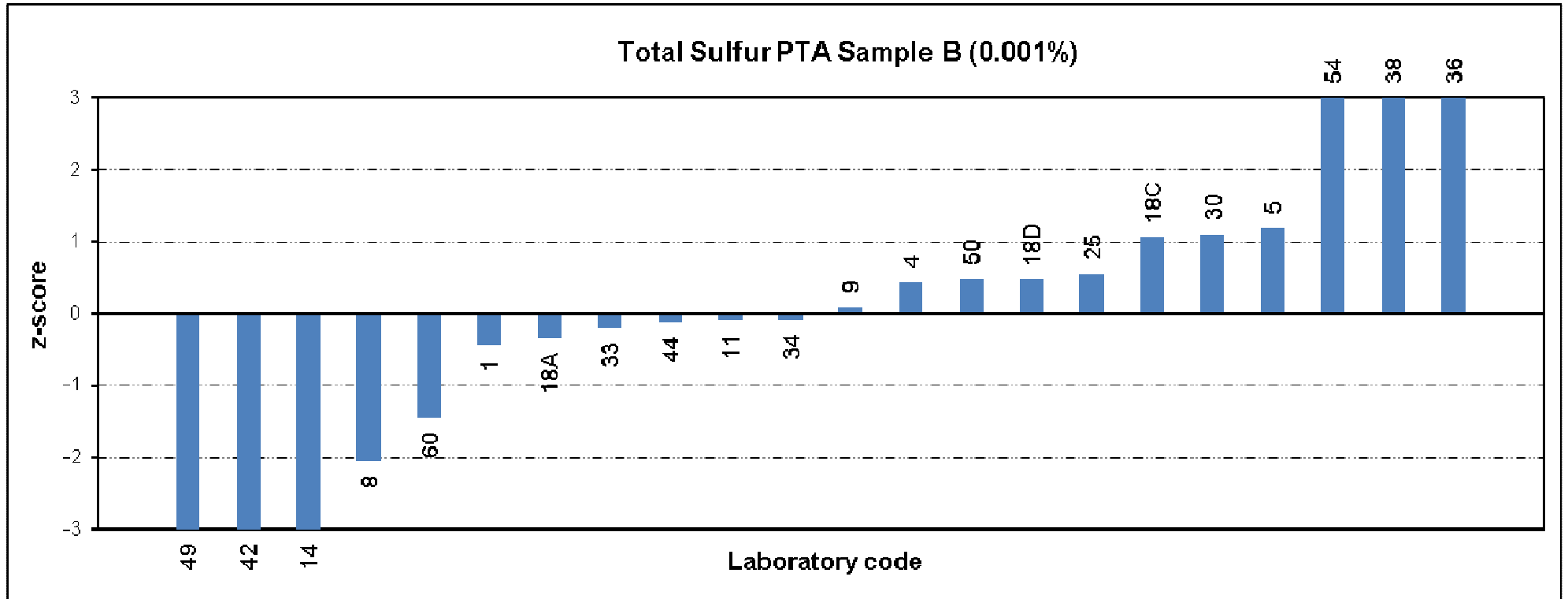


Total Sulfur PTA Sample B (0.001%)						
Laboratory code	Result 1	Result 2	Average	MU	Robust z-score	Method/Technique
1	0.350	0.346	0.348	± 0.03	-0.43	AS 1038.6.3.3
4	0.360	0.358	0.359	± 0.03	0.43	AS 1038.6.3.3
5	0.376	0.361	0.369	± 0.03	1.18	AS 1038.6.3.3
8	0.322	0.333	0.328	#	-2.05	AS 1038.6.3.3
9	0.369	0.340	0.355	± 0.2	0.08	AS 1038.6.3.3
11	0.355	0.350	0.353	± 0.03	-0.08	AS 1038.6.3.3
14	0.223	0.201	0.212	#	-11.15	§ a.SL25, b.cls analyser
18A	0.347	0.351	0.349	± 0.03	-0.35	AS 1038.6.3.3
18C	0.363	0.371	0.367	± 0.03	1.06	AS 1038.6.3.3
18D	0.362	0.357	0.360	± 0.03	0.47	AS 1038.6.3.3
25	0.366	0.355	0.361	#	0.55	ASTM D3177
30	0.361	0.374	0.368	#	1.10	AS 1038.6.3.3
33	0.345	0.357	0.351	± 0.1	-0.20	ISO 19579
34	0.353	0.352	0.353	± 0.03	-0.08	AS 1038.6.3.3
36	0.460	0.468	0.464	± 0.004	8.70	§ ASTM D3177
38	0.390	0.400	0.395	± 0.030	3.27	§ ASTM D4239 DETSC 5016
42	0.148	0.131	0.140	± 37.76%	-16.86	§ Microwave acid digest ICPOES analysis
44	0.351	0.353	0.352	± 0.03	-0.12	AS 1038.6.3.3
49	0.103	0.112	0.108	± 0.10	-19.38	§ XRF in house method validated against AS 1038.9.2
50	0.356	0.363	0.360	± 0.05	0.47	AS 1038.6.3.3
54	0.398	0.391	0.395	± 0.021	3.23	§ a.ASTM D4239-14 part 8.2.1 b.Method A: Calibration with coal reference materials
60	0.335	0.335	0.335	± 0.03	-1.46	AS 1038.6.3.3

No of Results:	22
Median:	0.3535
Uncertainty (median)	0.0034
Normalised IQR:	0.0127
Robust CV:	3.6%
Minimum:	0.108
Maximum:	0.464
Range:	0.357

Note:

A "§" indicates an outlier i.e. $|z\text{-score}| \geq 3.0$.
A "#" indicates no response was provided.



APPENDIX B

Homogeneity Testing

Homogeneity Testing

10 jars were randomly selected and tested for homogeneity by determining and recording the Ash and Moisture Content of each with results reported on a dry basis. The homogeneity for PTA's Coal Proficiency Testing Program Round 32 was determined on Ash in accordance with ISO Guide 35 and ISO 1171.

PTA Sample A			
Bottle number	Duplicate (a)	Duplicate (b)	Average
34	8.04	8.08	8.06
8	8.07	8.08	8.08
41	8.12	8.08	8.10
38	8.04	8.06	8.05
18	8.04	8.03	8.04
1	8.05	8.06	8.06
24	8.05	8.01	8.03
42	8.06	8.07	8.07

PTA Sample B			
Bottle number	Duplicate (a)	Duplicate (b)	Average
34	23.65	23.59	23.62
8	23.62	23.68	23.65
41	23.53	23.57	23.55
38	23.63	23.68	23.66
18	23.53	23.44	23.49
1	23.47	23.54	23.51
24	23.59	23.41	23.50
42	23.50	23.46	23.48

Statistical analysis of the results indicated that no notable sample variability existed. Therefore, it was concluded that any outlier results subsequently identified could not be attributed to sample variability.

APPENDIX C

Documentation

Instructions to Participants	C1
Results Sheet	C2

**PROFICIENCY TESTING AUSTRALIA
COAL PROFICIENCY TESTING PROGRAM (ROUND 32)
INSTRUCTIONS TO PARTICIPANTS**

To ensure that results from this program can be analysed properly, participants are asked to adhere carefully to the following instructions.

1. Two 125 gram coal samples labelled PTA Sample A and PTA Sample B have been supplied to each laboratory.

2. For the sample the following determinations are required in duplicate:

*Moisture, Ash, Volatile Matter, Gross Calorific Value and Total Sulfur.

Results for Moisture are to be reported to air-dry basis.

All other tests are to be reported to DRY basis.

(*Results obtained from proximate analysis)

3. These tests are to be conducted in accordance with AS 1038 (relevant sections). However laboratories may perform their tests to other methods and note this on the attached result sheet.

4. For each test on the sample, two replicate results are to be reported to the accuracy and reporting basis indicated on the result sheet.

5. The following additional information is required for each test:

a) Method - relevant AS, ISO or in-house method number (include part numbers, e.g. AS 1038 Part 6.3.2.)

b) Technique - if there is alternative options in a particular method, state which option is used.

6. Laboratories are also requested to calculate and report an estimate of measurement uncertainty (MU) for each reported replicated measurement result. All estimates of measurement uncertainty must be given as a 95% confidence interval (coverage factor $k \approx 2$).

7. Testing may commence as soon as the sample is received. All laboratories must return the results sheet no later than **24 October 2014** to:

Laura Galbraith
Proficiency Testing Australia
PO Box 7507
SILVERWATER NSW 2128
Phone: 02 9736 8397
Fax: 02 9743 6664
Email: laura.galbraith@pta.asn.au



C2

**PROFICIENCY TESTING AUSTRALIA
COAL PROFICIENCY TESTING PROGRAM (ROUND 32)**

RESULTS SHEET

Lab Code

The reporting basis for all tests is dry except for Moisture (air-dry).

TEST (report to)	SAMPLE A			SAMPLE B			a. Method b. Technique
	Result 1	Result 2	MU (±)	Result 1	Result 2	MU (±)	
Moisture (air-dry basis) (0.01%)							
Ash (0.01%)							
Volatile Matter (0.01%)							
Gross Calorific Value (0.001 MJ/kg)							
Total Sulfur (0.001%)							

Signed: _____

Date: _____

Please return to: Ms Laura Galbraith, Fax: +61 2 9743 6664, Email: laura.galbraith@pta.asn.au by the **24 October 2014**

- *End of Report* -