

**Report No. 1103**

**Coal Proficiency Testing Program**

**Round 37**

**September 2018**

**Acknowledgments**

PTA wishes to gratefully acknowledge the technical assistance provided for this program by Mr J Kelly of BHP, QLD and by Mr M Preston of ALS Coal Gladstone, QLD. The samples were supplied by Mr M Preston of ALS Coal Gladstone, QLD.

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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 37<sup>th</sup> round of an ongoing series of programs. This program is accredited to ISO/IEC 17043:2010 “*Conformity assessment - General requirements for proficiency testing*” by International Accreditation New Zealand (IANZ).

The program was conducted in July-August 2018 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 Dr E Cincu and the Technical Advisers were Mr J Kelly of BHP, QLD and Mr M Preston of ALS Coal Gladstone, QLD. This report was authorised by Mrs K Cividin, PTA Quality 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 22 laboratories received samples, comprising:
  - 14 Australian participants; and
  - 8 overseas participants, including:
    - Colombia (1);
    - Korea (3);
    - Malaysia (2); and
    - Philippines (2).

Of these 22 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 eight 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 one laboratory reported more than one set of results and, therefore, it’s code number (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 4). 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, 2016* (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 7.

### **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 and stability 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 7 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.

Please note that the average results have been displayed to one decimal place more than requested from the participants.

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).

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

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

$n$  = number of results

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	Sample	No. of Results	Median	Normalised IQR
<b>Moisture (air-dry basis) (0.01%)</b>	Sample A	20	Not calculated	
	Sample B	20	Not calculated	
<b>Ash (0.01%)</b>	Sample A	19	9.590	0.095
	Sample B	19	14.370	0.146
<b>Volatile Matter (0.01%)</b>	Sample A	19	24.325	0.712
	Sample B	19	29.730	0.797
<b>Gross Calorific Value (0.001 MJ/kg)</b>	Sample A	19	31.3650	0.1012
	Sample B	19	26.4500	0.2496
<b>Total Sulfur (0.001%)</b>	Sample A	19	0.4160	0.0065
	Sample B	19	0.1600	0.0165

**Note:** Statistics for each test are based on the average of the duplicate results reported for the sample.

## 5. PTA AND TECHNICAL ADVISER'S COMMENTS

Of the 22 participating laboratories, one laboratory did not submit results in time for inclusion in the final report. From the remaining 21 laboratories, 10 outliers were identified; in total resulting in 5.20% of outliers in 192 results. The overall performance was similar to previous rounds, as depicted in Table B.

The Moisture test 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.

Two participants (Laboratory Codes 14 and 16) reported a Gross Calorific Value (GCV) but either no Moisture or Sulfur results which are required for the correct reporting of a GCV result as per the relevant standard methods.

Table C shows a comparison of past program Robust CV (%) results for the coal sample. While the variability of the results was low for Ash, Volatile Matter and Gross Calorific Value, the identified outlier for Laboratory Code 28 Sulfur (Sample B) determined an increased CV value of 10.3%.

Table D shows the laboratories identified with outliers. All laboratories with a z-score greater than +/- 2 are encouraged to review their procedures.

**TABLE B: PREVIOUS ROUNDS OUTLIER RESULTS**

	<b>Round 33 Coal</b>	<b>Round 34 Coal</b>	<b>Round 35 Coal</b>	<b>Round 36 Coal</b>	<b>Round 37 Coal</b>
<b>Total Results</b>	200	212	172	237	192
<b>Number of Outliers</b>	22	15	5	15	10
<b>% outliers</b>	11.00%	7.08%	2.91%	6.32%	5.20%

**TABLE C: COMPARISON OF ROBUST CVS (%)**

Test	Round 34	Round 35		Round 36	Round 37	
	Sample	Sample A	Sample B	Sample A	Sample A	Sample B
<b>Ash (0.01%)</b>	1.0	1.2	0.9	0.8	1.0	1.0
<b>Volatile Matter (0.01%)</b>	1.0	1.0	2.7	1.5	2.9	2.7
<b>Gross Calorific Value (0.001 MJ/kg)</b>	0.2	0.3	0.4	0.4	0.3	0.9
<b>Total Sulfur (0.001%)</b>	2.7	3.8	5.5	4.0	1.6	10.3

#### 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.

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

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.



## 6. OUTLIER RESULTS

Laboratories reporting outlier results are listed in the following table:

**TABLE D: SUMMARY OF STATISTICAL OUTLIERS**

Test	Sample	Laboratory Code
<b>Ash (0.01%)</b>	Sample A	-
	Sample B	4, 29, 68B
<b>Volatile Matter (0.01%)</b>	Sample A	68B
	Sample B	31, 56, 68B
<b>Gross Calorific Value (0.001 MJ/kg)</b>	Sample A	-
	Sample B	-
<b>Total Sulfur (0.001%)</b>	Sample A	4, 29
	Sample B	29

## 7. REFERENCES

- [1] *Guide to Proficiency Testing Australia*, 2016 (This document can be found on the PTA website, [www.pta.asn.au](http://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(R2013) Coal and coke – Analysis and testing – Higher rank coal – Ultimate analysis – Total sulfur – Infrared method*
- [5] *ISO Guide 35 - 2017 Reference materials -- Guidance for characterization and assessment of homogeneity and stability*
- [6] *ISO 1171 - 2010 Solid mineral fuels - Determination of ash*
- [7] *ISO 11722 - 2013 Solid mineral fuels -- Hard coal -- Determination of moisture in the general analysis test sample by drying in nitrogen*
- [8] *ISO 562 - 2010 Hard coal and coke -- Determination of volatile matter*

- [9] *ISO 1928 - 2009 Solid mineral fuels -- Determination of gross calorific value by the bomb calorimetric method and calculation of net calorific value*
- [10] *ISO 19579 - 2006 Solid mineral fuels -- Determination of sulfur by IR spectrometry*

Precision data can be referenced from the following standards:

**TABLE E: STANDARDS FOR PRECISION DATA**

<b>Standard</b>	<b>Test</b>
AS 1038.3 (2000) ISO 1171 (2010)	Ash
AS 1038.3 (2000) ISO 562 (2010)	Volatile Matter
AS 1038.5 (1998) ISO 1928 (2009)	Gross Calorific Value
AS 1038.6.3.2 (2003) AS 1038.6.3.3 (1997) ISO 19579 (2006)	Total Sulfur

# APPENDIX A

## Results and Data Analysis

Moisture (air-dry basis) (0.01%).....	A1
Ash (0.01%).....	A3
Volatile Matter (0.01%).....	A6
Gross Calorific Value (0.001 MJ/kg).....	A9
Total Sulfur (0.001%).....	A12

<b>Moisture (air-dry basis) (0.01%) Sample A</b>					
<b>Laboratory Code</b>	<b>Result 1</b>	<b>Result 2</b>	<b>Average</b>	<b>MU (±)</b>	<b>Method/Technique</b>
<b>2</b>	1.98	1.94	1.960	0.06	ASTM D3173
<b>4</b>	1.53	1.44	1.485	0.05	ASTM D3173
<b>13</b>	2.07	2.00	2.035	0.1	AS 1038.3
<b>14</b>	2.39	2.27	2.330	0.083	AS 1038.3 Section 2
<b>17</b>	1.99	1.89	1.940	0.1	AS 1038.3
<b>20</b>	2.02	2.01	2.015	0.1	AS 1038.3
<b>22</b>	1.98	1.98	1.980	0.18	ASTM D3173-11
<b>26</b>	2.32	1.99	2.155	0.1	AS 1038.3
<b>27</b>	2.22	2.18	2.200	0.14	ASTM D7582-15
<b>29</b>	1.87	1.83	1.850	#	ASTM D3173
<b>31</b>	2.12	2.19	2.155	0.08	ASTM D3173/ D3173M-17a
<b>40</b>	2.10	2.06	2.080	#	AS 1038.4
<b>41</b>	2.24	2.31	2.275	0.10	ISO 11722
<b>46</b>	2.08	2.07	2.075	0.1	AS 1038.3
<b>53</b>	1.84	1.86	1.850	#	ISO 11722
<b>56</b>	2.16	2.15	2.155	0.03	ASTM D7582-15
<b>68A</b>	2.19	2.20	2.195	#	TGA
<b>68B</b>	1.78	1.77	1.775	#	AS 1038.3 Furnace, Lab method SL05
<b>69</b>	2.28	2.32	2.300	0.15	ISO 11722
<b>70</b>	2.42	2.42	2.420	#	ISO 11722

No of Results	20
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## Notes:

Moisture results are required purely as a correctional value, to enable calculation of other results to dry basis

"#" indicates no response was provided by the laboratory

<b>Moisture (air-dry basis) (0.01%) Sample B</b>					
<b>Laboratory Code</b>	<b>Result 1</b>	<b>Result 2</b>	<b>Average</b>	<b>MU (±)</b>	<b>Method/Technique</b>
<b>2</b>	10.60	10.51	10.555	0.06	ASTM D3173
<b>4</b>	9.41	9.23	9.320	0.09	ASTM D3173
<b>13</b>	9.40	9.43	9.415	0.1	AS 1038.3
<b>14</b>	11.23	11.21	11.220	0.014	AS 1038.3 Section 2
<b>17</b>	7.71	7.69	7.700	0.1	AS 1038.3
<b>20</b>	9.89	9.94	9.915	0.1	AS 1038.3
<b>22</b>	10.54	10.52	10.530	0.18	ASTM D3173-11
<b>26</b>	10.29	10.39	10.340	0.1	AS 1038.3
<b>27</b>	10.67	10.69	10.680	0.13	ASTM D7582-15
<b>29</b>	9.95	9.93	9.940	#	ASTM D3173
<b>31</b>	10.56	10.72	10.640	0.16	ASTM D3173/ D3173M-17a
<b>40</b>	9.30	9.24	9.270	#	AS 1038.4
<b>41</b>	10.53	10.50	10.515	0.10	ISO 11722
<b>46</b>	11.04	11.05	11.045	0.1	AS 1038.3
<b>53</b>	9.05	9.01	9.030	#	ISO 11722
<b>56</b>	10.66	10.64	10.650	0.03	ASTM D7582-15
<b>68A</b>	10.89	10.81	10.850	#	TGA
<b>68B</b>	10.19	10.20	10.195	#	AS 1038.3 Furnace, Lab method SL05
<b>69</b>	10.15	10.25	10.200	0.15	ISO 11722
<b>70</b>	11.12	11.09	11.105	#	ISO 11722

No of Results	20
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## Notes:

Moisture results are required purely as a correctional value, to enable calculation of other results to dry basis

"#" indicates no response was provided by the laboratory

Ash (0.01%) Sample A						
Laboratory Code	Result 1	Result 2	Average	MU ( $\pm$ )	Z-Score	Method/Technique
2	9.62	9.65	9.635	0.22	0.48	ASTM D3174
4	9.54	9.44	9.490	0.05	-1.06	ASTM D3174
13	9.71	9.69	9.700	0.1	1.16	AS 1038.3
17	9.69	9.70	9.695	0.1	1.11	AS 1038.3
20	9.56	9.60	9.580	0.1	-0.11	AS 1038.3
22	9.63	9.63	9.630	0.17	0.42	In-house method based on ASTM D3174-11
26	9.66	9.56	9.610	0.1	0.21	AS 1038.3
27	9.62	9.65	9.635	0.18	0.48	ASTM D7582-15
29	9.52	9.49	9.505	#	-0.90	ASTM D3174
31	9.47	9.64	9.555	0.25	-0.37	ASTM D3174-12
40	9.58	9.61	9.595	0.15	0.05	AS 1038.4
41	9.59	9.59	9.590	0.15	0.00	ISO 1171
46	9.69	9.67	9.680	0.1	0.95	AS 1038.3
53	9.54	9.60	9.570	0.60% Absolute	-0.21	ISO 1171
56	9.39	9.42	9.405	0.20	-1.96	ASTM D7582-15
68A	9.5	9.5	9.500	#	-0.95	TGA
68B	9.5	9.5	9.500	#	-0.95	AS 1038.3 Furnace, Lab method SL03
69	9.50	9.52	9.510	0.6	-0.85	ISO1171
70	9.67	9.67	9.670	#	0.85	ISO 1171

No of Results	19
Median	9.590
Norm IQR	0.095
Uncertainty (Median)	0.027
Robust CV	1.0%
Minimum	9.41
Maximum	9.70
Range	0.29

Note:

"#" indicates no response was provided by the laboratory

Ash (0.01%) Sample B						
Laboratory Code	Result 1	Result 2	Average	MU ( $\pm$ )	Z-Score	Method/Technique
2	14.31	14.41	14.360	0.22	-0.07	ASTM D3174
4	13.90	13.82	13.860	0.04	-3.48	§ ASTM D3174
13	14.48	14.50	14.490	0.1	0.82	AS 1038.3
17	14.57	14.46	14.515	0.1	0.99	AS 1038.3
20	14.39	14.36	14.375	0.1	0.03	AS 1038.3
22	14.43	14.46	14.445	0.25	0.51	In-house method based on ASTM D3174-11
26	14.38	14.40	14.390	0.1	0.14	AS 1038.3
27	14.28	14.26	14.270	0.20	-0.68	ASTM D7582-15
29	13.84	13.80	13.820	#	-3.76	§ ASTM D3174
31	14.26	14.38	14.320	0.20	-0.34	ASTM D3174-12
40	14.42	14.35	14.385	0.15	0.10	AS 1038.4
41	14.35	14.39	14.370	0.15	0.00	ISO 1171
46	14.41	14.51	14.460	0.1	0.61	AS 1038.3
53	14.39	14.36	14.375	0.60% Absolute	0.03	ISO 1171
56	14.12	14.17	14.145	0.26	-1.54	ASTM D7582-15
68A	14.0	14.0	14.000	#	-2.53	? TGA
68B	13.0	12.8	12.900	#	-10.04	§ AS 1038.3 Furnace, Lab method SL03
69	14.40	14.34	14.370	0.6	0.00	ISO1171
70	14.43	14.41	14.420	#	0.34	ISO 1171

No of Results	19
Median	14.370
Norm IQR	0.146
Uncertainty (Median)	0.042
Robust CV	1.0%
Minimum	12.90
Maximum	14.52
Range	1.62

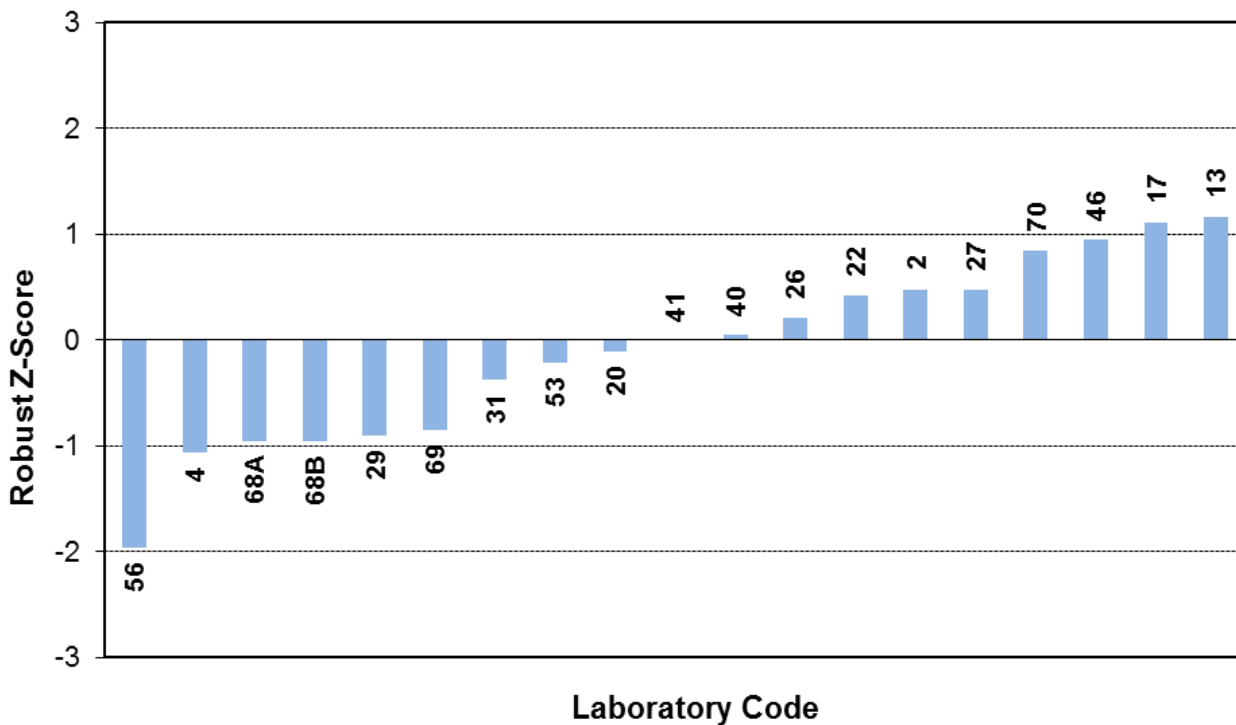
## Notes:

"#" indicates no response was provided by the laboratory

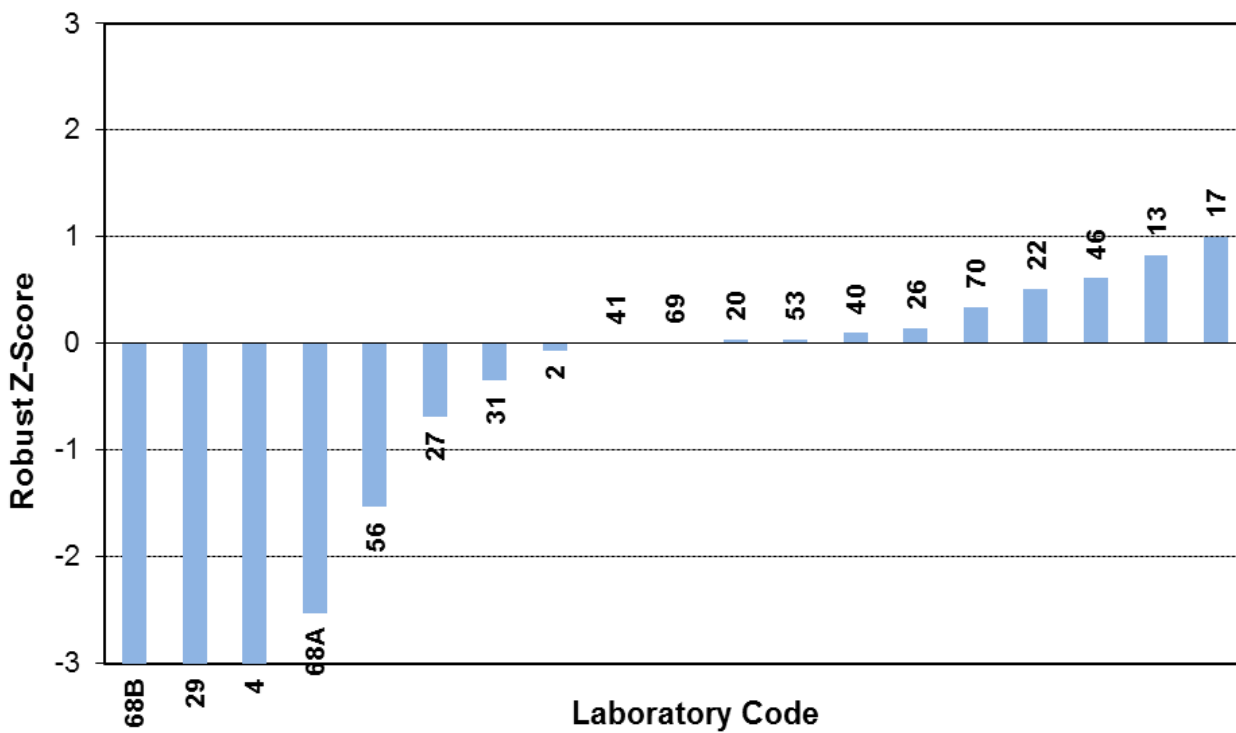
"?" indicates an absolute z-score greater than 2 but less than 3, i.e.  $2 < |z\text{-score}| < 3$

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

**Ash (0.01%) Sample A**



**Ash (0.01%) Sample B**





Volatile Matter (0.01%) Sample A						
Laboratory Code	Result 1	Result 2	Average	MU ( $\pm$ )	Z-Score	Method/Technique
2	24.64	24.575	24.608	1.51	0.40	ASTM D3175
4	25.47	25.09	25.280	0.19	1.34	ASTM D3175
13	24.18	24.21	24.195	0.2	-0.18	AS 1038.3
17	23.99	23.96	23.975	0.2	-0.49	AS 1038.3
20	24.43	24.47	24.450	0.2	0.18	AS 1038.3
22	24.95	24.92	24.935	0.69	0.86	In-house method based on ASTM D3175-11
26	24.18	24.30	24.240	0.2	-0.12	AS 1038.3
27	24.96	24.94	24.950	0.35	0.88	ASTM D7582-15
29	25.07	24.99	25.030	#	0.99	ASTM D3175
31	26.26	26.39	26.325	0.38	2.81	? ASTM D3175-17
40	23.95	23.99	23.970	0.50	-0.50	AS 1038.4
41	24.14	24.22	24.180	0.20	-0.20	ISO 562
46	23.98	24.00	23.990	0.2	-0.47	AS 1038.3
53	24.25	24.40	24.325	1.0% Absolute	0.00	ISO 562
56	25.80	25.84	25.820	0.54	2.10	? ASTM D7582-15
68A	23.9	23.9	23.900	#	-0.60	TGA
68B	10.2	10.2	10.200	#	-19.85	§ AS 1038.3 Furnace, Lab method SL04
69	24.48	24.54	24.510	1	0.26	ISO 562
70	23.81	23.78	23.795	#	-0.74	ISO 562

No of Results	19
Median	24.325
Norm IQR	0.712
Uncertainty (Median)	0.205
Robust CV	2.9%
Minimum	10.20
Maximum	26.33
Range	16.13

## Notes:

"#" indicates no response was provided by the laboratory

"?" indicates an absolute z-score greater than 2 but less than 3, i.e.  $2 < |z\text{-score}| < 3$

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

Volatile Matter (0.01%) Sample B						
Laboratory Code	Result 1	Result 2	Average	MU ( $\pm$ )	Z-Score	Method/Technique
2	30.01	30.00	30.005	1.51	0.35	ASTM D3175
4	32.09	31.76	31.925	0.17	2.75	? ASTM D3175
13	29.41	29.43	29.420	0.2	-0.39	AS 1038.3
17	29.74	29.74	29.740	0.2	0.01	AS 1038.3
20	29.77	29.63	29.700	0.2	-0.04	AS 1038.3
22	30.43	30.39	30.410	0.84	0.85	In-house method based on ASTM D3175-11
26	29.66	29.63	29.645	0.2	-0.11	AS 1038.3
27	31.46	31.54	31.500	0.39	2.22	? ASTM D7582-15
29	30.44	30.71	30.575	#	1.06	ASTM D3175
31	34.24	34.79	34.515	0.59	6.00	§ ASTM D3175-17
40	29.35	29.35	29.350	0.50	-0.48	AS 1038.4
41	29.94	29.98	29.960	0.20	0.29	ISO 562
46	29.39	29.30	29.345	0.2	-0.48	AS 1038.3
53	29.38	29.45	29.415	1.0% Absolute	-0.40	ISO 562
56	32.18	32.10	32.140	0.59	3.02	§ ASTM D7582-15
68A	29.5	29.4	29.450	#	-0.35	TGA
68B	24.6	24.7	24.650	#	-6.37	§ AS 1038.3 Furnace, Lab method SL04
69	29.70	29.76	29.730	1	0.00	ISO 562
70	29.30	29.24	29.270	#	-0.58	ISO 562

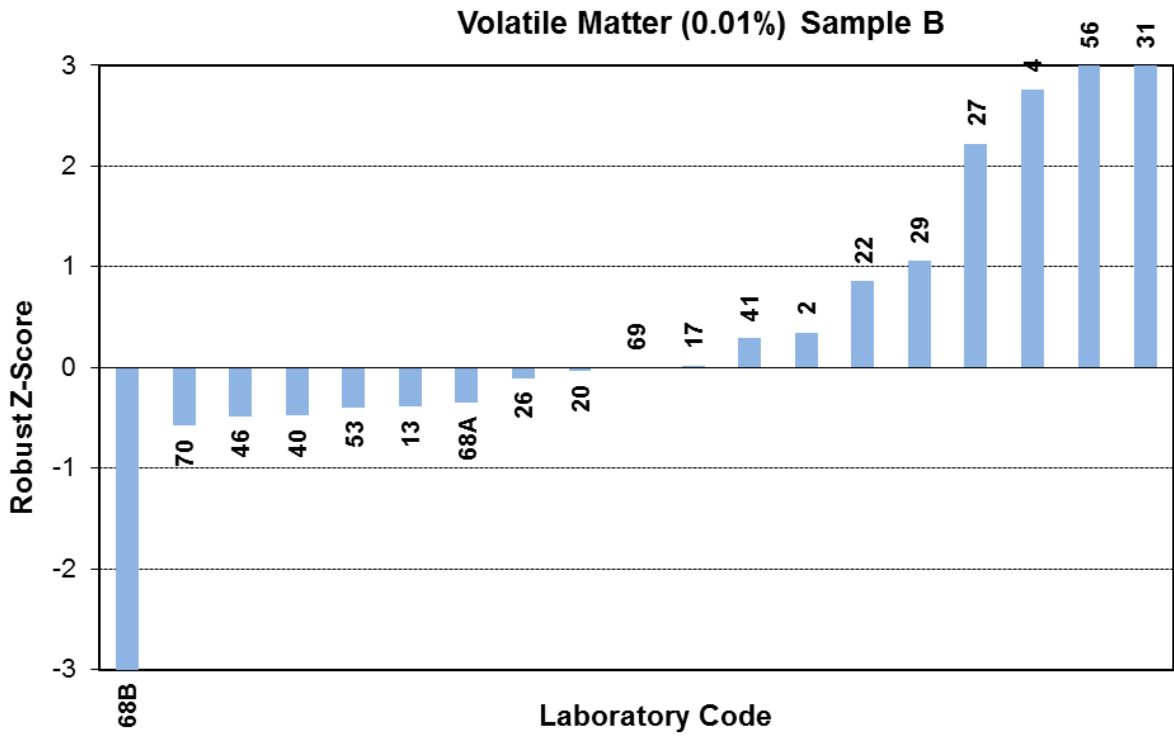
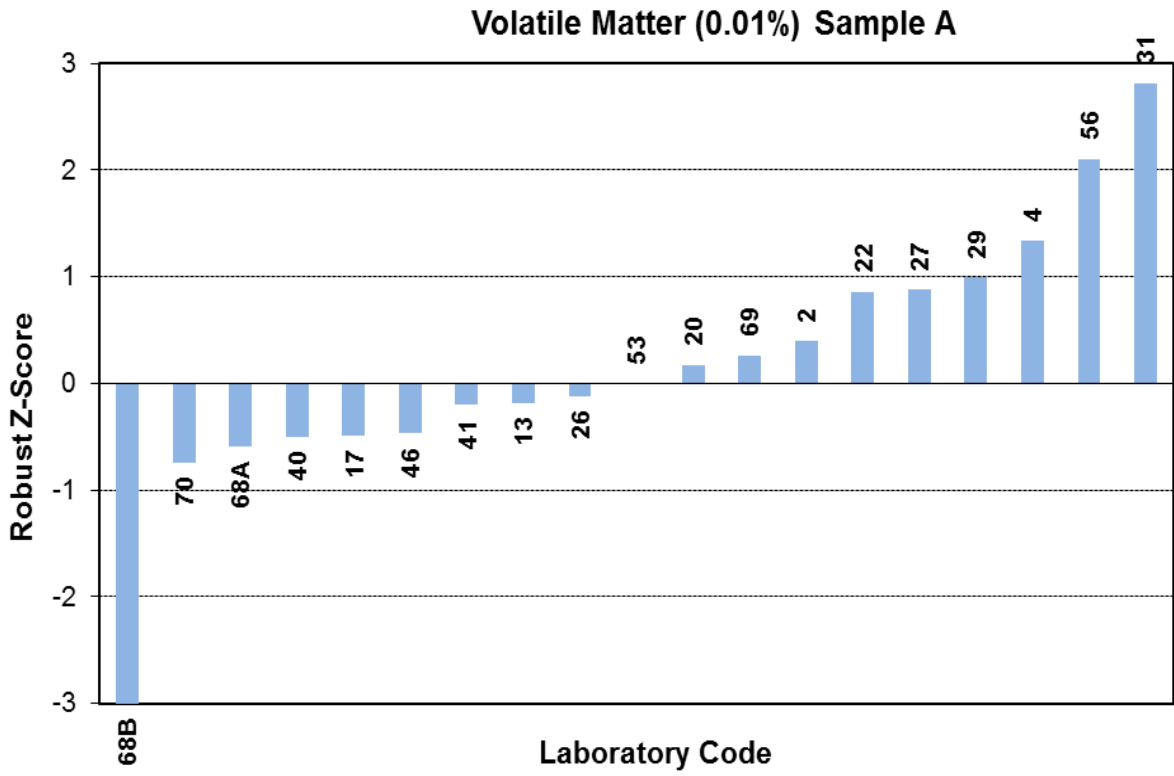
No of Results	19
Median	29.730
Norm IQR	0.797
Uncertainty (Median)	0.229
Robust CV	2.7%
Minimum	24.65
Maximum	34.52
Range	9.87

## Notes:

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"?" indicates an absolute z-score greater than 2 but less than 3, i.e.  $2 < |z\text{-score}| < 3$

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



Gross Calorific Value (0.001 MJ/Kg) Sample A						
Laboratory Code	Result 1	Result 2	Average	MU ( $\pm$ )	Z-Score	Method/Technique
2	31.173	31.180	31.1765	0.149	-1.86	ASTM D5865
4	31.19	31.05	31.1200	0.07	-2.42	? ASTM D5865
13	31.422	31.426	31.4240	0.13	0.58	AS 1038.5
14	31.547	31.440	31.4935	0.0741	1.27	AS 1038.5
16	31.351	31.269	31.3100	0.097	-0.54	ASTM D5865-13
17	31.414	31.393	31.4035	0.13	0.38	AS 1038.5
20	31.41	31.35	31.3800	0.13	0.15	AS 1038.5
22	31.280	31.292	31.2860	0.285	-0.78	ASTM D5865-11a
26	31.407	31.284	31.3455	0.13	-0.19	AS 1038.5
27	31.300	31.325	31.3125	0.216	-0.52	ASTM D5865-13
29	31.324	31.256	31.2900	#	-0.74	ASTM D5865
31	31.150	31.230	31.1900	0.067	-1.73	ASTM D5865-13
40	31.473	31.434	31.4535	0.30	0.87	AS 1038.5
41	31.364	31.366	31.3650	0.13	0.00	ISO 1928
46	31.42	31.43	31.4250	0.13	0.59	AS 1038.5
53	31.557	31.521	31.5390	0.30	1.72	ISO 1928
56	31.291	31.234	31.2625	0.073	-1.01	KS E3702
69	31.500	31.490	31.4950	0.3	1.28	ISO 1928
70	31.371	31.362	31.3665	#	0.01	ISO 1928

No of Results	19
Median	31.3650
Norm IQR	0.1012
Uncertainty (Median)	0.0291
Robust CV	0.3%
Minimum	31.120
Maximum	31.539
Range	0.419

## Notes:

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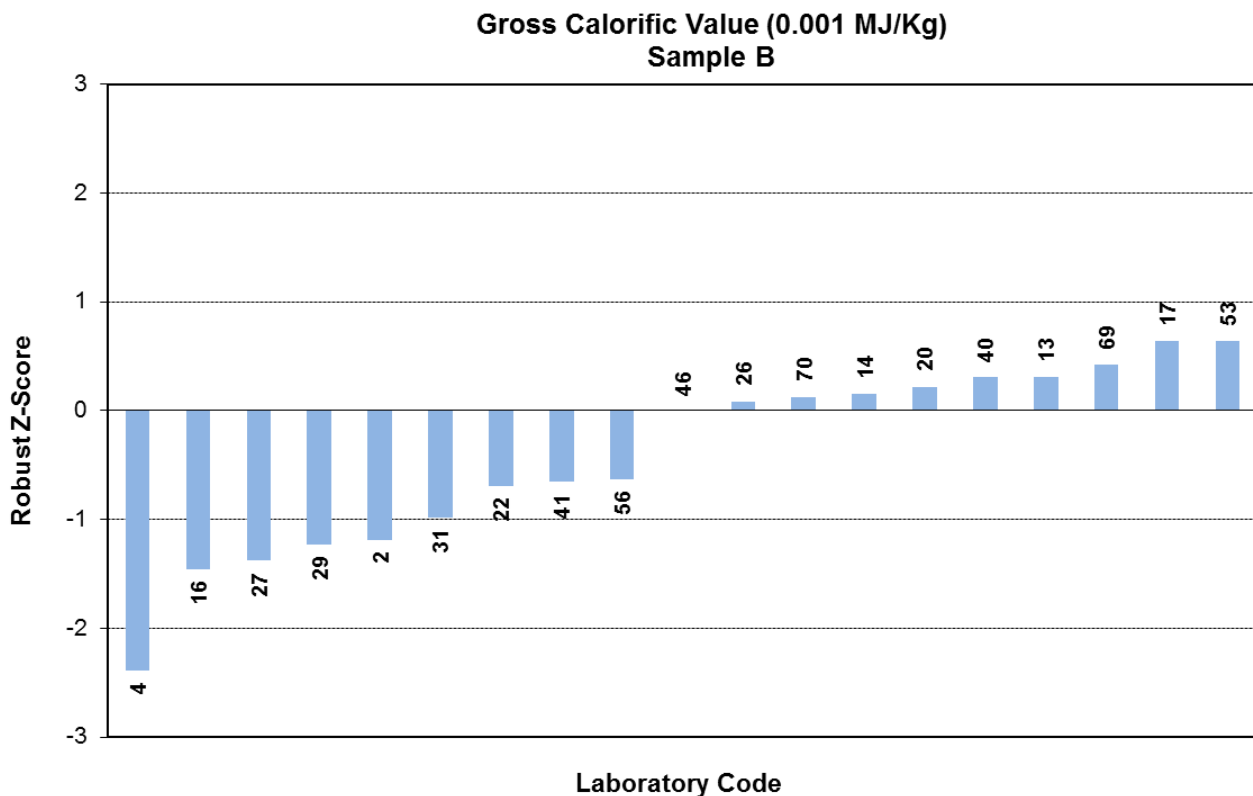
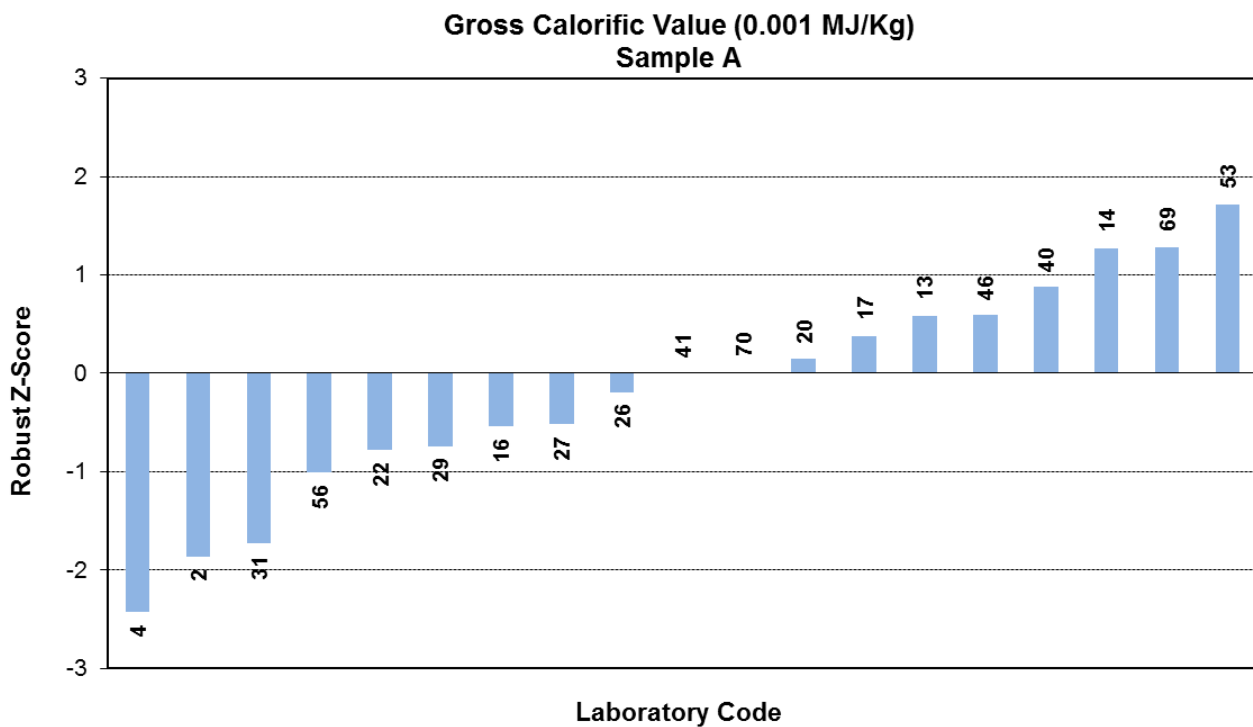
Gross Calorific Value (0.001 MJ/Kg) Sample B						
Laboratory Code	Result 1	Result 2	Average	MU ( $\pm$ )	Z-Score	Method/Technique
2	26.143	26.163	26.1530	0.149	- 1.19	ASTM D5865
4	25.91	25.80	25.8550	0.06	- 2.38	? ASTM D5865
13	26.532	26.523	26.5275	0.13	0.31	AS 1038.5
14	26.476	26.500	26.4880	0.0166	0.15	AS 1038.5
16	26.087	26.087	26.0870	0.051	- 1.45	ASTM D5865-13
17	26.590	26.628	26.6090	0.13	0.64	AS 1038.5
20	26.48	26.53	26.5050	0.13	0.22	AS 1038.5
22	26.245	26.309	26.2770	0.285	- 0.69	ASTM D5865-11a
26	26.448	26.494	26.4710	0.13	0.08	AS 1038.5
27	26.112	26.101	26.1065	0.224	- 1.38	ASTM D5865-13
29	26.154	26.132	26.1430	#	- 1.23	ASTM D5865
31	26.140	26.270	26.2050	0.072	- 0.98	ASTM D5865-13
40	26.558	26.495	26.5265	0.30	0.31	AS 1038.5
41	26.292	26.281	26.2865	0.13	- 0.65	ISO 1928
46	26.44	26.46	26.4500	0.13	0.00	AS 1038.5
53	26.630	26.589	26.6095	0.30	0.64	ISO 1928
56	26.304	26.281	26.2925	0.052	- 0.63	KS E3702
69	26.550	26.559	26.5545	0.3	0.42	ISO 1928
70	26.473	26.490	26.4815	#	0.13	ISO 1928

No of Results	19
Median	26.4500
Norm IQR	0.2496
Uncertainty (Median)	0.0718
Robust CV	0.9%
Minimum	25.855
Maximum	26.610
Range	0.754

## Notes:

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Total Sulfur (0.001%) Sample A							
Laboratory Code	Result 1	Result 2	Average	MU ( $\pm$ )	Z-Score	Method/Technique	
2	0.421	0.416	0.4185	0.038	0.39	ASRM D4239	
4	0.41	0.36	0.3850	0.03	-4.78	§	ASTM D3177
13	0.415	0.418	0.4165	0.03	0.08		AS 1038.6.3.3
16	0.417	0.415	0.4160	0.017	0.00		ASTM D4239-14E2
17	0.421	0.421	0.4210	0.03	0.77		AS 1038.6.3.3
20	0.407	0.406	0.4065	0.03	-1.46		AS 1038.6.3.3
22	0.428	0.424	0.4260	0.07	1.54		ASTM D3177-02/2007 ESCHKA Method
26	0.416	0.415	0.4155	0.03	-0.08		AS 1038.6.3.3
27	0.412	0.415	0.4135	0.046	-0.39		ASTM D4239-17
29	0.591	0.601	0.5960	#	27.75	§	ASTM D3177
31	0.408	0.413	0.4105	0.012	-0.85		ASTM D4239-17 Method A
40	0.405	0.418	0.4115	0.05	-0.69		AS 1038.6.3.3
41	0.439	0.427	0.4330	0.03	2.62	?	ISO 19579
46	0.409	0.409	0.4090	0.03	-1.08		AS 1038.6.3.3
53	0.415	0.417	0.4160	8% of Mean	0.00		ISO 19579
56	0.403	0.404	0.4035	0.02	-1.93		ASTM D4239-14
68A	0.41	0.42	0.4150	#	-0.15		CS Analyser (CS 2000)
69	0.430	0.430	0.4300	0.05	2.16	?	ISO 19579
70	0.413	0.423	0.4180	#	0.31		ISO 351

No of Results	19
Median	0.4160
Norm IQR	0.0065
Uncertainty (Median)	0.0019
Robust CV	1.6%
Minimum	0.385
Maximum	0.596
Range	0.211

## Notes:

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Total Sulfur (0.001%) Sample B							
Laboratory Code	Result 1	Result 2	Average	MU ( $\pm$ )	Z-Score	Method/Technique	
2	0.167	0.159	0.1630	0.038	0.18	ASRM D4239	
4	0.22	0.15	0.1850	0.04	1.52	ASTM D3177	
13	0.162	0.164	0.1630	0.03	0.18	AS 1038.6.3.3	
16	0.179	0.177	0.1780	0.017	1.09	ASTM D4239-14E2	
17	0.156	0.167	0.1615	0.03	0.09	AS 1038.6.3.3	
20	0.158	0.159	0.1585	0.03	-0.09	AS 1038.6.3.3	
22	0.143	0.143	0.1430	0.07	-1.03	ASTM D3177-02/2007 ESCHKA Method	
26	0.173	0.173	0.1730	0.03	0.79	AS 1038.6.3.3	
27	0.158	0.157	0.1575	0.044	-0.15	ASTM D4239-17	
29	0.333	0.333	0.3330	#	10.49	§	ASTM D3177
31	0.151	0.148	0.1495	0.010	-0.64		ASTM D4239-17 Method A
40	0.150	0.155	0.1525	0.05	-0.45		AS 1038.6.3.3
41	0.189	0.184	0.1865	0.03	1.61		ISO 19579
46	0.160	0.160	0.1600	0.03	0.00		AS 1038.6.3.3
53	0.148	0.148	0.1480	8% of Mean	-0.73		ISO 19579
56	0.152	0.151	0.1515	0.02	-0.52		ASTM D4239-14
68A	0.12	0.12	0.1200	#	-2.43	?	CS Analyser (CS 2000)
69	0.153	0.159	0.1560	0.05	-0.24		ISO 19579
70	0.178	0.173	0.1755	#	0.94		ISO 351

No of Results	19
Median	0.1600
Norm IQR	0.0165
Uncertainty (Median)	0.0047
Robust CV	10.3%
Minimum	0.120
Maximum	0.333
Range	0.213

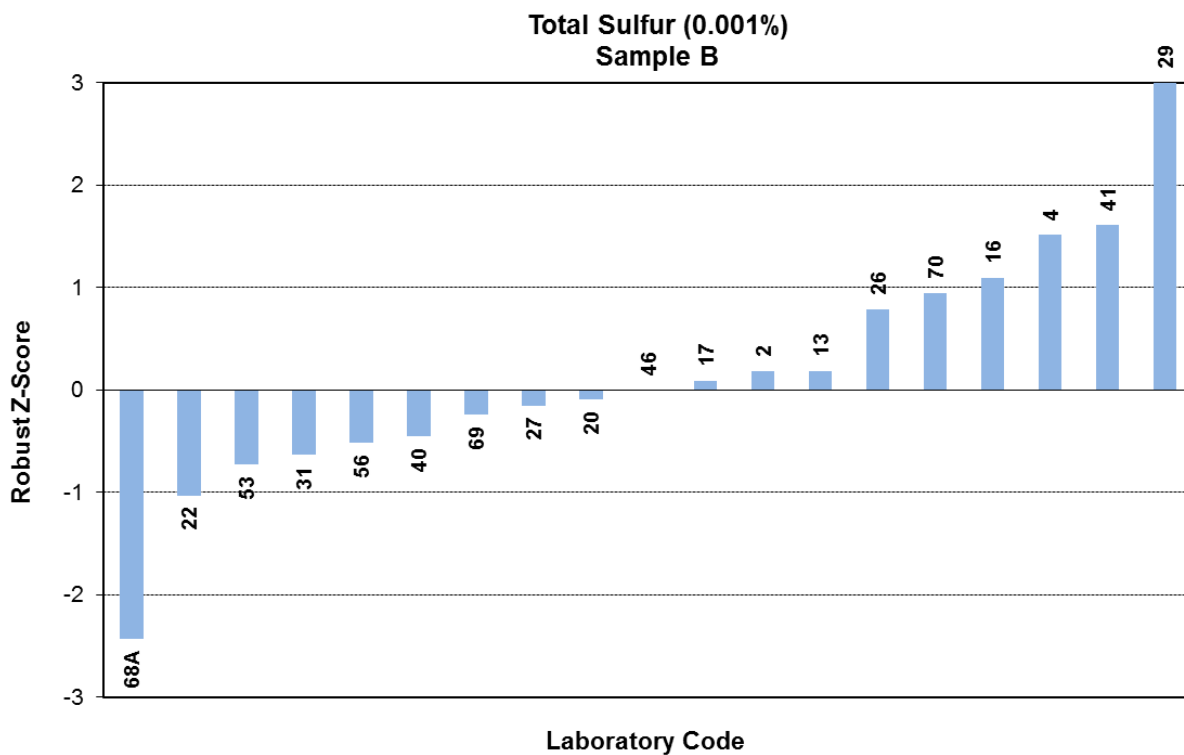
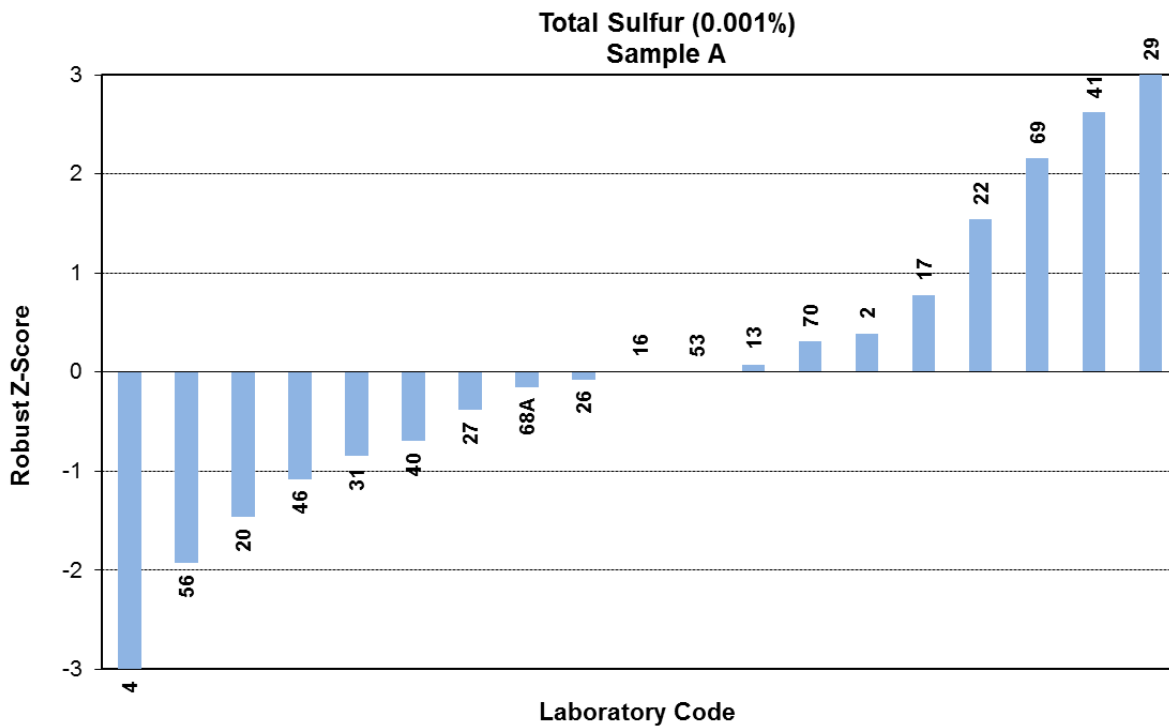
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"§" indicates an outlier, i.e.  $|z\text{-score}| \geq 3.0$





# **APPENDIX B**

## **Homogeneity Testing**

### **Homogeneity Testing**

For Coal round 37 Proficiency Testing Program, participants were supplied with two 125g coal samples (Sample A and Sample B). Homogeneity testing was performed on 8 randomly-selected A and B samples. Statistical analysis of the results indicated that no notable sample variability existed. Therefore, any outlier results identified could not be attributed to sample variability.

#### **HOMOGENEITY TEST (ASH) FOR PTA SAMPLE A**

	<b>Ash (dry) % Duplicate (a)</b>	<b>Ash (dry) % Duplicate (b)</b>	<b>Ash (dry) % Average</b>
1	9.60	9.62	9.61
2	9.53	9.52	9.52
3	9.57	9.62	9.60
4	9.53	9.58	9.56
5	9.56	9.56	9.56
6	9.54	9.62	9.58
7	9.52	9.57	9.54
8	9.52	9.62	9.57

#### **HOMOGENEITY TEST (ASH) FOR PTA SAMPLE B**

	<b>Ash (dry) % Duplicate (a)</b>	<b>Ash (dry) % Duplicate (b)</b>	<b>Ash (dry) % Average</b>
1	14.40	14.45	14.43
2	14.41	14.37	14.39
3	14.39	14.39	14.39
4	14.37	14.44	14.41
5	14.41	14.42	14.41
6	14.44	14.39	14.41
7	14.40	14.36	14.38
8	14.57	14.54	14.56

# APPENDIX C

## Documentation

Instructions to Participants .....	C1
Results Sheet .....	C2

## PROFICIENCY TESTING AUSTRALIA

### COAL PROFICIENCY TESTING PROGRAM (ROUND 37)

#### 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 ISO standards (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 ISO or in-house method number (include part numbers, e.g. ISO 562:2010 Determination of Volatile Matter.)
  - 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 **10 August 2018** to:

**Emilia Cincu**  
**Proficiency Testing Australia**  
**PO Box 7507**  
**SILVERWATER NSW 2128**  
**Phone: 02 9736 8397**  
**Fax: 02 9743 6664**  
**Email: emilia.cincu@pta.asn.au**

**PROFICIENCY TESTING AUSTRALIA  
COAL PROFICIENCY TESTING PROGRAM (ROUND 37)**

**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 ( $\pm$ )	Result 1	Result 2	MU ( $\pm$ )	
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: Dr Emilia Cincu, Fax: +61 2 9743 6664, Email: emilia.cincu@pta.asn.au by **10 August 2018**

*- End of Report -*