



# **BITUMEN (ROUND 1)**

## **PROFICIENCY TESTING PROGRAM**

**OCTOBER 2009**

**AMENDED REPORT – REPORT NO. 633B**  
**(Issued November 2011)**

### **ACKNOWLEDGEMENTS**

PTA wishes to gratefully acknowledge the technical assistance provided for this program by Mr B Chik of SAMI Bitumen Technologies (Camellia). This assistance included preparation and homogeneity testing of the sample, along with input into the design of the program, technical advice and discussion in the final report.

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**PROFICIENCY TESTING AUSTRALIA**  
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## 1. FOREWORD

This report summarises the results for Round 1 of a series of proficiency testing programs on the analysis of Bitumen.

The exercise was conducted by Proficiency Testing Australia in August 2009. The aim of the program was to assess laboratories' ability to competently perform the tests examined. The Program Coordinator was Mr R Sultan.

## 2. FEATURES OF THE PROGRAM

- a) A total of 12 laboratories were sent samples for the program. All laboratories returned results to be included in the final report.
- b) Participating laboratories were supplied two 800 mL samples of Bitumen labelled PTA Sample A and PTA Sample B.
- c) The following tests were performed on each sample:

### **PTA Sample A:**

Viscosity @ 135°C  
Density at 15C (bottle)  
Penetration 25°C, 100g, 5s

### **PTA Sample B:**

Vacuum Viscosity @ 60°C (recommended to be tested before RTFOT)  
RTFOT Viscosity Change (including the actual value after treatment)

- 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 any significant 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," both of which were distributed to participants with the sample (Appendix C).
- f) Each laboratory was randomly allocated a unique code number for the program to enable confidentiality of results. Reference to each laboratory in this report is made by its code number.

### 3. FORMAT OF APPENDICES

#### APPENDIX A

Appendix A lists all the results reported by the participating laboratories for each test.

Calculated robust z-scores, z-score charts and a table of robust statistics - number of results, median, normalised interquartile range (IQR), robust coefficient of variation (CV), minimum, maximum and range, are presented for the following tests:

- Viscosity @ 135°C
- Density at 15.0 °C
- Penetration 25.0 °C, 100g, 5s
- Viscosity @ 60.0°C
- RTFOT Viscosity Change (no z-score calculation)

The remaining tests are included in the report to assist PTA and the Technical Adviser to review results and are presented for information purposes only.

#### APPENDIX B

Contains details of sample preparation and homogeneity testing data.

#### APPENDIX C

Contains a copy of the “Instructions to Participants” and the “Results Sheet” supplied to participants.

### 4. STATISTICAL DESIGN OF THE PROGRAM

Robust statistical procedures were used to generate the z-scores and summary statistics for the sample. Summary statistics include:

- number of results;
- median;
- normalised IQR;
- robust CV;
- minimum;
- maximum; and
- range.

Robust z-scores were calculated for each laboratory based on the average of results submitted for each analyte. Where only one result was submitted, that result was used to calculate the z-score.

**TABLE A: Summary Statistics**

<b>Test</b>	<b>No. of Results</b>	<b>Median</b>	<b>Normalised IQR</b>
Viscosity @ 135°C	8	0.3935	0.0178
Density @ 15°C	7	1044.8	0.6
Penetration @ 25.0 °C, 100g, 5s	10	71.00	2.59
Viscosity @ 60.0 °C	9	205.0	6.7
RTFOT Viscosity Change (including actual value after treatment)	7	182.0	2.6

## 5. 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 three.

For each laboratory and for each sample, a single robust z-score was calculated. A single robust z-score (denoted by Z) for a laboratory's sample A would then be:

$$Z = \frac{A - \text{median}(A)}{\text{NormIQR}(A)}$$

For further information on the calculation and interpretation of z-scores, please see the *Guide to Proficiency Testing Australia (2008)*<sup>1</sup>.

**TABLE B: Outlier Results**

<b>Sample A Test</b>	<b>Lab Code</b>
Viscosity @ 135°C	3 and 11
Density @ 15°C	5
Penetration @ 25.0 °C, 100g, 5s	7

<b>Sample B Test</b>	<b>Lab Code</b>
Viscosity @ 60.0 °C	---
RTFOT Viscosity Change (including actual value after treatment)	No statistics performed

Note: --- indicates that no laboratories received outliers for this test

## 6. PTA AND TECHNICAL ADVISER'S COMMENTS

### General Comments

Of the 12 participating laboratories, 4 laboratories have been identified as having reported one outlier result each. Of the 43 total results, 4 outliers were reported, therefore 9.30% of the total results have been identified as outlier results. The RTFOT test results are not included in these calculations due to no z-scores being calculated.

For the Viscosity @ 135°C, Laboratory's 12 and 13 did not report viscosity in Pascal Seconds (Pa.s) so their results were not included in the statistics. A z-score calculation will be performed on laboratories 12 and 13 results and a summary sheet issued once the values are converted over.

No z-scores were performed on the RTFOT test because there is too much error involved resulting in unfair outlier allocation.

The majority of outliers were outside the repeatability/reproducibility values stated in the relevant AS2341 series. Therefore, laboratories reporting to Australian Standards repeatability/reproducibility values who obtained outliers should initiate an investigation. In addition, any laboratories reporting results with a z-score between 2 and 3 are encouraged to review their procedures. Outlier laboratories not only need to review their method but perhaps ensure results are to the correct basis or value.

The specified method for performing the tests was AS2341 (relevant sections). The majority of participating laboratories followed this method. Other methods reported included relevant ASTM and in-house methods.

## 7. REFERENCES

[1] "Guide to Proficiency Testing Australia" – 2008 (this document is located on the PTA website at [www.pta.asn.au](http://www.pta.asn.au) under Programs / Documents).

[2] AS/NZS 2341.0:2002 – *Methods of testing bitumen and related roadmaking products.*

# **APPENDIX A**

## **Summary of Results and Robust Z-Score Charts**

Viscosity @ 135°C (Pa.s)				
Lab Code	Result	MU	Robust Z-Score	Method / Technique
1	0.390	---	-0.20	MAT - TP652
2	0.397	0.012	0.20	P & S T61
3	0.467		4.13	Q343
4	---	---		---
5	---	---		---
6	0.375	---	-1.04	AS2341.21
7	0.429	---	2.00	AS2341.4
8	0.404	2%	0.59	AS2341.4
10	0.390	---	-0.20	AG:PT/T111
11	0.320	---	-4.13	---
12	354 (cst)	---	N/A	ASTM-D2170
13	385 mm <sup>2</sup> /s	19	N/A	AS2341.3

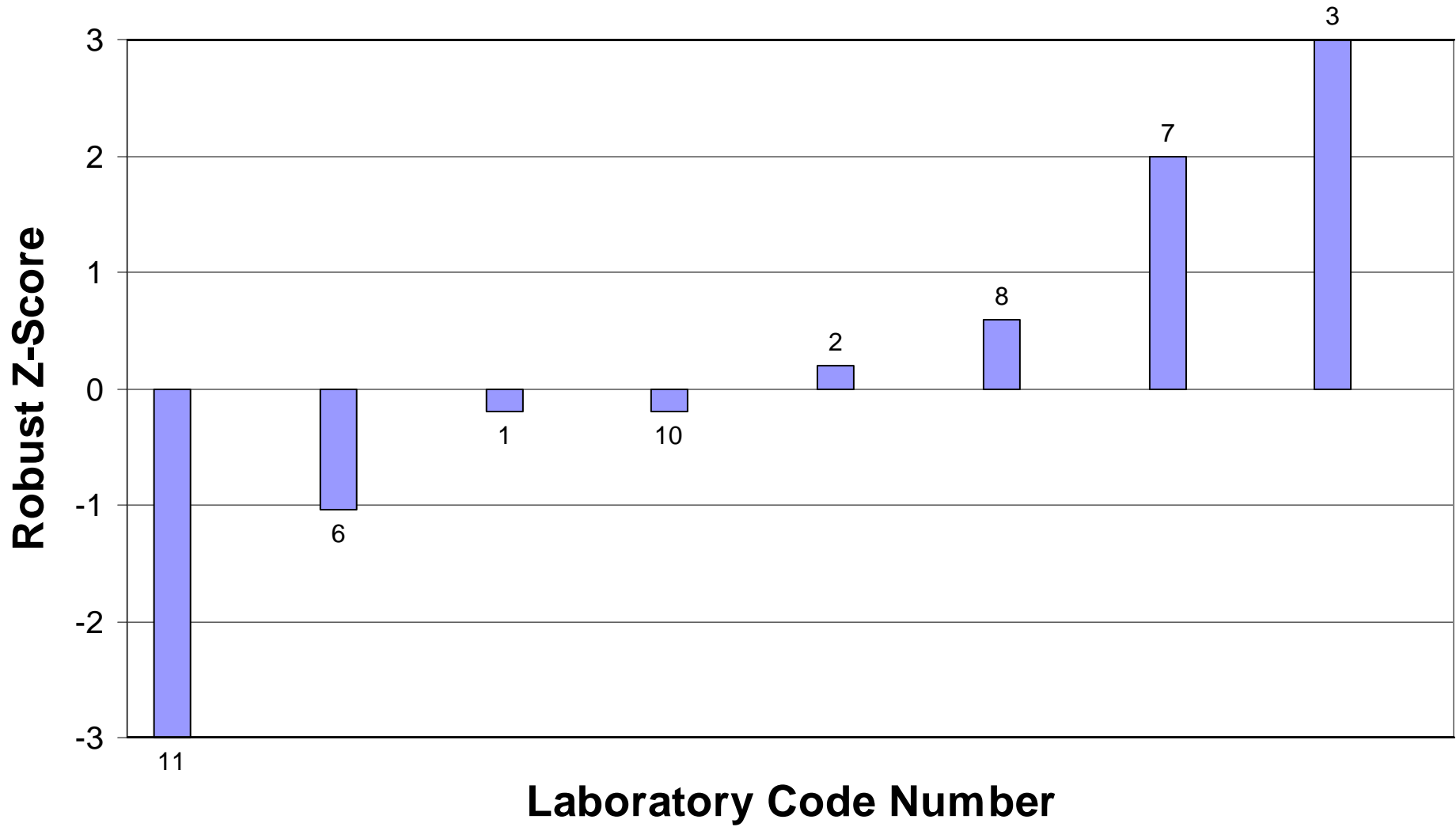
No. of Results	8
Median	0.3935
Normalised IQR	0.0178
Robust CV	4.5%
Minimum	0.320
Maximum	0.467
Range	0.147

**Note:** --- denotes that the laboratory left this space blank

**Note:** § denotes an outlier (i.e. |z-score| > 3)

**Note:** N/A denotes that the laboratory did not report the result in the correct units and therefore not included in the statistics.

## Viscosity @ 135°C (Pa.s)



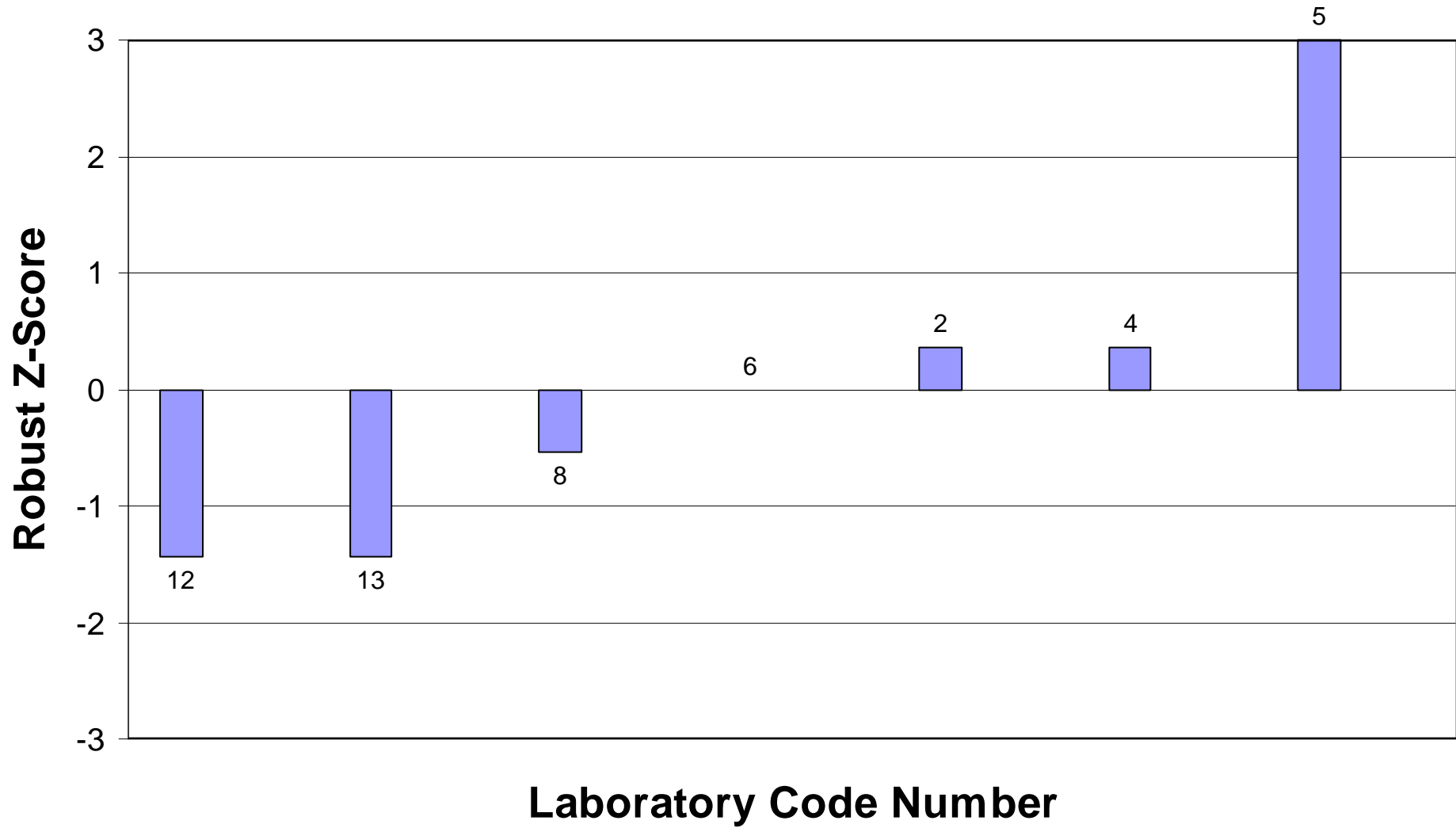
Density at 15.0C (bottle) Kg/m <sup>3</sup>				
Lab Code	Result	MU	Robust Z-Score	Method / Technique
1	---	---		---
2	1045	1	0.36	P & S T65
3	---	---		---
4	1045	---	0.36	AS2341.7
5	1047	0.8	4.50	AS2341.7
6	1045		0.00	AS2341.7
7	---	---		---
8	1045	2%	-0.54	AS2341.7
10	---	---		---
11	---	---		---
12	1044	---	-1.44	ASTM D - 70
13	1044	0.003	-1.44	ASTM 70:09

No. of Results	7
Median	1044.8
Normalised IQR	0.6
Robust CV	0.05%
Minimum	1044
Maximum	1047
Range	3

**Note:** --- denotes that the laboratory left this space blank

**Note:** § denotes an outlier (i.e. |z-score| > 3)

### Density at 15.0C (bottle) Kg/m<sup>3</sup>



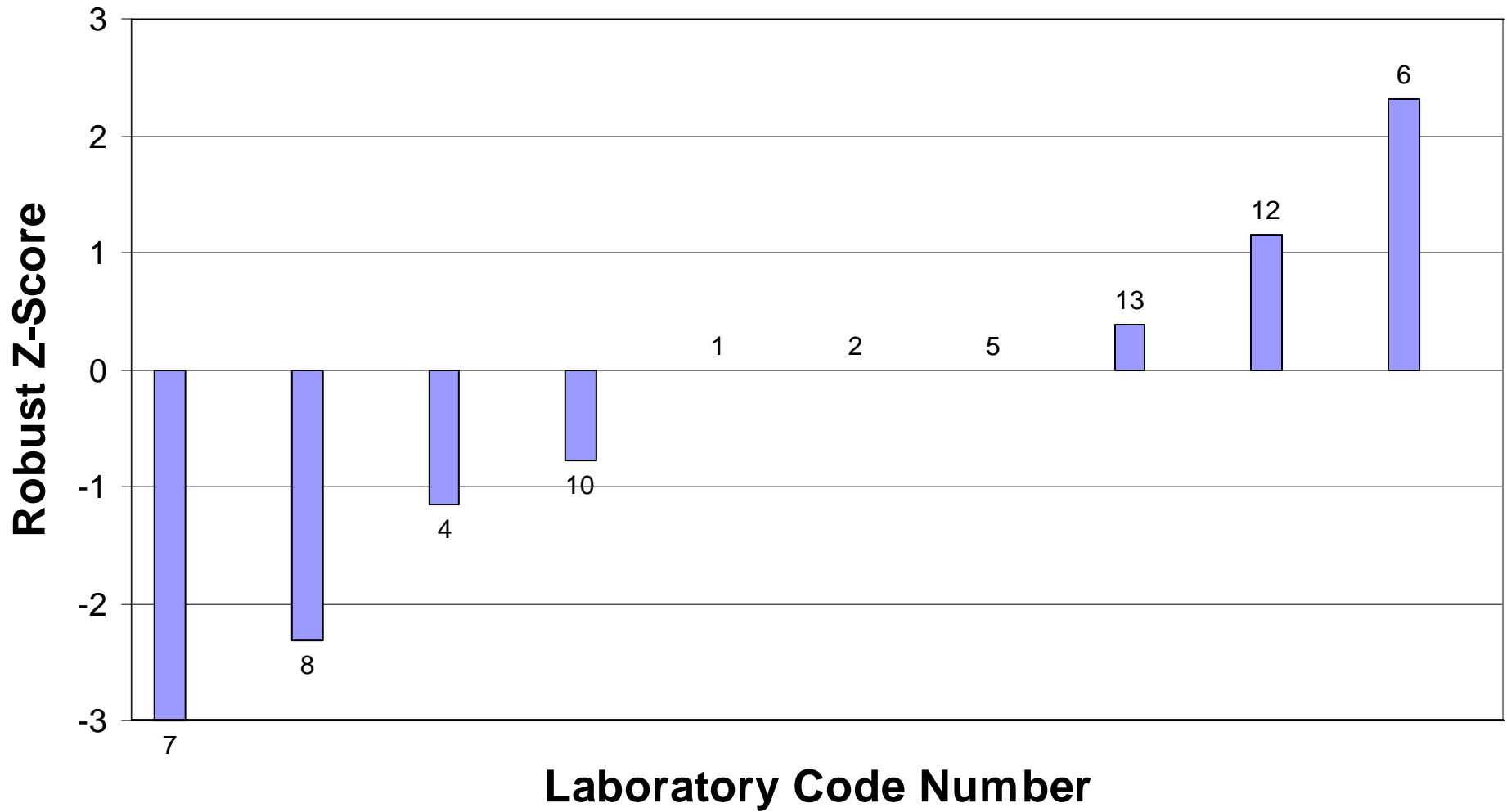
Penetration 25.0°C, 100g, 5s				
Lab Code	Result	MU	Robust Z-Score	Method / Technique
1	71.0	---	0.00	AS2341-12
2	71.0	2	0.00	AS2341.12
3	---	---		---
4	68.0	---	-1.16	AS2341.12
5	71.0	2.5	0.00	AS2341.12
6	77.0	---	2.31	AS2341.12
7	60.5		-4.05	AS2341.12
8	65.0	2	-2.31	AS2341.12
10	69.0	---	-0.77	AS2341.12
11	---	---		---
12	74.0	0.1mm(near value)	1.16	ASTM D - 5
13	72.0	2.3	0.39	ASTM 05:06

No. of Results	10
Median	71.00
Normalised IQR	2.59
Robust CV	3.7%
Minimum	60.5
Maximum	77.0
Range	16.5

**Note:** --- denotes that the laboratory left this space blank

**Note:** § denotes an outlier (i.e. |z-score| > 3)

### Penetration 25.0°C, 100g, 5s

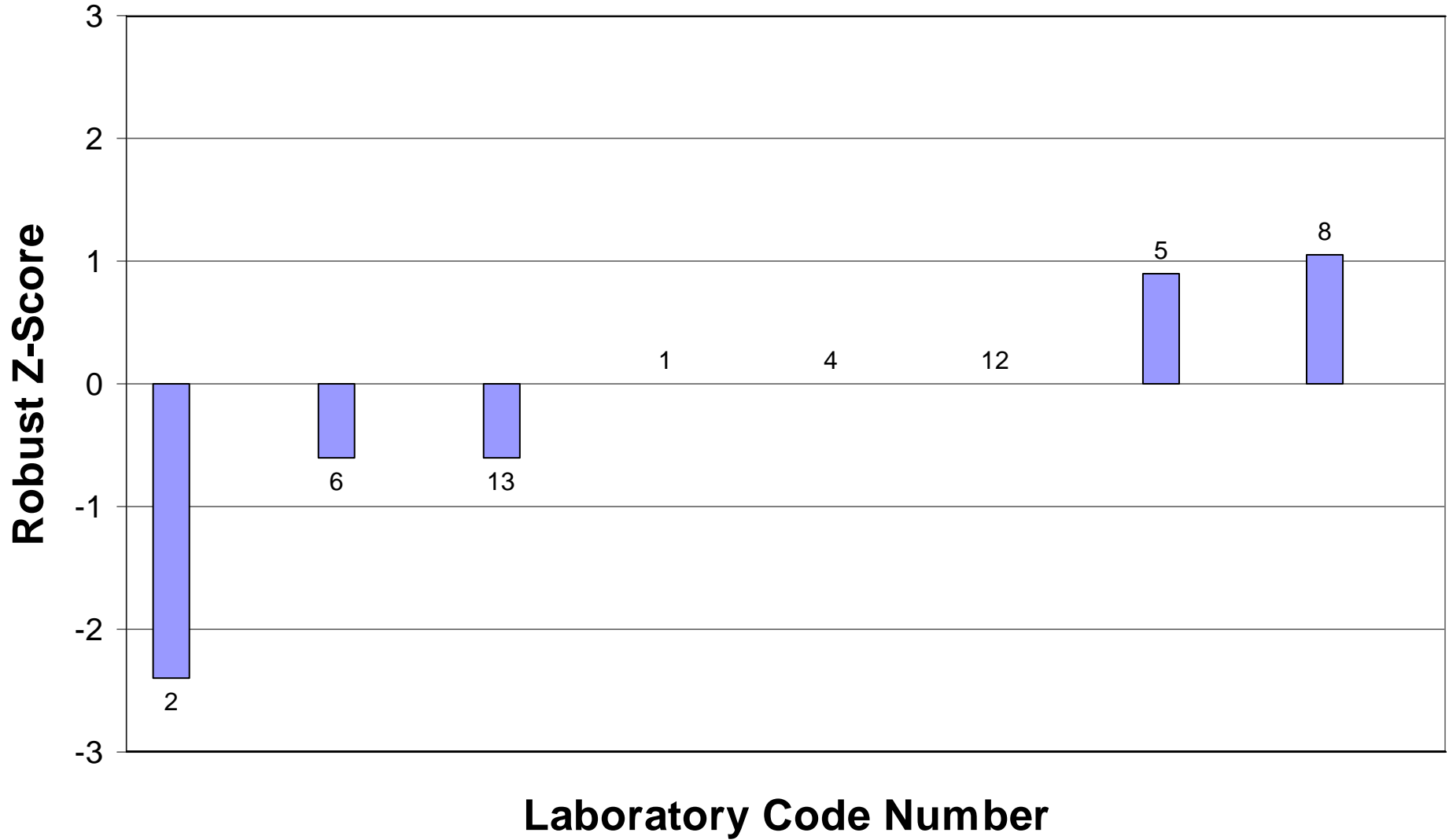


Viscosity @ 60°C				
Lab Code	Result	MU	Robust Z-Score	Method / Technique
1	205	---	0.00	MAT-TP652
2	189	3.9	-2.40	P & S T61
3	210	Not verified		AS2341.2
4	205	---	0.00	AS2341.2
5	211	3.5 Pas	0.90	AS2341.2
6	201	---	-0.60	AS2341.2
7	---	---		---
8	212	4	1.05	AS2341.2
10	---	---		---
11	---	---		---
12	205	---	0.00	ASTM D-2171
13	201	10	-0.60	AS2341.2

No. of Results	9
Median	205.0
Normalised IQR	6.7
Robust CV	3.3%
Minimum	189
Maximum	212
Range	23

**Note:** --- denotes that the laboratory left this space blank

### Viscosity @ 60°C



RTFOT Viscosity Change			
Lab Code	Result	MU	Method / Technique
1	181	---	AS2341-10 (MAT-TP652)
2	184	10 (8)	AS2341.10 (P&S T61)
3	---	---	---
4	186	---	AS2341.2 & 4S.2341.10 AS2341.10
5	182	5 (3.5)	AS2341.2
6	---	---	---
7	---	---	---
8	174	5	AS2341.10, AS2341.2
10	---	---	---
11	---	---	---
12	199	---	ASTM - D 287 2/ 2171
13	182	31	AS2341.10

No. of Results	7
Median	182.0
Normalised IQR	2.6
Robust CV	1.4%
Minimum	174
Maximum	199
Range	25

**Note:** --- denotes that the laboratory left this space blank

<b>Lab Code</b>	<b>i) Shear rates for all viscosities Tested a) @ 135.0°C and b) @ 60.0°C</b>	<b>ii) Partial filling method or Total filling method in the determination of density at 15°C</b>	<b>iii) a) If Cannon-Manning viscometer used or b) Asphalt Institute viscometer and / or c) make and model of rotational viscometer</b>
<b>1</b>	---	---	c) HAAKE VISCOBALANCE
<b>2</b>	a) 20s-1 and b) 2.8 s-1	Partial	c) Contraves Rheomat 15T
<b>3</b>	a) 10.2 s-1 and b) 2.43 s-1	---	b) Asphalt institute and c) Brookfield DU-ii Pro
<b>4</b>	b) 6.62 s-1	Partial	a) Cannon-Manning
<b>5</b>	b) Pre 2.381, post 1.308	Partial	b) Asphalt institute
<b>6</b>	a) 1037.4404 b) 6.4330	Partial	a) Cannon-Manning
<b>7</b>	a) 20s-1	---	c) Brookfield DU-ii Pro
<b>8</b>	a) 20s-1	Partial	---
<b>10</b>	a) 100rpm	---	---
<b>11</b>	---	---	Brookfield LVT
<b>12</b>	---	---	a) Cannon
<b>13</b>	a) 10.2 and b) 1.9	Partial	a) Cannon

**Note:** --- denotes that the laboratory left this space blank

## **APPENDIX B**

# **Sample Preparation and Homogeneity Testing**

### HOMOGENEITY TESTING

Ten samples were selected at random and analysed in duplicate for Penetration at 25°C (AS2341.12) and Viscosity @ 135°C (AS2341.4) . 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.

PTA HOMOGENIETY TESTING					
Sample No.	Penetration at 25C AS2341.12			Viscosity at 135C AS2341.4	
	Results p.u.	Deviation		Results Pa.s.	Deviation
62	66	-0.9174		0.405	-0.37175
72	65	0.6116		0.408	-1.11524
45	65	0.6116		0.407	-0.86741
43	66	-0.9174		0.402	0.37175
1	65	0.6116		0.405	-0.37175
24	65	0.6116		0.408	-1.11524
74	66	-0.9174		0.402	0.37175
90	65	0.6116		0.400	0.86741
19	65	0.6116		0.400	0.86741
10	66	-0.9174		0.398	1.36307
Mean	65.4			0.4035	
%CV		0.8%			0.9%

Water bath temperature checked = 25.0C

Thermosel temperature checked = 135.0C

Note: Deviation lies within test method requirements.

# **APPENDIX C**

**Instructions to Participants**

**and**

**Results Sheet**

## PROFICIENCY TESTING AUSTRALIA

### BITUMEN PROFICIENCY TESTING PROGRAM ROUND 1

#### 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. 2 x 800 ml samples labelled PTA Sample A and PTA Sample B are provided to each laboratory.
2. The following tests are to be conducted:

**PTA Sample A:**

Viscosity @ 135°C  
Density at 15C (bottle)  
Penetration 25°C, 100g, 5s

**PTA Sample B:**

Vacuum Viscosity @ 60°C (recommended to be tested before RTFOT)  
RTFOT Viscosity Change (including the actual value after treatment)

3. Participants can perform any or all of the above mentioned tests.
4. If performing both 'Sample B' tests it is recommended to perform the Vacuum Viscosity @ 60°C test first.
5. Participants should use the routine test methods which would normally be used to test samples. (The Australian Standard method AS 2341 is the preferred method). Please identify the method used on the Results Sheet.
6. Laboratories are also requested to calculate and report an estimate of uncertainty of measurement for each reported measurement result if possible. All estimates of uncertainty of measurement must be given as a 95% confidence interval (coverage factor  $k \approx 2$ ) and reported in  $\pm$  reporting unit basis.
7. The results for all determinations are to be recorded on the results sheet to the accuracy and reporting basis indicated.
8. Testing may commence as soon as the sample is received. All laboratories must return the results sheet no later than **Friday 21 August 2009** to:

Ray Sultan  
Proficiency Testing Australia  
PO Box 7507  
SILVERWATER NSW 2128  
Phone: 02 9736 8397  
Fax: 02 9743 6664  
Email: [rsultan@pta.asn.au](mailto:rsultan@pta.asn.au)

**PROFICIENCY TESTING AUSTRALIA  
BITUMEN PROFICIENCY TESTING PROGRAM  
ROUND 1**

**RESULTS SHEET**

Lab Code

PTA  
SAMPLE A

TEST (report to)	Result	MU ±	Method
Viscosity @ 135.0°C			
Density at 15.0C (bottle)			
Penetration 25.0°C, 100g, 5s			

PTA  
SAMPLE B

TEST (report to)	Result	MU ±	Method
Viscosity @ 60.0°C			
RTFOT Viscosity Change (including the actual value after treatment)			

If rotational viscometer is used for any viscosity determination, please report make and model \_\_\_\_\_

Also please report:

i) Shear rates for all viscosities tested: @ 135.0°C \_\_\_\_\_ @ 60.0°C \_\_\_\_\_

ii) Partial filling method or Total filling method in the determination of density at 15°C \_\_\_\_\_

iii) If Cannon-Manning viscometers or Asphalt Institute viscometers are used. \_\_\_\_\_

Date of tests: \_\_\_\_\_ Signature: \_\_\_\_\_

Send results by Friday 21 August to:  
 Ray Sultan  
 Proficiency Testing Australia  
 PO Box 7507  
 SILVERWATER NSW 2128  
 Phone: 02 9736 8397  
 Fax: 02 9743 6664  
 Email: [rsultan@pta.asn.au](mailto:rsultan@pta.asn.au)

----- End of report -----