



Report No. 798

Bitumen Proficiency Testing

Round 4

February 2013

Acknowledgments

PTA wishes to gratefully acknowledge the technical assistance and supply of samples for this program by Mr B Chik of SAMI Bitumen Technologies Pty Ltd.

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CONTENTS

1. FOREWORD	1
FEATURES OF THE PROGRAM	1
3. FORMAT OF THE APPENDICES	2
4. STATISTICAL DESIGN OF THE PROGRAM.....	2
Table A: Summary Statistics.....	4
5. PTA AND TECHNICAL ADVISOR’S COMMENTS	5
Table B: Comparison of Robuct CVs and Percentage of Outliers	5
6. OUTLIER RESULTS.....	7
Table C: Summary of Statistical Outliers	7
7. REFERENCE.....	7

APPENDIX A – Results and Data Analysis

Viscosity at 135.0°C (Pa S).....	A1
Density at 15.0°C (bottle) (Kg/m ³).....	A2
Penetration 25.0°C, 100g, 5s (p.u).....	A3
Softening Point °C.....	A4
Viscosity at 60.0°C (Pa S).....	A5
RTFOT Viscosity Change (%).....	A6
RTFOT Viscosity after treatment (Pa S).....	A7
Additional Information.....	A8

APPENDIX B – Homogeneity Testing

Homogeneity Testing.....	B1
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APPENDIX C – Documentation

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

1. FOREWORD

This report summarises the results of a proficiency testing program on the determination of selected chemical tests of bitumen. It constitutes the fourth round of an ongoing series of programs.

The program was conducted in December 2012 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 Advisor was Mr B Chik of SAMI Bitumen Technologies Pty Ltd. This report was authorised by Mrs F Watton, PTA Quality - Business Development Manager.

2. FEATURES OF THE PROGRAM

- (a) Participants were provided with two 800ml samples of bitumen labelled Sample A and Sample B.
- (b) A total of 15 laboratories received samples, comprising:
 - 14 Australian participants; and
 - 1 overseas participant from Singapore.

Of these 15 laboratories, 3 were 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, 2012* (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 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 “§”.

(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 significant figures) 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;
- 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	No. of Results	Median	Normalised IQR
Viscosity @ 135.0°C (Pa S)	11	0.4000	0.0089
Density @ 15.0°C (bottle) (Kg/m ³)	10	1038.20	4.95
Penetration 25.0°C, 100g, 5s (p.u)	12	66.0	1.9
Softening Point °C	9	48.5	0.9
Viscosity @ 60.0°C (Pa S)	10	196.5	3.8
RTFOT Viscosity Change (%)	9	190.0	4.3
RTFOT Viscosity after treatment (Pa S)	9	368.0	12.2

5. PTA AND TECHNICAL ADVISOR'S COMMENTS

Of the 12 laboratories that submitted results for inclusion in the final report four laboratories received an outlier. Therefore, of the 72 results returned for z-score analysis, 5.5% have been identified as outlier results. Any laboratories reporting outliers are encouraged to review their procedures.

The following table gives a comparison of the robust CVs for tests common to previous programs.

TABLE B: COMPARISON OF ROBUST CVs AND PERCENTAGE OF OUTLIERS

Test	Round 2		Round 3		Round 4	
	CV	% outliers	CV	% outliers	CV	% outliers
Viscosity at 135.0°C (Pa S)	3.9%	--	5.3%	--	2.2%	9.1
Density at 15.0°C	0.2%	7.7	0.3%	--	0.5%	--
Penetration at 25.0°C, 100g, 5s (p.u)	5.7%	7.1	3.4%	6.3	2.9%	8.3
Viscosity @ 60.0 °C (Pa S)	1.6 %	8.3	2.5%	12.5	1.9%	10.0
RTFOT Viscosity Change	2.3%	10.0	1.8%	--	2.3 %	11.1
RTFOT Viscosity after treatment (Pa S)	4.3%	11.1	3.8%	8.3	3.3%	--
Softening Point °C	~	~	1.6%	14.3	1.8%	--

Note: ~ indicates that this test was not offered previously.
-- indicates no outliers detected.

Comparison of robust CVs and percentage outliers from previous rounds demonstrates a fairly consistent spread of results. The percentage of outliers and CVs are relatively low throughout rounds two to four. This is indicative of the level of precision the participants have shown.

Any laboratories reporting results with an absolute 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.

Laboratory 5 has used the method ASTM D2872 for the RTFO test. It was decided that this method is not comparable to AS2341.10 as the air blowing time is 85 minutes compared to 60 minutes for AS2341.10. In addition, RTFO ovens made to US standard D2872 are now fitted with a big fan. It was therefore concluded that results with ASTM and AS methods could not be analysed together.

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.

The samples were taken from a bitumen storage tank at SAMI Bitumen Technologies Camellia, Sydney in 2012. The standard method AS2008 and other standards were used to prepare the samples. Certified reference materials used include calibration reports on reference thermometers. Calibrations were conducted using clean and fresh standard silicon oil.

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 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. The majority of tests returning results from the same method group were less than 11, therefore reliable conclusions cannot be drawn from analysing grouped methods on this occasion. For these tests results from all method groups have been pooled for analysis.

Results for Penetration returned at least 11 sets of results from the same method group, therefore results generated using these methods have been pooled for analysis. Method AS2341.12 was grouped for analysis for Penetration. The number of results was 11 with a median of 66.0 p.u and uncertainty of the median of 0.8 p.u.

The results of the grouped analysis compared to the consensus results show little or no variation.

6. OUTLIER RESULTS

Laboratories reporting outlier results are listed in the following table:

TABLE C: SUMMARY OF STATISTICAL OUTLIERS

Test	Laboratory Code No.
Viscosity @ 135.0°C (Pa S)	12
Density @ 15.0°C (bottle) (Kg/m ³)	--
Penetration 25.0°C, 100g, 5s (p.u)	24
Softening Point °C	--
Viscosity @ 60.0°C (Pa S)	4
RTFOT Viscosity Change (%)	27A
RTFOT Viscosity after treatment (Pa S)	--

7. Reference

- [1] *Guide to Proficiency Testing Australia*, 2012 (This document can be found on the PTA website, www.pta.asn.au).

APPENDIX A

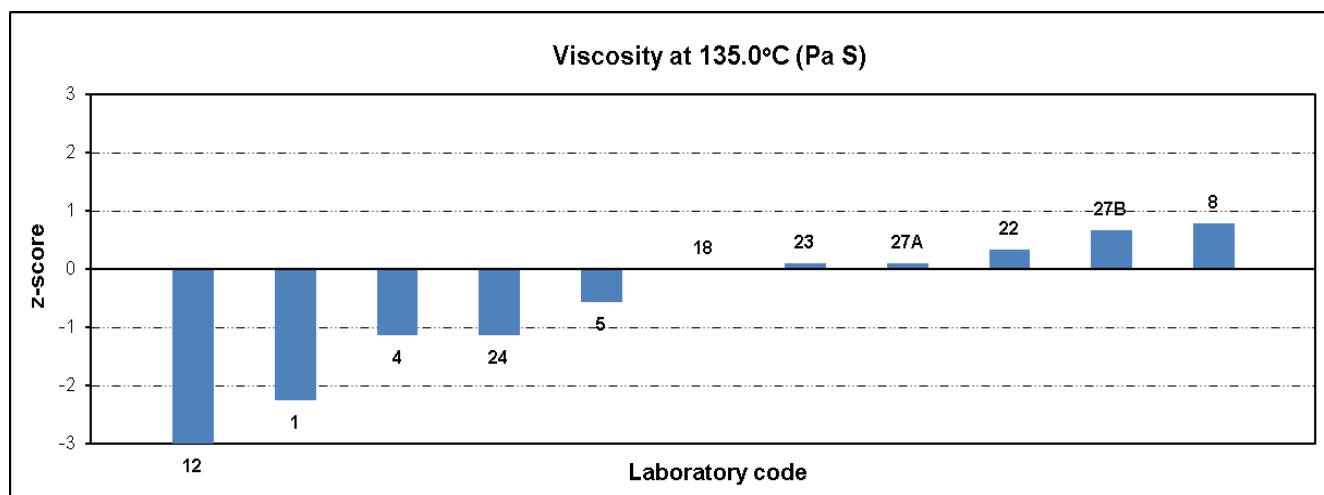
Results and Data Analysis

Viscosity at 135.0°C (Pa S).....	A1
Density at 15.0°C (bottle) (Kg/m ³).....	A2
Penetration 25.0°C, 100g, 5s (p.u).....	A3
Softening Point °C.....	A4
Viscosity at 60.0°C (Pa S).....	A5
RTFOT Viscosity Change (%).....	A6
RTFOT Viscosity after treatment (Pa S).....	A7
Additional Information.....	A8

Viscosity at 135.0°C (Pa S)				
Laboratory code	Result	MU	Robust z-score	Method
1	0.380	#	-2.25	AG/PT:T111
4	0.390 ±	3%	-1.12	AS2341.4
5	0.395	#	-0.56	ASTM D2171-10
8	0.407	#	0.79	AS2341.3
12	0.370	#	-3.37 §	AS2341.3
18	0.400 ±	0.048	0.00	AS2341.3
22	0.403 ±	2%	0.34	AS2341.4
23	0.401 ±	2%	0.11	AS2341.4
24	0.390	#	-1.12	AS2341.2
27A	0.401 ±	0.03	0.11	AS2341.4
27B	0.406	#	0.67	AS2341.4

Number of results	11
Median	0.4000
Uncertainty (median)	0.0034
Normalised IQR	0.0089
Robust CV	2.2%
Minimum	0.370
Maximum	0.407
Range	0.037

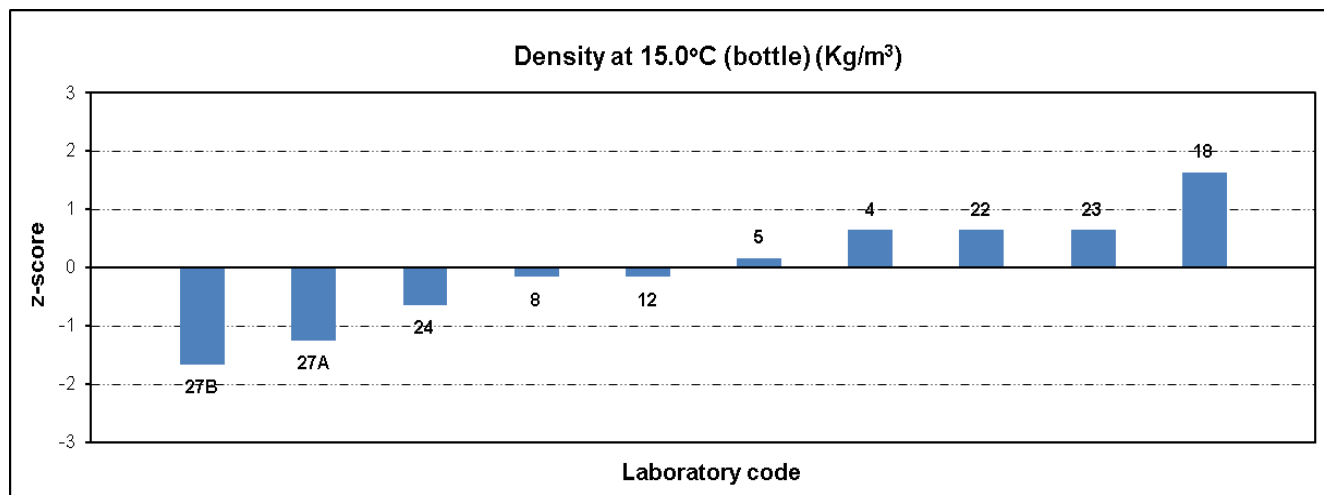
Note: A '#' indicates no response was provided.
 A '§' indicates an outlier.



Density at 15.0°C (bottle) (Kg/m ³)					
Laboratory code	Result	MU	Robust z-score	Method	
4	1041.4	± 0.08%	0.65	AS2341.7	
5	1039	#	0.16	ASTM D70-09 ^{e1}	
8	1037.4	#	-0.16	AS2341.7	
12	1037.4	#	-0.16	AS2341.7	
18	1046.3	± 0.9	1.64	AS2341.7	
22	1041.4	± 0.04%	0.65	AS2341.7	
23	1041.4	± 0.5	0.65	AS2341.7	
24	1035	#	-0.65	AS2341.7	
27A	1032	± 0.7	-1.25	AS2341.7	
27B	1030	#	-1.66	AS2341.7	

Number of results	10
Median	1038.20
Uncertainty (median)	1.96
Normalised IQR	4.95
Robust CV	0.5%
Minimum	1030.0
Maximum	1046.3
Range	16.3

Note: A '#' indicates no response was provided.

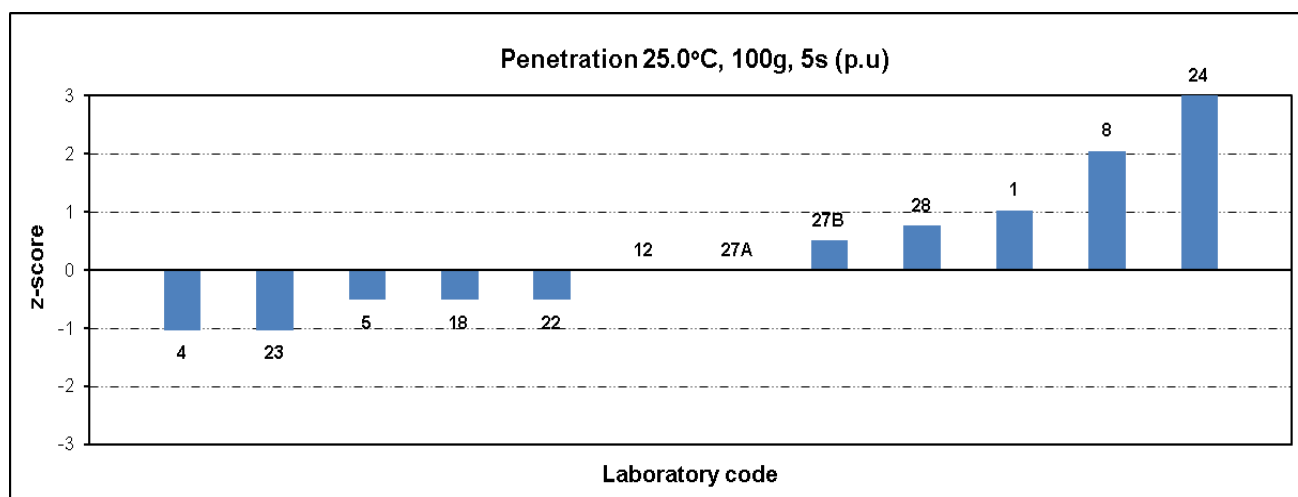


Penetration 25.0°C, 100g, 5s (p.u)				
Laboratory code	Result	MU	Robust z-score	Method
1	68	#	1.03	AS2341.12
4	64	± 3%	-1.03	AS2341.12
5	65	#	-0.51	ASTM D5-06 ^{e1}
8	70	#	2.06	AS2341.12
12	66	#	0.00	AS2341.12
18	65	± 4	-0.51	AS2341.12
22	65	± 3%	-0.51	AS2341.12
23	64	± 2%	-1.03	AS2341.12
24	80	#	7.19 §	AS2341.12
27A	66	± 0.66	0.00	AS2341.12
27B	67	#	0.51	AS2341.12
28	67.5	#	0.77	AS2341.12

Number of results	12
Median	66.0
Uncertainty (median)	0.7
Normalised IQR	1.9
Robust CV	2.9%
Minimum	64.0
Maximum	80
Range	16.0

Note: A '#' indicates no response was provided.

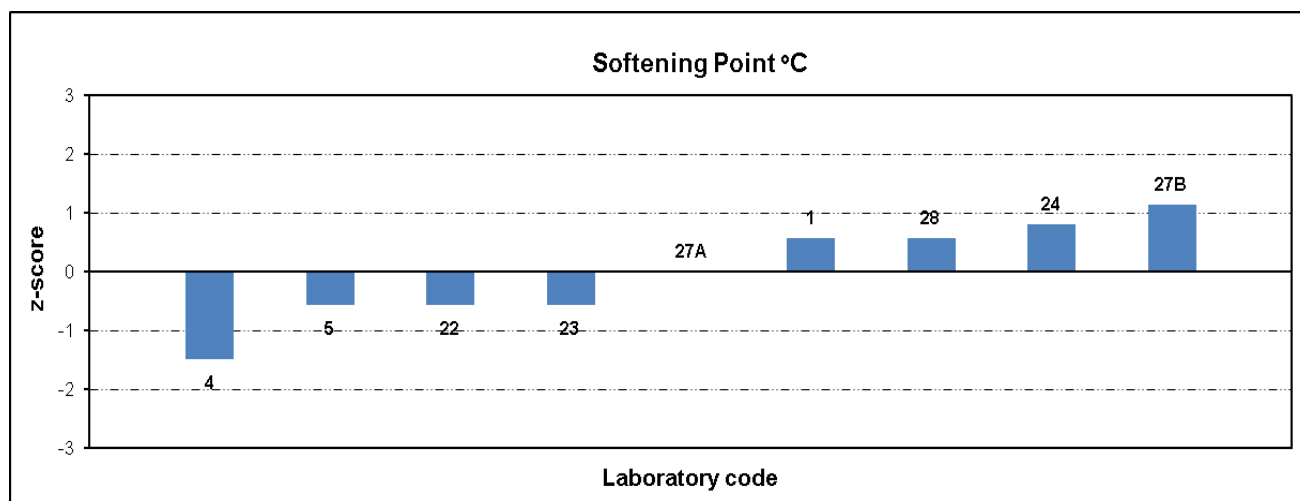
A '§' indicates an outlier.



Softening Point °C				
Laboratory code	Result	MU	Robust z-score	Method
1	49	#	0.57	AS2341.18
4	47.2	± 4%	-1.49	AS2341.18
5	48.0	#	-0.57	ASTM D36-12
22	48.0	± 3%	-0.57	AS2341.18
23	48.0	± 3%	-0.57	AS2341.18
24	49.2	#	0.80	AS2341.18
27A	48.5	± 1.2	0.00	AS2341.18
27B	49.5	#	1.14	AS2341.18
28	49.0	#	0.57	AS2341.18

Number of results	9
Median	48.5
Uncertainty (median)	0.4
Normalised IQR	0.9
Robust CV	1.8%
Minimum	47
Maximum	49.50
Range	2.30

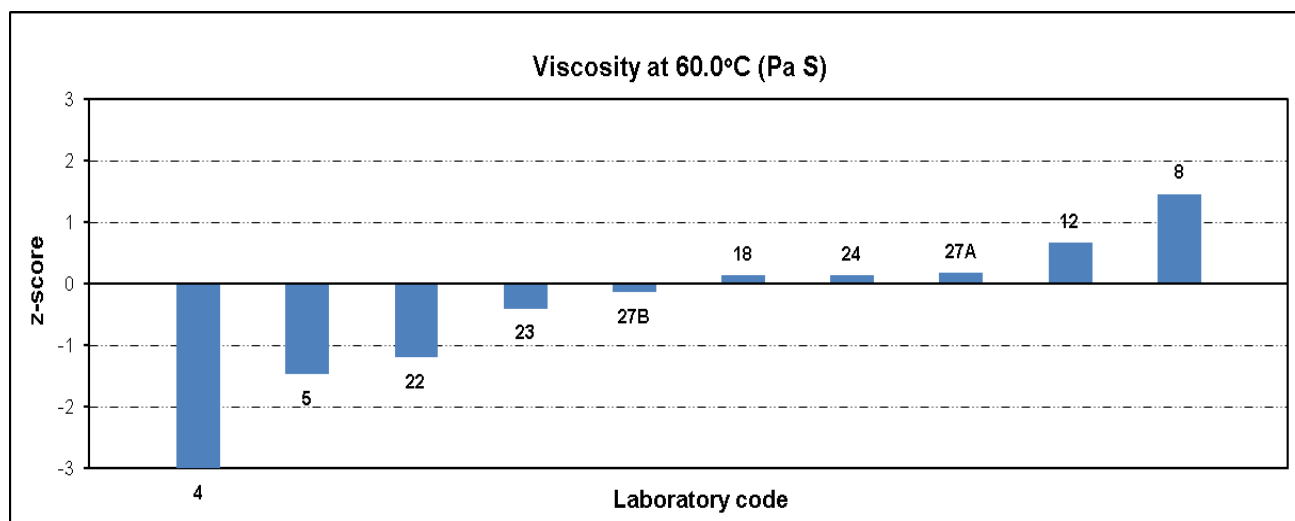
Note: A '#' indicates no response was provided.



Viscosity at 60.0°C (Pa S)					
Laboratory code	Result	MU	Robust z-score		Method
4	184	± 5%	-3.33	§	AS2341.2
5	191	#	-1.47		ASTM D2171- 10
8	202	#	1.47		AS2341.2
12	199	#	0.67		AS2341.2
18	197	± 23.6	0.13		AS2341.2
22	192	± 5%	-1.20		AS2341.2
23	195	± 5%	-0.40		AS2341.2
24	197.0	#	0.13		AS2341.2
27A	197.20	± 13.8	0.19		AS2341.2
27B	196	#	-0.13		AS2341.2

Number of results	10
Median	196.5
Uncertainty (median)	1.5
Normalised IQR	3.8
Robust CV	1.9%
Minimum	184
Maximum	202
Range	18

Note: A '#' indicates no response was provided.
A '§' indicates an outlier.



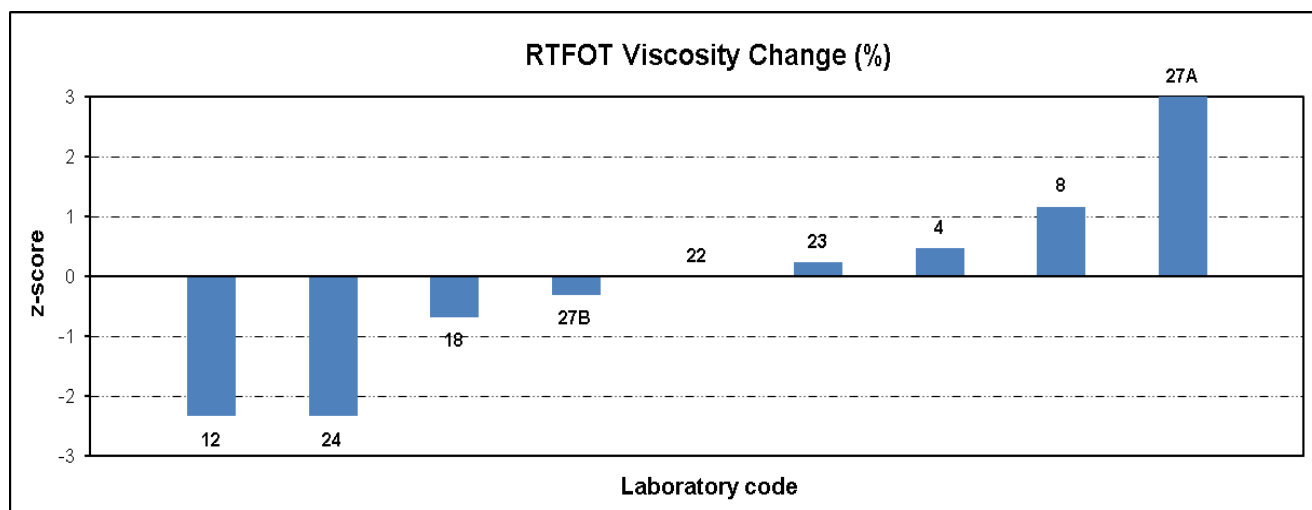
RTFOT Viscosity Change (%)				
Laboratory code	Result	MU	Robust z-score	Method
4	192	#	0.47	AS2341.2, AS2341.10
5	220*	#	Na*	ASTM D2872 / D2171
8	195	#	1.17	AS2341.2, AS2341.10
12	180	#	-2.34	AS2341.10
18	187.1 ± 22.4	#	-0.68	AS2341.10
22	190	#	0.00	AS2341.2, AS2341.10
23	191	#	0.23	AS2341.2, AS2341.10
24	180	#	-2.34	AS2341.10
27A	205	#	3.50 §	AS2341.2, AS2341.10
27B	188.7	#	-0.30	AS2341.2, AS2341.10

Number of results	9
Median	190.0
Uncertainty (median)	1.8
Normalised IQR	4.3
Robust CV	2.3%
Minimum	180
Maximum	205
Range	25

Note: A '#' indicates no response was provided.

A '§' indicates an outlier.

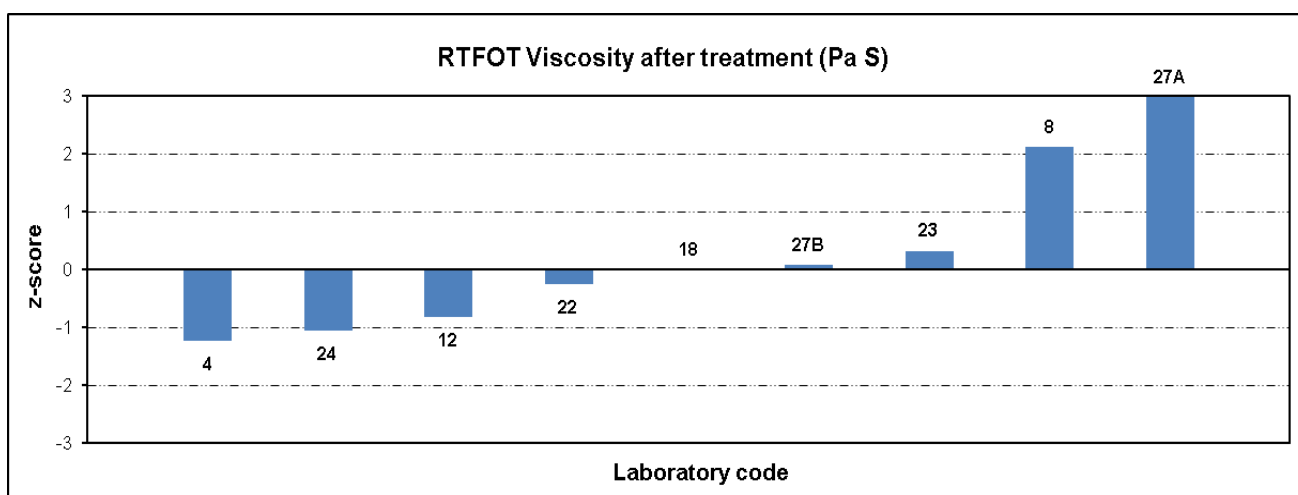
A '*' indicates this results was eliminated from the consensus as the method is not comparable to AS2341.10.



RTFOT Viscosity after treatment (Pa S)					
Laboratory code	Result	MU	Robust z-score	Method	
4	353 ±	4%	-1.23	AS2341.2, AS2341.10	
5	421*	#	Na*	ASTM D2872 / D2171	
8	394	#	2.12	AS2341.2, AS2341.10	
12	358	#	-0.82	AS2341.10	
18	368 ±	44.2	0.00	AS2341.10	
22	365 ±	5%	-0.25	AS2341.2, AS2341.10	
23	372 ±	6%	0.33	AS2341.2, AS2341.10	
24	355.0	#	-1.06	AS2341.10	
27A	404.47 ±	28.3	2.98	AS2341.2, AS2341.10	
27B	369	#	0.08	AS2341.2, AS2341.10	

Number of results	9
Median	368.0
Uncertainty (median)	5.1
Normalised IQR	12.2
Robust CV	3.3%
Minimum	353
Maximum	404
Range	51.47

Note: A '#' indicates no response was provided.
 A '*' indicates this results was eliminated from the consensus as the method is not comparable to AS2341.10.



Additional Information						
Laboratory Code	Measured RTFOT temperature change ($^{\circ}\text{C}$)	Make and model of rotational viscometer	Shear rates for all viscosities tested		Filling method in determination of density at 15°C	Cannon-Manning or Asphalt Institute viscometer used?
			135.0°C	60.0°C		
1	#	Brookfield DVII	112	#	#	#
4	163	LV Viscometer, Brookfield DV-II Pro for viscosity at 135°C	20.40 sec^{-1}	1.54 sec^{-1}	partial	AI
5	162.9	#	#	#	partial	135°C - Cannon, 60°C - AI
8	Digital sensor disconnected AS2341.10	#	#	#	partial	Cannon
12	163	#	#	1.869	partial	AIV
18	163	#	0.33	2.62	partial filling method	Cannon-Manning
22	163	Brookfield LVDVII for viscosity at 135°C	20.40 sec^{-1}	1.29 sec^{-1}	partial	AI
23	163	Brookfield Viscometer LVDVII for viscosity at 135°C	20.40 sec^{-1}	1.31 sec^{-1}	partial	AI
24	163.0	#	67200	#	partial	Asphalt Institute
27A	163	Brookfield / DV-II +	37.76 sec^{-1}	pre-RTFO = 1.074 sec^{-1} , post-RTFO = 0.83 sec^{-1}	partial	Asphalt Institute
27B	163	Brookfield - DV-II +	37.765 sec^{-1}	pre-RTFO = 1.235 sec^{-1} , post-RTFO = 0.9135 sec^{-1}	partial	Asphalt Institute
28	n/a	Brookfield DVII	112	#	#	#

APPENDIX B

Homogeneity Testing

Homogeneity Testing

PTA Homogeneity Testing --- 2012				
Sample no.	Penetration at 25°C AS2341.12		Viscosity at 135°C AS2341.4	
	Results p.u.	Deviation	Results Pa.s.	Deviation
7	65	-1.2461	0.407	0.34280
14	64	0.3115	0.409	-0.14691
21	64	0.3115	0.407	0.34280
23	65	-1.2461	0.409	-0.14691
31	64	0.3115	0.407	0.34280
40	64	0.3115	0.410	-0.39177
44	64	0.3115	0.407	0.34280
51	64	0.3115	0.408	0.09794
61	64	0.3115	0.410	-0.39177
66	64	0.3115	0.410	-0.39177
Average	64.2		0.4084	
<p>Water bath temperature checked = 25°C Thermosel temperature checked = 135°C</p> <p>Note: Deviation lies within test method requirements Sample numbers are randomly chosen by PTA Samples are homogeneous</p>				

APPENDIX C

Documentation

Instructions to Participants	C1
Results Sheet	C2

**PROFICIENCY TESTING AUSTRALIA
BITUMEN PROFICIENCY TESTING PROGRAM
ROUND 4
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:

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

PTA Sample B:

Dynamic Viscosity @ 60°C (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 Dynamic Viscosity @ 60°C test first.
5. Please report oven (RTFO) temperature applied during RTFOT. Measure RTFOT temperature using a calibrated thermometer because some ovens have misleading temperature readings.
6. 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.
7. 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.
8. The results for all determinations are to be recorded on the results sheet to the accuracy and reporting basis indicated.
9. Testing may commence as soon as the sample is received. All laboratories must return the results sheet no later than **17 December 2012** to:

Laura Galbraith
Proficiency Testing Australia
Phone: 02 9736 8397
Fax: 02 9743 6664
Email: laura.galbraith@pta.asn.au

**PROFICIENCY TESTING AUSTRALIA
BITUMEN PROFICIENCY TESTING PROGRAM
ROUND 4
RESULTS SHEET**

Lab Code

PTA
SAMPLE A

TEST (report to)	Result	MU ±	Method
Viscosity at 135.0°C Pa.s			
Density at 15.0C (bottle) Kg/m ³			
Penetration 25.0°C, 100g, 5s p.u. (0.1 mm)			
Softening Point, °C			

PTA
SAMPLE B

TEST (report to)	Result	MU ±	Method
Viscosity at 60.0°C Pa.s			
RTFOT Viscosity Change (including the actual value after treatment) %			
Measured RTFOT temperature, °C			

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

Also please report:

i) Mean 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 **17 December 2012** to:
 Laura Galbraith
 Proficiency Testing Australia
 Fax: 02 9743 6664 Email: laura.galbraith@pta.asn.au

- End of Report -